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HANDBOOK OF NATURAL PRODUCTS

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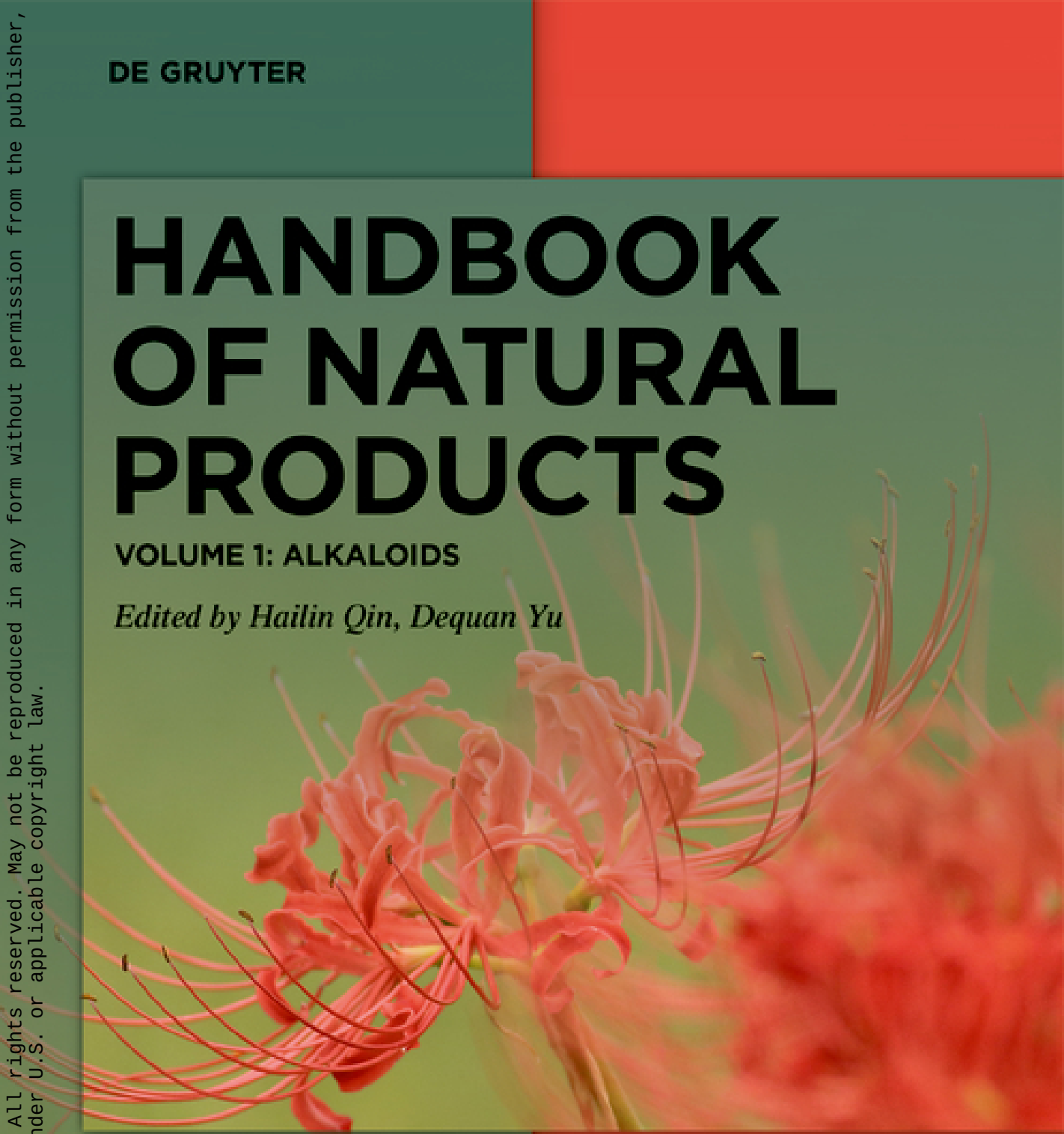

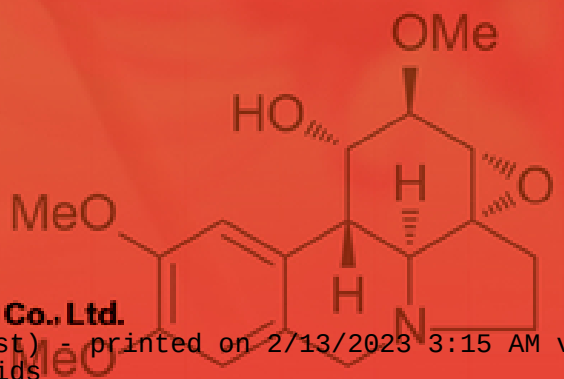

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Hailin Qin, Dequan Yu

^{1}H NMR Handbook of Natural Products

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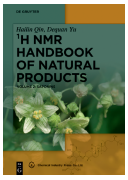

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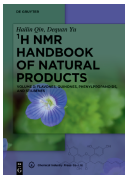

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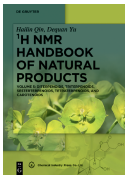

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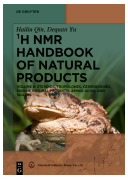

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Hailin Qin, Dequan Yu

^{1}H NMR Handbook of Natural Products

Volume 1: Alkaloids

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Authors
Prof. Dr. Hailin Qin
Institute of Materia Medica
Chinese Academy of Medical Sciences and
Peking Union Medical College
Beijing
China
qinhailin@imm.ac.cn

Prof. Dequan Yu
Academician of Chinese Academy of Engineering
Institute of Materia Medica
Chinese Academy of Medical Sciences and
Peking Union Medical College Beijing
China
dqyu@imm.ac.cn

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Preface

Proton Nuclear Magnetic Resonance (^{1}H NMR) is a science to study the relationship between the energy level transitions of hydrogen nuclei in organic compounds under the action of magnetic field and the chemical environment in molecules. The NMR data of organic compounds mainly include the chemical shift (resonance frequency), the number of hydrogen atoms, the peak shape (peak splitting), and the coupling constant of hydrogen functional groups, which are closely related to the structure of organic compounds. In the process of conducting structural identification of organic compounds, we deeply feel that although we have understood the basic principle of nuclear magnetic resonance phenomenon and the basic theory of chemical shift, peak integral area, spin coupling and spin splitting, coupling constant and so on, if there is no perceptual knowledge about nuclear magnetic resonance of various hydrogen functional groups in organic compounds, it is not enough to help us to analyze the structure of organic compounds skillfully. In addition, in the short 30 years from 1990s to now, with the theory and technology of NMR and computer science becoming more and more mature, the research on the structure of organic compounds has tended to be micro, fast, and accurate, which greatly shortens the research period of natural organic compounds. On the basis of the development and wide application of separation and purification technology of natural organic compounds represented by conventional chromatography and preparative liquid chromatography, a large number of natural organic compounds with relatively complex structures have been identified, and the NMR signals of these compounds have been fully assigned, thus accumulating a large number of spectral data of natural organic compounds. These data are very important for researchers engaged in the research of organic chemistry (including natural organic chemistry), because they not only help to simplify the structural identification of known compounds obtained in organic chemistry research, but also can be used as an important reference in the structural identification of new similar compounds and even novel compounds.

Natural organic chemistry is a basic subject to study the organic composition, structure, and change law of natural biological resources. It has been playing an important role in the research of organic chemistry, pharmaceutical chemistry, biochemistry, botany, and other disciplines, as well as the development of pharmaceutical industry and pesticide industry. For example, through the application of various natural organic compounds separation and purification methods and modern organic structure identification methods, tens of thousands of plant secondary metabolites have been identified in the field of phytochemistry, which not only greatly enriched the structure and types of organic compounds, but also proved that many components have significant physiological activities, or play an important role in the process of plant life. But, the chemical composition of many natural plant resources has not been fully elucidated, and the novel structure still attracts the majority of phytochemical

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researchers. On the other hand, at present, there are still many scientific questions, such as how these components are formed in plants, what is the relationship between genetic materials of related plants, and what is the overall level of secondary metabolites in different growth stages of plants. Although the number of natural organic compounds is relatively small in the vast number of organic compounds, due to the special source, structure, and function of natural organic compounds, especially in the discipline of natural pharmaceutical chemistry, the pharmaceutical properties of some natural organic compounds or their derivatives are much higher than those of other organic compounds, which makes natural organic compounds have been a very active research field since the beginning of organic chemistry.

Based on practical principles, this book embodies, as far as possible, various kinds of natural organic compounds that have been elucidated and completed the assignment of all ^{1}H NMR data in the field of natural organic chemistry. All the staffs responsible for compiling this manual are front-line researchers engaged in the research of Natural Pharmaceutical Chemistry (including extraction, separation, structural identification, structural modification, and total synthesis of natural organic compounds). This manual is based on consulting a large number of expertise journals on the research of natural organic compounds and sorting out the ^{1}H NMR data of natural organic compounds published in recent years according to the common classification method of natural organic compounds. Therefore, we sincerely thank the authors of the original documents included in the manual. The book consists of six volumes and sixteen chapters. The first chapter in the first volume is the introduction, which briefly introduces the development of natural organic compounds and the role of nuclear magnetic resonance spectroscopy in the identification of natural organic compounds. The remaining 15 chapters, which are scattered in Volumes 1 to 6, contain the ^{1}H NMR data of natural organic compounds published in recent years. The collected compounds include alkaloids, saponins, flavonoids, Quinones,Phenylpropanoids, stilbenes, monoterpenes, sesquiterpenes, Diterpenoids, Triterpenoids, and Sesterterpenoids, Tetraterpenoids, Carotenoids steroids, cycloheptatrone, cerebrosides, amino acids, and sugars from plants and marine organisms, a total of 17 categories of natural organic compounds. The contents include the name, molecular formula, ^{1}H NMR test solvent, structural formula, signal assignment, and literature sources of the compounds. The 1H NMR data include chemical shift, peak shape and coupling constant. Readers can refer to the structural formula to obtain the corresponding number of hydrogen atoms of functional groups containing hydrogen. It should be indicated that in the process of compiling this manual, in order to achieve the unification of data format and structure as much as possible, the authors have adjusted the atomic number of structural formula and the presented format of some structural simplification and stereochemistry in some original literatures. However, the atomic numbers of systematic nomenclature of some compounds are still in accordance with the original literature without corresponding changes. Readers can refer to the original literature for relevant contents.

The whole process of compiling this manual is strongly supported by editors at Chemical Industry Press Co., Ltd. Here, the authors would like to thank them in particular. The management departments of Institute of Materia Medica, Chinese Academy of medical sciences provide a superior environment and space for the compilation of this book. Ms. Li Zhihong and Ms. Yuan Guijie have provided strong support for the compilation of this manual from the beginning to the end, and we would also like to express our thanks here.

Due to the limited level of the author, short writing time, and heavy task, there are inevitably many shortcomings and errors in the book. We would like to ask the readers for criticism and correction, and give feedback on the problems found in the use process. The authors are very grateful.

Hailin Qin
September 12, 2020

A corresponding table of abbreviations of functional groups and structures

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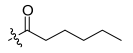
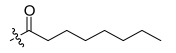
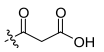
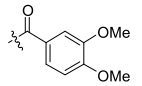
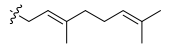
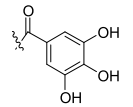
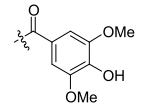
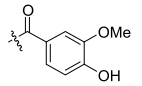
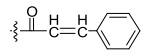
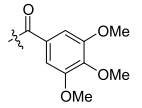
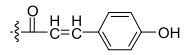
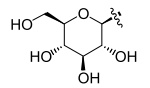
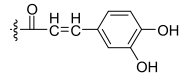
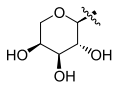
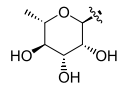
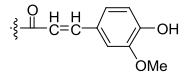
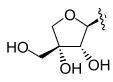
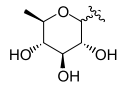
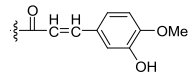
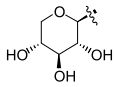
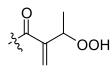
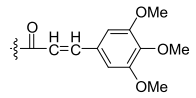
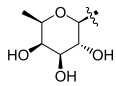
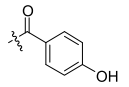
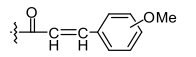
Ac		Val		Hex		Oct	
Prop		Mal		Vr		Ger	
But		MeBu		Gal		Hdmb	
^{i}But		Nic		Van		Cinn	
Sen		Pc		Tmb		Coum	
Ang		Mpc		Glc		Caff	
Tig		Ara		Rha		Fer	
Prenyl		Api		Qui		^{i}Fer	
Bn		Xyl		Hydrp		Tmc	
Bz		Fuc		*p*-HydrBz		MeOCinn	

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Contents

List of Participants

Editors in Chief: H.-L. Qin, D.-Q. Yu
Deputy Editors in Chief: A.-J. Deng, R.-Y. Yan, Q.-J. Zhang
Participants: J.-Q. Yu, Q. Zhou, Z.-Y. Hao, D. Liang, T. Sun, Z.-H. Zhang, Y. Yue, H.-L. Long, W. Zhao, D. Zhang

1 Introduction

1.1 Summary on natural organic compounds

Natural organic compounds (NOCs) are a series of organic compounds with structural diversity, which constitutes one branch of the most important substances in organic chemistry field. In a general concept, NOCs refer to all compounds existing natively in natural world. This generalization includes the organic constituents of all natural products, not only oil and coal, but also plants, animals, insects, marine organisms, and microorganisms, and the like. But, in general, the commonly accepted NOCs in the field of natural organic chemistry are those from plants, animals, insects, marine organisms, and microorganisms, especially the secondary metabolits and a little primary metabolits isolated from these biological organisms.

There are a great variety of NOCs with more or less, or even significantly, different natures and structures. Most NOCs contain carbon and hydrogen atoms as their major elements. In addition, on top of carbon and hydrogen, another common elements in NOCs are oxygen and nitrogen. By comparison, alkali metals, halogens, sulphur, and phosphorus, among a little others, are known to exist in a relatively small number of NOCs. With the advances of science and technology referring to exploration on natural products, the investigations on NOCs as one of the core areas in organic chemistry studies has made great contribution for promoting social development and human health in human history.

In everyday life and social production, NOCs has had a longstanding application. For example, they have been extensively used in prevention and treatment of diseases, pesticide chemicals, food, beverages, cosmetics, and toothpastes, among others. In the early 19th century, benzene, naphthalene, and anthracene, and some other aromatic compounds, were isolated from the byproducts from coke and coal gas production—coal tar, which in fact initiated the industrial application of coal tar. With the advance of organic structure theory, the cyclic structure theory of benzene was first put forward by Kekule, a German chemist, in 1865, significantly promoting the rapid development of dyestuff, medicine, and explosive, and the like, of organic syntheses industries. After long-term exploration by mankind in everyday life-practice and science research, natural organic chemistry has developed, from an earlier budding state of science exploring the composition and exploitation of natural products, into a subject of important practical significance, which is currently exploring the secret of nature, enriching the precious natural learnings reaped by mankind in the long-term social practice, and developing harmoniously with natural world.

The discovery and structural investigations of NOCs are significantly associated with the practical demand of human society development. In the earlier stage of the development of organic chemistry, the functional natural products were the major

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subjects of study, with the isolation of the functional organic compounds from natural products being appreciated mainly. For examples, the dyestuffs, medicines, and spices were isolated from plants. In the late 18th century, a Swedish chemist, Carl Wilhelm Scheele, isolated pure tartaric acid from grape juice, the by-product of brewing industry. In the isolation, Scheele took advantage of the hydrophobic physical property of calcium salts and lead salts of organic acids. The organic acid was precipitated from the juice as the salts, and the pure tartaric acid was obtained again after acidizing the precipitation with inorganic acids. With this case as a representative, the extensive isolation and purification of NOCs took off, citric acid being isolated from lemon, malic acid from apple, lactic acid from yoghurt, uric acid from urine, and gallic acid from gallnut. Many other kinds of NOCs were also isolated from natural products in the early stage of the development of natural organic chemistry, such as the isolation of morphine from opium in the 19th century and quinine from *Cinchona ledgeriana* (Howard) Moens ex Trim, as well as the isolation of emetine, strychnine, nicotine, atropine, cocaine, carotene, ephedrine, berberine, colchicine, eserine, rutin, strophanthin, and digitoxin, among others. Of these compounds, morphine was known as the first isolated alkaloid. In the earlier stage of time, the active NOCs which were isolated based on the learning of humanity on the bioactivities of natural products laid a foundation for the development of chemical drugs. Especially, based on the exploration on the chemical structures of NOCs, not only were the functional NOCs directly applied to drugs preparation, but also many drugs were acquired via artificial synthesis and structural modification. For instance, an Britain scientist, Fleming, discoveried an important active NOC, penicillin, in 1929, which was used to treat bacterial infections by humans and is regarded as the representative of NOCs in drugs. And the inventions of a series of topical anesthetics, such as benzocaine, procaine, and tetracaine, through the chemically structural modification of cocaine were also typical application of NOCs in drugs.

During the fad and ongoing process of studying the chemical structures of NOCs to search for hyper-effective, hypo-toxic or non-toxic, and high-quality new molecular entity to develop innovative drugs, the chemical structure modifying of lead compounds are conducted frequently based on the bioactive investigation. This is because of the reality that some, or even many, of the imperfections in druggability of considered lead compounds abounds, such as poor bioactivities failing to apply to treat diseases, structural instabilities, obvious toxicity to subjects, lack of selectivities, and undesirability of pharmacokinetic properties. Superior drugs are often developed through this common process which is usually called structural optimization of lead compounds. For years, based on the extensive studies on the structure study and optimization of NOCs performed by numerous investigators worldwide, many kinds of structures of NOCs have been enriched and developed to a large extent. Thus, close inner link exists between NOCs and synthetic organic compounds (SOCs).

From the beginning of the 20th century up to now, over more than 100 years, the development and interinfiltration of physics, organic chemistry, and analytic chem-

istry, among others, significantly contributed to the study on NOCs. Of the scientists of the period, Russian botanist, Tsvet (1872–1919), initiated isolation technology of chromatography in 1906; Austrian chemist, F. Pregl (1869–1930), developed the microdetermintion of carbon and hydrogen elements in organic compounds based on the elements analyses of organic compounds in 1916. These events are the important representatives of the development of modern separation and structural identification techniques for organic compounds. From 1930s through 1950, instrumental analysis technologies of optical rotatory dispersion (ORD), ultraviolet-visible absorption spectrometry (UV-VIS), infrared spectroscopy (IR), mass spectrometry (MS), and nuclear magnatic resonance (NMR) were developed in succession. These technologies, along with the advances in the isolation and purification technologies of NOCs, enabled the structure identifications of NOCs in level of milligrams or even lower. Especially, as of the 1980s, the development of structural identification technologies of organic compounds, represented by the development of new NMR technologies, made the structural research more micro, fast, and accurate, the research cycle of NOCs being significantly shortened. Ever since 2000, compared with the earlier academic papers, more and more new or novel structures of NOCs were clarified completely, and the NMR signals of structurally more complicated NOCs were assigned wholly and accurately, leadint to more NOCs published annually in various academic journals on natural product researches and further accumulation of spectroscopic data of ever more NOCs. Thus, based on the works of 'Handbook of Analytical Chemistry, Nuclear Magnetic Resonance Spectroscopy Analysis (Seventh fascicle, Second edition)' by academician Dequan Yu and professor Junshan Yang published by Chemical Industry Press in 1999, it will make a difference significantly to further supplement and perfect the spectroscopic data of NOCs.

Except for the conventional classification method mainly representing the sources of NOCs, the method related to carbon skeletons and functional groups is also a generally accepted classification method, which takes into account the biosynthetic pathways and properties of NOCs. In addition, the latter also aims at the goal of representing the structures and physicochemical properties of NOCs. The important NOCs of secondary metabolites reported in recent years mainly belong in alkaloids, saponins, flavonoids, terpenoids, phenylpropanoids, stilbenes, quinones, and steroids, etc., from plant, animal, and marine sources.

1.2 Proton nuclear magnetic resonance spectroscopy implicated in structures of NOCs

Ever since the mid-20th centure, the means of UV-VIS, IR, MS, and NMR as morden instrumental methods have gradually replaced the conventional chemical methods for the structures identifications of NOCs, as well as for the investigations on the details of configurations and conformations. The applications of these methods signifi-

cantly advances the development of organic chemistry study, which also represents one of the modern technology features for structure identification of organic compounds. Of these technologies, UV-VIS offers the informations of chromophores, such as conjugated structures, *via* measuring mainly the absorption spectrum of organic compounds between the optical wavelengths of 200 nm and 400 nm. IR offers the recognizable informations on functional groups containing specific chemical bonds *via* measuring the absorption spectrum of organic compounds between the optical wave numbers of 4000 cm^{-1} and 400 cm^{-1}. MS offers the informations of molecular weights and molecular formulae *via* making organic compounds ionized and fragmented and measuring the mass-to-charge ratios of ions. The informations of structure fragments are also learned through analyzing the fragmentation routes of organic compounds in the MS experiments. By comparison, NMR offers more abundant and corroboratory informations *via* measuring the resonance signals of magnatic nuclei of organic compounds under different magnatic field intensities, including the numbers and chemical environments of protons and carbon-13 nuclei in molecules, species of functional groups, the interconnection order of atoms, and the mode of spatial interaction, etc. In addition, the accurate structural identificantions and signal assignments can be achieved by extensively applying various advanced one- and two-dimentional NMR technologies to the target organic compounds. With respect to the proton-NMR (^{1}H NMR) technique, the available and very useful data mainly include integral area of spectral peaks, chemical shifts (δ values), spin coupling and spin splitting features, and coupling constants. These data are related to the numbers, chemical enviroments, interconnection orders, and the space interaction modes of hydrogens in the compounds, respectively. As indicated above, in general, the hydrogen numbers can be obtained directly from the integral operation of each proton signal in the process of ^{1}H NMR measurement. Thus, only the concepts of chemical shifts, spin coupling and spin splitting, and coupling constants are briefly introduced below.

1.2.1 Definition of chemical shift

The theoretical basis of the NMR technology is nuclear physics. NMR is an analytical technique based on the theory that atomic nuclei with magnetic moments, such as ^{1}H, ^{13}C, ^{15}N, ^{19}F, and ^{31}P, when placed in the environment of external magnetic field and affected by the change of magnetic field, can produce interlevel transitions of energy.

When the atomic nuclei with magnetic moment is placed in the environment of external magnetic field H_0, the energy-level splitting phenomenon is formed and the nuclei are actually distributed over two or more energy levels. At the same time, these nuclei have a certain precession angular velocity ω or precession frequency υ. If H_0 is changed, then the ω and υ will change accordingly. If a small alternating magnetic field H_1 is added from the vertical direction of H_0 and the frequency of this alternating

magnetic field is set as f, then, as the situation of $f = \upsilon$ was achieved, according to the concept of resonance in physics, resonances from these two vibrations occurs, making the energy-low protons to absorb the energy of alternating magnetic field H_1 and jump to the high energy state. This is called NMR.

In organic compounds, each hydrogen nucleus isn't separated. In the environment of external magnetic field, the induced magnetic field due to the electrical effects of the surrounding atoms or groups of the considered proton would produce positive or negative shielding effect to the external magnetic field intensity received by the given proton. Thus, under the given irradiation frequency, all the actual resonance frequencies of given protons are influenced by their chemical environments. That is to say that, in the molecules of organic compounds, hydrogen nuclei in different chemical environments would produce NMR phenomena under different magnetic field intensities. The phenomenon that protons, or other kinds of magnetic nuclei, duo to the different chemical environment in the molecule, show their different resonance signals under different magnetic field intensities is call chemical shift.

1.2.2 Manifestation and influencing factors of chemical shift

Compared to absolute resonance frequencies, the differences between the chemical shifts, i. e., the differences of resonance magnetic field intensities, of any two magnetic nuclei in different chemical environments are paltry. In terms of protons, it is around the range of 10×10^{-6}. Thus, it is very hard to accurately measure the absolute resonance frequencies of each magnetic nuclei. In the practical application, the chemical shifts are expressed using relative values. The resonance peak of a certain standard substance is used as original point to measure the distance between each peak of the sample and the original point. The relative values are easier to measure, with the accuracy of less than 1 Hz being achieveable.

1.2.2.1 Chemical shift values

In general, the values of chemical shifts are expressed as the parameter δ. The specific concept can be expressed by the following formulas:

$$\delta = \frac{\Delta H}{H_R} \times 10^6 = \frac{H_R - H_S}{H_R} \times 10^6$$

or

$$\delta = \frac{\Delta \nu}{\nu_R} \times 10^6 = \frac{\nu_S - \nu_R}{\nu_R} \times 10^6$$

where, H_R is the magnetic field intensity for resonance of standard substance; H_S is the magnetic field intensity for resonance of tested sample; υ_R is the resonance frequency of standard substance; and υ_S is the resonance frequency of tested sample.

The differences between ΔH and H_R, and $\Delta\upsilon$ and υ_R, are only a few parts per million, thus, in order to make the δ values easier to read and write, it is multiplied by 10^6.

1.2.2.2 Shielding effect

In the external magnetic field environment, the electron motion of a molecule circulating around the nuclei on a plane perpendicular to the external magnetic field would produce an induced magnetic field opposite or identical to the direction of the external magnetic field in a specific region. This induced magnetic field would affect the intensity of the external magnetic field in a specific area, which is called 'shielding effect' in the NMR spectroscopy. Shielding effect is one of the main contrbutors affecting the chemical shifts of hydrogen nuclei on different groups in molecules. In the ^{1}H NMR experiment, shielding effect includes local shielding effect and remote shielding effect.

① Local shielding effect

Some structural elements can influence the electron cloud density of the bonding electrons outside the considered hydrogen nuclei. The resulting shielding effect due to this situation is called the local shielding effect. Local shielding effect has its own rules. If one or more electron-withdrawing chemical groups exist near a hydrogen nucleus, then the electron cloud density around it decreases, the shielding effect is also reduced and the chemical shift moves to lower field, i. e., δ value increases. If one or more electron-donating chemical groups exist near a hydrogen nucleus, then the electron cloud density around it increases, the shielding effect is also increased and the chemical shift moves to higher field, i. e., δ value decreases.

② Remote shielding effect

The impact of shielding effects produced by the motion of electron(s) outside the nuclei of other atoms or atomic groups around the considered hydrogen nuclei is called remote shielding effect. One of the features of remote shielding effects is its directionality. The magnitude and the positive and negative values of remote shielding effect depend on distance and direction, i. e., on magnetic anisotropy of atoms or atomic groups. Thus, hydrogen nuclei in different orientations of the atoms or atomic groups with remote shielding effect possess different rules in chemical shift changes.

1.2.2.3 Hydrogen bond and chemical shift

Chemical shifts of hydrogen nuclei are very sensitive to hydrogen bonds. The change principle of chemical shift is mainly related to the strength of hydrogen bond and the nature of hydrogen bond donor. In the case of strong hydrogen bond, the electrostatic effect of the donor atoms is the main contributor affecting the chemical shift value. In most cases, the formation of intramolecular or intermolecular hydrogen bond would make the hydrogen nucleus being deshielded, and thus make its chemical shifts mov-

ing downfield. The greater the hydrogen bond strength, the more obvious the change of chemical shift.

1.2.2.4 Solvent effect

When the same sample is dissolved in different solvents and their ^{1}H NMR spectra are recorded, the obtained chemical shifts in different solvents are also different. The effect of changing chemical shifts due to different solvents is called solvent effect. There are many factors causing solvent effect, such as the formation of molecular complexes or intermolecular hydrogen bond between solvent and sample molecules. Solvent effect is an important factor. When reporting the chemical shift values, the used solvent must be noted.

At present, the theoretical study on chemical shift in the NMR spectroscopy has gone deeply. And in principle, theoretical calculations can be done. But, in practice, it is still hard to provide an accurate calculation value in advance. That is to say that, under the case of only understanding some theoretical knowledges but having no perceptual concept of chemical shift, it's not enough for helping us analyze the NMR spectra skillfully. Thus, for the structure identifications of NOCs, it is very important to learn the empirical data and empirical calculation formulas of chemical shifts of the common hydrogenated functional groups. On the other hand, in the research process of natural organic chemistry, the accurate structural identification can be achieved generally through comparing the NMR data of condidered compounds wih those of similar structures, in speediness and convenience. Just like the situation that every characteristic group has a certain absorption peak in the IR spectroscopy, the NMR chemical shifts of protons on various groups has been summarized in many literatures and monographs by generalizing and analyzing the ^{1}H NMR data of a large number of organic compounds [1, 2, 3].

1.2.3 Spin-spin coupling and spin-spin splitting

1.2.3.1 Defination of spin-spin coupling and spin-spin splitting

As has been noted above, the chemical shifts are related to the chemical environment around the considered magnetic nuclei (chemical environment is also called electronic environment). The shielding effect of electrons around the nucleus is the source of the chemical shift. But, in organic compounds, except for electrons around every nucleus, positively charged nuclei abound. There is also interference effect among these nuclei of spin phenomena in the external magnetic field. This interference does not affect the chemical shift of magnetic nuclei, but it has an important inffluence on the peaktype of the NMR spectra. In the NMR spectroscopy, the mutual interference among magnetic nuclei with spin phenomena is called spin-spin coupling, while, the phenomenon of increasing spectral lines of the given signals due to spin-spin coupling is called spin-spin splitting.

1.2.3.2 $N + 1$ rule

In general, spin-spin splitting follows the $n + 1$ rule, that is, when a certain hydrogen atom in a chemical functional group have n adjacent equibalent hydrogens, it will display $n+1$ multiple peaks. And a specific strength relationship of the multiple peaks exists which is listed in Table 1.

Table 1: $N + 1$ rule of spin-spin splitting and strength relationship of the multiple peaks.

Numbers of neighboring equivalent hydrogens (n)	Numbers of split peaks ($n + 1$)	Strength ratio of split peaks [Coefficient ratio of expanded form of $(a + b)^n$]
0	Singlet (s)	1
1	Doublet (d)	1 : 1
2	Triplet (t)	1 : 2 : 1
3	Quartet (q)	1 : 3 : 3 : 1
4	Quintet (quint)	1 : 4 : 6 : 4 : 1
5	Sextet (sext)	1 : 5 : 10 : 10 : 5 : 1
6	Septet (sept)	1 : 6 : 15 : 20 : 15 : 6 : 1

It should be indicated that the phenomenon of spin-spin splitting is not exhibited between those hydrogen nuclei exhibiting the same chemical shift due to chemical equivalence. But, if these adjacent hydrogen nuclei are in different chemical environments, for example, the number of hydrogen nuclei in one environment is n, while the number of hydrogen nuclei in another environment is $n', \ldots$, then, the signal of the considered hydrogen nucleus, which is affected by all those magnetic nuclei, would exhibit the multiple peaks of $(n+1)(n'+1)\ldots$. If the coupling constants of those adjacent hydrogen nuclei in different chemical environments to the considered hydrogen nucleus are the same by chance, then, the total number of adjacent hydrogen nuclei in these different chemical environments can be regarded as n, and the number of splitting peaks can be still calculated by the $n + 1$ rule. In the NMR spectroscopy, the spin-spin coupling that follows the $n + 1$ rule is called first-order coupling.

The first-order coupling of the ^{1}H NMR spectrum needs to meet certain conditions, i. e., the difference of chemical shift ($\Delta\delta$) between coupled spin-nuclei should be much larger than the values of their coupling constants (J). In general, the value of $\Delta\delta/J$ should be more than $6 \sim 10$. But, in practice, many structures which do not meet the above conditions also abound. Under these situations, the spin-spin coupling of these magnetic nuclei will not meet the $n + 1$ rule. This type of coupling which does not meet the $n + 1$ rule is called second-order coupling. The analysis of second-order coupling is more complicated. A certain complicated calculations must be conducted to accurately assign the chemical shifts and coupling constants. In practice, A received saying on this situation is to simply say that the coupled hydrogen nuclei split into multiple peaks (m).

1.2.4 Coupling constants

In a spin-spin coupling system, the signals of the NMR spectrum split to form doublet or multiplet. The distance between spectral lines caused by splitting reflects the magnitude of interference of magnetic nuclei, which is called coupling constant. Coupling constant is denoted by the letter J.

The interactions and the magnitudes of coupling constants between hydrogen nuclei are related to many factors, such as the number of chemical bonds, the connection order, and the spatial mode of action between these magnetic nuclei. Thus, many structural informations can be obtained from coupling constants. In general, according to the difference of the number of chemical bonds between the hydrogens interfering each other, the coupling constants are divided into geminal coupling constant, vicinal coupling constant, and long range coupling constants.

1.2.4.1 Geminal coupling constant

When two hydrogen nuclei are on the same carbon atom, i. e., the number of chemical bonds between these two hydrogen nuclei are two (H–C–H), the coupling constant is simply called germinal coupling constant. The variation range of the germinal coupling constant is very large, closely related to structures. Especially, the germinal coupling constants of most sp^3 hydrid group are significantly different from that of sp^2 hydrid group of terminal double bond. The specific numerical values of most coupling constats corresponding to common chemical functional groups can be consulted in relevant monographs [1, 2, 3].

The main factors affecting the magnitude of the germinal coupling constants include the carbon-hydrogen bond angle, electronegativity of connected groups, and π bond in ortho-position(s).

1.2.4.2 Vicinal coupling constant

The mutual coupling constant of protons with three chemical bonds apart is called vicinal coupling constant. The variation range of the vicinal coupling constant is also very large. When the related hydrogen-containing functional group can rotate freely, the vicinal coupling constant is about 7 Hz. When the conformation of the compound is fixed, the vicinal coupling constants can vary in the range of 0–18 Hz due to different structures. The main factors affecting the magnitude of the vicinal coupling constant is the magnitude of torsion angle, which represents the angle between the two respective H–C–C planes of the two explored hydrogen nuclei. Other factors, such as electronegativity of substituents, bond length, and bond angle, etc., have less influence [1, 2, 3].

The vicinal coupling constants are encountered frequently in practical work. Many structural problems, especially some stereochemistry structures, can be solved by using the vicinal coupling constants.

1.2.4.3 Long-range coupling constant

In most cases, when the two explored hydrogens are spaced by more than three chemical bonds, the coupling constant is zero. But, for some special structures, when this situation occurs, spin-spin coupling between the two explored hydrogen nuclei can still be observed. In general, the coupling constants between the two hydrogen nuclei spaced out by more than three chemical bonds are called long-range coupling constants. In NOCs, several common types of structures in which long-range couplings are sometimes observed are as follows.

① Coupling of allyl motif (H_a–C=C–C–H_b): The coupling constant range between H_a and H_b is from 0 to 3 Hz.

② Coupling of homoallyl motif (H_a–C–C=C–C–H_b): The coupling constant range between H_a and H_b is from 0 to 4 Hz.

③ Coupling between aromatic protons and the α protons from side chains: In aromatic compounds, there are spin-spin coupling between the methyl groups on aromatic rings and their ortho-hydrogens, the coupling constant range is around 0.6–0.9 Hz. There are also spin-spin coupling between the methyl groups on aromatic rings and their para-hydrogens, the coupling constant range is around 0.5–0.6 Hz.

In addition, in heterocyclic aromatic compounds, there are spin-spin coupling between the methyl groups on heteroaromatic rings and their ortho-hydrogens, the coupling constant range is around 0.5–1.3 Hz.

There are spin-spin coupling between meta-hydrogens of benzene, the coupling constant range is around 1–3 Hz.

The coupling constants of protons from some other specific functional groups, such as the vicinal coupling constants of aromatic rings and heteroaromatic rings, as well as the coupling constants between protons and other non-hydrogen magnetic nuclei, can be consulted in relevant monographs [1, 2, 3].

Bibliography

[1] Liang XT. Nuclear Magnetic Resonance–Analysis and Application of High Resolution Proton Nuclear Magnetic Resonance. Science Press, Beijing, 1976.

[2] Zhao T-Z. Proton Nuclear Magnetic Resonance. Peking University Press, Beijing, 1983.

[3] Yu DQ, Yang J-S. Handbook of Analytical Chemistry, Nuclear Magnetic Resonance Spectroscopy Analysis (Seventh fascicle, Second edition). Chemical Industru Press, Beijing, 1999.

2 Alkaloids

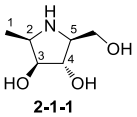
2.1 Pyrrolidine alkaloids

2.1.1 Polyhydroxylated pyrrolidine alkaloids

Table 2-1-1: Cos, MFs, and TSs of polyhydroxylated pyrrolidine alkaloids **2-1-1**~**2-1-19**[①].

No.	Compounds	MFs	Test solvents	References
2-1-1	2,5-imino-1,2,5-trideoxy-D-glucitol	$C_6H_{13}NO_3$	D_2O	[1]
2-1-2	2,5-dideoxy-2,5-imino-D-fucitol	$C_6H_{13}NO_3$	D_2O	[1]
2-1-3	2,5-imino-1,2,5-trideoxy-D-mannitol	$C_6H_{13}NO_3$	D_2O	[2]
2-1-4	7-deoxy-homoDMDP	$C_7H_{15}NO_4$	D_2O	[3]
2-1-5	2,5-dideoxy-2,5-imino-glycero-D-galacto-heptitol	$C_7H_{15}NO_5$	D_2O	[3]
2-1-6	homoDMDP	$C_7H_{15}NO_5$	D_2O	[4]
2-1-7	6-deoxyhomoDMDP	$C_7H_{15}NO_4$	D_2O	[5]
2-1-8	2,5-imino-2,5,6-trideoxy-D-gulo-heptitol	$C_7H_{15}NO_4$	D_2O	[5]
2-1-9	*α*-1-*C*-(2-hydroxypropyl)-1,4-dideoxy-1,4-imino-D-arabinitol	$C_8H_{17}NO_4$	D_2O	[6]
2-1-10	*α*-1-*C*-(1,2-dihydroxypropyl)-1,4-dideoxy-1,4-imino-D-arabinitol	$C_8H_{17}NO_5$	D_2O	[6]
2-1-11	2,5-dideoxy-2,5-imino-D-glucitol	$C_6H_{13}NO_4$	D_2O	[7]
2-1-12	1,4-dideoxy-1,4-imino-(hydroxyethyliminiumyl)-D-arabinitol	$C_7H_{15}NO_4$	D_2O	[1]
2-1-13	1,4-dideoxy-1,4-imino-D-arabinitol	$C_5H_{11}NO_3$	D_2O	[8]
2-1-14	1,4-dideoxy-1,4-imino-D-ribitol	$C_5H_{11}NO_3$	D_2O	[9]
2-1-15	6-*C*-butyl-DMDP	$C_{10}H_{21}NO_4$	CD_3OD	[10]
2-1-16	4-*O*-[*β*-D-mannopyranosyl-(1→4)-*β*-D-mannopyranosyl]-6-deoxy-homoDMDP	$C_{19}H_{35}NO_{14}$	D_2O	[3]
2-1-17	1,4-dideoxy-1,4-imino-(5-*O*-*β*-D-glucopyranosyl)-D-arabinitol	$C_{11}H_{21}NO_8$	D_2O	[1]
2-1-18	3-*O*-*β*-D-glucopyranosyl-DMDP	$C_{12}H_{23}NO_9$	D_2O	[7]
2-1-19	homoDMDP-7-*O*-*β*-D-xylopyranoside	$C_{12}H_{23}NO_9$	D_2O	[11]

[①] Co: compound, MF: molecular formula, TS: test solvent.



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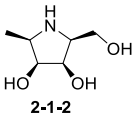

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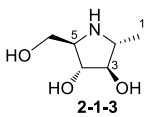

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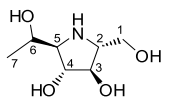

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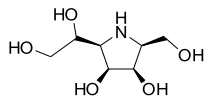

2-1-5

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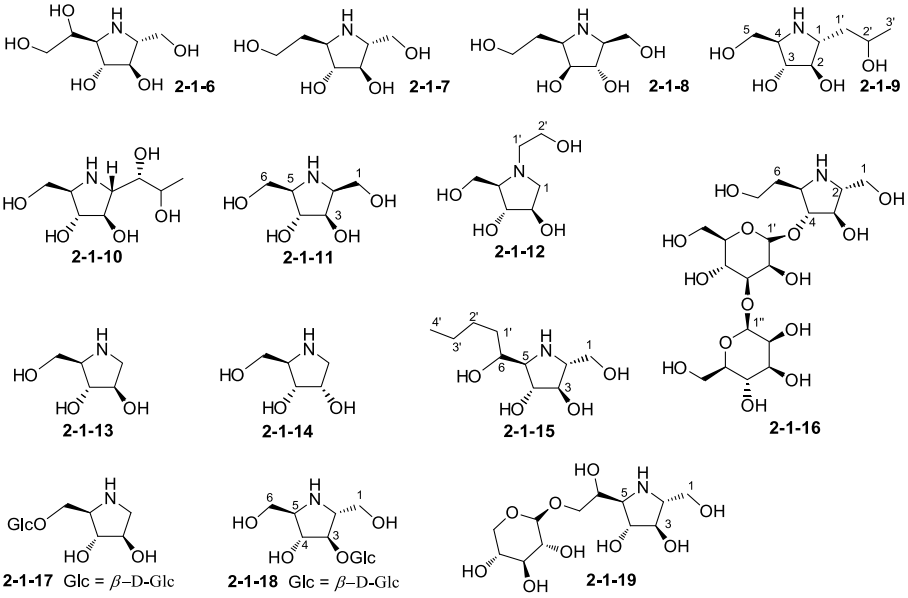


Table 2-1-2: ^{1}H NMR spectroscopic data of polyhydroxylated pyrrolidine alkaloids **2-1-1**~**2-1-4**.

H	2-1-1	2-1-2	2-1-3	2-1-4
1	1.16 d(6.8)	1.19 d(6.8)	1.20 d(6.7)	3.64 dd(6.1, 11.7) 3.72 dd(4.4, 11.7)
2	3.32 dq(4.4, 6.8)	3.53 dq(3.7, 6.8)	2.97 dq(8.3, 6.7)	3.04 ddd(4.4, 6.1, 7.6)
3	3.96 dd(2.2, 4.4)	4.02 dd(2.0, 3.7)	3.62 dd(8.3, 7.0)	3.86 dd(6.8, 7.6)
4	3.88 dd(2.2, 4.9)	4.27 dd(2.0, 4.6)	3.83 dd(7.0, 7.0)	4.03 dd(6.8, 7.1)
5	3.01 ddd(4.9, 4.9, 6.4)	3.60 ddd(4.6, 6.4, 7.1)	3.19 dt(7.0, 5.5, 7.0)	2.91 dd(5.8, 7.1)
6	3.68 dd(6.4, 11.5) 3.76 dd(4.9, 11.5)	3.69 dd(7.1, 11.2) 3.81 dd(6.4, 11.2)	3.68 dd(5.5, 11.0) 3.63 dd(7.0, 11.0)	3.89 dq(5.8, 6.3)
7				1.24 d(6.3)

Table 2-1-3: ^{1}H NMR spectroscopic data of polyhydroxylated pyrrolidine alkaloids **2-1-5**~**2-1-8**.

H	2-1-5	2-1-6	2-1-7	2-1-8
1	3.75 dd(5.9, 11.4) 3.79 dd(5.0, 11.4)	3.74 dd(11.8, 3.8) 3.69 dd(11.9, 6.1)	3.65 dd(6.1, 11.7) 3.71 dd(4.4, 11.7)	3.71 dd(6.4, 11.7) 3.78 dd(5.1, 11.7)
2	3.13 ddd(4.6, 5.0, 5.9)	3.16 br	3.05 ddd(4.4, 6.1, 7.3)	3.07 ddd(4.4, 5.1, 6.4)
3	3.94 dd(2.3, 4.6)	3.91 t(7.3)	3.84 t(7.3)	3.92 dd(1.7, 4.4)
4	4.11 dd(2.3, 5.0)	4.12 t(7.3)	3.74 dd(7.3, 7.8)	4.03 dd(1.7, 4.1)
5	3.29 dd(5.0, 6.9)	3.13 br	3.01 ddd(4.9, 7.8, 8.8)	3.34 ddd(4.1, 6.6, 7.8)
6	3.96 ddd(3.7, 6.9, 6.9)	3.83 br	1.71 m, 1.93 m	1.79 m, 1.90 m
7	3.62 dd(6.9, 11.9) 3.75 dd(3.7, 11.9)	3.74 dd(11.8, 3.8) 3.64 dd(11.7, 6.9)	3.64~3.77 m	3.72

Table 2-1-4: ^{1}H NMR spectroscopic data of polyhydroxylated pyrrolidine alkaloids **2-1-9**, **2-1-10** and **2-1-15**.

H	2-1-9	2-1-10	2-1-15
1	3.06 ddd(8.7, 7.8, 5.0)	3.15 dd(7.3, 6.0)	3.68 dd(6.4, 11.0) 3.78 dd(5.9, 11.0)
2	3.75 dd(7.8, 6.9)	4.10 dd(7.3, 6.0)	3.25 ddd(4.2, 5.9, 6.4)
3	3.85 dd(7.3, 6.9)	3.88 dd(7.3, 6.4)	4.07 dd(4.2, 4.9)
4	3.10 ddd(7.3, 6.4, 4.6)	3.08 ddd(6.4, 6.0, 4.6)	4.13 dd(4.9, 6.8)
5	3.73 dd(11.9, 4.6) 3.66 dd(11.9, 6.4)	3.73 dd(11.9, 4.6) 3.65 dd(11.9, 6.0)	3.05 dd(4.2, 6.8)
6			3.63 dt(4.2, 8.3)
1′	1.81 ddd(14.2, 6.4, 5.0) 1.70 ddd(14.2, 8.7, 6.9)	3.57 dd(6.4, 6.0)	1.48~1.66 m
2′	3.98 dq(6.9, 6.4)	3.91 q(6.4)	1.30~1.43 m 1.48~1.66 m
3′	1.21 d(6.4)	1.22 d(6.4)	1.30~1.43 m
4′			0.94 t(7.3)

Table 2-1-5: ^{1}H NMR spectroscopic data of polyhydroxylated pyrrolidine alkaloids **2-1-11**~**2-1-14**.

H	2-1-11	2-1-12	2-1-13	2-1-14
1	3.71 dd(4.9, 11.3) 3.82 dd(6.0, 11.3)	2.82 dd(5.9, 11.2) 3.08 br dd(2.2, 11.2)	2.86 dd(12.1, 4.0) 3.14 dd(12.1, 5.8)	2.88 dd(12.4, 3.7) 3.21 dd(12.4, 5.1)
2	3.38 ddd(2.9, 4.9, 6.0)	4.13 ddd(2.2, 2.9, 5.9)	4.16 ddd(4.0, 5.8, 3.7)	4.17 dt(3.7, 5.1)
3	4.14 dd(2.9, 5.2)	3.95 br dd(2.9, 5.2)	3.85 dd(3.7, 5.5)	3.91 dd(5.1, 7.4)
4	3.90 dd(2.9, 5.2)	2.61 m	3.01 ddd(5.5, 6.3, 4.8)	3.13 ddd(7.4, 6.2, 4.4)
5	3.08 ddd(2.9, 4.4, 4.9)	3.69~3.73	3.67 dd(6.3, 11.7) 3.75 dd(4.8, 11.7)	3.65 dd(6.2, 11.8) 3.77 dd(4.4, 11.8)
6	3.69 dd(4.4, 11.5) 3.77 dd(4.9, 11.5)			
1′		2.58 m, 3.02 m		
2′		3.69~3.73		

Table 2-1-6: ^{1}H NMR spectroscopic data of polyhydroxylated pyrrolidine alkaloids **2-1-16**~**2-1-19**.

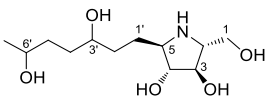
H	2-1-16	2-1-17	2-1-18	2-1-19
1	3.67 dd(6.3, 12.0) 3.73 dd(7.8, 11.9)	2.97 br dd(3.9, 12.5) 3.26 dd(5.6, 12.5)	3.75~3.80	3.65 dd(5.9, 11.7) 3.73 dd(4.4, 11.7)
2	3.14 ddd(4.7, 6.1, 6.7)	4.21 ddd(3.9, 3.9, 5.6)	3.34 m	3.04~3.08
3	4.06 dd(5.6, 6.7)	3.97 br dd(3.9, 4.9)	4.10 t(5.6)	3.86 dd(6.8, 7.8)
4	3.91 dd(5.6, 6.9)	3.26~3.30 m	4.13 t(5.6)	4.10 dd(6.8, 7.4)
5	3.26 m	3.82 dd(7.6, 10.7) 4.13 dd(4.4, 10.7)	3.21 m	3.04~3.08
6	1.80 m 1.92 m		3.74 dd(6.1, 11.7) 3.82 dd(4.4, 11.7)	3.93 ddd(3.2, 5.6, 6.8)

Table 2-1-6 (continued)

H	2-1-16	2-1-17	2-1-18	2-1-19
7	3.68~3.75			3.71 dd(7.1, 11.0) 4.02 dd(3.2, 11.0)
1′	4.8 (ov)	4.51 d(7.8)	4.61 d(8.1)	4.45 d(7.8)
2′	4.12 dd(0.9, 2.8)	3.33 dd(7.8, 9.3)	3.40 dd(8.1, 9.3)	3.32 dd(7.8, 9.3)
3′	3.82 dd(2.8, 9.3)	3.52 dd(9.0, 9.3)	3.59 t(9.3)	3.46 t(9.3)
4′	3.80 dd(9.3, 9.3)	3.40 dd(9.0, 9.8)	3.47 dd(9.3, 9.8)	3.63 ddd(5.4, 9.3, 10.5)
5′	3.57 m	3.49 ddd(2.2, 5.9, 9.8)	3.58 ddd(2.6, 6.6, 9.8)	ax 3.33 dd(10.5, 11.7) eq 3.97 dd(5.4, 11.7)
6′	3.74 dd(6.9, 11.9) 3.95 dd(2.3, 11.9)	3.74 dd(5.9, 12.5) 3.93 dd(2.2, 12.5)	3.78 dd(6.6, 12.0) 4.03 dd(2.6, 12.0)	
1″	4.73 d(0.9)			
2″	4.07 dd(0.9, 3.2)			
3″	3.66 dd(3.2, 9.7)			
4″	3.58 dd(9.7, 9.7)			
5″	3.45 ddd(2.3, 6.9, 9.7)			
6″	3.96 dd(6.9, 11.9) 3.96 dd(2.3, 11.9)			

Table 2-1-7: Cos, MFs, and TSs of polyhydroxylated pyrrolidine alkaloids **2-1-20**~**2-1-41**.

No.	Compounds	MFs	Test solvents	References
2-1-20	6-deoxy-6-*C*-(2,5-dihydroxyhexyl)-DMDP	$C_{12}H_{25}NO_5$	D_2O	[11]
2-1-21	broussonetine S	$C_{18}H_{37}NO_6$	C_5D_5N	[12]
2-1-22	broussonetine T	$C_{18}H_{35}NO_7$	C_5D_5N	[12]
2-1-23	broussonetine U	$C_{18}H_{33}NO_5$	C_5D_5N	[12]
2-1-24	broussonetine V	$C_{18}H_{33}NO_5$	C_5D_5N	[12]
2-1-25	broussonetine M	$C_{18}H_{37}NO_5$	C_5D_5N	[13]
2-1-26	broussonetine O	$C_{18}H_{33}NO_5$	C_5D_5N	[13]
2-1-27	broussonetine P	$C_{18}H_{33}NO_5$	C_5D_5N	[13]
2-1-28	broussonetine C	$C_{18}H_{35}NO_5$	C_5D_5N	[14]
2-1-29	broussonetine D	$C_{18}H_{35}NO_5$	C_5D_5N	[14]
2-1-30	broussonetine E	$C_{18}H_{35}NO_6$	C_5D_5N	[15]
2-1-31	broussonetine F	$C_{18}H_{35}NO_6$	C_5D_5N	[15]
2-1-32	broussonetinine A	$C_{18}H_{35}NO_5$	C_5D_5N	[15]
2-1-33	broussonetinine B	$C_{18}H_{35}NO_5$	C_5D_5N	[15]
2-1-34	*α*-1-*C*-(1,10,13-trihydroxytridecyl)-1,4-dideoxy-1,4-imino-D-arabinitol	$C_{18}H_{37}NO_8$	D_2O	[6]
2-1-35	morusimic acid B	$C_{18}H_{35}NO_4$	CD_3OD	[16]
2-1-36	broussonetine R	$C_{18}H_{31}NO_6$	C_5D_5N	[12]
2-1-37	broussonetine G	$C_{18}H_{33}NO_6$	C_5D_5N	[17]
2-1-38	broussonetine H	$C_{18}H_{33}NO_6$	C_5D_5N	[17]
2-1-39	broussonetine K	$C_{24}H_{45}NO_{11}$	C_5D_5N	[18]
2-1-40	broussonetine L	$C_{24}H_{45}NO_{11}$	C_5D_5N	[18]
2-1-41	morusimic acid A	$C_{24}H_{45}NO_9$	CD_3OD	[16]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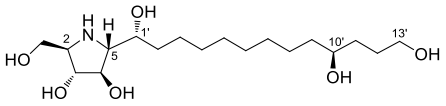
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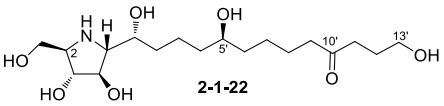
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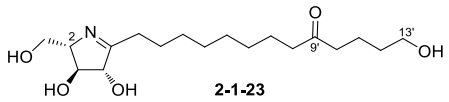
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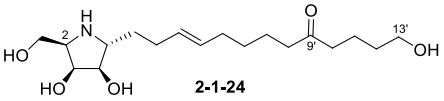
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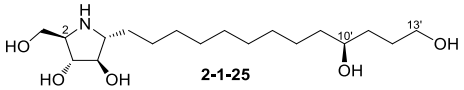
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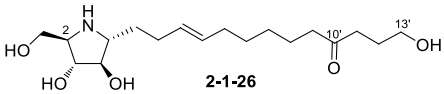
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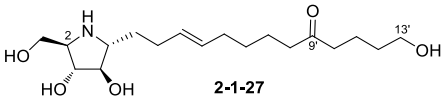
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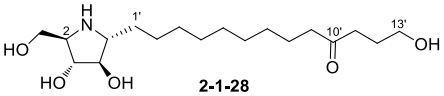
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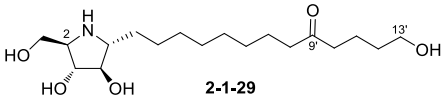
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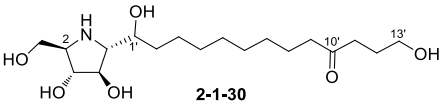
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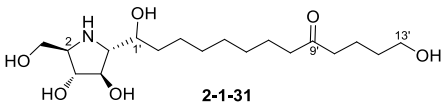
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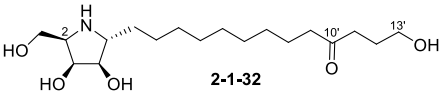
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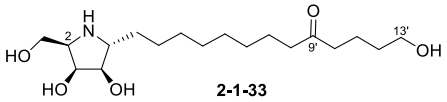
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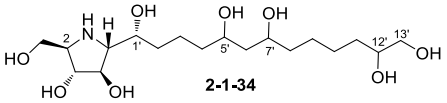
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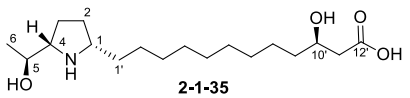
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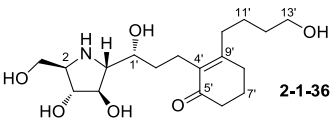
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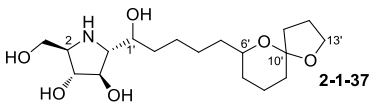
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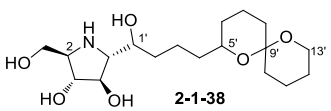
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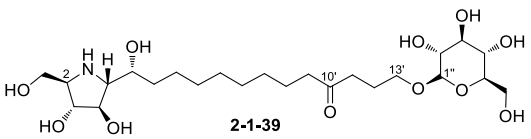
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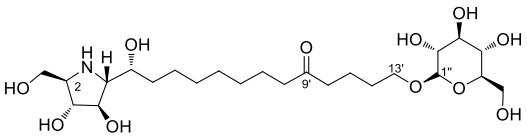
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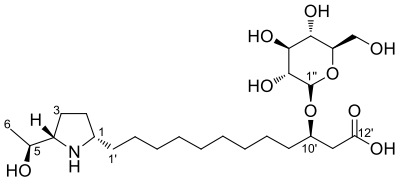


2-1-41

Table 2-1-8: ^{1}H NMR spectroscopic data of polyhydroxylated pyrrolidine alkaloids **2-1-20**~**2-1-23**.

H	2-1-20	2-1-21	2-1-22	2-1-23
1	3.66 dd(6.4, 11.7)	4.20 dd(11.0, 5.7)	4.18 dd(11.0, 5.5, ov)	4.22 dd(11.8, 3.2)
	3.73 dd(4.6, 11.7)	4.24 dd(11.0, 4.1)	4.23 dd(11.0, 4.1)	4.83 dd(11.8, 3.2)
2	3.10 ddd(4.6, 6.4, 7.1)	3.80 m	3.79 m(ov)	4.47 m
3	3.86 t(7.1)	4.70 t(6.4)	4.71 t(6.2)	5.01 t(3.4)
4	3.74 dd(7.1, 7.8)	4.94 t(6.4)	4.95 t(6.2)	5.21 d(3.4)
5	2.95 ddd(4.5, 7.8, 8.1)	3.65 t(6.4)	3.66 t(6.2)	
1′	1.44~1.76	4.14 m	4.18 (ov)	2.71 m, 2.91 m
2′	1.44~1.76	1.95 m(ov)	2.05 (ov)	1.74 (ov)
3′	3.68 m	1.55 m(ov), 1.76 m(ov)	1.98 (ov)	1.30 m
4′	1.44~1.76	1.19~1.36 (ov)	1.68 (ov)	1.08~1.19 (ov)
5′	1.44~1.76	1.19~1.36 (ov)	3.80 (ov)	1.08~1.19 (ov)
6′	3.84 m	1.19~1.36 (ov)	1.57 (ov)	1.08~1.19 (ov)
7′	1.18 d	1.19~1.36 (ov)	1.48 (ov), 1.63 (ov)	1.51 m
8′		1.47 m(ov), 1.60 m(ov)	1.65 (ov)	2.34 t(7.3)
9′		1.60 m(ov), 1.69 m(ov)	2.42 t(7.1)	
10′		3.89 m		2.47 t(7.3)
11′		1.85 m	2.64 t(7.3)	1.86 m
12′		2.01 m(ov), 2.12 m	2.05 (ov)	1.74 (ov)
13′		3.95 t(6.5)	3.85 t(6.4)	3.85 t(6.4)

Table 2-1-9: ^{1}H NMR spectroscopic data of polyhydroxylated pyrrolidine alkaloids **2-1-24**~**2-1-27**.

H	2-1-24	2-1-25	2-1-26	2-1-27
1	4.26 dd(10.8, 5.9)	4.15 dd(11.0, 5.9)	4.14 dd(11.0, 5.8)	4.14 dd(11.0, 5.9)
	4.32 dd(10.8, 5.9)	4.22 dd(11.0, 4.1)	4.19 dd(11.0, 4.2)	4.21 dd(11.0, 4.0)
2	3.72 m	3.75 m	3.72 m	3.74 m
3	4.58 t(4.8)	4.62 t(6.4)	4.62 t(6.4)	4.61 t(6.4)
4	4.09 m	4.36 t(6.4)	4.36 t(6.4)	4.36 t(6.4)
5	3.55 m	3.45 m	3.49 m	3.48 m
1′	1.69 m(ov), 1.98 m(ov)	1.70 (ov), 2.00 (ov)	1.82 m, 2.09 (ov)	1.83 (ov), 2.09 m
2′	2.29 m(ov), 2.37 m(ov)	1.16~1.66	2.29 m, 2.39 (ov)	2.26 m, 2.36 (ov)
3′	5.54 m	1.16~1.66	5.53 m	5.50 m
4′	5.46 m	1.16~1.66	5.45 m	5.43 m
5′	1.95 dd(14.0, 7.3, ov)	1.16~1.66	1.93 dd(13.5, 7.3)	1.93 dd(14.0, 7.3)
6′	1.30 q(7.3)	1.16~1.66	1.26 (ov)	1.29 quint(7.3)
7′	1.59 q(7.3)	1.16~1.66	1.22 (ov)	1.57 quint(7.3)

Table 2-1-9 (continued)

H	2-1-24	2-1-25	2-1-26	2-1-27
8′	2.35 t(7.3)	1.16~1.66	1.57 quint(7.3)	2.36 t(7.3)
9′		1.63 (ov), 1.70 (ov)	2.39 t(7.3)	
10′	2.46 t(7.3)	3.90 m		2.46 t(7.3)
11′	1.86 m	1.86 m	2.65 t(7.3)	1.83
12′	1.73 m(ov)	2.00 (ov), 2.12 m	2.06	1.72 m
13′	3.84 t(6.4)	3.95 t(6.4)	3.85 t(6.4)	3.83 t(6.4)

Table 2-1-10: ^{1}H NMR spectroscopic data of polyhydroxylated pyrrolidine alkaloids **2-1-28**~**2-1-32**.

H	2-1-28	2-1-29	2-1-30	2-1-31	2-1-32
1	4.28 dd(11.0, 5.0)	4.27 dd(11.3, 5.0)	4.22 m(ov)	4.27 m(ov)	4.32 dd(11.5, 5.0)
	4.22 dd(11.0, 5.0)	4.21 dd(11.3, 5.0)			4.26 dd(11.5, 5.0)
2	3.85 m	3.81 m	3.85 m(ov)	3.82 m	3.78 m
3	4.72 t(6.4)	4.71 t(8.3)	4.72 t(6.4)	4.71 t(6.4)	4.60 t(6.2)
4	4.44 t(6.4)	4.42 t(8.3)	4.96 t(6.4)	4.96 t(6.4)	4.10 dd(6.2, 6.6)
5	3.56 m	3.52 m	3.68 t(6.4)	3.65 t(6.4)	3.56 m
1′	2.04 (ov)	2.02 (ov)	4.15 m(ov)	4.15 m(ov)	1.95 (ov)
	1.75 (ov)	1.75 (ov)			1.65 (ov)
2′ ~6′	1.15~1.62 (ov)	1.15~1.70 (ov)	1.15~2.00	1.15~1.70	1.10~1.65
7′	1.15~1.62 (ov)	1.61 (ov)	1.15~2.00	1.55 (ov)	1.10~1.65
8′	1.62 (ov)	2.39 t(8.3)	1.55 (ov)	2.35 t(7.3)	1.60 (ov)
9′	2.44 t(7.3)		2.61 t(7.3)		2.49 t(7.3)
10′		2.51 t(8.3)		2.49 t(7.3)	
11′	2.71 t(7.3)	1.93 (ov)	2.71 t(7.3)	1.75 (ov)	2.69 (7.3)
12′	2.12 quint(7.3)	1.78 (ov)	1.95 q(7.3)	1.87 (ov)	2.08 quint(7.3)
13′	3.92 t(7.3)	3.90 t(8.3)	3.90 t(7.3, ov)	3.87 t(7.3)	3.90 t(7.3)

Table 2-1-11: ^{1}H NMR spectroscopic data of polyhydroxylated pyrrolidine alkaloids **2-1-33**~**2-1-37**.

H	2-1-33	2-1-34	2-1-35	2-1-36	2-1-37
1	4.40 dd(11.3, 5.0)	3.72 dd(11.7, 4.4)	3.43 (ov)	4.19 (11.0, 5.7, ov)	4.22 dd(10.8, 5.5)
	4.32 dd(11.3, 5.0)	3.64 dd(11.7, 6.3)		4.23 (11.0, 4.1, ov)	4.26 dd(10.8, 4.3)
2	3.82 m	3.06 ddd(7.7, 6.3, 4.4)	1.64 (ov), 2.15 m	3.79 m	3.81 m (ov)
3	4.65 t(6.3)	3.86 dd(7.7, 6.8)	2.01 m	4.70 t(6.4)	4.73 t(6.2)
4	4.14 m	4.05 dd(7.3, 6.8)	3.45 (ov)	4.95 t(6.4)	4.95 t(6.2)
5	3.52 m	2.95 dd(7.3, 5.2)	3.98 dddd (3.9, 6.4, 6.4, 6.4)	3.67 t(6.4)	3.65 t(6.2)
6			1.22 d(6.4)		
1′	1.95 (ov)	3.70 m	1.64 (ov)	4.20 (ov)	4.13 ddd
	1.68 (ov)		1.75 m		(6.2, 4.8, 4.8)

Table 2-1-11 (continued)

H	2-1-33	2-1-34	2-1-35	2-1-36	2-1-37
2′	1.20~1.70	1.62~1.40	1.30~1.49 (ov)	2.07 m, 2.16 m(ov)	1.99 (ov)
3′	1.20~1.70	1.62~1.40	1.30~1.49 (ov)	2.73 m, 2.94 m	1.86 m, 1.57 (ov)
4′	1.20~1.70	1.62~1.40	1.30~1.49 (ov)		1.46 (ov)
5′	1.20~1.70	3.84 m	1.30~1.49 (ov)		1.55 (ov), 1.41 m
6′	1.20~1.70	1.68 m, 1.63 m	1.30~1.49 (ov)	2.34 t(6.5, ov)	3.82 (ov)
7′	1.60 (ov)	3.84 m	1.30~1.49 (ov)	1.70 (ov)	1.45 (ov), 1.13 m
8′	2.39 t(8.0)	1.62~1.40	1.30~1.49 (ov)	2.16 m(ov)	1.94 (ov), 1.57 (ov)
9′		1.62~1.40	1.46 (ov)		1.66 (ov)
10′	2.51 t(8.0)	1.62~1.40	3.88 m	2.35 m(ov)	
11′	1.93 (ov)	1.62~1.40	2.26 dd(7.6, 14.9) 2.32 dd(5.5, 14.9)	1.70 (ov)	1.96 (ov) 1.58 (ov)
12′	1.80 (ov)	3.70 m		1.70 (ov)	1.95 (ov), 1.69 (ov)
13′	3.90 t(8.0)	3.60 dd(11.7, 4.4) 3.48 dd(11.7, 6.7)		3.86 t(6.2)	3.87 m(ov)

Table 2-1-12: ^{1}H NMR spectroscopic data of polyhydroxylated pyrrolidine alkaloids **2-1-38**~**2-1-41**.

H	2-1-38	2-1-39	2-1-40	2-1-41
1	4.21 dd(10.8, 5.5) 4.25 dd(10.8, 4.3)	4.19 dd(5.4, 15.3) 4.24 dd(4.2, 15.3)	4.19 dd(5.4, 15.3) 4.24 dd(4.2, 15.3)	3.48 m
2	3.81 m	3.85 m	3.81 m	1.65 (ov), 2.16 m
3	4.73 t(6.2)	4.72 t(6.4)	4.70 t(6.4)	2.02 m
4	4.96 t(6.2)	4.98 t(6.4)	4.93 t(6.4)	3.52 ddd(4.0, 8.5, 8.5)
5	3.67 t(6.2, ov)	3.72 t(6.4, ov)	3.67 t(6.4, ov)	4.03 dddd(4.0, 6.5, 6.5, 6.5)
6				1.22 d(6.5)
1′	4.17 ddd(6.2, 4.8, 4.8)	4.19 m(ov)	4.09 m(ov)	1.65 (ov), 1.78 m
2′	2.03 m	1.14~1.32	1.14~1.96	1.31~1.38 (ov)
3′	1.95 (ov)	1.14~1.32	1.14~1.96	1.31~1.38 (ov)
4′	1.47 (ov), 1.68 m	1.14~1.32	1.14~1.96	1.31~1.38 (ov)
5′	3.65 m	1.14~1.32	1.14~1.96	1.31~1.38 (ov)
6′	1.46 (ov), 1.14 m	1.14~1.32	1.14~1.96	1.31~1.38 (ov)
7′	1.92, 1.45 (ov)	1.14~1.32	1.50 m(ov)	1.40 (ov)
8′	1.63 (ov), 1.32 (ov)	1.50 m(ov)	2.28 t(7.3)	1.42(ov)
9′		2.30 t(7.3)		1.59 (ov), 1.62 (ov)
10′	1.63 (ov), 1.41 (ov)		2.37 t(7.3)	4.07 m
11′	1.92 (ov) 1.40 (ov)	2.59 t(7.3)	1.71 m(ov)	2.37 (6.0, 14.2) 2.44 dd(6.0, 14.2)
12′	1.47 (ov), 1.34 (ov)	2.02 m(ov)	1.92 m	
13′	3.71 ddd(10.7, 10.7, 2.2) 3.55 br dd(10.7, 4.3)	3.73 (ov) 4.10	3.69 (ov) 4.09	
1″		4.81 d(7.8)	4.78 d(7.8)	4.40 d(7.7)

Table 2-1-12 (continued)

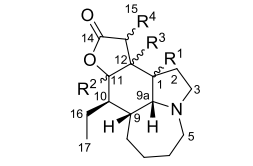
H	2-1-38	2-1-39	2-1-40	2-1-41
2″		4.02 dd(7.8, 8.5)	3.98 dd(7.8, 8.5)	3.16 dd(7.7, 8.9)
3″		4.21 dd(8.5, 8.5, ov)	4.21 dd(8.5, 8.5, ov)	3.38 t(8.9)
4″		4.17 dd(8.5, 8.5, ov)	4.17 dd(8.5, 8.5, ov)	3.29 (ov)
5″		3.95 m	3.91 m	3.27 (ov)
6″		4.39 dd(5.3, 11.8)	4.33 dd(5.3, 11.8)	3.68 dd(11.9, 5.1)
		4.54 dd(2.4, 11.8)	4.52 dd(2.4, 11.8)	3.84 dd(11.9, 2.2)

2.1.2 Stemona alkaloids

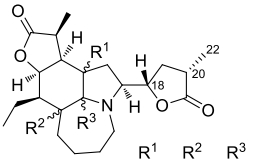
2.1.2.1 Stenine-type stemona alkaloids

Table 2-1-13: Cos, MFs, and TSs of stenine-type Stemona alkaloids **2-1-42**~**2-1-52**.

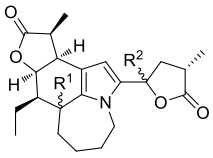
No.	Compounds	MFs	Test solvents	References
2-1-42	sessilifoline B	$C_{17}H_{27}NO_2$	$CDCl_3$	[19]
2-1-43	neostenine	$C_{17}H_{27}NO_2$	$CDCl_3$	[20]
2-1-44	tuberostemonine K	$C_{22}H_{33}NO_4$	C_5D_5N	[21]
2-1-45	neotuberostemonine	$C_{22}H_{33}NO_4$	$CDCl_3$	[22]
2-1-46	tuberostemonine J	$C_{22}H_{33}NO_4$	$CDCl_3$	[20]
2-1-47	tuberostemonine H	$C_{22}H_{33}NO_4$	$CDCl_3$	[20]
2-1-48	*epi*-bisdehydroneotuberostemonine J	$C_{22}H_{29}NO_4$	$CDCl_3$	[20]
2-1-49	bisdehydroneotuberostemonine	$C_{22}H_{29}NO_4$	$CDCl_3$	[22]
2-1-50	tuberostemonone	$C_{22}H_{31}NO_6$	$CDCl_3$	[23]
2-1-51	neotuberostemonone	$C_{22}H_{31}NO_6$	$CDCl_3$	[24]
2-1-52	epoxytuberostemonone	$C_{22}H_{29}NO_7$	$CDCl_3$	[24]



	R^1	R^2	R^3	R^4
2-1-42	α-H	β-H	β-H	α-Me
2-1-43	β-H	α-H	α-H	β-Me



	R^1	R^2	R^3
2-1-44	α-H	β-H	β-H
2-1-45	β-H	β-H	β-H
2-1-46	β-H	α-H	α-H
2-1-47	β-H	β-H	α-H



	R^1	R^2
2-1-48	α-H	α-H
2-1-49	β-H	β-H

Table 2-1-14: ^{1}H NMR spectroscopic data of stenine-type Stemona alkaloids **2-1-42**~**2-1-46**.

H	2-1-42	2-1-43	2-1-44	2-1-45	2-1-46
1	1.80~1.85 m	1.3~2.0	1.79 m	1.75 m	1.40~2.10
2	1.95~2.12 m	1.3~2.0	α 1.10 m	1.65 m	1.40~2.10
	1.38~1.44 m		β 2.25 m		

Table 2-1-14 (continued)

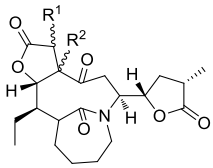
H	2-1-42	2-1-43	2-1-44	2-1-45	2-1-46
3	3.22 dd(15.3, 7.4) 2.42~2.49 m	2.45 m	3.22 dd(9.0, 12.1)	3.30 dd(7.7, 14.0)	3.02 m
5	2.85~2.91 m 2.34 t(7.9)	2.89 m 2.81 m	*α* 2.81 dd(9.3, 15.1) *β* 3.41 dd(5.8, 9.3)	*α* 3.05 dd *β* 2.92 dd	2.98 m 2.74 m
6	1.64~1.78 m 1.70~1.79 m	1.3~2.0	1.28 m	1.67 m	1.40~2.10
7	1.62~1.74 m	1.3~2.0	1.57 m	1.64 m, 1.48 m	1.40~2.10
8	1.66~1.73 m 1.69~1.80 m	1.3~2.0	*α* 1.06 m *β* 1.88 m	1.91 m 1.65 m	1.40~2.10
9	1.71~1.82 m	1.3~2.0	1.89 m	1.85 m	1.40~2.10
9a	2.38~2.45 m	3.22 m	3.11 dd(3.9, 11.2)	3.17 dd(3.8, 3.9)	3.02 m
10	1.73~1.86 m	1.3~2.0	1.89 m	1.72 m	1.40~2.10
11	4.51 d(2.2)	4.50 m	4.20 d(2.0)	4.51 dd(3.3, 3.0)	4.46 m
12	2.21~2.30 m	1.3~2.0	2.17 m	2.07 ddd(15.0, 3.3, 6.7)	1.40~2.10
13	2.80~2.87 m	2.27 m	2.88 dq(6.8, 7.4)	2.88 dq(7.1, 6.7)	2.74 m
15	1.21 d(7.2)	1.20 d(7.2)	1.32 d(7.4)	1.23 d(7.1)	1.18 d(7.5)
16	1.62~1.74 m 1.38~1.45 m	1.3~2.0	1.2~1.4 m	1.65 m 1.35 m	1.40~2.10
17	0.99 t(7.4)	0.97 t(7.5)	0.87 t(7.4)	0.99 t(7.3)	1.01 t(7.5)
18			4.26 m	4.38 ddd(11.2, 7.7, 5.5)	4.39 m
19			*α* 1.49 ddd(12.0) *β* 2.21 m	2.36 ddd(15.2, 13.3, 5.5) 1.45 dd(15.2, 11.2)	2.25 m 1.40~2.10
20			2.64 dq(4.0, 6.8)	2.59 ddq(7.0, 5.3, 12.1)	2.50 m
22			1.23 d(6.8)	1.26 d(7.0)	1.22 d(7.5)

Table 2-1-15: ^{1}H NMR spectroscopic data of stenine-type Stemona alkaloids **2-1-47**~**2-1-50**.

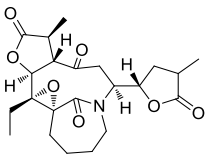
H	2-1-47	2-1-48	2-1-49	2-1-50
1	1.3~2.0			
2	1.3~2.0	5.97 s	5.95 s	*α* 2.39 dd(12.2, 3.7) *β* 3.19 dd(12.2, 12.2)
3	3.20 m			5.37ddd(12.2, 5.7, 3.7)
5	2.84 m 2.78 m	4.24 m 3.79 m	*α* 4.20 ddd(15.0, 5.0, 1.0) *β* 3.73 br dd(15.0, 11.4)	*α* 3.81 ddd(12.1, 9.2, 2.9) *β* 3.51 ddd(12.1, 4.6, 2.6)
6	1.3~2.0	3.06 m, 1.16 m	1.93 m, 1.30 m	1.91 m, 1.77 m
7	1.3~2.0	2.08 m, 1.47 m	2.03 m, 1.45 m	1.51 m, 1.75 m
8	1.3~2.0	1.95 m	1.10 ddd(11.7, 8.2, 3.2) 1.30 m	1.70 m, 1.52 m
9	1.3~2.0	3.01 m	2.73 m	3.08 ddd(11.0, 9.2, 1.8)
9a	3.01 m		–	
10	1.3~2.0	1.47 m	1.90 m	2.32 m
11	4.57 m	4.67 m	4.62 br d(5.0)	5.08 dd(9.9, 7.1)
12	1.3~2.0	3.56 dd(5.8, 5.0)	3.51 dd(7.1, 5.0)	3.56 dd(9.9, 7.7)

Table 2-1-15 (continued)

H	2-1-47	2-1-48	2-1-49	2-1-50
13	2.61 m	2.7~2.9 m	2.95 dq(7.1, 7.3)	2.91 dq(7.7, 7.0)
15	1.18 d(7.2)	1.37 d(7.0)	1.30 d(7.3)	1.30 d(7.0)
16	1.3~2.0	1.80 m	1.75~1.85 m	1.80 m, 1.27 m
17	1.00 t(7.2)	1.06 t(7.0)	1.03 t(7.5)	0.94 dd(7.4, 7.2)
18	4.37 ddd (10.5, 5.9, 4.6)	5.36 dd(11.0, 5.0)	5.33 dd(5.2, 11.2)	4.46 ddd (10.9, 5.7, 5.7)
19	2.35 m	2.80 m	2.77 ddd(11.7, 5.4, 5.2)	α 1.78 m
	1.3~2.0	2.07 m	2.15 ddd(11.7, 11.9, 11.2)	β 2.48 ddd(11.2, 5.7, 5.5)
20	2.45 m	2.7~2.9 m	2.80 ddq(11.9, 6.9, 5.4)	2.72 m
22	1.22 d(7.2)	1.35 d(7.0)	1.35 d(6.9)	1.23 d(7.1)



	R^1	R^2
2-1-50	β-Me	α-H
2-1-51	α-Me	β-H



2-1-52

Table 2-1-16: ^{1}H NMR spectroscopic data of stenine-type Stemona alkaloids **2-1-51** and **2-1-52**.

H	2-1-51	2-1-52	H	2-1-51	2-1-52
2	α 2.04 m, β 3.38 m	α 2.43 m, β 3.06 m	12	3.56 m	3.21 t(7.6)
3	4.85 m	5.21 m	13	2.81 m	2.79 m
5	α 3.18 m, β 3.43 m	α 3.58 m, β 3.94 m	15	1.02 d(7.5)	1.33 d(7.5)
6	1.82 m	1.70 m	16	1.63 m, 1.55 m	2.19 m, 1.43 m
7	α 1.77 m, β 1.15 m	α 1.92 m, β 1.70 m	17	0.96 t(7.5)	1.04 t(7.5)
8	1.54 m	1.67 m	18	4.56 m	4.73 m
9	2.74 m		19	α 2.72 m, β 1.54 m	α 1.66 m, β 2.52 m
10	2.39 m		20	2.52 m	2.81 m
11	4.54 t(3.5)	4.26 d(7.6)	22	1.27 d(7.5)	1.30 d(7.5)

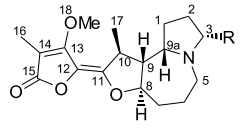
2.1.2.2 Stemoamide-type stemona alkaloids

Table 2-1-17: Cos, MFs, and TSs of stemoamide-type Stemona alkaloids **2-1-53**~**2-1-68**.

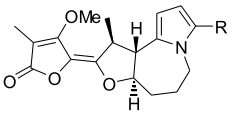
No.	Compounds	MFs	Test solvents	References
2-1-53	neostemonine	$C_{18}H_{25}NO_4$	$CDCl_3$	[25]
2-1-54	bisdehydroneostemonine	$C_{18}H_{21}NO_4$	$CDCl_3$	[25]
2-1-55	protostemonine	$C_{23}H_{31}NO_6$	$CDCl_3$	[25]
2-1-56	isoprotostemonine	$C_{23}H_{31}NO_6$	$CDCl_3$	[25]

Table 2-1-17 (continued)

No.	Compounds	MFs	Test solvents	References
2-1-57	bisdehydroprotostemonine	$C_{23}H_{27}NO_6$	$CDCl_3$	[25]
2-1-58	stemoninine	$C_{22}H_{31}NO_5$	$CDCl_3$	[26]
2-1-59	bisdehydrostemoninine	$C_{22}H_{27}NO_5$	DMSO-d_6	[27]
2-1-60	isobisdehydrostemoninine	$C_{22}H_{27}NO_5$	DMSO-d_6	[27]
2-1-61	bisdehydroneostemoninine	$C_{17}H_{21}NO_3$	$CDCl_3$	[27]
2-1-62	bisdehydrostemoninine A	$C_{22}H_{27}NO_6$	$CDCl_3$	[27]
2-1-63	bisdehydrostemoninine B	$C_{22}H_{29}NO_5$	$CDCl_3$	[27]
2-1-64	stemofoline	$C_{22}H_{29}NO_5$	C_5D_5N	[28]
2-1-65	oxystemofoline	$C_{22}H_{29}NO_6$	CD_3COCD_3	[28]
2-1-66	methylstemofoline	$C_{19}H_{23}NO_5$	$CDCl_3$	[29]
2-1-67	2′*R*-hydroxystemofoline	$C_{22}H_{29}NO_6$	$CDCl_3$	[29]
2-1-68	stemofolenol	$C_{22}H_{27}NO_6$	$CDCl_3$	[29]



2-1-53 R = H



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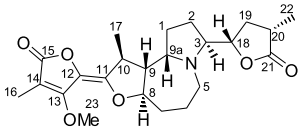
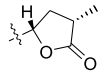
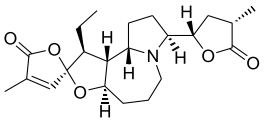


2-1-56

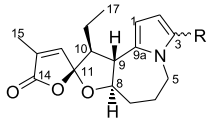
2-1-55 R =



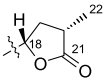
2-1-57 R =



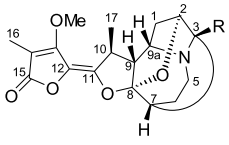
2-1-58



2-1-59 R =

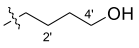


2-1-61 R = H



2-1-64 R =

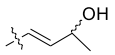
2-1-65 R =

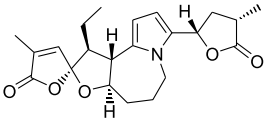


2-1-66 R = Me

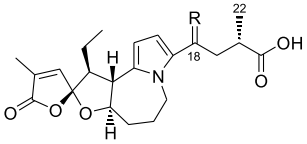
2-1-67 R =

2-1-68 R =





2-1-60



2-1-62 R = O
2-1-63 R = H_2

Table 2-1-18: ^{1}H NMR spectroscopic data of stemoamide-type Stemona alkaloids **2-1-53**~**2-1-56**.

H	2-1-53	2-1-54	2-1-55	2-1-56
1	2.24 m, 1.85 m	5.98 d(2.1)	1.92 m, 1.55 m	1.89 ddd, 1.55 m
2	2.25 m, 2.05 m	6.03 dd(2.1, 3.1)	1.89 m, 1.48 m	1.87 m, 1.48 m

Table 2-1-18 (continued)

H	2-1-53	2-1-54	2-1-55	2-1-56
3	3.67 ddd	6.60 dd(3.1)	3.27 ddd	3.24 ddd(7.3, 7.5, 11.5)
5	3.10 ddd(6.4, 3.0, 15.8)	4.07 dd(5.2, 10.4)	*α* 3.48 dd(4.0, 15.5)	*α* 3.50 dd(14.8, 4.8)
	3.35 m	3.85 dd(11.6, 14.4)	*β* 2.92 dd(7.1, 15.2)	*β* 2.89 dd(11.2, 14.8)
6	2.14 m, 1.85 m	1.76 m, 1.80 ddd	1.50 m, 1.65 m	1.50 m, 1.65 m
7	1.62 m, 2.57 m	2.57 m, 2.07 m	2.32 m, 1.50 m	2.32 m, 1.50 m
8	4.18 ddd(10.8, 10.7, 3.7)	3.77 ddd(10.3, 3.7, 14.3)	4.08 ddd(10.4, 3.4, 14.3)	4.18 ddd
9	2.22 ddd	2.91 dd(10.2, 10.3)	2.19 ddd(10.4, 4.1, 9.5)	2.12 ddd(10.4, 10.3, 5.3)
9a	4.27 m		3.73 m	3.69 ddd(5.6, 10.7, 10.6)
10	2.91 dq(6.8, 10.1)	3.49 dq(6.5, 10.2)	2.89 m	3.01 dq(6.7, 10.5)
16	2.05 s	2.07 s	2.04 s	2.01 s
17	1.40 d(6.8)	1.51 d(6.5)	1.41 d(6.6)	1.32 d(6.7)
18	4.10 s	4.16 s	4.15 ddd(11.1, 5.5, 5.4)	4.14 ddd
19			2.35 m, 1.52 m	*α* 2.36 ddd, *β* 1.52 m
20			2.60 ddq(12.0, 8.5, 7.0)	2.58 ddq
22			1.23 d(7.0)	1.24 d(6.9)
23			4.10 s	4.10 s

Table 2-1-19: ^{1}H NMR spectroscopic data of stemoamide-type Stemona alkaloids **2-1-57**~**2-1-60**.

H	2-1-57	2-1-58	2-1-59	2-1-60
1	5.95 d(3.8)	1.50 m, 1.75 m	5.88 d(3.5)	5.91 d(3.6)
2	6.13 d(3.8)	1.35 m, 1.80 m	6.13 d(3.5)	6.11 d(3.6)
3		3.25 ddd(5.5, 7.0, 10.0)		
5	*α* 4.37 dd(5.6, 14.7)	*α* 2.86 dd(11.5, 15.5)	4.20 dd(5.3, 14.6)	4.20 dd(5.3, 14.4)
	β 3.82 dd(11.3, 14.7)	*β* 3.41 dd(9.0, 15.5)	3.83 dd(11.8, 14.6)	3.78 dd(11.8, 14.4)
6	1.85 m, 1.55 m	*α* 1.57 m, *β* 1.33 m	2.02 m, 1.60 m	2.00 m, 1.63 m
7	2.47 m	*α* 2.03 br dd(3.5, 12.0)	2.19 m	2.20 m
	2.10 m	*β* 1.43 m	1.72 m	1.65 m
8	3.70 ddd(10.9, 10.2, 3.7)	3.93 ddd(3.5, 9.5, 11.0)	3.58 ddd(3.6, 9.9, 9.9)	3.68 ddd(3.6, 9.9, 9.9)
9	3.10 t(10.2)	2.43 ddd(5.5, 9.5, 11.0)	3.21 dd(9.9, 12.0)	3.05 dd(10.6, 20.9)
9a		3.68 dt(6.0, 6.0, 9.5)		
10	3.53 dq(10.2, 6.5)	1.93 ddd(5.5, 7.0, 12.0)	2.60 m	2.60 m
12		6.59 d(2.0)	7.18 d(1.3)	7.23 d(1.4)
15		1.85 d(2.0)	1.81 d(1.3)	1.81 d(1.4)
16	2.05 s	1.28 m, 1.60 m	1.43 m, 1.38 m	1.45 m, 1.38 m

Table 2-1-19 (continued)

H	2-1-57	2-1-58	2-1-59	2-1-60
17	1.51 d(6.5)	0.77 t(7.5)	0.81 t(7.6)	0.79 t(7.6)
18	5.50 dd(5.2, 11.0)	4.14 ddd(5.5, 7.0, 10.0)	5.53 dd(5.3, 11.1)	5.52 dd(5.4, 11.3)
19	2.70 ddd	*α* 2.31 ddd(5.5, 9.0, 12.0)	2.68 m	2.72 m
	2.15 m	*β* 1.43 m	2.11 m	2.12 m
20	2.85 m	2.54 ddq(7.0, 9.0, 12.0)	2.82 m	2.80 m
22	1.25 d(7.1)	1.17 d(7.0)	1.19 d(7.0)	1.18 d(6.9)
23	4.25 s			

Table 2-1-20: ^{1}H NMR spectroscopic data of stemoamide-type Stemona alkaloids **2-1-61**~**2-1-64**.

H	2-1-61	2-1-62	2-1-63	2-1-64
1	5.89 m	5.94 d(4.0)	5.80 d(4.7)	1.89 m
2	6.02 t(2.0)	6.95 d(4.1)	5.85 d(4.7)	4.13 s
3	6.58 t(3.3)			
5	4.05 dd(4.8, 14.3)	5.80 dd(5.7, 14.7)	4.18 dd(5.3, 14.8)	*α* 2.99 ddd(6.7, 7.4, 14.0)
	3.89 dd(11.6, 14.3)	3.68 dd(2.8, 14.3)	3.70 m	*β* 2.93 ddd(6.6, 6.9, 14.0)
6	2.08 m, 1.58 m	2.08 m, 1.62 m	2.08 m, 1.50 m	1.73 m
7	2.31 m, 1.80 m	2.31 m, 1.85 m	2.30 m, 1.80 m	2.64 dd(2.7, 6.2)
8	3.71 dt(3.4, 11.1)	3.70 m	3.67 dd(3.7, 15.2)	
9	3.20 dd(3.2, 11.0)	3.25 m	3.20 dd(10.0, 12.1)	1.89 dd(3.4, 3.5)
9a				3.43 m
10	2.72 dt(3.2, 9.3)	2.58 ddd(3.5, 8.9, 12.5)	2.55 m	3.14 dq(3.5, 6.6)
12	6.75 d(1.5)	6.75 d(1.6)	6.75 d(1.6)	1.61 ddd(3.2, 3.4, 12.0)
15	1.98 d(1.5)	1.98 d(1.5)	1.98 d(1.6)	
16	1.61 m, 1.77 m	1.72 m, 1.64 m	1.60 m, 1.80 m	1.91 s
17	0.90 t(7.6)	0.91 t(7.5)	0.87 t(7.6)	1.30 d(6.6)
18			2.00 m, 1.77 m	
19		3.25 m, 2.92 m	2.58 m	
20		3.12 dq(2.0, 7.3)	2.68 m	
22		1.27 d(7.2)	1.25 d(6.3)	
1′				1.43 m
2′				1.42 m, 1.11 m
3′				1.21 m
4′				0.91 t(6.0)
OMe				3.87 s

Table 2-1-21: ^{1}H NMR spectroscopic data of stemoamide-type Stemona alkaloids **2-1-65**~**2-1-68**.

H	2-1-65	2-1-66	2-1-67	2-1-68
1	*α* 1.98 m	1.93 d(12.5)	1.88 d(12.5)	1.98 dd(12.0, 3.0)
	β 1.73 m	1.84 m	1.81 m	1.80 m

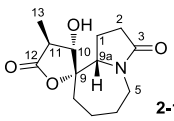
Table 2-1-21 (continued)

H	2-1-65	2-1-66	2-1-67	2-1-68
2	4.23 m	4.15 br s	4.42 br s	4.25 br s
5	*α* 3.01 m	3.20 ddd(14.0, 10.5, 5.0)	3.09 ddd(14.3, 10.0, 5.0)	3.05 m
	β 3.14 m	3.03 m	2.95 ddd(14.3, 8.8, 5.0)	3.01 m
6	*α* 1.96 m	1.97 m	1.88 m	1.83 m
	β 1.85 ddd(2.5, 7.4, 11.9)	1.82 m	1.77 m	
7	2.68 dd(2.5, 6.1)	2.69 d(5.0)	2.64 d(6.0)	2.89 d(5.4)
9	1.81 m	1.78 dd(9.8, 3.5)	1.72 dd(9.0, 4.0)	1.85 dd(5.9, 3.0)
9a	3.47 m	3.55 br s	3.43 br s	3.57 br s
10	3.10 dq(6.5, 7.5)	3.03 dq(9.8, 6.0)	3.02 dq(9.0, 6.8)	3.08 m
16	2.06 s	2.00 s	2.00 s	2.07 s
17	1.36 d(6.5)	1.32 d(6.5)	1.30 d(7.0)	1.39 d(6.3)
1′	1.72 m	1.35 s	1.76 dd(15.2, 3.8)	5.76 d(15.9)
	1.52 m		1.70 dd(15.2, 7.3)	
2′	1.57 m		3.69 m	5.87 dd(15.9, 5.1)
3′	1.61 m		1.43 quint(7.5)	4.36 quint(6.5)
4′	3.56 t(6.2)		0.88 t(7.5)	1.28 d(6.5)
OMe	4.13 s	4.08 s	4.07 s	4.15 s

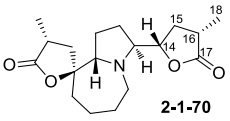
2.1.2.3 Tuberostemospironine-type stemona alkaloids

Table 2-1-22: Cos, MFs, and TSs of tuberostemospironin-type Stemona alkaloids **2-1-69**~**2-1-74**.

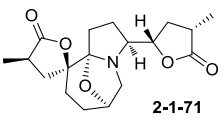
No.	Compounds	MFs	Test solvents	References
2-1-69	tuberostemospironine	$C_{13}H_{19}NO_4$	$CDCl_3$	[23]
2-1-70	croomine	$C_{18}H_{27}NO_4$	$CDCl_3$	[30]
2-1-71	stemotinine	$C_{18}H_{25}NO_5$	$CDCl_3$	[30]
2-1-72	isostemotinine	$C_{18}H_{25}NO_5$	$CDCl_3$	[30]
2-1-73	stemonidine	$C_{19}H_{29}NO_5$	$CDCl_3$	[30]
2-1-74	tuberospironine	$C_{18}H_{27}NO_5$	C_5D_5N	[21]



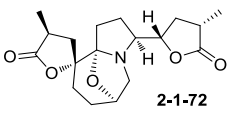
2-1-69



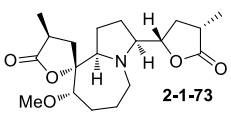
2-1-70



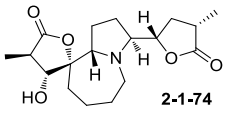
2-1-71



2-1-72



2-1-73



2-1-74

Table 2-1-23: ^{1}H NMR spectroscopic data of tuberostemospironine-type Stemona alkaloids **2-1-69**~**2-1-71**.

H	2-1-69	2-1-70	2-1-71
1	*α* 1.90 m, *β* 2.01 m	–	1.86 m, 1.91 m
2	*α* 2.24 m, *β* 2.25 m	–	1.72 m, 1.98 m
3		3.36 ddd(7.4, 6.8, 6.8)	2.86 ddd(10.8, 8.8, 5.8)
5	*α* 2.80 ddd(13.2, 12.7, 1.0) *β* 3.83 ddd(13.2, 3.6, 2.9)	3.12 m	*α* 3.00 ddd(10.7, 6.3, 1.4) *β* 3.22 d(10.7)
6	1.65 m, 1.29 m	–	4.59 m(6.3, 2.0, 2.0, 1.4)
7	1.51 m 1.90 m	–	*α* 1.81 m(13.5, 12.6, 5.9) *β* 1.62 br dd(12.6, 5.4, 1.8)
8	1.57 m	–	*α* 1.55 ddt(13.5, 5.9, 1.8, 1.8) *β* 2.34 dt(13.5, 13.5, 5.4)
9a	3.70 dd(6.4, 9.8)	3.50 dd(8.2, 7.8)	
10	3.77 d(10.2)	*α* 2.45 dd(13.5, 10.9) *β* 1.65 dd(13.5, 7.9)	*α* 2.61 dd(14.6, 11.6) *β* 1.70 dd(14.6, 6.3)
11	2.49 dq(10.2, 7.0)	2.72 ddq(10.9, 7.9, 7.8)	2.81 ddq(11.6, 7.7, 6.3)
13	1.15 d(7.0)	1.32 d(7.8)	1.34 d(7.7)
14		4.32 ddd(11.3, 8.8, 5.4)	4.26 ddd(11.3, 8.8, 5.4)
15*α*		1.51 ddd(12.6, 12.6, 11.3)	1.48 ddd(12.6, 12.6, 11.3)
15*β*		2.37 ddd(12.6, 9.0, 5.4)	2.36 ddd(12.6, 9.0, 5.4)
16		2.61 ddq(12.6, 9.0, 7.5)	2.67 ddq(12.6, 9.0, 7.5)
18		1.27 d(7.5)	1.26 d(7.5)

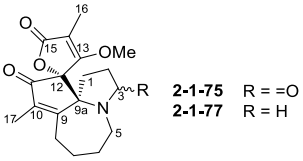
Table 2-1-24: ^{1}H NMR spectroscopic data of tuberostemospironine-type Stemona alkaloids **2-1-72**~**2-1-74**.

H	2-1-72	2-1-73	2-1-74
1	1.88 m, 1.92 m	–	2.07~2.10 m, 2.12~2.15 m
2	1.60 m, 2.15 m	–	1.75~1.81 m, 1.40~1.42 m
3	2.93 ddd(10.8, 7.8, 6.1)	3.30 ddd(7.4, 6.8, 6.8)	3.45 dd(6.8, 6.4)
5	*α* 3.04 dd(10.4, 6.3) *β* 3.20 d(10.4)	3.10 m	*α* 3.50 dd(1.0, 15.1) *β* 3.27 dd(15.1, 11.7)
6	4.68 ddd(6.3, 2.0, 2.0)	–	*α* 1.32~1.35 m, *β* 1.65~1.72 m
7	–	–	*α* 1.40~1.45 m *β* 1.85 dt(15.0, 4.5)
8	–	3.22 dd(6.8, 2.4)	*α* 2.03~2.08 m, *β* 2.20~2.25 m
9a		3.77 (8.0, 6.6)	3.64 t(8.4)
10	*α* 2.10 dd(13.1, 10.0) *β* 1.71 dd(13.1, 12.6)	*α* 2.47 dd(14.0, 11.0) *β* 1.63 dd(14.0, 8.0)	4.09 d(10.3)
11	2.80 ddq(12.6, 10.0, 7.7)	2.71 ddq(11.0, 8.0, 7.8)	2.87 dq(10.3, 6.8)
13	1.28 d(7.7)	1.31 d(7.8)	1.36 d(6.8)
14	4.14 ddd(11.3, 7.8, 5.4)	4.38 ddd(11.3, 8.8, 5.4)	4.18 ddd(5.3, 6.8, 1.5)
15*α*	1.58 ddd(12.6, 12.6, 11.3)	1.50 ddd(12.6, 12.6, 11.3)	1.44~1.50 m
15*β*	2.36 ddd(12.6, 9.0, 5.4)	2.38 ddd(12.6, 9.0, 5.4)	2.14~2.18 m
16	2.66 ddq(12.6, 9.0, 7.5)	2.62 ddq(12.6, 9.0, 7.5)	2.59 dq(12.4, 6.8)
18	1.28 d(7.5)	1.27 d(7.5)	1.2 d(6.8)
OMe		3.40 s	

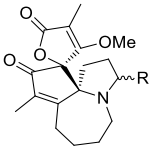
2.1.2.4 Stemonamine-type stemona alkaloids

Table 2-1-25: Cos, MFs, and TSs of stemonamine-type Stemona alkaloids **2-1-75**~**2-1-78**.

No.	Compounds	MFs	Test solvents	References
2-1-75	stemonamide	$C_{18}H_{21}NO_5$	$CDCl_3$	[31]
2-1-76	isostemonamide	$C_{18}H_{21}NO_5$	$CDCl_3$	[31]
2-1-77	stemonamine	$C_{18}H_{23}NO_4$	$CDCl_3$	[31]
2-1-78	isostemonamine	$C_{18}H_{23}NO_4$	$CDCl_3$	[31]



2-1-75 R = =O
2-1-77 R = H



2-1-76 R = =O
2-1-78 R = H

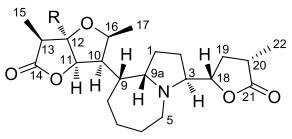
Table 2-1-26: ^{1}H NMR spectroscopic data of stemonamine-type Stemona alkaloids **2-1-75**~**2-1-78**.

H	2-1-75	2-1-76	2-1-77	2-1-78
1	*α* 1.95 ddd	*α* 1.90 ddd(9.2, 12.8, 13.2)	1.85 m	1.73 m
	β 2.59 ddd	*β* 2.59 dd(13.2, 7.2)	1.75 m	1.52 ddd
2	*α* 2.28 dd(14.6, 7.9)	*α* 2.26 ddd	1.85 m	2.34 dd(5.7, 12.7)
	β 2.37 dd(14.6, 8.8)	*β* 2.32 ddd(9.2, 16.4, 12.8)		1.73 m
3			3.05 ddd	2.82 dd(13.5, 6.3)
			3.10 m	3.17 m
5	*α* 4.18 br d(14.6)	*α* 4.14 br d(10.8)	2.75 m	3.17 m
	β 2.83 br t(12.9)	*β* 2.95 m	3.10 m	3.08 dd (2.7, 15.6)
6	*α* 1.40 m	*α* 1.36 m	2.10 m	1.75 m
	β 1.82 br d(10.5)	*β* 1.77 br d(10.5)	1.39 m	1.36 br dd(14.3, 3.2)
7	*α* 2.13 m, *β* 1.32 m	*α* 2.10 m, *β* 1.26 m	1.85 m, 1.20 ddd	1.98 m, 1.13 m
8	*α* 2.14 m	*α* 2.10 m	2.87 ddd	2.81 m
	β 2.98 dd(5.7, 13.0)	*β* 2.93 dd(12.2, 5.6)	2.10 m	1.98 m
16	2.00 s	2.06 s	2.00 s	2.06 s
17	1.85 s	1.74 s	1.76 s	1.73 s
OMe	3.97 s	4.13 s	3.96 s	4.11 s

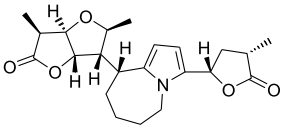
2.1.2.5 Parvistemoline-type stemona alkaloids

Table 2-1-27: Cos, MFs, and TSs of parvistemoline-type Stemona alkaloids **2-1-79**~**2-1-82**.

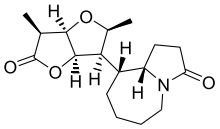
No.	Compounds	MFs	Test solvents	References
2-1-79	parvistemonine	$C_{22}H_{33}NO_5$	CD_3OD	[32]
2-1-80	12*α*-methoxylparvistemonine	$C_{23}H_{35}NO_6$	$CDCl_3$	[33]
2-1-81	didehydroparvistemonine	$C_{22}H_{29}NO_5$	$CDCl_3$	[34]
2-1-82	parvistemoline	$C_{17}H_{25}NO_4$	$CDCl_3$	[34]



2-1-79 R = H
2-1-80 R = OMe



2-1-81



2-1-82

Table 2-1-28: ^{1}H NMR spectroscopic data of parvistemoline-type Stemona alkaloids **2-1-79**~**2-1-82**.

H	2-1-79	2-1-80	2-1-81	2-1-82
1	2.04 m	1.40 m, 1.64 m	6.07 d(3.7)	2.33 m, 1.89 m
2	*α* 2.13 m, *β* 1.68 m	1.59 m, 1.75 m	5.81 d(3.7)	2.45 m, 2.23 m
3	3.79 ddd(6.5, 9.7, 11.0)	3.40 m		
5	*α* 3.30 m	*α* 2.85 m	*α* 3.84 ddd(12.8, 12.0, 2.0)	*α* 2.63 ddd(14.0, 12.7, 2.3)
	β 3.52 ddd(12.7, 4.0, 2.0)	*β* 3.37 m	*β* 4.19 ddd(12.8, 2.8, 2.2)	*β* 4.03 ddd(14.0, 8.0, 3.0)
6	1.80 m	1.42 m, 1.70 m	1.98 m	1.45 m
7	*α* 1.43 m, *β* 1.80 m	1.38 m, 1.77 m	0.88 m, 1.24 m	1.45 m
8	*α* 1.94 m, *β* 1.68 m	1.45 m, 1.91 m	1.57 m	1.72 m
9	3.25 m	2.15 ddd	3.10 ddd(10.4, 7.9, 2.5)	1.73 m
9a	3.72 ddd(12.4, 8.1, 4.3)	3.43 ddd		3.75 ddd(10.7, 5.5, 5.2)
10	2.27 ddd(8.8, 6.8, 7.4)	2.30 ddd	2.87 ddd(4.1, 7.9, 7.4)	2.42 m
11	4.91 dd(8.8, 3.9)	4.6 d(4.3)	5.02 dd(4.1, 4.1)	4.98 dd(4.3, 4.5)
12	4.49 dd(3.9, 3.8)		4.68 dd(5.3, 4.1)	4.64 dd(5.5, 4.5)
13	2.72 dq(5.8, 7.2)	2.89 q(7.2)	2.74 dq(7.4, 5.2)	2.73 dq(6.8, 5.5)
15	1.05 d(7.2)	1.34 d(7.2)	1.30 d(7.4)	1.00 d(6.8)
16	4.30 dq(6.7, 6.8)	4.48 dq(6.7, 7.2)	4.60 dq (7.4, 6.5)	4.31 dq(7.5, 6.9)
17	1.05 d(6.7)	1.19 d(6.7)	0.95 d(6.5)	1.03 d(7.5)
18	4.55 ddd(11, 5.5, 9.7)	4.18 m	5.37 dd(11.0, 5.2)	
19	*α* 1.56 ddd(12.5, 11, 12.2)	1.55 m	*α* 2.19 ddd(12.5, 11.5, 11.0)	
	β 2.45 ddd(12.5, 5.5, 8.4)	2.35 m	*β* 2.66 ddd(12.5, 8.3, 5.3)	
20	2.66 ddq(8.4, 12.2, 7.7)	2.59 m	2.74 ddq (11.5, 8.3, 7.0)	
22	1.09 d(7.7)	1.25 d(7.0)	1.33 d(7.0)	
OMe		3.35 s		

Bibliography

[1] Yasuda K, Kizu H, Yamashita T, et al. J Nat Prod, 2002, 65: 198.
[2] Molyneux RJ, Pan YT, Torpea JE, et al. J Nat Prod, 1993, 56: 1356.
[3] Yamashita T, Yasuda K, Kizu H, et al. J Nat Prod, 2002, 65: 1875.
[4] Watson AA, Nash RJ, Wormald MR, et al. Phytochemistry, 1997, 46: 255.
[5] Asano N, Kato A, Miyauchi M, et al. J Nat Prod, 1998, 61: 625.
[6] Asano N, Ikeda K, Kasahara M, et al. J Nat Prod, 2004, 67: 846.
[7] Asano N, Yamauchi T, Kagamifuchi K, et al. J Nat Prod, 2005, 68: 1238.

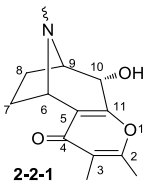
[8] Asano N, Tomioka E, Kizu H, et al. Carbohydr Res, 1994, 253: 235.
[9] Asano N, Oseki K, Tomioka E, et al. Carbohydr Res, 1994, 259: 243.
[10] Asano N, Nishida M, Miyauchi M, et al. Phytochemistry, 2000, 53: 379.
[11] Kato A, Adachi I, Miyauchi M, et al. Carbohydr Res, 1999, 316: 95.
[12] Tsukamoto D, Shibano M, Okamoto R, et al. Chem Pharm Bull, 2001, 49: 492.
[13] Shibano M, Tsukamoto D, Fujimoto R, et al. Chem Pharm Bull, 2000, 48: 1281.
[14] Shibano M, Kitagawa S, Kusano G. Chem Pharm Bull, 1997, 45: 505.
[15] Shibano M, Kitagawa S, Nakamura S, et al. Chem Pharm Bull, 1997, 45: 700.
[16] Kusano G, Orihara S, Tsukamoto D, et al. Chem Pharm Bull, 2002, 50: 185.
[17] Shibano M, Nakamura S, Akazawa N, et al. Chem Pharm Bull, 1998, 46: 1048.
[18] Shibano M, Nakamura S, Motoya N, et al. Chem Pharm Bull, 1999, 47: 472.
[19] Qian J, Zhan ZJ. Helv Chim Acta, 2007, 90: 326.
[20] Chung HS, Hon PM, Lin G, et al. Planta Med, 2003, 69: 914.
[21] Jiang RW, Hon PM, Zhou Y, et al. J Nat Prod, 2006, 69: 749.
[22] Ye Y, Qin GW, Xu RS. Phytochemistry, 1994, 37: 1201.
[23] Lin WH, Ye Y, Xu RS. J Nat Prod, 1992, 55: 571.
[24] Cai XH, Luo XD. Planta Med, 2007, 73: 170.
[25] Ye Y, Qin GW, Xu RS. Phytochemistry, 1994, 37: 1205.
[26] Cheng DL, Guo J, Chu TT, et al. J Nat Prod, 1988, 51: 202.
[27] Lin LG, Zhong QX, Cheng TY, et al. J Nat Prod, 2006, 69: 1051.
[28] Lin WH, Xu RS, Zhong QX. Acta Chim Sin, 1991, 49: 1034.
[29] Sastraruji T, Jatisatienr A, Pyne SG, et al. J Nat Prod, 2005, 68: 1763.
[30] Xu RS, Lu YJ, Chu JH. Tetrahedron, 1982, 38: 2667.
[31] Ye Y, Qin GW, Xu RS. J Nat Prod, 1994, 57: 655.
[32] Lin WH, Yin BP, Tang Z, et al. Acta Chim Sin, 1990, 48: 811.
[33] Xu RS. Chemistry of natural products. 2nd ed. Science Press, Beijing, 2004, 146.
[34] Lin WH, Xu RS, Zhong QX. Acta Chim Sin, 1991, 49: 927.

2.2 Tropane alkaloids

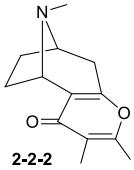
2.2.1 Tropanol-type tropane alkaloids

Table 2-2-1: Cos, MFs, and TSs of tropanol-type tropane alkaloids **2-2-1**~**2-2-11**.

No.	Compounds	MFs	Test solvents	References
2-2-1	10-hydroxydarlingine	$C_{13}H_{17}NO_3$	$CDCl_3$	[35]
2-2-2	(+)-darlingine	$C_{13}H_{17}NO_2$	$CDCl_3$	[36]
2-2-3	darlingine *N*-oxide	$C_{13}H_{17}NO_3$	DMSO-d_6	[36]
2-2-4	calystegin A_6	$C_7H_{13}NO_3$	D_2O	[37]
2-2-5	calystegin N_1	$C_7H_{14}N_2O_3$	D_2O	[37]
2-2-6	calystegin A_3	$C_7H_{13}NO_3$	D_2O	[38]
2-2-7	calystegin A_5	$C_7H_{13}NO_3$	D_2O	[38]
2-2-8	calystegin B_1	$C_7H_{13}NO_4$	D_2O	[38]
2-2-9	calystegin B_2	$C_7H_{13}NO_4$	D_2O	[38]
2-2-10	calystegin B_3	$C_7H_{13}NO_4$	D_2O	[38]
2-2-11	calystegin C_1	$C_7H_{13}NO_5$	D_2O	[38]



2-2-1



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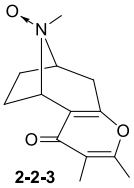
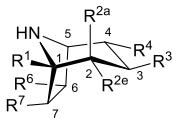


2-2-3

Table 2-2-2: ^{1}H NMR spectroscopic data of tropanol-type tropane alkaloids **2-2-1**~**2-2-3**.

H	2-2-1	2-2-2	2-2-3
6	4.17 d(5.4)	4.14 d(6.0)	4.18 d(6.0)
7α	1.76 ddd(12.0, 9.0, 3.0)	1.78 m	1.65 m
7β	2.23 m	2.17 m	2.75 m
8	α 2.16 m, β 1.98 m	α 2.19 m, β 1.48 m	α 1.71 m, β 2.52 m
9	3.52 dd(5.4, 5.4)	3.42 dd(6.0, 5.4)	3.65 dd(6.0, 5.4)
10α		2.07 d(18.6)	2.82 d(18.6)
10β	5.06 d(5.4)	2.96 dd(18.6, 5.4)	3.24 dd(18.6, 5.4)
2-Me	2.28 s	2.21 s	2.26 s
3-Me	1.99 s	1.90 s	1.82 s
NMe	2.43 s	2.30 s	3.07 s



	R^1	R^{2e}	R^{2a}	R^3	R^4	R^6	R^7
2-2-4	OH	OH	H	H	H	H	OH
2-2-5	NH_2	OH	H	OH	OH	H	H
2-2-6	OH	OH	H	OH	H	H	H
2-2-7	OH	H	H	OH	OH	H	H
2-2-8	OH	OH	H	OH	H	OH	H
2-2-9	OH	OH	H	OH	OH	H	H
2-2-10	OH	H	OH	OH	OH	H	H
2-2-11	OH	OH	H	OH	OH	OH	H

Table 2-2-3: ^{1}H NMR spectroscopic data of tropanol-type tropane alkaloids **2-2-4**~**2-2-7**.

H	2-2-4	2-2-5	2-2-6	2-2-7
2ax	3.697 dd(11.4, 6.2)	3.232 dd(8.5, 1.5)	3.40 dd(8.4, 1.5)	1.68 br t(12.3, 11.0)
2eq				2.26 dd(12.3, 6.4)
3ax	1.277 m	3.281 t(8.5)	3.70 ddd(11.0, 8.4, 6.6)	3.72 m
3eq	1.974 m			
4ax	1.56 m	3.515 dd(8.5, 4.0)	1.45~1.57①	3.47 br dd(8.8, 4.0)
4eq	1.50 m		1.99 ddd(13.2, 6.6, 2.6)	
5	3.450 m	3.263 dd(7.0, 4.0)	3.46 ddd(6.6, 3.6, 2.6)	3.34 dd(7.0, 4.0)
6	ex 1.866 dddd(14.3, 7.7, 3.3, 1.5)	ex 1.850 m	ex 1.45~1.57①	1.76~1.96①(2H)
	en 2.116 dd(14.3, 7.7)	en 1.732 m	en 2.01~2.12①	
7	4.076 dd(7.7, 3.3)	1.342 m, 1.910 m	1.45~1.57①, 2.01~2.128	1.76~1.96①(2H)

① Peak-type not shown in the literatures.
Notes: ax denotes axial, eq equatorial, en *endo*, and ex *exo*.

Table 2-2-4: ^{1}H NMR spectroscopic data of tropanol-type tropane alkaloids **2-2-8**~**2-2-11**.

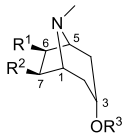
H	2-2-8	2-2-9	2-2-10	2-2-11
2ax	3.34 dd(8.4, 1.8)	3.42 dd(8.4, 1.8)		3.35 dd(8.8, 1.8)
2eq			3.83 d(3.6)	
3ax	3.45 ddd(11.0, 8.4, 6.6)	3.35 t(8.4)	3.61 dd(9.5, 3.6)	3.15 t(8.8)
3eq				
4ax	1.47 ddd(13.2, 11.0, 4.0)	3.58 ddd(8.4, 4.0. 5)	3.65 dd(9.5, 3.6)	3.54 dd(8.8, 4.7)
4eq	2.03 ddd(13.2, 6.6, 2.6)			
5	3.26 br s	3.32 br dd(7.0, 4.0, 1.5①)	3.28 dd(6.6, 3.6)	3.20 dd(4.7, 1.5)
6	4.11 dd(7.3, 2.6)	en 1.76 m, ex 1.95 m	en 1.74 m, ex 1.92 m	4.29 br dd(7.4, 2.9, 1.5①)
7	ex 1.40 ddd(14.7, 2.6, 1.8)	ex 1.54 m, en 2.00 m	1.76~1.81 (2H)	ex 1.47 ddd(14.3, 2.9, 1.8)
	en 2.52 dd(14.7, 7.3)			en 2.55 dd(14.3, 7.4)

① Confirmed by decoupling experiments.

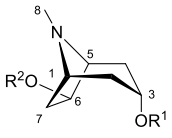
2.2.2 Hyoscyamine-type tropane alkaloids

Table 2-2-5: Cos, MFs, and TSs of hyoscyamine-type tropane alkaloids **2-2-12**~**2-2-46**.

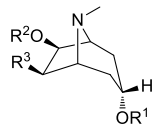
No.	Compounds	MFs	Test solvents	References
2-2-12	7*β*-hydroxy-6*β*-(3,4,5-trimethoxybenzoyloxy)-3*α*-(*E*)-(3,4,5-trimethoxycinnamoyloxy)tropane	$C_{30}H_{37}NO_{11}$	$CDCl_3$	[39]
2-2-13	6*β*-benzoyloxy-3*α*-(*Z*)-(3,4,5-trimethoxycinnamoyloxy)-tropane	$C_{27}H_{31}NO_7$	$CDCl_3$	[39]
2-2-14	(–)-6*β*-benzoyloxy-3*α*-hydroxytropane	$C_{15}H_{19}NO_3$	$CDCl_3$	[39]
2-2-15	6*β*-benzoyloxy-3*α*-(3,4,5-trimethoxycinnamoyloxy)-tropane	$C_{27}H_{31}NO_7$	$CDCl_3$	[39]
2-2-16	3*α*-(1-methylitaconyl)-6*β*-senecioyloxytropane	$C_{19}H_{27}NO_6$	CD_3OD	[40]
2-2-17	3*α*-(1-methylitaconyl)-6*β*-angeloyloxytropane	$C_{19}H_{27}NO_6$	CD_3OD	[40]
2-2-18	3*α*-(1-methylmesaconyl)-6*β*-senecioyloxytropane	$C_{19}H_{27}NO_6$	CD_3OD	[40]
2-2-19	3*α*-(1-methylmesaconyl)-6*β*-angeloyloxytropane	$C_{19}H_{27}NO_6$	CD_3OD	[40]
2-2-20	3*α*-(1-methylmesaconyl)-6*β*-tigloyloxytropane	$C_{19}H_{27}NO_6$	CD_3OD	[40]
2-2-21	3*α*-(1-methylcitraconyl)-6*β*-senecioyloxytropane	$C_{19}H_{27}NO_6$	CD_3OD	[40]
2-2-22	3*α*–(1-methylcitraconyl)-6*β*-angeloyloxytropane	$C_{19}H_{27}NO_6$	CD_3OD	[40]
2-2-23	catuabine D	$C_{19}H_{23}N_3O_4$	$CDCl_3$	[41]
2-2-24	7*β*-hydroxycatuabine D	$C_{19}H_{23}N_3O_5$	$CDCl_3$	[41]
2-2-25	catuabine E	$C_{20}H_{25}N_3O_4$	$CDCl_3$	[41]
2-2-26	7*β*-hydroxycatuabine E	$C_{20}H_{25}N_3O_5$	$CDCl_3$	[41]
2-2-27	7*β*-acetylcatuabine E	$C_{22}H_{27}N_3O_6$	$CDCl_3$	[41]
2-2-28	catuabine F	$C_{23}H_{28}N_2O_7$	$CDCl_3$	[41]
2-2-29	7*β*-hydroxycatuabine F	$C_{23}H_{28}N_2O_8$	$CDCl_3$	[41]
2-2-30	catuabine G	$C_{16}H_{21}NO_5$	$CDCl_3$	[41]
2-2-31	catuabine H	$C_{14}H_{20}N_2O_3$	$CDCl_3$	[42]
2-2-32	3-*αH*-catuabine H	$C_{14}H_{20}N_2O_3$	$CDCl_3$	[42]
2-2-33	7*β*-hydroxycatuabine H	$C_{14}H_{20}N_2O_4$	$CDCl_3$	[42]
2-2-34	7*α*-hydroxycatuabine H	$C_{14}H_{20}N_2O_4$	$CDCl_3$	[42]
2-2-35	catuabine I	$C_{14}H_{20}N_2O_3$	$CDCl_3$	[42]
2-2-36	7*β*-hydroxycatuabine I	$C_{14}H_{20}N_2O_4$	$CDCl_3$	[42]
2-2-37	vaccinine A	$C_{14}H_{20}N_2O_3$	$CDCl_3$	[42]
2-2-38	vaccinine B	$C_{14}H_{20}N_2O_3$	$CDCl_3$	[42]
2-2-39	catuabine E *N*-oxide	$C_{20}H_{25}N_3O_5$	$CDCl_3$	[42]
2-2-40	pervilleine A	$C_{30}H_{37}NO_{11}$	$CDCl_3$	[43]
2-2-41	pervilleine A *N*-oxide	$C_{30}H_{37}NO_{12}$	$CDCl_3$	[43]
2-2-42	pervilleine B	$C_{30}H_{37}NO_{10}$	$CDCl_3$	[43]
2-2-43	pervilleine C	$C_{32}H_{39}NO_{10}$	$CDCl_3$	[43]
2-2-44	pervilleine D	$C_{32}H_{39}NO_{11}$	$CDCl_3$	[43]
2-2-45	pervilleine E	$C_{28}H_{33}NO_8$	$CDCl_3$	[43]
2-2-46	pervilleine F	$C_{28}H_{33}NO_7$	$CDCl_3$	[43]



	R^1	R^2	R^3
2-2-12	OH	OTmb	Tmc
2-2-13	H	OBz	*cis*-Tmc
2-2-14	H	OBz	H
2-2-15	H	OBz	Tmc



	R^1	R^2
2-2-16	1-methylitaconyl	senecioye
2-2-17	1-methylitaconyl	Ang
2-2-18	1-methylmesaconyl	senecioye
2-2-19	1-methylmesaconyl	Ang
2-2-20	1-methylmesaconyl	Tig
2-2-21	1-methylcitraconyl	senecioye
2-2-22	1-methylcitraconyl	Ang



	R^1	R^2	R^3
2-2-23	Mpc	Pc	H
2-2-24	Mpc	Pc	OH
2-2-25	Mpc	Mpc	H
2-2-26	Mpc	Mpc	OH
2-2-27	Mpc	Mpc	OAc
2-2-28	Hdmb	Mpc	H
2-2-29	Hdmb	Mpc	OH
2-2-30	3-OHPhAc	H	OH

Table 2-2-6: ^{1}H NMR spectroscopic data of hyoscyamine-type tropane alkaloids **2-2-12**~**2-2-15**.

H	2-2-12	2-2-13	2-2-14	2-2-15
1	3.43 br s	3.30 br s	3.59 br s	3.40 br s
2ax	2.34 dd(11.7, 4.2)	2.17 m	2.46 m	2.20 m
2eq	1.73 d(11.7)	1.69 br d(14.7)	1.88 br d(14.6)	1.77 br d(15.4)
3β	5.26 br t(4.5)	5.11 br t(5.0)	4.18 br t(5.0)	5.22 br t(4.7)
4ax	2.29 dd(13.5, 4.4)	2.25 m	2.46 m	2.25 m
4eq	1.69 d(13.5)	1.95 br d(15.3)	2.14 br d(14.5)	1.99 br d(15.3)
5	3.20 br s	3.36 br s	3.66 br s	3.40 br s
6α	4.80 br t(6.0)	2.51 dd(14.1, 7.3)	3.12 dd(14.0, 7.9)	2.78 dd(13.8, 7.4)
6β		2.26 m	2.62 m	2.30 m
7α	5.80 d(6.0)	5.64 dd(7.3, 2.9)	6.02 dd(7.9, 2.7)	5.90 dd(7.4, 3.0)
NMe	2.63 s	2.56 s	2.82 s	2.61 s
OH	2.51 d(6.0)			
	Tmb	Bz	Bz	Bz
2′,6′	7.32 s	8.00 d(7.8)	7.99 d(7.4)	8.02 d(7.9)
3′,5′		7.43 dd(7.8, 7.8)	7.45 dd(7.4, 7.4)	7.46 dd(7.9, 7.9)
4′		7.56 dd(7.8, 7.8)	7.59 dd(7.4, 7.4)	7.57 dd(7.9, 7.9)
m-OMe	3.90			
p-OMe	3.91			
	Tmc	*cis*-Tmc		Tmc
α	6.36 d(16.0)	5.96 d(12.7)		6.36 d(16.0)
β	7.78 d(16.0)	6.88 d(12.7)		7.46 d(16.0)
2′,6′	6.96 s	7.06 s		6.91 s
m-OMe	3.96	3.88		3.95
p-OMe	3.90	3.87		3.90

Notes: ax denotes axial and eq equatorial.

Table 2-2-7: ^{1}H NMR spectroscopic data of hyoscyamine-type tropane alkaloids **2-2-16**~**2-2-19**.

H	2-2-16	2-2-17	2-2-18	2-2-19
1	3.32 br s	3.37 br s	3.34 br s	3.39 br s
2en	1.63 d(15.2)	1.69 d(16.3)	1.75 d(15.4)	1.75 d(15.4)

Table 2-2-7 (continued)

H	2-2-16	2-2-17	2-2-18	2-2-19
2ex	2.11 m	2.15 m	2.19 m	2.19 m
3	4.98 t(5.2)	5.01 t[①]	5.07 t(5.3)	5.08 t(5.3)
4en	1.83 d(14.6)	1.89 (ov)	1.91 d(15.1)	1.93 d(18.1)
4ex	3.13 m	2.19 m	2.21 m	2.22 m
5	3.15 br s	3.23 br s	3.17 br s	3.23 br s
6	5.37 dd(7.8, 3.2)	5.47 m[①]	5.44 dd(7.8, 3.2)	5.52 dd(7.8, 3.2)
7en	2.51 m(7.1)	2.57 m	2.56 m(7.1)	2.60 m(7.1)
7ex	2.13 m	2.23 m	2.19 m	2.23 m
8	2.46 s	2.51 s	2.48 s	2.51 s
10	3.38 d(0.8)	3.42 s	6.75 q(1.6)	6.77 q(1.6)
13	3.75 s	3.77 s	3.80 s	3.81 s
14	5.83 d(1.0), 6.33 d(1.0)	5.87 s, 6.36 s	2.27 d(1.6)	2.28 d(1.6)
10′	5.65 s		5.68 s	
11′		6.14 m[①]		6.13 qq(7.2, 1.5)
12′	2.17 d(1.2)	1.99 s	2.16 d(1.2)	1.97 dq(7.2, 1.5)
13′	1.91 d(1.2)	1.91 s	1.90 d(1.2)	1.87 m(1.5)

[①] Coupling constants not detected due to poor resolution.
Note: en denotes *endo* and ex *exo*, and the same below.

Table 2-2-8: ^{1}H NMR spectroscopic data of hyoscyamine-type tropane alkaloids **2-2-20**~**2-2-22**.

H	2-2-20	2-2-21	2-2-22	H	2-2-20	2-2-21	2-2-22
1	3.35 br s	3.33 br s	3.37 br s	8	2.51 s	2.50 s	2.51 s
2en	1.74 d(15.5)	1.71 d(15.6)	1.75 d(15.2)	10	6.76 q(1.6)	6.09 s	6.10 s
2ex	2.18 m	2.17 m	2.16 m	13	3.80 s	3.80 s	3.81 s
3	5.08 t(5.2)	5.04 t[①]	5.04 t[①]	14	2.27 d(1.6)	2.07 s	2.08 s
4en	1.93 d(15.2)	1.96 (ov)	1.98 (ov)	10′		5.70 s	
4ex	2.21 m	2.20 m	2.20 m	11′	6.87 qq(7.0, 1.5)		6.16 m[①]
5	3.22 br s	3.23 br s	3.24 br s	12′	1.80 dd(7.1, 1.0)	2.19 s	2.00 s
6	5.49 dd(7.7, 3.2)	5.43 m[①]	5.51 m[①]	13′	1.83 s	1.93 s	1.90 s
7en	2.59 m(7.1)	2.57 m	2.59 m				
7ex	2.22 m	2.22 m	2.23 m				

[①] Coupling constants not detected due to poor resolution.

Table 2-2-9: ^{1}H NMR spectroscopic data of hyoscyamine-type tropane alkaloids **2-2-23**~**2-2-26**.

H	2-2-23	2-2-24	2-2-25	2-2-26
1	3.40 br s	3.56 br s	3.50 br s	3.33 br s
2ex	2.22 br dd(15.1, 4.9)	2.43 m	2.34 m	2.27 br dd(17.1, 4.9)
2en	1.74 d(15.1)	1.93 d(16.1)	1.82 d(15.1)	1.72 d(17.1)
3β	5.23 t(4.9)	5.27 t(4.9)	5.23 t(4.9)	5.24 t(4.9)

Table 2-2-9 (continued)

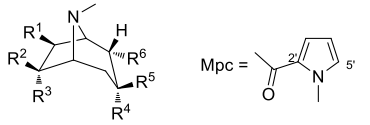
H	2-2-23	2-2-24	2-2-25	2-2-26
4ex	2.25 br dd(15.1, 4.9)	2.47 m	2.34 m	2.29 br dd(18.1, 4.9)
4en	1.96 d(15.1)	1.96 d(15.6)	2.06 d(15.6)	1.76 d(18.1)
5	3.34 br s	3.38 br s	3.44 br s	3.20 br s
6α	5.81 dd(7.3, 2.9)	5.80 d(6.4)	5.77 dd(7.6, 3.2)	5.71 d(5.9)
7α	2.76 dd(13.9, 7.3)	4.88 d(6.4)	2.77 dd(14.4, 7.6)	4.79 d(5.9)
7β	2.28 m		2.34 m	
NMe	2.58 s	2.78 s	2.63 s	2.61 s
	Mpc	Mpc	Mpc	Mpc
3′	7.08 dd(3.9, 2.0)	7.07 dd(3.9, 1.5)	7.05 dd(3.9, 2.0)	7.13 dd(3.9, 2.0)
4′	6.15 dd(3.9, 2.4)	6.30 dd(3.9, 2.4)	6.16 dd(3.9, 2.4)	6.18 dd(3.9, 2.0)
5′	6.80 t(2.0)	7.06 dd(2.4, 1.5)	6.80 t(2.4, 2.0)	6.81 t(2.0)
NMe	3.94 s	3.95 s	3.93 s	3.93 s
	Pc	Pc	Mpc	Mpc
3″	6.91 dd(3.9, 2.0)	7.10 dd(3.9, 2.0)	6.91 dd(3.9, 1.5)	7.03 dd(3.9, 2.0)
4″	6.25 dd(3.9, 2.0)	6.13 dd(3.9, 2.4)	6.10 dd(3.9, 2.4)	6.11 dd(3.9, 2.0)
5″	6.94 dd(3.9, 2.0)	6.86 t(2.0)	6.79 t(2.4, 2.0)	6.79 t(2.0)
NH	9.58 br s			
NMe			3.94 s	3.94 s

Table 2-2-10: ^{1}H NMR spectroscopic data of hyoscyamine-type tropane alkaloids **2-2-27**~**2-2-30**.

H	2-2-27	2-2-28	2-2-29	2-2-30
1	3.35 br s	3.45 br s	3.50 br s	3.02 br s
2ex	2.28 br dd(15.1, 5.1)	2.29 br dd(15.1, 4.9)	2.55 m	2.17 dd(15.6, 4.9)
2en	1.87 d(15.1)	1.86 d(15.1)	2.04 d(13.7)	1.52 d(15.6)
3β	5.28 t(5.1)	5.31 t(4.9)	5.36 t(4.9)	4.97 t(4.9)
4ex	2.25 br dd(14.6, 5.1)	2.32 br dd(15.6, 4.9)	2.58 m	2.17 dd(15.6, 4.9)
4en	1.92 d(14.6)	2.07 d(15.6)	2.07 d(15.1)	1.52 d(15.6)
5	3.35 br s	3.40 br s	3.72 br s	3.02 br s
6α	5.88 d(6.3)	5.86 dd(7.3, 3.2)	5.91 d(6.4)	4.18 s
7α	5.81 d(6.3)	2.77 dd(14.2, 7.3)	4.90 d(6.4)	4.18 s
7β		2.30 m		
NMe	2.65 s	2.62 s	2.90 s	2.51 s
	Mpc	Hdmb	Hdmb	3-OHPhCH_2CO
2′		7.39 s	7.39 s	6.74 m
3′	7.22 dd(3.9, 2.0)			
4′	6.18 dd(3.9, 2.0)			6.76 m
5′	6.80 t(2.0)			7.18 dd(8.8, 7.3)
6′		7.39 s	7.39 s	6.76 m
NMe	3.92 s			
b′				3.55 s
OMe		3.99 s	4.01 s	

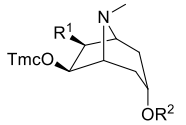
Table 2-2-10 (continued)

H	2-2-27	2-2-28	2-2-29	2-2-30
	Mpc	Mpc	Mpc	
3″	6.93 dd(3.9, 2.0)	6.91 dd(3.9, 2.0)	7.04 dd(3.9, 2.0)	
4″	6.09 dd(3.9, 2.0)	6.11 dd(3.9, 2.0)	6.13 dd(3.9, 2.0)	
5″	6.78 t(2.0)	6.81 t(2.0)	6.87 t(2.0)	
NMe	3.93 s	3.90 s	3.92 s	
	OAc			
b‴	2.04 s			



Mpc =

	R^1	R^2	R^3	R^4	R^5	R^6	Other
2-2-31	OMpc	H	H	OH	H	H	
2-2-32	OMpc	H	H	H	OH	H	
2-2-33	OMpc	OH	H	OH	H	H	
2-2-34	OMpc	H	OH	OH	H	H	
2-2-35	OH	H	H	OMpc	H	H	
2-2-36	OH	OH	H	OMpc	H	H	
2-2-37	H	H	H	OH	H	OMpc	
2-2-38	OH	H	H	H	H	OMpc	
2-2-39	OMpc	H	H	OMpc	H	H	N^+-O^-



	R^1	R^2	Other
2-2-40	OH	Tmb	
2-2-41	OH	Tmb	N^+-O^-
2-2-42	H	Tmb	
2-2-43	H	Tmc	
2-2-44	OH	Tmc	
2-2-45	H	3-OHPhAc	
2-2-46	H	PhAc	

Table 2-2-11: ^{1}H NMR spectroscopic data of hyoscyamine-type tropane alkaloids **2-2-31**~**2-2-35**.

H	2-2-31	2-2-32	2-2-33	2-2-34	2-2-35
1	3.35 m(3.9, 3.4)	3.60 br s	3.16 br s	3.35 m(6.4)	3.48 m(3.4)
2ex	2.12 m(4.9)	1.88 m	2.20 m	2.17 dt(15.1, 4.9)	2.32 m
2en	1.66 d(14.7)	1.88 m	1.61 d(17.6)	1.89 d(15.1)	1.69 d(15.6)
3*α*		3.78 tt(10.8, 5.9)			
3*β*	4.08 t(4.9)		4.11 t(4.9)	4.08 t(4.9)	5.15 t(5.4)
4ex	2.16 m(4.9)	2.14 m	2.20 m	2.20 dt(15.1, 4.9)	2.33 m
4en	1.91 d(14.7)	1.88 m	1.64 d(17.6)	1.84 d(15.1)	1.83 d(15.6)
5	3.26 br s	3.51 br s	3.27 br s	3.11 br s	3.23 br s
6*α*	5.79 dd(7.3, 3.4)	5.24 dd(5.4)①	5.75 d(6.4)	5.42 d(2.4)	4.72 dd(7.3, 2.9)
7*α*	2.75 dd(13.7, 7.3)	2.33 d(5.4)	4.84 d(6.4)		2.75 dd(14.2, 7.3)
7*β*	2.24 m	2.32 d(3.4)		4.62 d(6.4)	2.08 dd(14.2, 7.3)
NMe	2.55 s	2.67 s	2.58 s	2.54 s	2.68 s
	Mpc	Mpc	Mpc	Mpc	Mpc
3′	6.90 dd(3.9, 2.0)	6.90 dd(3.9, 2.0)	7.02 dd(3.9, 2.0)	7.00 dd(3.9, 2.0)	6.91 dd(3.9, 2.0)
4′	6.09 dd(3.9, 2.4)	6.11 dd(3.9, 2.4)	6.10 dd(3.9, 2.4)	6.11 dd(3.9, 2.4)	6.14 dd(3.9, 2.4)
5′	6.77 t(2.0)	6.82 t(2.0)	6.79 t(2.0)	6.81 t(2.0)	6.83 dd(2.4, 2.0)
NMe	3.93 s	3.92 s	3.92 s	3.92 s	3.93 s

① Only one coupling constant in the original literature.

Table 2-2-12: ^{1}H NMR spectroscopic data of hyoscyamine-type tropane alkaloids **2-2-36**~**2-2-39**.

H	2-2-36	2-2-37	2-2-38	2-2-39
1	3.21 br s	3.26 br d①	3.33 br d(6.4)	4.07 br s
2ex	2.32 m	1.86 m	1.88 m	2.53 m
2en	1.76 d(15.6)	1.96 m	1.33 m	2.20 d(16.6)
3*α*			1.36 m(6.4)	
3*β*	5.13 t(4.9)	3.87 dd(8.8)	1.97 m	5.27 t(5.4)
4ex	2.32 m	4.98 dd(8.8, 3.9)	5.12 m(7.3, 3.4)	2.51 m
4en	1.76 d(15.6)			2.51 m
5	3.21 br s	3.37 br d①	3.18 d(3.4)	4.07 br s
6*α*	4.62 s	1.92 m	4.55 dd(7.3, 3.4)	5.86 dd(8.3, 3.4)
6*β*		1.92 m		
7*α*	4.62 s	1.59 m	2.09 m(7.3)	2.74 dd(13.2, 8.8)
7*β*		2.08 m	2.05 m(3.4)	3.19 m
NMe	2.63 s	2.42 s	2.63 s	3.42 s
	Mpc	Mpc	Mpc	Mpc
3′	6.90 br s	6.90 dd(3.9, 2.0)	6.95 dd(3.9, 2.0)	7.07 dd(3.9, 1.5)
4′	6.13 br s	6.11 dd(3.9, 2.4)	6.10 dd(3.9, 2.4)	6.19 dd(3.9, 2.4)
5′	6.88 br s	6.80 dd(3.9, 2.0)	6.78 dd(2.4, 2.0)	6.86 t(2.0)
NMe	3.93 s	3.91 s	3.92 s	3.96 s
				Mpc
3″				7.12 dd(3.9, 1.5)
4″				6.11 dd(3.9, 2.4)
5″				6.78 t(2.0)
NMe				3.95 s

① No coupling constant in the original literature.

Table 2-2-13: ^{1}H NMR spectroscopic data of hyoscyamine-type tropane alkaloids **2-2-40**~**2-2-43**.

H	2-2-40	2-2-41	2-2-42	2-2-43
1	3.36 br s	4.32 br s	3.29 br s	3.31 br s
2ax	2.37 m	2.46~2.56 m	2.33 dd①	2.26 m
2eq	1.74 br d(12.6)	2.31 br d(17.3)②	1.93 br d(12.6)	1.90 br d(15.3)
3*β*	5.37 br t(4.5)	5.36 br t(4.5)	5.34 br dd(5.4,4.5)	5.21 br t(5.0)
4ax	2.37 m	2.46~2.56 m	2.33 br dd(11.4, 5.4)	2.24 m
4eq	1.74 br d(12.6)	2.38 br d(16.9)②	1.72 br d(11.7)	1.69 br d(15.3)
5	3.22 br s	3.85 br s	3.41 br d(7.4)	3.39 br d(7.2)
6*α*	4.77 d(6.9)	4.81 d(6.9)	2.77 dd(13.6, 7.4)	2.72 dd(14.1, 7.6)
6*β*			2.20~2.22 m	2.22~2.26 m
7*α*	5.72 d(6.9)	5.97 d(6.9)	5.77 dd(7.5, 2.7)	5.73 dd(7.6, 2.7)
NMe	2.62 s	3.31 s	2.61 s	2.59 s
Tmc				R^2/R^3
a	6.45 d(15.8)	6.54 d(15.9)	6.37 d(15.9)	6.35/6.38 d(15.8)
b	7.64 d(15.8)	7.67 d(15.9)	7.56 d(15.8)	7.31/7.56 d(15.8)
ortho	6.76 s	6.77 s	6.57 s	6.74/6.88 s
OMe	3.89~4.00 (3 s)	3.88~3.97 (3 s)	3.88~3.98 (3 s)	3.88~3.94 (6 s)

Table 2-2-13 (continued)

H	2-2-40	2-2-41	2-2-42	2-2-43
Tmb				
2′	7.35 s	7.36 s	7.38 s	
6′	7.35 s	7.36 s	–	
OMe	3.89~4.00 (3 s)	3.88~3.97 (3 s)	3.88~3.98 (3 s)	

① No coupling constant in the original literature. ② Assignments may be exchanged.

Table 2-2-14: ^{1}H NMR spectroscopic data of hyoscyamine-type tropane alkaloids **2-2-44**~**2-2-46**.

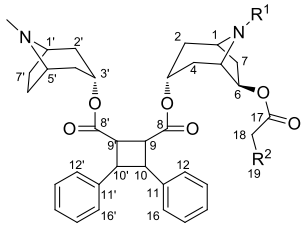
H	2-2-44	2-2-45	2-2-46
1	3.32 br s	3.27 br s	3.18 s
2ax	2.37 m	2.13 m	2.00~2.15 m
2eq	1.69 dd(15.3, 7.3)	1.45 br t(15.2)	1.48 br d(15.2)①
3β	5.24 br t(4.5)	5.07 br t(5.2)	5.05 br t(5.2)
4ax	2.31 m	2.20 m	2.00~2.15 m
4eq	1.69 dd(15.3, 7.3)	1.71 br d(15.2)	1.78 br d(15.2)①
5	3.18 br s	3.27 br s	3.26 br d(6.3)
6α	4.77 d(6.3)	2.41 dd(14.1, 7.6)	2.19 dd(14.1, 7.5)
6β		2.01 m	2.00~2.15 m
7α	5.72 d(6.3)	5.66 dd(7.5, 3.0)	5.35 dd(7.5, 3.0)
NMe	2.60 s	2.55 s	2.50 s
Tmc	R^2/R^3		
a	6.36/6.47 d(15.9)	6.40 d(15.9)	6.37 d(15.9)
b	7.62/7.74 d(15.9)	7.66 d(15.9)	7.58 d(15.9)
ortho	6.75/6.92 s	6.77 s	6.76 s
OMe	3.90~3.96 (6 s)	3.89~3.90 (3 s)	3.88~3.90 (3 s)
		3-OHPhCH_2CO	PhCH_2CO
CH_2		3.56 s	3.66 s
2′		7.19 d(2.2)	7.25~7.36 m
3′			7.25~7.36 m
4′,5′		6.80~6.84 m	7.25~7.36 m
6′		7.22 d(7.8)	7.25~7.36 m

① Assignments may be exchanged.

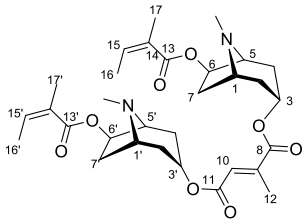
2.2.3 Dimeric tropane alkaloids

Table 2-2-15: Cos, MFs, and TSs of dimeric tropane alkaloids **2-2-47**~**2-2-49**.

No.	Compounds	MFs	Test solvents	References
2-2-47	mooniine A	$C_{36}H_{44}N_2O_6$	$CDCl_3$	[44]
2-2-48	mooniine B	$C_{36}H_{44}N_2O_6$	$CDCl_3$	[44]
2-2-49	schizanthine X	$C_{31}H_{44}N_2O_8$	$CDCl_3$, C_6D_6	[45]



	R^1	R^2
2-2-47	H	Me
2-2-48	Me	H



2-2-49

Table 2-2-16: ^{1}H NMR spectroscopic data of dimeric tropane alkaloids **2-2-47**~**2-2-48**.

H	2-2-47	2-2-48
1	4.13 br s	3.72 br m
2ax, 2eq	2.33 m①, 2.10 m①	2.21 m, 2.15 m
3	5.31 m	4.67 m($W_{1/2}$ = 16 Hz)
4ax, 4eq	2.30 m①, 2.10 m①	2.40 m, 1.75 m
5	4.14 br s	3.90 br m
6en	5.35 m	5.16 dd(7.5, 2.8)
7ex	2.78 br d(14)	2.75 br d(14.6)
7en	2.13 m	2.15 m
1′, 5′	4.11~4.16 br s	3.42 br s①, 3.52 br s①
2′ax, 2eq, 4′ax, 4′eq	2.40 m①, 2.20 m①, 1.83 m①	2.15 m①, 1.80 m①, 1.76 m①
3′	5.29 m	4.76 m($W_{1/2}$ = 15 Hz)
6′ex, 6′en, 7′ex, 7′en	2.30 m①, 2.00 m①	2.20 m①, 2.00 m①
9, 9′	2.34 m①, 2.30 m①	2.34 m①, 2.10 m①
10, 10′	2.15 m①, 2.05 m①	2.15 m
12, 12′, 16, 16′	8.06 dd(8.40, 1.36)①, 7.99 dd(8.47, 1.30)①	8.15 dd(8.40, 1.37)①
14, 14′	7.58 tt(7.40, 1.10)①	7.57 tt(7.31, 1.34)①
13, 13′, 15, 15′	7.45 t(7.64)	7.40~7.45 (ov)
18	2.10 m	2.13 s
19	1.25 m	
N′Me	2.38 s	2.65 s①
NMe		2.75 s①

① Assignments not certain and data may be exchanged in each column.
Notes: ax denotes axial, eq equatorial, en *endo*, and ex *exo*.

Table 2-2-17: ^{1}H NMR spectroscopic data of dimeric tropane alkaloids **2-2-49**.

H	2-2-49($CDCl_3$)	2-2-49(C_6D_6)	H	2-2-49($CDCl_3$)	2-2-49(C_6D_6)
1	3.16 br s($W_{1/2}$ = 15.0)	3.01 t	6′	5.51 dd(7.8, 2.4)	5.82 dd
2	1.70①, 2.20①		7′	2.20, 2.50 m	2.65 ddd
3	5.08 t	5.10 t	10	6.79 q(1.7)	–
4	1.90①, 2.20①	–	12	2.33 d(1.7)	–
5	3.30 m	3.08 m	15	6.01 qq(7.5, 1.6)	–
6	5.47 dd(7.8, 2.4)	5.70 dd	15′	6.01 qq(7.5, 1.6)	–

Table 2-2-17 (continued)

H	2-2-49($CDCl_3$)	2-2-49(C_6D_6)	H	2-2-49($CDCl_3$)	2-2-49(C_6D_6)
7	2.20, 2.50 m	2.65 dd	16	2.06 d(7.5)	–
1′	3.19 br s	3.01 t	16′	2.08 d(7.5)	–
2′	1.70①, 2.20①	–	17	1.91 d(1.6)	–
3′	5.12 t	5.14 t	17′	1.91 d(1.6)	–
4′	1.90①, 2.20①	–	NMe	2.48 s	–
5′	3.30 m	3.08 m	N′Me	2.49 s	–

① Peak-type not shown in the literatures.

Bibliography

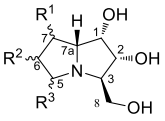
[35] Butler MS, Katavic PL, Davis RA, et al. J Nat Prod, 2000, 63: 688.
[36] Katavic PL, Butler MS, Quinn RJ, et al. Phytochemistry, 1999, 52: 529.
[37] Asano N, Kato A, Yokoyama Y, et al. Carbohydr Res, 1996, 284: 169.
[38] Asano N, Kato A, Oseki K, et al. Eur J Biochem, 1995, 229: 369.
[39] Chavez D, Cui B, Chai HB, et al. J Nat Prod, 2002, 65: 606.
[40] Cretton S, Glauser G, Humam M, et al. J Nat Prod, 2010, 73: 844.
[41] Zanolari B, Guilet D, Marston A, et al. J Nat Prod, 2003, 66: 497.
[42] Zanolari B, Guilet D, Marston A, et al. J Nat Prod, 2005, 68: 1153.
[43] Silva GL, Cui B, Chavez D, et al. J Nat Prod, 2001, 64: 1514.
[44] Att-ur-Rahman KKF, Nighat F, et al. Phytochemistry, 1998, 48: 377.
[45] Munoz O, Hartmann R, Breitmaier E. J Nat Prod, 1991, 54: 1094.

2.3 Pyrrolizidine alkaloids

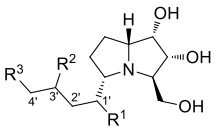
Table 2-3-1: Cos, MFs, and TSs of pyrrolizidine alkaloids **2-3-1**~**2-3-38**.

No.	Compounds	MFs	Test solvents	References
2-3-1	hyacinthacine B_7	$C_9H_{17}NO_4$	D_2O[①]	[46]
2-3-2	hyacinthacine C_2	$C_9H_{17}NO_5$	D_2O[①]	[46]
2-3-3	hyacinthacine C_3	$C_9H_{17}NO_5$	D_2O[①]	[46]
2-3-4	hyacinthacine C_4	$C_9H_{17}NO_5$	D_2O[①]	[46]
2-3-5	*α*-5-*C*-(3-hydroxybutyl)hyacinthacine A_1	$C_{12}H_{23}NO_4$	D_2O	[47]
2-3-6	*α*-5-*C*-(1,3-dihydroxybutyl)hyacinthacine A_1	$C_{12}H_{23}NO_5$	D_2O	[47]
2-3-7	*α*-5-*C*-(1,3,4-trihydroxybutyl)hyacinthacine A_1	$C_{12}H_{23}NO_6$	D_2O	[47]
2-3-8	hyacinthacine C_5	$C_9H_{17}NO_5$	D_2O[①]	[46]
2-3-9	*α*-5-*C*-(3-hydroxybutyl)-hyacinthacine A_2	$C_{12}H_{23}NO_4$	D_2O[①]	[46]
2-3-10	*α*-5-*C*-(3-hydroxybutyl)-7-*epi*-australine	$C_{12}H_{23}NO_5$	D_2O	[47]
2-3-11	bohemamine B	$C_{14}H_{20}N_2O_3$	DMSO-d_6	[48]
2-3-12	bohemamine C	$C_{14}H_{20}N_2O_3$	DMSO-d_6	[48]
2-3-13	5-chlorobohemamine C	$C_{14}H_{19}ClN_2O_3$	DMSO-d_6	[48]
2-3-14	bohemamine	$C_{14}H_{18}N_2O_3$	DMSO-d_6	[48]
2-3-15	amphorogynine A	$C_{19}H_{25}NO_6$	$CDCl_3$	[49]
2-3-16	amphorogynine B	$C_{19}H_{25}NO_6$	$CDCl_3$	[49]
2-3-17	amphorogynine C	$C_{19}H_{25}NO_6$	$CDCl_3$	[49]
2-3-18	amphorogynine D	$C_8H_{13}NO_3$	$CDCl_3$, CD_3OD	[49]
2-3-19	hadienecine	$C_8H_{15}NO_3$	C_5D_5N	[50]
2-3-20	rosmarinine	$C_{18}H_{27}NO_6$	$CDCl_3$	[50]
2-3-21	hadiensine	$C_{18}H_{27}NO_6$	$CDCl_3$	[50]
2-3-22	12-*O*-acetylhadiensine	$C_{20}H_{29}NO_7$	$CDCl_3$	[50]
2-3-23	12-*O*-acetylrosmarinine	$C_{20}H_{29}NO_7$	$CDCl_3$	[50]
2-3-24	petitianine	$C_{18}H_{27}NO_7$	CD_3OD	[50]
2-3-25	1-*O*-acetylhadiensine	$C_{20}H_{29}NO_7$	$CDCl_3$	[50]
2-3-26	1,12-di-*O*-acetylhadiensine	$C_{22}H_{31}NO_8$	$CDCl_3$	[50]
2-3-27	2-*O*-acetylrosmarinine	$C_{20}H_{29}NO_7$	$CDCl_3$	[50]
2-3-28	2,12-di-*O*-acetylrosmarinine	$C_{22}H_{31}NO_8$	$CDCl_3$	[50]
2-3-29	neorosmarinine	$C_{18}H_{27}NO_6$	$CDCl_3$	[50]
2-3-30	12-*O*-acetylneohadiensine	$C_{20}H_{29}NO_7$	$CDCl_3$	[50]
2-3-31	callosine	$C_{21}H_{31}NO_7$	$CDCl_3$	[51]
2-3-32	11-*O*-acetylbulgarsenine	$C_{20}H_{29}NO_6$	$CDCl_3$	[51]
2-3-33	11-*O*-acetyl bulgarsenine *N*-oxide	$C_{20}H_{29}NO_7$	$CDCl_3$	[51]
2-3-34	bulgarsenine	$C_{18}H_{27}NO_5$	$CDCl_3$	[51]
2-3-35	*N*-chloromethyl bulgarsenine chloride	$C_{19}H_{29}Cl_2NO_5$	CD_3OD	[51]
2-3-36	megalanthonine	$C_{15}H_{27}NO_5$	$CDCl_3$	[52]
2-3-37	echimidine	$C_{20}H_{31}NO_7$	$CDCl_3$	[53]
2-3-38	3′-acetylechimidine	$C_{22}H_{33}NO_8$	$CDCl_3$	[53]

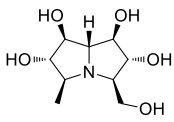
[①] TSP (sodium 3-(trimethylsilyl)propionate) as internal standard.



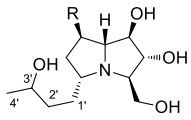
	R^1	R^2	R^3
2-3-1	β-OH	H	β-Me
2-3-2	α-OH	H	α-CH_2OH
2-3-3	β-OH	H	α-CH_2OH
2-3-4	α-OH	β-OH	α-Me



	R^1	R^2	R^3
2-3-5	H	OH	H
2-3-6	OH	OH	H
2-3-7	OH	OH	OH



2-3-8



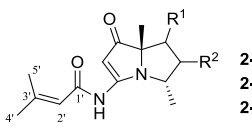
2-3-9 R = H
2-3-10 R = OH

Table 2-3-2: ^{1}H NMR spectroscopic data of pyrrolizidine alkaloids **2-3-1**~**2-3-4**.

H	2-3-1	2-3-2	2-3-3	2-3-4
1	4.35 t(4.4)	4.16 t(4.4)	4.32 t(4.4)	4.18 t(4.0)
2	3.97 dd(4.4, 7.6)	3.85 dd(4.4, 7.6)	4.04 dd(4.4, 9.5)	4.00 dd(4.0, 8.7)
3	3.29 ddd(7.6, 5.5, 3.5)	3.33 ddd(5.0, 5.7, 7.6)	3.50 m	3.38 dt (4.6, 8.7)
5	3.22 m	3.24 m	3.84 (ov)	2.96 dq(6.9, 9.2)
6	α 1.68 m, β 2.16 m	1.78 m, 2.03 m	1.93 m, 2.07 m	3.90 dd(8.2, 9.2)
7	4.50 m	4.40 m	4.56 ddd(2.5, 4.4, 2.5)	4.15 t(8.2)
7a	3.45 dd(4.4, 7.6)	3.39 dd(4.4, 6.9)	3.85 (ov)	3.57 dd(3.7, 8.2)
8	3.57 dd(3.5, 11.5)	3.54 dd(5.7, 12.0)	3.69 dd(3.2, 12.6)	3.65 d(4.6)
	3.63 dd(3.5, 11.5)	3.61 dd(5.0, 12.0)	3.85 dd(3.2, 12.6)	3.65 d(4.6)
1′	1.25 d(7.0)	3.66 dd(5.0, 12.0)	3.79 dd(6.2, 12.0)	1.29 d(6.9)
		3.85 dd(6.9, 12.0)	3.84 (ov)	

Table 2-3-3: ^{1}H NMR spectroscopic data of pyrrolizidine alkaloids **2-3-5**~**2-3-7**.

H	2-3-5	2-3-6	2-3-7
1	4.17 dd(4.6, 3.9)	3.99 t(3.7)	3.99 t(3.7)
2	4.12 dd(8.5, 4.6)	4.10 dd(9.1, 3.7)	4.10 dd(9.2, 3.7)
3	3.33 ddd(8.5, 5.0, 4.5)	3.39 ddd(9.1, 6.4, 3.7)	3.37 ddd(9.2, 6.9, 3.7)
5	3.28 m	3.16 m	3.15 m
6	α 1.99 m	α 2.12 m	α 2.11 m
	β 1.73~1.81 m	β 1.89~2.05 m	β 1.96 m
7	α 2.06 m	α 1.89~2.05 m	α 1.93 m
	β 1.73~1.81 m	β 1.76 m	β 1.75 m
7a	3.83 m	3.70 dt(7.8, 3.7)	3.67 m
8	3.78 dd(11.9, 5.0)	3.85 dd(11.9, 3.7)	3.84 dd(11.4, 4.1)
	3.75 dd(11.9, 4.5)	3.68 dd(11.9, 6.4)	3.67 dd(11.4, 6.9)
1′	1.88 m	4.38 ddd(9.6, 3.2, 1.8)	4.42 ddd(9.7, 2.8, 2.3)
	1.44~1.52 m		
2′	1.54 m	1.67 ddd(14.7, 9.6, 3.7)	1.66 ddd(14.6, 9.7, 3.2)
	1.44~1.52 m	1.49 ddd(14.7, 9.6, 3.2)	1.45 ddd(14.6, 10.1, 2.8)
3′	3.84 m	3.98 ddq(9.6, 6.4, 3.7)	3.87 dddd(10.1, 6.9, 4.2, 3.2)
4′	1.19 d(6.4)	1.22 d(6.4)	3.61 dd(11.4, 4.2)
			3.49 dd(11.4, 6.9)



	R^1	R^2
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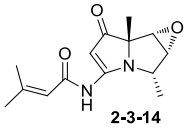
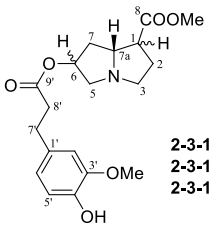
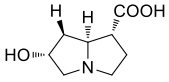
2-3-12	α-OH	H
2-3-13	α-OH	β-Cl



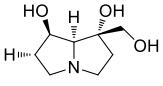
2-3-14



2-3-15	1β-H	6β-H
2-3-16	1α-H	6α-H
2-3-17	1β-H	6α-H



2-3-18



2-3-19

Table 2-3-4: ^{1}H NMR spectroscopic data of pyrrolizidine alkaloids **2-3-8**~**2-3-10** and **2-3-19**.

H	2-3-8	2-3-9	2-3-10	2-3-19
1	4.01 t (7.6)	3.76 t (6.9)	3.82 dd(6.4, 6.0)	
2	3.80 t (7.6)	3.96 t (6.9)	3.97 t(6.4)	2.70 ddd(9.0, 12, 12) *ca*. 2.10 m
3	3.01 m	2.18 ddd(5.0, 5.7, 6.9)	3.10 ddd(6.4, 6.0, 4.1)	4.21 ddd(7.2, 11, 11) 3.23 t(10)
5	2.81 dq (6.9, 7.6)	3.09 m	3.35 m	4.10 m, 3.37 m
6	3.60 t (7.6)	α 1.62 m β 1.93 m	α 1.79 ddd(13.7, 11.4, 5.5) β 1.89 ddd(13.7, 5.0, 1.8)	*ca*. 2.05 m
7	3.99 t (7.6)	α 1.87 m, β 1.95 m	4.33 dt(5.5, 1.8)	4.85 br s
7a	3.24 t (7.6)	3.40 m	3.22 dd(6.0, 1.8)	4.50 d(3.2)
8	3.50 dd (5.3, 12.0) 3.55 dd (4.1, 12.0)	3.73 dd (5.0, 12.0) 3.75 dd (5.7, 12.0)	3.71 dd(11.5, 4.1) 3.65 dd(11.5, 6.0)	4.56 d(11) 4.37 d(11)
1′	1.13 d(6.9)	1.46 m, 1.95 m	1.62~1.46 m	
2′		1.46 m, 1.62 m	1.82 m, 1.46~1.62 m	
3′		3.86 m	3.87 m	
4′		1.19 d(6.0)	1.20 d(6.4)	

Table 2-3-5: ^{1}H NMR spectroscopic data of pyrrolizidine alkaloids **2-3-11**~**2-3-14**.

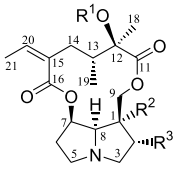
H	2-3-11	2-3-12	2-3-13	2-3-14
1	α 1.55 dd(12.2, 10.1) β 1.69 dd(12.2, 5.9)	3.84 t(3.9)	3.99 ddd (5.0, 4.4, 1.7)	3.60 d(3.4)
2	4.44 dddd (10.1, 5.9, 6.4, 3.5)	α 1.75 d(13.2) β 2.57 ddd(13.2, 8.7, 3.9)	4.21 t(2.4)	3.73 br d(3.4)
3	3.86 dq(6.5, 6.4)	3.92 dq(6.7, 8.7)	4.07 dq(6.8, 2.4)	3.8 br q(6.7)
6	5.56 s	5.40 s	5.55 s	5.34 s
8	1.13 s	1.07 s	1.41 s	1.20 s
9	0.90 d(6.5)	1.25 d(6.7)	1.32 d(6.8)	1.35 d(6.7)
2′	5.98 dq(1.0, 9.0)	5.98 br s	5.95 dq(1.0)	5.90 dq(1.3, 1.0)

Table 2-3-5 (continued)

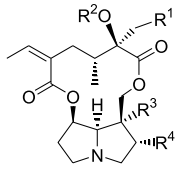
H	2-3-11	2-3-12	2-3-13	2-3-14
4′	1.88 d(0.9)	1.88 br s	1.89 d(1.0)	1.87 d(1.0)
5′	2.12 d(1.0)	2.12 br s	2.14 d(1.0)	2.12 d(1.3)
NH	10.24 br s	10.24 br s	10.33 br s	10.10 br s
OH	4.82 d(3.5)	4.92 d(3.5)	5.82 d(5.0)	

Table 2-3-6: ^{1}H NMR spectroscopic data of pyrrolizidine alkaloids **2-3-15**~**2-3-18**.

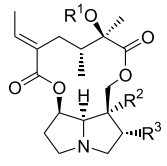
H	2-3-15	2-3-16	2-3-17	2-3-18($CDCl_3$)	2-3-18(CD_3OD)
1	3.18 ddd(8)	2.63 m	3.14 ddd(8)	3.09 m	3.31 m
2	*α* 2.22 m	2.20 m	*α* 2.09 m	2.34 m	*α* 2.44 m
	β 1.90 m		*β* 1.95 m		*β* 2.54 m
3	*α* 2.78 m	*α* 2.65 m	*α* 2.79 m	2.96 m	3.23 m
	β 3.05 ddd(11, 8, 6)	*β* 3.28 ddd(6)	*β* 3.06 m	3.23 m	3.37 m
5	*α* 2.72 dd(12, 6)	*α* 2.77 dd(12, 4.5)	*α* 2.86 m	*α* 2.85 dd(13, 3)	*α* 3.08 dd(13, 2)
	β 3.26 dd(12, 5)	*β* 3.16 dd(12)	*β* 3.21 dd(12)	*β* 3.48 dd(13)	*β* 3.62 dd(13, 3)
6	5.15 dddd(6)	5.36 m	5.32 m	4.74 br s	4.95 m
7	*α* 1.48 ddd(14, 8.5)	*α* 1.88 ddd(13, 7.5)	*α* 1.65 ddd(14, 7.5)	2.18 m	*α* 2.44 m
	β 2.18 ddd(14, 8)	*β* 2.16 m	*β* 1.86 ddd(14, 7)		*β* 2.30 m
7a	3.75 ddd(8)	3.85 m	3.86 ddd(6)	3.50 m	3.72 m
COOMe	3.64 s	3.68 s	3.66 s		
2′	6.64 d(2)	6.67 d(2)	6.65 d(2)		
5′	6.74 d(8)	6.78 d(8)	6.77 d(8)		
6′	6.62 dd(8, 2)	6.63 dd(8, 2)	6.64 dd(8)		
7′	2.18 t(7)	2.83 t(7.5)	2.18 t(7.5)		
8′	2.53 t(7)	2.55 t(7.5)	2.53 t(7.5)		
3′-OMe	3.81 s	3.83 s	3.83 s		



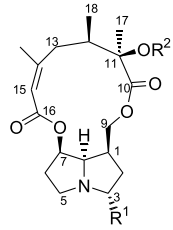
	R^1	R^2	R^3
2-3-20	H	H	OH
2-3-21	H	OH	H
2-3-22	Ac	OH	H
2-3-23	Ac	H	OH



	R^1	R^2	R^3	R^4
2-3-24	OH	H	H	OH
2-3-25	H	H	OAc	H
2-3-26	H	Ac	OAc	H
2-3-27	H	H	H	OAc
2-3-28	H	Ac	H	OAc



	R^1	R^2	R^3
2-3-29	H	H	OH
2-3-30	Ac	OH	H



	R^1	R^2
2-3-31	CH_2COOMe	H
2-3-32	H	Ac
2-3-34	H	H

Table 2-3-7: ^{1}H NMR spectroscopic data of pyrrolizidine alkaloids **2-3-20**~**2-3-23**.

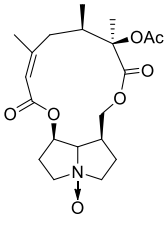
H	2-3-20	2-3-21	2-3-22	2-3-23
1	2.50 m			2.45 m
2	4.23 m	2.21 m, 2.19 m	*ca*. 2.08 m	4.31 q(*ca*. 8)
3	3.07 dd(7.6, 11.4)	3.27 m	3.15 m	3.09 dd(7.4, 11.1)
	2.92 dd(8.1, 11.1)	2.78 m	2.84 m	2.91 dd(8, 11.1)
5	3.27 m, 2.58 m	3.11 m, 2.72 m	3.02 m, 2.78 m	3.30 t(8.7), *ca*. 2.5 m
6	*ca*. 2.0 m	2.19 m	2.26 m	2.30 dd(6.4, 14)
	2.26 m		2.08 m	2.05 m
7	5.05 m	5.30 q(4.9)	5.30 q(*ca*. 5)	4.98 m
8	3.55 dd(3.2, 7.9)	3.41 d(4.9)	3.31 d(4.9)	3.56 dd(3.1, 7.6)
9	4.90 dd(5.2, 12.6)	4.57 d(11.6)	4.41 d(11.6)	4.88 dd(5, 12)
	4.12 d(12.6)	4.04 d(11.6)	4.17 d(11.6)	4.10 dd(*ca*. 1, 12)
13	*ca*. 1.8 m	*ca*. 1.95 m	1.83 m	*ca*. 1.72 m
14	1.94 dd(9.6, 13.2)	*ca*. 1.97 m	2.53 dd(4.5, 14.3)	*ca*. 2.5 m
	2.26 m		*ca*. 2.05 m	1.91 dd(9.8, 14)
18	1.34 s	1.32 s	1.72 s	1.72 s
19	0.97 d(6.7)	0.97 d(6.8)	1.01 d(6.8)	0.99 d(6.8)
20	5.78 q(7.1)	5.88 q(7.1)	5.89 q(7.1)	5.79 dq(1.1, 7.1)
21	1.84 d(7.1)	1.83 d(7.1)	1.89 d(7.1)	1.86 dd(1.5, 7.1)
OAc			2.10 s	2.11 s

Table 2-3-8: ^{1}H NMR spectroscopic data of pyrrolizidine alkaloids **2-3-24**~**2-3-27**.

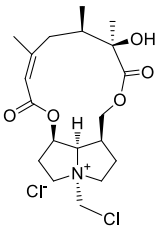
H	2-3-24	2-3-25	2-3-26	2-3-27
1	2.43 m			2.68 m
2	4.23 m	2.49 m, 2.35 m	*ca*. 2.40 m	5.06 ddd(5, 6.8, 6.8)
3	*ca*. 2.94 m	*ca*. 2.46 m	*ca*. 2.83 m	3.08 dd(5.1, 11.3)
		ca. 2.8 m		2.91dd(6.8, 11.2)
5	3.19 m, 2.63 m	*ca*. 2.46 m, *ca*. 2.80 m	*ca*. 2.83 m	*ca*. 3.14 m, *ca*. 2.69 m
6	2.68 m, 2.21 m	2.13 m, *ca*. 2.47 m	*ca*. 2.39 m, *ca*. 2.08 m	*ca*. 2.23 m
7	5.32 m	5.55 ddd(3.3, 4.9, 4.9)	5.45 m	5.19 t(*ca*. 4)
8	3.72 dd(4.7, 7.8)	3.38 d(4.9)	3.51 d(4.1)	3.60 dd(4.2, 7.2)
9	4.78 dd(7.5, 12)	4.63 d(11.7)	4.63 d(12.2)	4.69 dd(7.6, 11.8)
	4.10 dd(1.9, 12)	4.42 d(11.7)	4.35 d(12.2)	4.00 dd(1.7, 11.8)
13	2.02 m	*ca*. 2.00 m	*ca*. 1.84 m	1.90 m
14	2.34 dd(4.1, 13)	*ca*. 2.30 m	2.63 dd(5.2, 14.4)	2.26 m
	2.07 dd(7.7, 13)	*ca*. 2.26 m	*ca*. 2.08 m	2.12 dd(7.1, 13.9)
18	3.64 d(11.2)	1.25 s	1.65 s	1.29 s
	3.60 d(11.2)			
19	0.89 d(6.6)	0.98 d(6.8)	1.03 d(6.8)	0.96 d(6.8)
20	5.91 q(7.2)	5.85 q(7.2)	5.87 q(7.1)	5.83 q(7.1)
21	1.83 d(7.2)	1.83 d(7.2)	1.88 d(7.1)	1.82 d(7.1)
OAc		2.01 s	2.09 s, 2.02 s	2.08 s

Table 2-3-9: ^{1}H NMR spectroscopic data of pyrrolizidine alkaloids **2-3-28**~**2-3-31**.

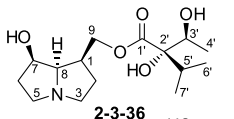
H	2-3-28	2-3-29	2-3-30	2-3-31
1	*ca*. 2.7 m	2.47 dq(1.1, *ca*. 7.5)		2.65 m
2	5.19 m	4.20 q(7.3)	1.95 m	2.06 ddd(5.5, 6.5, 12.5)
			1.85 m	1.99 dt(8.5, 12.5)
3	3.16 m	3.01 dd(7.2, 11.2)	3.15 ddd(6.5, 6.5, 10.6)	3.21 br dd(7.0, 8.5)
	2.92 dd(5.3, 11.2)	2.92 dd(7.3, 11.2)	2.87 ddd(2.6, 7.2, 10.7)	
5	*ca*. 3.18 m	3.20 ddd(2.5, 8, 10)	3.01 m	3.15 ddd(2.0, 7.5, 9.5)
	2.94 m	2.59 ddd(6.6, 10, 10)	2.77 ddd(4.1, 10, 11.5)	2.72 ddd(6.5, 9.5, 11.0)
6	*ca*. 2.22 m	2.09 m	2.24 m	2.17 m
		2.26 m	*ca*. 1.90 m	2.12 ddt(2.0, 7.0, 13.5)
7	5.19 m	5.10 m	5.45 q(*ca*. 6.3)	5.38 td(2.0, 3.5)
8	3.59 dd(4.2, 7.9)	3.65 dd(3.9, 7.6)	3.38 d(6.1)	3.76 dd (4.0, 7.5)
9	4.49 dd(7.8, 12)	4.75 dd(5.9, 12.3)	4.41 d(11.4)	4.51 dd (11.0, 11.5)
	4.21 dd(1.5, 12)	4.09 dd(1.1, 12.3)	4.15 d(11.4)	4.05 dd (3.0, 11.5)
12				2.44 dt (6.5, 9.5)
13	*ca*. 1.7 m	*ca*. 2.0 m	*ca*. 2.10 m	2.65 br dd (5.0, 13.5)
				2.22 dd (9.5, 13.5)
14	*ca*. 2.7 m	2.32 dd(8.4, 13.9)	2.41 dd(6.2, 14.2)	
	2.01 dd(7.3, 14.4)	2.18 dd(4.2, 13.9)	2.28 dd(7.2, 14.2)	
15				5.66 t (1.5)
17				1.29 s
18	1.66 s	1.33 s	1.76 s	0.99 d(6.8)
19	1.01 d(6.8)	0.99 d(6.8)	1.05 d(6.8)	1.88 d(1.5)
20	5.85 q(7.1)	6.51 q(7.1)	6.78 q(7.1)	
21	1.87 d(7.1)	1.78 d(7.1)	1.80 d(7.1)	
1a′				2.56 dd (6.0, 15.0)
				2.36 dd (7.0, 15.0)
OAc	2.08 s, 2.08 s		2.13 s	
OMe				3.69 s



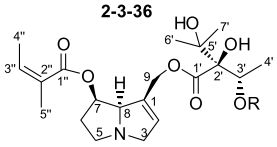
2-3-33



2-3-35



2-3-36



2-3-37 R = H
2-3-38 R = Ac

Table 2-3-10: ^{1}H NMR spectroscopic data of pyrrolizidine alkaloids **2-3-32**~**2-3-35**.

H	2-3-32 · HCl	2-3-33	2-3-34 · HCl	2-3-35
1	2.94 dtd-like (3.3, 9.0, 16.8)	3.37 m	2.94 dtd-like (3.0, 9.0, 17.5)	3.13 m

Table 2-3-10 (continued)

H	2-3-32 · HCl	2-3-33	2-3-34 · HCl	2-3-35
2	2.37 m	2.74 m	2.49 m	2.33 m
	2.11 m	2.10 m	2.13 ddd-like (7.0, 13.5, 14.0)	
3	3.78 dt(7.3, 11.4)	4.51 dt(6.0, 12.0)	3.88 dt(6.5, 11.5)	3.9~4.1 m
	3.10 m	3.83 m	3.08 dt(7.5, 11.5)	
5	3.98 ddd(1.7, 7.7, 11.3)	4.64 br dd(7.5, 12.3)	3.94 ddd(2.5, 8.0, 11.0)	3.9~4.1 m
	3.07 m	3.86 m	3.12 td(6.0, 11.5)	
6	2.50 m	3.04 dddd (4.5, 7.5, 12.3, 14.4)	2.51 m	2.4~2.6 m
	2.30 m	2.34 m	2.37 m	
7	5.70 td(1.8, 4.2)	5.86 br t(4.2)	5.69 td(2.5, 4.5)	5.84 dt(6.0, 7.2)
8	4.43 t (4.2)	4.96 dd (5.4, 9.3)	4.47 dd (4.5, 8.5)	4.65 dd (7.2, 8.4)
9	4.46 dd (9.3, 12.2)	4.38 dd (10.0, 12.0)	4.57 dd (9.0, 12.0)	4.44 dd (9.9, 11.4)
	4.19 dd (3.3, 12.2)	4.16 dd (3.9, 12.0)	4.18 dd (3.5, 12.0)	4.23 dd (4.2, 11.4)
12	2.34 m	2.34 m	2.35 m	2.61 m
13	2.80 br dd (3.9, 13.8)	2.75 m	2.57 br dd (4.5, 12.0)	2.74 ddd(1.5, 5.4, 13.2)
	2.41 m	2.38 m	2.32 dd (9.0, 12.0)	2.27 dd (9.6, 13.2)
15	5.66 t (1.2)	5.66 t (1.2)	5.67 br s	5.73 br s
17	1.66 s	1.65 s	1.33 s	1.31 s
18	1.15 d(6.6)	1.14 d(6.7)	1.03 d(6.5)	1.0 d(6.9)
19	1.95 d(1.2)	1.93 d(0.9)	1.93 d(1.0)	1.96 d(1.2)
1′				5.44 s
OAc	2.07 s	2.08 s		
OH			3.01 br s(OH)	

Table 2-3-11: ^{1}H NMR spectroscopic data of pyrrolizidine alkaloids **2-3-36**~**2-3-38**.

H	2-3-36	2-3-37	2-3-38
1	2.78 m		
2	*α* 1.89 m, *β* 2.15 m	5.82 m	5.84 m
3	*α* 3.26 m	u 3.36 m	u 3.41 m
	β 2.61 ddd(10.8, 8.6, 6.0)	d 3.94 dm(15.6)	d 4.00 dm(15.5)
5	*α* 3.21 m, *β* 2.73 m	u 2.65 m, d 3.33 m	u 2.71 m, d 3.41 m
6	*α* 2.03 m, *β* 2.14 m	2.08 m	2.15 m
7	4.29 br t(3.6)	5.41 m	5.45 m
8	3.48 dd(7.8, 4.0)	4.39 m	4.42 m
9u	4.09 dd(10.8, 7.8)	4.62 dm(13)	4.62 dm(13.3)
9d	4.46 dd(10.8, 4.5)	4.88 dm(13)	4.76 dm(13.3)
3′	3.98 q(6.8)	4.14 q(6.3)	5.44 q(6.3), 1.95 s(OAc)
4′	1.33 d(6.7)	1.22 d(6.3)	1.37 d(6.3)
5′	2.08 m		
6′	0.93 d(6.8)	1.19 s	1.17 s

Table 2-3-11 (continued)

H	2-3-36	2-3-37	2-3-38
7′	0.83 d(6.8)	1.26 s	1.39 s
3″		6.06 qq(1.5, 7.3)	6.10 qq(1.5, 7.3)
4″		1.93 quint(1.5)	1.80 quint(1.5)
5″		1.77 dq(1.5, 7.3)	1.96 dq(1.5, 7.3)

Bibliography

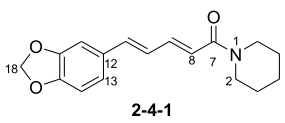
[46] Kato A, Kato N, Adachi I, et al. J Nat Prod, 2007, 70: 993.
[47] Asano N, Ikeda K, Kasahara M, et al. J Nat Prod, 2004, 67: 846.
[48] Bugni TS, Woolery M, Kauffman CA, et al. J Nat Prod, 2006, 69: 1626.
[49] Huong DTT, Martin MT, Litaudon M, et al. J Nat Prod, 1998, 61: 1444.
[50] Were O, Benn M, Munavu RM. J Nat Prod, 1991, 54: 491.
[51] Pérez-Castorena AL, Arciniegas A, Alonso RP, et al. J Nat Prod, 1998, 61: 1288.
[52] Reina M, Gonzalez-Coloma A, Gutierrez C, et al. J Nat Prod, 1998, 61: 1418.
[53] El-Shazly A, Sarg T, Ateya A, et al. J Nat Prod, 1996, 59: 310.

2.4 Piperidine alkaloids

2.4.1 N-acyl piperidine alkaloids

Table 2-4-1: Cos, MFs, and TSs of *N*-acyl piperidine alkaloids **2-4-1**~**2-4-15**.

No.	Compounds	MFs	Test solvents	References
2-4-1	piperine	$C_{17}H_{19}NO_3$	$CDCl_3$	[54]
2-4-2	3α,4α-epoxy-5β-pipermethystine	$C_{16}H_{17}NO_5$	CD_3COCD_3	[55]
2-4-3	awaine	$C_{14}H_{17}NO_2$	CD_3COCD_3	[55]
2-4-4	pipermethystine	$C_{16}H_{17}NO_4$	$CDCl_3$	[56]
2-4-5	*N*-(3-methoxy-4,5-methylenedioxydihydrocinnamoyl)-$\triangle^3$-pyridin-2-one	$C_{16}H_{17}NO_5$	$CDCl_3$	[57]
2-4-6	piplartine	$C_{17}H_{19}NO_5$	$CDCl_3$	[58]
2-4-7	*N*-(3,4-dimethoxycinnamoyl)-$\triangle^3$-pyridin-2-one	$C_{16}H_{17}NO_4$	$CDCl_3$	[58]
2-4-8	*N*-(3-methoxy-4,5-methylenedioxycinnamoyl)-$\triangle^3$-pyridin-2-one	$C_{16}H_{15}NO_5$	$CDCl_3$	[58]
2-4-9	8,9-dihydropiplartine	$C_{17}H_{21}NO_5$	$CDCl_3$	[59]
2-4-10	4′-desmethylpiplartine	$C_{16}H_{17}NO_5$	$CDCl_3$	[60]
2-4-11	cenocladamide	$C_{16}H_{17}NO_5$	$CDCl_3$	[60]
2-4-12	piplaroxide	$C_{16}H_{19}NO_5$	$CDCl_3$	[61]
2-4-13	piperdardine	$C_{19}H_{23}NO_3$	$CDCl_3$	[62]
2-4-14	piperettine	$C_{19}H_{21}NO_3$	$CDCl_3$	[62]
2-4-15	piplartine-dimer A	$C_{34}H_{38}N_2O_{10}$	$CDCl_3$	[63]



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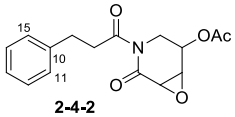

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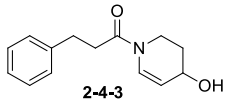

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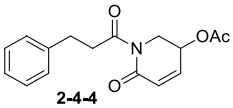

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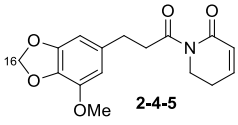

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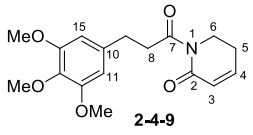

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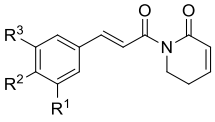
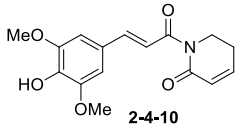


	R^1	R^2	R^3
2-4-6	OMe	OMe	OMe
2-4-7	OMe	OMe	H
2-4-8	OMe	-O-CH_2-O-	



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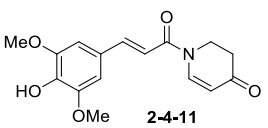

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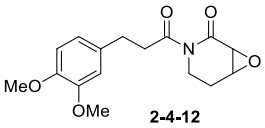

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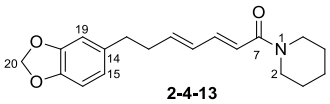

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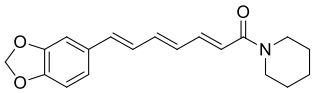

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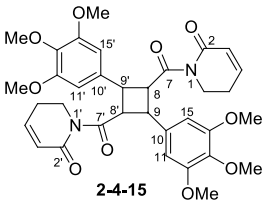


2-4-15

Table 2-4-2: ^{1}H NMR spectroscopic data of piperidine alkaloids **2-4-1**~**2-4-4**.

H	2-4-1	2-4-2	2-4-3	2-4-4
2	3.52 br s or 3.63 br s		6.83 d(8.2)	
3	1.55~1.70 br m	3.67 d(4.0)	6.97 dd(8.2, 4.0)	6.15 d(10)
4	1.55~1.70 br m	3.88 ddd(4.0, 3.1, 1.4)	4.15 m	6.84 ddd(10, 5, 1)
5	1.55~1.70 br m	5.64 ddd(3.1, 2.6, 2.4)	1.78 m	5.42 q(5)
6	3.63 br s or 3.52 br s	4.45 ddd (14.9, 2.4, 1.4)	3.93 dt(13.0, 5.1)	3.86 dd(15, 5)
		3.33 dd(14.9, 2.6)	3.40 ddd(13.0, 8.7, 5.6)	4.32 ddd(15, 5, 1)
8	6.77 d(15.0)	3.24 ddd(17.5, 9.1, 6.6)	2.76 t(7.3)	3.01[①]
		3.17 ddd (17.5, 8.6, 6.6)		
9	7.40 ddd(14.7, 8.5, 1.8)	2.91 m	2.91 t(7.3)	3.38[①]
10	6.76 dd(15.0, 8.5)			
11	6.44 d(14.7)	7.26 m	7.26 m	7.2 br s
12		7.26 m	7.26 m	7.2 br s
13	6.89 dd(8.0, 1.6)	7.17 m	7.17 m	7.2 br s
14	6.78 d(8.0)	7.26 m	7.26 m	7.2 br s
15		7.26 m	7.26 m	7.2 br s
17	6.98 d(1.6)			
18	5.98 s			
OAc		2.02 s		2.06 s
OH			3.81 d(5.4)	

[①] Methylene groups at 8- and 9-positions showed the signals of A_2B_2 system.

Table 2-4-3: ^{1}H NMR spectroscopic data of piperidine alkaloids **2-4-5**~**2-4-9**.

H	2-4-5	2-4-6	2-4-7	2-4-8	2-4-9[①]
2	3.95 t(6.5)	4.04 t(6.3)	4.01 t(6.5)	4.01 t (6.4)	
3	2.37 m	2.45 m	2.44 m	2.45 m	6.02 dt(9, 2)
4	6.88 dt(9.7, 4.2)	6.95 dt(9.7, 4.2)	6.91 dt(9.7, 4.1)	6.92 dt(9.7, 4.2)	6.92 m
5	5.98 dt(9.7, 1.8)	6.02 dt(9.7, 1.8)	6.02 dt(9.7, 1.8)	6.02 dt(9.7, 1.8)	2.40 m
6					3.99 t(6)

Table 2-4-3 (continued)

H	2-4-5	2-4-6	2-4-7	2-4-8	2-4-9[①]
8	3.20 t(7.6)	7.41 d(15.6)	7.38 d(15.6)	7.34 d(15.5)	3.26 m
9	2.88 t(7.6)	7.68 d(15.6)	7.70 d(15.6)	7.62 d(15.5)	2.95 m
11	6.40 d(1.4)	6.80 s	7.07 d(2.0)	6.73 d(1.5)	6.50 s
12					3.88 s(OMe)
13					3.85 s(OMe)
14			6.83 d(8.3)		3.88 s(OMe)
15	6.42 d(1.4)	6.80 s	7.14 dd(8.3, 2.0)	6.80 d(1.5)	6.50 s
16	5.90 s			6.00 s	
OMe	3.86 s	3.87, 3.88 2×s (3×OMe)	3.88, 3.89 2×s (2×OMe)	3.90 s	

[①] On the numbering system in the original literature.

Table 2-4-4: ^{1}H NMR spectroscopic data of piperidine alkaloids **2-4-10**~**2-4-14**.

H	2-4-10	2-4-11	2-4-12	2-4-13	2-4-14
2		7.97 br d(8.4)	4.29 dddd(13.5, 5.6, 1.5, 1.5) 3.13 dd(13.3, 4.0)	3.61 br s	3.61 br s
3	6.03 dt(9.6, 2.0)	5.43 d(8.4)	2.31 dm(15.0) 1.88 ddd(15.0, 13.2, 5.7)	1.45~1.60 m	1.48~1.60 m
4	6.93 dt(9.6, 4.2)		3.58 dd(4.0, 4.0)	1.61~1.70 m	1.61~1.70 m
5	2.56 m	2.62 t(7.4)	3.51 d(4.0)	1.45~1.60 m	1.48~1.60 m
6	4.03 t(6.6)	4.16 t(7.4)		3.48 br s	3.48 br s
8	7.38 d(15.6)	6.76 d(15.3)	3.20 m	6.26 d(14.8)	6.34 d(14.6)
9	7.68 d(15.6)	7.73 d(15.3)	2.90 t(7.2)	7.21 dd(14.8, 10.8)	7.33 dd(14.6, 11.4)
10				6.18 dd(15.1,10.8)	6.39 dd(14.0, 11.4)
11	6.81 s	6.79 s	6.76 s	6.06 dt(15.1, 7.0)	6.62 dt(14.0, 10.0)
12				2.42 dt(7.3, 7.0)	6.64 dd(15.0, 10.0)
13				2.66 t(7.3)	6.57 t(15.0)
14			6.76 s		
15	6.81 s	6.79 s	6.76 s	6.61 dd(7.9, 1.5)	6.84 dd(8.0, 1.6)
16				6.70 d(7.9)	6.74 d(8.0)
19				6.66 d(1.5)	6.94 d(1.6)
20				5.92 s	5.94 s
OMe	3.91 s(2×OMe)	3.82 s(2×OMe)	3.81, 3.84 2×s (2×OMe)		

Table 2-4-5: ^{1}H NMR spectroscopic data of piperidine alkaloids **2-4-15**.

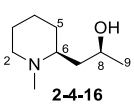
H	2-4-15	H	2-4-15	H	2-4-15
3, 3′	5.72 dq(10, 1.5)	6eq, 6′eq	3.66~4.15 m	11, 11′, 15, 15′	6.5 s
4, 4′	6.45~6.75 m	6ax, 6′ax	3.04~3.6 m	OMe-12, 12′, 14, 14′	3.86 s
5, 5′	1.44~2.40 m	8, 8′, 9, 9′	4.46~5.06 AA'BB'	OMe-13, 13′	3.77 s

2.4.2 Short-chain substituted piperidine alkaloids

Table 2-4-6: Cos, MFs, and TSs of short-chain substituted piperidine alkaloids **2-4-16**~**2-4-43**.

No.	Compounds	MFs	Test solvents	References
2-4-16	*N*-methylsedridine	$C_9H_{19}NO$	$CDCl_3$	[64]
2-4-17	pinidinol	$C_9H_{19}NO$	$CDCl_3$	[65]
2-4-18	(+)-6-*epi*-9-epipinidinol	$C_9H_{19}NO$	$CDCl_3$	[66]
2-4-19	(+)-1,2-dehydropinidinol	$C_9H_{17}NO$	$CDCl_3$	[66]
2-4-20	1,2-dehydropinidinone	$C_9H_{15}NO$	$CDCl_3$	[66]
2-4-21	adalinine	$C_{13}H_{23}NO_2$	$CDCl_3$	[67]
2-4-22	α-7-deoxyhomonojirimycin	$C_7H_{15}NO_4$	D_2O	[68]
2-4-23	α-7-deoxyhomomannojirimycin	$C_7H_{15}NO_4$	D_2O	[68]
2-4-24	α-homomannojirimycin	$C_7H_{15}NO_5$	D_2O	[69]
2-4-25	β-homomannojirimycin	$C_7H_{15}NO_5$	D_2O	[69]
2-4-26	α-homonojirimycin	$C_7H_{15}NO_5$	D_2O	[69]
2-4-27①	*N*-methyl-α-homonojirimycin	$C_8H_{17}NO_5$	D_2O	[70]
2-4-28①	*N*-butyl-α-homonojirimycin	$C_{11}H_{23}NO_5$	D_2O	[70]
2-4-29	β-homonojirimycin	$C_7H_{15}NO_5$	D_2O	[69]
2-4-30	β-4,5-di-*epi*-homonojirimycin	$C_7H_{15}NO_5$	D_2O	[70]
2-4-31	α-3,4-di-*epi*-homonojirimycin	$C_7H_{15}NO_5$	C_5D_5N-D_2O(3:1)	[69]
2-4-32	7-*O*-β-D-glucopyranosyl-α-homonojirimycin	$C_{13}H_{25}NO_{10}$	D_2O	[69]
2-4-33	5-*O*-α-D-galactopyranosyl-α-homonojirimycin	$C_{13}H_{25}NO_{10}$	D_2O	[69]
2-4-34	1,6-dideoxynojirimycin	$C_6H_{13}NO_3$	D_2O	[71]
2-4-35	1,3,4-trideoxynojirimycin	$C_6H_{13}NO_2$	D_2O	[71]
2-4-36	*N*-methyl-1-deoxynojirimycin	$C_7H_{15}NO_4$	D_2O	[72]
2-4-37	2-*O*-α-D-galactopyranosyl-1-deoxynojirimycin	$C_{12}H_{23}NO_9$	D_2O	[72]
2-4-38	fagomine	$C_6H_{13}NO_3$	D_2O	[72]
2-4-39	α-1-*C*-ethyl-fagomine	$C_8H_{17}NO_3$	D_2O	[73]
2-4-40	3-*epi*-fagomine	$C_6H_{13}NO_3$	D_2O	[74]
2-4-41	6-*O*-(α-D-galactopyranosyl)-1-deoxynojirimycin	$C_{12}H_{23}NO_9$	D_2O	[74]
2-4-42	6-*O*-β-D-glucopyranosyl-1-deoxynojirimycin	$C_{12}H_{23}NO_9$	D_2O	[74]
2-4-43	3-*O*-β-D-glucopyranosyl-1-deoxynojirimycin	$C_{12}H_{23}NO_9$	D_2O	[74]

① Synthesized compounds.



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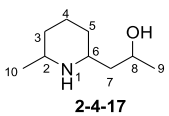

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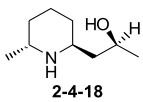

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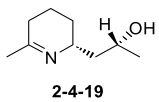

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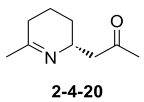

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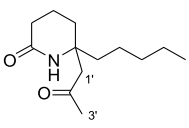
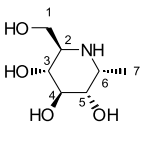
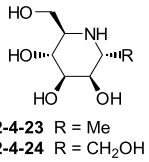


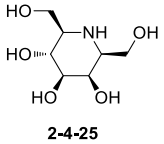
2-4-21



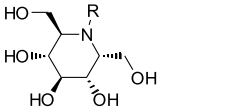
2-4-22



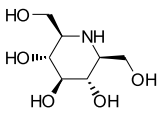
2-4-23 R = Me
2-4-24 R = CH_2OH



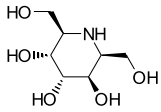
2-4-25



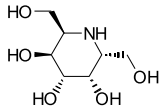
2-4-26 R = H
2-4-27 R = Me
2-4-28 R = $CH_2CH_2CH_2CH_3$ (1′ 2′ 3′ 4′)



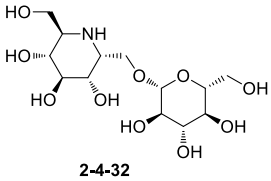
2-4-29



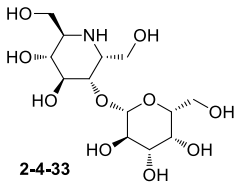
2-4-30



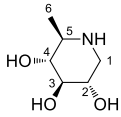
2-4-31



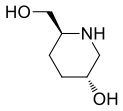
2-4-32



2-4-33



2-4-34



2-4-35

Table 2-4-7: ^{1}H NMR spectroscopic data of piperidine alkaloids **2-4-16**~**2-4-21**.

H	2-4-16	2-4-17	2-4-18	2-4-19	2-4-20	2-4-21
2	2.11~2.20 m①	2.60 dqd(11.0, 6.2, 2.6)	3.10 m			
3	1.45~1.62 m	1.0 m	1.63 m	2.13 m (2H)	1.40~1.70 m(2H)	2.30 m
	1.67~1.79 m	1.35 m③	1.21 m			
4	1.67~1.79 m	1.35 m, 1.85 m	1.59 m (2H)	1.69 m, 1.60 m	1.40~1.70 m(2H)	1.70 m, 1.78 m
	1.20~1.34 m					
5	1.45~1.62 m	1.49 m	1.50 m, 1.35 m	1.60 m, 1.21 m	1.40~1.70 m(2H)	1.54 m, 1.57 m
6	2.90 br d(11.3)②	2.95 dtd(8.1, 5.4, 2.7)	3.29 m	3.66 m	2.00 m	
7	1.20~1.34 m	1.47 ddd(14.4, 9.1, 3.3)	1.25 ddd(14, 6.7, 3.4)	1.66 dd	2.38 dd(16, 7.9)	1.60 m, 1.73 m
	1.90~2.02 m	1.60 ddd(14.4, 5.6, 3.2)	1.84 ddd(14, 9.0, 3.8)	(6.7, 4.7, 2H)	2.72 dd(16, 5.8)	
8	4.22 dqd(11, 6, 3)	4.13 qdd(9.2, 6.1, 3.1)	4.04 m	4.09 m		1.10 m, 1.22 m
9	1.15 d(6.0)	1.16 d(6.1)	1.16 d(6.3)	1.25 d(6.4)	2.07 s	1.20 m, 1.22 m
10		1.04 d(6.3)	1.07 d(6.7)	1.92 d(2.0)	1.80 d(1.9)	1.22 m, 1.28 m
11						0.85 t(7.0)
1′						2.63 AB q(17.7)
2′						2.10 s
NH			3.46 br s(NH or OH)			6.5 br s
NMe	2.34 s					

① Only one proton signal from the 2-postion was indicated in the literature.
② Signals for 1.90~2.02 m (H-6) was listed in the literature.
③ Signals for 1.60 ddd (H-3) was listed in the literature.

Table 2-4-8: ^{1}H NMR spectroscopic data of piperidine alkaloids **2-4-22**~**2-4-25**.

H	2-4-22	2-4-23	2-4-24	2-4-25
1	3.69 dd(11.7, 5.6)	3.73 dd(11.7, 3.0)	3.751 dd(11.5, 5.8)	3.74 dd(11.7, 5.4)
	3.81 dd(11.7, 2.9)	3.85 dd(11.7, 4.4)	3.80 dd(11.5, 3.4)	3.84 dd(11.7, 3.2)
2	2.88 ddd(9.8, 5.6, 2.9)	2.76 ddd(9.5, 4.4, 2.9)	2.75 m	2.59 ddd(10.3, 5.4, 3.2)
3	3.27 dd(9.8, 9.3)	3.67 dd(9.5, 9.5)	3.65 t(9.3)	3.59 t(10.3)
4	3.59 dd(9.3, 9.3)	3.79 dd(9.5, 3.0)	3.68 dd(9.3, 2.9)	3.56 dd(10.3, 2.5)
5	3.62 dd(9.3, 5.5)	3.86 dd(3.0, 3.0)	4.02 t(2.9)	4.01 dd(2.5, 1.7)
6	3.37 qd(7.1, 5.5)	3.21 qd(7.3, 3.0)	3.15 ddd(8.1, 6.8, 2.9)	2.87 dt(6.6, 1.7)
7	1.17 d(7.1)	1.19 d(7.3)	3.69 dd(11.5, 6.8)	3.62~3.71(2H)
			3.748 dd(11.5, 8.1)	

Table 2-4-9: ^{1}H NMR spectroscopic data of piperidine alkaloids **2-4-26**~**2-4-29**.

H	2-4-26	2-4-27	2-4-28	2-4-29
1	3.57 dd(11.5, 7.1)	3.86 dd(12.7, 3.4)	3.84 dd(12.4, 5.4)	3.64 dd(11.5, 6.6)
	3.90 dd(11.5, 3.2)	3.89 dd(12.7, 2.9)	3.94 dd(12.4, 3.2)	3.89 dd(11.5, 2.9)
2	2.87 ddd(10.0, 7.1, 3.2)	2.59 dt(10.0, 3.2)	2.79~2.81	2.66 ddd(9.9, 6.6, 2.9)
3	3.21 dd(10.0, 9.0)	3.41 dd(10.0, 9.0)	3.41 dd(10.3, 9.0)	3.25 dd(9.9, 9.2)
4	3.50 dd(10.0, 9.0)	3.50 dd(10.0, 9.0)	3.51 dd(10.3, 9.0)	3.39 t(9.2)
5	3.75 dd(10.0, 6.1)	3.76 dd(10.0, 5.8)	3.80 dd(10.3, 5.9)	3.25 dd(9.9, 9.2)
6	3.29 ddd(9.1, 6.1, 5.3)	3.09 dt(5.8, 4.4)	3.30 m	2.66 ddd(9.9, 6.6, 2.9)
7	3.79~3.86 m (2H)	3.90 dd(12.2, 4.2)	3.85 (2H)	3.64 dd(11.5, 6.6)
		3.94 dd(12.2, 5.4)		3.89 dd(11.5, 2.9)
1′		2.53 s	2.79~2.81	
2′			1.44 m, 1.55 m	
3′			1.32 m	
4′			0.91 s①	

① A mistake may be exist in the literature.

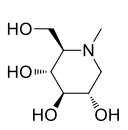
Table 2-4-10: ^{1}H NMR spectroscopic data of piperidine alkaloids **2-4-30**~**2-4-33**.

H	2-4-30	2-4-31	2-4-32	2-4-33
1	3.72 dd(11.7, 5.4)	4.21 dd(10.6, 7.7)	3.60 dd(11.4, 6.9)	3.59 dd(11.4, 7.3)
	3.82 dd(11.7, 3.2)	4.30 dd(10.6, 4.8)	3.88 dd(11.4, 3.2)	3.90 dd(11.4, 2.9)
2	2.90 ddd(10.5, 5.4, 3.2)	3.87 ddd(7.7, 6.2, 4.8)	2.90 ddd(10.0, 6.9, 3.2)	2.87 ddd(9.9, 7.3, 2.9)
3	3.78 dd(10.5, 3.2)	4.26 dd(6.2, 3.3)	3.24 dd(10.0, 9.0)	3.27 dd(9.9, 9.2)
4	4.01 dd(3.9, 3.2)	4.39 t(3.3)	3.53 dd(10.0, 9.0)	3.64 dd(9.9, 9.2)
5	3.93 dd(3.9, 1.7)	4.48 dd(4.0, 3.3)	3.75 dd(10.0, 6.1)	3.81 dd(9.9, 5.9)
6	3.09 dt(6.6, 1.7)	3.65 ddd(8.1, 4.8, 4.0)	3.44~3.50	3.50 ddd(9.1, 5.9, 2.9)
7	3.64 dd(11.2, 6.6)	4.42 dd(11.0, 4.8)	3.933 dd(10.7, 9.5)	3.83 dd(11.4, 9.1)
	3.68 dd(11.2, 6.6)	4.61 dd(11.0, 8.1)	4.13 dd(10.7, 3.7)	3.91 dd(11.4, 2.9)
1′			4.50 d(7.8)	5.13 d(3.7)

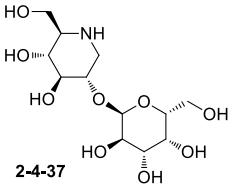
Table 2-4-10 (continued)

H	2-4-30	2-4-31	2-4-32	2-4-33
2′			3.32 dd(9.1, 7.8)	3.87 dd(10.6, 3.7)
3′			3.51 t(9.1)	3.93 dd(10.6, 3.3)
4′			3.40 dd(9.8, 9.1)	4.00 dd(3.3, 1.1)
5′			3.44~3.50	4.17 dt(6.2, 1.1)
6′			3.74 dd(12.2, 5.9) 3.927 dd(12.2, 2.2)	3.75 d(2H)①

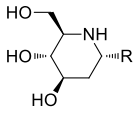
① No coupling constant was given in the literature.



2-4-36

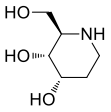


2-4-37

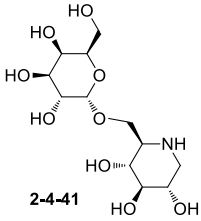


2-4-38 R = H

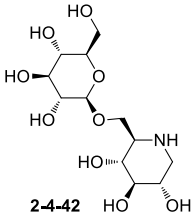
2-4-39 R = CH_2CH_3 (1′, 2′)



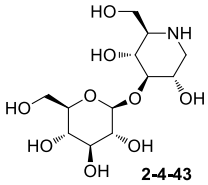
2-4-40



2-4-41



2-4-42



2-4-43

Table 2-4-11: ^{1}H NMR spectroscopic data of piperidine alkaloids **2-4-34**~**2-4-38**.

H	2-4-34	2-4-35	2-4-36	2-4-37	2-4-38
1ax	2.46 dd(12.5, 10.7)	2.40 dd(11.5, 10.2)	2.23 dd(11.6, 10.8)	2.46 dd(12.5, 10.6)	2.60 dt(13.0, 2.6)
1eq	3.07 dd(12.5, 5.1)	3.16 ddd(11.5, 4.6, 2.2)	2.97 dd(11.6, 4.9)	3.32 dd(12.5, 4.8)	3.00 ddd(13.0, 4.5, 2.2)
2ax	3.51 ddd(10.7, 9.3, 5.1)	3.65 m	3.57 ddd(10.8, 9.5, 4.9)	3.58 ddd(10.6, 9.5, 4.8)	1.45 ddt(13.0, 11.6, 4.5)
2eq					1.99 dddd (13.0, 5.1, 2.6, 2.2)
3ax	3.28 dd(9.3, 9.3)	1.35 m	3.29 t(9.5)	3.47 t(9.5)	3.54 ddd(11.6, 9.2, 5.1)
3eq		2.07 m			
4ax	3.01 dd(9.3, 9.3)	1.18 m	3.40 t(9.5)	3.30 t(9.5)	3.17 dd(9.5, 9.2)
4eq		1.76 m			

Table 2-4-11 (continued)

H	2-4-34	2-4-35	2-4-36	2-4-37	2-4-38
5	2.52 dq(6.4, 9.3)	2.63 m	1.99 dt(9.5, 2.6)	2.57 ddd(9.5, 6.2, 2.9)	2.52 ddd(9.5, 6.6, 3.0)
6	1.16 d(6.4)	3.43 dd(11.3, 7.3)	3.83 dd(12.8, 2.6)	3.65 dd(11.7, 6.2)	3.63 dd(11.7, 6.6)
		3.55 dd(11.3, 4.9)	3.91 dd(12.8, 2.6)	3.84 dd(11.7, 2.9)	3.86 dd(11.7, 3.0)
NMe			2.35 s		
1′				5.09 d(3.7)	
2′				3.83 dd(10.2, 3.7)	
3′				3.91 dd(10.2, 3.3)	
4′				4.01 br d(3.3, 1.0)①	
5′				4.19 br t(6.2, 1.0)	
6′				3.75 d(2H, 6.2)	

① Coupling constant being confirmed by decoupling experiment.

Table 2-4-12: ^{1}H NMR spectroscopic data of piperidine alkaloids **2-4-39**~**2-4-43**.

H	2-4-39	2-4-40	2-4-41	2-4-42	2-4-43
1ax	3.05 m	2.79 dt(11.7, 2.6)	2.48 dd(12.5, 10.6)	2.46 dd(12.4, 10.9)	2.50 dd(12.4, 10.7)
1eq		2.77 ddd(11.7, 5.9, 2.6)	3.12 dd(12.5, 5.1)	3.12 dd(12.4, 5.1)	3.14 dd(12.4, 5.1)
2ax	1.55~1.67 m	1.73 dddd (14.3, 11.7, 5.9, 2.6)	3.50 m	–	3.36 ddd (10.7, 10.7, 5.1)
2eq	2.02 ddd(13.4, 4.9, 2.2)	1.85 ddt(14.3, 3.3, 2.6)			
3ax	3.78 ddd(11.5, 9.0, 4.9)		3.32①	3.33 t(9.0)	3.57 t(9.0)
3eq		4.09 dt(3.3, 2.6)			
4ax	3.20 dd(10.0, 9.0)	3.48 dd(9.9, 3.3)	3.32①	3.32 t(9.0)	3.68 dd(9.9, 9.0)
4eq					
5	2.85 ddd(10.0, 6.8, 2.9)	2.87 ddd(9.9, 6.6, 3.3)	2.75 ddd(9.0, 5.1, 2.6)	2.69 m	2.58 ddd(9.9, 5.9, 2.9)
6	3.63 dd(11.5, 6.8)	3.63 dd(11.7, 6.6)	3.64 dd(10.3, 2.6)	3.76 dd(10.7, 6.3)	3.67 dd(11.8, 5.9)
	3.89 dd(11.5, 2.9)	3.82 dd(11.7, 3.3)	3.884 dd(10.3, 5.1)	4.14 dd(10.7, 2.7)	3.83 dd(11.8, 2.9)
NMe					
1′	1.55~1.67 m (2H)		4.95 d(3.7)	4.45 d(8.0)	4.73 d(8.1)

Table 2-4-12 (continued)

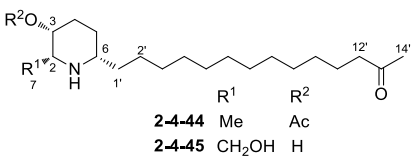
H	2-4-39	2-4-40	2-4-41	2-4-42	2-4-43
2′	0.91 t(7.3)		3.82 dd(10.2, 3.7)	3.30 dd(9.2, 8.0)	3.37 dd(9.2, 8.1)
3′			3.878 dd(10.2, 2.8)	3.51 t(9.2)	3.53 t(9.2)
4′			3.99 dd(2.8, 1.0)	3.39 dd(9.5, 9.2)	3.42 dd(9.5, 9.2)
5′			3.94 br t②	3.47 ddd(9.5, 5.9, 2.4)	3.49 ddd(9.5, 5.9, 2.2)
6′			3.75 d② (2H)	3.72 dd(12.4, 5.9)	3.73 dd(12.4, 5.9)
				3.95 dd(12.4, 2.4)	3.92 dd(12.4, 2.2)

① Signal type not given in literature.
② Coupling constants not given in literature.

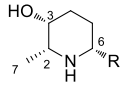
2.4.3 Long-chain substituted piperidine alkaloids

Table 2-4-13: Cos, MFs, and TSs of long-chain substituted piperidine alkaloids **2-4-44**~**2-4-57**.

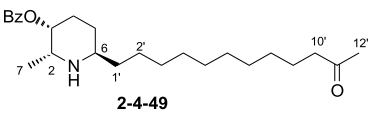
No.	Compounds	MFs	Test solvents	References
2-4-44	(–)-3-*O*-acetylspectaline	$C_{22}H_{41}NO_3$	$CDCl_3$	[75]
2-4-45	(–)-7-hydroxyspectaline	$C_{20}H_{39}NO_3$	$CDCl_3$	[75]
2-4-46	(–)-spectaline	$C_{20}H_{39}NO_2$	$CDCl_3$	[76]
2-4-47	(–)-spectalinine	$C_{20}H_{41}NO_2$	$CDCl_3$	[76]
2-4-48	canavaline	$C_{18}H_{37}NO_2$	$CDCl_3$	[76]
2-4-49	3(*R*)-benzoyloxy-2(*R*)-methyl-6(*R*)-(11′-oxododecyl)-piperidine	$C_{25}H_{39}NO_3$	–	[77]
2-4-50	*iso*-6-spectaline	$C_{20}H_{39}NO_2$	$CDCl_3$	[75]
2-4-51	*iso*-6-canavaline	$C_{18}H_{37}NO_2$	$CDCl_3$	[76]
2-4-52	leptophyllin B	$C_{17}H_{33}NO_3$	CD_3OD	[76]
2-4-53	leptophyllin A	$C_{18}H_{37}NO_3$	$CDCl_3$	[76]
2-4-54	3-acetylleptophyllin A	$C_{20}H_{39}NO_4$	$CDCl_3$	[76]
2-4-55	micropine	$C_{17}H_{29}NO_2$	$CDCl_3$	[78]
2-4-56	uoamine A	$C_{22}H_{41}NO_3S$	$CDCl_3$	[79]
2-4-57	uoamine B	$C_{22}H_{41}NO_3S$	$CDCl_3$	[79]



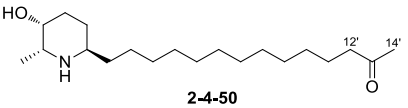
2-4-44 Me Ac
2-4-45 CH_2OH H



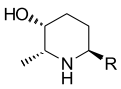
2-4-46 R= $-(CH_2)_{12}COCH_3$
2-4-47 R= $-(CH_2)_{12}CH(OH)CH_3$
2-4-48 R= $-(CH_2)_{10}CH(OH)CH_3$



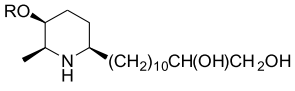
2-4-49



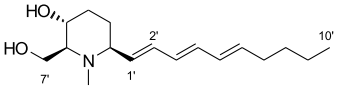
2-4-50



2-4-51 R = -$(CH_2)_{10}CH(OH)CH_3$
2-4-52 R = -$(CH_2)_{10}CO_2H$



2-4-53 R=H
2-4-54 R=Ac



2-4-55

Table 2-4-14: ^{1}H NMR spectroscopic data of piperidine alkaloids **2-4-44**~**2-4-48**.

H	2-4-44	2-4-45	2-4-46	2-4-47	2-4-48
2	2.82 br d(6.5, 2.0)①	2.70 dq(7.5, 2.0)	2.72 dq(6.5, 1.2)	2.72 dq(6.5, 1.2)	2.72 dq(6.5, 1.2)
3	4.75 br s	3.76 br s	3.57 br s	3.57 br s	3.57 br s
4	1.98 ddd(11.5, 6.0, 4.5)	1.96 ddd(12.5, 6.5, 6.0)	1.84 ddd(13, 6, 6)	1.89 ddd(12.3, 6, 6)	1.89 ddd(12, 6, 6)
	1.41 dddt(11.5, 6.0, 3.2)②	1.60 ddd(12.5, 6.5, 3.0)②	1.45 m	1.50 m	1.55 m
5	1.52 m, 1.36 m	1.50 m, 1.36 m	1.40 m	1.51 m, 1.32 m	1.50 m, 1.32 m
6	2.53 m	2.52 m	2.50 m	2.48 m	2.48 m
7	1.00 d(6.5)	3.74 dd(11.0, 4.5) 3.66 dd(11.0, 6.5)	1.06 d(6.5)	1.06 d(6.5)	1.05 d(6.5)
1′	1.36 m	1.46 m	1.32 m	1.35 m	1.35 m
2′	1.26 m	1.26 m	1.22 br s	1.12 br s	1.12 br s
3′	1.23 m	1.19~1.20 br s	1.22 br s	1.12 br s	1.12 br s
4′ ~9′	1.20~1.22 br s	1.19~1.20 br s	1.22 br s	1.12 br s	1.12 br s
10′	1.25 m③	1.23 m	1.22 br s	1.12 br s	1.12 br s
11′	1.50 m③	1.25 m	1.50 dt(7.4, 6)	1.25 m	3.75 m
12′	2.34 t(7.5)	2.40 t(7.5)	2.36 t(7.4)	1.45 m	1.15 d(6.5)
13′				3.75 m	
14′	2.06 s	2.06 s	2.16 s	1.14 d(6.1)	
Ac	2.00 s				

① Two coupling constants were given in the original literature.
② The peaktype was given as dddt in the original literature.
③ There's a print error in the original literature, i. e. both of these two data were assigned to H-10′.

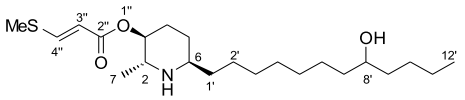
Table 2-4-15: ^{1}H NMR spectroscopic data of piperidine alkaloids **2-4-49**~**2-4-53**.

H	2-4-49	2-4-50	2-4-51	2-4-52	2-4-53
2	3.43 dq(6.6, 4.3)	2.98 br d(6.5, 2.5)①	3.21 dq②(6.5, 2.6)	3.28 dq(6.8, 2.8)②	2.72 dq(6.5, 1.2)②
3	5.12 td(8.11, 4.3)	3.66 br s	3.81 br s	3.82 br s	3.53 br s

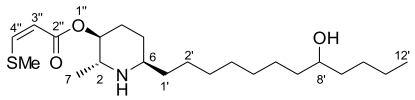
Table 2-4-15 (continued)

H	2-4-49	2-4-50	2-4-51	2-4-52	2-4-53
4	~1.90 (2H)	1.95 ddd(13.0, 10.8, 4.5) 1.49 ddd(10.8, 6.6, 5.0)	1.95 ddd(13, 4, 6.6) 1.70 dd(4, 6.6)	1.95 m	1.85 ddd(13, 6, 6) 1.55 dt(13, 6)
5	~1.51 (2H)	1.50 ddd(13.0, 6.6, 5.5) 1.36 br dd(13.0, 5.0)	1.76 m	1.42 m	– 1.33 m
6	2.92 br s	3.02 ddd(7.2, 5.5, 5.0)	3.02 m	3.02 m	2.51 m
7	1.24 d(6.4)	1.26 d(6.5)	1.30 d(6.5)	1.31 dt(6.8)	1.08 d(6.5)
1′	1.27 m	1.46 dt(11.0, 7.2)	2.33 dt(7.0)②	2.14 dt(6.9, 5.9)	1.35 m
2′	1.27 m	–	1.20 br s	1.22 m	1.20 br s
3′	1.27 m	1.23 m	1.20 br s	1.22 m	1.20 br s
4′ ~8′	1.27 m	1.20~1.22 br s	1.20 br s	1.22 m	1.20 br s
9′	1.54 p(7.3)	1.20~1.22 br s	1.20 br s	1.22 m	1.20 br s
10′	2.38 t(7.3)	1.25 m	1.20 br s	1.22 m	1.20 br s
11′		1.25 m	3.45 m	2.46 t(9)	3.69 m
12′	2.10 s	2.35 t(7.5)	1.30 d(7.6)		3.40 dd(11, 8) 3.62 dd(11, 3)
14′		2.06 s			
3″,7″	8.06 d(7.4)				
4″,6″	7.42 dt(7.4, 1.6)				
5″	7.53 tt(7.4, 1.6)				

① The peaktype of 'br d' is the original peaktype given in the original literature.
② The peaktype in the orignal literature isn't consistent with the structure.



2-4-56



2-4-57

Table 2-4-16: ^{1}H NMR spectroscopic data of piperidine alkaloids **2-4-54**~**2-4-57**.

H	2-4-54	2-4-55	2-4-56	2-4-57
2	2.81 dq(6.5, 1.2)	ax 1.89 ddd(9.1, 3,8, 1.4)	3.35 dq(7, 4)	3.47 br s
3	4.80 br s	ax 3.69 ddd(10.7, 9.2, 4.8)	4.96 ddd(8, 4, 3.5)	5.06 br s
4	1.98 ddd(12.5, 6, 6.5) 1.60 dddt(12.5, 6.5, 3)	ax 1.25~1.45 m eq 2.02 m	*ca.* 1.78 m	–
5	– –	ax 1.25~1.45 m eq 1.64 ddd(9.7, 3.2, 3.2)	*ca.* 1.30 m	–
6	2.48 m	ax 2.61 ddd(11.4, 8.7, 3.2)	2.97 m	3.25 br m

Table 2-4-16 (continued)

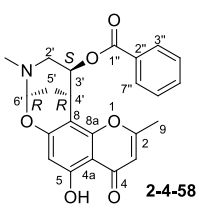
H	2-4-54	2-4-55	2-4-56	2-4-57
7	1.05 d(6.5)	3.84 dd(11.3, 3.8) 3.95 dd(11.3, 1.6)	1.18 d(7)	1.25 d(7)
1′	1.39 m	5.47 dd(15.0, 8.6)	1.29 br s	–
2′ ~5′	1.20 br s	6.0~6.2 m	1.29 br s	–
6′	1.20 br s	5.70 dt(15.0, 7.1)	1.29 br s	–
7′	1.20 br s	2.09 dt(7.1, 7.0)	1.40 m	–
8′	1.20 br s	1.25~1.45 m	3.58 m	3.57 m①
9′	1.20 br s	1.25~1.45 m	1.40 m	–
10′	1.20 br s	0.89 t(7.0)	1.29 br s	–
11′	3.88 m		1.29 br s	–
12′	3.63 dd(10, 3) 3.41 dd(10, 7.6)		0.91 t(7.0)	0.90 t(7)
3″			5.72 d(15)	6.02 d(10)
4″			7.84 d(15)	7.09 d(10)
OH		2.02~2.13 (2H)		
NMe		2.25 s		
SMe			2.33 s	2.40 s
NH			1.80 br s	–

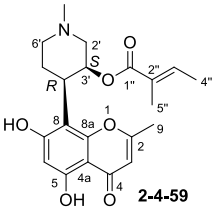
① There's an assignment error in the original literature.

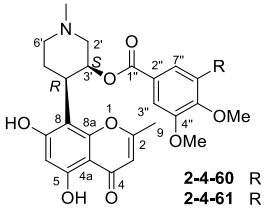
2.4.4 Chromone-type piperidine alkaloids

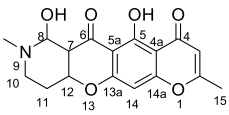
Table 2-4-17: Cos, MFs, and TSs of chromone-type piperidine alkaloids **2-4-58**~**2-4-66**.

No.	Compounds	MFs	Test solvents	References
2-4-58	chrotacumine A	$C_{23}H_{21}NO_6$	CD_3OD	[80]
2-4-59	chrotacumine B	$C_{21}H_{25}NO_6$	CD_3OD	[80]
2-4-60	chrotacumine C	$C_{26}H_{29}NO_9$	CD_3OD	[80]
2-4-61	chrotacumine D	$C_{25}H_{27}NO_8$	CD_3OD	[80]
2-4-62	*N*-methylschumannificine	$C_{17}H_{17}NO_6$	–	[81]
2-4-63	anhydroschumannificine	$C_{16}H_{13}NO_5$	–	[81]
2-4-64	*N*-methylanhydroschumannificine	$C_{17}H_{15}NO_5$	–	[81]
2-4-65	*N*-methylschumagnine	$C_{16}H_{17}NO_4$	$CDCl_3$	[82]
2-4-66	schumagnine	$C_{15}H_{15}NO_4$	$CDCl_3$	[82]

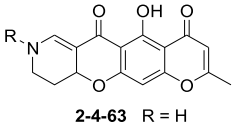




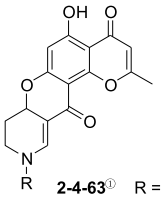




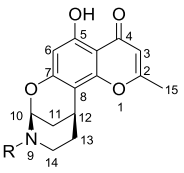
2-4-62



2-4-63 R = H
2-4-64 R = Me



2-4-63① R = H
2-4-64② R = Me



2-4-65 R = Me
2-4-66 R = H

① Proposed structure of compound **2-4-63** to be modified in the literature (Houghton PJ. Planta Med, 1987, 53: 264).

② Proposed structure of compound **2-4-64** to be modified in the literature (Houghton PJ. Planta Med, 1987, 53: 264).

Table 2-4-18: ^{1}H NMR spectroscopic data of piperidine alkaloids **2-4-58**~**2-4-61**.

H	2-4-58	2-4-59	2-4-60	2-4-61
3	5.86 s	6.03 s	5.82 s	5.78 s
6	6.29 s	6.20 s	6.19 s	6.15 s
9	1.63 s	2.41 s	2.23 s	2.23 s
2′	2.45 t(11.0)	2.45 d(12.7)	2.56 d(12.8)	2.50 d(12.7)
	2.92 dd(11.0, 5.2)	3.14 d(12.7)	3.19 d(12.8)	3.18 br d(12.7)
3′	5.51 ddd(11.0, 5.2, 5.2)	5.17 br s	5.53 br s	5.40 br s
4′	3.96 br s	3.48 d(13.1)	3.58 d(13.6)	3.49 d(12.8)
5′	2.29 br d(13.3)	1.81 d(13.1)	1.81 d(13.6)	1.77 d(12.4)
	2.03 ddd(13.3, 3.7, 3.7)	3.24 m	3.30 m	3.27 m
6′	5.11 br s	2.21 t(11.5)	2.29 d(12.8)	2.21 m
		3.11 m	3.19 d(12.8)	3.12 (10.6)①
3″	7.79 d(7.9)	6.89 br q(6.8)	7.33 s	7.48 br s
4″	7.42 t(7.9)	1.72 d(6.8)		
5″	7.60 t(7.9)	1.65 s		
6″	7.42 t(7.9)			6.82 d(8.4)
7″	7.79 d(7.9)		7.33 s	7.60 d(8.4)
NMe	2.61 s	2.33 s	2.41 s	2.32 s
4″-OMe			3.86 s	3.79 s
5″-OMe			3.81 s	3.79 s
6″-OMe			3.86 s	

① Typographic error exists in the literature, no peaktype being given.

Table 2-4-19: ^{1}H NMR spectroscopic data of piperidine alkaloids **2-4-62**~**2-4-66**.

H	2-4-62	2-4-63	2-4-64	2-4-65	2-4-66
3	6.09 s	6.20 s	6.12 s	6.03 s	6.03 s
6				6.28 s③	6.27 s③
7	3.1~3.3 m				
8	5.60 d(6.0)	7.77 dd(6.0, 0.3)	7.72 d(0.3)		
10	3.1~3.3 m	1.5~3.6 m	3.0~3.6 m	5.06 s	5.27 s

Table 2-4-19 (continued)

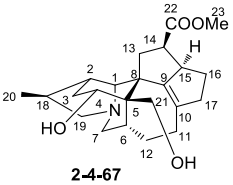
H	2-4-62	2-4-63	2-4-64	2-4-65	2-4-66
11	2.2 m	1.5~3.6 m (2H)	3.0~3.6 m (2H)	1.7~2.8 m	1.8~2.3 m
	2.65 m				2.82 m
12	3.7 m	4.0 dd(9.0, 0.3)①	3.85 dd(12, 0.3)	3.46 m	3.53 m
13				1.7~2.8 m	1.8~2.3 m
14	6.35 s	6.27 s	6.46 s	1.7~2.8 m	1.8~2.3 m
15	2.39 s	2.45 s	2.41 s	2.36 s	2.35 s
NMe	2.97 s		3.14 s	2.52 s	
NH		7.2 br s②			2.82 m
5-OH	12.6 s	12.7 s	12.73 s	12.56 s	12.57 s
8-OH	6.87 s				

① Typographic error exists in the literature, H-12 being typed as OH-12.
② Typographic error exists in the literature, NH being typed as OH-5.
③ Typographic error exists in the literature, H-6 being typed as H-8.

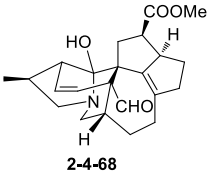
2.4.5 Alkaloids containing piperidine moiety in molecular structure

Table 2-4-20: Cos, MFs, and TSs of piperidine alkaloids **2-4-67**~**2-4-74**.

No.	Compounds	MFs	Test solvents	References
2-4-67	4,21-deacetyl-deoxyyuzurimine	$C_{23}H_{33}NO_4$	$CDCl_3$	[83]
2-4-68	daphhimalenine B	$C_{23}H_{29}NO_4$	$CDCl_3$-TFA(99:1)	[84]
2-4-69	dapholdhamine B	$C_{22}H_{35}NO_3$	$CDCl_3$-CD_3OD(2:1)	[85]
2-4-70	dapholdhamine A	$C_{22}H_{33}NO_3$	$CDCl_3$	[85]
2-4-71	macropodumine L	$C_{25}H_{33}NO_5$	$CDCl_3$	[83]
2-4-72	dapholdhamine C	$C_{23}H_{25}NO_6$	$CDCl_3$-CD_3OD(2:1)	[85]
2-4-73	dapholdhamine D	$C_{26}H_{29}NO_6$	$CDCl_3$	[85]
2-4-74	daphenylline	$C_{21}H_{27}N$	$CDCl_3$	[86]



2-4-67



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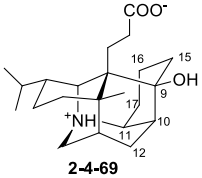

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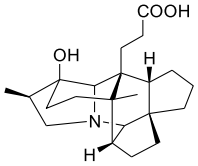

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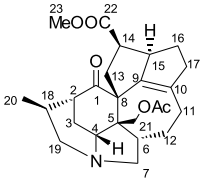

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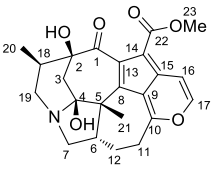

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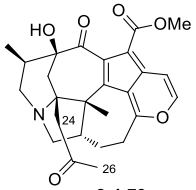

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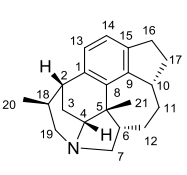


2-4-74

Table 2-4-21: ^{1}H NMR spectroscopic data of piperidine alkaloids **2-4-67**~**2-4-70**.

H	2-4-67	2-4-68	2-4-69	2-4-70
1	2.60 d(3.1)		3.35 d(3.2)	3.15 s
2	2.22 m	3.05 m	1.60 m	
3	*α* 1.51 m, *β* 1.93 m	5.85 dd(10.5, 3.0)	1.80 m, 2.28 m	1.69 m, 1.84 m
4	4.32 dd(12.0, 6.6)	6.16 dd(10.5, 2.0)	1.48 m, 2.04 m	1.67 m, 2.16 m
6	2.65 m	2.18 m	1.70 m	2.40 m
7	3.22 d(12.9)	*α* 3.64 m, *β* 3.28 m	3.45 br d(12.8)	3.58 m
	3.05 dd(12.4, 9.2)		3.91 d(12.8)	
9				2.06 m
10			2.32 m	
11	2.48 m, 2.04 m	*α* 1.94 m, *β* 1.70 m	3.89 d(6.4)	1.88 m, 2.01 m
12	1.90 m, 1.50 m	*α* 2.23 d(16.5)	1.41 m, 1.92 m	1.55 m, 1.37 m
		β 2.62 m		
13	*α* 1.85 m	*α* 2.55 dd(15.5, 9.5)	1.33 m, 2.06 m	1.54 m, 1.79 m
	β 2.89 m	*β* 2.33 dd(15.5, 3.0)		
14	2.94 m	3.08 m	2.16 m, 2.29 m	2.35 m, 2.44 m
15	3.52 m	3.87 br s	1.57 m, 1.92 m	1.56 m, 1.80 m
16	*α* 1.95 m	*α* 1.97 m	1.38 m, 2.63 m	1.77 m, 1.93 m
	β 1.18 m	*β* 1.27 (ov)		
17	*α* 2.75 m	*α* 2.37 d(7.5)	1.39 m, 1.80 m	1.69 m, 1.85 m
	β 2.38 m	*β* 2.64 m		
18	2.38 m	3.18 m	1.58 m	2.37 m
19	3.51 m, 2.28 m	*α* 4.19 dt(12.0, 3.5)	0.93 d(5.0)	3.53 m
		β 2.69 dd(12.0, 3.5)		3.60 dd(11.2, 8.0)
20	1.09 d(6.6)	1.26 d(7.5)	1.00 d(5.1)	1.01 d(6.0, ov)
21	4.24 d(11.6)	10.37 s	0.96 s	1.02 s(ov)
	3.88 d(11.6)			
23	3.66 s	3.61 s		

Table 2-4-22: ^{1}H NMR spectroscopic data of piperidine alkaloids **2-4-71**~**2-4-74**.

H	2-4-71	2-4-72	2-4-73	2-4-74
2	2.16 m			2.75 m
3	*α* 2.05 m, *β* 2.11 m	2.41 d(14.2)	2.10 d(15.6)	*α* 2.37 m
		3.54 d(14.2)	2.74 d(15.6)	*β* 1.95 m
4	3.52 d(4.5)			3.73 m
6	2.57 m	2.89 m	2.66 m	2.45 m
7	2.98 m, 2.66 dd(9.0, 6.5)	2.24 dd(11.6, 8.0)	2.32 dd(11.2, 8.8)	*α* 3.74 m
		3.12 m	3.08 m	*β* 2.46 m
10				3.43 m
11	2.02 m (2H)	2.94 dd(5.2, 2.8)	2.92 m	*α* 2.04 m
		3.61 ddd(16.8, 13.6, 2.8)	3.55 ddd(16.4, 14.0, 2.8)	*β* 1.57 m
12	1.92 m, 1.72 m	1.68 m, 2.55 m	1.66 m	*α* 1.86 m
			2.58 m	*β* 1.30 m
13	*α* 2.22 dd(14.0, 8.0)			6.87 d(7.6)
	β 2.85 t(6.9)			
14	2.74 m			7.05 d(7.6)

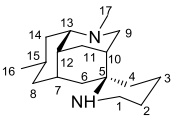
Table 2-4-22 (continued)

H	2-4-71	2-4-72	2-4-73	2-4-74
15	3.42 m			
16	α 1.91 m, β 1.25 m	7.68 br s	7.69 d(4.8)	α 2.76 m, β 2.68 m
17	α 2.66 m	7.68 br s	7.67 d(4.8)	α 1.60 m
	β 2.35 dd(15.0, 9.0)			β 2.33 m
18	2.80 m	2.55 m	2.66 m	1.87 m
19	2.79 m, 2.50 m	2.68 dd(15.6, 2.4)	2.57 m, 3.08 m	α 2.71 m
		3.24 dd(15.6, 10.4)		β 2.97 dd(13.2, 6.0)
20	1.00 d(6.5)	0.90 d(6.8)	0.86 d(6.8)	1.26 d(7.2)
21	4.48 d(11.5), 4.28 d(11.5)	1.53 s	1.38 s	1.45 s
23	3.65 s	3.82 s	3.81 s	
24			2.15 d(12.8), 2.93 m	
26			2.18 s	
OAc	2.07 s			

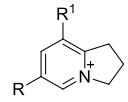
2.4.6 Coupled piperidine alkaloids

Table 2-4-23: Cos, MFs, and TSs of piperidine alkaloids **2-4-75**~**2-4-78**.

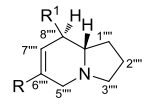
No.	Compounds	MFs	Test solvents	References
2-4-75	nankakurine A	$C_{17}H_{30}N_2$	CD_3OD	[87]
2-4-76	prosopilosidine	$C_{40}H_{72}N_3O_2$	CD_3OD	[88]
2-4-77	prosopilosine	$C_{40}H_{75}N_3O_2$	CD_3OD	[88]
2-4-78	juliprosopine	$C_{40}H_{75}N_3O_2$	CD_3OD	[88]



2-4-75



2-4-76 R = R^1 = Y



2-4-77 R = R^1= Y
2-4-78 R = R^1 = X

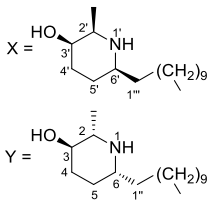


Table 2-4-24: ^{1}H NMR spectroscopic data of piperidine alkaloids **2-4-75**.

H	2-4-75	H	2-4-75	H	2-4-75
1	2.82 t(5.1)	8	1.49 m	13	2.03 m
2	1.58 m, 1.53 m		1.20 ddd(12.6, 12.6, 5.0)	14	2.02 m
3	1.57 m	9	3.00 ddd(12.1, 2.4, 2.4)		0.89 ddd(12.1, 12.1, 2.1)
4	1.66 m		2.14 dd(12.1, 2.8)	15	1.95 m
6	2.29 dd(12.7, 12.7)	10	1.81 m	16	0.85 d(6.6)
	1.64 m	11	1.83 m, 1.53 m	17	2.12 s
7	1.85 m	12	1.53 m		

Table 2-4-25: ^{1}H NMR spectroscopic data of piperidine alkaloids **2-4-76**~**2-4-78**.

H	2-4-76	2-4-77	2-4-78
2,2′	3.19 dq(6)	3.17 q(7.8)	2.75 q(6.5)
3,3′	3.75 ddd(4.8)	3.66 br s	3.48 br s
4,4′	1.70 m, 1.59 m	1.72 m, 1.62 m	1.84 m, 1.46 m
5,5′	1.82 m, 1.21 m	1.49 m, 1.22 m	1.40 m, 1.20 m(ov)
6,6′	2.80 m(ov)	2.85 m	2.56 q(8)
7,7′	1.14 d(7)	1.12 d(6.5)	1.07 d(6.4)
1″,1‴	1.37 m	1.40 m	1.20~1.40 m(ov)
2″ ~8″	1.27~1.35 m(ov)	1.27~1.39 m(ov)	1.20~1.30 m(ov)
2‴ ~8‴	1.27~1.35 m(ov)	1.27~1.39 m(ov)	1.20~1.30 m(ov)
9″,9‴	1.27~1.35 m(ov)	1.43 m	1.20~1.30 m(ov)
10″	2.80 m(ov)	2.00 t(7.5)	2.00 t(7.4)
10‴	2.80 m(ov)	1.27~1.39 m(ov)	1.20~1.40 m(ov)
1′′′′	2.80 m(ov)	1.52 m	1.20~1.40 m(ov)
2′′′′	2.50 m	2.05 m, 1.80 m	2.04 m, 1.76 m
3′′′′	4.88 m(ov)	3.13 dd(18, 7.8), 2.10 dd(18, 9)	3.11 ddd(17.6, 7.8), 2.08 dd(17.6, 9)
5′′′′	8.64 s	3.29 d(15), 2.64 d(15)	3.26 d(15.2), 2.58 d(15.2)
7′′′′	8.18 s	5.37 s	5.33 s
8′′′′		2.00 m	2.00 m
8′′′′a		1.82 m	1.74 m

Bibliography

[54] Ternes W, Krause EL. Anal Bioanal Chem, 2002, 374: 155.
[55] Dragull K, Yoshida WY, Tang CS. Phytochemistry, 2003, 63: 193.
[56] Smith RM. Tetrahedron, 1979, 35: 437.
[57] Duh CY, Wu YC, Wang SK. J Nat Prod, 1990, 53: 1575.
[58] Duh CY, Wu YC, Wang SK. Phytochemistry, 1990, 29: 2689.
[59] Maxwell A, Rampersad D. J Nat Prod, 1991, 54: 1150.
[60] Dodson CD, Dyer LA, Searcy J, et al. Phytochemistry, 2000, 53: 51.
[61] Capron MA, Wiemer DF. J Nat Prod, 1996, 59: 794.
[62] Araujo-Junior JXD, Da-Cunha EVL, Chaves MCDO, et al. Phytochemistry. 1997;44:559.
[63] Filho RB, Souza MPD, Mattos MEO. Phytochemistry, 1981, 20: 345.
[64] Schneider MJ, Brendze S, Montali JA. Phytochemistry, 1995, 39: 1387.
[65] Schneider MJ, Stermitz FR. Phytochemistry, 1990, 29: 1811.
[66] Tawara JN, Blokhin A, Foderaro TA, et al. J Org Chem, 1993, 58: 4813.
[67] Lognay G, Hemptinne JL, Chan FY, et al. J Nat Prod, 1996, 59: 510.
[68] Yamashita T, Yasuda K, Kizu H, et al. J Nat Prod, 2002, 65: 1875.
[69] Asano N, Nishida M, Kizu H, et al. J Nat Prod, 1997, 60: 98.
[70] Asano N, Nishida M, Kato A, et al. J Med Chem, 1998, 41: 2565.
[71] Yasuda K, Kizu H, Yamashita T, et al. J Nat Prod, 2002, 65: 198.
[72] Asano N, Tomioka E, Kizu H, et al. Carbohydr Res, 1994, 253: 235.
[73] Asano N, Nishida M, Miyauchi M, et al. Phytochemistry, 2000, 53: 379.
[74] Asano N, Oseki K, Tomioka E, et al. Carbohydr Res, 1994, 259: 243.
[75] Viegas C, Bolzani VS, Furlan M, et al. J Nat Prod, 2004, 67: 908.

[76] Bolzani VS, Gunatilake AAL, Kingston DGI. Tetrahedron, 1995, 51: 5929.
[77] Sriphong L, Sotanaphun U, Limsirichaikul S, et al. Planta Med, 2003, 69: 1054.
[78] Aguinaldo AM, Read RW. Phytochemistry, 1990, 29: 2309.
[79] McCoy MC, Faulkner DJ. J Nat Prod, 2001, 64: 1087.
[80] Ismail IS, Nagakura Y, Hirasawa Y, et al. J Nat Prod, 2009, 72: 1879.
[81] Houghton PJ, Yang H. Planta Med, 1985, 51: 23.
[82] Houghton PJ, Yang H. Planta Med, 1987, 53: 262.
[83] Li ZY, Xu HG, Zhao ZZ, et al. J Asian Nat Prod Res, 2009, 11: 153.
[84] Zhang Y, Di YT, Zhang Q, et al. Org Lett, 2009, 11: 5414.
[85] Zhang Y, Di YT, Mu SZ, et al. J Nat Prod, 2009, 72: 1325.
[86] Zhang Q, Di YT, Li CS, et al. Org Lett, 2009, 11: 2357.
[87] Hirasawa Y, Morita H, Kobayashi J. Org Lett, 2004, 6: 3389.
[88] Samoylenko V, Ashfaq MK, Jacob MR, et al. J Nat Prod, 2009, 72: 92.

2.5 Licopodium alkaloids

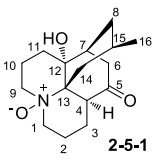
2.5.1 Licopodine-type licopodium alkaloids

Table 2-5-1: Cos, MFs, and TSs of licopodine-type licopodium alkaloids **2-5-1**~**2-5-26**.

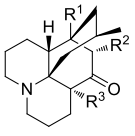
No.	Compounds	MFs	Test solvents	References
2-5-1	12-*epi*lycodoline *N*-oxide	$C_{16}H_{25}NO_3$	$CDCl_3$	[89]
2-5-2	7-hydroxylycopodine	$C_{16}H_{25}NO_2$	CD_3OD	[89]
2-5-3	4,6*α*-dihydroxylycopodine	$C_{16}H_{25}NO_3$	CD_3OD	[89]
2-5-4	serratezomine C	$C_{16}H_{25}NO_3$	CD_3OD	[90]
2-5-5	lycoposerramine G	$C_{16}H_{25}NO_3$	$CDCl_3$	[91]
2-5-6	lycoposerramine L	$C_{16}H_{25}NO_2$	$CDCl_3$	[91]
2-5-7	lycoposerramine M	$C_{16}H_{25}NO_2$	$CDCl_3$	[91]
2-5-8	lycoposerramine N	$C_{18}H_{27}NO_4$	$CDCl_3$	[91]
2-5-9	obscurumine B	$C_{18}H_{27}NO_3$	$CDCl_3$	[92]
2-5-10	2-chlorohuperzine E	$C_{16}H_{20}ClNO_2$	$CDCl_3$	[93]
2-5-11	huperzine E'	$C_{16}H_{19}NO_2$	$CDCl_3$	[93]
2-5-12	huperzine F'	$C_{16}H_{19}NO_3$	$CDCl_3$	[93]
2-5-13	lycopodatine A	$C_{17}H_{28}NO_2$	CD_3OD	[94]
2-5-14	lycopodatine B	$C_{17}H_{26}NO$	CD_3OD	[94]
2-5-15	lycopodatine C	$C_{16}H_{23}NO$	CD_3OD	[94]
2-5-16	lycoposerramine K	$C_{16}H_{23}NO_2$	$CDCl_3$	[91]
2-5-17①	miyoshianine A	$C_{16}H_{25}NO_4$	CD_3OD	[95]
2-5-18①	lycoposerramine F	$C_{16}H_{25}NO_4$	CD_3OD	[91]
2-5-19	obscurumine A	$C_{16}H_{25}NO_3$	CD_3OD	[92]
2-5-20②	miyoshianine B②	$C_{16}H_{25}NO_2$	CD_3OD	[95]
2-5-21	lycoposerramine H	$C_{16}H_{23}NO_2$	$CDCl_3$	[91]
2-5-22	lycoposerramine I	$C_{16}H_{23}NO_2$	$CDCl_3$	[91]
2-5-23②	lycoposerramine J	$C_{16}H_{25}NO_2$	$CDCl_3$	[91]
2-5-24	lycoposerramine O	$C_{28}H_{39}NO_6$	$CDCl_3$	[91]
2-5-25	lyconesidine C	$C_{29}H_{39}NO_6$	CD_3OD	[96]
2-5-26	complanadine C	$C_{32}H_{42}N_2O_3$	CD_3OD	[97]

① Compounds **2-5-17** and **2-5-18** possess the exact same structure and the same solvent was used for their NMR test. But, their ^{1}H NMR data are very different. Thus, the two compounds are embodied in this handbook.

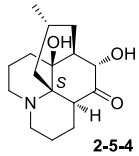
② Compounds **2-5-20** and **2-5-23** possess the exact same structure and the same solvent was used for their NMR test. But, their ^{1}H NMR data are very different. Thus, the two compounds are embodied in this handbook.



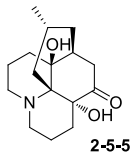
2-5-1



	R^1	R^2	R^3
2-5-2	OH	H	H
2-5-3	H	OH	OH



2-5-4



2-5-5

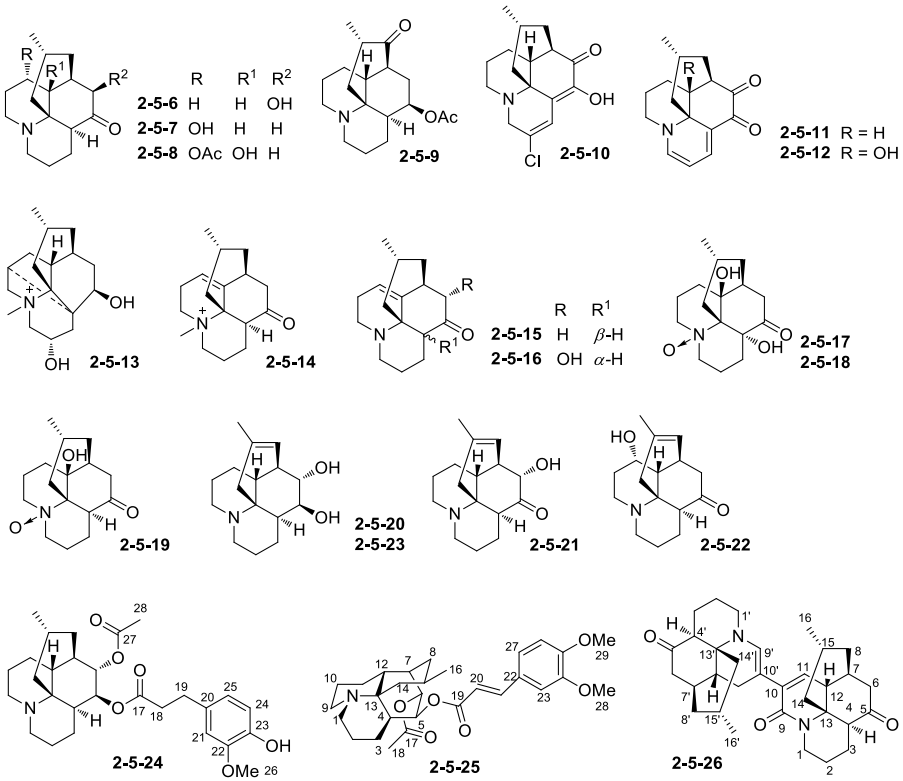


Table 2-5-2: ^{1}H NMR spectroscopic data of licopodine-type licopodium alkaloids **2-5-1**~**2-5-4**.

H	2-5-1	2-5-2	2-5-3	2-5-4
1	α 2.89 dd(13.3, 4.5)	α 3.15 dt(13.1, 2.1)	α 3.97 dt(14.0, 5.4)	2.48 br d(11.8)
	β 3.58 td(13.7, 4.9)	β 3.78 td(13.2, 2.0)	β 3.68 td(14.0, 4.1)	3.25 m
2	α 1.81 qt(14.1, 4.6)	α 1.61 qt(13.2, 5.0)	α 2.30 qt(14.4, 5.2)	2.01 m, 1.42 m
	β 1.90 br d(14.3)	β 2.11 br d(13.2)	β 1.77 br d(14.8)	
3	α 2.16 br d(15.5)	α 1.94 qt(13.2, 4.2)	α 1.95 br d(13.3)	1.63 m, 1.99 m
	β 1.64 qd(15.5, 4.7)	β 1.88 br d(13.2)	β 1.87 td(14.3, 4.5)	
4	2.84 dd(12.0, 3.1)	3.26 dd(13.2, 3.0)		3.48 br d(11.9)
6	α 2.59 dd(17.0, 6.1)	α 2.72 d(15.9)		3.85 d(1.5)
	β 2.38 dd(17.0, 1.3)	β 2.37 dd(15.9, 1.3)	β 3.98 br s	
7	2.04 br s		2.33 br s	1.97 m
8	ex 2.07 td(13.1, 3.5)	ex 1.33 td(12.1, 1.9)	ex 1.30 td(12.6, 4.2)	1.32 m, 2.00 m
	en 1.29 dt(13.4, 1.9)	en 1.84 dd(13.4, 3.6)	en 1.74 dd(12.6, 2.2)	
9	α 3.01 dt(12.7, 4.8)	α 3.70 td(13.8, 4.5)	α 4.40 ddd(13.3, 11.9, 3.8)	3.39 m
	β 4.03 td(12.7, 3.5)	β 3.00 dd(13.7, 4.8)	β 2.98 br d(12.8)	2.61 br d(11.7)
10	α 2.97 q(13.5, 4.5)①	α 2.11 br d(13.5)	α 1.98 br d(14.5)	1.61 m, 2.22 m
	β 1.74 br d(14.1)	β 1.93 qt(13.5, 4.6)	β 1.87 m	

Table 2-5-2 (continued)

H	2-5-1	2-5-2	2-5-3	2-5-4
11	*α* 1.61 dd(13.4, 5.0) *β* 2.21 td(13.4, 4.4)	*α* 1.78 qd(12.8, 3.3) *β* 2.14 br d(13.0)	*α* 2.87 qd(13.4, 4.3) *β* 1.56 dq(13.3, 3.0)	2.79 dt(14.5, 3.3) 1.48 br d(14.5)
12		1.96 dd(13.4, 3.6)	1.86 dd(9.9, 4.4)	
14	ex 2.72 t(13.2) en 1.86 dd(13.0, 5.6)	ex 1.28 t(12.2) en 2.68 dd(12.2, 3.0)	ex 1.18 t(12.7) en 2.54 dd(13.5, 3.8)	2.25 dd(13.0, 4.9) 1.58 t(13.0)
15	1.31 m	1.39 m	1.33 m	1.27 m
16	0.92 d(6.2)	0.95 d(5.7)	0.84 d(5.6)	0.87 d(6.1)

① Typographic error for the peaktype exists in the literature.
Note: en denotes *endo* and ex *exo*.

Table 2-5-3: ^{1}H NMR spectroscopic data of licopodine-type licopodium alkaloids **2-5-5**~**2-5-7**.

H	2-5-5	2-5-6	2-5-7
1	3.35 ddd(14.0, 14.0, 3.9) 2.56 m	3.38 ddd(14.0, 14.0, 3.7) 2.53 dd(14.6, 4.9)	3.36 ddd(14.3, 14.3, 3.7) 2.57 dd(14.2, 4.4)
2	1.44 m 2.28 ddddd(13.7, 13.7, 13.7, 5.2, 5.2)	1.86 m 1.37 br d(14.6)	1.40 br d(14.3) 1.87 ddddd(13.7, 13.7, 13.7, 4.9, 4.9)
3	2.17 m, 1.73 m	1.73 m, 2.04 br d(10.7)	1.53 m, 2.05 m
4		3.02 br d(11.9)	3.50 m
6	3.29 ddd(15.9, 5.8, 1.5) 2.23 dd(15.9, 2.1)	4.24 dd(6.4, 1.5) –	3.31 dd(15.1, 6.0) 2.16 d(16.2)
7	2.10 m	2.41 br s	2.33 m
8	2.00 dddd(12.4, 12.4, 4.1, 1.4) 1.37 m	2.10 br d(16.8) 1.01 ddd(12.9, 12.9, 4.1)	1.68 br d(12.8) 1.28 ddd(12.5,12.5,3.7)
9	2.55 m, 3.99 ddd(13.1, 10.9, 4.6)	2.66 m3.12 ddd(11.9, 11.9, 2.7)	3.54 ddd(12.7, 12.7, 2.3)2.48 m
10	1.73 m, 2.10 m	1.60~1.86 m (2H)	2.05 m, 1.75 dddd(14.0, 2.7, 2.7, 2.7)
11	1.44 m 2.91 ddd(13.1, 13.1, 5.2)	1.60~1.86 m (2H)	4.22 ddd(2.8, 2.8, 2.8) –
12		1.60~1.86 m	1.53 m
14	2.10 m, 1.54 dd(13.1, 13.1)	2.63 dd(13.4, 4.6), 0.91 m	2.64 dd(13.6, 4.4), 0.85 m
15	1.44 m	1.22 m	1.53 m
16	0.85 d(6.1)	0.83 d(6.1)	0.85 d(6.1)

Table 2-5-4: ^{1}H NMR spectroscopic data of licopodine-type licopodium alkaloids **2-5-8**~**2-5-10**.

H	2-5-8	2-5-9	2-5-10
1	3.22 ddd(14.2, 14.2, 3.8) 2.49 dd(14.7, 5.2)	2.64 m 3.29 dt(14.0, 3.3)	*α* 3.09 d(19.3) *β* 4.15 dd(19.3, 2.6)
2	1.40 br d(15.0) 1.90 ddddd(13.7, 13.7, 13.7, 5.4, 5.4)	1.41 m, 1.90 m	

Table 2-5-4 (continued)

H	2-5-8	2-5-9	2-5-10
3	2.10 br d(12.5), 1.66 m	1.44 m, 1.74 m	6.83 d(2.5)
4	3.44 dd(11.9, 3.7)	2.40 br d(12.5)	
5		4.91 br d(1.9)	
6	2.85 dd(17.2, 6.9), 2.32 dd(17.4, 1.2)	1.93 m, 1.96 m	
7	–	2.29 br d(2.2)	2.56~2.59 m
8	2.20 dd(3.5, 3.5)		ex 1.30 dd(12.9, 4.6)
	2.00 ddd(13.1, 13.1, 4.0)		en 1.79 d(13.1)
8[①]	1.31 br d(11.9)[①]		
9	2.46 m	2.66 m	*α* 2.72~2.78 m
	3.33 ddd(12.8, 12.8, 3.1)	3.19 td(12.4, 2.9)	*β* 2.66 d(11.3)
10	1.75 dddd(15.0, 2.5, 2.5, 2.5)	1.79 m, 1.87 m	*α* 1.61~1.66 m
	2.42 m		*β* 1.61~1.66 m
11	4.98 dd(2.7, 2.7)	1.38 m, 1.58 m	*α* 1.33~1.37 m,
			β 1.47 ddd(9.9, 8.7, 3.2)
12		2.19 br d(11.4)	1.75 dd(14.3, 2.2)
14	2.38 m	1.69 m	ex 1.05 dd(12.3, 12.2)
	1.26 dd(13.1, 1.31[②])	2.98 dd(14.0, 9.7)	en 2.07 dd(12.4, 4.4)
15	1.48 m	2.77 m	1.55~1.62 m
16	0.87 d(6.1)	1.28 d(7.7)	0.92 d(6.5)
OH	2.97 s		6.29 s
OAc	2.07 s	1.89 s	

[①] Typographic errors exist in the literature and this is the redundant data given in the literature.
[②] Typographic errors exist in the literature.

Table 2-5-5: ^{1}H NMR spectroscopic data of licopodine-type licopodium alkaloids **2-5-11**~**2-5-14**.

H	2-5-11	2-5-12	2-5-13	2-5-14
1	6.83 d(5.8)	6.81 d(5.8)	3.25 dd(14.2, 1.8)	3.40 br d(13.6)
			3.82 dd(14.2, 6.9)	3.79 ddd(13.6, 13.6, 3.7)
2	5.36 dd(7.1, 5.9)	5.45 dd(7.1, 5.9)	4.33 m	1.94 m, 2.00 m
3	7.68 d(7.1)	7.64 d(7.1)	1.58 m,	1.70 m,
			3.08 dd(15.9, 8.9)	2.18 dd(14.9, 2.0)
4				3.37 m
5			3.95 d(8.6)	
6			1.42 d(16.2)	2.51 d(16.5)
			2.57 ddd(16.2, 8.1, 8.1)	2.72 dd(16.5, 6.6)
7	2.87~2.92 m	2.82 dd(5.3, 2.0)	2.20 m	3.04 m
8	ex 1.39 td(13.3, 4.8)	ex 1.82 m	1.14 ddd (13.0, 13.0, 3.0)	1.37 ddd(12.9, 12.8, 3.2)
	en 1.85 m (ov)	en 1.61~1.64 dm	1.65 m	1.81 br d(12.9)

Table 2-5-5 (continued)

H	2-5-11	2-5-12	2-5-13	2-5-14
9	*α* 3.83 ddd(12.6, 12.5, 5.8)	*α* 4.49 ddd(12.1, 11.9, 6.0)	3.04 d(11.4)	3.23 dd(12.6, 6.1)
	β 3.33 dd(12.6, 6.9)	*β* 3.24 dd(11.5, 7.3)	4.66 ddd(11.4, 3.8, 3.8)	4.29 ddd(12.6, 12.3, 4.7)
10	*α* 1.75 m (ov)*β* 1.75 m (ov)	*α* 1.84 m (ov)*β* 1.74 m (ov)	2.30 m	2.52 m, 2.78 m
11	*α* 1.77 m (ov)*β* 1.91 m (ov)	*α* 2.04~2.78 m (ov)*β* 1.77 m (ov)	1.62 m, 1.89 br d(13.9)	5.90 d(5.5)
12	2.32~2.37 m		2.37 m	
14	ex 0.95 dd(12.1, 11.8)	ex 1.46 dd(11.9, 11.8)	1.37 dd(12.6, 12.4)	1.66 dd(11.8, 11.8)
	en 2.40 ddd(11.7, 4.1, 1.5)	en 2.09 dd(12.0, 8.1)	1.93 dd(12.6, 5.3)	2.50 m
15	1.97~2.03 m	1.28~1.31 m	2.70 m	1.73 m
16	0.92 d(6.3)	0.90 d(6.3)	1.01 d(6.4)	0.99 d(5.8)
17			3.00 s	3.16 s

Table 2-5-6: ^{1}H NMR spectroscopic data of licopodine-type licopodium alkaloids **2-5-15**~**2-5-17**.

H	2-5-15	2-5-16	2-5-17②
1	2.79 m	3.08 dd(12.5, 12.5)	*α* 3.04 br d(12.0)
	3.01 ddd(12.0, 12.0, 2.9)	2.62 m	*β* 3.66 td(12.0, 4.0)
2	1.62 m, 1.80 m	1.69 m, 1.60 m	*α* 2.23 qt(14.0, 4.6), *β* 1.88 (ov)
3	1.42 dddd (13.2, 13.2, 13.2, 3.5)	1.98 br d(11.0), 1.51 m	*α* 1.89 (ov)
	1.65 m		*β* 2.15 td(14.5, 4.6)
4	2.42 dd(13.6, 3.6)	2.99 br d(9.5)	
6	2.28 d(18.3), 2.78 m	3.85 d(2.4)	*α* 3.37 ddd(16.2, 6.1, 1.6)
			β 2.28 dd(16.1, 2.1)
7	2.79 m	2.68 ddd(4.9, 2.4, 2.4)	2.06 m
8	1.28 ddd(12.4, 12.4, 4.2)	1.83 dddd (13.4, 4.2, 2.1, 2.1)	ex 2.09 td(12.4, 1.5)
	1.82 m	1.32 ddd(13.1, 13.1, 4.9)	en 1.43 br d(12.5)
9	2.76 m	2.71 m, 2.86 m	*α* 5.02 ddd(12.8, 11.6, 4.1)
	3.43 ddd(13.6, 11.7, 4.9)		*β* 3.05 br d(12.5)
10	1.90 m, 2.61 m	2.34 m (2H)①	*α* 1.78 br d(13.5)
			β 2.75 qt(13.3, 5.3)
11	5.81 d(5.7)	5.73 dd(3.8, 3.8)	*α* 3.09 td(13.3, 5.3), *β* 1.42 m
14	1.72 m, 1.83 m	2.37 dd(13.4, 3.4), 1.10 m	en 1.97 dd(13.7, 5.0), ex 2.76 t(13.6)

Table 2-5-6 (continued)

H	2-5-15	2-5-16	2-5-17[②]
15	1.83 m	1.51 m	1.75 m
16	0.89 d(5.7)	0.84 d(6.1)	0.98 d(6.1)

[①] This data was assigned to H-12 incorrectly in the literature.
[②] Compounds **2-5-17** and **2-5-18** possess the exact same structure and the same solvent was used for their NMR test. But, their ^{1}H NMR data are very different. Thus, the two compounds are embodied in this handbook.

Table 2-5-7: ^{1}H NMR spectroscopic data of licopodine-type licopodium alkaloids **2-5-18**~**2-5-20**.

H	2-5-18[①]	2-5-19	2-5-20[②]
1	2.93 m	2.92 dd(13.3, 4.8)	*α* 3.0 br d(13.3)
	3.52 ddd(13.8, 13.8, 4.2)	3.62 dt(13.5, 5.0)	*β* 3.60 td(13.4, 3.8)
2	1.75 m, 2.05 m	1.91 m, 1.98 m	*α* 2.15 qt(13.6, 4.4), *β* 1.86 m
3	1.75 m, 2.05 m	2.12 br d(14.3), 1.69 m	*α* 1.57 br d(13.0), *β* 2.10 (ov)
4		3.30 m	2.73 m
5			3.74 dd(3.3, 1.3)
6	2.14 dd(16.0, 2.0), 3.24 m	2.36 dd(17.1, 1.7)[③]	*α* 3.76 br s
7	1.93 m	2.77 m	2.43 br d(7.6)
8	1.30 m, 1.93 m	1.37 br d(12.4), 2.01 m	en 5.58 dd(6.8, 1.9)
9	2.93 m	2.98 dd(12.8, 4.7)	*α* 3.79 td(13.2, 3.3)
	4.89 ddd(15.4, 10.2, 4.0)	4.31 dt(13.0, 3.7)	*β* 3.02 br d(13.1)
10	1.64 m, 2.62 m	1.81 br d(6.7), 2.79 m	*α* 1.97 dt(13.5, 2.8)
			β 1.72 qt(13.6, 3.8)
11	2.93 m, 1.30 m	1.53 br d(13.5)	*α* 2.50 qt(13.5, 3.6)
		2.48 dt(13.7, 4.6)	*β* 1.62 dd(13.4, 2.0)
12			2.01 dt(13.7, 2.9)
14	2.62 m	2.08 dd(13.4, 4.5)	en 2.06 br d(16.9)
	1.84 dd(13.9, 5.0)	2.52 t(12.2)	ex 3.00 br d(17.0)
15	1.60 m	1.49 m	
16	0.84 d(6.1)	0.97 d(6.2)	1.65 s

[①] Compounds **2-5-17** and **2-5-18** possess the exact same structure and the same solvent was used for their NMR test. But, their ^{1}H NMR data are very different. Thus, the two compounds are embodied in this handbook.
[②] Compounds **2-5-20** and **2-5-23** possess the exact same structure and the same solvent was used for their NMR test. But, their ^{1}H NMR data are very different. Thus, the two compounds are embodied in this handbook.
[③] The data of 6b was not given in the literature.

Table 2-5-8: ^{1}H NMR spectroscopic data of licopodine-type licopodium alkaloids **2-5-21**~**2-5-23**.

H	2-5-21	2-5-22	2-5-23[①]
1	2.59 m	2.61 m	2.64 br d(14.0)
	3.18 m	3.18 ddd(14.0, 14.0, 3.5)	3.27 ddd(13.4, 13.4, 3.4)

Table 2-5-8 (continued)

H	2-5-21	2-5-22	2-5-23[①]
2	1.34 br d(12.2), 1.88 m	1.35 br d(14.6), 1.85 m	1.38 br d(10.4), 2.00 m
3	1.79 m, 1.86 m	1.80 m, 1.69 m	1.55 m, 2.00 m
4	3.65 dd(9.5, 5.8)	3.63 dd(12.1, 3.5)	2.59 m
5			3.56 dd-like(11.1, 4.2)
6	3.78 d(2.7)	3.45 dd(13.4, 5.2), 2.08 m	3.90 s
7	2.52 br s	2.63 m	2.37 d(4.9)
8	5.35 d(5.2)	5.49 d(5.2)	5.72 dd-like(6.4, 1.5)
9	2.62 m	2.46 ddd(12.0, 4.2, 2.8)	2.54 br d(12.5)
	3.20 m	3.58 ddd(12.7, 12.7, 2.3)	3.22 ddd(12.2, 12.2, 2.4)
10	1.63 m, 1.85 m	1.80 m, 2.08 m	1.56 m, 1.74 m
11	2.45 dddd (13.4, 13.4, 13.4, 3.7)	4.34 ddd(2.8, 2.8, 2.8)	2.17 dddd (13.3, 13.3, 13.3, 3.5)
	1.69 m		1.50 m
12	1.95 m	1.87 m	1.78 m
14	2.70 d(18.3), 1.98 m	1.94 d(17.7), 2.73 d(18.0)	2.96 d(18.3), 2.05 m
16	1.58 s	1.57 s	1.69 s
5-OH			1.85 (11.0)

[①] Compounds **2-5-20** and **2-5-23** possess the exact same structure and the same solvent was used for their NMR test. But, their ^{1}H NMR data are very different. Thus, the two compounds are embodied in this handbook.

Table 2-5-9: ^{1}H NMR spectroscopic data of licopodine-type licopodium alkaloids **2-5-24**~**2-5-26**.

H	2-5-24	2-5-25	H	2-5-26
1	2.52 m[①]	3.00 dd(13.4, 4.8)	1	4.42 dd(13.6, 5.3)
		3.77 ddd(13.4, 13.4, 3.6)		3.08 ddd(13.2, 13.2, 2.4)
2[①]	3.37 ddd(14.2, 14.2, 3.5)		2	1.65 m, 1.49 m
2	1.28 m	1.81 m	3	2.04 m, 1.68 m
	1.92 ddddd (13.6, 13.6, 13.6, 4.7, 4.7)	2.09 m	4	2.70 m
3	1.30 m, 1.47 m	1.90 m, 1.58 m	6	2.59 m, 2.29 m
4	2.66 ddd(11.3, 8.5, 2.7)	3.15 m	7	2.50 m
5	5.06 d(7.0)	5.28 d(7.1)	8	1.78 m
6	4.60 s	4.77 br s		1.38 ddd(12.6, 12.6, 4.2)
7	1.86 br s	2.10 m	11	5.96 d(2.3)
8	1.74 m	1.40 ddd(13.3, 13.3, 4.8)	12	2.73 m
	1.23 m	1.93 m	14	1.16 t(12.6), 2.59 m
9	2.53 m, 3.16 ddd(12.2, 12.2, 2.4)	3.10 m, 3.90 m		
10	1.74 m	1.82 m, 2.02 m	15	1.55 m

Table 2-5-9 (continued)

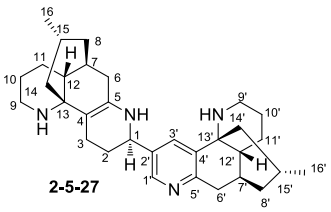
H	2-5-24	2-5-25	H	2-5-26
11	1.76 m, 1.30 m	1.57 m, 2.16 m	16	0.91 d(6.2)
12	1.42 m	1.83 m	1′	3.37 ddd(13.8, 13.8, 3.0)
14	0.85 m, 2.61 m	1.22 dd(12.9, 12.9)		2.92 dd(13.8, 4.8)
		2.90 dd(12.9, 6.0)	2′	1.52 m (2H)
15	2.35 m	2.79 m	3′	2.04 m, 1.62 m
16	0.88 d(6.4)	1.08 d(6.3)	4′	2.62 m
18	2.61 dd(7.6, 7.6)	2.09 s	6′	2.75 m, 2.23 m
19	2.88 dd(7.8, 7.8)		7′	2.31 m
20		6.40 d(15.9)	8′	1.75 m
21	6.70 s	7.64 d(15.9)		1.30 ddd(12.6, 12.6, 3.6)
23		7.23 d(2.0)	9′	7.22 s
24	6.85 d(7.6)		11′	2.55 m, 2.19 m
25	6.69 dd(8.2, 1.8)		12′	1.99 m
26	3.89 s	6.99 d(8.3)	14′	2.56 m, 0.91 m
27		7.19 dd(8.3, 2.0)	15′	1.50 m
28	2.04 s②	3.87 s	16′	0.87 d(6.2)
29		3.87 s		

① Typographic errors exist in the literature, giving one more H-2 (δ 3.37) and one less H-1.
② Typographic error exists in the literature, designating this signal as H-27 which is discrepant with the structure.

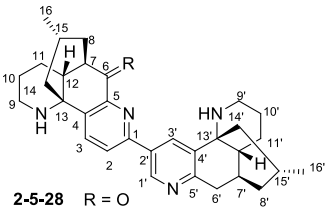
2.5.2 Lycodine-type licopodium alkaloids

Table 2-5-10: Cos, MFs, and TSs of lycodine-type licopodium alkaloids **2-5-27**~**2-5-42**.

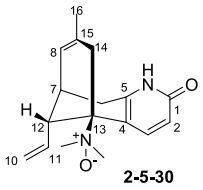
No.	Compounds	MFs	Test solvents	References
2-5-27	complanadine D	$C_{32}H_{46}N_4$	CD_3OD	[97]
2-5-28	complanadine B	$C_{32}H_{40}N_4O$	CD_3OD	[92]
2-5-29	complanadine A	$C_{32}H_{42}N_4$	CD_3OD	[98]
2-5-30	huperzinine *N*-oxide	$C_{17}H_{22}N_2O_2$	$CDCl_3$	[99]
2-5-31	huperzinine	$C_{17}H_{22}N_2O$	$CDCl_3$	[100]
2-5-32	huperzine C	$C_{15}H_{18}N_2O$	$CDCl_3$	[100]
2-5-33	huperzine D	$C_{17}H_{22}N_2O_2$	$CDCl_3$-CD_3OD	[100]
2-5-34	8,15-dihydrohuperzinine	$C_{17}H_{24}N_2O$	CD_3OD	[99]
2-5-35	huperzine U	$C_{16}H_{22}N_2O_2$	CD_3OD	[101]
2-5-36	carinatumin A	$C_{15}H_{18}N_2O_2$	CD_3OD	[102]
2-5-37	carinatumin B	$C_{16}H_{20}N_2O_2$	CD_3OD	[102]
2-5-38	11-hydroxylycodine	$C_{16}H_{22}N_2O$	$CDCl_3$	[103]
2-5-39	lycopladine F	$C_{20}H_{29}N_3O_2$	CD_3OD	[104]
2-5-40	lycopladine G	$C_{21}H_{28}N_2O_3$	CD_3OD	[104]
2-5-41	hydroxypropyllycodine	$C_{19}H_{28}N_2O$	$CDCl_3$	[105]
2-5-42	himeradine A	$C_{29}H_{45}N_3O$	CD_3OD	[106]



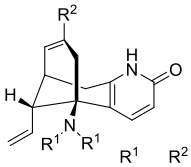
2-5-27



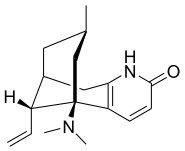
2-5-28 R = O
2-5-29 R = H_2



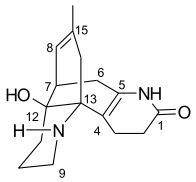
2-5-30



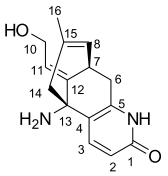
	R^1	R^2
2-5-31	Me	Me
2-5-32	H	Me
2-5-33	Me	CH_2OH



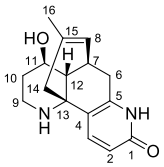
2-5-34



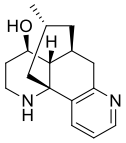
2-5-35



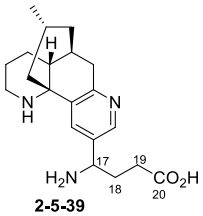
2-5-36



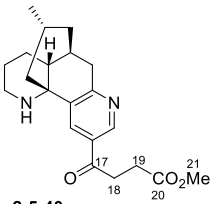
2-5-37



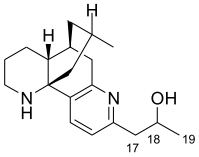
2-5-38



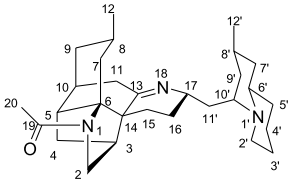
2-5-39
(17*S*/17*R*, 3.5:1)



2-5-40



2-5-41



2-5-42

Table 2-5-11: ^{1}H NMR spectroscopic data of lycodine-type licopodium alkaloids **2-5-27**~**2-5-29**.

H	2-5-27	2-5-28	2-5-29
1	4.44 dd(4.3, 4.3)		
2	2.14 m, 1.96 m	8.31 d(8.3)	7.89 d(8.2)
3	1.96 m, 1.57 m	8.26 d(8.3)	8.00 d(8.2)
6	2.50 dd(16.8, 5.8)		2.82 d(19.2)
	1.77 m		3.27 dd(19.2, 7.2)
7	1.92 m	2.82 m	2.26 m
8	1.70 m	1.61 dt(13.0, 3.9)	1.43 dt(13.0, 3.9)
	1.26 ddd(12.6, 12.6, 3.8)	1.93 br d(13.4)	1.86 br d(12.7)

Table 2-5-11 (continued)

H	2-5-27	2-5-28	2-5-29
9	2.76 m	2.48 dt(12.5, 2.7)	2.73 m
	2.31 ddd(11.5, 11.5, 2.8)	2.82 t(14.3)	3.05 br d(13.5)
10	1.61 m, 1.47 m	1.68 m (2H)	1.80 m (2H)
11	1.66 m, 1.55 m	1.22 m, 1.60 m	1.67 m (2H)
12	1.55 m	2.04 br d(12.3)	1.96 br s
14	1.70 m	1.52 t(12.3)	1.53 dd(12.1, 4.8)
	0.96 dd(11.5, 11.5)	1.70 m	1.74 m
15	1.80 m	1.31 m	1.29 m
16	0.94 d(6.6)	0.87 d(6.4)	0.86 d(6.4)
1′	8.24 d(2.2)	9.07 d(2.1)	9.03 d(2.1)
3′	7.89 d(2.2)	8.81 d(2.1)	8.61 d(2.1)
6′	3.13 dd(18.7, 7.1)	2.74 m	2.77 d(19.2)
	2.65 d(18.2)	3.22 dd(19.0, 6.9)	3.24 dd(19.2, 7.2)
7′	2.11 m	2.18 m	2.26 m
8′	1.80 m	1.43 dt(13.0, 3.9)	1.43 dt(13.0, 3.9)
	1.39 ddd(12.6, 12.6, 3.8)	1.83 br d(13.4)	1.86 br d(12.7)
9′	2.74 m	2.50 dt(12.5, 2.7)	2.73 m
	2.42 ddd(12.6, 12.6, 2.7)	2.79 t(14.3)	3.07 br d(13.5)
10′	1.64 m (2H)	1.68 m(2H)	1.80 m(2H)
11′	1.56 m, 1.14 m	1.20 m, 1.60 m	1.67 m(2H)
12′	1.70 m	1.79(12.6)①	1.94 br s
14′	1.47 m	1.39 t(12.2)	1.51 dd(12.1, 4.8)
	1.30 dd(12.1, 12.1)	1.59 m	1.74 m
15′	1.14 m	1.20 m	1.29 m
16′	0.80 d(6.6)	0.82 d(6.4)	0.86 (6.4)

① Peaktype was not given in the original literature.

Table 2-5-12: ^{1}H NMR spectroscopic data of lycodine-type licopodium alkaloids **2-5-30**~**2-5-33**.

H	2-5-30	2-5-31	2-5-32	2-5-33
2	6.42 d(9.7)	6.43 d(9.4)	6.43 d(9.4)	6.24 d(9.5)
3	9.45 d(9.7)	7.64 d(9.4)	7.81 d(9.4)	7.47 d(9.5)
6*α*	3.02 dd(17.8, 5.2)	3.96 dd(17.4, 4.8)	2.94 dd(17.6, 5.3)	2.72 dd(17.6, 4.8)
6*β*	2.46 d(17.8)	2.77 d(17.4)	2.45 d(17.6)	2.65 d(17.6)
7	2.56 br d(3.9)	2.40(ov)	2.59 ddd(5.3, 5.2, 3.8)	2.31 ddd(4.8, 4.2, 3.6)
8	x 5.38 br d(4.0)	5.34 d(4.1)	5.45 d(5.2)	5.45 d(4.2)
10	5.25 d(17.1)	5.17 dd(17.1,1.2)	5.25 dd(17.1, 1.9)	5.06 dd(16.9, 1.3)
	5.26 d(10.2)	5.03 dd(9.9, 1.2)	5.18 dd(10.1, 1.9)	4.93 dd(10.1, 1.3)
11	5.96 ddd(17.1, 10.2, 10.2)	5.93 ddd(17.1, 10.0, 9.9)	5.66 ddd(17.1, 10.1, 10.0)	5.77 ddd(16.9, 10.1, 10.1)
12	2.78 dd(10.2, 3.9)	2.83 dd(10.0, 3.8)	2.39 dd(10.0, 3.8)	2.71 (ov)
14en	2.84 s (2H)	1.59 d(17.2)	2.00 d(17.1)	1.53 d(17.3)
14ex		2.39(ov)	2.15 d(17.1)	2.08 d(17.3)
16	1.60 s	1.52 s	1.54 s	3.60, 3.66 AB(13.5)

Table 2-5-12 (continued)

H	2-5-30	2-5-31	2-5-32	2-5-33
NMe	3.18 s	2.40 s		2.23 s
NMe	3.28 s	2.40 s		
NH		12.85 br s	12.67 br s	3.82 br s
OH				3.82 s

Table 2-5-13: ^{1}H NMR spectroscopic data of lycodine-type licopodium alkaloids **2-5-34**~**2-5-37**.

H	2-5-34	2-5-35	2-5-36	2-5-37
2	6.41d(9.4)	*α* 2.52 td(10.7, 3.3)	6.45 d(9.3)	6.50 d(9.6)
		β 2.41 dd(10.5, 6.5)		
3	7.66 d(9.4)	*α* 2.34 (ov), *β* 2.32 (ov)	7.65 d(9.3)	7.59 d(9.6)
6	*α* 2.95 dd(18.5, 6.9)	*α* 2.49 br d(17.2)	2.93 dd(17.0, 5.1)	2.96 dd(18.6, 5.4)
	β 2.26 d(18.5)	*β* 1.96 d(17.2)	2.69 d(17.0)	2.46 d(18.6)
7	2.02~2.07 m	2.41 d(5.9)	3.76 br t①	3.03 m
8	ex 1.25~1.28 m		5.51 br d(4.8)	5.66 d(5.4)
	en 1.66~1.70 m	en 5.36 br d(5.5)		
9		*α* 2.81 td(13.0, 3.1)		2.87 br t(12.3)
		β 3.09 dt(13.0, 3.2)		3.34 m
10	5.24 d(17.0)	*α* 1.67 br d(14.0)	4.25 d(2H, 6.2)	2.13 br d(9.0)
	5.02 d(10.1)	*β* 2.13 tt(13.5, 4.0)		1.81 m
11	6.00 ddd(17.0, 10.1, 10.1)	*α* 1.52 br d(12.7)	5.49 t(6.2)	3.60 ddd(10.8, 10.4, 4.4)
		β 1.97 dd(13.4, 4.4)		
12	2.83 dd(10.1, 3.3)			1.94 dd(10.4, 3.6)
14	ex 1.62~1.66 m	ex 2.02 d(15.8)	2.42 d(16.2)	2.63 d(16.2)
	en 1.31~1.36 m	en 2.34 d(15.6)	2.58 d(16.2)	2.24 d(16.2)
15	1.19~1.24 m			
16	0.85 d(6.0)	1.66 s	1.62 s	1.65 s
NMe	2.43 s			
NMe	2.43 s			

① No specific coupling constant was given in the literature.

Note: ex denotes *exo* and en *endo*.

Table 2-5-14: ^{1}H NMR spectroscopic data of lycodine-type licopodium alkaloids **2-5-38**~**2-5-40**.

H	2-5-38	2-5-39	2-5-40
1	8.33 dd(4.7, 1.3)	8.59 (0.78H s), 8.61 (0.22H, s)	9.07 s
2	7.11 dd(7.7, 4.7)		
3	7.78 dd(7.7, 1.1)	8.24 (0.78H s), 8.16 (0.22H s)	8.50 s
6	3.24 dd(18.9, 7.2)	3.28 m	3.28 m
	2.76 m	2.82 (0.78H 19.2), 2.83 (0.22H d 19.2)	2.86 d(19.8)

Table 2-5-14 (continued)

H	2-5-38	2-5-39	2-5-40
7	2.76 d(18.5)	2.35 m	2.34 m
8	1.30 dt(12.2, 3.5)	1.87 m	1.88 m
	1.84 m	1.47 ddd(13.2, 12.6, 3.6)	1.47 ddd(12.6, 12.6, 3.6)
9	2.46 dt(13.4, 2.4)	3.28 m	3.21 br d(13.2)
	2.87 br d(13.4)	2.94 ddd(13.2, 12.6, 3.6)	2.83 ddd(13.2, 13.2, 4.2)
10	1.86 m, 1.48 m	1.88 m(2H)	1.84 m(2H)
11	3.37 dt(10.7, 4.5)	1.73 br d(13.2), 1.34 m	1.71 br d(13.8), 1.29 m
12	1.45 dd(10.7, 2.3)	2.09 br d(12.6)	2.05 br d(12.6)
14	1.18 t(11.6)	1.89 m	1.83 m
	1.52 br d(11.6)	1.63 dd(12.0, 12.0)	1.60 dd(12.0, 12.0)
15	1.23 m	1.23 m	1.23 m
16	0.78 d(6.0)	0.87 (2.34H d 6.6), 0.88 (0.66H d 6.6)	0.88 d(6.6)
17		4.50 (0.78H m), 4.51 (0.22H m)	
18		2.38 m (2H)	3.39 m (2H)
19		2.42 m, 2.36 m	2.78 (2H t 6.0)
21			3.68 s

Table 2-5-15: ^{1}H NMR spectroscopic data of lycodine-type licopodium alkaloids **2-5-41**~**2-5-42**.

H	2-5-41	2-5-42	H	2-5-42
2	6.96 d(8)	3.34 m, 3.80 m	17	4.09 m
3	7.71 d(8)	2.70 br s	20	2.02 s
4		1.73 m, 1.97 m	2′	3.19 m, 3.33 m
5		2.57 br s	3′	1.82 m, 1.93 m
6	3.09 dd(19, 7), 2.66 d(19)		4′	1.54 m, 1.82 m
7	2.06 m	1.78 m, 3.01 br t(12.9)	5′	1.55 m, 1.93 m
8	1.77 br d(12)	1.26 m	6′	3.24 m
	1.33 dddd(12, 12, 4, 1)		7′	1.19 br q(13.2)
9	2.77 dm(12)	1.30 m		1.90 m
	2.43 dddd(12, 12, 3, 1)	1.65 m	8′	1.92 m
10	1.46~1.60 m(2H)	2.35 br s	9′	1.65 m, 1.95 m
11	1.17~1.27 m(2H)	2.35 m①, 2.79 m①	10′	3.77 m
12	1.46~1.60 m	0.96 d(4.6)	11′	2.10 m
14	1.43 ddd(12, 3.5, 2)			2.49 br d(11.8)
	1.13 dd(12, 11)		12′	0.98 d(5.9)
15	1.17~1.28 m	1.80 m, 1.96 m		
16	0.78 d(6)	1.75 m, 2.29 m		
Others	2.89 dd(15, 3)			
	2.71 dd(15, 3)			
	4.21 ddq(9, 6, 3)			
	1.27 d(6)			

① Tested in C_5D_5N.

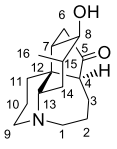
2.5.3 Fawcettimine-type licopodium alkaloids

2.5.3.1 Carbinol-amine fawcettimine-type licopodium alkaloids

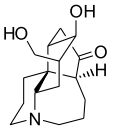
Fawcettidine-type carbinol-amine fawcettimine-type licopodium alkaloids

Table 2-5-16: Cos, MFs, and TSs of fawcettidine-type carbinol-amine fawcettimine-type licopodium alkaloids **2-5-43**~**2-5-55**.

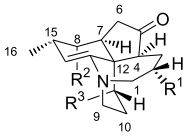
No.	Compounds	MFs	Test solvents	References
2-5-43	lyconesidine A	$C_{16}H_{25}NO_2$	CD_3OD	[96]
2-5-44	lyconesidine B	$C_{16}H_{25}NO_3$	CD_3OD	[96]
2-5-45	11α-hydroxyfawcettidine	$C_{16}H_{23}NO_2$	$CDCl_3$	[107]
2-5-46	2α,11α-dihydroxyfawcettidine	$C_{16}H_{23}NO_3$	$CDCl_3$	[107]
2-5-47	8α,11α-dihydroxyfawcettidine	$C_{16}H_{23}NO_3$	$CDCl_3$	[107]
2-5-48	2β-hydroxylycothunine	$C_{16}H_{23}NO_3$	$CDCl_3$	[107]
2-5-49	8α-hydroxylycothunine	$C_{16}H_{23}NO_3$	$CDCl_3$	[107]
2-5-50	lycothunine	$C_{16}H_{23}NO_2$	$CDCl_3$	[107]
2-5-51	lycoposerramine C	$C_{16}H_{23}NO_2$	$CDCl_3$	[108]
2-5-52	lycoposerramine P	$C_{16}H_{25}NO_3$	CD_3COCD_3	[108]
2-5-53	lycoposerramine Q	$C_{16}H_{25}NO$	$CDCl_3$	[108]
2-5-54	huperzine Q	$C_{16}H_{25}NO_2$	$CDCl_3$	[109]
2-5-55	*N*-oxyhuperzine Q	$C_{16}H_{25}NO_3$	$CDCl_3$	[109]



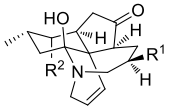
2-5-43



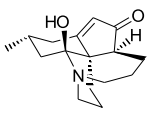
2-5-44



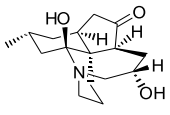
	R^1	R^2	R^3
2-5-45	H	H	OH
2-5-46	OH	H	OH
2-5-47	H	OH	OH



	R^1	R^2
2-5-48	OH	H
2-5-49	H	OH
2-5-50	H	H



2-5-51



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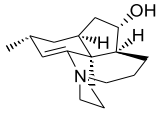

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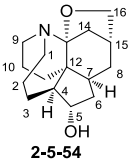
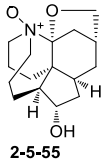


2-5-54



2-5-55

Table 2-5-17: ^{1}H NMR spectroscopic data of fawcettidine-type carbinol-amine fawcettimine-type licopodium alkaloids **2-5-43**~**2-5-45**.

H	2-5-43	2-5-44	2-5-45
1	2.89 br d(15.1) 3.23 m	2.81 br d(15.4) 3.20 m	α 3.15 br d(14.3) β 3.00 ddd(12.9, 12.9, 3.5)

Table 2-5-17 (continued)

H	2-5-43	2-5-44	2-5-45
2	1.81 m, 1.87 m	1.80 m (2H)	*α* 1.70 m, *β* 1.76 m
3	1.68 m, 2.22 m	1.67 dd(12.3, 12.3), 2.20 m	*α* 2.32 m, *β* 1.43 m
4	2.31 m	2.29 dd(12.4, 3.1)	2.04 m
6	2.43 d(16.5)	2.36 d(18.1)	*α* 2.72 dd(16.7, 7.9)
	2.74 dd(16.5, 7.0)	2.75 dd(18.1, 7.2)	*β* 2.06 m
7	1.90 m	1.86 d(10.2)	2.80 m
8	3.09 m	3.23 m	*α* 1.25 m, *β* 1.40 m
9	3.11 m, 3.21 m	3.04 m	*α* 3.35 ddd(14.0, 14.0, 3.8)
		3.14 ddd(13.6, 13.6, 3.6)	*β* 2.83 dd(14.5, 6.0)
10	1.66 m	1.59 m	*α* 1.65 m
	2.01 m	1.94 m	*β* 2.23 dddd(13.9, 13.9, 6.3, 2.6)
11	1.58 ddd(13.3, 13.3, 3.7)	1.57 ddd(13.1, 13.1, 3.2)	3.87 br s
	2.02 m	2.01 d(13.1)	
13	3.12 m	3.07 m	
14	1.52 ddd(13.3, 3.8, 3.8)	1.77 m	5.97 d(5.2)
	2.28 m	2.13 ddd(13.5, 13.5, 4.6)	
15	2.20 m	2.17 m	2.32 m
16	1.05 d(7.1)	3.71 dd(10.9, 8.9)	1.09 d(7.1)
		3.90 dd(10.9, 5.8)	

Table 2-5-18: ^{1}H NMR spectroscopic data of fawcettidine-type carbinol-amine fawcettimine-type licopodium alkaloids **2-5-46**~**2-5-48**.

H	2-5-46	2-5-47	2-5-48
1*α*	3.15 d(15.1)	3.12 ddd(13.8, 2.8, 2.8)	2.83 d(14.0)
1*β*	3.23 dd(15.3, 2.7)	3.03 m	3.27 dd(14.1, 10.5)
2*α*	3.99 d(6.6)	1.77 m	4.22 dddd(11.8, 10.4, 2.7, 2.7)
2*β*		1.77 m	
3*α*	2.34 m	2.17 m	2.26 m
3*β*	1.67 dd(13.4, 13.4)	1.51 m	2.10 m
4	2.53 d(12.3)	2.17 m	2.28 m
6*α*	2.80 m	2.51 m	2.19 m
6*β*	2.07 d(17.0)	2.51 m	2.54 dd(18.1, 12.6)
7	2.85 m	2.70 ddd(7.5, 7.5, 7.5)	2.15 m
8*α*	1.22 ddd(13.3, 13.3, 5.3)	3.59 dd(4.9, 4.9)	1.20 ddd(13.0, 13.0, 5.2)
8*β*	1.42 br d(12.3)		1.61 d(15.6)
9*α*	3.33 ddd(13.4, 13.4, 3.7)	3.45 ddd(13.9, 13.9, 4.0)	3.83 d(19.2)
9*β*	2.85 m	2.80 dd(14.2, 5.2)	3.33 dd(19.3,3.9)
10	*α* 1.50 ddd(13.7, 3.5, 3.5)	*α* 1.63 ddd(14.5, 3.2, 3.2)	5.84 ddd(9.8, 3.8, 1.6)
	β 2.80 m	*β* 2.17 m	
11	3.91 s	3.79 br s	5.53 d(9.8)
14	5.95 d(5.5)	5.65 d(3.2)	*α* 1.71 dd(12.9, 12.9), *β* 1.32 d(13.7)
15	2.31 m	2.51 m	1.99 m
16	1.10 d(7.1)	1.14 d(7.1)	0.92 d(6.5)

Table 2-5-19: ^{1}H NMR spectroscopic data of fawcettidine-type carbinol-amine fawcettimine-type licopodium alkaloids **2-5-49**~**2-5-51**.

H	2-5-49	2-5-50	2-5-51
1	*α* 2.82 dd(14.5, 4.1)	*α* 2.80 d(14.5)	2.73 br d(14.1)
	β 3.27 dd(12.7, 12.7)	*β* 3.33 dd(13.3, 13.3)	3.43 ddd(14.1, 14.1, 4.6)
2	*α* 1.35 m, *β* 2.01 m	*α* 1.36 m, *β* 1.99 m	1.75 m (2H)
3	*α* 2.19 m, *β* 1.97 m	*α* 2.18 m, *β* 1.97 m	2.14 m, 2.00 m
4	2.27 m	2.23 m	2.14 m
6	*α* 2.30 m, *β* 2.34 m	*α* 2.18 m, *β* 2.54 dd(17.8, 12.9)	5.82 d(1.5)
7	2.32 m	2.11 m	
8	3.75 br s	*α* 1.20 ddd(13.0, 13.0, 4.8)	2.05 ddd(13.2, 13.2, 1.7)
		β 1.60 d(13.5)	2.57 ddd(13.2, 4.3, 1.5)
9	*α* 3.91 d(19.5)	*α* 3.82 ddd(19.4, 2.1, 2.1)	3.34 ddd(14.6, 14.6, 4.1)
	β 3.22 dd(19.3, 3.7)	*β* 3.23 br d(19.2)	2.98 dd(14.6, 5.5)
10	5.80 dd(10.7, 3.0)	5.80 ddd(9.9, 3.9, 1.8)	1.75 m, 1.53 br d(13.7)
11	5.75 d(9.8)	5.53 ddd(9.9, 2.0, 2.0))	2.23 ddd(13.6, 13.6, 4.9)
			1.26 ddd(13.6, 2.3, 2.3)
14	*α* 2.23 m	*α* 1.70 dd(13.6, 12.2)	2.35 dd(14.0, 11.9)
	β 1.09 m	*β* 1.36 m	1.31 dd(14.0,2.8)
15	2.25 m	1.99 m	2.00 m
16	1.01 d(6.3)	0.92 d(6.6)	1.01 d(6.1)

Table 2-5-20: ^{1}H NMR spectroscopic data of fawcettidine-type carbinol-amine fawcettimine-type licopodium alkaloids **2-5-52**~**2-5-55**.

H	2-5-52	2-5-53	2-5-54	2-5-55
1	2.27 m	2.97 m	*α* 3.02 dt(15.2, 1.9)	*α* 3.62 br d(14.4)
	3.57 dd(13.9, 5.6)	3.09 br d(12.8)	*β* 3.12 ddd(15.2, 12.8, 2.8)	*β* 3.33 br t(14.4)
2	4.37 m	1.75 m, 1.69 m	*α* 1.84 (ov), *β* 1.63 (ov)	*α* 2.08 (ov), *β* 1.87 (ov)
3	2.54 m, 1.92 m	1.92 m, 1.35 q(12.7)	*α* 2.19 (ov), *β* 1.69 (ov)	*α* 2.29 (ov), *β* 1.58 (ov)
4	1.92 m	1.85 m	1.64 (ov)	1.56 (ov)
5		3.96 q(4.9)	3.95 ddd(10.0, 6.7, 5.5)	4.00 ddd(9.8, 6.6, 5.6)
6	2.04 m,	1.75 m	*α* 1.81 (ov), *β* 1.69 (ov)	*α* 1.77 dd(11.1, 6.6)
	2.37 dd(17.9, 11.7)			*β* 1.67 (ov)
7	1.92 m	2.13 m	1.82 (ov)	2.02 (ov)
8	1.38 m, 1.53 m	1.26 m, 1.51 m	en1.81 (ov), ex1.24 m	en1.88(ov), ex1.31 br t(13.6)
9	3.18 ddd(14.3, 14.3, 3.2)	2.97 m	*α* 2.73 dd(14.0, 5.4)	*α* 3.40 dd(13.6, 4.0)
	2.70 dd(14.3, 5.2)		*β* 3.45 td(14.0, 4.2)	*β* 3.76 td(13.6, 3.8)
10	1.38 m, 2.04 m	1.51 m, 1.92 m	*α* 2.08 (ov)	–
			β 1.44 br d(13.5)	*β* 1.65 (ov)

Table 2-5-20 (continued)

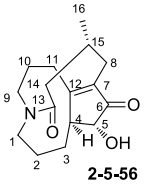
H	2-5-52	2-5-53	2-5-54	2-5-55
11	1.53 m, 2.27 m	1.85 m 1.57 ddd(12.5, 12.5, 4.3)	α 1.48 br d(12.8) β 2.19 td(12.8, 4.2)	α 1.43 br d(12.8) β 2.19 td(12.8, 3.1)
14	1.30 m,2.04 m	5.57 d(3.4)	en 1.87 dd(11.2, 5.0) ex 2.06 d(11.2)	en 2.87 ddd(9.8, 3.2, 1.3) ex 1.68 d(9.8)
15	2.10 m	2.23 m	2.49 q(4.1)	2.54 br s
16	0.89 d(6.4)	0.98 d(7.0)	en 4.00 ddd(7.5, 4.9, 1.5) ex 3.74 d(7.5)	en 4.14 ddd(7.2, 3.6, 2.4) ex 3.85 d(7.2)

Phlegmariurine B-type carbinol-amine fawcettimine-type licopodium alkaloids

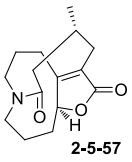
Table 2-5-21: Cos, MFs, and TSs of phlegmariurine B-type carbinol-amine fawcettimine-type licopodium alkaloids **2-5-56**~**2-5-66**.

No.	Compounds	MFs	Test solvents	References
2-5-56	huperzine T	$C_{16}H_{23}NO_3$	$CDCl_3$	[101]
2-5-57	huperzine R	$C_{15}H_{21}NO_3$	$CDCl_3$	[110]
2-5-58	2α-hydroxyphlegmariurine B	$C_{16}H_{23}NO_3$	$CDCl_3$	[111]
2-5-59	2-oxo-phlegmariurine B	$C_{16}H_{21}NO_3$	$CDCl_3$	[111]
2-5-60	11-oxo-phlegmariurine B	$C_{16}H_{21}NO_3$	$CDCl_3$	[111]
2-5-61	phlegmariurine B	$C_{16}H_{23}NO_2$	$CDCl_3$	[112]
2-5-62	11α-hydroxyphlegmariurine B	$C_{16}H_{23}NO_3$	CD_3OD	[112]
2-5-63	7α-hydroxyphlegmariurine B	$C_{16}H_{23}NO_3$	$CDCl_3$	[112]
2-5-64	7α,11α-hydroxyphlegmariurine B	$C_{16}H_{23}NO_4$ ①	CD_3OD	[112]
2-5-65	huperzine P	$C_{16}H_{21}NO_3$	$CDCl_3$	[113]
2-5-66	lycoposerramine E	$C_{16}H_{23}NO_3$	$CDCl_3$	[108]

① The molecular formula given in the literature is wrong.



2-5-56



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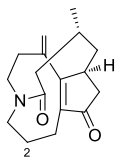
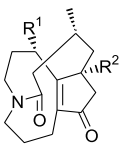
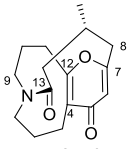


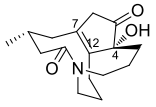
2-5-58 2α-OH
2-5-59 2-oxo
2-5-60 11-oxo



	R^1	R^2
2-5-61	H	H
2-5-62	OH	H
2-5-63	H	OH
2-5-64	OH	OH



2-5-65



2-5-66

Table 2-5-22: ^{1}H NMR spectroscopic data of phlegmariurine B-type carbinol-amine fawcettimine-type licopodium alkaloids **2-5-56**~**2-5-59**.

H	2-5-56	2-5-57	2-5-58	2-5-59
1*α*	2.45 td(13.8, 2.0)	2.40 td(14.0, 1.3)	2.66 dd(13.3, 9.9)	3.18 d(15.7)
1*β*	3.91 dt(13.8, 3.2)	3.94 dt(14.0, 3.1)	4.07 dd(13.3, 5.6)	5.14 d(15.7)
2*α*	2.10 (ov)	2.21 qd(13.8, 3.0)		
2*β*	1.51 (ov)	1.34 m	4.60 tt(9.9, 5.3)	
3*α*	2.40 (ov)	1.91 t(14.2)	2.68 dd(13.2, 9.8)	3.29 d(17.4)
3*β*	2.09 (ov)	2.53 m	2.73 dd(13.2, 5.0)	3.69 d(17.4)
4	2.66 br s	4.79 br d(3.8)		
5	3.84 d(1.5)			
6*α*			2.37 dd(19.3, 7.1)	2.45 (ov)
6*β*			2.20 dd(19.1, 1.9)	2.45 (ov)
7			2.76 br s	2.88 br s
8	en 2.44 dd(12.7, 2.0) ex 1.94 t(12.7)	*α* 2.01 t(12.1) *β* 2.41 dd(12.1, 3.2)	en 1.77 dd(14.9, 4.6) ex 1.90 dd(15.0, 11.2)	1.93 (2H, ov)
9*α*	3.11 br d(13.9)	3.10 br d(14.2)	3.27 dt(15.1, 3.0)	3.46 dt(15.5, 4.5)
9*β*	4.03 td(13.9, 3.3)	4.02 td(14.2, 3.2)	3.89 ddd(15.5, 12.5, 3.0)	4.07 ddd(15.2, 11.0, 4.0)
10*α*	1.96 br d(13.1)	2.15 qd(13.9, 3.0)	2.82 qt(13.6, 4.1)	2.44 (ov)
10*β*	2.40 qt(12.8, 4.4)	1.89 br d(13.9)	1.91 (ov)	2.11 dt(15.7, 4.6)
11*α*	2.41 br d(13.7)	2.41 dd(14.0, 1.3)	2.64 td(14.1, 3.7)	2.78 (ov)
11*β*	2.99 td(13.7, 4.4)	2.91 td(14.0, 4.1)	2.74 ddd(14.0, 5.0, 3.4)	2.78 (ov)
14	1.95 br d(13.6) 2.53 dd(13.6, 11.2)	en 1.99 dd(12.9, 3.0) ex 2.56 dd(12.9, 11.2)	en 1.84 d(14.7) ex 2.40 dd(14.7, 8.7)	en 1.94 d(13.8) ex 2.46 (ov)
15	2.25 m	2.48 m	en 2.11 m	en 2.46 m
16	1.09 d(6.8)	1.10 d(6.2)	1.05 d(7.0)	1.09 d(6.4)

Table 2-5-23: ^{1}H NMR spectroscopic data of phlegmariurine B-type carbinol-amine fawcettimine-type licopodium alkaloids **2-5-60**~**2-5-63**.

H	2-5-60	2-5-61	2-5-62	2-5-63
1*α*	2.90 ddd(14.2, 12.5, 4.6)	2.81 ddd(14.1, 11.4, 4.7)	3.01 td(13.7, 4.6)	2.83 dd(14.1, 11.6)
1*β*	4.19 dd(14.2, 4.7)	4.05 dt(14.1, 2.5)	3.89 dd(13.7, 4.9)	4.01 ddd(14.1, 2.6, 1.7)
2*α*	1.59 dtt(13.7, 6.0, 1.5)	1.41 dtt(14.0, 4.7, 2.5)	1.37 dtt(14.0, 6.0, 2.3)	1.42 dtt(14.0, 6.8, 2.4)
2*β*	2.49 (ov)	2.44 (ov)	2.30 qdd(14.0, 4.9, 1.9)	2.40 qdd(14.0, 5.1, 1.6)
3*α*	2.82 td(14.2, 1.5)	2.42 (ov)	2.65 td(13.8, 2.1)	2.42 td(13.8, 2.4)
3*β*	2.49 (ov)	2.60 (ov)	2.46 dd(13.8, 5.9)	2.59 (ov)
6*α*	2.49 dd(19.8, 6.9)	2.35 dd(19.0, 7.1)	2.41 dd(19.4, 6.7)	2.63 d(18.8)
6*β*	2.28 dd(19.8, 2.5)	2.20 dd(19.0, 2.2)	2.15 dd(19.4, 1.9)	2.30 d(18.6)
7	3.47 br s	2.75 br s	3.14 dt(4.4, 2.2)	

Table 2-5-23 (continued)

H	2-5-60	2-5-61	2-5-62	2-5-63
8en	1.67 ddd(14.9, 4.2, 1.5)	1.76 dd(15.0, 4.6)	1.77 dd(13.0, 5.0)	1.90 d(12.2)
8ex	1.79 ddd(15.0, 11.2, 3.8)	1.90 dd(15.0, 7.7)	1.93 dd(13.0, 3.0)	1.99 dd(12.2, 8.4)
9*α*	3.31 ddd(15.4, 6.6, 1.9)	3.16 dt(15.2, 3.7)	3.19 ddd(15.5, 3.8, 3.4)	3.19 dt(15.3, 3.6)
9*β*	4.05 ddd(15.3, 13.5, 3.3)	3.87 ddd(15.2, 11.5, 3.7)	4.02 ddd(15.5, 13.5, 2.8)	3.88 ddd(15.3, 11.7, 3.1)
10*α*	3.59 td(13.5, 6.6)	2.73 (ov)	2.82 dddd(14.2, 13.5, 12.2, 4.1)	2.71 (ov)
10*β*	2.82 ddd(13.8, 3.3, 2.0)	1.92 (ov)	2.09 ddd(14.2, 4.9, 2.8)①	1.98 (ov)
11*α*		2.64 (ov)		2.71 ddd(16.0, 12.9, 3.8)
11*β*		2.73 (ov)	4.58 dd(12.2, 4.9)	2.81 dt(16.0, 2.3)
14en	1.86 d(15.2)	1.86 d(14.8)	1.75 d(14.8)	1.85 d(14.5)
14ex	1.97 dd(15.2, 7.8)	2.43 dd(14.8, 8.5)	2.49 dd(14.8, 8.0)	2.50 dd(14.5, 7.6)
15n	2.10 m	2.12 m	1.93 m	2.00 m
16	1.03 d(7.0)	1.07 d(7.0)	1.04 d(6.5)	1.10 d(6.4)

① The peaktype of dddd was given in the original literature, but only three coupling constants were given.

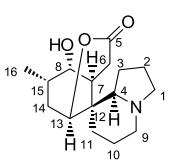
Table 2-5-24: ^{1}H NMR spectroscopic data of phlegmariurine B-type carbinol-amine fawcettimine-type licopodium alkaloids **2-5-64**~**2-5-66**.

H	2-5-64	2-5-65	2-5-66
1	*α* 3.02 td(13.9, 4.5)	*α* 2.64 ddd(14.3, 8.2, 3.3)	4.00 ddd(13.8, 3.3, 3.3)
	β 3.86 dd(13.9, 4.0)	*β* 4.09 ddd(14.3, 8.3, 3.5)	2.54 m
2	*α* 1.39 dtt(14.0, 6.1, 2.3)	*α* 1.88 dddd(15.1, 8.4, 5.1, 3.3)	2.02 m
	β 2.37 qdd(14.0, 5.2, 2.4)	*β* 2.01 (ov)	
3	*α* 2.68 td(13.7, 2.3)	*α* 3.22 ddd(14.1, 8.5, 5.6)	2.14 m, 2.24 m
	β 2.48 ddd(13.7, 6.1, 2.3)	*β* 2.38 dt(14.6, 5.6)	
6	*α* 2.59 d(18.8), *β* 2.33 d(18.8)	6.02 s	3.22 dd(22.3, 2.6), 2.65 m
8	en 1.90 d(14.4)	en 2.74 dd(13.0, 5.3)	2.02 m, 2.54 m
	ex 2.04 dd(14.4, 10.6)	ex 1.99 dd(12.8, 10.1)	
9	*α* 3.21 dt(15.4, 3.7)	*α* 3.75 ddd(15.3, 5.6, 4.8)	4.12 ddd(13.9, 13.9, 3.0)
	β 4.03 ddd(15.4, 13.2, 2.8)	*β* 3.17 ddd(15.4, 10.0, 5.4)	3.18 br d(15.6)
10	*α* 2.76 dddd(14.6, 13.2, 11.8, 3.7)	*α* 2.01 (ov)	2.14 m
	β 2.14 dddd(14.6, 5.2, 3.7, 2.8)	*β* 2.46 (ov)	1.70 ddd(15.3, 3.0, 3.0)
11	*β* 4.71 dd(11.8, 5.2)	*α* 2.61 dt(15.0, 5.7)	2.54 m
		β 2.99 ddd(15.0, 10.0, 5.0)	2.77 ddd(14.0, 14.0, 4.2)
14	en 1.77 d(14.5)	en 1.97 dd(14.8, 1.7)	2.02 m
	ex 2.59 dd(14.5, 8.7)	ex 2.50 dd(15.1, 10.1)	2.70 m
15	en 1.81 m	en 2.82 m	2.39 m
16	1.09 d(7.1)	1.14 d(6.8)	1.11 d(6.6)

2.5.3.2 Keto-amine fawcettimine-type licopodium alkaloids

Table 2-5-25: Cos, MFs, and TSs of keto-amine fawcettimine-type licopodium alkaloids **2-5-67**~**2-5-76**.

No.	Compounds	MFs	Test solvents	References
2-5-67	serratezomine A	$C_{16}H_{25}NO_3$	CD_3OD	[90]
2-5-68	serratezomine B	$C_{16}H_{25}NO_4$	CD_3OD	[90]
2-5-69	huperine H	$C_{16}H_{21}NO_2$	$CDCl_3$	[114]
2-5-70	lycoposerramine D	$C_{17}H_{25}NO_3$	$CDCl_3$	[108]
2-5-71	lycoposerramine U	$C_{17}H_{25}NO_3$	$CDCl_3$	[108]
2-5-72	lycoposerramine B	$C_{17}H_{28}N_2O_2$	$CDCl_3$	[115]
2-5-73	sieboldine A	$C_{16}H_{23}NO_4$	CD_3OD	[116]
2-5-74	lycoposerramine A	$C_{18}H_{29}N_3O_2$	$CDCl_3$	[117, 128]
2-5-75	lycoposerramine S	$C_{17}H_{30}N_2$	$CDCl_3$	[108]
2-5-76	palhinine A	$C_{17}H_{25}NO_3$	$CDCl_3$	[118]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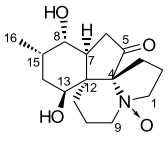
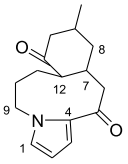

2-5-68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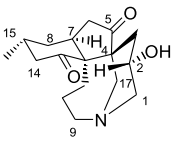
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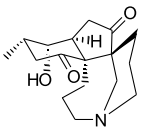
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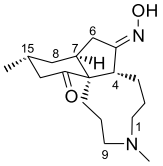
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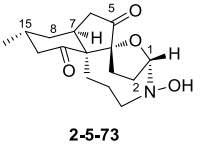
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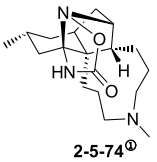
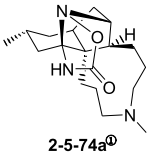
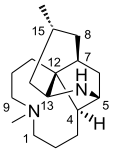
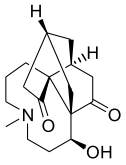

2-5-74①


2-5-74a①


2-5-75


2-5-76

① The structure **2-5-74** was amended as the structure **2-5-74a** later on in the literature of Takayama H, Katakawa K, Kitajima, M, et al. Org Lett, 2002, 4: 1243.

Table 2-5-26: ^{1}H NMR spectroscopic data of keto-amine fawcettimine-type licopodium alkaloids **2-5-67**~**2-5-70**.

H	2-5-67	2-5-68	2-5-69	2-5-70
1	3.35 m 3.54 ddd(9.2, 9.2, 9.2)	3.90 br t(9.7) 4.04 ddd(10.8, 10.8, 10.8)	6.77 dd(4.4, 2.1)	2.99 dd(13.5, 5.1) 2.71 dd(13.5, 10.4)

Table 2-5-26 (continued)

H	2-5-67	2-5-68	2-5-69	2-5-70
2	2.20 m (2H)	2.12 m, 2.38 m	6.18 dd(4.1, 4.1)	4.12 tt(10.4, 5.1)
3	2.25 m	2.27 ddd(15.0, 11.5, 5.0)	6.99 dd(4.1, 2.1)	2.32 m
	2.15 m	2.67 ddd(15.0, 11.5, 4.6)		1.75 m
4	3.81 dd(10.9, 6.0)			
6	2.46 d(20.0)	2.42 d(10.9)	3.02 dd(12.5, 9.5)	2.30 m
	3.14 dd(20.0, 8.0)		2.66 br d(12.5)	2.25 m
7	2.61 m	3.23 br t(10.9)	2.36 m	2.63 m
8	3.77 t(3.4)	3.79 br s	1.88 dd(12.11, 4.2)	1.65 m
			1.82 m	1.83 br d(11.4)
9	2.98 dt(13.0, 3.5)	3.56 m (2H)	4.46 br t(13.4)	3.06 m
	3.26 m		4.21 dm(13.4)	2.83 ddd(13.9, 6.7, 3.4)
10	1.84 m, 2.03 m	1.42 m, 2.57 dt(14.0, 3.3)	1.79 m, 1.56 m	1.75 m, 1.65 m
11	1.40 dt(13.6, 3.3)	1.82 dd(12.7, 1.5)	2.76 dd(16.0, 3.4)	2.20 m
	2.83 br d(13.6)	2.46 m	1.04 br d(16.0)	2.05 dd(14.7, 9.5)
12			2.05 br d(12.3)	
13	4.32 br d(2.7)	3.59 br s		
14	1.83 m (2H)	1.39 m	2.48 dd(10.6, 5.9)	2.30 m (2H)
		1.84 dd(12.9, 1.4)	2.22 d(10.6)	
15	1.78 m	2.23 m	2.42 m	2.17 m
16	1.01 d(6.4)	1.00 d(7.0)	0.96 d(7.2)	1.03 d(6.6)
17				3.09 d(14.2)
				2.57 d(14.2)

Table 2-5-27: ^{1}H NMR spectroscopic data of keto-amine fawcettimine-type licopodium alkaloids **2-5-71**~**2-5-73**.

H	2-5-71	2-5-72	2-5-73
1	2.88 m, 2.95 ddd(13.7, 13.7, 3.7)	2.03 m, 2.66 dd(13.4, 13.4)	4.89 m
2	1.80 m, 1.34 m	1.46 m, 1.69 m	1.98 m, 2.12 m
3	1.98 m (2H)	1.28 br d(14.4), 2.00 m	2.08 m, 2.40 m
4		3.18 d-like(2.8)	
6	2.63 m	2.20 m	1.93 dd(19.6, 10.9)
	2.03 dd(18.2, 8.2)	2.54 ddd(19.0, 9.5, 1.1)	2.45 dd(19.6, 9.2)
7	2.70 ddd(10.5, 8.2, 2.4)	2.41 m	3.25 m
8	3.86 t(2.4)	1.73 m (2H)	1.76 m, 1.77 m
9	2.88 m, 3.03 ddd(13.3, 9.5, 3.8)	2.27 m, 2.31 m	2.91 ddd(14.8, 8.0, 3.7), 3.19 m
10	2.20 m, 1.80 m	1.20 m, 1.36 m	1.63 m, 2.57 m
11	2.20 m, 2.43 dd(13.9, 9.3)	2.00 m, 2.20 m	1.77 m, 2.46 m
14	2.20 m, 2.53 dd(18.0, 13.1)	2.20 m, 2.27 m	2.03 m, 2.54 dd(12.7, 12.7)
15	2.20 m	2.10 m	2.06 m
16	1.06 d(6.4)	1.03 d(6.4)	1.06 d(6.2)
17	2.66 m, 3.24 br d(14.3)		
NMe		2.27 s	

Table 2-5-28: ^{1}H NMR spectroscopic data of keto-amine fawcettimine-type licopodium alkaloids **2-5-74**~**2-5-76**.

H	2-5-74	2-5-75	2-5-76
1	1.99 dd(13.9, 3.5) 2.66 ddd(13.0, 4.1)①	2.68 ddd(13.4, 13.4, 4.1) 2.01 ddd(13.4, 4.8, 1.9)	2.64 m 2.43 m
2	1.12 br t(13.3, 13.3), 1.66 m	1.20 m, 1.69 m	2.04 m, 1.48 m
3	1.84 m, 2.40 m	1.34 m, 1.75 m	4.43 d(9.6)
4	1.61 br s	1.57 m	
5	3.66 d(4.3)	3.00 br s	
6	1.84 m, 1.28 dd(13.1, 4.6)	1.75 m, 1.34 m	2.65 m, 2.23 d(19.6)
7	2.06 m	1.94 m	2.14 dd(9.0, 9.0)
8	1.02 ddd(13.1, 13.1, 3.4), 1.42 m	1.43 m, 1.05 ddd(13.0, 13.0, 3.2)	1.94 m, 1.41 ddd(13.6, 3.8, 2.7)
9	2.28 m, 2.40 m	2.21 m, 2.36 m	2.42 m, 2.07 m
10	1.42 m, 1.56 m	1.48 m, 1.34 m	1.54 m (2H)
11	1.56 m, 1.84 m	1.43 m, 1.49 m	α 1.22 ddd(14.8, 3.7, 3.7), β 2.31 m
13		2.97 br s	
14	1.20 dd(12.7, 12.7), 1.84 m	1.69 m, 1.00 ddd(12.2, 12.2, 2.7)	2.49 m, 2.28 m
15	1.93 m	1.87 m	2.36 m
16	0.91 d(6.4)	0.88 d(6.4)	2.44 m, 1.64 ddd(15.4, 2.7, 2.7)
NMe	2.25 s	2.24 s	1.93 s
NH	5.08 br s		
OH			4.59 br s

① Typographic error exists in the literature.

2.5.4 Other licopodium alkaloids

Table 2-5-29: Cos, MFs, and TSs of other licopodium alkaloids **2-5-77**~**2-5-102**.

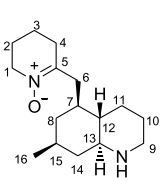
No.	Compounds	MFs	Test solvents	References
2-5-77	carinatumin C	$C_{16}H_{28}N_2O$	CD_3OD	[102]
2-5-78	huperzine J	$C_{17}H_{30}N_2O$	$CDCl_3$	[119]
2-5-79	huperzine L	$C_{17}H_{30}N_2O_2$	$CDCl_3$	[119]
2-5-80	huperzine K	$C_{16}H_{28}N_2O$	$CDCl_3$	[119]
2-5-81	cermizine A	$C_{19}H_{34}N_2O_2$	CD_3OD	[120]
2-5-82	cermizine B	$C_{17}H_{32}N_2$	CD_3OD	[120]
2-5-83	cermizine D	$C_{16}H_{30}N_2$	CD_3OD	[120]
2-5-84	cernuine *N*-oxide	$C_{16}H_{26}N_2O_2$	CD_3OD	[120]
2-5-85	lycocernuine *N*-oxide	$C_{16}H_{26}N_2O_3$	CD_3OD	[120]
2-5-86	cernuine	$C_{16}H_{26}N_2O$	CD_3OD	[120]
2-5-87	lycocernuine	$C_{16}H_{26}N_2O_2$	CD_3OD	[120]

Table 2-5-29 (continued)

No.	Compounds	MFs	Test solvents	References
2-5-88	serratezomine D	$C_{29}H_{49}N_3O$	$CDCl_3$	[121]
2-5-89①	serratezomine E①	$C_{18}H_{32}N_2O$	$CDCl_3$	[121]
2-5-90②	lycoperine A②	$C_{31}H_{49}N_3O_2$	–	[122]
2-5-90a②	tetrahydrodeoxylycoperine A②	$C_{31}H_{53}N_3$	C_6D_6	[122]
2-5-91	cermizine C	$C_{11}H_{21}N$	CD_3OD	[120]
2-5-92	senepodine G	$C_{11}H_{20}N$	CD_3OD	[120]
2-5-93	senepodine H	$C_{14}H_{26}NO$	CD_3OD	[120]
2-5-94	lycojapodine A	$C_{16}H_{23}NO_3$	$CDCl_3$	[123]
2-5-95	lyconadin B	$C_{16}H_{22}N_2O$	CD_3OD	[124]
2-5-96	lyconadin A	$C_{16}H_{20}N_2O$	CD_3OD	[125]
2-5-97	lycopladine A	$C_{16}H_{21}NO_2$	CD_3OD	[126]
2-5-98	lycopladine B	$C_{17}H_{23}NO_3$	CD_3OD	[124]
2-5-99	lycopladine D	$C_{16}H_{23}NO_3$	CD_3OD	[124]
2-5-100	senepodine A	$C_{23}H_{40}N_2$	CD_3OD	[127]
2-5-101	senepodine C	$C_{22}H_{38}N_2$	CD_3OD	[127]
2-5-102	senepodine B	$C_{23}H_{39}N_2$	CD_3OD	[127]

① Due to the rotation of N-acetyl group, compound **2-5-89** existed as a mixture of two rotamers. Two sets of signals were observed in the ^{1}H NMR spectrum.

② Due to the rotation of N-acetyl group, the ^{1}H NMR spectrum of **2-5-90** showed broad signals. Treatment of **2-5-90** with $LiAlH_4$ afforded **2-5-90a**, which provided sharp signals on the ^{1}H NMR spectrum.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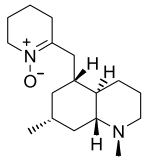
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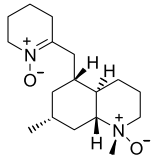
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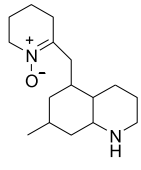
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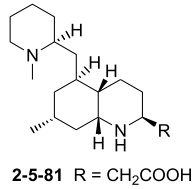
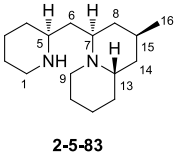
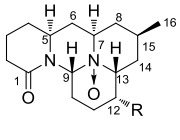
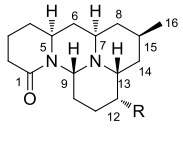
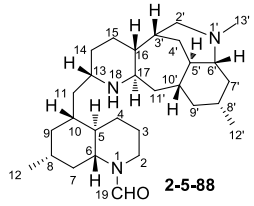
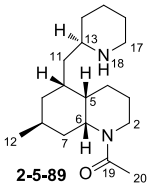

2-5-81 R = CH_2COOH
2-5-82 R = H

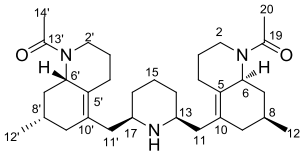

2-5-83


2-5-84 R = H
2-5-85 R = OH


2-5-86 R = H
2-5-87 R = OH


2-5-88


2-5-89


2-5-90

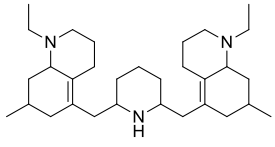
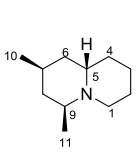
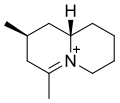
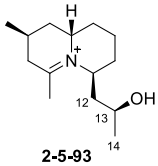

2-5-90a

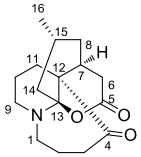
Table 2-5-30: ^{1}H NMR spectroscopic data of other licopodium alkaloids **2-5-77**~**2-5-80**.

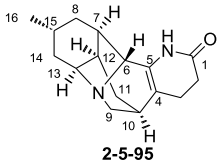
H	2-5-77	2-5-78	2-5-79	2-5-80
1	3.75 br t(5.7) (2H)	3.75 t(5.5) (2H)	3.76 t(6.1) (2H)	3.78 t(5.5) (2H)
2	1.95 m (2H)	1.88 m (2H)	1.90 m (2H)	1.90 m (2H)
3	1.75 m (2H)	1.70 m (2H)	1.74 m (2H)	1.71 m (2H)
4	2.53 m (2H)	2.36 t(6.2) (2H)	2.40 t(5.5) (2H)	2.36 t(5.8) (2H)
6	2.26 dd(13.1, 10.6)	2.84 dd(10.4, 4.1)	3.03 br d(13.2)	2.74 dd(13.0, 3.4)
	2.80 dd(13.1, 3.7)	2.08 d(10.4)	1.82 dd(13.2, 3.2)	2.29 d(13.0)
7	1.81 m	1.72 m	2.08 m	1.90 m
8	0.86 ddd(12.5, 12.5, 12.5), 1.55 m	1.44 m, 0.75 m	1.43 m, 0.80 m	1.84 m, 1.41 m
9	2.77 ddd(12.7, 11.5, 2.2)	2.81 m, 2.04 m	3.34 d(12.0)	2.89 br d(11.3)
	3.16 br d(11.5)		3.14 dd(12.0, 3.0)	2.09 m
10	1.61 m, 1.86 m	1.62 m	2.36 m	1.66 m
11	1.13 dddd(12.5, 12.5, 11.4, 3.6)	1.98 m, 1.05 m	2.14 m, 1.12 m	2.00 br d(14.5)
	2.07 br d(11.4)			1.03 m
12	1.05 m	0.94 m	1.78 m	1.03 m
13	2.53 m	1.57 m	2.68 ddd(11.0, 10.6, 3.4)	1.88 br d(13.0)
14	1.01 ddd(12.1, 12.1, 12.1)	2.01 dd(12.2, 4.1)	1.94 m, 1.59 m	1.32 m, 1.30 m
	1.81 m	0.78 m		
15	1.55 m	1.40 m	1.46 m	2.11 m
16	0.94 d(6.0)	0.86 d(6.4)	0.93 d(6.3)	0.99 d(7.3)
17		2.22 s	3.10 s	

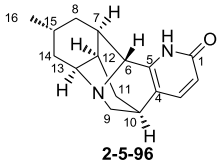

2-5-91

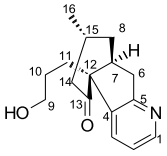

2-5-92

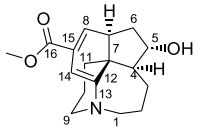

2-5-93

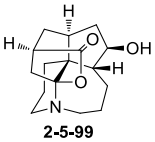

2-5-94

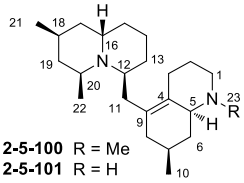

2-5-95


2-5-96


2-5-97


2-5-98


2-5-99


2-5-100 R = Me
2-5-101 R = H

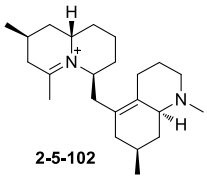

2-5-102

Table 2-5-31: ^{1}H NMR spectroscopic data of other licopodium alkaloids **2-5-81**~**2-5-83**.

H	2-5-81	2-5-82	2-5-83
1	2.29 ddd(11.7, 11.7, 3.9)	2.17 ddd(11.1, 11.1, 3.6)	3.05 ddd(12.2, 11.8, 2.3)
	2.91 ddd(11.7, 4.0, 4.0)	2.84 br d(11.1)	3.42 br d(12.2)
2	1.61 m (2H)	1.41 m, 1.60 m	1.69 m, 1.93 m
3	1.34 m, 1.74 m	1.32 m, 1.73 m	1.70 m, 1.91 m
4	1.27 m, 1.80 m	1.24 m, 1.78 m	1.57 m, 2.01 m
5	2.08 ddd(9.2, 9.2, 3.7)	1.92 m	3.30 m
6	1.24 m	1.19 m, 1.97 m	1.66 m, 2.32 m
	1.99 ddd(13.6, 9.6, 3.7)		
7	1.69 m	1.56 m	3.96 br t(10.7)
8	1.24 m	1.18 m	1.17 q(12.8)
	1.40 br d(14.1)	1.32 m	2.22 br d(13.4)
9	3.44 ddd(8.6, 8.3, 4.3)	2.67 br d(12.6)	3.14 ddd(12.8, 12.4, 3.2)
		2.78 ddd(12.6, 12.6, 2.9)	3.71 br d(12.8)
10	1.51 m, 1.88 m	1.52 m, 1.66 m	1.73 m, 1.82 m
11	1.51 m, 1.89 m	1.38 m, 1.76 m	1.65 m, 1.93 m
12	1.77 m	1.61 m	1.69 m, 2.17 br q(13.0)
13	3.62 ddd(12.2, 4.8, 4.8)	3.09 ddd(12.1, 4.5, 4.5)	3.68 br d(13.0)
14	1.65 m,1.69 m	1.41 m, 1.61 m	1.60 m, 1.80 m
15	1.72 m	1.63 m	1.97 m
16	0.98 d(6.3)	0.94 d(6.2)	0.98 d(6.3)
17	2.37 dd(16.2, 8.6)		
	2.42 dd(16.2, 4.3)		
NMe	2.33 s	2.26 s	

Table 2-5-32: ^{1}H NMR spectroscopic data of other licopodium alkaloids **2-5-84**~**2-5-87**.

H	2-5-84	2-5-85	2-5-86	2-5-87
2	2.38 m, 2.45 m	2.38 m, 2.46 m	2.35 m, 2.36 m	2.35 m, 2.36 m
3	1.65 m, 1.90 m	1.69 m, 1.90 m	1.65 m, 1.82 m	1.65 m, 1.80 m
4	1.65 m, 2.03 m	1.62 m, 2.04 m	1.48 dddd(11.2, 11.2, 11.2, 3.0)	1.50 m, 2.04 m
			2.02 m	
5	3.78 m	3.84 m	3.64 m	3.67 m
6	1.52 ddd(13.7, 3.1, 3.1)	1.50 ddd(13.7, 3.1, 3.1)	1.20 ddd(12.7, 12.2, 11.8)	1.16 m, 1.72 m
	2.02 m	1.95 m	1.73 ddd(12.7, 3.0, 2.9)	
7	4.03 dddd	4.36 dddd	3.24 dddd(11.8, 11.7, 2.9, 2.8)	3.76 m
	(11.8, 11.8, 3.1, 3.0)	(11.8, 11.8, 3.1, 3.1)		
8	1.46 m, 1.65 m	1.43 br d(13.7), 1.69 m	0.81 ddd(12.1, 12.1, 11.7)	0.84 m, 1.70 m
			1.66 m	
9	5.64 dd(13.1, 3.6)	5.67 dd(13.5, 3.2)	5.44 dd(12.5, 2.6)	5.47 dd(12.0, 2.3)

Table 2-5-32 (continued)

H	2-5-84	2-5-85	2-5-86	2-5-87
10	1.72 m, 2.42 m	2.74 dddd (13.9, 13.9, 13.5, 3.5) 1.60 m	1.19 m 2.12 dddd (12.8, 12.8, 12.5, 4.2)	1.21 m, 2.42 m
11	1.77 m, 1.95 m	1.90 m, 1.99 m	1.66 m, 1.94 m	1.90 m, 2.39 m
12	1.74 m 2.22 dddd (13.5, 13.5, 13.5, 3.1)	4.12 br s	1.12 br d(14.6) 1.89 m	3.78 m
13	3.43 br d(13.5)	3.29 br d(5.9)	3.07 br d(12.2)	2.97 br d(6.6)
14	1.46 m 2.32 ddd(13.1, 13.1, 4.4)	1.66 m 2.29 ddd(13.1, 13.1, 5.9)	1.40 ddd(13.0, 12.9, 5.2) 1.58 br d(13.0)	1.40 m, 1.79 m
15	1.92 m	2.54 m	1.78 m	2.45 m
16	0.93 d(6.5)	0.90 d(6.5)	0.89 d(6.4)	0.88 d(6.5)

Table 2-5-33: ^{1}H NMR spectroscopic data of other licopodium alkaloids **2-5-88**, **2-5-89a**, **2-5-89b**, and **2-5-90a**.

H	2-5-88	2-5-89a[①]	2-5-89b[①]	2-5-90a[②]
2	4.63 d(12.6), 2.37 m	3.56 dd(13.5, 4.3) 3.31 dt(13.5, 3.0)	4.47 dd(12.9, 3.7) 2.59 dt(13.8, 3.0)	2.17 ddd(11.6, 11.3, 2.6) 2.91 br d(11.3)
3	1.71 m, 1.43 m	1.75 m, 1.38 m	1.75 m, 1.32 m	1.68 m
4	2.19 m, 1.02 m	1.48 m	1.47 m	1.76 m, 2.99 br d(13.2)
5	0.89 m	1.57 m	1.70 m	
6	2.87 ddd(11.1, 11.1, 3.0)	4.85 dt(13.8, 4.6)	3.90 dt(12.6, 4.6)	2.84 m
7	2.06 m, 1.31 m	1.93 m, 1.18 m	2.13 m, 1.18 m	1.27 m, 2.01 m
8	1.50 m	2.13 m	2.13 m	1.62 m
9	1.78 m, 0.65 ddd(12.0, 12.0, 12.0)	1.38 m, 1.10 m	1.38 m, 1.18 m	1.78 m, 1.98 m
10	1.10 m	1.94 m	1.94 m	
11	1.97 m, 0.84 m	1.61 m, 1.29 m	1.61 m, 1.29 m	2.26 dd(13.1, 6.5), 2.37 m
12	1.01 d(6.6)	1.09 d(7.8)	1.07 d(7.2)	0.98 m
13	3.07 br s	2.76 m	2.76 m	2.70 m
14	1.71 m, 1.32 m	1.87 m, 1.41 m	1.87 m, 1.41 m	1.26 m, 1.71 m
15	1.58 m (2H)	1.94 m, 1.28 m	1.83 m, 1.39 m	1.35 m, 1.83 m
16	2.17 m	1.75 m, 1.67 m	1.75 m, 1.67 m	1.32 m, 1.58 m
17	2.96 m	3.25 m, 2.59 m	3.25 m, 2.59 m	2.69 m
19	8.18 s			2.42 m, 2.83 m
20		2.04 s	2.06 s	1.00 m
2′	2.96 m, 2.26 m			2.13 ddd(11.2, 11.2, 1.6), 2.85 m
3′	2.33 m			1.62 m, 1.71 m

Table 2-5-33 (continued)

H	2-5-88	2-5-89a[①]	2-5-89b[①]	2-5-90a[②]
4′	2.82 d(10.8), 1.61 m			1.58 m, 3.04 br d(13.9)
5′	1.75 m			
6′	2.56 br s			2.75 m
7′	2.06 m, 1.15 m			1.22 m, 1.98 m
8′	1.54 m			1.55 m
9′	1.93 br d(18.6), 1.71 m			1.85 m, 1.98 m
10′	0.91 m			
11′	1.65 m (2H)			1.92 br d(12.8), 2.56 dd(12.8, 10.0)
12′	0.97 d(6.6)			0.97 m
13′	2.33 s			2.43 m, 2.78 m
14′				1.00 m

[①] Due to the rotation of N-acetyl group, compound **2-5-89** existed as a mixture of two rotamers. Two sets of signals were observed in the ^{1}H NMR spectrum.
[②] Due to the rotation of N-acetyl group, the ^{1}H NMR spectrum of **2-5-90** showed broad signals. Treatment of **2-5-90** with $LiAlH_4$ afforded **2-5-90a**, which provided sharp signals on the ^{1}H NMR spectrum.

Table 2-5-34: ^{1}H NMR spectroscopic data of other licopodium alkaloids **2-5-91**~**2-5-94**.

H	2-5-91	2-5-92	2-5-93	2-5-94
1	3.08 ddd(13.7, 13.5, 2.7) 3.65 br d(13.7)	3.52 br t(12.9) 4.50 br d(12.9)	4.90 ddd(10.5, 4.9, 4.4)	3.79 m 2.91 dt(15.2, 1.6)
2	1.69 m, 1.79 m	1.75 m, 2.01 m	1.89 m, 1.98 m	1.97 m, 1.62 m
3	1.65 m, 1.94 m	1.80 m, 1.94 m	1.75 m, 2.01 m	2.68 m, 2.62 m
4	1.62 m, 2.17 br q(13.3)	1.85 m, 1.91 m	1.89 m (2H)	
5	3.60 br d(13.2)	3.96 m	4.17 m	
6	1.56 ddd(14.1, 14.1, 5.1), 1.79 m	1.76 m, 1.82 m	1.72 m, 1.84 br d(14.1)	2.43 m, 2.40 m
7	1.96 m	2.02 m	2.02 m	2.66 m
8	1.21 q(13.2), 1.95 m	2.45 m 2.98 dd(19.9, 3.8)	2.52 dd(20.8, 9.9) 3.04 dd(20.8, 2.9)	1.48 m, 1.46 m
9	3.82 m			3.35 m, 3.03 dd(15.2, 5.3)
10	0.95 d(6.2)	1.04 d(6.7)	1.04 d(6.6)	1.44 m, 1.41 m
11	1.31 d(6.3)	2.44 s	2.58 s	2.05 m, 2.00 m
12			1.65 ddd(14.9, 10.6, 4.4) 2.39 ddd(14.9, 10.5, 2.6)	
13			3.54 ddq(10.6, 6.1, 2.6)	
14			1.24 d(6.1)	2.18 dd(19.1, 12.2), 1.69 m
15				1.77 m
16				0.95 d(6.3)

Table 2-5-35: ^{1}H NMR spectroscopic data of other licopodium alkaloids **2-5-95**~**2-5-98**.

H	2-5-95	2-5-96	2-5-97	2-5-98
1			8.30 dd(5.0, 1.4)	3.26 br d(13.6), 3.09 m
2	2.47 m (2H)	6.35 d(8.9)	7.24 dd(7.6, 5.1)	1.84 m, 1.76 m
3	2.37 m (2H)	7.42 d(8.9)	7.67 dd(7.7, 1.4)	2.10 m, 1.21 q(12.3)
4				1.73 m
5				3.59 m
6	3.45 s	4.19 br s	3.09 dd(16.5, 8.2)	2.08 m
			2.83 dd(16.5, 9.1)	1.96 dd(10.3, 5.3)
7	2.25 d(4.8)	2.25 br d(4.4)	2.97 m	2.76 br s
8	1.84 m, 0.94 t(13.2)	1.05 t(13.0), 1.94 m	1.90 m, 1.83 m	6.51 d(2.0)
9	3.25 m	2.88 d(13.7)	3.53 m(2H)	3.11 m(2H)
	2.83 d(12.0)	3.55 dd(13.7, 3.1)		
10	2.10 m	2.81 m	1.56 m, 1.35 m	2.04 m, 1.61 m
11	1.95 m	1.74 br d(13.9)	2.06 ddd(13.6, 13.6, 4.6)	2.02 m, 1.82 m
	1.68 d(13.5)	2.14 ddd(13.9, 5.6, 3.9)	1.88 m	
12	1.89 s	2.04 m		
13	3.26 d(3.1)	3.54 d(2.7)		
14	1.95 m	1.18 t(12.1)	2.29 m (2H)	6.22 s
	1.04 ddd(12.6, 12.6, 2.4)	2.07 m		
15	1.74 m	1.86 m	2.12 m	
16	0.89 d(6.5)	0.95 d(6.4)	1.08 d(6.5)	
17				3.74 s

Table 2-5-36: ^{1}H NMR spectroscopic data of other licopodium alkaloids **2-5-99**~**2-5-102**.

H	2-5-99	2-5-100	2-5-101	2-5-102
1	3.46 dt(15.4, 4.2)	2.31 m	3.09 ddd(12.9, 12.8, 3.0)	3.19 m
	2.62 ddd(15.4, 5.2, 1.6)	2.92 br d(11.8)	3.38 br d(12.8)	3.53 m
2	2.02 m, 1.73 m	1.54 m, 1.73 m	1.66 m, 2.00 m	1.74 m, 2.02 m
3	2.04 m, 1.52 m	1.75 m, 2.86 br d(13.6)	2.93 br d(14.5), 2.05 m	2.02 m, 2.98 m
4	2.19 m			
5	3.43 m	2.63 dd(9.2, 8.0)	3.80 dd(9.2, 8.0)	3.82 m
6	1.22 m, 1.96 m	1.04 ddd(12.5, 12.5, 9.2)	1.28 ddd(12.3, 12.3, 9.2)	1.35 m, 2.34 m
		2.09 m	2.11 m	
7	1.70 m	1.58 br d(14.0)	1.75 br d(13.8)	1.69 m
8	2.09 m, 1.67 m	1.79 m, 1.92 br d(16.1)	1.92 m, 2.02 br d(16.8)	1.96 m, 2.09 br d(15.6)
9	3.20 m, 3.05 dd(14.2, 6.1)			

Table 2-5-36 (continued)

H	2-5-99	2-5-100	2-5-101	2-5-102
10	1.88 m, 1.37 m	0.99 d(6.6)	1.03 d(6.4)	1.07 d(6.4)
11	1.86 m,1.91 m	2.13 br d(12.5)	2.37 br d(12.2)	2.43 br d(11.5)
		3.03 dd(12.5, 12.2)	3.17 dd(12.5, 12.2)	3.11 dd(11.5, 11.1)
12		3.48 m	3.79 m	4.70 m
13		1.34 br d(12.6), 1.70 m	1.55 br d(10.6), 1.84 m	1.87 m(2H)
14	2.62 d[①], 2.00 m	1.68 m, 1.77 m	1.70 m, 1.83 m	1.72 m, 1.87 m
15	2.73 br s	1.28 br d(15.5)	1.60 br d(16.1)	1.88 m(2H)
		2.04 ddd(15.5, 13.1, 4.1)	2.17 m	
16		3.46 m	3.85 br d(13.3)	4.28 m
17		1.47 ddd(13.1, 12.8, 4.9)	1.60 m	1.73 m
		1.59 br d(12.8)	1.70 m	1.87 m
18		1.82 m	1.98 m	1.96 m
19		1.13 ddd(12.5, 12.5, 12.5)	1.38 m	2.54 m
		1.72 m	1.93 m	3.03 m
20		3.44 m	3.89 m	
21		0.90 d(6.5)	0.94 d(6.3)	1.04 d(6.3)
22		1.18 d(6.1)	1.38 d(6.3)	2.49 s
23		2.30 s		2.89 s

[①] The coupling constant was not given in the original literature.

Bibliography

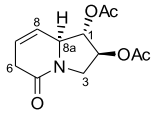
[89] Tan CH, Zhu DY. Helv Chim Acta, 2004, 87: 1963.
[90] Morita H, Arisaka M, Yoshida N, et al. J Org Chem, 2000, 65: 6241.
[91] Takayama H, Katakawa K, Kitajima M, et al. Chem Pharm Bull, 2003, 51: 1163.
[92] Morita H, Ishiuchi K, Haganuma A, et al. Tetrahedron, 2005, 61: 1955.
[93] Wang HB, Tan CH, Tan JJ, et al. Helv Chim Acta, 2007, 90: 153.
[94] Morita H, Hirasawa Y, Kobayashi J, et al. J Nat Prod, 2005, 68: 1809.
[95] Tong XT, Tan CH, Ma XQ, et al. Planta Med, 2003, 69: 576.
[96] Hirasawa Y, Morita H, Kobayashi J. Tetrahedron, 2002, 58: 5483.
[97] Ishiuchi K, Kubota T, Mikami Y, et al. Bioorg Med Chem, 2007, 15: 413.
[98] Kobayashi J, Hirasawa Y, Yoshida N, et al. Tetrahedron Lett, 2000, 41: 9069.
[99] Yin S, Fan CQ, Wang XN, et al. Helv Chim Acta, 2006, 89: 138.
[100] Liu JS, Huang MF. Phytochemistry, 1994, 37: 1759.
[101] Tan CH, Ma XQ, Chen GF, et al. Can J Chem, 2003, 81: 315.
[102] Choo CY, Hirasawa Y, Karimata C, et al. Bioorg Med Chem, 2007, 15: 1703.
[103] Kobayashi J, Hirasawa Y, Yoshida N, et al. J Org Chem, 2001, 66: 5901.
[104] Ishiuchi K, Kubota T, Hayashi S, et al. Tetrahedron Lett, 2009, 50: 4221.
[105] Ayer WA, Kasitu GC. Can J Chem, 1989, 67: 1077.
[106] Morita H, Hirasawa Y, Kobayashi J. J Org Chem, 2003, 68: 4563.

[107] Katakawa K, Nozoe A, Kogure N, et al. J Nat Prod, 2007, 70: 1024.
[108] Takayama H, Katakawa K, Kitajima M, et al. Tetrahedron Lett, 2002, 43: 8307.
[109] Tan CH, Ma XQ, Chen GF, et al. Helv Chim Acta, 2002, 85: 1058.
[110] Tan CH, Chen GF, Ma XQ, et al. J Nat Prod, 2002, 65: 1021.
[111] Tan CH, Chen GF, Ma XQ, et al. J Asian Nat Prod Res, 2002, 4: 227.
[112] Tan CH, Wang BD, Jiang SH, et al. Planta Med, 2002, 68: 188.
[113] Tan CH, Jiang SH, Zhu DY, et al. Tetrahedron Lett, 2000, 41: 5733.
[114] Gao WY, Li YM, Wang BD, et al. Chin Chem Lett, 1999, 10: 463.
[115] Katakawa K, Kitajima M, Aimi N, et al. J Org Chem, 2005, 70: 658.
[116] Hirasawa Y, Morita H, Shiro M, et al. Org Lett, 2003, 5: 3991.
[117] Takayama H, Katakawa K, Kitajima M, et al. Org Lett, 2001, 3: 4165.
[118] Zhao FW, Sun QY, Yang FM, et al. Org Lett, 2010, 12: 3922.
[119] Gao WY, Li YM, Jiang SH, et al. Planta Med, 2000, 66: 664.
[120] Morita H, Hirasawa Y, Shinzato T, et al. Tetrahedron, 2004, 60: 7015.
[121] Kubota T, Yahata H, Yamamoto S, et al. Bioorg Med Chem Lett, 2009, 19: 3577.
[122] Hirasawa Y, Kobayashi J, Morita H. Org Lett, 2006, 8: 123.
[123] He J, Chen XQ, Li MM, et al. Org Lett, 2009, 11: 1397.
[124] Ishiuchi K, Kubota T, Hoshino T, et al. Bioorg Med Chem, 2006, 14: 5995.
[125] Kobayashi J, Hirasawa Y, Yoshida N, et al. J Org Chem, 2001, 66: 5901.
[126] Ishiuchi K, Kubota T, Morita H, et al. Tetrahedron Lett, 2006, 47: 3287.
[127] Hirasawa Y, Morita H, Kobayashi J. Tetrahedron, 2003, 59: 3567.
[128] Hsiao Y, Rivera NR, Yasuda N, et al. Org Lett, 2002, 4: 1243.

2.6 Indolizidine alkaloids

Table 2-6-1: Cos, MFs, and TSs of indolizidine alkaloids **2-6-1**~**2-6-22**.

No.	Compounds	MFs	Test solvents	References
2-6-1	(1*S*,2*S*,8a*S*)-1,2-diacetyloxy-2,3,6,8a-tetrahydro-5(1*H*)-indolizinone	$C_{12}H_{15}NO_5$	$CDCl_3$	[129]
2-6-2	(1*S*,2*S*,8a*S*)-1,2-dihydroxy-2,3,6,8a-tetrahydro-5(1*H*)-indolizinone	$C_8H_{11}NO_3$	D_2O	[129]
2-6-3	(1*S*,2*S*,8a*S*)-1,2-dihydroxy-1,2,3,5,6,8a-hexahydroind-olizine	$C_8H_{13}NO_2$	D_2O	[129]
2-6-4	(1*S*,2*S*,8a*S*)-1,2-dihydroxy-hexahydro-5(1*H*)-indolizin-one	$C_8H_{13}NO_3$	D_2O	[129]
2-6-5	(1*S*,2*R*,6*S*,7*R*,8*R*,8a*R*)-octahydroindolizine-1,2,6,7,8-pentol	$C_8H_{15}NO_5$	D_2O	[130]
2-6-6	(1*S*,2*R*,6*S*,7*R*,8*R*,8a*R*)-octahydroindolizine-1,2,6,7,8-pentylpentaacetate	$C_{18}H_{25}NO_{10}$	$CDCl_3$	[130]
2-6-7	(2*S*,6*S*,7*R*,8*R*,8a*R*)-2,6,7,8-tetrahydroxyindolizidine	$C_8H_{15}NO_4$	D_2O	[131]
2-6-8	(2*R*,6*S*,7*R*,8*R*,8a*R*)-2,6,7,8-tetrahydroxyindolizidine	$C_8H_{15}NO_4$	D_2O	[131]
2-6-9	(2*R*,6*S*,7*R*,8*R*,8a*S*)-2,6,7,8-tetrahydroxyindolizidine	$C_8H_{15}NO_4$	D_2O	[131]
2-6-10	(2*S*,6*S*,7*R*,8*R*,8a*S*)-2,6,7,8-tetrahydroxyindolizidine	$C_8H_{15}NO_4$	D_2O	[131]
2-6-11	tetrahydroxyindolizidine	$C_8H_{15}NO_4$	CD_3OD	[132]
2-6-12	–	$C_8H_{15}NO_4$	CD_3OD	[132]
2-6-13	–	$C_8H_{15}NO_4$	D_2O	[133]
2-6-14	–	$C_8H_{15}NO_4$	D_2O	[133]
2-6-15	(1*R*,2*S*,7*R*,8*R*,8a*S*)-1,2,7,8-tetraacetoxyindolizidin-5-one	$C_{16}H_{21}NO_9$	$CDCl_3$	[133]
2-6-16	(1*R*,2*S*,7*S*,8*S*,8a*S*)-1,2,7,8-tetraacetoxyindolizidin-5-one	$C_{16}H_{21}NO_9$	$CDCl_3$	[133]
2-6-17	(1*S*,2*S*,8a*S*)-1-benzyloxy-2-hydroxy-indolizidine(1-*O*-benzyl-lentiginosine)	$C_{15}H_{21}NO_2$	$CDCl_3$	[134]
2-6-18	(1*S*,2*R*,8*S*,8a*R*)-1,2,8-triacetoxyindolizidine[(–)-8-*epi*-swainsonine triacetate]	$C_{14}H_{21}NO_6$	$CDCl_3$	[135]
2-6-19	(1*R*,2*S*,8*R*,8a*R*)-1,2,8-triacetoxyindolizidine[(+)-1,2-di-*epi*-swainsonine triacetate]	$C_{14}H_{21}NO_6$	$CDCl_3$	[135]
2-6-20	(1*R*,2*S*,8*R*,8a*R*)-1,2,8-trihydroxyindolizidine[(+)-1,2-di-*epi*-swainsonine]	$C_8H_{15}NO_3$	$CDCl_3$	[135]
2-6-21	1-deoxy-castanospermine	$C_8H_{15}NO_3$	–	[136]
2-6-22	1-deoxy-8a-*epi*-castanospermine	$C_8H_{15}NO_3$	C_5N_5D-D_2O	[136]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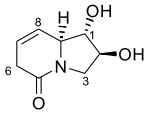
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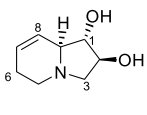
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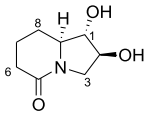
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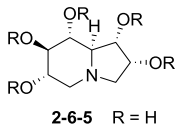
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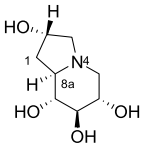
2-6-6 R = Ac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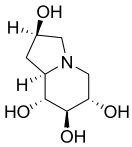
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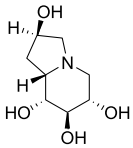
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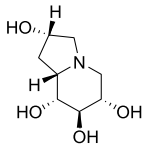
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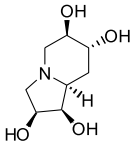
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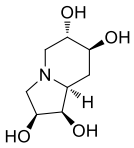
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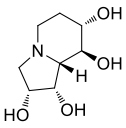
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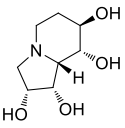
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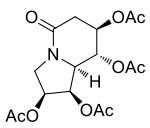
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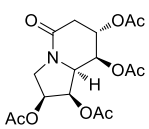

2-6-16

Table 2-6-2: ^{1}H NMR spectroscopic data of indolizidine alkaloids **2-6-1**~**2-6-4**.

H	2-6-1	2-6-2	2-6-3	2-6-4
1	5.07 dd(7.8, 4.3)	3.67 m	3.72 dd(6.8, 4.5)	3.61 dd(8.8, 7.2)
2	5.22 ddd(7.8, 3.9, 3.8)	4.18 dt(8.0, 6.4)	4.06 dt(7.2, 4.9)	4.09 qd(7.0, 1.0)
3	4.09 dd(13.4, 3.4)	3.67 m	2.98 dd(11.1, 7.2)	3.62 dd(12.6, 8.2)
	3.67 dd(13.7, 7.0)	3.39 dd(12.5, 6.4)	2.71 dd(11.1, 4.9)	3.22 dd(12.6, 7.0)
5			2.80 ddd(12.3, 5.7, 3.9)	
			2.61 ddd(12.3, 9.0, 5.1)	
6	2.96~2.98 m	2.89~2.96 m	2.10~2.21 dm(18.2)	2.29 dd(18.3, 6.5)
		2.77~2.84 m	1.90~1.98 dm(18.2)	2.19 ddd(18.3, 11.3, 7.2)
7	5.96 dq(10.1, 1.8)	5.82 dddd(12.5, 5.0, 2.5, 2.3)	5.81 ddq(10.2, 1.0, 2.9)	1.81~1.88 m, 1.51~1.63 m
8	5.86~5.91 m	5.93 m	5.73 dq(10.2, 2.0)	2.10~2.04 m, 1.29 qd(13.3, 3.3)
8a	4.12~4.14 m	3.98~3.96 m	3.05 dquint(7.0, 2.5)	3.29 td(10.0, 3.8)
OAc	2.14 s, 2.06 s			

Table 2-6-3: ^{1}H NMR spectroscopic data of indolizidine alkaloids **2-6-5**~**2-6-7**.

H	2-6-5	2-6-6	2-6-7①
1	3.82 t(7.5)	5.02 t(7.3)	–
2	4.11 q(7.0)	5.25 q(6.3)	4.34~4.44 m

Table 2-6-3 (continued)

H	2-6-5	2-6-6	2-6-7[①]
3*α*	3.26 dd(6.5, 10.5)	2.42 dd(5.0, 10.0)	–
3*β*	2.20 dd(6.5, 10.5)	3.57 dd(6.8, 10.3)	–
5	*α* 2.09 t(10.8)	*α* 2.27 t(10.3)	eq 3.12 dd(5.4)
	β 3.01 dd(5.5, 10.5)	*β* 3.28 dd(5.5, 10.5)	ax 2.25 t(11.0)
6	3.46 ddd(5.5, 9.0, 11.0)	4.97 dd(5.8, 9.8)	3.51 ddd(11.0, 5.4, 9.3)
7	3.20 t(9.0)	5.11 t(9.3)	3.23 t(9.3)
8	3.25 t(9.0)	4.96 t(9.3)	3.14 t(9.3)
8a	2.08 dd(7.5, 9.3)	2.60 t(8.5)	2.54 ddd(7.3, 6.4)
OAc		2.04 s, 2.028 s, 2.025 s, 2.02 s, 2.01 s	

[①] Synthetic compound, part of the NMR data was not assigned in the literature.

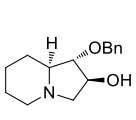
Table 2-6-4: ^{1}H NMR spectroscopic data of indolizidine alkaloids **2-6-8**~**2-6-10**.

H	2-6-8[①]	2-6-9[①]	2-6-10[①]
2	4.38~4.48 m	4.47~4.58 m	4.45~4.50 m
5eq	3.24 dd(5.0)	2.65 dd(3.3)	3.08~3.25 m
5ax	2.29 t(11.0)	2.72 dd(5.6, 12.2)	2.9~3.05 m
6	3.61 ddd(11.0, 5.0, 9.3)	3.82 dt(5.6, 3.3)	3.74~3.85 m
7	3.20 t(9.3)	3.74 t(5.6)	3.74~3.85 m
8	3.35 t(9.3)	3.69 dd(5.6, 3.6)	3.74~3.85 m
8a	2.45 ddd(8.4, 6.3)	3.04 ddd(6.0, 12.0)	3.08~3.25 m

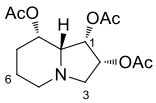
[①] Synthetic compound, part of the NMR data was not assigned in the literature.

Table 2-6-5: ^{1}H NMR spectroscopic data of indolizidine alkaloids **2-6-11**~**2-6-14**.

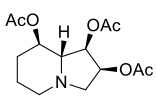
H	2-6-11	2-6-12	2-6-13	2-6-14
1			4.20 dd(3.6)	4.37 dd(4.3,6.0)
2			4.36 ddd(2.6,6.0,8.2)	4.29 ddd(7.5,2.2)
3	3.23~3.06 m	–	2.85 dd(11.2)	2.90 dd(11.2)
			2.57 dd	2.52 dd
5	3.23~3.06 m	*α* 2.04 t(10.5),	2.92 ddd(2.3,4.4,11.6)	2.84 m
		β 3.17 dd(10.8)		
			2.12 td(12.5)	2.34 td(2.5,13.3)
6	3.76 m	3.50 ddd(10.0, 8.9, 4.8)	1.93 dddd	2.05 m(14.8)
			1.51 dddd(12.9)	1.62 m
7	3.94 q(3.0)	3.40 ddd(4.9, 11.0)	3.46 ddd(5.0,11.0)	3.84 ddd
8	*α* 1.79 dt(2.7)	*α* 1.97 ddd(2.6, 13.1)	3.56 t(9.2)	4.09 m
	β 2.30 ddd(12.5, 14.5)	*β* 1.68 ddd(1.2)		
8a	3.06~3.23 m	2.28 ddd(3.9, 11.7)	2.06 dd	2.48 dd



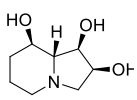
2-6-17



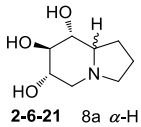
2-6-18



2-6-19



2-6-20



2-6-21 8a α-H
2-6-22 8a β-H

Table 2-6-6: ^{1}H NMR spectroscopic data of indolizidine alkaloids **2-6-15**~**2-6-19**.

H	2-6-15	2-6-16	2-6-17	2-6-18	2-6-19
1	5.52 dd(3.9, 3.0)	5.61 t(4.6)	3.52 dd(1.5, 7.5)	5.40 t(6.5, 5.8)	5.01 t(7.7, 7.7)
2	5.32 ddd(8.6, 9.2)①	5.35 ddd(6.9, 7.4)①	4.07 dd(1.5, 6.0)	5.26 dt(6.5, 1.8, 6.8)	5.20 ddd(7.7, 6.9, 5.7)
3	3.87 dd(11.9) 3.52 dd	3.77 d	2.87 d(10.5) 2.53 dd(6.0, 10.5)	3.27 dd(1.8, 11.2) 2.43 dd(6.8, 11.2)	3.57 dd(6.9, 10.0) 2.33 dd(5.7, 10.0)
5			2.98 m 1.98 m	ax 2.10 m eq 3.22 m	ax 2.10 m(10.7) eq 2.93 br d(10.7)
6	3.04 dd(17.8)② 2.49 dd	2.82 dd(18.5)② 2.53 dd	1.62 m 1.54 m	ax 1.54 m eq 2.00 m	ax 1.63 dddt(13.6) eq 1.72 m(13.6)
7	5.21 ddd (6.9, 8.8)	5.17 td(5.2, 2.8)	1.88 m 1.26 m	ax 1.46 m eq 1.90 m	ax 1.29 m(11.0) eq 2.10 m(4.6)
8	5.27 t(9.4)	5.32 dd(3.1)	1.89 m 1.26 m	5.36 br t(1.3)	4.69 ddd (7.7, 11.0, 4.6, 9.2)
8a	3.80 dd	4.21 dd	1.98 m	2.26 dd(5.8, 1.3)	2.29 t(9.2,7.7)
OAc	2.13 s, 2.04 s 2.03 s, 2.03 s	2.09 s, 2.09 s 2.06 s, 2.04 s			
OBn			4.70 d(12.0) 4.59 d(12.0)		

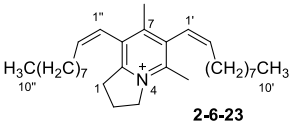
① The original literature gave only two coupling constants.
② The original literature gave only one coupling constant.

Table 2-6-7: ^{1}H NMR spectroscopic data of indolizidine alkaloids **2-6-20**~**2-6-22**.

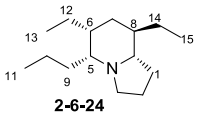
H	2-6-20	2-6-21	2-6-22
1	3.94 t(7.5, 7.5)	–	1.72~2.00 m, 2.22~2.40 m
2	4.20 q(7.5, 7.4, 6.4)	–	1.72~2.00 m, 2.22~2.40 m
3	3.41 dd(7.4, 9.8), 2.27 dd(6.4, 9.8)	–	2.84~3.00 m, 3.49~3.67 m
5ax	2.13 t(10.2, 1.2, 11.1)	2.30 t(10.5)	3.44 br d(12.3)
5eq	2.93 br d(10.2)	3.43 dd(5.2)	3.69 br d(12.3)
6	ax 1.54 m(13.8, 11,1), eq 1.78 br d(1.2, 13.8, 3.7)	4.33 ddd(8.6)	4.55 br s
7	ax 1.34 dq(3.7, 12.4, 9.9), eq 2.06 m(12.4, 4.2)	3.91 t(8.6)	4.42 br s
8	3.54 dt(9.9, 4.2, 8.6)	3.86 t(8.6)	4.42 br s
8a	1.99 t(7.5, 8.6)	2.94 dt(8.6, 2.0)	3.49~3.67 m

Table 2-6-8: Compounds, MFs, and test solvents of indolizidine alkaloids **2-6-23**~**2-6-25**.

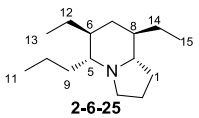
No.	Compounds	MFs	Test solvents	References
2-6-23	6,8-didec-(1*Z*)-enyl-5,7-dimethyl-2,3-dihydro-1*H*-indolizinium	$C_{30}H_{50}N$	CD_2Cl_2	[137]
2-6-24	(5*R**,6*R**,8*R**,9*S**)-6,8-diethyl-5-propyloctahydroindolizine	$C_{15}H_{29}N$	D_2O	[138]
2-6-25	(5*R*,6*S*,8*R*,9*S*)-6,8-diethyl-5-propyloctahydroindolizine	$C_{15}H_{29}N$	D_2O	[138]



2-6-23



2-6-24



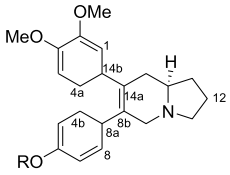
2-6-25

Table 2-6-9: ^{1}H NMR spectroscopic data of indolizidine alkaloids **2-6-23**~**2-6-25**.

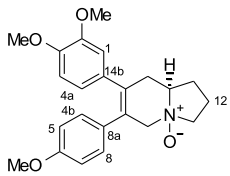
H	2-6-23	2-6-24 (DCl salt)	2-6-25 (DCl salt)
1	3.31 t	*α* 1.56 m, *β* 2.28 m	*α* 1.52 m, *β* 2.16 m
2	2.49 p(7.7)	*α* 1.97 m, *β* 1.88 m	*α* 1.90 m, *β* 1.82 m
3	4.73 t(7.7)	*α* 3.58 dt(9.6, 2.7), *β* 2.95 q(9.1)	*α* 3.48 dt(9.3, 2.6), *β* 2.87 q(9.3)
5	–	3.15 dt(11.0, 3.6)	2.74 dd(10.8, 3.9)
5-Me	2.63 s		
6		1.93 m	1.37 m
7*α*		2.04 m	1.78 m
7*β*		1.20 m	0.85 q(12.0)
7-Me	2.31 s		
8		1.55 m	1.46
8a		2.86 dt(11.5, 6.0)	2.78 dt(11.5, 6.3)
9		1.68, 1.52	1.60, 1.48
10		1.34, 1.15	1.28, 1.14
11,13,15		0.81, 0.84, 0.86	0.68, 0.71, 0.73
12		1.44, 1.12	1.36, 1.12
14		1.39, 1.17	1.37, 1.02
1′	6.23 br AB(11.3)		
2′	6.10 AB(11.3, 7.4)		
3′	1.74 br q(7.4)		
1″	6.23 br AB(11.4)		
2″	6.07 AB(11.4, 7.4)		
3″	1.80 dq(7.4, 1.6)		
4′, 4″	1.36 m		
9′, 9″	1.27 sext		
5′,5″,6′,6″ 7′,7″,8′,8″	1.24~1.18 m		
10′, 10″	0.86 t(7.0)		

Table 2-6-10: Cos, MFs, and TSs of indolizidine alkaloids **2-6-26**~**2-6-29**.

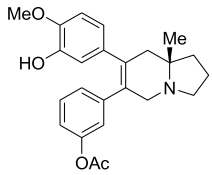
No.	Compounds	MFs	Test solvents	References
2-6-26	(–)-(*R*)-13a*α*-secoantofine	$C_{23}H_{27}NO_3$	C_6D_6-$CDCl_3$ (7:9)	[139]
2-6-27	(–)-(*R*)-13a*α*-6-*O*-desmethylsecoantofine	$C_{22}H_{25}NO_3$	C_6D_6-$CDCl_3$ (7:9)	[139]
2-6-28	(–)-10*β*,13a*α*-secoantofine *N*-oxide	$C_{23}H_{27}NO_4$	C_6D_6-$CDCl_3$ (7:9)	[140]
2-6-29	tyloindicine B	$C_{24}H_{27}NO_4$	DMSO-d_6	[141]



2-6-26 R = Me
2-6-27 R = H



2-6-28



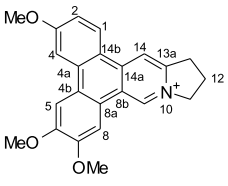
2-6-29

Table 2-6-11: ^{1}H NMR spectroscopic data of indolizidine alkaloids **2-6-26**~**2-6-29**.

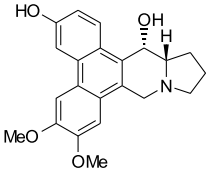
H	2-6-26	2-6-27	2-6-28	2-6-29
1	6.51 d(2.0)	6.33 d(1.7)	6.29 d(1.9)	7.90 dd(9, 3)
2				7.16 m
4	6.47 d(8.3)	6.38 d(8.3)	6.39 d(8.2)	
4a	6.66 dd(8.3, 2.0)	6.46 dd(8.3, 1.7)	6.43 dd(8.2, 1.9)	8.14 d(3)
4b	6.99	6.67	6.97 d(8.5)	–
5	6.61	6.47	6.50 d(8.5)	7.16 m
5a				7.90 dd(9, 3)
6				7.70 m
7	6.61	6.47	6.50 d(8.5)	
8	6.99	6.67	6.97 d(8.5)	6.97 d(3)
9*α*	3.03 d(15.9)	3.00 d(15.4)	4.69 br s	–
9*β*	3.84 d(15.9)	3.67 d(15.4)	5.28 br s	–
11*α*	2.11 m	1.81 m	2.92 br m	–
11*β*	3.21 m	3.14 m	4.46 br s	–
12	*α*1.66 m, *β*1.86 m	*α*1.77 m, *β*1.56 m	*α*1.54 m, *β*1.98 m	–
13*α*	1.93 m	1.84 m	1.66 m(ov)	–
13*β*	1.53 m	1.49 m	1.62 m(ov)	–
13a	2.32 m	2.19 m	2.82 m	
14*α*	2.68 d(16.4)	2.46 m	2.35 dd(17.6, 3.9)	–
14*β*	2.48 dd(16.4, 11.3)		2.51 dd(17.6, 11.8)	–
Me	3.39 s(2-OMe)	3.30 s(2-OMe)	3.27 s(2-OMe)	3.80 s
	3.49 s(3-OMe)	3.45 s(3-OMe)	3.41 s(3-OMe)	1.60 s(C-Me)
	3.39 s(6-OMe)		3.31 s(6-OMe)	
OAc				2.52 s
OH				6.30 br s

Table 2-6-12: Cos, MFs, and TSs of indolizidine alkaloids **2-6-30**~**2-6-59**.

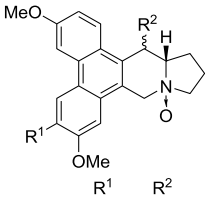
No.	Compounds	MFs	Test solvents	References
2-6-30	tylophoridicine D	$C_{23}H_{22}NO_3$	DMSO-d_6	[142]
2-6-31	tylophoridicine E	$C_{22}H_{23}NO_4$	DMSO-d_6	[142]
2-6-32	tylophoridicine C	$C_{22}H_{23}NO_5$	DMSO-d_6	[142]
2-6-33	tylophoridicine F	$C_{22}H_{23}NO_5$	DMSO-d_6	[142]
2-6-34	ficuseptine B	$C_{23}H_{23}NO_4$	CD_3COCD_3	[143]
2-6-35	ficuseptine C	$C_{22}H_{21}NO_3$	CD_3COCD_3	[143]
2-6-36	ficuseptine D	$C_{23}H_{25}NO_3$	CD_3COCD_3	[143]
2-6-37	10*R*,13a*R*-tylophorine *N*-oxide	$C_{24}H_{27}NO_5$	CD_3OD	[143]
2-6-38	10*R*,13a*R*-tylocrebrine *N*-oxide	$C_{24}H_{27}NO_5$	CD_3OD	[143]
2-6-39	10*S*,13a*R*-tylocrebrine *N*-oxide	$C_{24}H_{27}NO_5$	CD_3OD	[143]
2-6-40	10*S*,13a*R*-isotylocrebrine *N*-oxide	$C_{24}H_{27}NO_5$	CD_3OD	[143]
2-6-41	10*S*,13a*S*-isotylocrebrine *N*-oxide	$C_{24}H_{27}NO_5$	CD_3OD	[143]
2-6-42	antofine	$C_{23}H_{25}NO_3$	$CDCl_3$	[144]
2-6-43	ficuseptine A	$C_{25}H_{29}NO_7$	$CDCl_3$	[145]
2-6-44	tylophoridicine A	$C_{22}H_{23}NO_3$	DMSO-d_6	[146]
2-6-45	tylophorinine	$C_{23}H_{25}NO_4$	$CDCl_3$	[146]
2-6-46	*O*-methyl-tylophorinidine	$C_{23}H_{25}NO_4$	$CDCl_3$	[146]
2-6-47	tylophorinidine	$C_{22}H_{23}NO_4$	DMSO-d_6	[146]
2-6-48	tyloindicine A	$C_{24}H_{27}NO_4$	DMSO-d_6+CF_3COOD	[141]
2-6-49	(+)-14-hydroxyisotylocrebrine	$C_{24}H_{27}NO_5$	DMSO-d_6	[141]
2-6-50	4,6-desmethylistylocrebrine	$C_{22}H_{23}NO_4$	DMSO-d_6	[141]
2-6-51	tyloindicine C	$C_{23}H_{25}NO_4$	DMSO-d_6	[141]
2-6-52	tyloindicine D	$C_{24}H_{27}NO_5$	DMSO-d_6	[141]
2-6-53	tyloindicine E	$C_{22}H_{23}NO_3$	DMSO-d_6	[141]
2-6-54	3-demethyl-14α-hydroxyisotylocrebrine *N*-oxide	$C_{23}H_{25}NO_6$	$CDCl_3$-CD_3OD	[147]
2-6-55	3-demethyl-14α-hydroxyisotylocrebrine	$C_{23}H_{25}NO_5$	$CDCl_3$-CD_3OD	[147]
2-6-56	isotylocrebrine *N*-oxide	$C_{24}H_{27}NO_5$	CD_3OD	[147]
2-6-57	3.4-didemethyl-14α-hydroxyisotylocrebrine *N*-oxide	$C_{22}H_{23}NO_6$	CD_3OD	[147]
2-6-58	*trans*-(+)-3,14α-dihydroxy-6,7-dimethoxyphenanth-roindolizidine	$C_{22}H_{23}NO_4$	DMSO-d_6	[148]
2-6-59	*trans*-(+)-3,14α-dihydroxy-4,6,7-trimethoxyphenan-throindolizidine	$C_{23}H_{25}NO_5$	$CDCl_3$	[148]



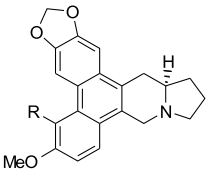
2-6-30



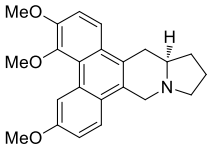
2-6-31



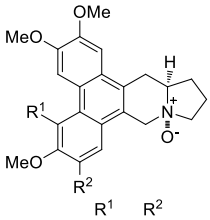
	R^1	R^2
2-6-32	OH	α-OH
2-6-33	OMe	β-OH



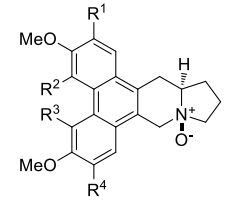
2-6-34 R = OMe
2-6-35 R = H



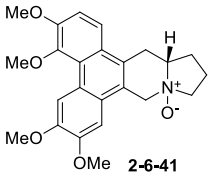
2-6-36



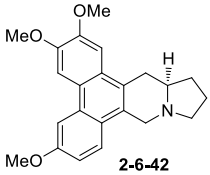
	R^1	R^2
2-6-37	H	OMe
2-6-38	OMe	H



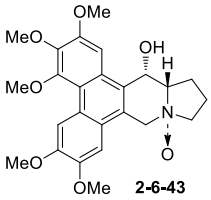
	R^1	R^2	R^3	R^4
2-6-39	OMe	H	OMe	H
2-6-40	H	OMe	H	OMe



2-6-41



2-6-42



2-6-43

Table 2-6-13: ^{1}H NMR spectroscopic data of indolizidine alkaloids **2-6-30**~**2-6-33**.

H	2-6-30	2-6-31	2-6-32	2-6-33
1	8.70 d(9.2)	8.13 d(9.0)	8.04 d(9)	8.14 d(9.0)
2	7.34 dd(9.2, 2.5)	7.10 dd(9.0, 2.0)	7.16 dd(9.0, 2.0)	7.24 dd(9.0, 1.0)
4	7.85 d(2.5)	7.90 d(2.0)	7.69 d(2.0)	7.67 d(1.0)
5	7.78 s	7.87 s	7.92 s	7.56 s
8	7.99 s	7.03 s	7.07 s	6.64 s
9	10.36 s	4.34 d(14.5), 3.36 d(14.5)	5.20 d(15), 4.83 d(15)	5.07 d(15.0), 4.47 d(15.0)
11	4.92 m	2.29 m, 3.25 m	3.62 m, 3.71 m	3.54 m, 3.41 m
12	2.54 m	1.79 m, 1.9 m	2.07 m, 2.31 m	2.14 m
13	3.53 m	2.18 m, 1.79 m	2.10 m, 2.69 m	2.96 m, 2.54 m
13a		2.34 m	3.71 m	3.85 m
14	8.98 s	4.86 s	5.16 d(2.5)	4.96 s
OMe	4.05 s, 4.04 s, 4.03 s	3.98 s, 3.99 s	3.09 s, 3.09 s	4.02 s, 4.00 s, 3.91 s

Table 2-6-14: ^{1}H NMR spectroscopic data of indolizidine alkaloids **2-6-34**~**2-6-38**.

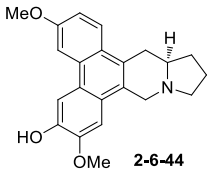
H	2-6-34	2-6-35	2-6-36	2-6-37	2-6-38
1	7.45 s	7.43 s	7.83 d(8.4)	7.43 s	7.39 s
2			7.49 d(8.4)		
4	9.18 s	8.18 s		8.03 s	9.28 s
5		8.05 d(2.8)	9.31 d(2.8)	8.02 s	
7	7.42 d(9.2)	7.21 dd(8.8, 2.8)	7.25 dd(7.2, 2.8)		7.45 d(7.8)
8	7.71 d(9.2)	7.86 d(8.8)	7.87 d(7.2)	7.28 s	7.69 d(7.8)

Table 2-6-14 (continued)

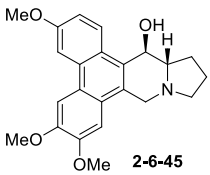
H	2-6-34	2-6-35	2-6-36	2-6-37	2-6-38
9	4.58 d(14.8)	4.65 d(17.6)	4.58 d(14.6)	5.17 d(15.8)	5.13 d(16.0)
	3.57 d(14.8)	3.67 d(17.6)	3.57 d(14.6)	4.86 d(15.8)	4.81 d(16.0)
11	3.35 td(9.0, 3.0)	3.59 td(14.4, 2.0)	3.25 td(14.8, 3.2)	3.80 td(9.0, 3.0)	3.72 br t(16.0)
	2.45 q(9.0)	3.10 q(14.4)	2.41 q(14.8)	3.72 q(9.0)	3.65 q(16.0)
12	1.91 m, 2.05 m	1.89 m, 2.07 m	1.82 m, 2.01 m	2.14 m, 2.33 m	1.88 m, 2.60 m
13	1.69 m, 2.21 m	1.71 m, 2.28 m	1.70 m, 2.20 m	1.89 m, 2.95 m	2.14 m, 2.30 m
13a	2.36 m	2.43 m	2.34 m	3.94 m	3.98 m
14	2.63 dd(16.0, 4.0)	3.30 dd(16.0, 5.2)	2.94 dd(15.0, 11.0)	3.57 dd(16.5, 12.0)	3.48 dd(16.0, 11.0)
	3.28 dd(16.0, 4.0)	3.35 dd(16.0, 5.2)	3.32 dd(15.0, 3.0)	3.37 dd(16.5, 4.0)	3.29 dd(16.0, 2.0)
OMe	3.88 s(5-OMe)	4.02 s(6-OMe)	3.98 s(3-OMe)	4.02 s(2-OMe)	4.01 s(2-OMe)
	4.01 s(6-OMe)		4.05 s(4-OMe)	4.09 s(3-OMe)	4.01 s(3-OMe)
			3.92 s(6-OMe)	4.09 s(6-OMe)	3.90 s(5-OMe)
				4.04 s(7-OMe)	4.01 s(6-OMe)
OCH_2O	6.15 s	6.15 s			

Table 2-6-15: ^{1}H NMR spectroscopic data of indolizidine alkaloids **2-6-39**~**2-6-43**.

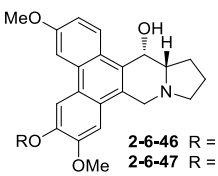
H	2-6-39	2-6-40	2-6-41	2-6-42	2-6-43
1	7.29 s	7.85 d(9.2)	7.87 d(9.4)	7.32 s	7.61 s
2		7.45 d(9.2)	7.47 d(9.4)		
4	9.26 s			7.92 s	
5		9.30 s	9.34 s	7.90 d(2.5)	9.24 s
7	7.40 d(8.8)			7.20 dd(2.5, 9.0)	
8	7.50 d(8.8)	6.98 s	7.25 s	7.82 d(9.0)	7.02 s
9	5.04 d(15.0)	5.03 d(15.2)	5.18 d(15.8)	3.70 d(14.7)	α5.52 d(14.9)
	4.66 d(15.0)	4.65 d(15.2)	4.81 d(15.8)	4.70 dd(2.5, 9.0)	β4.64 d(14.9)
11	3.85 td(18.0, 2.0)	3.87 td(19.0, 3.0)	3.80 td(19.2, 3.1)	2.45 m	α4.05 m
	3.65 q(18.0)	3.68 q(19.0)	3.29 q(19.2)	3.45 dd(1.8, 8.6)	β3.62 m
12	2.11 m, 2.37 m	2.13 m, 2.35 m	1.84 m, 2.59 m	1.92 m, 2.03 m	α2.25 m
					β2.66 m
13	2.19 m, 2.31 m	2.18 m, 2.28 m	2.16 m, 2.32 m	1.78 m, 2.24 m	α3.11 m
					β2.25 m
13a	3.45 m	3.51 m	4.08 m	2.51 m	3.47 m
14	3.52 dd(16.0, 3.0)	3.47 dd(14.0, 7.0)	3.53 dd(16.0, 8.0)	2.91 m	5.26 d(2.6)
	3.15 dd(16.0, 11.0)	3.24 dd(14.0, 3.0)	3.29 dd(16.0, 2.0)	3.36 dd(2.7, 15.7)	
2-OMe	3.99 s			4.11 s	4.06 or 4.07 s
3-OMe	3.93 s	4.02 s	4.02 s	4.07 s	4.06 or 4.07 s
4-OMe		4.01 s	4.02 s		3.98 s
5-OMe	4.02 s				
6-OMe	4.02 s	3.92 s	3.91 s	4.01 s	4.09 s
7-OMe		3.99 s	4.04 s		4.04 s



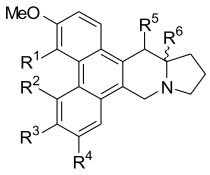
2-6-44



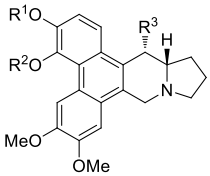
2-6-45



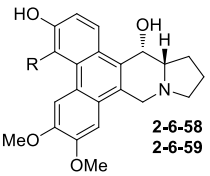
2-6-46 R = Me
2-6-47 R = H



	R^1	R^2	R^3	R^4	R^5	R^6
2-6-48	OMe	OMe	OMe	H	H	H
2-6-49	OMe	H	OMe	OMe	OH	H
2-6-50	OH	H	OH	OMe	H	H
2-6-51	OH	H	OH	OMe	H	β-Me
2-6-52	OMe	OH	OMe	OMe	H	H
2-6-53	H	H	OH	OMe	H	H



	R^1	R^2	R^3
2-6-54 *N*-oxide	H	Me	OH
2-6-55	H	Me	OH
2-6-56 *N*-oxide	Me	Me	H
2-6-57 *N*-oxide	H	H	OH



2-6-58 R = H
2-6-59 R = OMe

Table 2-6-16: ^{1}H NMR spectroscopic data of indolizidine alkaloids **2-6-44**~**2-6-47**.

H	2-6-44	2-6-45	2-6-46	2-6-47
1	7.88 d(9)	–	8.44 d(9)	8.17 d(9)
2	7.17 dd(9, 2)	7.25 dd(9, 2)	7.25 dd(9, 2)	7.18 dd(9, 2)
4	7.85 d(2)	–	7.69 d(2)	7.83 d(2)
5	8.03 s	–	7.50 s	8.03 s
8	7.17 s	–	6.01 s	7.13 s
9	4.53 d(16), 3.49 d(16)	–	3.23 d(16), 2.91 d(16)	4.48 d(16), 3.40 d(16)
11	2.30 m, 3.38 m	2.40 m, 3.32 m	2.18 m, 3.29 m	2.30 m, 3.33 m
12	1.81~1.84 m	2.02, 2.40 m	1.87 m, 2.05 m	1.77~1.85 m
13	1.61 m, 2.12 m	1.90 m, 2.25 m	1.87 m, 2.41 m	1.77~1.85 m, 2.16 m
13a	2.36 m	2.40 m	2.20 m	2.33 m
14	2.74 m, 3.03 m	4.93 br d	4.87 br d	4.88 br d
3-OMe	3.95 s	4.00 s	4.04 s	3.95
6-OMe		4.09 s	4.08 s	
7-OMe	3.94 s		3.77 s	3.94

Table 2-6-17: ^{1}H NMR spectroscopic data of indolizidine alkaloids **2-6-48**~**2-6-50**.

H	2-6-48	2-6-49	2-6-50
1	7.94 d(9)	8.00 d(9)	8.04 d(9)
2	7.36 d(9)	7.28 d(9)	7.26 d(9)

Table 2-6-17 (continued)

H	2-6-48	2-6-49	2-6-50
5		8.24 s	8.33 s
7	7.70 d(9)		
8	7.10 d(9)	7.10 s	7.10 s
13a	5.40 m	3.40 m	4.70 br s
14	–	6.04 br s	
OMe	4.00 br s(12H, 4×OMe)	4.0 s, 3.92 s, 3.90 s, 3.80 s	4.00 s, 3.92 s
OH		4.60 br s	7.83 br s, 6.96 s
CH_2		2.60 m, 2.40 m, 1.80 m	2.52 m, 2.20 m, 1.80 m

Table 2-6-18: ^{1}H NMR spectroscopic data of indolizidine alkaloids **2-6-51**~**2-6-55**.

H	2-6-51	2-6-52	2-6-53	2-6-54	2-6-55
1	7.90 d(9)	8.00 d(9)	7.96 d(9)	8.01 d(9)	8.09 d(9)
2	7.00 d(9)	7.20 d(9)	7.20 d(9)	7.32 d(9)	7.40 d(9)
4			7.62 d(3)		
5	8.03 s		7.72 d(9)	9.30 s	9.28 s
8	6.78 s	7.16 s	7.04 s	7.11 s	7.20 s
9	–	–	–	4.82 d(15), 5.34 d(15)	3.60 d(15), 4.20 d(15)
11	–	–	–	3.75 q(9), 3.90 br t(9)	–
13a	–	4.60 br s	4.23 br s	–	–
14	–	–	–	β 5.35 d(3)	β 5.04 br s
Me	3.80 s, 3.70 s 1.60 s(C-Me)	4.10 s, 3.96 s(9H)	3.94 s, 3.42 s	3.89 s(4-OMe) 4.09 s(6-OMe) 4.08 s(7-OMe)	3.88 s(4-OMe) 4.12 s(6-OMe) 4.07 s(7-OMe)
OH	7.66 br s, 6.60 br s	7.80 br s	8.14 br s		

Table 2-6-19: ^{1}H NMR spectroscopic data of indolizidine alkaloids **2-6-56**~**2-6-59**.

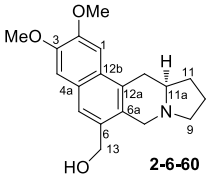
H	2-6-56	2-6-57	2-6-58	2-6-59
1	7.81 d(9)	8.15 d(9)	8.13 d(9.0)	8.15 d(9)
2	7.26 d(9)	7.21d(9)	7.09 dd(9.0, 1.8)	7.34 d(9)
4		–	7.92 d(1.8)	
5	9.27 s	7.95 s	7.91 s	9.00 s
8	7.16 s	7.08 s	7.16 s	7.07 s
9	4.97 d(15) 5.13 d(15)	4.88 d(15) 5.33 d(15)	3.43 ABq(15.0) 4.50 ABq(15.0)	3.59 ABq(15) 4.46 ABq(15)
11	–	–	2.36 m, 3.30 m	2.61 m, 3.41 m
12	–	–	1.75~1.85 m	1.96 m
13	–	–	1.82 m, 2.17 m	2.35 m, 2.50 m
13a	–	–	2.39 m	2.90 m

Table 2-6-19 (continued)

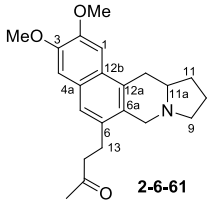
H	2-6-56	2-6-57	2-6-58	2-6-59
14	–	β 5.35 br s	4.90 d(9.6)	5.10 m
3-OMe	3.76 s			
6-OMe	3.85 s	4.09 s	3.99 s	4.08 s
4-OMe	4.030 s or 4.033 s			3.84 s
7-OMe	4.030 s or 4.033 s	4.06 s	3.91 s	4.04 s
14-OH			4.59 d(9.6)	

Table 2-6-20: Cos, MFs, and TSs of indolizidine alkaloids **2-6-60** and **2-6-61**.

No.	Compounds	MFs	Test solvents	References
2-6-60	fistulosine	$C_{19}H_{23}NO_3$	$CDCl_3$	[149]
2-6-61	2,3-dimethoxy-6-(3-oxo-butyl)-7,9,10,11,11a,12-hexahy-drobenzo[*f*]pyrrolo[1,2-*b*]isoquinoline	$C_{22}H_{27}NO_3$	$CDCl_3$	[150]



2-6-60



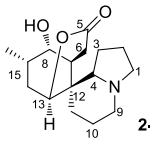
2-6-61

Table 2-6-21: ^{1}H NMR spectroscopic data of indolizidine alkaloids **2-6-60** and **2-6-61**.

H	2-6-60	2-6-61	H	2-6-60	2-6-61
1	7.15 s	7.14 s	11a	2.43 m	3.28 m
4	7.11 s	7.06 s	12	2.87 dd(15, 11), 3.37 m	3.30 m, 2.86 m
5	7.56 s	7.34 s	13	4.70 s	2.91 m
7	3.43 d(15), 4.28 d(15)	4.29 d(14.9), 3.45 d(14.9)	14		2.82 m
9	2.35 q(9), 3.37 m	2.39 m	16		2.15 s
10	1.90 m, 2.01 m	1.95 m	2-OMe	4.02 s	3.98 s
11	1.72 m, 2.23 m	1.25 m, 2.22 m	3-OMe	4.00 s	4.00 s

Table 2-6-22: Cos, MFs, and TSs of indolizidine alkaloid **2-6-62**.

No.	Compound	MF	Test solvent	Reference
2-6-62	serratezomine A	$C_{16}H_{25}NO_3$	CD_3OD	[151]



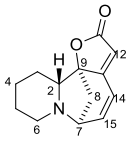
2-6-62

Table 2-6-23: ^{1}H NMR spectroscopic data of indolizidine alkaloid **2-6-62**.

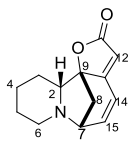
H	2-6-62	H	2-6-62
1	3.35 m, 3.54 ddd(9.2, 9.2, 9.2)	9	2.98 dt(3.5, 13.0), 3.26 m
2	2.20 m	10	1.84 m, 2.03 m
3	2.25 m, 2.15 m	11	1.40 dt(3.3, 13.6), 2.83 br d(13.6)
4	3.81 dd(6.0, 10.9)	13	4.32 br d(2.7)
6	2.46 d(20.0), 3.14 dd(8.0, 20.0)	14	1.83 m
7	2.61 m	15	1.78 m
8	3.77 t(3.4)	16	1.01 d(6.4)

Table 2-6-24: Cos, MFs, and TSs of indolizidine alkaloids **2-6-63** and **2-6-64**.

No.	Compounds	MFs	Test solvents	References
2-6-63	securinine	$C_{13}H_{15}NO_2$	$CDCl_3$	[152]
			D_2O	[152]
2-6-64	allosecurinine	$C_{13}H_{15}NO_2$	$CDCl_3$	[152]



2-6-63



2-6-64

Table 2-6-25: ^{1}H NMR spectroscopic data of indolizidine alkaloids **2-6-63** and **2-6-64**.

H	2-6-63 ($CDCl_3$)	2-6-63 · HCl (D_2O)	2-6-64
2	2.10 dd(11.3, 2.5)	3.72 dd(12.7, 4.8)	3.65 dd(13.2, 3.5)
3	1.67~1.48 m	1.99~1.73 m	ax 1.15 m, eq 1.34 m
4	1.24 m, 1.88 m	1.40~1.31 m, 1.99~1.73 m	1.42 m, 1.70 m
5	1.67~1.48 m	1.99~1.73 m	1.70 m
6	ax 2.42 ddd(10.5, 7.5, 7.0) eq 2.97 dt(10.5, 3.7)	3.39 m	2.75 m
7	3.83 dd(5.3, 4.1)	4.47 dd(6.4, 4.8)	3.91 dd(4.4, 5.3)
8	1.78 d(9.2), 2.50 dd(4.1, 9.2)	2.22 d(12.2), 2.99 dd(4.8, 12.2)	1.93 d(9.8), 2.68 dd(4.4, 9.8)
12	5.56 s	6.02 s	5.73 s
14	6.61 d(9.2)	6.96 d(9.0)	6.66 d(9.1)
15	6.43 dd(9.2, 5.3)	6.74 dd(9.0, 6.4)	6.83 dd(9.1, 5.3)

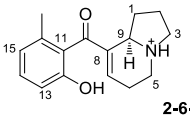
Table 2-6-26: Cos, MFs, and TSs of indolizidine alkaloids **2-6-65**~**2-6-97**.

No.	Compounds	MFs	Test solvents	References
2-6-65	elaeocarpenine	$C_{16}H_{20}NO_2$	DMSO-d_6	[153]
2-6-66	isoelaeocarpicine	$C_{16}H_{22}NO_3$	DMSO-d_6	[153]
2-6-67	isoelaeocarpine	$C_{16}H_{20}NO_2$	DMSO-d_6	[153]
2-6-68	elaeocarpine	$C_{16}H_{20}NO_2$	DMSO-d_6	[153]
2-6-69	–	$C_{15}H_{25}NO$	D_2O	[154]
2-6-70	kinganone	$C_{13}H_{19}NO_2$	C_5D_5N	[155]
2-6-71	3′′′′-oxo-juliprosopine①	$C_{40}H_{73}N_3O_3$	CD_3OD	[156]
2-6-72	3-oxo-juliprosine①	$C_{40}H_{70}N_3O_2$	CD_3OD	[156]
2-6-73	3′-oxo-juliprosine①	$C_{40}H_{70}N_3O_2$	CD_3OD	[156]
2-6-74	serratezomine B	$C_{16}H_{25}NO_4$	CD_3OD	[151]
2-6-75	(2*S*,3*S*,4*R*)-7-acetoxy-1-benzyloxy-2-*tert*-butyldimeth-ylsiloxy-6-chloro-5-oxo-6-eno-indolizine	$C_{23}H_{32}ClNO_5Si$	$CDCl_3$	[134]
2-6-76	(1*S*,2*R*,8a*S*)-1-benzyloxy-2-*tert*-butyldimethylsiloxy-7-hydroxy-6-methoxycarbonyl-6-eno-indolizidine	$C_{23}H_{35}NO_5Si$	$CDCl_3$	[134]
2-6-77	(1*S*,2*R*,8a*S*)-1-benzyloxy-2-*tert*-butyldimethylsiloxy-7-hydroxy-8-methoxycarbonyl-7-eno-indolizidine	$C_{23}H_{35}NO_5Si$	$CDCl_3$	[134]
2-6-78	(1*S*,2*R*,6*S*,7*S*,8a*S*)-3-benzyloxy-2-*tert*-butyldimethyl-siloxy-7-hydroxy-6-methoxycarbonyl-indolizidine	$C_{23}H_{37}NO_5Si$	$CDCl_3$	[134]
2-6-79	(1*S*,2*R*,6*S*,7*S*,8a*S*)-7-acetoxy-6-acetoxymethyl-1-benz-yloxy-2-*tert*-butyldimethylsiloxy-indolizidine	$C_{26}H_{41}NO_6Si$	$CDCl_3$ C_6D_6	[134] [134]
2-6-80	(1*S*,2*R*,8a*S*)-1-benzyloxy-2-*tert*-butyldimethylsiloxy-7-oxo-indolizidine	$C_{21}H_{33}NO_3Si$	$CDCl_3$	[134]
2-6-81	(1*S*,2*R*,7*R*,7a*R*)-7-acetoxy-1-benzyloxy-2-*tert*-butyldi-methylsiloxy-indolizidine	$C_{23}H_{37}NO_4Si$	C_6D_6	[134]
2-6-82	(1*S*,2*R*,8a*S*)-1-benzyloxy-2-*tert*-butyldimethylsiloxy-indolizidine	$C_{21}H_{35}NO_2Si$	$CDCl_3$	[134]
2-6-83	(1*S*,2*R*,8a*S*)-1-benzyloxy-2-hydroxy-indolizidine	$C_{15}H_{21}NO_2$	$CDCl_3$	[134]
2-6-84	pumiliotoxin 251 D	$C_{16}H_{29}NO$	$CDCl_3$	[157]
2-6-85	(+)-allopumiliotoxin 267A	$C_{16}H_{29}NO_2$	$CDCl_3$	[158]
2-6-86	(+)-allopumiliotoxin 339B	$C_{19}H_{33}NO_4$	$CDCl_3$	[158]
2-6-87	(8*R*,8a*S*)-8-hydroxy-8-methyl-6-((*Z*)-2(*R*)-methylhex-ylidene)octahydroindolizin-7-one	$C_{16}H_{27}NO_2$	$CDCl_3$	[158]
2-6-88	–	$C_{16}H_{29}NO_2$	$CDCl_3$	[158]
2-6-89	(8*R*,8a*S*)-8-hydroxy-8-methyl-6-[(*Z*)-2(*R*)-methyl-4-(benzyloxy)butylidene]octahydroindolizin-7-one	$C_{21}H_{29}NO_3$	$CDCl_3$	[158]
2-6-90	(8*R*,8a*S*)-8-hydroxy-8-methyl-6-[(*Z*)-2(*R*)-methyl-4-(benzyloxy)butylidene]octahydroindolizin-7-ol	$C_{21}H_{31}NO_3$	$CDCl_3$	[158]
2-6-91	(8*R*,8a*S*)-7-{[(dimethylethyl)dimethylsilyl]oxy}-8-hyd-roxy-8-methyl-6-[(*Z*)-2(*R*)-methy1-4-(benzy1oxy)but-ylidene]octahydroindolizine	$C_{27}H_{45}NO_3Si$	$CDCl_3$	[158]

Table 2-6-26 (continued)

No.	Compounds	MFs	Test solvents	References
2-6-92	(8*R*,8a*S*)-7-{[(dimethylethyl)dimethylsilyl]oxy}-8-hydr-oxy-8-methyl-6-[(*Z*)-2(*R*)-methyl-4-hydroxy-butyli-dene]octahydroindolizine	$C_{20}H_{39}NO_3Si$	$CDCl_3$	[158]
2-6-93	(8*R*,8a*S*)-7-{[(dimethylethyl)dimethylsilyl]oxy}-8-hydr-oxy-8-methyl-6-[(*Z*)-2(*R*)-methyl-4-oxobutylidene]octahydroindolizine	$C_{20}H_{37}NO_3Si$	$CDCl_3$	[158]
2-6-94	(8*R*,8a*S*)-7-{[(dimethylethyl)dimethylsilyl]oxy}-8-hydr-oxy-8-methyl-6-{(*Z*)-2(*R*),5-dimethyl-7-[[(dimethyl-ethyl)diphenylsilyl]oxy]-6-oxo-4-octen-1-ylidene}octa-hydroindolizine	$C_{41}H_{63}NO_4Si_2$	$CDCl_3$	[158]
2-6-95	(7*R*,8*R*,8a*S*)-8-(benzyloxy)-7-hydroxy-8-methyl-6-[(*E*)-(*R*)-2-methylhexylidene]-octahydroindolizine	$C_{23}H_{35}NO_2$	$CDCl_3$	[159]
2-6-96	(7*R*,8*R*,8a*S*)-8-(benzyloxy)-7-hydroxy-6(*Z*)-[6(*R*),7(*R*)-dihydroxy-2(*R*),5-dimethyl-4(*E*)-octenylidene)-8-meth-yloctahydroindolizine	$C_{26}H_{39}NO_4$	$CDCl_3$	[159]
2-6-97	(+)-allopumiliotoxin 339A	$C_{19}H_{33}NO_4$	$CDCl_3$	[159]

① Renumbering for the carbon skeleton of the compound was done in this handbook.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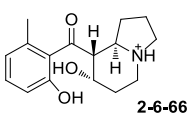
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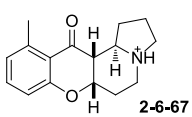
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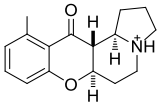
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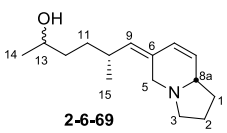
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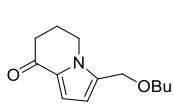
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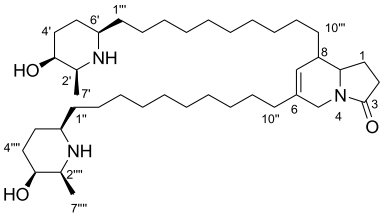
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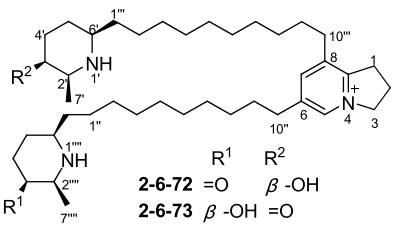
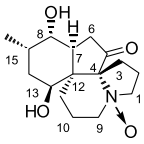

2-6-72 =O β-OH
2-6-73 β-OH =O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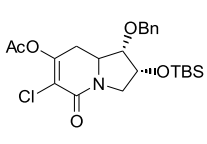
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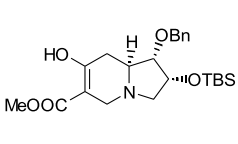
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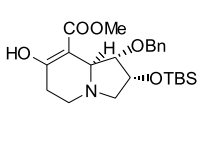
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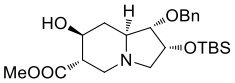

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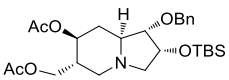

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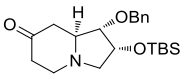

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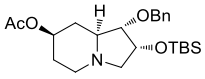


2-6-81

Table 2-6-27: ^{1}H NMR spectroscopic data of indolizidine alkaloids **2-6-65**~**2-6-68**.

H	2-6-65 (DCl)	2-6-66	2-6-67	2-6-68
1	2.53 m	2.49 m	1.78 ddd(2.4, 10.8, 17.4)	1.77 ddd(1.8, 11.4, 18.0)
	1.81 ddd(6.0, 10.8, 15.6)	1.61 ddd(2.4, 10.8, 18.0)	1.90 m	2.68 br
2	2.07 t(8.4)	2.03 dddd(1.8, 6.0, 6.0, 15.6)	1.90 m	1.96 br dd(10.8, 10.8)
		1.84 dddd(2.4, 11.4, 11.4, 18.0)		2.09 br
3	3.31 m, 3.57 dd(2.4, 13.2)	3.59 ddd(2.4, 2.4, 9.6)	3.07 ddd(2.4, 9.6, 18.0)	2.95 br dd(9.6, 18.0)
		3.08 m	3.60 ddd(2.1, 2.1, 10.2)	3.50 m
4	10.82 s	9.56 br	10.23 br s	10.36 br
5	3.16 br, 3.31 m	3.41 dd(2.4, 12.6)	3.28 ddd(3.3, 12.0, 12.0)	3.03 ddd(3.0, 12.6, 18.0)
		3.08 m	3.65 br dd(2.4, 13.8)	3.69 br dd(4.8, 13.2)
6	2.53 m	1.87 t(16.8)	2.29 br dd(3.8, 15.6)	2.15 ddd(4.8, 12.0, 12.0)
			2.08 br dd(4.8, 15.6)	2.40 br dd(3.6, 13.8)
7	6.60 dd(4.8, 4.8)	4.12 br	4.91 br s	4.60 ddd(4.4, 12.0, 12.0)
8		3.82 br d(10.8)	2.82 br d(12.0)	3.21 dd(11.4, 11.4)
9	4.51 br dd(9.6, 9.6)	3.53 ddd(2.4, 11.4, 11.4)	3.45 ddd(6.0, 12.0, 12.0)	3.32 br dd(10.8, 10.8)
13	6.72 d(9.6)	6.74 d(7.8)	6.96 d(7.8)	6.90 d(7.8)
14	7.12 dd(9.6, 9.0)	7.13 dd(7.8, 7.8)	7.47 dd(7.8, 7.8)	7.43 dd(7.8, 7.8)
15	6.68 d(9.0)	6.68 d(7.8)	6.91 d(7.8)	6.90 d(7.8)
17	2.02 s	2.19 s	2.55 s	2.52 s
7-OH		5.15 s		
12-OH		10.13 s		

Table 2-6-28: ^{1}H NMR spectroscopic data of indolizidine alkaloids **2-6-69**~**2-6-73**.

H	2-6-69 · DCl	2-6-70	2-6-71	2-6-72	2-6-73
1	α 2.20 m, β 1.90 m	7.19 d(4.0)	2.40 m	3.50 t(7.7)	3.50 t(7.7)
2	α 2.0 m, β 1.95 m	6.33 d(4.0)	(ov)	2.50~2.60 m	2.50~2.60 m
3	α 3.31 m, β 3.05 m		(ov)	4.90 t(7.9)	4.90 t(7.9)
5	α 3.89 d(13.9), β 3.68 d(13.9)	3.95 t(6.0)	ax 3.40 d(18.3) eq 4.06 d(18.3)	8.65 s	8.65 s

Table 2-6-28 (continued)

H	2-6-69 · DCl	2-6-70	2-6-71	2-6-72	2-6-73
6		1.96 m			
7	6.71 d(11.3*ca*.1)	2.48 t(6.5)	5.46 s	8.20 s	8.20 s
8	5.72 dt(11.3)		–		
8a	4.25 br s				
9	5.40 d(10.1)		–		
10	2.68 m	4.43 s			
11	1.50 m	3.39 t(7.2)			
12	1.38 m	1.50 m			
13	3.79 m	1.31 m			
14	1.15 d(6.3)	0.82 t(7.4)			
15	1.03 d(6.6)				
2′			3.19~3.25 m	3.85~3.95 m	3.24~3.30 m
3′			3.83 br s	(ov)	3.85 br s
4′			1.71~1.80 m	2.63~2.77 m	(ov)
5′			1.55~1.65 m	(ov)	(ov)
6′			3.00~3.08 m	4.05~4.10 m	3.05~3.15 m
7″			1.91~1.99 m	(ov)	(ov)
10″			2.05 t(7.3)	2.80 t(7.3)	2.80 t(7.3)
1‴			1.91~1.99 m	(ov)	(ov)
10‴			(ov)	2.80 t(7.3)	2.80 t(7.3)
2⁗			3.19~3.25 m	3.24~3.30 m	3.85~3.95 m
3⁗			3.83 br s	3.85 br s	
4⁗			1.71~1.80 m	(ov)	2.63~2.77 m
5⁗			1.55~1.65 m	(ov)	(ov)
6⁗			3.00~3.08 m	3.05~3.15 m	4.05~4.10 m

Table 2-6-29: ^{1}H NMR spectroscopic data of indolizidine alkaloids **2-6-74**~**2-6-76**.

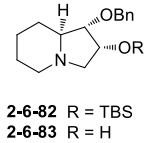
H	2-6-74	2-6-75	2-6-76
1	3.90 br t(9.7) 4.04 ddd(10.8, 10.8, 10.8)	3.59 m	3.50 t(7.0)
2	2.12 m, 2.38 m	4.73 m	4.36 q(6.0)
3	2.27 ddd(5.0, 11.5, 15.0) 2.67 ddd(4.6, 11.5, 15.0)	3.59 m	3.45 dd(6.0, 10.0) 2.41 dd(5.5, 10.0)
5			3.60 d(13.5), 2.91 dt(2.0, 13.5)
6	2.42 d(10.9)		
7	3.23 br t(10.9)		
8	3.79 br s	2.67 dd(5.7, 16.6), 2.60 dd(13.6, 16.6)	2.54 ddd(1.5, 4.0, 17.5), 2.23 m
8a		4.11 m	2.64 m
9	3.56 m		
10	1.42 m, 2.57 dt(14.0, 3.3)		
11	1.82 dd(1.5, 12.7), 2.46 m		
13	3.59 br s		
14	1.39 m, 1.84 dd(1.4, 12.9)		
15	2.23 m		

Table 2-6-29 (continued)

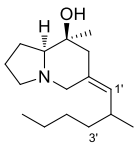
H	2-6-74	2-6-75	2-6-76
16	1.00 d(7.0)		
OAc		2.26 s	
OBn		4.73 d(11.8), 4.43 d(11.8)	4.77 d(12.0), 4.47 d(12.0)
TBS		0.90 s, 0.12 s, 0.11 s	0.92 s(*t*-Bu)
			0.11 s, 0.10 s($SiMe_2$)
OH			11.98 s
OMe			3.75 s

Table 2-6-30: ^{1}H NMR spectroscopic data of indolizidine alkaloids **2-6-77**~**2-6-79**.

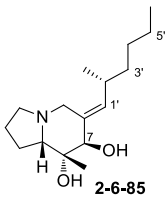
H	2-6-77	2-6-78	2-6-79($CDCl_3$)	2-6-79(C_6D_6)
1	3.66 m	3.73 m	3.44 t(7.5)	3.34 m
2	4.42 q(6.0)	4.35 q(6.5)	4.34 q(6.5)	4.25 m
3	3.41 dd(6.5, 9.5)	3.36 m	3.34 dd(6.5, 9.5)	3.29 m
	2.44 m	2.35 dd(6.0, 9.5)	2.25 dd(6.0, 9.5)	2.30 dd(5.5, 9.0)
5	3.05 dd(7.0, 11.0)	3.01 dd(3.5, 10.0)	3.07 dd(10.5, 17.0)	2.96 dd(4.5, 11.0)
	2.44 m	2.53 d(2.5)	2.05 m	1.93 t(11.0)
6	2.61 dd(3.5, 12.0)	3.36 m	2.05 m	2.20 m
	2.52 dd(6.5, 12.0)			
7		4.39 m	4.69 m	4.93 dt(5.0, 11.0)
8	3.17 m	2.63 m	2.34 m	2.61 ddd
		1.24 m	1.25 m	(2.5, 5.0, 11.5)
				1.40 m
8a	3.27 m	2.15 dt(3.0, 13.5)	2.34 m	2.50 ddd
				(2.5, 8.0, 11.5)
1′			4.06 dd(3.0, 11.5)	4.15 dd(3.5, 11.5)
			3.98 dd(6.0, 11.5)	4.11 dd(6.5, 11.5)
OAc			2.05 s, 2.03 s	1.78 s, 1.76 s
OBn	4.74 d(11.0)	4.75 d(12.0)	4.74 d(12.0)	4.75 d(12.0)
	4.34 d(11.0)	4.44 d(12.0)	4.43 d(12.0)	4.43 d(12.0)
TBS	0.90 s(*t*-Bu)	0.91 s(*t*-Bu)	0.91 s (*t*-Bu)	1.07 s (*t*-Bu)
	0.11 s, 0.09 s($SiMe_2$)	0.09 s, 0.08 s($SiMe_2$)	0.08 s, 0.09 s($SiMe_2$)	0.13 s, 0.15 s($SiMe_2$)
OMe	3.57 s	3.71 s		



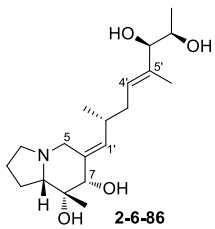
2-6-82 R = TBS
2-6-83 R = H



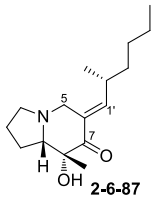
2-6-84



2-6-85



2-6-86



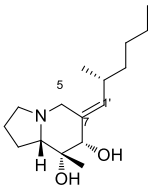
2-6-87

Table 2-6-31: ^{1}H NMR spectroscopic data of indolizidine alkaloids **2-6-80**~**2-6-83**.

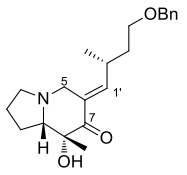
H	2-6-80	2-6-81	2-6-82	2-6-83
1	3.52 t(7.0)	3.44 dd(6.6, 7.9)	3.42 m	3.52 dd(7.0, 8.0)
2	4.42 q(6.5)	4.23 m(5.4, 6.5, 6.6)	4.31 dd(6.5, 12.0)	4.19 m
3	3.41 dd(6.5, 9.5)	3.25 dd(6.5, 9.0)	3.34 dd(6.5, 9.5)	3.45 dd(6.5, 10.0)
	2.35 dd(7.0, 9.5)	2.26 dd(3.4, 9.0)	2.22 dd(6.0, 9.5)	2.18 dd(5.0, 10.0)
5	2.33 m, 3.18 m	2.73 m, 1.95 m	2.95 m, 2.05 m	2.96 m, 2.03 m
6	2.47 m	1.70 m, 1.50 m	1.62 m, 1.47 m	1.62 m, 1.47 m
7		4.90 m	1.77 m, 1.26 m	1.78 m, 1.21 m
8	2.61 m	2.46 m	1.96 m	1.93 m, 1.21 m
	2.13 dd(11.5, 13.5)	1.95 m	1.14 m	
8a	2.61 m	2.46 m	2.12 m	2.03 m
OAc		1.78 s		
OBn	4.76 d(12.0)	4.75 d(11.9)	4.77 d(12.0)4.44 d(12.0)	4.67 d(11.5)
	4.43 d(12.0)	4.41 d(11.9)		4.59 d(11.5)
TBS	0.92 s(*t*-Bu)	1.04 s, 0.12 s	0.91 s(*t*-Bu)	
	0.10 s, 0.12 s	0.10 s	0.09 s,0.10 s($SiMe_2$)	

Table 2-6-32: ^{1}H NMR spectroscopic data of indolizidine alkaloids **2-6-84**~**2-6-86**.

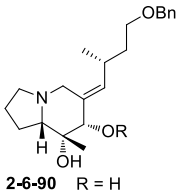
H	2-6-84	2-6-85	2-6-86
1	1.66~1.78 m	1.6~1.8 m	1.6~1.8 m
2	1.66~1.78 m	1.6~1.8 m	1.6~1.8 m
3	α 2.17 m, β 3.08 m	3.1 m, 2.2~2.4 m	3.07 m, 1.9~2.3 m
5	α 2.35 d(12), β 3.79 d(12)	3.60 d(12.5), 2.71 br d(12)	3.79 d(12), 2.39 dd(1.8, 12)
7	2.14 br s	3.71 s	3.66 dd(1.1, 1.6)
8		2.45~2.50 m	
8a	1.98 m		1.9~2.3 m
1′	5.04 d(9.5)	5.33 dd(9.5, 1.5)	5.52 ddd(1.9, 1.9, 10)
2′	2.37 m	2.37~2.41 m	2.5 m
3′	1.11~1.34 m	1.1~1.4 m	1.9~2.3 m
4′	1.11~1.34 m	1.1~1.4 m	5.39 dt(1.1, 7.6)
5′	1.11~1.34 m	1.1~1.4 m	
6′	0.88 t(7)	0.87 t(7.2)	3.71 d(6.9)
7′			3.78 dd(6.9, 13)
8′			1.11 d(6.1)
8-OH	1.11~1.34 m	2.91 br s	
8-Me	1.13 s	1.21 s	1.22 s
2′- Me	0.97 d(6.5)	0.97 d(6.5)	1.01 d(6.6)
5′- Me			1.60 d(1.2)



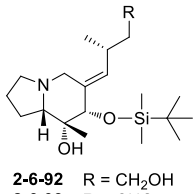
2-6-88



2-6-89



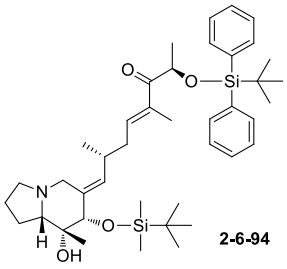
2-6-90 R = H
2-6-91 R =TBS



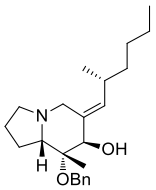
2-6-92 R = CH_2OH
2-6-93 R = CHO

Table 2-6-33: ^{1}H NMR spectroscopic data of indolizidine alkaloids **2-6-87**~**2-6-89**.

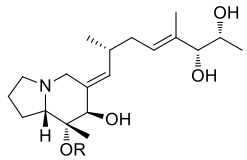
H	2-6-87	2-6-88	2-6-89
1	1.8~2.0 m	1.7~1.85 m	1.5~2.0 m
2	1.8~2.0 m	1.7~1.85 m	1.5~2.0 m
3	3.25~3.35 m, 2.25~2.5 m	3.0~3.1 m, 2.3~2.45 m	3.18 m, 2.4~2.2 m
5	4.03 dd(1.6, 14), 2.97 dd(2.6, 14)	3.80 d(12), 2.38 ddd(1.2,1.2,1.2)	4.02 dd(1.4, 14), 2.94 dd(2.6, 14)
7		3.67 br s	
8a	2.25~2.5 m	2.0~2.1 m	2.2~2.4 m
1′	6.52 ddd(1.6, 2.6, 11)	5.47 ddd(1.8,1.8,9.9)	6.48 ddd(1.6, 2.5, 11)
2′	2.5~2.25 m	2.15~2.3 m	2.64 m
3′	1.1~1.4 m	1.1~1.4 m	1.5~2.0 m
4′	1.1~1.4 m	1.1~1.4 m	3.3~3.5 m
5′	1.1~1.4 m	1.1~1.4 m	
6′	0.87 t(6.8)	0.86 t(7.1)	
8-OH	3.7 br s	2.91 br s	3.7 br s
8-Me	1.26 s	1.22 s	1.25 s
2′- Me	1.02 d(6.6)	0.98 d(6.6)	1.04 d(6.7)
1″			4.45 s
Ar-H			7.3 m



2-6-94



2-6-95



2-6-96 R = Bn
2-6-97 R = H

Table 2-6-34: ^{1}H NMR spectroscopic data of indolizidine alkaloids **2-6-90**~**2-6-94**.

H	2-6-90	2-6-91	2-6-92	2-6-93	2-6-94
1	1.6~1.85 m	1.6~2.0 m	1.4~1.9 m	1.6~1.9 m	1.6~1.9 m
2	1.6~1.85 m	1.6~2.0 m	1.4~1.9 m	1.6~1.9 m	1.6~1.9 m

Table 2-6-34 (continued)

H	2-6-90	2-6-91	2-6-92	2-6-93	2-6-94
3	3.03 m	2.93 m	3.06 m	3.1 m	3.1 m
	1.9~2.2 m	2.0~2.2 m	2.0~2.3 m	1.9~2.3 m	2.00~2.4 m
5	3.83 d(12)	3.83 d(14)	3.82 d(15)	3.87 d(12)	3.70 d(11)
	2.30 ddd(1.3, 1.3, 12)	2.27 d(12)	2.42 br d(15)	1.9~2.3 m	2.00~2.4 m
7	3.65 m	3.76 d(2.0)	3.80 s	3.76 d(2.1)	3.74 d(2.0)
8a	1.9~2.2 m	2.0~2.2 m	2.0~2.3 m	1.9~2.3 m	2.00~2.4 m
1′	5.46 ddd(1.8, 1.8, 10)	5.41 ddd(1.0, 1.8, 10)	5.44 dd(1.5, 10)	5.46 ddd(1.1, 2.0, 10)	5.46 dd(1.3, 9.3)
2′	2.68 m	2.70 m	2.7 m	3.1 m	2.5 m
3′	1.6~1.85 m	1.6~2.0 m	1.4~1.9 m	2.39 ddd(2.0, 3.5, 7.6)	2.00~2.4 m
4′	3.3~3.5 m	3.3~3.4 m	3.6~3.8 m		6.23 dt(1.2, 7.5)
7′					4.87 q(6.7)
8′					1.0 m
8-OH	2.38 br s		2.28 s		
8-Me	1.21 s	1.14 s	1.15 s	1.14 s	1.19 s
2′-Me	1.01 d(6.7)	1.03 d(6.6)	1.05 d(6.5)	1.10 d(6.7)	1.0 m
5′-Me					1.37 d(6.7)
1″	4.45 s	4.46 s			
Ar-H	7.3 m	7.2~7.3 m			7.3~7.8 m
^{t}Bu		0.96 s	0.97 s	0.97 s	1.19 s
Si-Me		0.16 s, 0.08 s	0.09 s, 0.03 s	0.08 s, 0.01 s	0.11 s, 0.06 s
CHO				9.68 t(2.1)	

Table 2-6-35: ^{1}H NMR spectroscopic data of indolizidine alkaloids **2-6-95**~**2-6-97**.

H	2-6-95	2-6-96	2-6-97
1	1.62~2.03 m	1.65~1.92 m	1.65~1.75 m
2	1.62~2.03 m	1.65~1.92 m	1.65~1.75 m
3	ax 2.15 m, eq 3.12 t(8.3)	2.34 m, 3.00 m	2.26 m, 3.02 dd(6.3, 2.3)
5ax	2.75 d(12.4)	2.85 d(12.3)	2.68 dd(12.1, 1.1)
5eq	3.70 d(12.4)	3.51 d(12.3)	3.56 d(12.1)
7	4.06 s	4.07 s	3.66 s
8a	2.42~2.47 m	2.65 m	2.45~2.58 m
1′	5.25 d(9.5)	5.22 d(10.0)	5.24 dd(10.1, 1.1)
2′	2.42~2.47 m	2.57 m	2.45~2.58 m
3′	1.12~1.37 m	1.94~2.10 m	1.96~2.09 m
4′	1.12~1.37 m	5.37 br dd(9.3, 6.0)	5.32 br t(7.7)
5′	1.12~1.37 m		
6′	0.87 t(7.1)	3.66 d(6.8)	3.70 d(6.2)
7′		3.75 m	3.77 quint(6.3)
8′		1.11 d(6.3)	1.12 d(6.3)
2′-Me	0.99 d(6.5)	1.07 d(6.5)	1.05 d(6.5)

Table 2-6-35 (continued)

H	2-6-95	2-6-96	2-6-97
8-Me	1.25 s	1.28 s	1.19 s
5′-Me		1.56 s	1.55 s
1″	4.57 ABq(12.7), 4.58 ABq(12.7)	4.54 s	
Ar-H	7.17~7.32 m	7.20~7.33 m	

Bibliography

[129] Cardona F, Moreno G, Guarna F, et al. J Org Chem, 2005, 70: 6552.
[130] Davis AS, Pyne SG, Skelton BW, et al. J Org Chem, 2004, 69: 3139.
[131] Karanjule NS, Markad SD, Sharma T, et al. J Org Chem, 2005, 70: 1356.
[132] Carmona AT, Fuentes J, Robina I. Tetrahedron Lett, 2002, 43: 8543.
[133] Carmona AT, Fuentes J, Robina I, et al. J Org Chem, 2003, 68: 3874.
[134] Rabiczko J, Urbańczyk-Lipkowska Z, Chmielewski M. Tetrahedron, 2002, 58: 1433.
[135] Razavi H, Polt R. J Org Chem, 2000, 65: 5693.
[136] Patil NT, Tilekar JN, Dhavale DD. Tetrahedron Lett, 2001, 42: 747.
[137] Klausmeyer P, Chmurny GN, McCloud TG, et al. J Nat Prod, 2004, 67: 1732.
[138] Pu XT, Ma DW. J Org Chem, 2003, 68: 4400.
[139] Stærk D, Lykkeberg AK, Christensen J, et al. J Nat Prod, 2002, 65: 1299.
[140] Stærk D, Christensen J, Lemmich E, et al. J Nat Prod, 2000, 63: 1584.
[141] Ali M, Bhutani KK. Phytochemistry, 1989, 28: 3513.
[142] Huang XS, Gao S, Fan LH, et al. Planta Med, 2004, 70: 441.
[143] Damu AG, Kuo PC, Shi LS, et al. J Nat Prod, 2005, 68: 1071.
[144] Lee SK, Nam KA, Heo YH. Planta Med, 2003, 69: 21.
[145] Wu PL, Rao KV, Su CH, et al. Heterocycles, 2002, 57: 2401.
[146] Zhen YY, Huang XS, Yu DQ, et al. Acta Bot Sin, 2002, 44: 349.
[147] Abe F, Yamauchi T, Honda K, et al. Phytochemistry, 2001, 56: 697.
[148] Komatsu H, Watanabe M, Ohyama M, et al. J Med Chem, 2001, 44: 1833.
[149] Subramaniam G, Ang KKH, Ng S, et al. Phytochem Lett, 2009, 2: 88.
[150] An TY, Huang RQ, Yang Z, et al. Phytochemistry, 2001, 58: 1267.
[151] Morita H, Arisaka M, Yoshida N, et al. J Org Chem, 2000, 65: 6241.
[152] Livant PD, Beutler JA. Tetrahedron, 1987, 43: 2915.
[153] Katavic PL, Venables DA, Rali T, et al. J Nat Prod, 2007, 70: 872.
[154] Andriamaharavo NR, Andriantsiferana M, Stevenson PA, et al. J Nat Prod, 2005, 68: 1743.
[155] Wang YF, Lu CH, Lai GF, et al. Planta Med, 2003, 69: 1066.
[156] Nakano H, Nakajima E, Hiradate S, et al. Phytochemistry, 2004, 65: 587.
[157] Fox DNA, Lathbury D, Mahon MF, et al. J Am Chem Soc, 1991, 113: 2652.
[158] Goldstein SW, Overman LE, Rabinowitz MH. J Org Chem, 1992, 57: 1179.
[159] Aoyagi S, Wang TC, Kibayashi C. J Am Chem Soc, 1993, 115: 11393.

2.7 Quinolizidine alkaloids

2.7.1 Lupinine-type alkaloids

Table 2-7-1: Cos, MFs, and TSs of lupinine-type alkaloids **2-7-1**~**2-7-13**.

No.	Compounds	MFs	Test solvents	References
2-7-1	cermizine C	$C_{11}H_{21}N$	CD_3OD	[160]
2-7-2	senepodine G	$C_{11}H_{20}N$	CD_3OD	[160]
2-7-3	senepodine H	$C_{14}H_{26}NO$	CD_3OD	[160]
2-7-4	(+)-*epi*-lupinine acetate *N*-oxide	$C_{12}H_{21}NO_3$	$CDCl_3$	[161]
2-7-5	jussiaeiine B	$C_{16}H_{24}N_2O_2$	$CDCl_3$	[162]
2-7-6	jussiaeiine C	$C_{16}H_{24}N_2O_3$	$CDCl_3$	[162]
2-7-7	jussiaeiine D	$C_{16}H_{24}N_2O_3$	$CDCl_3$	[162]
2-7-8	3-(4-hydroxyphenyl)-4-(3-methoxy-4-hydroxy-phenyl)-3,4-dehydroquinolizidine	$C_{22}H_{25}NO_3$	C_5D_5N	[163]
2-7-9	cermizine D	$C_{16}H_{30}N_2$	CD_3OD	[160]
2-7-10	hydrachine A	$C_{17}H_{19}N_3O_3$	$CDCl_3$	[164]
2-7-11	senepodine A	$C_{23}H_{40}N_2$	CD_3OD	[165]
2-7-12	senepodine C	$C_{22}H_{38}N_2$	CD_3OD	[165]
2-7-13	senepodine B	$C_{23}H_{39}N_2$	CD_3OD	[165]

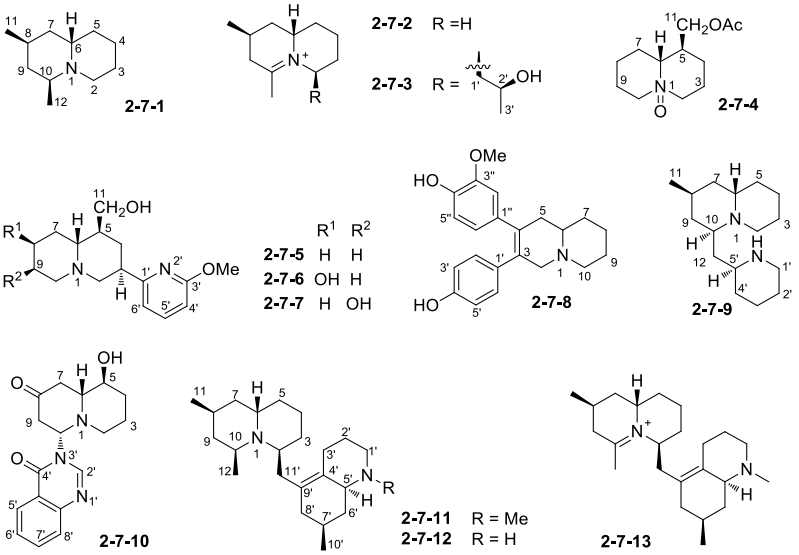


Table 2-7-2: ^{1}H NMR spectroscopic data of lupinine-type alkaloids **2-7-1**~**2-7-4**.

H	2-7-1	2-7-2	2-7-3	2-7-4
2	3.08 ddd(13.7, 13.5, 2.7) 3.65 br d(13.7)	3.52 br t(12.9) 4.50 br d(12.9)	4.90 ddd (10.5, 4.9, 4.4)	ax 3.07 m eq 3.36 t(13.5)

Table 2-7-2 (continued)

H	2-7-1	2-7-2	2-7-3	2-7-4
3	1.69 m, 1.79 m	1.75 m, 2.01 m	1.89 m, 1.98 m	–
4	1.65 m, 1.94 m	1.80 m, 1.94 m	1.75 m, 2.01 m	–
5	1.62 m, 2.17 br q(13.3)	1.85 m, 1.91 m	1.89 m	2.44 m
6	3.60 br d(13.2)	3.96 m	4.17 m	2.86 dt(11.4, 2.3)
7	1.56 ddd(14.1, 14.1, 5.1)	1.76 m	1.72 m	–
	1.79 m	1.82 m	1.84 br d(14.1)	
8	1.96 m	2.02 m	2.02 m	–
9	1.21 q(13.2)	2.45 m	2.52 dd(20.8, 9.9)	–
	1.95 m	2.98 dd(19.9, 3.8)	3.04 dd(20.8, 2.9)	
10	3.82 m			ax 3.07 m
				eq 3.36 t(13.5)
11	0.95 d(6.2)	1.04 d(6.7)	1.04 d(6.6)	4.06 dd(11.5, 4.4)
				4.02 dd(11.5, 3.6)
12	1.31 d(6.3)	2.44 s	2.58 s	
1′			1.65 ddd(14.9, 10.6, 4.4)	
			2.39 ddd(14.9, 10.5, 2.6)	
2′			3.54 ddq(10.6, 2.6, 6.1)	
3′			1.24 d(6.1)	
OAc				2.06 s

Table 2-7-3: ^{1}H NMR spectroscopic data of lupinine-type alkaloids **2-7-5**~**2-7-9**.

H	2-7-5	2-7-6	2-7-7	2-7-8	2-7-9
2	*α* 2.97 br d(11.2)	3.02~3.08 m	*α* 2.98~3.05 m	3.76 d(11.6)	3.14 ddd(12.8, 12.4, 3.2)
	β 2.26 br t(11.0)	2.30~2.40 m	*β* 2.34 br t(12.2)	3.11 d(11.6)	3.71 br d(12.8)
3	3.02 m	3.02~3.08 m	2.98~3.05 m		1.73 m, 1.82 m
4	*α* 2.07~2.12 m	*α* 1.99 br d(12.8)	*α* 2.06 br d(12.0)		1.65 m
	β 1.61~1.67 m	*β* 1.81 q(12.8)	*β* 1.65 q(12.0)		1.93 m
5	1.61~1.67 m	1.47~1.55 m	1.56 br s	2.65 m	1.69 m
				2.52 m	2.17 br q(13.0)
6	1.78 br s	2.40, 2.30 m	1.76 q(8.7)	2.21 m	3.68 br d(13.0)
7	*α* 1.19~1.28 m	*α* 1.47~1.55 m	*α* 1.21~1.33 m	1.31 m	1.60 m
	β 1.95 br d(9.7)	*β* 2.13 br d(14.0)	*β* 2.06 br d(11.9)		1.80 m
8	*α* 1.78 br s	4.20 br s	2.06 br d(11.9)	1.31 m	1.97 m
	β 1.19~1.28 m	2.30~2.40 m(OH)	1.21~1.33 m		
9	1.61~1.67 m	*α* 1.92 br t(13.6)	3.81 m	1.69 m	1.17 q(12.8)
		β 1.72 br d(14.0)	1.86 br s(OH)		2.22 br d(13.4)
10	*α* 2.86 br d(11.2)	*α* 2.68 br d(18.8)	*α* 2.98~3.05 m	3.03 d(11.6)	3.96 br t(10.7)
	β 2.07~2.12 m	*β* 2.60 br t(11.4)	*β* 1.98 br t(10.4)	2.00 d(11.6)	
11	3.71 dd(2.7, 10.8)	3.77 dd(14.6, 11.4)	3.73 br d(8.4)		0.98 d(6.3)
	3.61 dd(5.1, 10.8)	3.61 dd(2.6, 11.4)	3.67 dd(5.3, 10.3)		
	2.26 br t(OH)	2.30~2.40 m(OH)	1.86 br s(OH)		

Table 2-7-3 (continued)

H	2-7-5	2-7-6	2-7-7	2-7-8	2-7-9
12					1.66 m, 2.32 m
1′					3.05 ddd(12.2, 11.8, 2.3) 3.42 br d(12.2)
2′	3.87 s(OMe)	3.89 s(OMe)	3.90 s(OMe)	7.25 d(8)	1.69 m, 1.93 m
3′	6.52 d(8.2)	6.55 d(8.4)	6.55 d(8.2)	7.07 d(8)	1.70 m, 1.91 m
4′	7.43 t(7.7)	7.46 t(7.8)	7.47 t(7.7)		1.57 m, 2.01 m
5′	6.69 d(7.2)	6.73 d(7.2)	6.72 d(7.2)	7.07 d(8)	3.30 m
6′				7.25 d(8)	
2″				6.92 d(1.6)	
3″				3.59 s(OMe)	
5″				7.12 d(8.4)	
6″				6.94 dd(8.4, 1.6)	

Table 2-7-4: ^{1}H NMR spectroscopic data of lupinine-type alkaloids **2-7-10**~**2-7-13**.

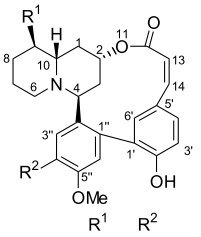
H	2-7-10	2-7-11	2-7-12	2-7-13
2	ax 2.93 br dd(13.0, 4.0) eq 2.18 br dt(13.0, 4.0)	3.48 m	3.79 m	4.70 m
3	ax 1.80 m, eq 1.74 m	1.34 br d(12.6), 1.70 m	1.55 br d(10.6), 1.84 m	1.87 m
4	ax 1.29 m, eq 2.07 m	1.68 m, 1.77 m	1.70 m, 1.83 m	1.72 m, 1.87 m
5	3.46 ddd(11.0, 9.0, 5.0)	1.28 br d(15.5) 2.04 ddd(15.5, 13.1, 4.1)	1.60 br d(16.1) 2.17 m	1.88 m
6	2.28 ddd(11.2, 9.0, 3.2)	3.46 m	3.85 br d(13.3)	4.28 m
7	ax 2.50 dd(15.2, 11.2) eq 3.18 dd(15.2, 3.2)	1.47 ddd(13.1, 12.8, 4.9) 1.59 br d(12.8)	1.60 m, 1.70 m	1.73 m, 1.87 m
8		1.82 m	1.98 m	1.96 m
9	ax 2.84 dd(11.2, 10.4) eq 3.34 dd(10.4, 6.4)	1.13 ddd(12.5, 12.5, 12.5) 1.72 m	1.38 m, 1.93 m	2.54 m, 3.03 m
10	5.70 dd(11.2, 6.4)	3.44 m	3.89 m	
11		0.90 d(6.5)	0.94 d(6.3)	1.04 d(6.3)
12		1.18 d(6.1)	1.38 d(6.3)	2.49 s
1′		2.31 m 2.92 br d(11.8)	3.09 ddd(12.9, 12.8, 3.0) 3.38 br d(12.8)	3.19 m, 3.53 m
2′	7.88 s	1.54 m, 1.73 m	1.66 m, 2.00 m	1.74 m, 2.02 m
3′		1.75 m, 2.86 br d(13.6)	2.93 br d(14.5), 2.05 m	2.02 m, 2.98 m
5′	8.28 dd(8.0, 1.6)	2.63 dd(9.2, 8.0)	3.80 dd(9.2, 8.0)	3.82 m
6′	7.50 ddd(8.0, 7.2, 1.6)	1.04 ddd(12.5, 12.5, 9.2) 2.09 m	1.28 ddd(12.3, 12.3, 9.2) 2.11 m	1.35 m 2.34 m
7′	7.80 ddd(8.4, 7.2, 1.6)	1.58 br d(14.0)	1.75 br d(13.8)	1.69 m
8′	7.71 dd(8.4, 1.6)	1.79 m 1.92 br d(16.1)	1.92 m 2.02 br d(16.8)	1.96 m 2.09 br d(15.6)

Table 2-7-4 (continued)

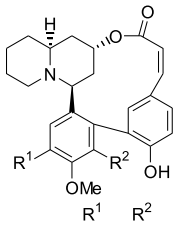
H	2-7-10	2-7-11	2-7-12	2-7-13
10′		0.99 d(6.6)	1.03 d(6.4)	1.07 d(6.4)
11′		2.13 br d(12.5) 3.03 dd(12.5, 12.2)	2.37 br d(12.1) 3.17 dd(12.5, 12.2)	2.43 br d(11.5) 3.11 dd(11.5, 11.1)
NMe		2.30 s		2.89 s

Table 2-7-5: Cos, MFs, and TSs of lupinine-type alkaloids **2-7-14**~**2-7-24**.

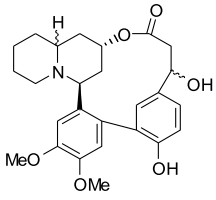
No.	Compounds	MFs	Test solvents	References
2-7-14	9β-hydroxyvertine	$C_{26}H_{29}NO_6$	CD_3OD	[166]
2-7-15	lythrine	$C_{26}H_{29}NO_5$	$CDCl_3$	[166]
2-7-16	dehydrodecodine	$C_{25}H_{27}NO_5$	CD_3OD	[166]
2-7-17	vertine	$C_{26}H_{29}NO_5$	CD_3OD	[166]
2-7-18	lyfoline	$C_{25}H_{27}NO_5$	CD_3OD	[166]
			$CDCl_3$	[167]
2-7-19	*epi*-lyfoline	$C_{25}H_{27}NO_5$	CD_3OD	[166]
	10-*epi*-lyfoline	$C_{25}H_{27}NO_5$	$CDCl_3$	[167]
2-7-20	lythridine	$C_{26}H_{31}NO_6$	CD_3OD	[166]
2-7-21	heimidine	$C_{26}H_{31}NO_6$	CD_3OD	[166]
2-7-22	5-*epi*-dihydrolyfoline	$C_{25}H_{29}NO_5$	CD_3OD-$CDCl_3$	[168]
2-7-23	dihydrolyfoline	$C_{25}H_{29}NO_5$	$CDCl_3$	[168]
2-7-24	lagerine	$C_{25}H_{29}NO_5$	$CDCl_3$	[168]



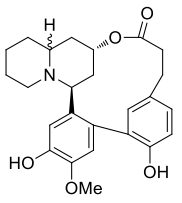
	R^1	R^2
2-7-14	OH	OMe
2-7-17	H	OMe
2-7-19	H	OH



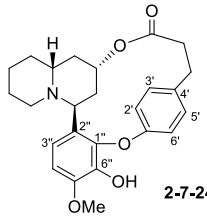
	R^1	R^2
2-7-15	OMe	H
2-7-16	H	OH
2-7-18	OH	H



2-7-20 10α-H
2-7-21 10β-H



2-7-22 10β-H
2-7-23 10α-H



2-7-24

Table 2-7-6: ^{1}H NMR spectroscopic data of lupinine-type alkaloids **2-7-14**~**2-7-17** and **2-7-20**.

H	2-7-14	2-7-15	2-7-16	2-7-17	2-7-20
1	1.85~1.96	1.73	1.81	2.15~2.18	1.69
	2.49 d(15.0)	1.69		1.66~1.70	
2	5.30 br s	5.32 br s	5.22 br s	5.29 br s	5.01 br s
3	1.97 t(13.0)	2.23 d(14.8)	2.00	2.21~2.25	2.28 d(13.2)
	2.18 d(14.5)	2.04 t(14.0)		1.97~2.03	1.97
4	4.57 d(11.5)	3.65 d(10.8)	4.13 d(9.6)	4.73 d(11.2)	3.12 d(10.4)
6	2.75 d(13.5)	2.66 d(11.2)	2.72 d(9.2)	2.84 d(13.4)	2.68 d(12.8)
	2.29 t(12.5)	1.13	1.37~1.39	2.41 d(11.2)	
7	0.93 d(14.0)	1.40	1.37~1.39	0.65~0.78	1.46
	0.78 d(13.5)				
8	1.61 d(10.0)	1.60	1.49~1.58	1.33~1.35	1.67
	1.00 d(13.0)	1.57			
9	3.52	1.45, 1.40	1.49~1.58	1.02~1.05	1.51, 1.44
10	2.63	1.95	2.29 d(12.1)	3.09	3.11
13	5.76 d(12.5)	5.83 d(12.2)	5.74 d(12.1)	5.79 d(12.4)	2.81, 2.40
14	6.74 d(12.5)	6.76 d(12.2)	6.78 d(12.1)	6.79 d(12.4)	5.01 dd(2.4)
3′	6.89 d(8.5)	6.97 d(8.4)	6.89 d(8.8)	6.93 d(8.8)	6.97 d(8.8)
4′	7.10 d(8.0)	7.16 dd(8.2, 1.8)	7.14 d(8.0)	7.16 dd(8.2, 1.8)	7.42 dd(8.4, 2.0)
6′	6.94	7.10 d(2.0)	6.86	7.02 d(2.4)	6.58 d(2.4)
3″	7.02 s	7.04 s	6.93	7.08 s	7.17 s
4″	3.78 s(OMe)	3.85 s(OMe)	6.89	3.82 s(OMe)	3.82 s(OMe)
5″	3.82 s(OMe)	3.89 s(OMe)	3.82 s(OMe)	3.86 s(OMe)	3.87 s(OMe)
6″	7.07 s	6.92 s		7.12 s	6.92 s

Table 2-7-7: ^{1}H NMR spectroscopic data of lupinine-type alkaloids **2-7-18** and **2-7-19**.

H	2-7-18 (CD_3OD)	2-7-18 ($CDCl_3$)	2-7-19 (CD_3OD)	2-7-19 ($CDCl_3$)
1	1.69 d(5.2)	ax 1.72 br t, eq 1.75 br d	2.21~2.29, 1.66~1.72	ax 1.67 m, eq 2.27 m
2	5.25 br s	5.34 m	5.28 br s	5.36 br m
3	2.18 d(14.8)	ax 2.03 td	2.21~2.29	ax 2.06 m
	2.04 d(13.2)	eq 2.24 br d	2.09~2.12	eq 2.27 br d
4	3.87 d(11.2)	3.61 dd(10.0, 1.1)	4.80 d(10.8)	4.56 d(11.1)
6	2.69 d(11.6)	ax 1.14 m	2.91 d(13.6)	ax 2.47 td
	1.21	eq 2.69	2.49	eq 2.90 br d
7	1.54 d(11.2)	ax 1.40, eq 1.48	1.17, 1.68	ax 0.57 tt, eq 0.83 d
8	1.37	ax 1.18, eq 1.61	1.32~1.43	ax 1.11 m, eq 1.28 m
9	1.37	ax 1.27~1.30 dd	1.32~1.65	ax 1.58
	1.21	eq 1.40~1.43 m		eq 1.42
10	1.99	1.96 br t	3.18~3.20	3.21 br
13	5.71 d(12.4)	5.87 d(12.5)	5.78 d(12.8)	5.86 d(12.5)
14	6.64 d(12.4)	6.79 d(12.5)	6.80 d(12.8)	6.80 d(12.5)
3′	6.88 d(8.8)	7.00 d(8.3)	6.94	7.01 d(8.3)
4′	7.05 d(8.4)	7.19 dd(8.3, 2.2)	7.15 dd(8.4, 2.0)	7.21 dd(8.3, 2.1)
6′	6.90 s	7.12 d(2.2)	7.08	7.15 d(2.1)

Table 2-7-7 (continued)

H	2-7-18 (CD_3OD)	2-7-18 ($CDCl_3$)	2-7-19 (CD_3OD)	2-7-19 ($CDCl_3$)
3″	7.02 s	7.18 s	7.09	7.23 s
5″	3.78 s(OMe)	3.90 s(OMe)	3.84 s(OMe)	3.91 s(OMe)
6″	6.95 s	6.93 s	6.98	6.96 s

Table 2-7-8: ^{1}H NMR spectroscopic data of lupinine-type alkaloids **2-7-21**~**2-7-24**.

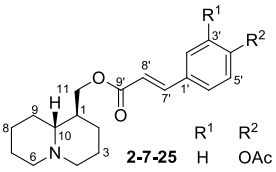
H	2-7-21	2-7-22	2-7-23	2-7-24
1	2.09~2.14	ax 1.99 ddd(15.0, 6.5, 3.5)	1.65	ax 1.99
	1.65~1.69	eq 1.57 br d(15.0)		eq 1.45 br d(15.5)
2	4.95	4.89 br s	4.97 br s	4.88 br s
3	2.33 d(13.2)	ax 1.66 br t(13.5)	ax 1.80 br t(13.5)	ax 1.57
	1.74~1.77	eq 2.33 br d(15.0)	eq 2.36 br d(15.0)	eq 1.37 br d(13.0)
4	4.13 d(11.2)	4.00 d(11.0)	2.99 d(11.0)	3.37 d(12.5)
6	2.90 d(14.4)	ax 2.29 br td(10.0, 4.0)	ax 1.21	ax 2.42 br d(13.5)
	2.44 d(10.8)	eq 2.81 br d(12.5)	eq 2.66 br d(11.0)	eq 2.56
7	0.82~0.86	0.82~0.78 m	1.44	ax 1.64 dt(13.5, 4.0)
				eq 1.08 br t(15.0)
8	1.52 d(10.4)	ax 1.14~1.11 m	ax 1.14	ax 1.37 br d(13.0)
	1.06	eq 1.44 br d(12.5)	eq 1.61	eq 1.86 br d(13.0)
9	1.79 d(12.4)	ax 1.66 br t(13.5)	ax 1.25	ax 1.75 dd(13.5, 4.0)
		eq 0.98 br d(13.5)	eq 1.47	eq 1.08 br t(15.0)
10	3.09	2.99 dd(13.5, 2.5)	1.97 dd(14.5, 9.0)	3.04
13	2.86 d(2.8)	2.54 ddd(12.5 5.5, 2.5)	2.61 ddd(12.5, 5.5, 2.5)	2.58 ddd(11.5, 4.5, 2.5)
	2.38 d(11.2)	2.19 td(13.0, 2.5)	2.28 td(13.0, 2.5)	2.28 td(13.0, 5.5)
14	5.05 dd,(10.8, 2.8)	3.00 td(13.5, 2.5)	3.14 td(13.5, 2.5)	3.04 ddd(13.0, 5.5, 3.0)
		2.71 ddd(13.5, 5.5, 2.5)	2.79 ddd(13.5, 5.5, 2.5)	2.85 td(13.5, 5.0)
2′				7.34 dd(8.0, 2.0)
3′	6.98 d(8.4)	6.82 d(8.0)	6.96 d(8.0)	7.26 dd(8.0, 2.0)
4′	7.42 dd(8.8, 2.0)	6.95 dd(8.0, 2.0)	7.08 dd(8.0, 2.0)	
5′				6.95 dd(8.0, 2.0)
6′	6.62 d(2.0)	6.69 d(2.0)	6.84 d(2.0)	6.51 dd(8.0, 2.0)
3″	7.09 s	6.86 s	7.17 s	6.91 d(8.0)
4″	3.84 s(OMe)			6.74 d(8.0)
5″	3.87 s(OMe)	3.77 s(OMe)	3.88 s(OMe)	3.93 s(OMe)
6″	6.96 s	6.80 s	6.83 s	

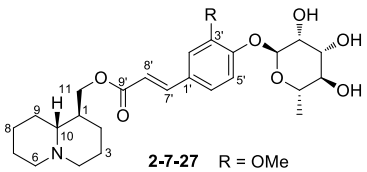
Table 2-7-9: Cos, MFs, and TSs of lupinine-type alkaloids **2-7-25**~**2-7-32**.

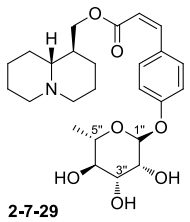
No.	Compounds	MFs	Test solvents	References
2-7-25	(+)-(*trans*-4′-acetoxycinnamoyl)*epi*lupinine	$C_{21}H_{27}NO_4$	$CDCl_3$	[161]
2-7-26	(+)-(*trans*-4′-hydroxy-3′-methoxy-cinnamoyl)*epi*lupinine	$C_{20}H_{27}NO_4$	$CDCl_3$	[169]

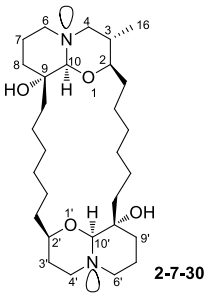
Table 2-7-9 (continued)

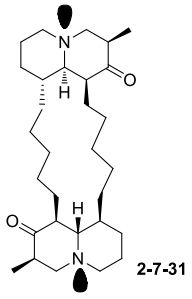
No.	Compounds	MFs	Test solvents	References
2-7-27	(–)-(*trans*-3′-methoxy-4′-*α*-L-rhamnosyloxycinnamoyl)-*epi*lupinine	$C_{26}H_{37}NO_8$	$CDCl_3$	[169]
2-7-28	(–)-(*trans*-4′-*α*-L-rhamnosyloxycinnamoyl)*epi*lupinine	$C_{25}H_{35}NO_7$	$CDCl_3$	[169]
2-7-29	(–)-(*cis*-4′-*α*-L-rhamnosyloxycinnamoyl)*epi*lupinine	$C_{25}H_{35}NO_7$	$CDCl_3$	[170]
2-7-30	3*α*-methylaraguspongine C	$C_{29}H_{52}N_2O_4$	$CDCl_3$	[171]
2-7-31	xestosin A	$C_{30}H_{50}N_2O_2$	$CDCl_3$	[172]
2-7-32	pictamine	$C_{20}H_{33}NO_2$	C_6D_6	[173]











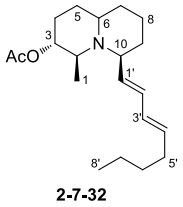


Table 2-7-10: ^{1}H NMR spectroscopic data of lupinine-type alkaloids **2-7-25**~**2-7-29**.

H	2-7-25	2-7-26	2-7-27	2-7-28	2-7-29
1	–	1.87 m	1.87 m	1.78 m	–
2	–	1.31 m	1.34 m	1.33 m	–
3	–	1.71 m	1.37~1.77 m	1.67 m	–
4	ax 2.21 m	3.0 m	3.06 m	2.94 m	ax 2.17 d-like(11.5)
	eq 3.01 m	2.19 m	2.22 m	2.15 m	eq 2.99 m
6	ax 2.21 m	3.0 m	3.06 m	2.94 m	ax 2.17 d-like(11.5)
	eq 3.01 m	2.19 m	2.22 m	2.15 m	eq 2.99 m
7	–	1.37 m	1.44 m	1.40 m	–
8	–	1.84 m	1.84 m	1.77 m	–

Table 2-7-10 (continued)

H	2-7-25	2-7-26	2-7-27	2-7-28	2-7-29
9	–	1.36 m	1.37 m	1.3 m	–
		2.19 m	1.9~2.0 m	1.79~1.89 m	
10	–	2.19 m	1.9~2.0 m	1.79~1.89 m	–
11	4.21 dd(11.3, 3.9)	4.19 m	4.14 dd(11.2, 3.5)	4.17 m	4.13 d-like(11.6)
	4.17 dd(11.3, 5.4)		4.17 dd(11.2, 5.3)	4.12 m	4.03 dd(11.6, 5.2)
2′	7.56 d(8.5)	7.0 d(1.2)	7.5 d(1.1)	7.43 d(8.6)	7.55 d(8.5)
3′	7.13 d(8.5)	3.95 s(OMe)	3.86 s(OMe)	7.03 d(8.6)	7.02 d(7.2)
5′	7.13 d(8.5)	6.94 d(8.3)	7.14 d(8.3)	7.03 d(8.6)	7.02 d(7.2)
6′	7.56 d(8.5)	7.10 d(8.3)	7.08 d(8.3)	7.43 d(8.6)	7.55 d(8.5)
7′	7.66 d(15.9)	7.63 d(16.0)	7.6 d(15.8)	7.6 d(16.0)	6.87 d(12.8)
8′	6.40 d(15.9)	6.30 d(16.0)	6.31 d(15.8)	6.30 d(16.0)	5.85 d(12.8)
1″			5.54 d(1.5)	5.5 d(1.6)	5.54 s
2″			4.18 m	4.15 m	–
3″			4.0 m	3.99 dd(9.3, 2.5)	–
4″			3.6 m	3.6 t(9.3)	–
5″			3.8 m	3.7 m	–
6″			1.29 d(6.2)	1.28(6.0)	1.26 s
OAc	2.31 s				

Table 2-7-11: ^{1}H NMR spectroscopic data of lupinine-type alkaloids **2-7-30**~**2-7-32**.

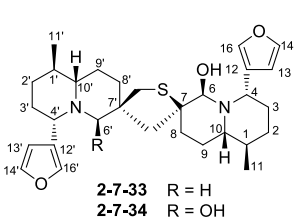
H	2-7-30	2-7-31	2-7-32
1		2.58 (ov)	1.03 d(7)
2	2.95~3.20 m		3.47 qd(7, 3.5)
3	–	*ca.*2.87 m	4.72 q(3.5)
4	*α* 2.64 br t(13.0)	*α ca.*1.84 (ov)	1.69 m
	β 2.87 br dd(13.0, 4.5)	*β ca.*3.00 (ov)	
5			1.86 m, 0.85 m
6	*α* 2.95~3.20 m, *β* 2.36 br t(10.0)	*α ca.*1.86 (ov), *β ca.*2.91 (ov)	3.01 dq(10.5, 3.5)
7	–	–	1.46 m, 1.37 m
8	–	0.72 (ov), 1.88 (ov)	1.42 m
9		*ca.*1.43 (ov)	1.61 (ov), 1.34 (ov)
10	–	*ca.*1.77 br d(9.4)	3.81 dt(8.0, 3.0)
16	0.68 d(6.5)	0.94 d(6.6)	
1′		2.75 br s	5.63 dd(15, 8)
2′	3.56 br t(10.6)		6.22 dd(15, 10)
3′	–	*ca.*2.64 (ov)	6.09 dd(15, 10)
4′	2.95~3.20 m	*α ca.*2.64 (ov), *β ca.*3.06 m	5.59 dt(15, 7)
5′			1.99 q(7)
6′	*α* 2.95~3.20 m, *β* 2.36 br t(10)	*α* 2.54 m, *β ca.*2.98 (ov)	1.26 m
7′	–	–	1.26 m
8′	–	–	0.83 t(7.5)

Table 2-7-11 (continued)

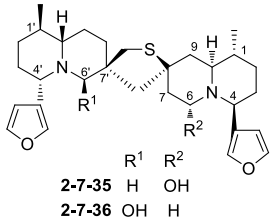
H	2-7-30	2-7-31	2-7-32
9′		1.62 (ov)	
10′	–	2.75 br s	
16′		0.94 d(6.0)	
OAc			1.82 s

Table 2-7-12: Cos, MFs, and TSs of lupinine-type alkaloids **2-7-33**~**2-7-40**.

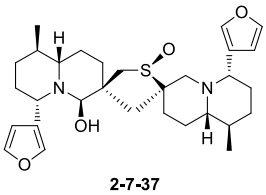
No.	Compounds	MFs	Test solvents	References
2-7-33	6-hydroxythiobinupharidine	$C_{30}H_{42}N_2O_3S$	CD_3OD	[174]
2-7-34	6,6′-dihydroxythiobinupharidine	$C_{30}H_{42}N_2O_4S$	CD_3OD	[174]
2-7-35	6-hydroxythionuphlutine B	$C_{30}H_{42}N_2O_3S$	CD_3OD	[174]
2-7-36	6′-hydroxythionuphlutine B	$C_{30}H_{42}N_2O_3S$	CD_3OD	[174]
2-7-37	nupharpumilamine A	$C_{30}H_{42}N_2O_4S$	CD_3OD	[175]
2-7-38	nupharpumilamine B	$C_{30}H_{42}N_2O_4S$	CD_3OD	[175]
2-7-39	nupharpumilamine C	$C_{30}H_{42}N_2O_3S$	$CDCl_3$	[175]
2-7-40	nupharpumilamine D	$C_{30}H_{42}N_2O_3S$	$CDCl_3$	[175]



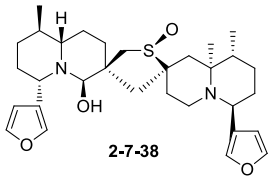
2-7-33 R = H
2-7-34 R = OH



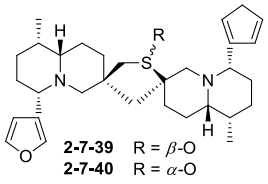
	R^1	R^2
2-7-35	H	OH
2-7-36	OH	H



2-7-37



2-7-38



2-7-39 R = β-O
2-7-40 R = α-O

Table 2-7-13: ^{1}H NMR spectroscopic data of lupinine-type alkaloids **2-7-33**~**2-7-36**.

H	2-7-33	2-7-34	2-7-35	2-7-36
1	1.26 (ov)	1.26 (ov)	1.18 (ov)	1.47 (ov)
2α	1.64 (ov)	1.62 (ov)	1.56 (ov)	1.70 (ov)
2β	1.09 (ov)	1.10 (ov)	1.04 (ov)	1.17 (ov)
3	1.66 (ov)	1.65 (ov)	1.48 (ov), 1.60 (ov)	1.66 (ov), 1.76 (ov)
4	3.63 dd(4.5, 10.5)	3.70 dd(5.0, 8.0)	3.63 dd(3.0, 11.5)	3.12 dd-like

Table 2-7-13 (continued)

H	2-7-33	2-7-34	2-7-35	2-7-36
6	3.77 s	3.77 s	3.85 s	*α* 1.83 d(11.5), *β* 2.77 d(11.5)
8*α*	1.31 (ov)	1.40 (ov)	1.70 (ov)	1.56 (ov)
8*β*	1.84 (ov)	1.86 (ov)	1.88 (ov)	1.88 (ov)
9*α*	1.26 (ov)	1.21 (ov)	1.77 (ov)	1.94 (ov)
9*β*	1.80 (ov)	1.82 (ov)	1.53 (ov)	1.41 (ov)
10	2.28 (ov)	2.32 (ov)	2.31 (ov)	1.77 (ov)
11	0.90 d(6.0)	0.92 d(6.5)	0.87 d(6.5)	0.92 d(6.5)
13	6.43 d(1.7)	6.43 d(1.0)	6.44 d(1.5)	6.44 d(1.2)
14	7.41 t(1.7)	7.45 t(1.0)	7.40 t(1.5)	7.38 t-like
16	7.35 br s	7.43 br s	7.41 br s	7.33 br s
17*α*	1.63 d(13.5)	1.79 d(13.0)	1.62 d-like	1.61 s
17*β*	2.07 d(13.5)	1.84 d(13.0)	1.69 d-like	1.61 s
1′	1.50 d(2.9)	1.28 (ov)	1.36 (ov)	1.17 (ov)
2′*α*	1.72 (ov)	1.62 (ov)	1.63 (ov)	1.59 (ov)
2′*β*	1.16 (ov)	1.10 (ov)	1.11 (ov)	1.07 (ov)
3′	1.66 (ov), 1.79 (ov)	1.65 (ov)	1.58 (ov)	1.50 (ov), 1.62 (ov)
4′	2.92 d-like	3.65 dd(5.0, 8.0)	2.92 dd(3.5, 10.5)	3.63 dd(3.0, 11.0)
6′	*α* 2.92 d(11.5) *β* 1.52 d(11.5)	3.93 s	*α* 2.68 d(11.0) *β* 1.53 d(11.0)	3.57 s
8′*α*	1.53 (ov)	1.60 (ov)	1.20 (ov)	1.73 (ov)
8′*β*	1.19 (ov)	1.27 (ov)	1.76 (ov)	1.58 (ov)
9′*α*	1.31 (ov)	1.21 (ov)	1.20 (ov)	1.16 (ov)
9′*β*	1.89 (ov)	1.79 (ov)	1.88 (ov)	1.84 (ov)
10′	1.53 (ov)	2.35 (ov)	1.53 (ov)	2.32 (ov)
11′	0.94 d(6.5)	0.93 d(6.5)	0.90 d(6.5)	0.88 d(6.5)
13′	6.41 d(1.7)	6.42 d(1.0)	6.23 d(1.5)	6.26 d(1.2)
14′	7.46 t(1.7)	7.45 t(1.0)	7.17 t(1.5)	7.36 t-like
16′	7.43 br s	7.43 br s	7.27 br s	7.33 br s
17′*α*	2.25 d(12.0)	2.19 d(12.0)	2.20 d(11.5)	2.30 d(12.5)
17′*β*	2.30 d(12.0)	2.64 d(12.0)	2.39 d(11.5)	2.54 d(12.5)

Table 2-7-14: ^{1}H NMR spectroscopic data of lupinine-type alkaloids **2-7-37**~**2-7-40**.

H	2-7-37	2-7-38	2-7-39	2-7-40
1	–	–	1.75 m	–
4	3.04 dd(3.1, 11.6)	2.91 dd(6.4, 7.3)	2.87 dd(3.4, 11.6)	2.94 dd(3.4, 11.0)
6*α*	2.54 dd(2.7, 11.3)	1.67 dd(0.9, 12.8)	2.53 dd(2.4,11.3)	1.95 d(11.3)
6*β*	1.89 dd(1.2, 11.3)	3.20 dd(2.4, 12.8)	1.85 d(11.3)	2.97 dd(2.4, 11.3)
8	*α* 1.66 m, *β* 1.53 m		–	–
9	*α* 1.33 m, *β* –	–	–	–
10	1.64 m	1.56 m	2.01 m	2.03 m
11	0.98 d(6.4)	0.81 d(6.4)	1.13 d(7.0)	1.16 d(6.7)
13	6.40 dd(0.6, 1.5)	6.30 dd(0.6, 1.8)	6.34 d(1.5)	6.38 d-like

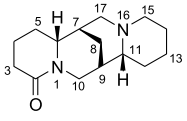
Table 2-7-14 (continued)

H	2-7-37	2-7-38	2-7-39	2-7-40
14	7.43 dd(1.5, 1.5)	7.31 dd(1.5, 1.8)	7.32 t(1.5)	7.34 t-like
16	7.37 dd(0.6, 1.5)	7.35 dd(0.6, 1.5)	7.24 br s	7.28 br s
17α	2.28 d(15.0)	1.56 d(14.3)	1.92 d(14.3)	2.03 d(14.3)
17β	2.02 d(15.0)	1.89 d(14.3)	2.15 d(14.3)	2.33 d(14.3)
1′	–	–	1.69 m	
4′	3.68 dd(3.4, 10.7)	3.53 dd(3.1, 11.6)	2.80 dd(3.4, 11.6)	2.83 dd(3.4, 11.9)
6′	4.14 s	4.08 s	α 2.90 dd(2.4, 11.6)	α 2.47 dd(2.4, 11.3)
			β 1.48 d(11.6)	β 1.47 d(11.3)
10′	2.35 m	2.24 ddd(2.4, 8.5, 11.0)	1.90 m	1.94 m
11′	0.92 d(6.4)	0.78 d(6.4)	1.11 d(7.0)	1.14 d(7.0)
13′	6.40 dd(0.6, 1.5)	6.01 dd(0.6, 1.5)	6.35 d(1.5)	6.38 d-like
14′	7.43 dd(1.5, 1.5)	6.89 t(1.5)	7.33 t(1.5)	7.34 t-like
16′	7.40 dd(0.6, 1.5)	7.17 dd(0.6, 1.5)	7.24 br s	7.25 br s
17′	α 2.32 d(14.3)	α 2.35 d(15.6)	α 2.56 d(13.4)	2.51 s
	β 2.92 d(14.3)	β 2.97 d(15.6)	β 2.43 d(13.4)	

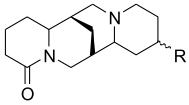
2.7.2 Sparteine-type alkaloids

Table 2-7-15: Cos, MFs, and TSs of sparteine-type alkaloids **2-7-41**~**2-7-52**.

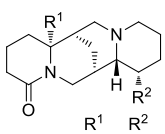
No.	Compounds	MFs	Test solvents	References
2-7-41	α-isolupanine	$C_{15}H_{24}N_2O$	$CDCl_3$	[176]
2-7-42	13α-hydroxylupanine	$C_{15}H_{24}N_2O_2$	$CDCl_3$	[177]
2-7-43	13β-hydroxylupanine	$C_{15}H_{24}N_2O_2$	$CDCl_3$	[177]
2-7-44	(–)-6α-hydroxylupanine	$C_{15}H_{24}N_2O_2$	$CDCl_3$	[178]
2-7-45	(–)-6α-methoxylupanine	$C_{16}H_{26}N_2O_2$	$CDCl_3$	[178]
2-7-46	(+)-12α-hydroxylupanine	$C_{15}H_{24}N_2O_2$	$CDCl_3$	[179]
2-7-47	(+)-2β-hydroxyaphylline	$C_{15}H_{24}N_2O_2$	$CDCl_3$	[180]
2-7-48	(+)-15β-hydroxy-17-oxolupanine	$C_{15}H_{22}N_2O_3$	$CDCl_3$	[181]
2-7-49	3α-hydroxylupanine	$C_{15}H_{24}N_2O_2$	$CDCl_3$	[182]
2-7-50	13β-methoxylupanine	$C_{16}H_{26}N_2O_2$	$CDCl_3$	[183]
2-7-51	4α-hydroxy-13β-methoxylupanine	$C_{16}H_{26}N_2O_3$	$CDCl_3$	[183]
2-7-52	3β,4α-dihydroxy-13β-methoxylupanine	$C_{16}H_{26}N_2O_4$	$CDCl_3$	[183]



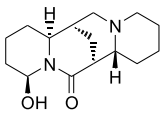
2-7-41



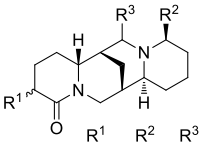
2-7-42 R = α-OH
2-7-43 R = β-OH



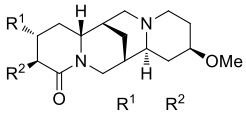
	R^1	R^2
2-7-44	OH	H
2-7-45	OMe	H
2-7-46	H	OH



2-7-47



	R^1	R^2	R^3
2-7-48	H	OH	=O
2-7-49	α-OH	H	H



	R^1	R^2
2-7-50	H	H
2-7-51	OH	H
2-7-52	OH	OH

Table 2-7-16: ^{1}H NMR spectroscopic data of sparteine-type alkaloids **2-7-41**~**2-7-43**.

H	2-7-41	2-7-42	2-7-43
3	2.36 m	2.24 ddddd(17.4, 2.3, 2.3, 2.3, 2.3)	2.50 ddddd(17.3, 2.3, 2.3, 2.3, 2.3)
	2.23 m	2.13 ddd(17.3, 13.0, 5.8)	2.33 ddd(17.4, 13.0, 5.4)
4	1.80 m	1.61~1.68 m	1.83 m
	1.59 m	1.40~1.51 m	1.60 ddddd(13.3, 13.3, 13.3, 4.5, 2.8)
5	1.76 m	1.61~1.68 m, 1.37 m	1.80 m, 1.48 m
6	3.45 d	3.20 m	3.33 ddd(11.1, 5.3, 1.6)
7	1.60 m	1.93 m	2.13 m
8	1.78 m	1.97 dddd(12.6, 4.2, 4.2, 4.2)	2.26 dm(13.0)
		1.13 ddd(12.6, 2.4, 2.4)	1.38 ddd(13.1, 2.4, 2.4)
9	1.56 m	1.44 m	1.80 m
10	4.86 br d	4.21 ddd(13.2, 2.4, 2.4)	4.54 ddd(13.3, 2.3, 2.3)
	2.54 m	2.36 dd(13.9, 2.6)	2.56 dd(13.3, 2.5)
11	1.93 m	1.90 ddd(11.7, 3.2, 3.2)	2.06 ddd(12.1, 2.5, 2.5)
12	1.30 m, 1.62 m	1.51~1.40 m	1.89 m, 1.70 m
13	1.68 m, 1.17 m	3.84 dddd(2.8, 2.8, 2.8, 2.8)	3.63 m
14	1.45 m	1.59 m, 1.61~1.40 m	1.89 m, 1.72 m
15	2.67 m	2.37 ddd(11.8, 4.7, 2.3)	3.05 ddd(12.1, 4.0, 2.5)
	1.68 m	2.18 ddd(13.2, 11.8, 2.9)	2.21 ddd(12.4, 12.4, 2.5)
17	2.97 m	2.69 dd(12.1, 10.1)	3.22 dd(11.9, 9.8)
	2.13 m	1.79 dd(12.1, 3.7)	2.08 dd(12.0, 3.6)

Table 2-7-17: ^{1}H NMR spectroscopic data of sparteine-type alkaloids **2-7-44** and **2-7-45**.

H	2-7-44	2-7-45	H	2-7-44	2-7-45
3α	2.48 ddd(13.1, 6.8, 5.4)	2.46 m	10α	2.95 d(13.2)	2.95 dd(12.8, 2.3)
3β	2.35 ddd(13.1, 2.5, 2.5)	2.38 m	10β	4.22 dd(13.2, 2.1)	4.30 dd(12.8, 2.0)
6		3.48 s(OMe)	15	α 2.76 m	α 2.75 d(11.5)
7	2.09 m	2.20 m	17	α 2.92 dd(11.8, 7.8)	α 2.82 ddd(13.2, 7.4, 2.9)

Table 2-7-18: ^{1}H NMR spectroscopic data of sparteine-type alkaloids **2-7-46**~**2-7-48**.

H	2-7-46	2-7-47	2-7-48
2		eq 6.13 dd(2.9, 1.8)	
3	α 2.46 d(11.3)	ax 1.61 m	1.39~1.46 m
	β 2.33 m	eq 1.67 m	

Table 2-7-18 (continued)

H	2-7-46	2-7-47	2-7-48
4	*α* 1.85~1.90 m, *β* 1.62 m	ax 1.70 m, eq 1.88~1.81 m	ax 1.39~1.46 m, eq 1.65~1.72 m
5	*α* 1.75~1.79 m, *β* 1.49~1.57 m	ax 1.41~1.47 m, eq 1.24~1.38 m	ax 2.26 m, eq 2.37 br d(13.2)
6	3.3 m	3.27 ddd(12.1, 5.1, 2.0)	3.46 m
7	2.09~2.05 m	1.70 m	2.50 br s
8	*α* 2.12 br d	ax 1.74 m	ax 1.87~1.99 m
	β 1.34 m	eq 2.11 dd(12.1, 3.0)	eq 2.19 m
9	2.12 br d	2.19 m	1.77 br s
10	*α* 4.5 dt(13.1, 2.3)		ax 4.81 dt(13.4, 2.4)
	β 2.53 d(15.2)		eq 2.69 dd(13.4, 2.4)
11	1.75~1.79 m	1.88~1.81 m	3.79 dd(12.3, 2.7)
12	*β* 3.6 br s	ax1.24~1.38 m, eq 1.41~1.47 m	ax 1.52~1.59 m, eq 1.65~1.72 m
13	*α* 1.82 m, *β* 1.44 d-like	ax 1.24~1.38 m, eq 1.81~1.88 m	ax 1.65~1.72 m, eq 2.19 m
14	*α* 1.85~1.90 m, *β* 1.47 d-like	1.53~1.58 m	ax 1.52~1.59 m, eq 1.87~1.99 m
15	*α* 2.7 d(8.5)	ax 1.89 d-like	6.18 dd(6.2, 3.3)
	β 1.95 d(3.5)	eq 2.63 br d(14.7)	
17	*α* 2.85 t(11.2)	ax 2.31 dd(11.9, 3.2)	
	β 1.97 d(3.4)	eq 2.87 dd(11.9, 8.9)	

Table 2-7-19: ^{1}H NMR spectroscopic data of sparteine-type alkaloids **2-7-49**~**2-7-52**.

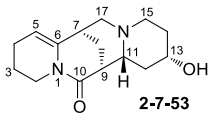
H	2-7-49	2-7-50	2-7-51	2-7-52
3	4.02 dt(4.2, 1.2)	2.41 m	2.64 ddd	3.85 d(9.5)
		2.23 m	2.31 dd(16.2, 10.1)	
4	ax 1.88 m, eq 1.89 m	1.88 m, 1.62 m	3.92 m	3.82 m
5	ax 1.72 m, eq 1.85 m	1.83 m, 1.52 m	1.96 m, 1.64 m	2.04 m, 1.76 m
6	3.39 ddd(12.3, 6.3, 2.0)	3.28 m	3.36 m	3.38 ddd(11.3, 5.5, 2.2)
7	1.96 m	1.97 m	1.91 m	1.97 m
8	ax 1.28 dt(12.5, 2.3)	2.08 m	2.10 m	2.19 m
	eq 2.12 dt(12.5, 2.0)	1.25 m	1.33 m	1.37 m
9	1.58 m(2.3)	1.65 m	1.66 m	1.72 m
10ax	2.53 dm(13.2, 2.3)	2.53 dd(13.3, 2.7)	2.55 dd(13.3, 2.7)	2.71 dd(13.6, 2.9)
10eq	4.52 dt(13.2, 2.3)	4.46 dt(13.3, 2.2)	4.42 dt(13.3, 2.2, 2.2)	4.28 dt(13.6, 2.2, 2.2)
11	1.88 m	1.88 m	1.96 m	1.96 m
12	ax 1.41 m, eq 1.42 m	1.82 m, 1.32 m	1.76 m, 1.37 m	1.82 m, 1.43 m
13	ax 1.51 m	3.09 m	3.10 m	3.12 m
	eq 1.55 m	3.34 s(OMe)	3.27 s(OMe)	3.32 s(OMe)
14	ax 1.25 dt(13.0, 3.9)	1.84 m	1.78 m	1.86 m
	eq 1.68 m	1.40 m	1.39 m	1.48 m

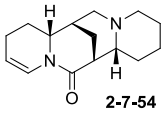
Table 2-7-19 (continued)

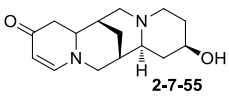
H	2-7-49	2-7-50	2-7-51	2-7-52
15	ax 2.05 dt(15.1, 2.8) eq 2.72 dm(15.1)	2.77 m 1.99 m	ax 2.11 m eq 2.75 ddd(11.6)	ax 2.15 m eq 2.82 ddd(12.5, 4.0, 2.9)
17	ax 2.28 dd(12.0, 3.4) eq 2.89 dd(12.0, 10.5)	2.84 m 1.96 m	ax 2.13 m eq 2.87 dd(11.8, 8.0)	ax 2.12 m eq 2.96 dd(12.1, 8.4)

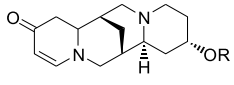
Table 2-7-20: Cos, MFs, and TSs of sparteine-type alkaloids **2-7-53**~**2-7-62**.

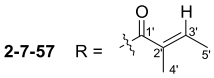
No.	Compounds	MFs	Test solvents	References
2-7-53	(+)-13α-hydroxyaphyllidine	$C_{15}H_{22}N_2O_2$	$CDCl_3$	[180]
2-7-54	(+)-2,3-dehydro-10-oxo-α-isoparteine	$C_{15}H_{22}N_2O$	C_6D_6	[184]
2-7-55	13β-hydroxymultiflorine	$C_{15}H_{22}N_2O_2$	$CDCl_3$	[180]
2-7-56	13α-hydroxymultiflorine	$C_{15}H_{22}N_2O_2$	$CDCl_3$	[180]
2-7-57	(–)-13α-tigloyloxymultiflorine	$C_{20}H_{28}N_2O_3$	$CDCl_3$	[161]
2-7-58	(–)-Δ^5-dehydromultiflorine	$C_{15}H_{20}N_2O$	$CDCl_3$	[185]
2-7-59	(–)-clathrotropine	$C_{17}H_{24}N_2O_3$	CD_3OD	[186]
2-7-60	baptifoline	$C_{15}H_{20}N_2O_2$	$CDCl_3$	[176]
2-7-61	(–)-*O*-acetylbaptifoline	$C_{17}H_{22}N_2O_3$	$CDCl_3$	[187]
2-7-62	(±)-termisine	$C_{16}H_{28}N_2O_3$	CD_3OD	[188]

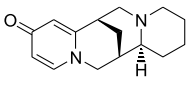


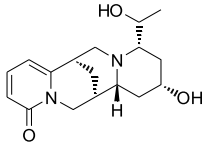


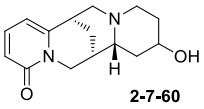


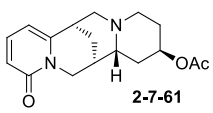












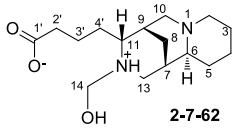


Table 2-7-21: ^{1}H NMR spectroscopic data of sparteine-type alkaloids **2-7-53**~**2-7-55**.

H	2-7-53	2-7-54	2-7-55
2	ax 3.66 ddd(13.1, 8.1, 3.9) eq 3.77 ddd(12.9, 4.3, 1.8)	7.74 d(8.4)	6.93 d(7.7)

Table 2-7-21 (continued)

H	2-7-53	2-7-54	2-7-55
3		4.89 m	4.94 d(7.7)
4	ax 2.11 ddd(14.1, 5.0, 1.8) eq 2.07 dd(13.9, 4.9)	1.87~1.90 m	
5	4.77 dd(5.7, 1.8)	ax 1.76 dd(11.5, 2.5) eq 1.30 m	ax 1.77 m eq 1.83 m
6		3.11 dd(11.5, 2.7)	3.27 ddd(16.2, 5.3, 2.7)
7	2.56 d-like	1.32 m	1.99 m
8 ax	1.63~1.77 m	1.61 m	1.77 m
8 eq	1.83 dd(11.8, 3.6)	1.40 m	1.22~1.47 m
9	2.43 br s	2.32 m	1.60 m
10			ax 3.07 d(13.2) eq 2.99 dd(13.1, 3.2)
11	3.09 m	1.68 dd(13.4, 2.7)	2.07 ddd(13.2, 3.9, 3.4)
12	ax 1.22~1.47 m eq 1.87 m	ax 2.00 qd(13.4, 3.1) eq 1.61 m	1.22~1.47 m
13	4.11 t(2.7)	ax 1.07 qt(13.4, 3.9) eq 1.61 m	3.87 m
14	ax 1.22~1.47 m eq 1.63~1.77 m	ax 1.41 qt(13.4, 3.4) eq 1.34 m	1.22~1.47 m
15 ax	2.82 ddd(12.9, 12.9, 1.9)	1.70 dt(2.7, 13.4)	2.11 m
15 eq	2.78 ddd(12.9, 2.1, 2.1)	2.55 m	2.41 br d(11.9)
17 ax	2.33 d(11.3)	1.81 dd(12.0, 2.7)	2.32 dd(13.7, 2.9)
17 eq	3.36 dd(11.3, 2.6)	2.67 dd(12.0, 2.4)	2.81 dd(11.4, 3.2)

Table 2-7-22: ^{1}H NMR spectroscopic data of sparteine-type alkaloids **2-7-56**~**2-7-58**.

H	2-7-56	2-7-57	2-7-58
2	6.85 d(7.7)	6.86 d(7.4)	7.19 d(7.6)
3	4.96 d(7.7)	4.99 d(7.4)	6.36 dd(7.6, 2.7)
5	ax 1.93 m eq 2.11 ddd(16.7, 5.6, 5.2)	–	6.19 d(2.7)
6	3.49 ddd(14.7, 5.0, 2.5)	3.50 br d(15.7)	
7	1.93 m	–	2.90 m
8	ax 1.93 m, eq 1.32~1.47 m	–	2.0 br s
9	1.71 d-like	–	–
10	ax 3.27 d(12.1) eq 3.13 dd(12.7, 3.0)	ax 2.52 d(11.3) Eq 3.00 t-like	α 3.92 d(12.7) β 4.12 dd(12.7, 6.3)
11	2.26 ddd(12.9, 3.7, 3.0)	–	2.93 d(11.8)
12	ax 1.32~1.47 m eq 1.53~1.65 m	–	–
13	4.13 br s	eq 5.17 br s	–
14	1.65~1.53 m	–	–

Table 2-7-22 (continued)

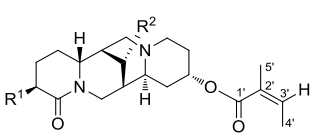
H	2-7-56	2-7-57	2-7-58
15	ax 2.42 br d(14.7) eq 2.73 br d(11.7)	–	2.76 ddd(13.8, 13.8, 2.8) 2.68 ddd(13.8, 1.9, 1.9)
17	ax 2.62 dd(12.3, 2.7) eq 2.96 dd(11.2, 3.3)	ax 3.26 d(12.1) eq 3.21 d(12.1)	3.35 dd(11.0, 2.8) 2.49 d(8.3)
3′		6.90 br d(6.9)	
4′		1.83 d(6.6)	
5′		1.87 s	

Table 2-7-23: ^{1}H NMR spectroscopic data of sparteine-type alkaloids **2-7-59**~**2-7-62**.

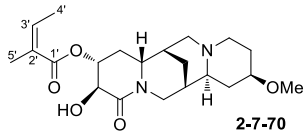
H	2-7-59	2-7-60	2-7-61	2-7-62
2				ax 2.96 td(13.0, 1.3) eq 3.13 t(9.5)
3	6.41 dd(1.3, 9.0)	6.40 dd(9, 1)	6.44 dd(9.1, 1.1)	ax 1.94 d(9.2) eq 2.30 dd(10.4, 3.1)
4	7.47 dd(7.0, 8.9)	7.26 dd(9, 7)	7.27 dd(9.1, 6.7)	1.71 m 1.98 d(13.7)
5	6.30 dd(1.2, 7.0)	5.97 dd(7, 1.3)	5.97 dd(6.9, 1.1)	eq 1.74 m
6			–	eq 3.10 t(6.4)
7	3.12 br s	2.95 m	–	eq 1.73 s
8	*α* 1.85 br d(13.2) *β* 2.15 br d(13.2)	*R* 1.67 br d(13) *S* 1.97 br d(13)	–	ax 1.68 m eq 1.79 br s
9	2.29 m	2.08 m	–	ax 1.79 br s
10	*α* 4.13 d(15.4) *β* 3.92 dd(6.3, 15.4)	*α* 3.88 dd(15.4, 6.6) *β* 4.06 br d(15.4)	*α* 3.91 dd(15.4, 6.6) *β* 4.12 d(15.4)	ax 3.36 d(13.4) eq 3.50 d(13.4)
11	2.96 br d(12.5)	3.44 br dt(13.0, 2, 2)	–	3.72 d(12.8)
12	*α* 1.94 m(12.5) *β* 1.56 m(ov)	*α* 2.10 br dt(14, 2.5) *β* 1.25 br d(14)	–	
13	3.75 m(ov)	4.26 m	5.20 t(2.8) 2.08 s(OAc)	ax 3.33 td(12.2, 1.8) eq 4.12 dd(12.8, 2.6)
14	*α* 1.36 m(12.5) *β* 1.58 m(ov)	*α* 1.85 dt(13.5, 13.5, 2) *β* 1.31 br d(13.5)	–	ax 4.34 dd(11.8, 2.6) eq 4.60 d(11.7)
15	2.36 ddd(2.1, 8.5, 12.3)	*α* 2.37 br d(14) *β* 3.19 dt(14, 14, 2.8)	*α* 3.10 ddd(14.3, 14.3, 2.8)	
17	*α* 3.06 dd(2.0, 11.2) *β* 2.74 dd(2.1, 11.4)	*α* 3.33 br d(10.7) *β* 2.45 br d(10.7)	*α* 2.49 m *β* 3.32 m	
1′	3.57 m			
2′	0.99 d(6.2)			2.19 dd(7.0, 1.3)
3′				ax 1.54 m eq 1.60 dd(6.4, 1.7)
4′				2.24 m, 1.54 m
5′				1.54 m

Table 2-7-24: Cos, MFs, and TSs of sparteine-type alkaloids **2-7-63**~**2-7-70**.

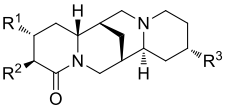
No.	Compounds	MFs	Test solvents	References
2-7-63	lupanine-13α-*O*-angelate	$C_{20}H_{30}N_2O_3$	$CDCl_3$	[189]
2-7-64	cajanifoline	$C_{20}H_{30}N_2O_4$	$CDCl_3$	[189]
2-7-65	pearsonine	$C_{20}H_{30}N_2O_5$	$CDCl_3$	[189]
2-7-66	cryptanthine	$C_{20}H_{30}N_2O_4$	$CDCl_3$	[189]
2-7-67	sessilifoline	$C_{20}H_{30}N_2O_4$	$CDCl_3$	[189]
2-7-68	cineverine	$C_{24}H_{32}N_2O_5$	$CDCl_3$	[177]
2-7-69	cineroctine	$C_{23}H_{36}N_2O_4$	$CDCl_3$	[177]
2-7-70	4α-angeloyloxy-3β-hydroxy-13β-methoxylupanine	$C_{21}H_{32}N_2O_5$	$CDCl_3$	[190]



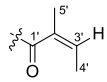
	R^1	R^2
2-7-63	H	H
2-7-64	OH	H
2-7-65	OH	OH
2-7-66	H	OH

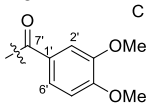


2-7-70



	R^1	R^2	R^3
2-7-67	OAng	OH	H
2-7-68	H	H	OB
2-7-69	H	H	OC





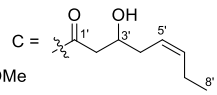


Table 2-7-25: ^{1}H NMR spectroscopic data of sparteine-type alkaloids **2-7-63**~**2-7-66**.

H	2-7-63	2-7-64	2-7-65	2-7-66
3	1.47~1.80 m	3.96 dd(11.4, 5.6) 4.65 br s(OH)	4.00 dd(11.1, 5.3) 1.59~1.96 m	1.30~2.0 m
4	1.47~1.80 m	ax 1.61~1.81 m eq 2.21 m	1.59~1.96 m	1.30~2.0 m
5	1.47~1.80 m	1.61~1.86 m	eq 2.27 m	1.30~2.0 m
6	3.26 m	3.33 ddd(10.6, 5.1, 1.7)	3.32 m	3.22 ddd(10.5, 4.5, 1.2)
7	2.00~2.40 m	1.99 m	2.27 m	2.05~2.30 m
8	ax 1.25 dt(2.4, 2.4, 12.3) eq 2.40~2.00 m	ax 1.27 dt(2.3, 2.3, 12.5) eq 2.12 m	3.61 t(2.9, 2.9)	3.55 t(2.8, 2.8) 4.0 br s(OH)
9	1.47~1.80 m	1.59 m	1.59~1.96 m	1.30~2.0 m
10ax	2.49 dd(2.8, 13.3)	2.65 dd(2.5, 13.4)	2.59 dd(2.5, 13.6)	2.40 dd(2.3, 13.8)
10eq	4.30 dt(2.2, 2.2, 13.3)	4.26 dt(2.2, 2.2, 13.4)	4.30 dd(3.0, 13.6)	4.45 dd(3.0, 13.8)
11	2.00~2.40 m	2.12 m	1.59~1.96 m	2.05~2.30 m
12	1.47~1.80 m	1.61~1.86 m	1.59~1.96 m	1.30~2.0 m
13	5.11 quint(2.8, 2.8)	5.13 quint(2.8, 2.8)	5.11 quint(2.8, 2.8)	5.04 quint(2.8, 2.8)
14	1.47~1.80 m	1.86~1.61 m	1.59~1.96 m	1.30~2.0 m

Table 2-7-25 (continued)

H	2-7-63	2-7-64	2-7-65	2-7-66
15ax	2.00~2.40 m	2.35 dt(12.5, 12.5, 2.6)	2.14 m	2.05~2.30 m
15eq	2.58 ddd(2.3, 4.3, 12.4)	2.60 ddd(1.9, 4.6, 12.5)	2.67 ddd(4.0, 2.9, 11.5)	2.59 dt(3.6, 3.6, 11.5)
17ax	2.00~2.40 m	2.04 dd(3.8, 11.6)	2.13 dd(2.3, 11.7)	2.05~2.30 m
17eq	2.85 dd(10.4, 12.3)	2.91 dd(9.2, 11.6)	2.99 dd(10.5, 11.7)	2.81 dd(10.6, 11.4)
3′	6.01 dd(7.4, 1.4)	6.04 qq(7.2, 1.4)	6.06 qq(7.2, 1.4)	5.99 q(7.2, 1.4)
4′	1.93 dq(7.4, 1.5)	1.95 dq(7.2, 1.4)	1.96 dq(7.3, 1.6)	1.90 dq(7.3, 1.5)
5′	1.85 dq(1.4, 1.5)	1.86 quint(1.4, 1.4)	1.86 dq(1.4, 1.6)	1.81 dq(1.4, 1.5)
OH			3.77 br s	

Table 2-7-26: ^{1}H NMR spectroscopic data of sparteine-type alkaloids **2-7-67**~**2-7-70**.

H	2-7-67	2-7-68	2-7-69	2-7-70
3	4.14 d(10.1) 3.70 br s(OH)	2.44 ddddd (17.2, 2.1, 2.1, 2.1, 2.1) 2.26 ddd(17.4, 12.5, 5.4)	2.47 ddddd (17.6, 2.1, 2.1, 2.1, 2.1) 2.29 ddd(17.2, 13.2, 5.5)	4.15 d(10)
4	5.00 ddd (10.1, 11.9, 4.1)	1.79 m 1.59 dddd (13.1, 13.1, 13.1, 3.0)	1.82 m 1.58 dddddd	5.05 ddd(4, 10, 12)
5	ax 2.07 m eq 2.23 ddd (4.1, 13.2, 5.0)	1.52 dddd, 1.75 m	1.78 m 1.49 dddd (13.2, 13.4, 13.4, 3.2)	1.75 m, 2.2 m
6	3.44 dd(11.7, 5.0)	3.29 ddd(10.9, 5.2, 1.5)	3.31 ddd(11.0, 5.1, 1.2)	3.5 ddd(11.5, 5, 2)
7	1.40~1.75 m	2.05 ddddd	2.10 ddddd	1.9 m
8	ax 1.38 m eq 2.36 m	2.24 dddd 1.31 dm(12.6)	1.29 dm(12.0), 2.26 m	1.3 m, 2.1 m
9	1.40~1.75 m	1.63 dddddd	1.61 m	1.7 m
10	ax 2.71 dd(2.2, 13.4) eq 4.24 dt(2.0, 2.0, 13.4)	4.50 ddd(13.4, 2.5, 2.5) 2.52 dd(13.4, 2.5)	4.42 ddd(13.3, 2.4, 2.4) 2.46 dd(13.0, 2.6)	4.35 dt(13.5, 2.2) 2.7 m
11	1.40~1.75 m	2.34 m	–	1.95 m
12	1.40~1.75 m	1.82 m	1.92 dm(14.1)	1.75 m, 1.35 m
13	1.40~1.75 m	5.24 dddd (2.7, 2.7, 2.7, 2.7)	5.03 dddd (2.8, 2.8, 2.8, 2.8)	3.15 tt(11, 4.5) 3.35 s(OMe)
14	1.40~1.75 m	1.95 dddd 1.85 br s	1.72 dm(14.4)	1.8 m 1.4 m
15	ax 1.40~1.75 m, eq 2.83 m	2.68 ddd, 2.42 ddd	2.71 br s, 2.32 dd	2.75 m, 2.1 m
17	ax 1.40~1.75 m eq 2.96 dd(10.2, 11.8)	2.94 dd 2.12 dd(11.8, 2.9)	2.93 dd 1.96 ddd	2.95 dd(12, 8) 2.0 m
2′		7.49 d(1.9)	2.53 dd(14.7, 3.1) 2.32 dd(14.7, 10.3)	

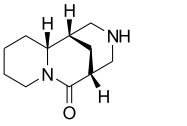
Table 2-7-26 (continued)

H	2-7-67	2-7-68	2-7-69	2-7-70
3′	6.09 qq(7.2, 1.4)		4.01 ddddd 4.17 d(2.4, OH)	6.1 qq(7.3, 1.5)
4′	1.99 dq(7.2, 1.5)		2.33 ddddd 2.21 ddddd (18.3, 7.2, 7.2, 1.0, 1.0)	2.0 dq(1, 7.3)
5′	1.89 dq(1.4, 1.5)	6.88 d(8.4)	5.39 dddd(10.8)	1.9 d(1)
6′		7.64 dd(8.4, 1.9)	5.50 dddd(10.8)	
7′			2.05 ddddd (7.5, 7.5, 7.5, 1.0, 1.0)	
8′			0.95 t(7.5)	
OMe		3.89 s, 3.90 s		

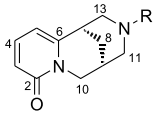
2.7.3 Cytisine-type alkaloids

Table 2-7-27: Cos, MFs, and TSs of cytisine-type alkaloids **2-7-71**~**2-7-80**.

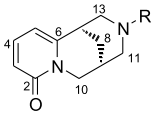
No.	Compounds	MFs	Test solvents	References
2-7-71	virgilidone	$C_{11}H_{18}N_2O$	$CDCl_3$	[191]
2-7-72	(–)-*N*-methylcytisine	$C_{12}H_{16}N_2O$	$CDCl_3$	[192]
2-7-73	(–)-12-hydroxycytisine	$C_{11}H_{14}N_2O_2$	CD_3OD	[193]
2-7-74	(–)-12-cytisineacetamide	$C_{13}H_{17}N_3O_2$	CD_3OD	[193]
2-7-75	(–)-*N*-(*N*-acetylaminomethyl)cytisine	$C_{14}H_{19}N_3O_2$	$CDCl_3$	[192]
2-7-76	(–)-*N*-(2-oxopyrrolidinomethyl)cytisine	$C_{16}H_{21}N_3O_2$	$CDCl_3$	[192]
2-7-77	sophorasine A	$C_{15}H_{20}N_2O_3$	$CDCl_3$	[194]
2-7-78	sophorasine B	$C_{15}H_{20}N_2O_3$	$CDCl_3$	[194]
2-7-79	(–)-11,12-*seco*-12,13-didehydromultiflorin	$C_{15}H_{22}N_2O$	$CDCl_3$	[195]
2-7-80	angustifoline *N*-carboxymethyl ester	$C_{16}H_{24}N_2O_3$	$CDCl_3$	[196]



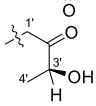
2-7-71



2-7-72 R = Me
2-7-73 R = OH
2-7-74 R = CH_2CONH_2
2-7-75 R = $CH_2NHCOCH_3$

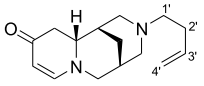


2-7-76 R =

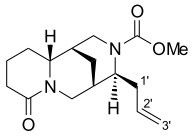


2-7-77 R =

2-7-78 R =



2-7-79



2-7-80

Table 2-7-28: ^{1}H NMR spectroscopic data of cytisine-type alkaloids **2-7-71**~**2-7-74**.

H	2-7-71	2-7-72	2-7-73	2-7-74
2	ax 2.44 dt(12.9, 12.9, 3.1) eq 4.83 dm(13.5)			
3	1.76 dm(10), 1.40~1.49 m	6.43 dd(9.0, 1.5)	6.39 dd(8.8, 1.1)	6.43 dd(8.8, 1.4)
4	1.93 m, 1.40~1.49 m	7.26 dd(9.0, 7.0)	7.45 dd(8.8, 6.9)	7.48 dd(8.8, 6.9)
5	ax 1.54 dq(12, 12, 12, 3.5) eq 1.69 dm(12.5)	5.98 dd(7.0, 1.5)	6.29 dd(6.9, 0.8)	6.32 dd(6.9, 1.1)
6	3.36 ddd(11.4, 5.3, 2.5)			
7	1.78 m	2.98 m	3.18 d(2.5)	3.14 d(2.2)
8	1.99 br d(5.5) 1.91 dt(12.8, 2.6, 2.6)	1.92 m 1.75 m	1.83 dd(13.9, 3.0)	ax 1.87 d(12.9) eq 1.96 dt(12.9, 1.5)
9	2.55 br s	2.47 m	2.60 dd(6.1, 3.0)	2.50 d(2.5)
10		4.06 d(15.5) 3.89 dd(15.0, 7.5)	ax 3.87 dd(15.4, 6.7) eq 4.05 d(15.4)	ax 3.91 dd(15.5, 6.0) eq 4.12 d(15.5)
11	3.27 br d(13.5) 2.78 dd(13.4, 2.5)	2.89 d(12.0) 2.55 d(12.0)	ax 2.72 br d(9.6) eq 3.32 dm(12.1)	ax 2.60 d(12.0) eq 3.00 d(12.0)
13	3.20 br d(13) 2.89 dd(12.7, 2.7)	2.87 dd(12.0, 2.4) 2.52 dd(12.0, 2.4)	ax 2.72 br d(9.6) eq 3.26 dt(9.6, 1.8)	ax 2.57 dd(11.1, 2.1) eq 2.90 dm(11.1)
1′		2.12 s		2.97 d(16.2) 2.87 d(16.2)

Table 2-7-29: ^{1}H NMR spectroscopic data of cytisine-type alkaloids **2-7-75**~**2-7-78**.

H	2-7-75	2-7-76	2-7-77	2-7-78
3	6.44 dd(9.1, 1.2)	6.43 dd(9.0, 1.5)	6.42 dd(9.0, 1.4)	6.46 dd(9.0, 1.3)
4	7.28 dd(9.1, 6.8)	7.26 dd(9.0, 6.6)	7.24 dd(9.0, 6.7)	7.27 dd(9.0, 6.8)
5	5.98 dd(6.8, 1.2)	5.98 dd(6.6, 1.5)	5.94 dd(6.7, 1.4)	5.98 dd(6.8, 1.3)
7	2.96 m	2.98 m	–	2.89 m
8	1.88 dd(12.8, 2.8) 1.75 dd(12.8, 1.6)	1.92 m 1.75 m	*α* 1.75 ddd, (12.8, 7.8, 7.8) *β* 1.78 ddd, (12.8, 4.0, 3.8)	*α* 1.77 ddd, (12.6, 7.6, 7.6) *β* 1.81 dd, (12.6, 4.0, 4.0)
9	2.46 m	2.47 m	2.45 m	2.42 m
10	4.01 d(14.8) 3.89 dd(14.8, 6.7)	4.06 d(15.2) 3.89 dd(15.2, 6.7)	*α* 3.91 dd(15.4, 6.8) *β* 4.03 d(15.4)	*α* 3.95 dd(15.3, 6.6) *β* 4.06 d(15.3)
11	2.87 dd(11.0, 1.9) 2.55 dd(11.0, 2.1)	2.89 d(12.5) 2.55 d(12.5)	*α* 2.72 br d(13.2) *β* 2.86 m	*α* – *β* 2.90~2.96 m
13	2.89 d(11.0) 2.52 d(11.0)	2.80 dd(12.5, 1.8) 2.52 dd(12.5, 1.8)	*α* 2.79 m *β* 2.91 m	*α* – *β* 2.90~2.96 m
1′	4.03 dd(12.2, 5.7) 3.84 dd(12.5, 5.7)	3.91 d(12.2) 3.77 d(12.2)	*α* 3.11 d(10.5) *β* 3.21 d(10.5)	*α* 3.19 d(9.3) *β* 3.21 d(9.3)
2′	5.73 m(NH)			
3′			4.16 q(6.8)	4.11 q(7.1)
4′	1.98 s	2.33 m	1.12 d(6.8)	1.20 d(7.1)
5′		1.92 m, 1.80 m		
6′		3.17 m, 3.00 m		

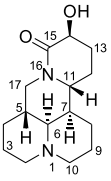
Table 2-7-30: ^{1}H NMR spectroscopic data of cytisine-type alkaloids **2-7-79** and **2-7-80**.

H	2-7-79	2-7-80	H	2-7-79	2-7-80
2	6.85 d(7.1)		10	–	4.76 dt(13.7, 2.0, 2.0)
					2.79 dd(13.0, 2.5)
3	4.93 d(7.1)	2.36 m	11	–	4.26 ddd(8.0, 8.0)
4		1.85~1.90 m	13	–	4.50 ddd(14.0, 3.0, 3.0)
		1.55~1.62 m			2.94 dd(14.0, 2.0)
5	–	2.09~2.15 m	1′	–	2.38 m
		1.76~1.81 m			2.28 m
6	3.60 dt(16.8, 3.8, 3.6)	3.43 ddd(11.0, 2.0, 2.0)	2′	–	5.69 dddd
					(17.0, 10.0, 7.0, 7.0)
7	–	1.61 m	3′	4.79 m	*trans* 5.04 dm(15.0)
					cis 5.02 dm(10.0)
8	–	2.15~2.09 m	4′	5.75 m	
		1.67 dddd(13.0, 2.5, 2.5, 2.5)			
9	–	1.81 m	OMe		3.60 s

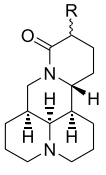
2.7.4 Matrine-type alkaloids

Table 2-7-31: Cos, MFs, and TSs of matrine-type alkaloids **2-7-81**~**2-7-89**.

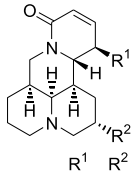
No.	Compounds	MFs	Test solvents	References
2-7-81	(–)-14β-hydroxylsophoridine	$C_{15}H_{24}N_2O_2$	$CDCl_3$	[197]
2-7-82	(–)-14β-acetoxymatrine	$C_{17}H_{26}N_2O_3$	$CDCl_3$	[198]
2-7-83	(+)-14α-acetoxymatrine	$C_{17}H_{26}N_2O_3$	$CDCl_3$	[198]
2-7-84	(–)-14β-hydroxymatrine	$C_{15}H_{24}N_2O_2$	$CDCl_3$	[198]
2-7-85	(–)-12β-hydroxylsophocarpine	$C_{15}H_{22}N_2O_2$	$CDCl_3$	[197]
2-7-86	(–)-9α-hydroxylsophocarpine	$C_{15}H_{22}N_2O_2$	$CDCl_3$	[197]
2-7-87	(–)-sophocarpine	$C_{15}H_{22}N_2O$	$CDCl_3$	[197]
2-7-88	(–)-5α-hydroxysophocarpine	$C_{15}H_{22}N_2O_2$	$CDCl_3$	[199]
2-7-89	7α-hydroxysophoramine	$C_{15}H_{20}N_2O_2$	$CDCl_3$	[200]



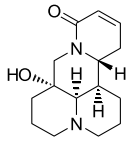
2-7-81



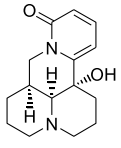
2-7-82 R = β-OAc
2-7-83 R = α-OAc
2-7-84 R = β-OH



	R^1	R^2
2-7-85	OH	H
2-7-86	H	OH
2-7-87	H	H



2-7-88



2-7-89

Table 2-7-32: ^{1}H NMR spectroscopic data of matrine-type alkaloids **2-7-81** and **2-7-85**~**2-7-88**.

H	2-7-81	2-7-85	2-7-86	2-7-87	2-7-88
2	–	–	–	–	2.76 br d(11.2)
9	–	–	*ca*. 3.83	–	–
10	–	–	–	–	2.85 br d(11.5)
11	3.42 m	4.33 dd(11.3, 11.3)	*ca*. 3.83	4.01 dm(11.6)	3.98 dd(18.8, 7.8)
12	–	4.27 dd(11.3, 5.5)	–	–	2.63 ddd (19.5, 5.1, 5.1)
13	–	6.60 dd(9.8, 5.5)	6.47 dm(9.8)	6.46 ddd (17.1, 17.1, 7.9)	6.49 ddd (9.8, 4.4, 4.4)
14	3.95 dd(12.2, 4.9)	6.01 d(9.8)	5.90 d(9.8)	5.89 d(9.9)	5.92 ddd (9.8, 1.7, 1.7)
17α	3.10 dd(12.2, 12.2)	4.22 dd(12.5, 4.3)	4.14 dd(13.0, 4.3)	4.14 dd(12.8, 4.9)	4.15 d(14)
17β	3.58 dd(12.2, 4.3)	3.20 dd(12.5, 2.5)	3.10 dd(13.0, 12.9)	3.09 dd(12.8, 12.8)	3.35 d(14)

Table 2-7-33: ^{1}H NMR spectroscopic data of matrine-type alkaloids **2-7-82**~**2-7-84** and **2-7-89**.

H	2-7-82	2-7-83	2-7-84	2-7-89
2	2.80 dm(12.8)	2.82 dm(11.6)	2.80 dm(13.6)	α 2.62 br d(10.4) β 1.94 t(11.1)
5	–	–	–	2.74 br s
6	–	–	–	1.99 br s
8	–	–	–	2.47 br d(13.6)
10	2.80 dm(12.8)	2.82 dm(11.6)	2.80 dm(13.6)	α 1.94 t(11.1) β 2.72 br s
11	3.82 ddd(10.4, 10.4, 5.5)	3.90 ddd(10.4, 4.9, 4.9)	*ca*. 3.89 m	
12	–	–	–	6.40 dd(7.2, 1.2)
13	–	–	–	7.13 dd(8.9, 7.2)
14	5.20 dd(11.9, 5.5) 2.12 s(OAc)	5.21 dd(5.5, 5.5) 2.11 s(OAc)	*ca*. 3.89 m	6.19 dd(8.9, 1.2)
17	4.32 dd(12.8, 4.9) 3.09 dd(12.8, 12.8)	4.30 dd(12.8, 4.9) 3.13 dd(12.8, 12.8)	4.25 dd(12.8, 4.3) 3.15 dd(12.8, 12.8)	α 3.61 dd(14.2, 13.0) β 3.99 dd(14.2, 7.0)

Bibliography

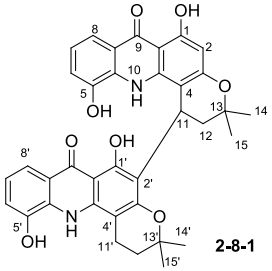
[160] Morita H, Hirasawa Y, Shinzato T, et al. Tetrahedron, 2004, 60: 7015.
[161] Takamatsu S, Saito K, Murakoshi I, et al. J Nat Prod, 1991, 54: 477.
[162] Máximo P, Lourenço A. J Nat Prod, 2000, 63: 201.
[163] Luo YG, Li BG, Zhang GL. Chin Chem Lett, 2001, 12: 337.

[164] Patnam R, Chang FR, Chen CY, et al. J Nat Prod, 2001, 64: 948.
[165] Hirasawa Y, Morita H, Kobayashi J. Tetrahedron, 2003, 59: 3567.
[166] Rumalla CS, Jadhav AN, Smillie T, et al. Phytochemistry, 2008, 69: 1756.
[167] Xie XQ, Rother A, Edwards JM. J Nat Prod, 1995, 58: 1876.
[168] Kim HJ, Lee IS, Youn UJ, et al. J Nat Prod, 2009, 72: 749.
[169] Abdel-Halim OB, El-Gammal AA, Abdel-Fattah H, et al. Phytochemistry, 1999, 51: 5.
[170] Takamatsu S, Saito K, Sekine T, et al. Phytochemistry, 1990, 29: 3923.
[171] Venkateswarlu Y, Reddy MVR, Rao JV. J Nat Prod, 1994, 57: 1283.
[172] Iwagawa T, Kaneko M, Okamura H, et al. J Nat Prod, 2000, 63: 1310.
[173] Kong F, Fanlkner DJ. Tetrahedron Lett, 1991, 32: 3667.
[174] Yoshikawa M, Murakami T, Wakao S, et al. Heterocycles, 1997, 45: 1815.
[175] Yoshikawa M, Murakami T, Ishikado A, et al. Heterocycles, 1997, 46: 301.
[176] Kennelly EJ, Flynn TJ, Mazzola EP, et al. J Nat Prod, 1999, 62: 1385.
[177] Rensen IV, Wray V, Witte L, et al. Phytochemistry, 1994, 35: 421.
[178] Wang YH, Li JS, Jiang ZR, et al. Chem Pharm Bull, 2000, 48: 641.
[179] Abdel-Halim OB, Sekine T, Saito K, et al. Phytochemistry, 1992, 31: 3251.
[180] Mohamed MH, Hassanean HA. Phytochemistry, 1997, 46: 365.
[181] Mohamed MH, Ibraheim ZZ, Abdallah OM, et al. Phytochemistry, 1994, 37: 1751.
[182] Al-Tel TH, Sabri SS, Zarga MHA, et al. Phytochemistry, 1991, 30: 2393.
[183] Veitch NC, Goodwin BL, Kite GC, et al. Phytochemistry, 1997, 45: 847.
[184] Nihei K, Shibata K, Kubo I. Phytochemistry, 2002, 61: 987.
[185] Mohamed MH, Saito K, Murakoshi I, et al. J Nat Prod, 1990, 53: 1578.
[186] Sagen AL, Gertsch J, Becker R, et al. Phytochemistry, 2002, 61: 975.
[187] Saito K, Takamatsu S, Murakoshi I, et al. J Nat Prod, 1989, 52: 1032.
[188] Mohamed MH, El-Shorbagi ANA. J Nat Prod, 1993, 56: 1999.
[189] Verdoorn GH, Wyk BEV. Phytochemistry, 1990, 29: 1297.
[190] Nuzillard JM, Connolly JD, Dclaude C, et al. Tetrahedron, 1999, 55: 11511.
[191] Veen G, Greinwald R, Witte L, et al. Phytochemistry, 1991, 30: 1891.
[192] Wang YH, Li JS, Kubo H, et al. Chem Pharm Bull, 1999, 47: 1308.
[193] Takamatsu S, Saito K, Ohmiya S, et al. Phytochemistry, 1991, 30: 3793.
[194] Rahman AU, Pervin A, Perveen S, et al. Phytochemistry, 1991, 30: 1001.
[195] Mohamed MH, Saito K, Kadry HA, et al. Phytochemistry, 1991, 30: 3111.
[196] Veen G, Schmidt C, Witte L, et al. Phytochemistry, 1992, 31: 4343.
[197] Xiao P, Kubo H, Komiya H, et al. Phytochemistry, 1999, 50: 189.
[198] Xiao P, Kubo H, Komiya H, et al. Chem Pharm Bull, 1999, 47: 448.
[199] Saito K, Arai N, Sekine T, et al. Planta Med, 1990, 56: 487.
[200] Rahman-Atta-ur A, Choudhary MI, Parvez K, et al. J Nat Prod, 2000, 63: 190.

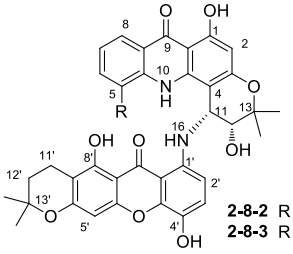
2.8 Acridone alkaloids

Table 2-8-1: Cos, MFs, and TSs of acridone alkaloids **2-8-1**~**2-8-11**.

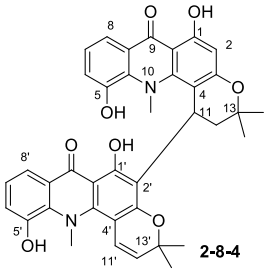
No.	Compounds	MFs	Test solvents	References
2-8-1	oriciacridone F	$C_{36}H_{32}N_2O_8$	CD_3OD	[201]
2-8-2	oriciacridone A	$C_{36}H_{32}N_2O_9$	DMSO-d_6	[202]
2-8-3	oriciacridone B	$C_{36}H_{32}N_2O_{10}$	DMSO-d_6	[202]
2-8-4	bis-5-hydroxynoracronycine	$C_{38}H_{34}N_2O_8$	DMSO-d_6	[203]
2-8-5	neoacrimarine H	$C_{33}H_{28}NO_8$	$CDCl_3$	[204]
2-8-6	glycobismine F	$C_{38}H_{32}N_2O_9$	CD_3COCD_3	[205]
2-8-7	glycobismine E	$C_{39}H_{34}N_2O_9$	$CDCl_3$	[206]
2-8-8	glycobismine G	$C_{38}H_{32}N_2O_9$	DMSO-d_6	[205]
2-8-9	buxifoliadine D	$C_{23}H_{23}NO_3$	CD_3COCD_3	[207]
2-8-10	1-oxo-2-hydroxy-1,2-dihydro-12-desmethyl-noracronycine	$C_{18}H_{15}NO_5$	DMSO-d_6	[208]
2-8-11	5-hydroxynoracronycine alcohol	$C_{19}H_{17}NO_5$	CD_3COCD_3	[209]



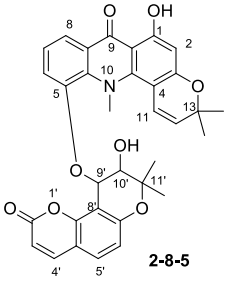
2-8-1



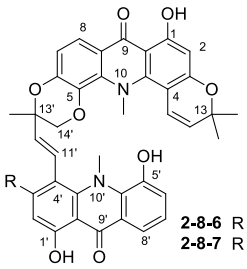
2-8-2 R = H
2-8-3 R = OH



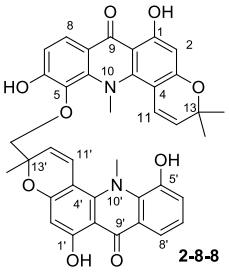
2-8-4



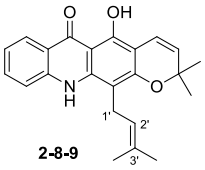
2-8-5



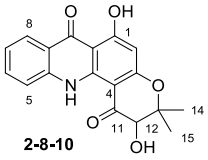
2-8-6 R = OH
2-8-7 R = OMe



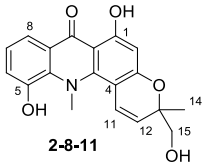
2-8-8



2-8-9



2-8-10



2-8-11

Table 2-8-2: ^{1}H NMR spectroscopic data of acridone alkaloids **2-8-1**~**2-8-5**.

H	2-8-1	2-8-2	2-8-3	2-8-4	2-8-5
1	14.86 s(OH)①	14.40 s(OH)	14.60 s(OH)	13.78 s(OH)	14.08 s(OH)
2	6.28 s	5.98 s	5.99 s	5.87 s	6.21 s
5	10.10 br s(OH)①	7.82 td(8.7, 1.5)	10.59 br s(OH)	9.24 or 10.14br s(OH)	
6	6.68 dd(7.5, 1.3)	7.78 br d(5.7)	7.17 d(9.0)	7.00 dd(1.8, 7.7)	7.78 dd(7.9, 1.2)
7	6.95 t(7.9)	7.30 ddd (6.8, 5.6, 2.3)	7.22 d(9.0)	6.97 t(7.7)	7.38 t(7.9)
8	7.55 dd(7.9, 1.3)	8.14 br d(7.8)	7.57 dd(9.0, 3.0)	7.50 dd(1.8, 7.7)	8.12 dd(7.9, 1.2)
10		10.70 br s(NH)	10.42 br s(NH)	3.28 s(NMe)	3.46 s(NMe)
11	5.31 dd(12.0, 7.1)	5.29 dd(3.7, 3.8)	5.72 m	4.66 dd(7.0, 12.5)	5.89 d(9.2)
12	2.29 dd(13.8, 7.1) 2.14 dd(13.8, 12.0)	4.30 d(3.7) 4.14 br s(OH)	4.13 d(3.9) 10.72 br s(OH)	1.79 dd(7.0, 13.6) 1.53 dd(12.5, 13.6)	5.34 d(9.2)
14, 15	1.45 s, 1.34 s	1.42 s, 1.62 s	1.39 s, 1.62 s	1.22 s, 1.15 s	1.44 s,1.43 s
16		9.56 d(5.2, NH)	9.28 m(NH)		
1′	14.35 s(OH)①			15.14 s(OH)	
2′		6.50 d(8.8)	6.58 d(11.4)		
3′		7.13 d(8.8)	7.18 d(11.4)		5.96 d(9.2)
4′		11.32 br s(OH)	10.84 br s(OH)		7.47 d(9.2)
5′	10.10 br s(OH)①	6.28 s	6.30 s	9.24 or 10.14 br s(OH)	7.37 d(8.5)
6′	6.64 dd(7.5, 0.9)			6.58 dd(1.1, 7.7)	6.87 d(8.5)
7′	6.93 t(7.5)			6.70 t(7.7)	
8′	7.52 dd(8.2, 0.9)	13.15 s(OH)	13.12 s(OH)	7.18 dd(1.1, 7.7)	
9′					5.77 d(4.9)
10′				3.20 s(NMe)	4.07 dd(7.9, 4.9) 2.78 d(7.9, OH)
11′	2.99 m, 2.92 m	2.54 m	2.57 m	6.07 d(9.9)	1.61 s(Me), 1.53 s(Me)
12′	1.88 m, 1.84 m	1.48 m	1.45 m	4.94 d(9.9)	
14′,15′	1.39 s, 1.51 s	1.14 s, 1.14 s	1.18 s, 1.18 s	0.86 s, 0.74 s	1.44 s, 1.43 s

① Recorded in DMSO-d_6 as solvent.

Table 2-8-3: ^{1}H NMR spectroscopic data of acridone alkaloids **2-8-6**~**2-8-10**.

H	2-8-6	2-8-7	2-8-8	2-8-9	2-8-10
1	14.49 s(OH)	14.15 s(OH)	14.49 s(OH)	14.80 s(OH)	15.43 s(OH)
2	6.09 s	6.25 s	6.03 s		6.10 s
5				7.70 dd(4.0, 1.2)	7.88 d(8)
6			10.76 s(OH)	7.70 dd(4.0, 1.2)	7.85 t(8)
7	7.05 d(8.8)	7.11 d(8.8)	7.00 d(8.3)	7.28 dt(8.0, 4.0)	7.45 t(8)
8	7.85 d(8.8)	7.98 d(8.8)	7.82 d(8.3)	8.26 d(8.0)	8.23 d(8)
10	3.79 s(NMe)	3.68 s(NMe)	3.61 s(NMe)	9.84 s(NMe)	12.88 s(NH)
11	6.70 d(9.8)	6.64 d(9.9)	6.23 d(9.8)	6.74 d(10.0)	

Table 2-8-3 (continued)

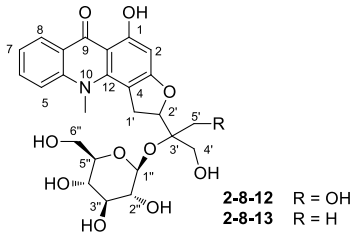
H	2-8-6	2-8-7	2-8-8	2-8-9	2-8-10
12	5.66 d(9.8)	5.53 d(9.9)	5.26 d(9.8)	5.67 d(10.0)	4.27 d(5) 6.19 d(5, OH)
14, 15	1.46 s, 1.49 s	1.58 s	1.27 s, 1.29 s	1.46 br s, 1.46 br s	1.33 s, 1.47 s
1′	14.35 s(OH)	14.34 s(OH)	14.49 s(OH)	3.53 d(6.8)	
2′	6.25 s	6.34 s	6.05 s	5.15 br t(6.8)	
3′	6.65 s(OH)	3.91 s(OMe)			
4′				1.87 br s	
5′		6.94 br(OH)	10.46 s(OH)	1.68 br s	
6′	7.25 dd(7.8, 1.0)	7.02 br d(7.7)	7.27 dd(7.8, 1.5)		
7′	7.15 t(7.8)	7.11 t(7.7)	7.23 t(7.8)		
8′	7.74 t(7.8, 1.0)	7.80 br d(7.7)	7.69 dd(7.8, 1.5)		
10′	3.47 s(NMe)	2.98 s(NMe)	3.73 s(NMe)		
11′	6.86 d(16.1)	6.57 d(16.1)	6.85 d(10.3)		
12′	6.53 d(16.1)	6.36 d(16.1)	5.75 d(10.3)		
13′-Me	1.65 s	1.47 s	1.61 s		
14′	4.25 d(11.2)	4.42 d(11.0)	4.04 d(10.8)		
	4.57 d(11.2)	4.07 d(11.0)	4.39 d(10.8)		

Table 2-8-4: ^{1}H NMR spectroscopic data of acridone alkaloid **2-8-11**.

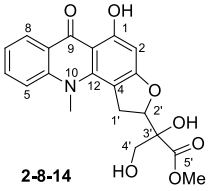
H	2-8-11	H	2-8-11
1	14.42 br(OH)	10	3.84 s(NMe)
2	6.10 s	11	6.82 d(10)
6	7.33 d(7.6)	12	5.66 d(10)
7	7.21 t(7.6)	14	1.47 s
8	7.78 d(7.6)	15	3.64 d(12), 3.76 d(12)

Table 2-8-5: Cos, MFs, and TSs of acridone alkaloids **2-8-12**~**2-8-23**.

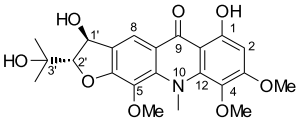
No.	Compounds	MFs	Test solvents	References
2-8-12	gravacridontriol-*O*-18-*β*-D-glucoside	$C_{25}H_{29}NO_{11}$	CD_3OD	[210]
2-8-13	gravacridondiol-*O*-18-*β*-D-glucoside	$C_{25}H_{29}NO_{10}$	CD_3OD	[210]
2-8-14	rhodesiacridone	$C_{20}H_{19}NO_7$	C_5D_5N	[211]
2-8-15	glycocitrine V	$C_{22}H_{25}NO_8$	$CDCl_3$	[206]
2-8-16	rutacridon epoxide	$C_{19}H_{17}NO_4$	$CDCl_3$	[212]
2-8-17	rutacridone	$C_{19}H_{17}NO_3$	$CDCl_3$	[212]
2-8-18	oriciacridone C	$C_{18}H_{15}NO_4$	CD_3OD	[201]
2-8-19	oriciacridone D	$C_{18}H_{15}NO_4$	CD_3OD	[201]
2-8-20	buxifoliadine E	$C_{23}H_{25}NO_5$	CD_3COCD_3	[207]
2-8-21	buxifoliadine F	$C_{16}H_{11}NO_4$	CD_3COCD_3	[207]
2-8-22	citbismine F	$C_{36}H_{34}N_2O_{10}$	DMSO-d_6	[213]
2-8-23	buxifoliadine G	$C_{22}H_{21}NO_4$	CD_3COCD_3	[207]



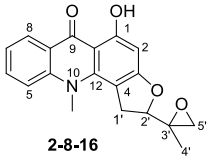
2-8-12 R = OH
2-8-13 R = H



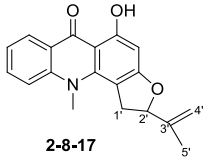
2-8-14



2-8-15



2-8-16



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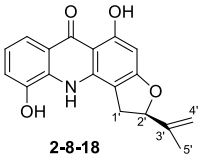

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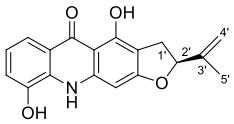

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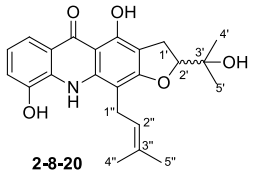
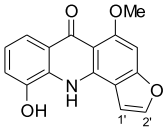


2-8-20



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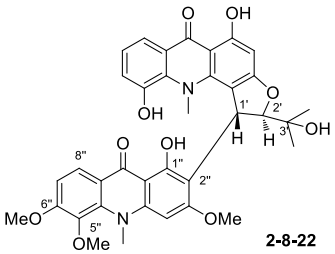
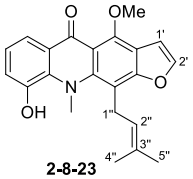


2-8-22



2-8-23

Table 2-8-6: ^{1}H NMR spectroscopic data of acridone alkaloids **2-8-12**~**2-8-16**.

H	2-8-12	2-8-13	2-8-14	2-8-15	2-8-16
1				14.05 s(OH)	14.85 s(OH)
2	6.18 s	6.16 s	6.58 s	6.37 s	6.22 s
3				3.95 s(OMe)	
4				3.77 s(OMe)	
5	7.65 d(8.5)	7.64 d(8.5)	7.32 dd(8.8, 1.6)	3.99 s(OMe)	7.40 d(8.8)
6	7.79 dd(8.5, 8.5)	7.79 dd(8.5, 8.5)	7.64 ddd (8.6, 8.0, 1.6)		7.71 ddd (8.8, 7.4, 1.3)
7	7.31 dd(7.9, 8.5)	7.31 dd(7.9, 8.5)	7.26 ddd (8.8, 7.9, 1.6)		7.29 dd(7.9, 7.4)

Table 2-8-6 (continued)

H	2-8-12	2-8-13	2-8-14	2-8-15	2-8-16
8	8.30 d(7.9)	8.30 d(7.9)	8.62 dd(7.9, 1.6)	8.11 s	8.38 dd(7.9, 1.3)
1′	3.74 dd(9.5, 14.6)	3.74 dd(9.1, 14.7)	4.11 dd(14.2, 7.6)	5.48 d(4.4)	3.69 m
	4.17 dd(8.8, 14.6)	3.95 dd(8.7, 14.7)	3.90 dd(14.2, 9.5)		
2′	5.16 t(9.5)	5.02 t(9.1)	5.69 dd(9.5, 7.9)	4.52 d(4.4)	4.94 t(8.9)
4′	4.01 d(12.3)	3.85 d(11.6)	4.60 dd(10.7, 4.4)	1.36 s	1.39 s
	3.88 d(12.3)	3.69 d(11.6)	4.66 dd(10.7, 4.4)		
5′	3.87 d(12.0)	1.39 s	3.79 s(COOMe)	1.40 s	3.69 m
	3.86 d(12.0)				
1″	4.88 d(7.8)	4.70 d(7.8)			
2″	3.19 dd(8.0, 9.2)	3.20 dd(8.0, 9.2)			
3″	3.27 (ov)	3.35[①]			
4″	3.27 (ov)	3.30[①]			
5″	3.27 (ov)	3.42[①]			
6″	3.77 d(11.7)	3.89 d(12.0)			
	3.64 dd(11.7, 5.3)	3.64 dd(12.0, 5.1)			
NMe	4.06 s	4.06 s	3.66 s	3.77 s	3.99 s

[①] Overlayed by residual signal of MeOD, values obtained from HMBC and HMQC spectra.

Table 2-8-7: ^{1}H NMR spectroscopic data of acridone alkaloids **2-8-17**~**2-8-21**.

H	2-8-17	2-8-18	2-8-19	2-8-20	2-8-21
1	15.20 s(OH)	14.50 s(OH)	14.53 s(OH)	14.49 s(OH)	4.13 s(OMe)
2	6.09 s	6.12 s			6.56 s
4			6.12 s		
5	7.23 br d(8.4)	10.10 s(OH)	9.98 br s(OH)	9.82 s(OH)	12.43 s(OH)
6	7.58 m	7.12 dd(7.9, 1.2)	7.06 dd(7.6, 2.4)	7.20 dd(8.0, 1.2)	7.49 dd(7.8, 1.6)
7	7.13 m	7.10 t(7.9)	7.08 t(7.9)	7.08 t(8.0)	7.36 dd(8.0, 7.8)
8	8.21 ddd (8.0, 1.2, 0.6)	7.71 dd(7.7, 1.2)	7.92 dd(6.1, 2.4)	7.74 dd(8.0, 1.2)	8.04 dd(8.0, 1.6)
1′	3.59 m	3.13 dd(14.5, 7.6)	3.05 dd(14.2, 6.9)	3.16 dd(15.2, 9.2)	6.93 d(3.6)
		3.51 dd(14.5, 9.7)	3.39 dd(14.2, 9.8)	3.21 dd(15.2, 8.0)	
2′	5.11 m	5.40 dd(9.7, 7.6)	5.38 dd(9.8, 6.9)	4.79 dd(9.2, 8.0)	8.70 d(3.6)
3′				3.82 s(OH)	
4′	5.10 m, 4.95 m	4.94 s, 5.12 s	4.95 s, 5.11 s	1.28 br s	
5′	1.78 s	1.79 s	1.72 s	1.25 br s	
1″				3.55 d(6.8)	
2″				5.21 m	
4″				1.99 br s	
5″				1.68 br s	
NMe	3.80 s(Me)				
NH				9.02 s	10.09 s

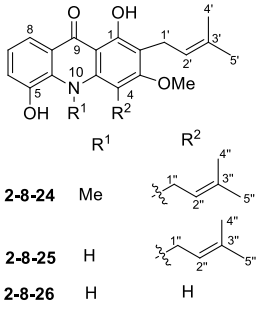
Table 2-8-8: ^{1}H NMR spectroscopic data of acridone alkaloids **2-8-22** and **2-8-23**.

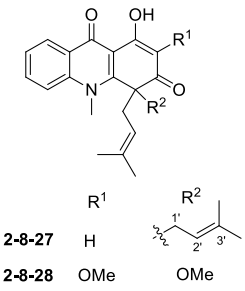
H	2-8-22	2-8-23	H	2-8-22	2-8-23
1	14.86 s(OH)	4.24 s(OMe)	1″	15.62 s(OH)	3.69 d(6.8)
2	6.18 s		2″		5.39 br t(6.8)
5	9.97 s(OH)	9.22 s(OH)	3″	3.52 s(OMe)	
6	6.94 br d(7.7)	7.23 dd(8.0, 1.8)	4″	6.38 s	1.80 br s
7	7.03 t(7.7)	7.15 t(8.0)	5″	3.70 s(OMe)	1.68 br s
8	7.56 dd(1.1, 7.7)	7.75 dd(8.0, 1.8)	6″	3.97 s(OMe)	
10	3.79 s(OMe)	3.63 s(OMe)	7″	7.25 d(9.2)	
1′	5.57 d(5.1)	7.24 d(2.0)①	8″	8.12 d(9.2)	
2′	4.35 d(5.1)	7.90 d(2.0)	10″	3.91 s(NMe)	
3′	4.73 s(OH)				
4′, 5′	1.27 s, 1.22 s				

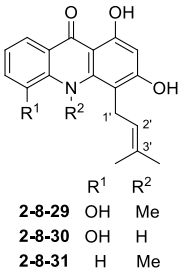
① Typographic errors exist in the literature.

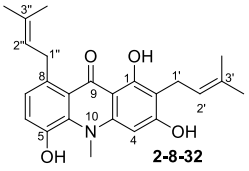
Table 2-8-9: Cos, MFs, and TSs of acridone alkaloids **2-8-24**~**2-8-33**.

No.	Compounds	MFs	Test solvents	References
2-8-24	buxifoliadine A	$C_{25}H_{29}NO_4$	CD_3COCD_3	[207]
2-8-25	buxifoliadine B	$C_{24}H_{27}NO_4$	CD_3COCD_3	[207]
2-8-26	buxifoliadine C	$C_{19}H_{19}NO_4$	CD_3COCD_3	[207]
2-8-27	glycocitrine VI	$C_{24}H_{27}NO_3$	$CDCl_3$	[206]
2-8-28	megistophylline I	$C_{21}H_{23}NO_5$	$CDCl_3$	[214]
2-8-29	oriciacridone E	$C_{19}H_{19}NO_4$	CD_3OD	[201]
2-8-30	1,3,5-trihydroxyl-4-prenylacridone	$C_{18}H_{17}NO_4$	CD_3OD	[201]
2-8-31	glycocitrine II	$C_{19}H_{19}NO_3$	DMSO-d_6	[215]
2-8-32	1,3,5-trihydroxy-2,8-bis(3-methylbut-2-enyl)-10-methyl-9-acridone	$C_{24}H_{26}NO_4$	DMSO-d_6	[216]
2-8-33	glycocitrine IV	$C_{20}H_{21}NO_5$	CD_3COCD_3	[206]

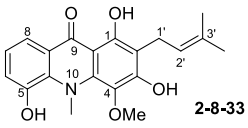








2-8-32



2-8-33

Table 2-8-10: ^{1}H NMR spectroscopic data of acridone alkaloids **2-8-24**~**2-8-28**.

H	2-8-24	2-8-25	2-8-26	2-8-27	2-8-28
1	14.38 s(OH)	14.47 s(OH)	14.49 s(OH)	16.07 s(OH)	16.22 s(OH)
2				5.67 s	3.84 s(OMe)
3	3.84 s(OMe)	3.66 s(OMe)	3.92 s(OMe)		
4			6.88 s		3.23 s(OMe)
5	9.23 s(OH)	9.80 s(OH)	9.62 s(OH)	7.75 br d(7.8)	7.85 d(8.3)
6	7.28 dd(8.0, 1.6)	7.23 dd(8.0, 1.2)	7.18 dd(7.8, 1.2)	7.86 br t(7.8)	7.94 td(8.3, 1.5)
7	7.16 t(8.0)	7.11 t(8.0)	7.07 t(7.8)	7.60 br t(7.8)	7.65 t(8.3)
8	7.78 dd(8.0, 1.6)	7.78 dd(8.0, 1.2)	7.78 dd(8.0, 1.6)	8.54 dd(1.5, 7.8)	8.57 dd(8.3, 1.5)
1′	3.39 d(6.8)	3.40 d(6.1)	3.34 d(6.1)	3.29 dd(15.1, 6.4)	2.93 dd(14.5, 8.3)
				2.94 dd(15.1, 7.3)	3.18 dd(14.5, 8.3)
2′	5.28 br t(6.8)	5.22 br t(6.1)	5.24 br t(6.1)	4.68 m	4.78 t(8.3)
4′, 5′	1.65 br s,	1.78 br s,	1.78 br s,	1.50 s, 1.47 s	1.38 s, 1.64 s
	1.75 br s	1.65 br s	1.63 br s		
1″	3.64 d(6.2)	3.66 d(6.1)		3.29 dd(15.1,6.4)	
				2.94 dd(15.1,7.3)	
2″	5.33 br t(6.2)	5.29 br t(6.1)		4.68 m	
4″, 5″	1.66 br s,	2.00 br s,		1.50 s, 1.47 s	
	1.79 br s	1.80 br s			
NMe	3.71 s			4.22 s	4.52 s
NH		9.11 s	10.23 s		

Table 2-8-11: ^{1}H NMR spectroscopic data of acridone alkaloids **2-8-29**~**2-8-33**.

H	2-8-29	2-8-30	2-8-31	2-8-32	2-8-33
1	14.08 s(OH)	14.10 s(OH)	14.78 s(OH)	15.07 s(OH)	14.18 s(OH)
2	6.20 s	6.19 s	6.33 s		
3	10.78 br s(OH)	10.72 br s(OH)			6.67 s(OH)
4				6.28 s	3.81 s(OMe)
5	10.78 br s(OH)	10.72 br s(OH)	7.55 d(9)		6.21 br(OH)
6	7.20 d(2.5)	7.17 d(2.5)	7.73 t(9)	7.07 d(8.2)	7.10 br
7	7.11 d(8.3)	7.10 d(8.2)	7.26 t(9)	6.84 d(8.2)	7.10 br
8	7.60 dd(8.3, 2.5)	7.59 dd(8.2, 2.5)	8.17 d(9)		7.92 br
1′	3.48 d(8.4)	3.50 d(8.2)	3.37 br d(4)	3.21 d(6.57)	3.43 d(7.3)
2′	5.13 t(8.3)	5.10 t(8.2)	5.33 br s	5.21 br tr(6.83)①	5.33 m
4′, 5′	1.79 s, 1.98 s	1.78 s, 1.98 s	1.73 s, 1.75 s	1.73 s, 1.61 s	1.71 s, 1.83 s
1″				3.93 d(6.96)	

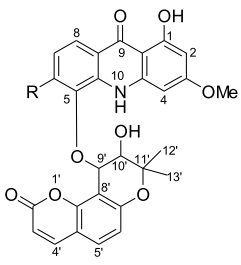
Table 2-8-11 (continued)

H	2-8-29	2-8-30	2-8-31	2-8-32	2-8-33
2″				5.33 br tr(6.87)[①]	
3″				1.64 s, 1.68 s	
NMe	3.63 s		3.77 s	3.77 s	3.80 s

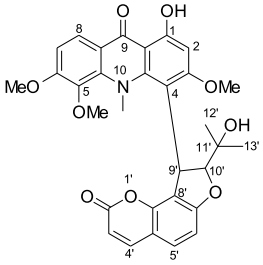
[①] The peaktype of br tr was given in the literature.

Table 2-8-12: Cos, MFs, and TSs of acridone alkaloids **2-8-34**~**2-8-52**.

No.	Compounds	MFs	Test solvents	References
2-8-34	neoacrimarine I	$C_{29}H_{25}NO_9$	DMSO-d_6	[204]
2-8-35	neoacrimarine J	$C_{28}H_{23}NO_9$	$CDCl_3$	[204]
2-8-36	neoacrimarine K	$C_{31}H_{29}NO_9$	$CDCl_3$	[204]
2-8-37	neoacrimarine F	$C_{29}H_{25}NO_9$	DMSO-d_6	[217]
2-8-38	neoacrimarine G	$C_{29}H_{25}NO_8$	$CDCl_3$	[217]
2-8-39	glycocitrine III	$C_{24}H_{27}NO_4$	$CDCl_3$	[218]
2-8-40	megistophylline II	$C_{26}H_{31}NO_5$	$CDCl_3$	[214]
2-8-41	glycobismine D	$C_{35}H_{30}N_2O_{10}$	CD_3COCD_3	[206]
2-8-42	1,3-dihydroxy-4-[(*Z*)-3′-hydroxy-3′-methyl-buten-1′-yl]-*N*-methylacridone	$C_{19}H_{19}NO_4$	$CDCl_3$	[215]
2-8-43	1,3-dihydroxy-4-(2′-hydroxy-3′-hydroxymethyl-3′,4′-epoxy-butyl)-*N*-methylacridone	$C_{19}H_{19}NO_6$	DMSO-d_6	[215]
2-8-44	toddaliopsin A	$C_{16}H_{15}NO_4$	$CDCl_3$	[219]
2-8-45	toddaliopsin B	$C_{19}H_{19}NO_6$	$CDCl_3$	[219]
2-8-46	toddaliopsin C	$C_{18}H_{17}NO_6$	$CDCl_3$	[219]
2-8-47	toddaliopsin D	$C_{18}H_{19}NO_5$	$CDCl_3$	[219]
2-8-48	buxifoliadine H	$C_{16}H_{15}NO_6$	CD_3COCD_3	[207]
2-8-49	1-hydroxy-10-methylacridone	$C_{14}H_{11}NO_2$	$CDCl_3$	[212]
2-8-50	arborinine	$C_{16}H_{15}NO_4$	$CDCl_3$	[212]
2-8-51	1-hydroxy-3-methoxy-9-(10-methyl)-acridone	$C_{15}H_{13}NO_3$	–	[220]
2-8-52	1-methoxy-10-methylacridan-9-one	$C_{15}H_{13}NO_2$	$CDCl_3$	[221]



2-8-34 R = OMe
2-8-35 R = OH



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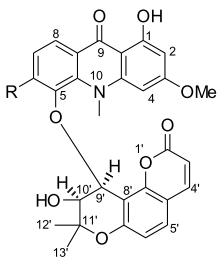
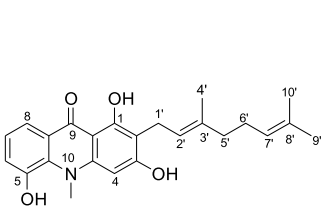


2-8-37 R = OH
2-8-38 R = H



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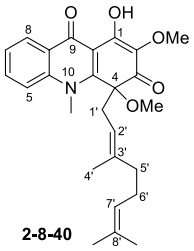
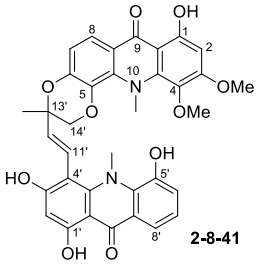
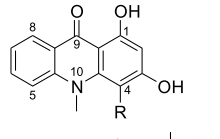


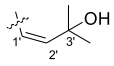
2-8-40



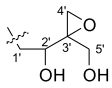
2-8-41

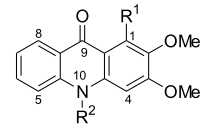


2-8-42 R =

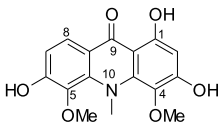


2-8-43 R =

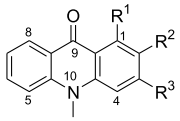




	R^1	R^2
2-8-44	OMe	H
2-8-45	OMe	CH_2OAc
2-8-46	OH	CH_2OAc
2-8-47	OMe	CH_2OMe



2-8-48



	R^1	R^2	R^3
2-8-49	OH	H	H
2-8-50	OH	OMe	OMe
2-8-51	OH	H	OMe
2-8-52	OMe	H	H

Table 2-8-13: ^{1}H NMR spectroscopic data of acridone alkaloids **2-8-34**~**2-8-38**.

H	2-8-34	2-8-35	2-8-36	2-8-37	2-8-38
1	14.31 s(OH)	14.13 s(OH)	14.36 s(OH)	14.54 s(OH)	14.44 s(OH)
2	6.16 d(2.2)	6.23 s	6.30 s	6.11 d(1.5)	6.26 d(2.2)
3	3.88 s(OMe)	3.83 s(OMe)	3.57 s(OMe)	3.76 s(OMe)	3.84 s(OMe)
4	6.43 d(2.2)	6.23 s		5.57 d(1.5)	5.90 d(2.2)
5			3.98 s(OMe)		
6	3.42 s(OMe)		4.01 s(OMe)	10.30 s(OH)	7.70 br d(8.1)
7	6.96 d(9.2)	6.93 d(8.6)	6.98 d(8.6)	6.95 d(8.8)	7.31 t(8.1)
8	7.92 d(9.2)	8.12 d(8.5)	8.07 d(8.6)	7.93 d(8.8)	8.22 dd(8.1, 1.1)
3′	6.17 d(9.2)	6.33 d(9.2)	6.10 d(9.2)	5.54 d(9.5)	5.88 d(9.5)
4′	7.97 d(9.2)	7.70 d(9.2)	7.60 d(9.2)	7.64 d(9.5)	7.44 d(9.5)
5′	7.61 d(8.8)	7.48 d(8.6)	7.28 d(8.5)	7.64 d(8.8)	7.35 d(8.8)
6′	6.85 d(8.8)	6.92 d(8.5)	6.80 d(8.5)	6.91 d(8.8)	6.84 d(8.8)
9′	5.45 d(3.7)	5.43 d(4.3)	5.49 d(7.3)	5.13 d(3.7)	5.65 d(4.8)
10′	4.24 t(3.7)	3.86 d(4.3)	4.46 d(7.3)	4.17 d(3.7)	4.11 dd(4.8, 9.2)
					2.66 d(9.2, OH)
12′, 13′	1.55 s, 1.61 s	1.59 s, 1.53 s	1.27 s, 1.05 s	1.53 s, 1.64 s	1.57 s, 1.59 s
NMe			4.16 s	3.18 s	3.56 s
NH	12.15 s	10.13 s			

Table 2-8-14: ^{1}H NMR spectroscopic data of acridone alkaloids **2-8-39**~**2-8-43**.

H	2-8-39	2-8-40	2-8-41	2-8-42	2-8-43
1	14.89 s(OH)	16.20 s(OH)	14.21 s(OH)	14.67 s(OH)	15.28 s(OH)
2		3.85 s(OMe)	6.37 s	6.26 s	6.18 s
3	6.27 s(OH)		3.95 s(OMe)		
4	6.30 s	3.23 s(OMe)	3.75 s(OMe)		
5		7.85 d(8.3)		7.43 d(9)	7.71 br d(9)
6	7.08 d(8.1)	7.92 td(8.3, 1.5)		7.71 m	7.82 ddd(2, 8, 9)
7	7.07 t(8.1)	7.65 t(8.3)	6.99 d(8.9)	7.30 m	7.33 t-like(8)
8	8.03 d(8.1)	8.55 dd(8.3, 1.5)	7.82 d(8.9)	8.38 dd(2, 8)	8.24 dd(2, 8)
1′	3.52 d(7.3)	2.97 dd(14.5, 8.3)	14.36 s(OH)	6.55 d(10)	3.72 dd(9, 14)
		3.09 dd(14.5, 8.3)			3.77 dd(8, 14)
2′	5.34 m	4.81 t(8.3)	6.26 s	5.50 d(10)	4.94 t-like(9)
4′	1.85 s	1.34 s		1.53 s	3.82 d(1)
5′	2.11 m	1.83 s		1.53 s	3.49 d(11)
					3.55 d(11)
6′	2.11 m	1.83 br s	7.25 br d(7.9)		
7′	5.07 m	4.93 br s	7.15 t(7.9)		
8′			7.73 br d(7.9)		
9′, 10′	1.60 s, 1.68 s	1.62 s, 1.51 s			
11′			6.86 d(16.5)		
12′			6.54 d(16.5)		
13′			1.65 s(Me)		
14′			4.54 d(11.3)		
			4.23 d(11.3)		
NMe	3.98 s	4.52 s	3.74 s	3.90 s	4.00 s
N'Me			3.48 s		

Table 2-8-15: ^{1}H NMR spectroscopic data of acridone alkaloids **2-8-44**~**2-8-48**.

H	2-8-44	2-8-45	2-8-46	2-8-47	2-8-48
1	4.00 s(OMe)	4.01 s(OMe)		3.90 s(OMe)	14.15 s(OH)
2	3.84 s(OMe)	3.99 s(OMe)	3.90 s(OMe)	4.01 s(OMe)	6.21 s
3	3.74 s(OMe)	4.01 s(OMe)	3.99 s(OMe)	4.00 s(OMe)	9.13 s(OH)
4	6.75 s	6.88 s	6.59 s	6.80 s	3.79 s(OMe)
5	7.53[①]	7.50 d(8.6)	7.58 d(8.6)	7.49 d(8.6)	3.86 s(OMe)
6	7.53[①]	7.65 dd(8.6, 7.0)	7.73 dd(8.6, 7.1)	7.62 dd(8.6, 7.1)	9.13 s(OH)
7	7.17 m	7.30 dd(8.1, 7.0)	7.34 dd(8.1, 7.1)	7.26 dd(8.1, 7.1)	6.94 d(9.2)
8	8.41 d(8.1)	8.43 d(8.1)	8.41 d(8.1)	8.43 d(8.1)	7.93 d(9.2)
NH	10.39 br s				
NMe					3.81 s
NCH_2		6.29 br s	6.33 br s	5.52 s	
OAc		2.20 s	2.19 s		
OMe				3.58 s	

[①] The peaktype was not given in the literature.

Table 2-8-16: ^{1}H NMR spectroscopic data of acridone alkaloids **2-8-49**~**2-8-52**.

H	2-8-49	2-8-50	2-8-51	2-8-52
1		15.0 br s(OH)		4.03 s(OMe)
2	6.67 d(8.4)	6.27 s	6.33 s	6.72 br d(8.2)
3	7.55 dd(8.4, 8.4)	4.15 or 4.19 s(OMe)	3.90 s(OMe)	7.05 br d(8.7)①
4	6.84 d(8.4)	4.15 or 4.19 s(OMe)	6.33 s	7.59 br t(8.5)①
5	7.50 d(8.4)	7.59 d(8.8)	7.48 d(8.7)	7.43 br d(8.6)
6	7.73 ddd(8.4, 6.8, 1.6)	7.86 dd(8.8, 7.4)	7.72 dtd(1.6, 1.7, 8.7)	7.66 td(7.0, 1.7)
7	7.28 dd(8.4, 6.8)	7.39 dd(8.0, 7.4)	7.29 t(0.6, 7.2)	7.26 td(7.9, 0.7)
8	8.44 dd(8.4, 1.6)	8.43 d(8.0)	8.46 dd(1.6, 1.7)	8.52 dd(8.0, 1.6)
NMe	3.89 s	3.88 s	3.78 s	3.84 s

① Typographic errors exist in the literature, assigning both of the data to H-3.

Bibliography

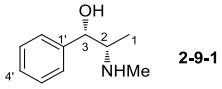
[201] Wansi JD, Wandji J, Meva'a LM, et al. Chem Pharm Bull, 2006, 54: 292.
[202] Wansi JD, Wandji J, Waffo AFK, et al. Phytochemistry, 2006, 67: 475.
[203] Takemura Y, Wada M, Ju-ichi M, et al. Chem Pharm Bull, 1998, 46: 693.
[204] Takemura Y, Takaya J, Okamura Y, et al. Chem Pharm Bull, 1998, 46: 1518.
[205] Negi N, Jinguji Y, Ushijima K, et al. Chem Pharm Bull, 2004, 52: 362.
[206] Ito C, Kondo Y, Wu TS, et al. Chem Pharm Bull, 2000, 48: 65.
[207] Wu TS, Chen CM. Chem Pharm Bull, 2000, 48: 85.
[208] Minh NT, Michel S, Tillequin F, et al. Z Naturforsch, 2003, 58b: 1234.
[209] Teng WY, Huang YL, Shen CC. J Chin Chem Soc, 2005, 52: 1253.
[210] Kuzovkina I, Al'terman I, Schneider B. Phytochemistry, 2004, 65: 1095.
[211] Ahua KM, Ioset JR, Ransijn A, et al. Phytochemistry, 2004, 65: 963.
[212] Wu TS, Shi LS, Wang JJ, et al. J Chin Chem Soc, 2003, 50: 171.
[213] Takemura Y, Ju-ichi M, Omura M, et al. Heterocycles, 1999, 51: 851.
[214] Papageorgiou M, Fokialakis N, Mitaku S, et al. J Nat Prod, 2000, 63: 385.
[215] Chaya N, Terauchi K, Yamagata Y, et al. Biol Pharm Bull, 2004, 27: 1312.
[216] Weniger B, Um BH, Valentin A, et al. J Nat Prod, 2001, 64: 1221.
[217] Takemura Y, Kuwahara J, Ju-ichi M, et al. Heterocycles, 1997, 45: 1411.
[218] Ito C, Kondo Y, Rao KS, et al. Chem Pharm Bull, 1999, 47: 1579.
[219] Naidoo D, Coombes PH, Mulholland DA, et al. Phytochemistry, 2005, 66: 1724.
[220] Simpson DS, Jacobs H. Biochem Syst Ecol, 2005, 33: 841 (supporting information).
[221] Gibbons S, Craven L, Dunlop C, et al. Phytochemistry, 1997, 44: 1109.

2.9 Amphetamine alkaloids

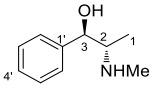
2.9.1 Amine-type amphetamine alkaloids

Table 2-9-1: Cos, MFs, and TSs of amine-type amphetamine alkaloids **2-9-1** and **2-9-2**.

No.	Compounds	MFs	Test solvents	References
2-9-1	*d*-pseudoephedrine	$C_{10}H_{15}NO$	D_2O	[222]
2-9-2	ephedrine	$C_{10}H_{15}NO$	D_2O	[222]



2-9-1



2-9-2

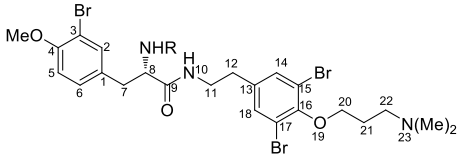
Table 2-9-2: ^{1}H NMR spectroscopic data of amine-type amphetamine alkaloids **2-9-1** and **2-9-2**.

H	2-9-1	2-9-2	H	2-9-1	2-9-2
1	0.92 d(6.4)	0.84 d(6.6)	Ar-H	7.10~7.50 m(5H)	7.2~8.0 m(5H)
2	2.82 m	2.83 m	OH, NH	2.50~2.70 br(2H, NH, OH)	1.90~2.30 br(1H, NH)
3	4.19 d(8.0)	4.80 d(3.8)	NCH_3	2.42 s	2.51 s

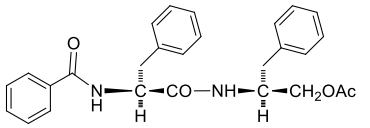
2.9.2 Amide-type amphetamine alkaloids

Table 2-9-3: Cos, MFs, and TSs of amide-type amphetamine alkaloids **2-9-3**~**2-9-6**.

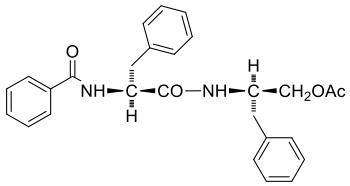
No.	Compounds	MFs	Test solvents	References
2-9-3	suberedamine A	$C_{23}H_{30}Br_3N_3O_3$	CD_3OD	[223]
2-9-4	suberedamine B	$C_{24}H_{32}Br_3N_3O_3$	CD_3OD	[223]
2-9-5	*O*-acetate of *N*-(*N*′-benzoyl-*S*-phenylalaninyl)-*S*-phenylalaninol	$C_{27}H_{28}N_2O_4$	$CDCl_3$	[224]
2-9-6	*O*-acetate of *N*-(*N*′-benzoyl-*S*-phenylalaninyl)-*R*-phenylalaninol	$C_{27}H_{28}N_2O_4$	$CDCl_3$	[224]



2-9-3 R = H
2-9-4 R = Me



2-9-5



2-9-6

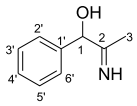
Table 2-9-4: ^{1}H NMR spectroscopic data of amide-type amphetamine alkaloids **2-9-3**~**2-9-6**.

H	2-9-3	2-9-4	2-9-5	2-9-6
1			2.03 s	2.0 s
2	7.47 d(1.9)	7.44 d(1.1)		
3			3.84 dd(11, 5)	3.90 dd(11, 6.5)
			3.95 dd(11, 5)	3.96 dd(11, 5.5)
4			4.2~4.47 m	4.28~4.31 m
5	7.04 d(8.5)	7.04 d(8.4)	6.1 d(7.5)	6.31 d(8)
6	7.20 dd(1.9, 8.5)	7.18 dd(1.1, 8.4)		
7	3.08 dd(7.8, 14.0)	3.12 dd(5.7, 13.6)	4.78 m	4.81 m
	2.98 dd(7.3, 14.0)	3.05 dd(8.6, 13.6)		
8	4.00 t(7.3)	3.90 m	6.85 d(7.5)	6.9 d(7.5)
10			3.07 dd(14.5, 8)	2.99 dd(13.6, 9)
			3.22 dd(15, 6)	3.12 dd(14.1, 6.5)
11	3.56 m	3.47 m	2.74 d(7)	2.79 dd(14.8)
	3.32 dd(6.6, 13.2)	3.38 m		2.85 dd(13.8, 5.6)
12	2.74 t(7.2)	2.73 m, 2.63 m		
14	7.51 s	7.47 s		
18	7.51 s	7.47 s		
20	4.13 t(5.6)	4.12 t(5.5)		
21	2.32 tt(5.6,7.9)	2.32 tt(5.5, 7.5)		
22	3.54 t(7.9)	3.54 t(7.4)		
OMe	3.91 s	3.90 s		
$N(Me)_2$	3.00 s	3.00 s		
NMe		2.62 s		
Ar-H			7.12~7.78 (15H)	7.1~7.72 (15H)

2.9.3 Imine-type amphetamine alkaloids

Table 2-9-5: Co, MF, and TS of imine-type amphetamine alkaloid **2-9-7**.

No.	Compound	MF	Test solvent	Reference
2-9-7	(±)-1-phenyl-2-imido1-propanol	$C_9H_{11}NO$	$CDCl_3$	[225]



2-9-7

Table 2-9-6: ^{1}H NMR spectroscopic data of imine-type amphetamine alkaloid **2-9-7**.

H	2-9-7	H	2-9-7
1	5.15 d(4.0)	1-OH	5.80 d(4.0)
3	1.54 s	2-NH	10.59 s
2′,6′	7.23~7.33 m		

Bibliography

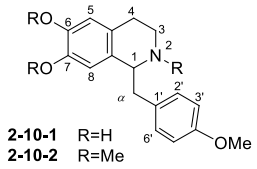
[222] Wang FP. Chemistry of alkaloids. Chemical Industry Press, Beijing, 2008, 159.
[223] Tsuda M, Sakuma Y, Kobayashi J. J Nat Prod, 2001, 64: 980.
[224] Wahidulla S, D'Souza L, Kamat SY. Phytochemistry, 1991, 30: 3323.
[225] Zhao W, Deng AJ, Du GH, et al. J Asian Nat Prod Res, 2009, 11: 168.

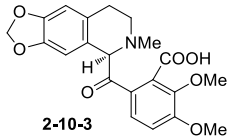
2.10 Isoquinoline alkaloids

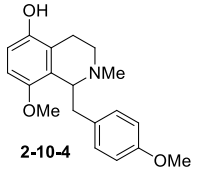
2.10.1 Benzylated isoquinoline alkaloids

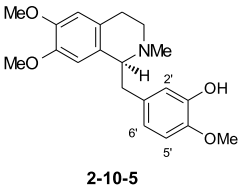
Table 2-10-1: Cos, MFs, and TSs of benzylated isoquinoline alkaloids **2-10-1**~**2-10-15**.

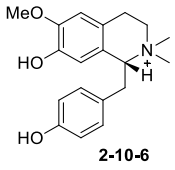
No.	Compounds	MFs	Test solvents	References
2-10-1	argemexirine	$C_{17}H_{19}NO_3$	$CDCl_3$-CD_3OD(4:1)	[226]
2-10-2	*O*-methylarmepavine	$C_{20}H_{25}NO_3$	$CDCl_3$-CD_3OD(4:1)	[226]
2-10-3	(–)-berbervirine	$C_{21}H_{21}NO_7$	$CDCl_3$	[227]
2-10-4	1,2,3,4-tetrahydro-5-hydroxyl-8-methoxyl-2-methyl-4′-methoxyl-benzylisoquinoline	$C_{19}H_{23}NO_3$	$CDCl_3$	[228]
2-10-5	(+)-laudanidine	$C_{20}H_{25}NO_4$	$CDCl_3$	[229]
2-10-6	(–)-magnocurarine	$C_{19}H_{24}NO_3$	CD_3OD	[230]
2-10-7	(–)-8-*O*-methyloblongine	$C_{20}H_{26}NO_3$	CD_3OD	[230]
2-10-8	(+)-parvinine	$C_{19}H_{21}NO_4$	$CDCl_3$	[231]
2-10-9	xylopinidine	$C_{20}H_{26}NO_4$	CD_3COCD_3	[232]
2-10-10	*N*,*N*-dimethylanomurine	$C_{22}H_{30}NO_4$	$CDCl_3$	[232]
2-10-11	1*S*,2*S*-reticuline N_α-oxide	$C_{19}H_{23}NO_5$	CD_3OD	[233]
2-10-12	1*S*,2*R*-reticuline N_β-oxide	$C_{19}H_{23}NO_5$	CD_3OD	[233]
2-10-13	*R*-(+)-isotembetarine	$C_{20}H_{26}NO_4$	CD_3COCD_3	[234]
2-10-14	(±)-*N*-methylhigenamine	$C_{17}H_{19}NO_3$	DMSO-d_6	[235]
2-10-15	(–)-*N*-Methylhigenamine *N*-oxide	$C_{17}H_{19}NO_4$	D_2O	[235]

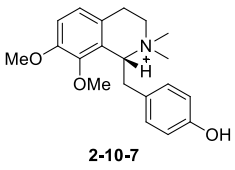


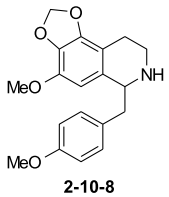


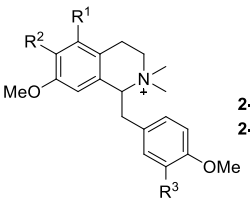




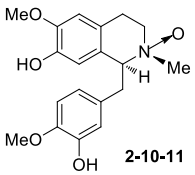


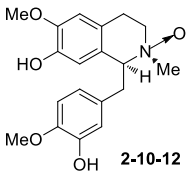


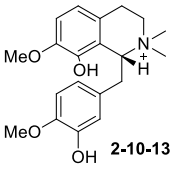




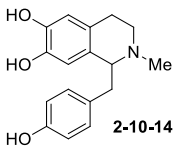
	R^1	R^2	R^3
2-10-9	H	OH	OH
2-10-10	OMe	OMe	H







2-10-13



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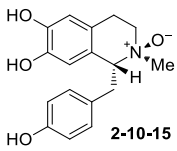


2-10-15

Table 2-10-2: ^{1}H NMR spectroscopic data of benzylated isoquinoline alkaloids **2-10-1**~**2-10-5**.

H	2-10-1	2-10-2	2-10-3	2-10-4	2-10-5
1	4.45 dd(3, 10)	4.34 dd(3, 10)		4.30 dd(2.8, 7.2)	3.67 dd(5.2, 7.8)
2	2.75 m	2.54 s			
3	2.75 m	2.66 m	–	3.32 m	3.14-3.2 m(ov)
	3.35 m	3.24 m		2.90 m	2.73-2.78 m(ov)
4	2.95 m	2.86 m	–	2.59 m	2.80-2.86 m(ov)
				2.88 m	2.57 dt(4.5, 16)
5	6.80 s	6.76 s	6.70 s		6.53 s
6				6.37 d(8.1)	
7				6.60 d(8.1)	
8	6.75 s	6.64 s	7.25 s		6.03 s
α	2.95 m	2.86 m		3.09 m	3.11 dd(5.2, 13.7)
	3.35 m	3.24 m			2.68 dd(7.9, 13.7)
2′	7.30 d(8.5)	7.20 d(8)	10.32 br s(COOH)	7.19 d(8.4)	6.50 d(2.1)
3′	7.00 d(8.5)	6.89 d(8)		6.76 d(8.4)	
5′	7.00 d(8.5)	6.89 d(8)	7.50 d(8.0)	6.76 d(8.4)	6.70 d(8.2)
6′	7.30 d(8.5)	7.20 d(8)	7.30 d(8.0)	7.19 d(8.4)	6.50 dd(2.1, 8.2)
NMe			2.50 s	2.47 s	2.49 s
OCH_2O			6.00 s		
OMe	3.85 s(4′-OMe)	3.88 s(4′-OMe)	3.95 s(3′-OMe)	3.87 s	3.82 s(4′-OMe)
		3.70 s(6-OMe)	3.90 s(4′-OMe)	3.76 s	3.80 s(6-OMe)
		3.82 s(7-OMe)			3.54 s(7-OMe)

Table 2-10-3: ^{1}H NMR spectroscopic data of benzylated isoquinoline alkaloids **2-10-6**~**2-10-10**.

H	2-10-6 · ClO_4	2-10-7 · ClO_4	2-10-8	2-10-9 · ClO_4	2-10-10
1	4.54 dd(3.8, 8.8)	5.01 t(5.1)	4.09 dd(9.5, 4.5)	4.82 dd(3.5, 10.0)	4.71 dd(4.0, 10.0)
3	–	–	2.91 dd(12.5, 6.0)	3.85 m	3.66 m
			3.21 td(12.5, 6.0)	4.10 ddd(7.5, 11.5, 13.0)	
4	–	–	2.65 br t(6.0)	3.26 m	3.00 m, 3.11 m
5	6.80 s	6.97 d(8.5)		6.74 s	
6		7.13 d(8.5)			
8	5.89 s	–	6.34 s	5.88 s	5.61 s
α	–	–	2.88 dd(14.0, 9.5)	3.00 dd(10, 12.5)	2.85 dd(10.0, 13.0)
			3.14 dd(14.0, 4.5)	3.78 br dd(3.5, 12.5)	3.62 dd(4.0, 13.0)

Table 2-10-3 (continued)

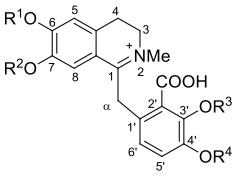
H	**2-10-6 · ClO_4**	**2-10-7 · ClO_4**	**2-10-8**	**2-10-9 · ClO_4**	**2-10-10**
2′	6.85 d(8.8)	6.92 d(8.5)	7.16 d-like (9.0)	6.66 d(2.0)	6.90 d(8.5)
3′	6.68 d(8.8)	6.67 d(8.5)	6.87 d-like (9.0)		6.80 d(8.5)
5′	6.68 d(8.8)	6.67 d(8.5)	6.87 d-like (9.0)	6.89 d(8.0)	6.80 d(8.5)
6′	6.85 d(8.8)	6.92 d(8.5)	7.16 d-like (9.0)	6.54 dd(2.0, 8.0)	6.90 d(8.5)
NMe	3.11 s, 3.38 s	3.20 s, 3.03 s		3.37 s, 3.64 s	3.25 s, 3.55 s
OCH_2O			5.96 s		
OMe	3.83 s(6-OMe)	3.86 s(7-OMe)	3.80 s(4′-OMe)	3.83 s(4′-OMe)	3.77 s(4′-OMe)
		3.82 s(8-OMe)	3.85 s(7-OMe)	3.42 s(7-OMe)	3.83 s(5-OMe)
					3.93 s(6-OMe)
					3.42 s(7-OMe)

Table 2-10-4: ^{1}H NMR spectroscopic data of benzylated isoquinoline alkaloids **2-10-11**~**2-10-15**.

H	**2-10-11**	**2-10-12**	**2-10-13**	**2-10-14**	**2-10-15**
1	5.08 dd	4.82 br d	5.22 m	3.48 t(5.1)	5.53 br s
3	–	–	3.72 m	2.57~2.99 m	3.15~4.13 m
			4.04 ddd(13.5, 12.5, 6.5)		2.55~3.01 m
4	–	–	3.30 m, 3.15 m	2.48~2.55 m	–
5	6.77 s	6.79 s	6.83 d(8.5)	6.38 s	6.79 s
6			7.08 d(8.5)	–	
8	6.05 s	5.75 s		6.34 s	6.66 s
α	–	–	3.28 m	2.81 dd(14.2, 5.1)	3.78 dd(11.0, 3.2)
			3.52 dd(16.0, 7.5)	2.71 dd(14.2, 5.1)	3.75 dd(11.0, 2.8)
2′	6.63 d	6.54 d	6.91 d(2.0)	6.89 d(8.3)	6.80 d(8.4)
3′				6.58 d(8.3)	6.71 d(8.4)
5′	6.85 d	6.82 d	6.87 d(8.5)	6.58 d(8.3)	6.71 d(8.4)
6′	6.56 br d	6.48 br d	6.81 dd(8.5, 2.0)	6.89 d(8.3)	6.80 d(8.4)
NMe	3.59 s	3.46 s	3.31 s, 3.34 s	2.31 s	3.07 s
OMe	3.81 s(4′-OMe)	3.81 s(4′-OMe)	3.82 s(4′-OMe)		
	3.82 s(6-OMe)	3.82 s(6-OMe)	3.89 s(7-OMe)		
OH			7.65 s,8.53 s		

Table 2-10-5: Cos, MFs, and TSs of benzylated isoquinoline alkaloids **2-10-16**~**2-10-18**.

No.	Compounds	MFs	Test solvents	References
2-10-16	leptopine	$C_{20}H_{18}NO_6$	CD_3OD	[236]
2-10-17	leptopinine	$C_{20}H_{22}NO_6$	CD_3OD	[236]
2-10-18	leptopidine	$C_{20}H_{20}NO_6$	CD_3OD	[236]



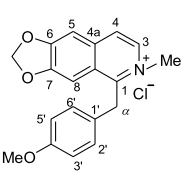
	R^1	R^2	R^3	R^4
2-10-16	-CH_2-		-CH_2-	
2-10-17	H	H	Me	Me
2-10-18	-CH_2-		H	Me

Table 2-10-6: ^{1}H NMR spectroscopic data of benzylated isoquinoline alkaloids **2-10-16**~**2-10-18**.

H	2-10-16 · Cl	2-10-17 · Cl	2-10-18 · Cl	H	2-10-16 · Cl	2-10-17 · Cl	2-10-18 · Cl
3	4.07 t(7.5)	4.03 t(7.5)	4.01 t(7.5)	NMe	3.77 s	3.70 s	3.70 s
4	3.16 t(7.5)	3.06 t(7.5)	3.09 t(7.5)	3′-OMe		3.83 s	
5	6.91 s	6.78 s	6.82 s	4′-OMe		3.65 s	3.80 s
8	7.42 s	7.21 s	7.11 s	6,7-OCH_2O	6.06 s		5.98 s
5′	6.65 d(8)	7.01 d(8.5)	6.90 d(7.5)	3′,4′-OCH_2O	5.92 s		
6′	6.49 d(8)	6.58 d(8.5)	6.50 d(7.5)				

Table 2-10-7: Cos, MFs, and TSs of benzylated isoquinoline alkaloids **2-10-19**~**2-10-28**.

No.	Compounds	MFs	Test solvents	References
2-10-19	2-methyl-1-(*p*-methoxybenzyl)-6,7-methylenedioxyiso-quinolinium chloride	$C_{19}H_{18}ClNO_3$	$CDCl_3$	[237]
2-10-20	1-(4-hydroxybenzyl)-6,7-methylenedioxy-2-methyliso-quinolinium trifluoroacetate	$C_{20}H_{16}F_3NO_5$	DMSO-d_6	[238]
2-10-21	1-(4-hydroxybenzoyl)-7-hydroxy-6-methoxyisoquino-line	$C_{17}H_{13}NO_4$	CD_3OD	[239]
2-10-22	thalprzewalskiinone	$C_{20}H_{20}NO_5$	DMSO-d_6	[240]
2-10-23	annocherine A	$C_{17}H_{15}NO_4$	$CDCl_3$	[241]
2-10-24	annocherine B	$C_{18}H_{17}NO_4$	$CDCl_3$	[241]
2-10-25	neolitacumonine	$C_{17}H_{13}NO_3$	C_5D_5N	[242]
2-10-26	tetradehydroreticuline	$C_{19}H_{20}NO_4$	CD_3OD	[243]
2-10-27	fumaflorine	$C_{20}H_{15}NO_7$	CD_3OD	[244]
2-10-28	1-(2-methoxycarbonyl-3,4-methylenedioxyphenylcarb-onyl)-6,7-dimethoxyisoquinoline	$C_{21}H_{17}NO_7$	$CDCl_3$	[244]



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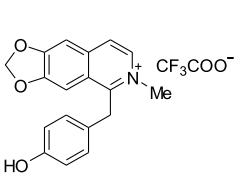

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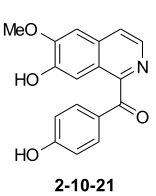

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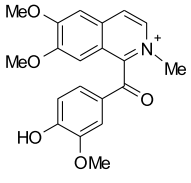

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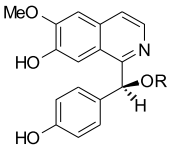
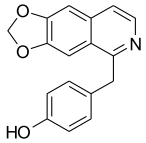


2-10-23 R = H
2-10-24 R = Me



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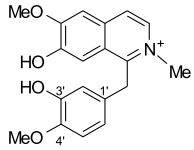

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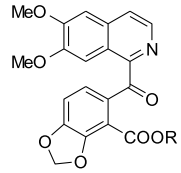


2-10-27 R = H
2-10-28 R = Me

Table 2-10-8: ^{1}H NMR spectroscopic data of benzylated isoquinoline alkaloids **2-10-19**~**2-10-23**.

H	2-10-19	2-10-20	2-10-21	2-10-22	2-10-23
3	8.98 d(6.6)	8.52 d(7.2)	8.26 d(5.6)	8.70 d(6.8)	8.29 d(6.0)
4	8.09 d(6.6)	8.18 d(7.2)	7.77 d(5.6)	8.45 d(6.8)	7.51 d(6.0)
5	7.39 s	7.70 s	7.36 s	7.88 s	7.16 s
8	7.56 s	8.08 s	7.22 s	6.89 s	7.88 s
α	4.87 s	4.84 s			4.45 s
2′	6.92 d(9.0)	6.90 d(8.4)	7.73 d(9)	7.49	7.01 d(8.8)
3′	6.83 d(9.0)	6.70 d(8.4)	6.86 d(9)		6.65 d(8.8)
5′	6.83 d(9.0)	6.70 d(8.4)	6.86 d(9)	7.24	6.65 d(8.8)
6′	6.92 d(9.0)	6.90 d(8.4)	7.73 d(9)	7.06	7.01 d(8.8)
6,7-OCH_2O	6.28 s	6.41 s			
2-Me	4.54 s	4.25 s			
OMe	3.76 s(4′-OMe)		4.06 s(6-OMe)	3.89 s(3′-OMe) 3.74 s(6-OMe) 4.08 s(7-OMe)	3.94 s(6-OMe)
NMe	4.54 s	4.25 s		4.14 s	
OH		9.44 s			

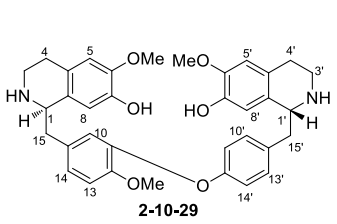
Table 2-10-9: ^{1}H NMR spectroscopic data of benzylated isoquinoline alkaloids **2-10-24**~**2-10-28**.

H	2-10-24	2-10-25	2-10-26	2-10-27	2-10-28
3	8.24 d(5.6)	8.54 d(5.6)	8.28 d(7.0)	8.29 d	8.31 d
4	7.46 d(5.6)	7.48 d(5.6)	8.10 d(7.0)	7.71 d	7.69 d
5	7.03 s	7.17 s	7.64 s	8.11 s	8.20 s
8	7.67 s	7.71 s	7.75 s	7.22 s	7.19 s
α	5.78 s	4.69 s	4.80 s		
2′	7.19 d(8.4)	7.42 d(8.4)	6.54 d(2.0)		
3′	6.67 d(8.4)	7.11 d(8.4)			
5′	6.67 d(8.4)	7.11 d(8.4)	6.86 d(8.5)	7.16 d	7.26 d
6′	7.19 d(8.4)	7.42 d(8.4)	6.46 dd(8.5, 2.0)	6.95 d	6.99 d
OMe	3.96 s(6-OMe)		4.16 s(6-OMe)	4.04 s(6-OMe)	4.07 s(6-OMe)
	3.36 s(*α*-OMe)		3.80 s(4′-OMe)	4.07 s(7-OMe)	4.08 s(7-OMe)
2′-COOMe					3.36 s
OCH_2O		6.05 s		6.19 s	6.19 s
NMe			4.28 s		

2.10.2 Dimeric benzylated isoquinoline alkaloids

Table 2-10-10: Cos, MFs, and TSs of dimeric benzylated isoquinoline alkaloids **2-10-29**~**2-10-42**.

No.	Compounds	MFs	Test solvents	References
2-10-29	(+)-costaricine	$C_{35}H_{38}N_2O_6$	$CDCl_3$	[245]
2-10-30	3-hydroxy-6′-desmethyl-9-*O*-methyl-thalifaboramine	$C_{39}H_{44}N_2O_8$	$CDCl_3$	[246]
2-10-31	3-hydroxythalifaboramine	$C_{39}H_{44}N_2O_8$	$CDCl_3$	[246]
2-10-32	6′-desmethylthalifaboramine	$C_{38}H_{42}N_2O_7$	$CDCl_3$	[246]
2-10-33	3,5′-dihydroxythalifaboramine	$C_{39}H_{44}N_2O_9$	$CDCl_3$	[246]
2-10-34	5′-hydroxythalifaboramine	$C_{39}H_{44}N_2O_8$	$CDCl_3$	[246]
2-10-35	3-hydroxy-6′-desmethylthalifaboramine	$C_{38}H_{42}N_2O_8$	$CDCl_3$	[246]
2-10-36	fauridine	$C_{40}H_{46}N_2O_8$	$CDCl_3$	[247]
2-10-37	faurithaline	$C_{39}H_{44}N_2O_8$	$CDCl_3$	[247]
2-10-38	3-methoxyfaurithaline	$C_{40}H_{46}N_2O_9$	$CDCl_3$	[247]
2-10-39	faurithaline diacetate	$C_{41}H_{46}N_2O_9$	$CDCl_3$	[247]
2-10-40	isothalictrine	$C_{41}H_{44}N_2O_{10}$	$CDCl_3$	[248]
2-10-41	isothalicrine	$C_{42}H_{52}N_2O_{10}$	$CDCl_3$	[248]
2-10-42	isothalirine	$C_{42}H_{52}N_2O_9$	$CDCl_3$	[248]



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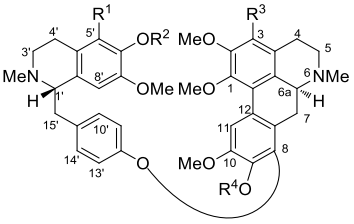
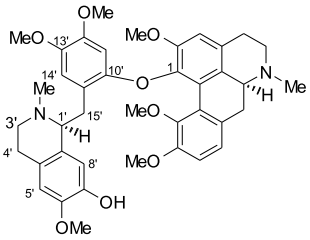


	R^1	R^2	R^3	R^4
2-10-30	H	H	OH	Me
2-10-31	H	Me	OH	H
2-10-32	H	H	H	H
2-10-33	OH	Me	OH	H
2-10-34	OH	Me	H	H
2-10-35	H	H	OH	H



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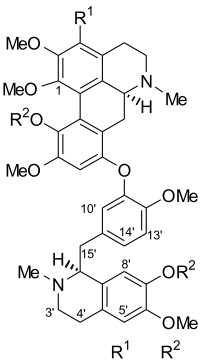
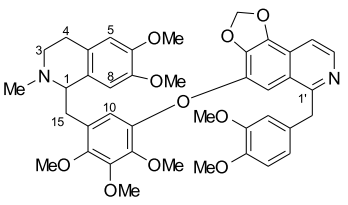
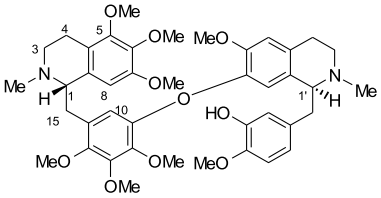


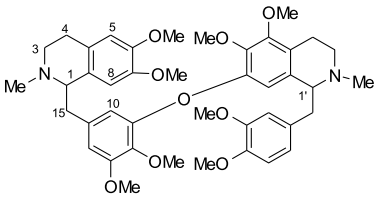
	R^1	R^2
2-10-37	H	H
2-10-38	OMe	H
2-10-39	H	Ac



2-10-40



2-10-41



2-10-42

Table 2-10-11: ^{1}H NMR spectroscopic data of dimeric benzylated isoquinoline alkaloids **2-10-29**~**2-10-33**.

H	2-10-29	2-10-30	2-10-31	2-10-32	2-10-33
1	4.05 dd(9.0, 3.5)				
3	2.64 m, 3.16 m			6.59	
4	2.63 m, 2.88 m	2.84 m, 2.75 m	2.87 m, 2.73 m	2.84 m, 2.75 m	2.83 m, 2.78 m
5	6.51 s	2.99 m	3.01 dd(5.5, 10.0)	2.99 m	3.02 dd(5.0, 13.0)
		2.37 m	2.37 dd(4.0, 11.5)	2.35 m	2.38 m
6a	–	2.81 m	2.82 m	2.81 m	2.85 m
7	–	3.19 m	3.21 m	3.22 dd(4.0, 14.0)	3.19 m
		1.99 t(14.0)	2.01 t(14.0)	2.00 t(14.0)	1.98 t(13.5)
8	6.69 s				
10	6.73 d(2.0)				
11		8.01	7.81	8.01	7.81 s
13	6.92 d(8.5)				
14	6.96 dd(8.5, 2.0)				
15	2.85 m				
	3.04 dd(14.0, 3.5)				
1′	4.08 dd(10.0, 3.7)	3.63 m	3.66 m	3.63 m	3.62 m
3′	2.72 m, 3.23 m	3.16 m, 2.73 m	3.18 m, 2.84 m	3.13 m, 2.72 m	3.16 m, 2.74 m
4′	2.76 m	2.73 m	2.75 m	2.71 m	2.74 m
	2.92 m	2.50 m	2.59 dt(16.0, 4.0)	2.50 m	2.45 m
5′	6.56 s	6.55 s	6.54 s	6.54 s	
8′	6.73 s	5.93 s	5.98 s	5.92 s	5.69 s
10′	7.13 br d(8.5)	6.95 d(8.5)	6.97 d(8.5)	6.93 d(8.5)	7.00 d(8.5)
11′	6.86 br d(8.5)	6.76 d(8.5)	6.81 d(8.5)	6.77 d(8.5)	6.80 d(8.5)
13′	6.86 br d(8.5)	6.76 d(8.5)	6.81 d(8.5)	6.77 d(8.5)	6.80 d(8.5)
14′	7.13 br d(8.5)	6.95 d(8.5)	6.97 d(8.5)	6.93 d(8.5)	7.00 d(8.5)
15′	2.80 dd(13.5, 3.7)	3.07 m	3.14 dd(4.5, 13.0)	3.08 m	3.08 dd(5.5, 14.0)
	3.14 m	2.75 m	2.72 m	2.75 m	2.76 m
OMe	3.81 s(6-OMe)	3.78 s(1-OMe)	3.75 s(1-OMe)	3.70 s(1-OMe)	3.76 s(1-OMe)
	3.84 s(12-OMe)	3.96 s(2-OMe)	3.98 s(2-OMe)	3.89 s(2-OMe)	3.99 s(2-OMe)
	3.82 s(6′-OMe)	3.78 s(9-OMe)	3.96 s(10-OMe)	3.92 s(10-OMe)	3.97 s(10-OMe)
		3.92 s(10-OMe)	3.82 s(6′-OMe)	3.53 s(7′-OMe)	3.84 s(6′-OMe)
		3.53 s(7′-OMe)	3.55 s(7′-OMe)		3.57 s(7′-OMe)
6-NMe		2.31 s	2.28 s	2.30 s	2.28 s
2′-NMe		2.48 s	2.52 s	2.48 s	2.49 s

Table 2-10-12: ^{1}H NMR spectroscopic data of dimeric benzylated isoquinoline alkaloids **2-10-34**~**2-10-36**.

H	2-10-34	2-10-35	2-10-36
3	6.60 s		6.75
4	2.95 m, 2.73 m	2.85 m, 2.73 m	*α* 2.78 br dd(16.3, 4), *β* 3.22 ddd(16.2, 11.8, 6.3)
5	2.98 m, 2.44 m	2.98 m, 2.37 m	*α* 2.60 ddd(11.8, 11.8, 3.8), *β* 3.10 (ov)
6a	2.86 m	2.82 m	2.97 dd(12.3, 3.7)
7	3.22 m, 2.01 t(14.5)	3.20 dd(4.0, 14.0), 2.01 t(14.0)	*α* 3.09 dd(13.5, 3.7), *β* 2.36 t(12.5)
8			6.89 d(8.1)
9			6.73 d(8.2)
11	8.02 s	7.82 s	
1′	3.68 m	3.66 m	4.28 br s
3′	3.16 m, 2.82 m	3.18 m, 2.83 m	–(ov)
4′	2.82 m, 2.48 m	2.74 m, 2.58 m	–(ov)
5′		6.59 s	6.55
8′	5.65 s	5.96 s	6.56 br
10′	7.00 d(8.0)	6.98 d(8.0)	
11′	6.81 d(8.0)	6.81 d(8.0)	5.65 br
13′	6.81 d(8.0)	6.81 d(8.0)	
14′	7.00 d(8.0)	6.98 d(8.0)	6.45 br
15′	3.16 m, 2.76 m	3.14 dd(4.5, 13.0), 2.78 m	3.14 dd(14.3, 8.1), –(ov)
OMe	3.70 s(1-OMe)	3.77 s(1-OMe)	3.71 (2-OMe)
	3.89 s(2-OMe)	3.97 s(2-OMe)	3.67 (10-OMe)
	3.94 s(10-OMe)	3.99 s(10-OMe)	3.69 (11-OMe)
	3.84 s(6′-OMe)	3.57 s(7′-OMe)	3.84 (6′-OMe)
	3.55 s(7′-OMe)		3.41 (12′-OMe)
			3.64 (13′-OMe)
6-NMe	2.28 s	2.31 s	2.57
2′-NMe	2.52 s	2.51 s	2.53 br

Table 2-10-13: ^{1}H NMR spectroscopic data of dimeric benzylated isoquinoline alkaloids **2-10-37**~**2-10-39**.

H	2-10-37	2-10-38	2-10-39
3	6.70		6.69
4*α*	2.67 dd(16.9, 2.8)	2.77 (ov)	2.67 dd(16.4, 3.3)
4*β*	3.21 ddd(16.9, 11.2, 5.6)	2.95 ddd(16.8, 11.0, 5.5)	3.11 ddd(17.2, 11.2, 5.9)
5*α*	2.48 ddd(11.9, 11.9, 3.5)	2.39 (ov)	2.46 ddd(11.9, 11.9, 3.9)
5*β*	3.01 dd(11.5, 5.6)	3.05 (ov)	2.98 dd(11.4, 5.5)
6a	2.82 br d(12.4)	2.77 dd(13.6, 4.0)	2.78 dd(12.6, 3.5)
7*α*	3.31 dd(13.8, 3.4)	3.31 dd(14.0, 3.5)	3.48 dd(14.0, 3.5)
7*β*	1.89 t(13.5)	1.88 t(13.6)	1.98 t(13.3)
9	6.63	6.60	6.56
1′	3.60 br t(6.5)	3.58 t(5.3)	3.58 t(6.0)

Table 2-10-13 (continued)

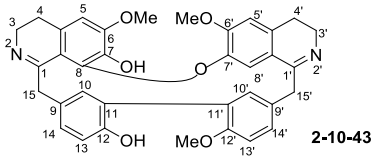
H	2-10-37	2-10-38	2-10-39
3′	3.08 (ov), 2.67 (ov)	3.06 (ov), 2.67 (ov)	3.05 ddd(12.8, 8.0, 4.8), 2.66 (ov)
4′	2.66 (ov), 2.47 (ov)	2.67 (ov), 2.47 (ov)	2.75 (ov), 2.52 ddd(16.2, 5.0, 5.0)
5′	6.39	6.39	6.59
8′	6.14	6.13	6.43
10′	6.35 br	6.34 d(1.4)	6.71 (ov)
13′	6.87 d(8.3)	6.88 dd(8.3, 1.3)	6.83 d(8.8)
14′	6.78 br d(8.2)	6.78 dd(8.3, 1.3)	6.71 (ov)
15′	3.07 (ov), 2.65 (ov)	3.05 (ov), 2.65 dd(14.0, 7.7)	2.93 dd(14.2, 6.2), 2.77 dd(14.2, 6.2)
OMe	3.67(1-OMe), 3.91(2-OMe), 3.81(10-OMe), 3.71(6′-OMe) 3.91(12′-OMe)	3.71(1-OMe), 3.97(2-OMe) 3.93(3-OMe), 3.81(10-OMe) 3.71(6′-OMe), 3.91(12′-OMe)	3.44(1-OMe), 3.87(2-OMe) 3.71(10-OMe), 3.76(6′-OMe) 3.86(12′-OMe)
6-NMe	2.37	2.36	2.42
2′-NMe	2.42	2.41	2.35
OAc			2.23 (11-OAc), 2.24 (7′-OAc)
11-OH	8.57		

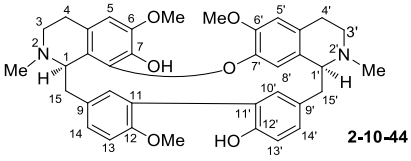
Table 2-10-14: ^{1}H NMR spectroscopic data of dimeric benzylated isoquinoline alkaloids **2-10-40**~**2-10-42**.

H	2-10-40	2-10-41	2-10-42	H	2-10-40	2-10-41	2-10-42
1	3.77~3.81 m	3.54~3.62 m	3.88~3.92 m	14′	6.52 dd (8.1, 1.8)	6.54 dd (8.2, 1.8)	6.49 dd (8.2, 1.8)
5	6.73 s	3.85 s(OMe)	6.55 s	15′	4.35 d(14.6) 4.43 d(14.6)		
8	6.46 s	5.81 s	6.14 s	6-OMe	3.71 s	3.82 s	3.83 s
10	6.01 s	5.98 s	–	7-OMe	3.73 s	3.49 s	3.56 s
11			6.38 s	12-OMe	3.98 s	3.74 s	3.71 s
14			6.57 s	13-OMe	3.87 s	3.85 s	3.77 s
15	3.48~3.60 m	–	–	14-OMe	3.88 s	3.84 s	
1′		3.88~3.92 m	3.60~3.62 m	6′-OMe		3.78 s	3.86 s
3′	8.29 d(5.8)	–	–	2-NMe	2.47 s	2.50 s	2.47 s
4′	7.43 d(5.8)	–	–	2′-NMe		2.50 s	2.47 s
5′		6.53 s	3.87 s(OMe)	11′-OMe	3.76 s		3.73 s
8′	7.37 s	6.42 s	6.02 s	12′-OMe	3.83 s	3.75 s	3.78 s
10′	6.57 d(1.8)	6.38 d(1.8)	6.59 d(1.8)	OCH_2O	6.19 s		
13′	6.67 d(8.1)	6.59 d(8.2)	6.60 d(8.2)	CH_2	2.52~3.14 m(4H)	2.54~2.60 m(1H) 2.62~2.98 m(7H) 3.00~3.30 m(4H)	2.50~2.66 m(2H) 2.70~2.90 m(5H) 2.92~3.36 m(5H)

Table 2-10-15: Cos, MFs, and TSs of dimeric benzylated isoquinoline alkaloids **2-10-43**~**2-10-45**.

No.	Compounds	MFs	Test solvents	References
2-10-43	philogaline	$C_{35}H_{32}N_2O_6$	$CDCl_3$	[249]
2-10-44	(–)-antioquine	$C_{37}H_{40}N_2O_6$	$CDCl_3$	[249]
2-10-45	(+)-isothalmidine	$C_{37}H_{36}N_2O_7$	$CDCl_3$	[250]





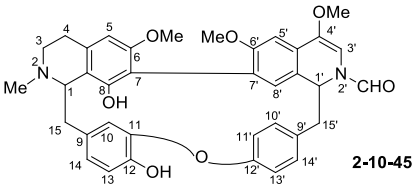


Table 2-10-16: ^{1}H NMR spectroscopic data of dimeric benzylated isoquinoline alkaloids **2-10-43**~**2-10-45**.

H	2-10-43	2-10-44	2-10-45
1		4.00 d(8)	3.72 m
3	ax 3.19 m, eq 3.89 m	ax 2.93 m, eq 3.48 m	2.87 m, 2.53 m
4	2.48 m	ax 2.47 m, eq 2.93 m	2.58 m, 2.87 m
5	6.56 s	6.34 s	6.31 s
10	6.60 d(2)	7.12 br s	6.22 d(2.2)
13	6.87 d(8)	6.85 d(8)	5.97 d(7.7)
14	7.47 dd(8, 2)	7.27 dd(8, 2)	6.52 dd(7.7, 2.2)
15	3.87 m, 4.58/4.59 2d(12)①	3.05 m, 2.78 m	2.58 m
1′		3.71 t(3)	5.61 dd(5.6, 10.8)
3′	ax 3.19 m, eq 3.89 m	3.12 m	6.90 s
4′	2.48 m	2.71 m	
5′	6.74 s	6.48 s	6.57 s
8′	7.52 s	6.94 s	6.21 s
10′	7.15 d(2)	6.63 br s	6.86 dd(8.2, 2.2)
11′			6.79 dd(8.2, 2.6)
13′	6.80 d(8)	6.86 d(8)	7.15 dd(8.2, 2.6)
14′	7.28 dd(8, 2)	7.20 dd(8, 2)	7.45 dd(8.2, 2.2)
15′	3.72 m, 4.04/4.05 2d(13)①	3.05 m	2.87 m, 3.04 m
2-NMe		2.35 s	2.35 s

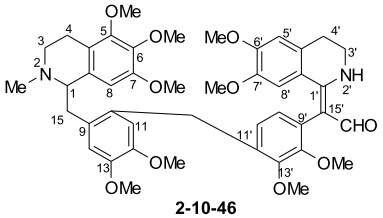
Table 2-10-16 (continued)

H	2-10-43	2-10-44	2-10-45
2′-NMe		2.64 s	
2′-CHO			8.39 s
OMe	3.92 s(6-OMe)	3.82 s(6-OMe)	3.78 s(6-OMe)
	3.90 s(6′-OMe)	3.87 s(12-OMe)	3.93 s(4′-OMe)
	3.85 s(12′-OMe)	3.49 s(6′-OMe)	3.42 s(6′-OMe)

① Two conformers in equal amount.

Table 2-10-17: Cos, MFs, and TSs of dimeric benzylated isoquinoline alkaloids **2-10-46** and **2-10-47**.

No.	Compounds	MFs	Test solvents	References
2-10-46	isopyruthaldine	$C_{44}H_{52}N_2O_{10}$	$CDCl_3$	[250]
2-10-47	isopythaldine	$C_{43}H_{48}N_2O_{10}$	$CDCl_3$	[250]



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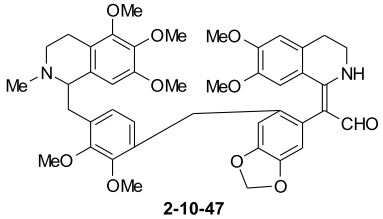


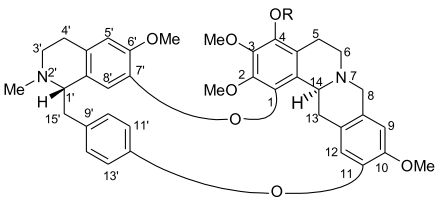
2-10-47

Table 2-10-18: ^{1}H NMR spectroscopic data of dimeric benzylated isoquinoline alkaloids **2-10-46** and **2-10-47**.

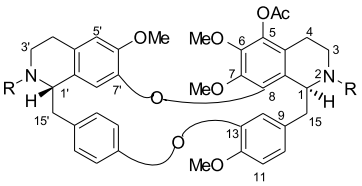
H	2-10-46	2-10-47	H	2-10-46	2-10-47
1	3.94 m	3.92 m	14′		7.83 s
3	2.81 m, 3.28 m	2.82 m, 3.15 m	2-NMe	2.55 s	2.56 s
4	3.28 m	3.15 m	5-OMe	3.82 s or 3.97 s	3.96 s
8	5.98 s	5.95 s	6-OMe	3.79 s	3.80 s
10		6.91 br s	7-OMe	3.57 s	3.56 s
11	7.31 s	7.91 br s	12-OMe	4.13 s	
14	7.96 s		13-OMe	4.10 s	3.82 s
15	3.28 m	3.15 m	14-OMe		3.97 s
3′	2.81 m	2.82 m	6′-OMe	3.86 s	3.86 s
4′	2.81 m, 3.28 m	2.82 m, 3.15 m	7′-OMe	3.80 s	3.77 s
5′	6.80 s	6.78 s	13′-OMe	3.97 s or 3.82 s	
8′	6.51 s	6.44 s	14′-OMe	3.99 s	
10′	6.92 br s		15′-CHO	10.68 s	10.60 s
11′	7.89 br s	7.31 s	CH_2-bridge	5.07 br s	5.03 br s
			OCH_2O		6.27 s

Table 2-10-19: Cos, MFs, and TSs of dimeric benzylated isoquinoline alkaloids **2-10-48**~**2-10-67**.

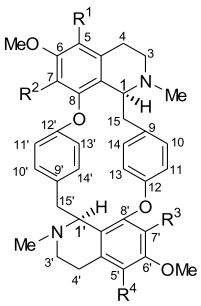
No.	Compounds	MFs	Test solvents	References
2-10-48	longiberine	$C_{38}H_{40}N_2O_7$	$CDCl_3$	[251]
2-10-49	*O*-methyllongiberine	$C_{39}H_{42}N_2O_7$	$CDCl_3$	[251]
2-10-50	longiberine acetate	$C_{40}H_{42}N_2O_8$	$CDCl_3$	[251]
2-10-51	thalidezine acetate	$C_{40}H_{44}N_2O_8$	$CDCl_3$	[251]
2-10-52	dinorthalidezine acetate	$C_{38}H_{40}N_2O_8$	$CDCl_3$	[251]
2-10-53	(–)-cycleanine	$C_{38}H_{42}N_2O_6$	$CDCl_3$	[252]
2-10-54	(–)-isochondodendrine	$C_{36}H_{38}N_2O_6$	$CDCl_3$-CD_3OD (9:1)	[252]
2-10-55	(–)-norcycleanine	$C_{37}H_{40}N_2O_6$	$CDCl_3$	[252]
2-10-56	(–)-jollyanine	$C_{38}H_{42}N_2O_7$	$CDCl_3$	[252]
2-10-57	(+)-fastrine	$C_{38}H_{42}N_2O_8$	$CDCl_3$	[252]
2-10-58	(+)-limacusine-2′-β-*N*-oxide	$C_{37}H_{40}N_2O_7$	$CDCl_3$	[253]
2-10-59	racemosidine B	$C_{37}H_{40}N_2O_6$	$CDCl_3$	[254]
2-10-60	racemosidine C	$C_{37}H_{40}N_2O_6$	$CDCl_3$	[254]
2-10-61	racemosinine A	$C_{35}H_{36}N_2O_6$	$CDCl_3$-CD_3OD	[254]
2-10-62	racemosinine B	$C_{35}H_{32}N_2O_6$	$CDCl_3$-CD_3OD	[254]
2-10-63	racemosinine C	$C_{35}H_{32}N_2O_7$	$CDCl_3$-CD_3OD	[254]
2-10-64	cavanine	$C_{36}H_{34}N_2O_7$	$CDCl_3$-CD_3OD (9:1)	[255]
2-10-65	puertogaline A	$C_{34}H_{30}N_2O_6$	$CDCl_3$	[249]
2-10-66	puertogaline B	$C_{35}H_{32}N_2O_6$	$CDCl_3$	[249]
2-10-67	(+)-guatteboline	$C_{35}H_{34}N_2O_6$	$CDCl_3$	[249]



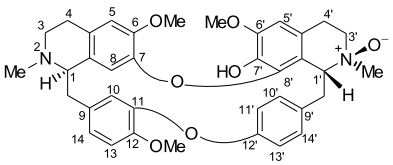
2-10-48 R = H
2-10-49 R = Me
2-10-50 R = Ac



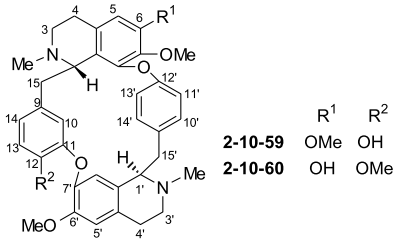
2-10-51 R = Me
2-10-52 R = H



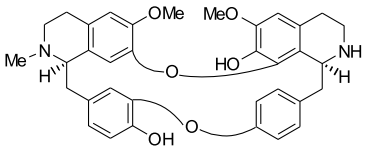
	R^1	R^2	R^3	R^4
2-10-53	H	OMe	OMe	H
2-10-54	H	OH	OH	H
2-10-55	H	OMe	OH	H
2-10-56	H	OMe	OMe	OH
2-10-57	OH	OMe	OMe	OH



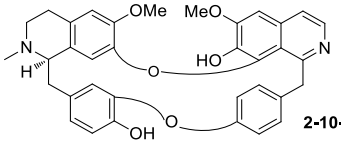
2-10-58



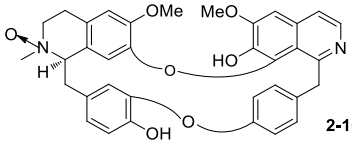
	R^1	R^2
2-10-59	OMe	OH
2-10-60	OH	OMe



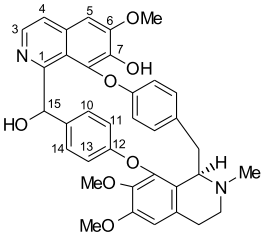
2-10-61



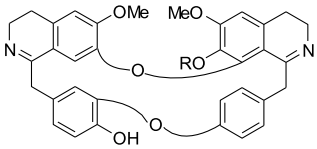
2-10-62



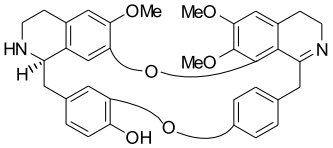
2-10-63



2-10-64



2-10-65 R = H
2-10-66 R = Me



2-10-67

Table 2-10-20: ^{1}H NMR spectroscopic data of dimeric benzylated isoquinoline alkaloids **2-10-48**~**2-10-52**.

H	2-10-48	2-10-49	2-10-50	2-10-51	2-10-52
1					4.03 d(9.7)
5	2.85 (ov), 2.79 (ov)	–	–		
6	2.99 (ov), 2.77 (ov)	–	–		
8	4.06 d(15.2) 3.97 d(15.2)	4.05 d(15.3) 3.99 d(15.3)	–		
9	6.68 s	6.68 s	6.72 s	–	*α* 2.60 d(13.2) *β* 2.79 dd(13.6, 10.4)
11				6.85 br s	6.75 dd(8.1, 1.8)
12	6.26 s	6.23 s	6.28 s	6.85 br s	6.88 d(8.1)
13*α*	3.95 br d(13.9)	3.06 d(13.4)	–		
13*β*	2.58 dd(13.1, 9.9)	2.57 dd(12.7, 10.3)	–		
14	3.51 br d(10.9)	3.50 d(10.0)			
15				6.55 br s	6.423 d(1.8)

Table 2-10-20 (continued)

H	2-10-48	2-10-49	2-10-50	2-10-51	2-10-52
1′	3.67 dd, (11.4, 3.6)	3.66 dd(1.5, 3.4)	–		4.27 dd (11.1, 5.4)
3′	3.37 (ov), 2.87 (ov)	3.40 (ov)	–		
4′	2.96 (ov), 2.73 (ov)	–	–		
5′	6.52 s	6.53 s	6.54 s	6.52 s	6.52 s
8′	5.96 s	5.97 s	5.99 s	6.01 s	6.02 s
15′α	2.75 dd, (12.2, 12.2)	2.75 dd, (13.4, 13.4)	–	–	3.04 dd(11.4, 11.4)
15′β	3.20 dd(12.4, 3.8)	3.20 dd(13.4, 3.6)	–	–	3.30 dd(11.4, 5.0)
10′	6.23 dd(8.8, 1.5)	6.23 br d(8.8)	6.22 dd(8.2, 1.8)	7.36 dd(8.3, 1.9)	7.43 dd(8.2, 2.0)
11′	6.33 dd(8.8, 1.5)	6.34 br d(8.8)	6.36 dd(8.2, 2.2)	7.14 dd(8.3, 2.2)	7.17 dd(8.2, 2.5)
13′	7.28 br s	7.28 br s	7.28 dd(8.0, 2.2)	6.79 dd(8.3, 2.2)	6.85 dd(8.2, 2.5)
14′	7.28 br s	7.28 br s	7.33 dd(8.0, 1.8)	6.30 dd(8.3, 1.9)	6.42 dd(8.0, 2.0)
OMe	3.27 s(2-OMe)	3.25 s(2-OMe)	3.26 s(2-OMe)	3.70 s(6-OMe)	3.73 s(6-OMe)
	3.82 s(3-OMe)	3.82 s(3-OMe)	3.74 s(3-OMe)	3.24 s(7-OMe)	3.34 s(7-OMe)
	3.92 s(10-OMe)	3.81 s(4-OMe)	3.92 s(10-OMe)	3.93 s(13-OMe)	3.96 s(13-OMe)
	3.46 s(6′-OMe)	3.92 s(10-OMe)	3.49 s(6′-OMe)	3.36 s(6′-OMe)	3.40 s(6′-OMe)
		3.48 s(6′-OMe)			
2-NMe				2.29 s	
2′-NMe	2.56 s	2.56 s	2.62 s	2.64 s	
OAc			2.33 s(4-OAc)	2.31 s(5-OAc)	2.32 s(5-OAc)
4-OH	5.50br s				

Table 2-10-21: ^{1}H NMR spectroscopic data of dimeric benzylated isoquinoline alkaloids **2-10-53**~**2-10-57**.

H	2-10-53	2-10-54	2-10-55	2-10-56	2-10-57
1	4.30 d(10)	4.40 d(10)	4.25 t(10)	4.32 t(9.8)	4.35 d(10)
3α	3.25 m	3.25 m	2.90 m	2.90 m	2.96 m
3β	2.90①	2.97 m①	2.90 m①	3.25 m①	3.29 m①
4α	3.05 m	3.00 m	2.90 m	2.90 m	2.74 m
4β	2.90①	2.97 m①	3.00 m①	3.05 m①	3.05 m①
5	6.58 s	6.60 s	6.60 s	6.58 s	
10	7.10 dd(8.6, 2.2)	7.10 dd(8.0, 2.2)	7.01 dd(8.6, 2.0)	7.05 dd(8.6, 2.2)	7.02 dd(8.6, 2.2)
11	6.60 dd(8.5, 2.7)	6.65 dd(8.6, 2.8)	6.62 dd(8.4, 2.7)	6.61 dd(8.5, 2.8)	6.58 dd(8.5, 2.8)
13	5.81 dd(8.3, 2.6)	5.82 dd(8.4, 2.8)	5.81 dd(8.3, 2.7)	5.85 dd(8.3, 2.8)	5.78 dd(8.4, 2.8)
14	6.27 dd(8.3, 2.2)	6.31 dd(7.9, 2.0)	6.25 dd(8.4)	6.30 dd(8.3, 2.3)	6.25 dd(8.2, 2.2)
15α	2.52 m	2.48 m	2.49 m	2.52 m	2.55 m
15β	3.25 m①	3.25 m①	3.20 m①	3.21 m①	3.29 m①
1′	4.30 d(10)	4.40 d(10)	4.25 t(10)	4.32 t(9.8)	4.35 d(10)

Table 2-10-21 (continued)

H	2-10-53	2-10-54	2-10-55	2-10-56	2-10-57
3′α	3.25 m	2.97 m	2.90 m	2.98 m	2.96 m
3′β	2.90 m①	2.97 m①	2.90 m①	3.29 m①	3.29 m①
4′α	2.90 m	2.97 m	2.91 m	2.74 m	2.74 m
4′β	2.90①	2.97①	3.00①	3.10①	3.05①
5′	6.58 s	6.60 s	6.48 s		
10′	7.10 dd(8.6, 2.2)	7.10 dd(8.0, 2.2)	7.05 dd(8.6, 2.0)	7.06 dd(8.6, 2.2)	7.02 dd(8.6, 2.2)
11′	6.60 dd(8.5, 2.7)	6.65 dd(8.6, 2.8)	6.62 dd(8.4, 2.7)	6.58 dd(8.5, 2.8)	6.58 dd(8.5, 2.8)
13′	5.81 dd(8.3, 2.6)	5.82 dd(8.4, 2.8)	5.80 dd(8.3, 2.7)	5.77 dd(8.4, 2.8)	5.78 dd(8.4, 2.8)
14′	6.27 dd(8.2, 2.2)	6.31 dd(7.9, 2.0)	6.25 dd(8.4)	6.28 dd(8.2, 2.2)	6.25 dd(8.2, 2.2)
15′α	2.53 m	2.50 m	2.50 m	2.52 m	2.55 m
15′β	3.26 m①	3.22 m①	3.21 m①	3.25 m①	3.29 m①
OMe	3.82 s(6-OMe)	3.87 s(6-OMe)	3.81 s(6-OMe)	3.82 s(6-OMe)	3.88 s(6-OMe)
	3.40 s(7-OMe)	3.87 s(6′-OMe)	3.40 s(7-OMe)	3.41 s(7-OMe)	3.46 s(7-OMe)
	3.82 s(6′-OMe)		3.82 s(6′-OMe)	3.87 s(6′-OMe)	3.88 s(6′-OMe)
	3.40 s(7′-OMe)			3.46 s(7′-OMe)	3.46 s(7′-OMe)
NMe	2.53 s	2.48 s	2.30 s	2.54 s	2.54 s
N’Me	2.53 s	2.50 s	2.54 s	2.54 s	2.54 s

① Signals not assigned in the original literature.

Table 2-10-22: ^{1}H NMR spectroscopic data of dimeric benzylated isoquinoline alkaloids **2-10-58**~**2-10-60**.

H	2-10-58	2-10-59	2-10-60
1	3.50 m	4.01 br d(9.6)	3.89 br d(8.0)
3	–	3.38 m, 2.85 m	3.25 m, 2.77 m
4	–	2.94 m, 2.55 m	2.92 m, 2.50 m
5	6.36 s	6.56 s	6.59 s
8	6.71 s		
10	6.79 br s	6.38 d(1.6)	6.26 br s
13	6.79 br s	6.78 d(8.0)	6.74 d(8.4)
14	6.89 br s	6.85 dd(8.0, 1.6)	6.81 br d(8.4)
15	–	2.75 m, 2.58 m	2.75 m
1′	4.63 m	3.50 dd(10.4, 3.2)	3.52 dd(8.4, 4.8)
3′	–	3.25 m, 2.75 m	3.30 m, 2.77 m
4	–	2.92 m, 2.85 m	2.90 m, 2.78 m
5′	6.38 s	6.63 s	6.60 s
8′		5.88 s	5.75 s
10′	6.86 dd(2.0, 8.0)	6.60 dd (8.8, 2.0)	6.58 dd(8.0, 2.4)
11′	6.86 dd(2.1, 6.6)	6.88 dd(8.8, 2.0)	6.78 dd(8.0, 2.4)
13′	7.78 dd(2.1, 4.5)	6.74 dd(8.8, 2.0)	6.64 dd(8.0, 2.4)
14′	7.80 dd(2.0, 7.9)	7.12 dd(8.8, 2.0)	6.96 br d(8.0, 2.4)
15′	–	3.20 dd(13.2, 4.0)	3.10 dd(13.2, 3.6)
		2.75 m	2.85 m

Table 2-10-22 (continued)

H	2-10-58	2-10-59	2-10-60
2-NMe	2.53 s	2.27 s	2.15 s
2′-NMe	3.19 s	2.50 s	2.49 s
OMe	3.56 s(6-OMe)	3.87 s(6-OMe)	3.74 s(7-OMe)
	3.81 s(6′-OMe)	3.70 s(7-OMe)	3.72 s(12-OMe)
	3.89 s(12-OMe)	3.84 s(6′-OMe)	3.80 s(6′-OMe)

Table 2-10-23: ^{1}H NMR spectroscopic data of dimeric benzylated isoquinoline alkaloids **2-10-61**~**2-10-63**.

H	2-10-61	2-10-62	2-10-63
1	3.57 br s	3.58 br s	4.23 br s
3	2.78 m, 2.46 m	2.66 m, 2.40 m	2.99 m, 2.90 m
4	2.51 m, 2.51 m	2.66 m, 2.20 m	3.22 m, 2.32 m
5	6.40 s	6.55 s	6.61 s
8	6.64 s	5.95 s	5.92 s
10	5.69 br s	4.74 d(2.0)	4.74 d(1.6)
13	6.78 d(8.0)	6.66 d(8.0)	6.70 d(8.0)
14	6.74 dd(8.0, 1.6)	6.60 dd(8.0, 2.0)	6.49 dd(8.0, 1.6)
15	3.18 m	2.94 dd(14.4, 3.2)	3.07 dd(16.0, 4.4)
	3.00 (14.4, 3.6)	2.40 m	2.77 (16.0, 4.4)
1′	4.54 dd(6.4, 2.0)		
3′	3.33 m, 2.90 m	8.30 d(5.6)	8.30 d(5.6)
4	2.92 m, 2.77 m	7.54 d(5.6)	7.54 d(5.6)
5′	6.37 s	7.03 s	7.03 s
10′	6.82 dd(8.4, 1.6)	6.88 dd(8.4, 1.6)	6.75 d(8.0)
11′	6.45 dd(8.4, 1.6)	6.60 dd(8.4, 2.4)	6.51 dd(8.0, 2.0)
13′	6.87 dd(8.4, 1.6)	6.49 dd(8.0, 2.4)	6.56 dd(8.0, 2.0)
14′	7.43 dd(8.4, 1.6)	7.43 dd(8.0, 1.6)	7.47 br d(8.0)
15′	3.22 m, 3.04 dd(14.0, 6.8)	5.43 d(14.4), 4.43 d(14.4)	5.46 d(14.4), 4.44 d(14.4)
OMe	3.82 s(6′-OMe),3.60 s(6-OMe)	4.05 s(6H)	4.04 s(6′-OMe),4.06 s(6-OMe)
NMe	2.51 s	2.28 s	3.32 s

Table 2-10-24: ^{1}H NMR spectroscopic data of dimeric benzylated isoquinoline alkaloids **2-10-64**~**2-10-67**.

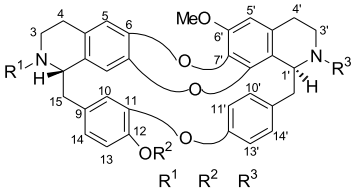
H	2-10-64	2-10-65	2-10-66	2-10-67
1				4.14 br s
3	8.30 d(5.6)	3.69~3.72 m	ax 3.24 m, eq 3.86 m	ax 2.79 m, eq 3.07 m
4	7.51 d(5.6)	2.47~2.51 m	2.53 m	ax 2.35 br d(16)
				eq 2.43 m
5	7.02	6.73 s	6.70 s	6.49 s
8		6.61 s	6.76 s	6.26 s
10	6.13 dd(2.3, 8.6)	5.61 br s	5.85 d(2)	5.13 br s

Table 2-10-24 (continued)

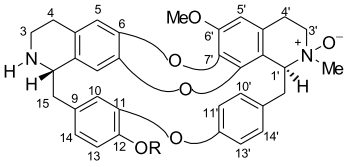
H	2-10-64	2-10-65	2-10-66	2-10-67
11	6.38 dd(2.3, 8.6)			
13	5.64 dd(2.3, 8.6)	6.71 br s	6.79 d(10)	6.75 d(8)
14	6.87 dd(2.3, 8.6)	6.71 br s	6.86 dd(10, 2)	6.64 br d(8)
15	6.38	3.72 m, 3.25 m	3.48 m, 3.80 m	2.76 m, 2.94 dd(14, 3)
1′	4.10 dd(1.1, 11.4)			
3′	3.17 m	3.50~3.60 m	ax 3.56 m	ax 3.63 m
3′	2.77 m		eq 3.88 m	eq 3.88 m
4′	2.90 m	2.65~2.75 m	2.71 m	2.71 m
5′	6.56	6.54 s	6.51 s	6.54 s
10′	6.50 dd(2.3, 8.6)	6.72 m	6.75 dd(8, 2)	7.19 br d(8)
11′	6.66 dd(2.3, 8.6)	6.33 dd(8, 2)	6.35 dd(8, 2)	6.39 dd(8, 2)
13′	5.29 dd(2.3, 8.6)	6.86 dd(8, 2)	6.96 dd(8, 2)	6.78 dd(8, 2)
14′	6.52 dd(2.3, 8.6)	7.12 dd(8, 2)	7.33 dd(8, 2)	7.42 br d(8)
15′	3.03 dd(1.1, 13.3)	4.67 d(15)	4.01 d(14)	4.02 d(14)
	2.49 dd(11.4, 13.3)	3.92 d(15)	4.42 d(14)	4.50 d(14)
2′-NMe	2.25			
OMe	3.94 (6-OMe)	3.95 s(6-OMe)	3.92 s(6-OMe)	3.90 s(6-OMe)
	3.76 (6′-OMe)	3.85 s(6′-OMe)	3.85 s(6′-OMe)	3.86 s(6′-OMe)
	3.40 (7′-OMe)		3.19 s (7′-OMe)	3.33 s (7′-OMe)

Table 2-10-25: Cos, MFs, and TSs of dimeric benzylated isoquinoline alkaloids **2-10-68**~**2-10-76**.

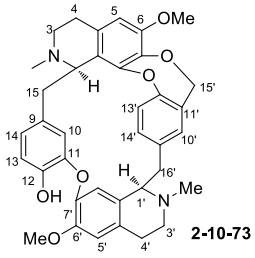
No.	Compounds	MFs	Test solvents	References
2-10-68	(+)-2′-norcocsoline	$C_{33}H_{30}N_2O_5$	$CDCl_3$-CD_3OD (4:0.04)	[256]
2-10-69	(+)-cocsoline	$C_{35}H_{34}N_2O_5$	$CDCl_3$	[256]
2-10-70	(+)-12-*O*-methylcocsoline	$C_{35}H_{34}N_2O_5$	$CDCl_3$	[256]
2-10-71	(+)-12-*O*-methylrorsoline-2′-*β*-*N*-oxide	$C_{35}H_{34}N_2O_6$	$CDCl_3$-CD_3OD (4:0.04)	[256]
2-10-72	(+)-cocsoline-2′-*β*-*N*-oxide	$C_{34}H_{32}N_2O_6$	$CDCl_3$-CD_3OD (4:0.04)	[256]
2-10-73	racemosidine A	$C_{37}H_{38}N_2O_6$	$CDCl_3$	[254]
2-10-74	kurramine-2′-*β*-*N*-oxide	$C_{33}H_{28}N_2O_6$	CD_3OD	[257]
2-10-75	kurramine-2′-*α*-*N*-oxide	$C_{33}H_{28}N_2O_6$	CD_3OD	[257]
2-10-76	1,2-dehydroapateline	$C_{34}H_{30}N_2O_5$	$CDCl_3$-CD_3OD	[257]



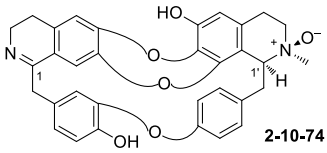
	R^1	R^2	R^3
2-10-68	H	H	H
2-10-69	Me	H	Me
2-10-70	H	Me	Me



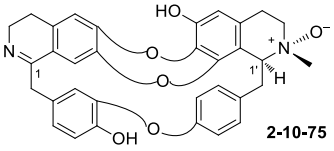
2-10-71 R = Me
2-10-72 R = H



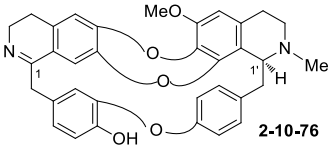
2-10-73



2-10-74



2-10-75



2-10-76

Table 2-10-26: ^{1}H NMR spectroscopic data of dimeric benzylated isoquinoline alkaloids **2-10-68**~**2-10-72**.

H	2-10-68	2-10-69	2-10-70	2-10-71	2-10-72
1	3.73 m	3.40 m	3.62 m	3.78 m	3.72 m
5	6.64 s	6.66 s	6.63 s	6.65 s	6.69 s
8	6.21 s	6.23 s	6.20 s	6.24 s	6.28 s
10	6.49 br s	6.54	6.63	6.57 d(1.2)	6.50 br s
13	6.92 br s	6.94 br s	6.90 br s	6.92 br s	6.92 br s
14	6.92 br s	6.94 br s	6.90 br s	6.92 br s	6.92 br s
1′	4.37 m	4.04 m	4.01 m	4.42 m	4.45 m
5′	6.35 s	6.35 s	6.33 s	6.39 s	6.43 s
10′	7.06 dd(8.3, 2.5)	7.14 dd(8.3, 2.0)	7.11 dd(8.2, 2.0)	7.06 dd(8.3, 2.0)	7.05 dd(8.3, 2.0)
11′	6.79 dd(8.3, 2.5)	6.80 dd(8.3, 2.5)	6.86 dd(8.5, 2.6)	6.81 dd(8.5, 2.6)	6.77 dd(8.5, 2.5)
13′	7.17 dd(8.3, 2.5)	7.22 dd(8.3, 2.5)	7.22 dd(8.3, 2.5)	7.20 dd(8.5, 2.5)	7.20 dd(8.2, 2.0)
14′	7.62 dd(8.3, 1.9)	7.63 dd(7.6, 1.8)	7.54 dd(8.4, 2.0)	8.04 dd(7.5, 1.8)	7.94 dd(8.4, 1.9)
2-Me		2.42			
2′-Me		2.61	2.60	3.30	3.22
6′-OMe	3.85	3.87	3.86	3.88	3.89
12-OMe			3.96	3.95	

Table 2-10-27: ^{1}H NMR spectroscopic data of dimeric benzylated isoquinoline alkaloids **2-10-73**~**2-10-76**.

H	2-10-73	2-10-74	2-10-75	2-10-76
1	4.25 m			
3	3.05 m	3.19 dd(3.0, 15.7)	3.39 m	2.95 m
	2.58 m	3.74 dd(3.9, 15.1)	3.83 m	3.65 m

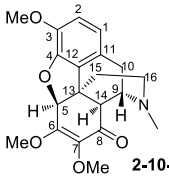
Table 2-10-27 (continued)

H	2-10-73	2-10-74	2-10-75	2-10-76
4	2.61 m	2.33 dt(5.7, 15.9)	2.53 dt(5.9, 15.6)	2.82 m
	2.33 m	2.47 br d(13.8)	2.70 m	2.90 m
5	6.31 s	6.67 s	6.79 s	6.46 s
8		6.79 s	7.00 s	6.52 s
10	5.76 d(1.6)	6.60 br s	6.61 br s	6.38 d(1.3)
13	6.68 d(8.0)	6.77 d(8.0)	6.76 d(8.2)	6.68 d(8.1)
14	6.96 dd(8.0, 1.6)	6.75 dd(8.0, 1.4)	6.78 dd(8.2, 1.3)	6.56 dd(8.3, 1.3)
15	3.13 dd(14.4, 2.4)	3.46 m	3.52 m	3.35 m
	3.40 dd(14.4, 5.2)	3.99 br d(13.2)	3.56 m	3.55 m
1′	3.44 dd(8.0, 3.2)	4.55 br d(10.3)	4.34 br s	4.02 s
3′	3.19 m, 2.75 m	3.46 m, 3.90 m	3.52 m, 3.83 m	2.53 m, 3.76 m
4′	2.85 m	3.04 dd(7.3, 17.3)	3.19 m	2.38 m
	2.65 m	3.24 m	3.25 m	3.52 m
5′	6.58 s	6.45 s	6.53 s	6.30 s
8′	5.36 s			
10′	6.29 br s	6.89 br d(7.9)	6.93 br d(6.5)	6.78 dd(8.0, 1.7)
11′		6.73 br d(7.8)	6.75 br d(7.8)	6.58 dd(8.0, 1.4)
13′	7.29 d(8.4)	7.26 br d(7.7)	7.27 br d(6.5)	7.04 dd(8.1, 1.4)
14′	7.19 dd(8.4, 1.2)	7.55 br d(8.0)	7.64 br d(7.7)	7.28 dd(8.1, 1.7)
15′	4.93 d(14.2)	2.85 t(11.7)	2.70 m	2.65 m
	5.18 d(14.2)	3.52 br d(12.7)	4.28 d(11.9)	3.45 m
16′	3.29 dd(14.4, 3.2)			
	2.58 m			
2-NMe	2.63 s			
2′-NMe	2.50 s	3.64 s	3.16 s	2.50 s
6-OMe	3.80 s			
6′-OMe	3.84 s			3.75 s

2.10.3 Morphine-type isoquinoline alkaloids

Table 2-10-28: Co, MF, and TS of morphine-type isoquinoline alkaloid **2-10-77**.

No.	Compound	MFs	Test solvent	Reference
2-10-77	cephasamine	$C_{20}H_{23}NO_5$	$CDCl_3$	[258]



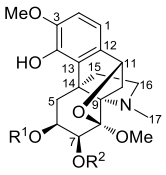
2-10-77

Table 2-10-29: ^{1}H NMR spectroscopic data of morphine-type isoquinoline alkaloid **2-10-77**.

H	2-10-77	H	2-10-77	H	2-10-77
1	6.60 d(7.9)	10	2.52 dd(18.3, 5.8), 3.93 d(18.3)	N-Me	2.44 s
2	6.71 d(7.9)	14	2.95 d(3.1)	3-OMe	3.84 s
5	5.40 s	15	1.84 ddd (12.2, 3.6, 1.6), 2.01 ddd (12.2, 12.2, 4.9)	6-OMe	4.08 s
9	3.81 dd(5.8, 3.1)	16	2.24 ddd (12.2, 12.2, 3.6), 2.54 ddd (12.2, 4.9, 1.6)	7-OMe	3.54 s

Table 2-10-30: Cos, MFs, and TSs of morphine-type isoquinoline alkaloids **2-10-78** and **2-10-79**.

No.	Compounds	MFs	Test solvents	References
2-10-78	*N*-methylstephisoferulin	$C_{30}H_{35}NO_9$	DMSO-d_6	[259]
2-10-79	6-cinnamoylhernandine	$C_{28}H_{31}NO_7$	DMSO-d_6	[259]



	R^1	R^2
2-10-78	Y	Me
2-10-79	X	H

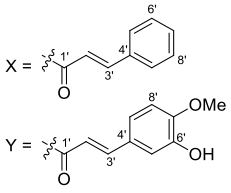
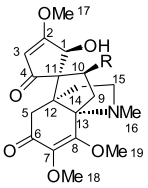


Table 2-10-31: ^{1}H NMR spectroscopic data of morphine-type isoquinoline alkaloids **2-10-78** and **2-10-79**.

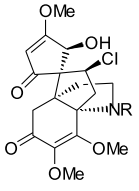
H	2-10-78	2-10-79	H	2-10-78	2-10-79
1	6.67 d(8.6)	6.66 d(8.4)	3-OMe	3.35 s	3.32 s
2	6.44 d(8.6)	6.40 d(8.4)	7-OMe	3.41 s	
5	2.12 dd(2.4, 15.0)	2.20 dd(2.4, 15.6)	8-OMe	3.57 s	3.60 s
	3.14 dd(3.6, 15.0)	3.20 dd(3.6, 15.6)	4-OH	5.70 s	5.70 s
6	5.39 ddd(2.4, 3.6, 3.6)	5.14 ddd(2.4, 3.6, 3.6)	2′	5.33 d(16.2)	5.53 d(15.6)
7	3.78 d(3.6)	4.20 d(3.6)	3′	7.06 d(16.2)	7.12 d(15.6)
10	1.52 d(10.8)	1.57 d(10.8)	5′	6.91 d(1.8)	7.29 m
	2.68 dd (6.6, 10.8)	2.75 dd (6.6, 10.8)			
11	4.96 d(6.6)	4.95 d(6.0)	6′	5.90 br s(OH)	7.29 m
15	1.86 ddd(7.8, 10.8, 13.2)	1.84 m	7′	3.94 s(OMe)	7.29 m
	2.50 ddd(3.6, 10.8, 12.6)	2.51 m			
16	2.46 ddd(3.6, 9.0,12.6)	2.50 m	8′	6.81 d(8.4)	7.29 m
	3.39 m	3.40 m			
NMe	2.57 s	3.07 s	9′	6.86 dd (1.8, 8.4)	7.29 m

Table 2-10-32: Cos, MFs, and TSs of morphine-type isoquinoline alkaloids **2-10-80**~**2-10-87**.

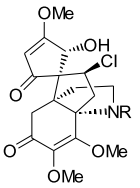
No.	Compounds	MFs	Test solvents	References
2-10-80	dechloroacutumine	$C_{19}H_{25}NO_6$	CD_3OD	[260]
2-10-81	acutumine	$C_{19}H_{24}ClNO_6$	C_5D_5N	[260]
2-10-82	acutumidine	$C_{18}H_{22}ClNO_6$	C_5D_5N	[261]
2-10-83	acutumine	$C_{19}H_{24}ClNO_6$	C_5D_5N	[261]
2-10-84	dauricumidine	$C_{18}H_{22}ClNO_6$	C_5D_5N	[261]
2-10-85	dauricumine	$C_{19}H_{24}ClNO_6$	C_5D_5N	[261]
2-10-86	dechloroacutumidine	$C_{18}H_{23}NO_6$	$CDCl_3$	[262]
2-10-87	1-*epi*dechloroacutumine	$C_{19}H_{25}NO_6$	C_5D_5N	[262]



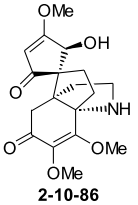
2-10-80 R = H
2-10-81 R = Cl



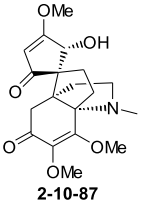
2-10-82 R = H
2-10-83 R = Me



2-10-84 R = H
2-10-85 R = Me



2-10-86



2-10-87

Table 2-10-33: ^{1}H NMR spectroscopic data of morphine-type isoquinoline alkaloids **2-10-80**~**2-10-83**.

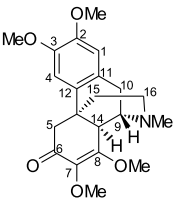
H	2-10-80	2-10-81	2-10-82	2-10-83
1	4.57 s	5.00 s	5.00	5.00 s
3	5.28 s	5.58 s	5.49 s	5.58 s
5	2.26 d(17), 2.49 d(17)	2.50 d(15), 3.02 d(15)	2.42 d(17.3), 2.75 d(17.3)	2.50 d(15), 3.02 d(15)
9	1.92 m, 2.25 m	2.64 dd(7, 12)	2.78 dd(7.0, 12.3)	2.64 dd(7, 12)
		3.14 dd(12, 12)	3.17 dd(12.3, 12.3)	3.14 dd(12, 12)
10	1.51 m, 2.24 m	5.16 dd(7, 12)	5.23 dd(7.0, 12.3)	5.16 dd(7, 12)
14	1.53 m, 2.30 m	1.61 m, 2.65 m	1.60 dd(3.9, 11.6)	1.61 m, 2.65 m
			2.59 ddd(6.3, 11.6, 11.6)	
15	2.51 m	2.43 m	2.81 ddd(3.9, 9.6, 11.6)	2.43 m
	2.74 m	2.65 m	2.92 dd(6.3, 9.6)	2.65 m
16	2.40 s	2.38 s		2.38 s
17	3.88 s	3.71 s	3.64 s	3.71 s
18	3.61 s	3.78 s	3.88 s	3.78 s
19	4.08 s	4.03 s	4.05 s	4.03 s
OH			8.37 br d(5.5)	8.30 br s

Table 2-10-34: ^{1}H NMR spectroscopic data of morphine-type isoquinoline alkaloids **2-10-84**~**2-10-87**.

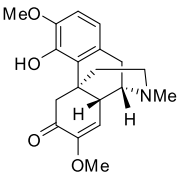
H	2-10-84	2-10-85	2-10-86	2-10-87
1	4.90 s	4.89 d(6.0)	5.03 s	4.54 s
3	5.53 s	5.61 s	5.42 s	5.20 s
5	2.66 d(17.7), 3.44 d(17.7)	2.99 d(16.3), 3.39 d(16.3)	2.54 d(17.1), 2.92 d(17.1)	2.10 d(15.1), 2.87 d(15.1)
9	2.62 dd(6.7, 12.1)	2.45 dd(6.7, 12.0)	2.32 m	1.23 m
	3.08 dd(12.1, 12.1)	3.03 dd(12.0, 12.0)	2.79 m	2.29 m
10	4.87 dd(6.7, 12.1)	4.81 dd(6.7, 12.0)	1.63 m, 2.70 m	1.80 m, 2.57 m
14	2.32 ddd(6.2,11.3, 12.7)	2.28 m, 2.42(ov)	1.93 m, 2.94 m	1.97 m, 2.18 m
	2.40 dd(4.5, 12.7)			
15	2.86 ddd(4.5, 9.6, 11.3)	2.45(ov)	2.94 m,3.03 m	2.41 m,2.93 m
	2.98 dd(6.2, 9.6)	2.62 m		
16		2.35 s		2.33 s
17	3.64 s	3.68 s	3.74 s	3.85 s
18	3.89 s	3.77 s	3.98 s	3.63 s
19	4.06 s	4.07 s	4.17 s	4.02 s
OH	8.15 br s	8.20 d(6.0)	7.93 br s	

Table 2-10-35: Cos, MFs, and TSs of morphine-type isoquinoline alkaloids **2-10-88**~**2-10-94**.

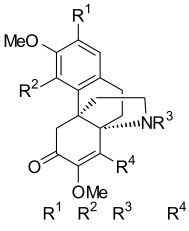
No.	Compounds	MFs	Test solvents	References
2-10-88	tannagine	$C_{21}H_{27}NO_5$	$CDCl_3$	[258]
2-10-89	14-*epi*sinomenine	$C_{19}H_{23}NO_4$	$CDCl_3$	[258]
2-10-90	cephatonine	$C_{20}H_{25}NO_5$	$CDCl_3$	[258]
2-10-91	cepharamine	$C_{19}H_{23}NO_4$	$CDCl_3$	[258]
2-10-92	aknadinine	$C_{20}H_{25}NO_5$	$CDCl_3$	[258]
2-10-93	aknadicine	$C_{19}H_{23}NO_5$	$CDCl_3$	[258]
2-10-94	aknadilactam	$C_{20}H_{23}NO_6$	$CDCl_3$	[258]



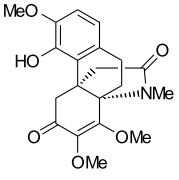
2-10-88



2-10-89



	R^1	R^2	R^3	R^4
2-10-90	OH	H	Me	OMe
2-10-91	H	OH	Me	H
2-10-92	H	OH	Me	OMe
2-10-93	H	OH	H	OMe



2-10-94

Table 2-10-36: ^{1}H NMR spectroscopic data of morphine-type isoquinoline alkaloids **2-10-88**~**2-10-91**.

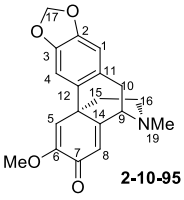
H	2-10-88	2-10-89	2-10-90	2-10-91
1	6.54 s	6.66 d(8.6)	6.56 s	6.59 d(8.2)
2	3.82 s(OMe)	6.73 d(8.6)		6.69 d(8.2)
4	6.63 s		6.65 s	
5	2.49 d(15.9)	2.67 d(17.6)	2.63 d(15.9)	2.49 d(16.8)
	3.17 d(15.9)	4.23 d(17.6)	3.01 d(15.9)	3.73 d(16.8)
8	4.01 s(OMe)	5.76 d(2.1)	4.08 s(OMe)	5.62 s
9	3.52 dd(5.3, 3.1)	3.13 dd (6.1, 2.1)	1.98 ddd	1.78 ddd
			(13.7, 9.8, 4.9)	(14.0, 13.4, 4.7)
			2.10 ddd	1.98 ddd
			(13.7, 5.5, 4.9)	(14.0, 5.0, 2.3)
10	2.65 dd(18.3, 5.8)	2.84ddd (18.0, 6.1, 0.9)	2.51 ddd(16.5, 5.5, 4.9)	2.58 ddd(16.2, 4.7, 2.3)
	2.94 d(18.3)	3.14 d(18.0)	2.72 ddd(16.5, 9.8, 4.9)	2.90 ddd(16.2, 13.4, 5.0)
14	3.06 d(3.1)	2.97 br s		
15	1.49 ddd	1.57 ddd	2.03 ddd	2.00 ddd
	(12.5, 3.4, 1.8)	(12.8, 3.1, 1.5)	(13.1, 9.5, 4.6)	(13.7, 9.8, 3.1)
	1.90 ddd	2.19 ddd	2.16 ddd	2.59 ddd
	(12.5, 12.2, 4.9)	(12.8, 12.3, 4.8)	(13.1, 9.8, 6.1)	(13.7, 9.1, 7.0)
16	2.15 ddd	2.04 ddd	2.78 ddd	2.36 ddd
	(12.2, 11.9, 3.4)	(12.3, 11.9,3.1)	(9.5, 9.2 6.1)	(9.8, 9.1, 7.0)
	2.48 ddd	2.41 ddd	2.82 ddd	2.87 ddd
	(11.9, 4.9,1.8)	(11.9, 4.8, 1.5)	(9.8, 9.2, 4.6)	(9.1, 9.1, 3.1)
NMe	2.45 s	2.36 s	2.52 s	2.41 s
3-OMe	3.81 s	3.87 s	3.84 s	3.85 s
7-OMe	3.32 s	3.71 s	3.63 s	3.65 s

Table 2-10-37: ^{1}H NMR spectroscopic data of morphine-type isoquinoline alkaloids **2-10-92**~**2-10-94**.

H	2-10-92	2-10-93	2-10-94
1	6.56 d(8.2)	6.59 d(8.2)	6.57 d(8.2)
2	6.66 d(8.2)	6.69 d(8.2)	6.72 d(8.2)
5	2.64 d(16.0)	2.50 d(16.8)	2.79 d(16.8)
	3.50 d(16.0)	3.63 d(16.8)	3.48 d(16.8)
9	1.90 ddd(13.4, 11.3, 4.6)	1.82 ddd(13.4, 13.1, 5.2)	2.17 ddd(14.1, 11.0, 5.4)
	2.15 ddd(13.4, 4.9, 4.9)	2.14 ddd(13.4, 4.9, 1.8)	2.32 ddd(14.1, 5.1, 4.9)
10	2.56 ddd(16.2, 4.9, 4.6)	2.61 ddd(17.0, 4.9, 1.8)	2.64 ddd(16.8, 11.0, 4.9)
	2.79 ddd(16.2, 11.3, 4.9)	3.06 ddd(17.0, 13.1, 5.2)	2.71 ddd(16.8, 5.1, 5.1)
15	2.11 ddd(14.0, 9.5, 4.0)	2.17 m	2.76 d(17.1)
	2.47 ddd(14.0, 10.1, 6.4)	2.64 m	3.04 d(17.1)
16	2.67 ddd(9.7, 9.5, 6.4)	2.84 m	–
	2.83 ddd(10.1, 9.7, 4.0)	2.86 m	
NMe	2.53 s		2.96 s
3-OMe	3.83 s	3.85 s	3.85 s
7-OMe	3.65 s	3.69 s	3.69 s
8-OMe	4.07 s	4.13 s	4.11 s

Table 2-10-38: Cos, MFs, and TSs of morphine-type isoquinoline alkaloids **2-10-95**~**2-10-100**.

No.	Compounds	MFs	Test solvents	References
2-10-95	(–)-amurine	$C_{19}H_{19}NO_4$	$CDCl_3$	[229]
2-10-96	cephakicine	$C_{23}H_{29}NO_7$	$CDCl_3$	[258]
2-10-97	sinoacutine	$C_{19}H_{21}NO_4$	$CDCl_3$	[258]
2-10-98	9*S*,17*S*-pallidine N_α-oxide	$C_{19}H_{21}NO_5$	CD_3CN	[233]
2-10-99	(+)-14-hydroxy-isostephodeline	$C_{21}H_{27}NO_6$	$CDCl_3$	[263]
2-10-100	(+)-isostephodeline	$C_{21}H_{27}NO_5$	$CDCl_3$	[263]



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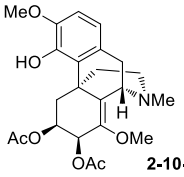

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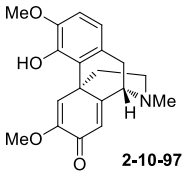

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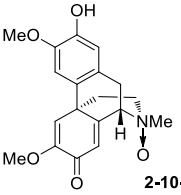
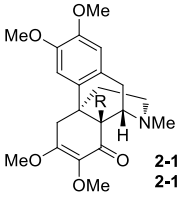


2-10-98



2-10-99 R = OH
2-10-100 R = H

Table 2-10-39: ^{1}H NMR spectroscopic data of morphine-type isoquinoline alkaloids **2-10-95**~**2-10-97**.

H	2-10-95	2-10-96	2-10-97
1	6.52 s	6.62 d(8.2)	6.67 d(8.2)
2		6.72 d(8.2)	6.75 d(8.2)
4	6.77 s		
5	6.23 s	2.35 dd(13.3, 12.8) 2.86 dd(12.8, 3.8)	7.55 s
6	3.71 s(OMe)	5.24 ddd(13.3, 3.8, 3.8)	3.75 s(OMe)
7		5.92 dd(3.8, 0.7)	
8	6.25 s	3.55 s(OMe)	6.33 s
9	3.60 d(6)	4.15 d(5.2)	3.69 d(5.8)
10	3.24 d(18) 2.92 dd(6,18)	2.87 ddd(17.7, 5.8, 1.0) 3.14 d(17.7)	2.98 ddd(17.7, 5.8, 1.2) 3.33 d(17.7)
15	1.85 td(6.4,11.5) 1.75 dt(2.5,12.6)	1.87 ddd(12.5, 3.1, 1.8) 2.10 ddd(12.5, 12.2, 4.6)	1.77 ddd(12.8,3.1,1.8) 2.37 ddd(12.8, 12.6, 4.6)
16	2.5 d(2.5), 2.52 m	2.55 ddd(12.5, 4.6, 1.8) 2.38 ddd(12.5,12.2,3.1)	2.49 ddd(12.6, 12.5 3.1) 2.61 ddd(12.5, 4.6,1.8)

Table 2-10-39 (continued)

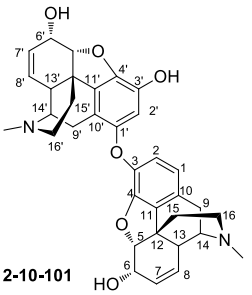
H	2-10-95	2-10-96	2-10-97
17	5.86 d(1.3), 5.83 d(1.3)		
NMe	3.37 s	2.40 s	2.45 s
3-OMe		3.87 s	3.89 s
OAc		2.01 s(6-OAc), 2.04 s(7-OAc)	

Table 2-10-40: ^{1}H NMR spectroscopic data of morphine-type isoquinoline alkaloids **2-10-98**~**2-10-100**.

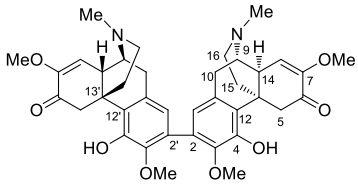
H	2-10-98 · CF_3COOH	2-10-99	2-10-100
1	6.66 s	6.51	6.52
4	7.08 s	6.61	6.63
5	6.74 s	3.15 d, 2.83 d(17.5)	3.11 d, 2.68 d(17.4)
8	6.45 s		
9	4.80 d	3.43 d(6.6)	3.66 m
10	–	3.00 d, 2.91 dd(18.4, 6.6)	2.87 d, 2.79 dd(18.3, 6.0)
15	–	2.16 m, 1.33 m	1.92 m, 1.53 m
16	–	2.40 m, 2.16 m	2.48 m, 2.10 m
NMe	3.61 s	2.41	2.45
2-OMe		3.81	3.81
3-OMe	3.89 s	3.82	3.83
6-OMe	3.78 s	4.00	3.98
7-OMe		3.41	3.35

Table 2-10-41: Cos, MFs, and TSs of morphine-type isoquinoline alkaloids **2-10-101**~**2-10-104**.

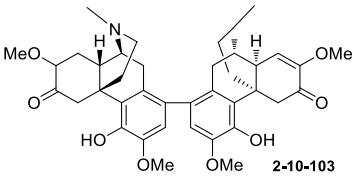
No.	Compounds	MFs	Test solvents	References
2-10-101	bismorphine B	$C_{34}H_{36}N_2O_6$	DMSO-d_6	[264]
2-10-102	2,2′-disinomenine	$C_{38}H_{44}N_2O_8$	$CDCl_3$	[265]
2-10-103	7′,8′-dihydro-1,1′-disinomenine	$C_{38}H_{46}N_2O_8$	$CDCl_3$	[265]
2-10-104	1,1′-disinomenine	$C_{38}H_{44}N_2O_8$	$CDCl_3$	[265]



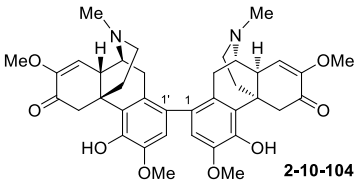
2-10-101



2-10-102



2-10-103



2-10-104

Table 2-10-42: ^{1}H NMR spectroscopic data of morphine-type isoquinoline alkaloids **2-10-101**~**2-10-104**.

H	2-10-101	2-10-102	2-10-103	2-10-104
1	6.57 d(8.2)	6.44 s		
2	6.50 d(8.2)		6.38 s	6.26 s
5*α*		2.48 d(15.6)	2.47 d(15.6)	2.47 d(15.2)
5*β*	4.68 d(6.2)	4.42 d(15.6)	4.39 d(15.6)	4.40 d(15.2)
6	4.08 m			
7	5.27 ddd(9.5, 3.0, 2.9)			
8	5.54 br d(9.5)	5.27 d(2.0)	5.37 d(2.0)	5.43 d(2.0)
9	2.79 d(17.5) 2.07 dd(17.5, 6.5)	2.98 t(2.0)	3.09 t(4.0)	3.08 t(4.0)
10*α*	–	2.53 d(18.8)	2.62 d(18.8)	2.32 d(10.8)
10*β*	–	1.75 dd(5.2, 18.8)	1.80 dd (18.8, 5.2)	2.39 dd(5.2, 18.0)
13	2.50 m			
14	3.19 dd(6.5, 3.0)	2.99 br d(2.0)	2.98 br s	2.99 br s
15	1.93 ddd(11.2, 11.0, 3.8)	*α* 2.01 dd(3.7, 12.4)	*α* 2.01 m	*α* 2.01 dd (2.4, 12.4)
	1.61 br d(11.2)	*β* 1.91 dd(4.0, 12.4)	*β* 1.92 m	*β* 1.92 ddd(4.4, 6.0, 12.4)
16	2.44 dd(11.2, 3.8)	*α* 2.05 dd(3.7, 11.8)	*α* 2.09 m	*α* 2.17 ddd(2.4, 6.0, 12.4)
	2.25 ddd(11.2, 11.0, 3.8)	*β* 2.57 dd(4.0, 11.8)	*β* 2.58 m	*β* 2.55 dd(4.4, 12.4)
NMe	2.28 s	2.31 s	2.34 s	2.33 s
3-OMe		3.76 s	3.78 s	3.73 s
7-OMe		3.51 s	3.44 s	3.50 s
1′		6.44 s		
2′	5.87 s		6.47 s	6.26 s
5′*α*		2.48 d(15.6)	2.29 d(15.2)	2.47 d(15.2)
5′*β*	4.74 d(6.2)	4.42 d(15.6)	4.37 d(15.2)	4.40 d(15.2)
6′	4.13 m			
7′	5.31 ddd(9.5, 3.0, 2.9)	–	3.92 dd(10.0, 6.8)	–
8′	5.57 br d(9.5)	5.27 d(2.0)	*α* 1.53 dd(12.4, 10.0) *β* 2.07 m	5.43 d(2.0)
9′	2.94 d(17.6) 2.27 dd(17.6, 6.5)	2.98 t(2.0)	2.81 br s	3.08 t(4.0)
10′*α*		2.53 d(18.8)	2.52 m	2.32 d(18.0)
10′*β*		1.75 dd(5.2, 18.8)	2.48 m	2.39 dd(5.2, 18.0)
13′	2.58 m			
14′	3.29 dd(6.5, 3.0)	2.99 br d(2.0)	2.35 m	2.99 br s

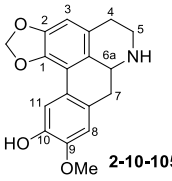
Table 2-10-42 (continued)

H	2-10-101	2-10-102	2-10-103	2-10-104
15′	2.01 ddd(11.2, 11.0, 3.8)	*α* 1.91 dd(4.0, 12.4)	*α* 2.00 m	*α* 2.01 dd(2.4, 12.4)
	1.66 br d(11.2)	*β* 2.01 dd(3.7, 12.4)	*β* 1.99 m	*β* 1.92 ddd(4.4, 6.0 12.4)
16′	2.48 dd(11.2, 3.8)	*α* 2.05 dd(3.7, 11.8)	*α* 2.06 m	*α* 2.17 ddd(2.4, 6.0, 12.4)
16′	2.28 ddd(11.2, 11.0, 3.8)	*β* 2.57 dd(4.0, 11.8)	*β* 2.57 m	*β* 2.55 dd(4.4, 12.4)
NMe	2.28 s	2.31 s	2.31 s	2.33 s
3′-OMe		3.76 s	3.81 s	3.73 s
7′-OMe		3.51 s	3.46 s	3.50 s

2.10.4 Aporphine-type isoquinoline alkaloids

Table 2-10-43: Cos, MFs, and TSs of aporphine-type isoquinoline alkaloids **2-10-105**~**2-10-130**.

No.	Compounds	MFs	Test solvents	References
2-10-105	litseglutine A	$C_{18}H_{17}NO_4$	CD_3OD	[266]
2-10-106	litseglutine B	$C_{20}H_{23}NO_4$	CD_3OD	[266]
2-10-107	1,2-methylenedioxy-9-methoxy-*N*-methoxycarbonyl-aporphine	$C_{20}H_{19}NO_6$	$CDCl_3$	[267]
2-10-108	1,2-methylenedioxy-8,9-dimethoxy-*N*-methoxy-carbonyl-aporphine	$C_{21}H_{21}NO_6$	$CDCl_3$	[267]
2-10-109	*β*-magnoflorine	$C_{20}H_{24}NO_4$	CD_3OD	[268]
2-10-110	*α*-magnoflorine	$C_{20}H_{24}NO_4$	CD_3OD	[268]
2-10-111	*N*,*O*-diacetylnoroliveroline	$C_{21}H_{19}NO_5$	$CDCl_3$	[269]
2-10-112	*N*,*O*-diacetyl-(–)-nornuciferidine	$C_{22}H_{23}NO_5$	$CDCl_3$	[269]
2-10-113	*N*-nitrosoanonaine	$C_{17}H_{14}N_2O_3$	$CDCl_3$	[270]
2-10-114	*N*-nitrosoxylopine	$C_{18}H_{16}N_2O_4$	$CDCl_3$	[270]
2-10-115	stesakine-9-*O*-*β*-D-glucopyranoside	$C_{25}H_{29}NO_9$	C_5D_5N	[271]
2-10-116	*N*-methylasimilobine-2-*O*-*β*-D-glucopyranoside	$C_{24}H_{29}NO_7$	C_5D_5N	[271]
2-10-117	stesakine	$C_{19}H_{19}NO_4$	C_5D_5N	[271]
2-10-118	*N*-methylasimilobine	$C_{18}H_{19}NO_2$	C_5D_5N	[271]
2-10-119	(+)-6*R*,6a*S*-isocorydine N_β-oxide	$C_{20}H_{23}NO_5$	$CDCl_3$	[272]
2-10-120	(+)-6*R*,6a*S*-corydine N_β-oxide	$C_{20}H_{23}NO_5$	$CDCl_3$	[272]
2-10-121	(+)-*N*-methyl-laurotetanine	$C_{20}H_{23}NO_4$	CD_3OD	[272]
2-10-122	cathafiline	$C_{20}H_{19}NO_6$	$CDCl_3$	[273]
2-10-123	cathaformine	$C_{21}H_{21}NO_7$	$CDCl_3$	[273]
2-10-124	(+)-2,l0-dimethoxyaporphine	$C_{19}H_{21}NO_2$	$CDCl_3$	[274]
2-10-125	romucosine F	$C_{22}H_{24}ClNO_6$	$CDCl_3$	[275]
2-10-126	romucosine G	$C_{23}H_{27}NO_7$	$CDCl_3$	[275]
2-10-127	10,11-dihydroxy-1,2-dimethoxynoraporphine	$C_{18}H_{19}NO_4$	$CDCl_3$	[231]
2-10-128	artabonatine B	$C_{18}H_{17}NO_4$	CD_3OD	[276]
2-10-129	artabonatine A	$C_{18}H_{13}NO_4$	$CDCl_3$	[276]
2-10-130	artabonatine E	$C_{19}H_{15}NO_5$	$CDCl_3$	[277]



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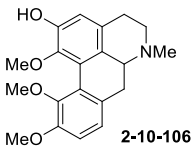

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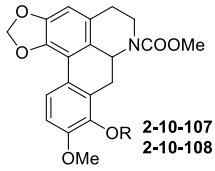
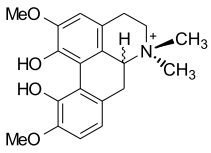
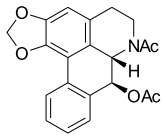


2-10-107 R = H
2-10-108 R = Me



2-10-109 6aβ-H
2-10-110 6aα-H



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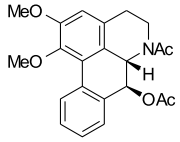

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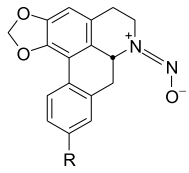
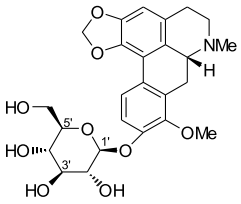


2-10-113 R = H
2-10-114 R = OMe



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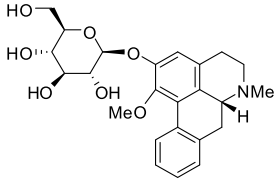

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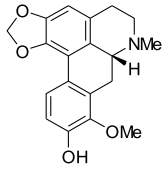

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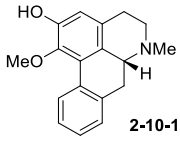
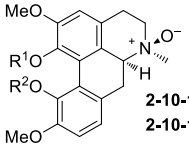
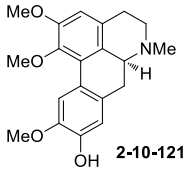


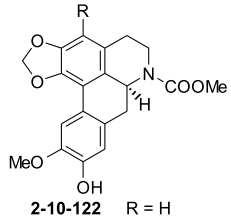
2-10-118



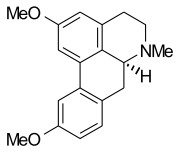
2-10-119 Me H
2-10-120 H Me



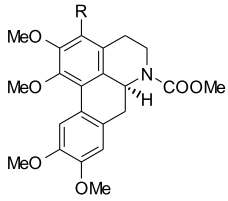
2-10-121



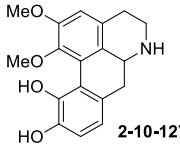
2-10-122 R = H
2-10-123 R = OMe



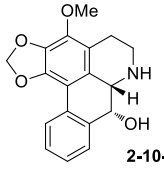
2-10-124



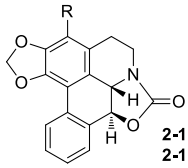
2-10-125 R = Cl
2-10-126 R = OMe



2-10-127



2-10-128



2-10-129 R = H
2-10-130 R = OMe

Table 2-10-44: ^{1}H NMR spectroscopic data of aporphine-type isoquinoline alkaloids **2-10-105**~**2-10-109**.

H	2-10-105	2-10-106	2-10-107	2-10-108	2-10-109
3	6.42 s	6.69 s	6.54 s	6.56 s	6.40 s
4	2.91 m, 2.58 m	2.82 m, 2.76 m	2.60 m, 2.87 m	2.58 m, 2.82 m	*α* 3.09 m, *β* 2.49 m
5	3.26 m, 2.91 m	3.21 m, 2.80 m	3.00 m, 4.42 m	2.99 m, 4.41 m	*α* 3.06 m, *β* 3.38 m
6a	3.80 dd(9.7, 4.6)	3.31 dd(9.5, 4.3)	4.85 m	4.76 m	3.67 m
7	2.70 dd(14.1, 4.9)	3.18 dd(13.8,4.3)	2.87 m	2.52 m,3.50 m	*α* 2.26 t(12.0)
	2.52 dd(14.1, 4.9)	2.45 dd(13.8,4.3)			*β* 2.86 m
8	6.59 s	7.05 d(8.2)	6.81 d(2.5)	3.83 s(OMe)	6.43 d(7.9)
9	3.74 s(OMe)	6.95 d(8.2)	3.84 s(OMe)	3.91 s(OMe)	6.64 d(8.0)
10		3.88 s(OMe)	6.86 dd(8.5, 2.5)	6.88 d(8.7)	3.80 s(OMe)
11	7.54 s	3.61 s(OMe)	8.03 d(8.5)	7.84 d(8.7)	
OMe		3.44 s(1-OMe)			3.72 s(2-OMe)
OCH_2O	5.95 br s		6.06 d(1.3)	6.07 d(1.2)	
	5.81 br s		5.95 d(1.3)	5.95 d(1.2)	
NMe		2.70 s			*α* 2.68 s, *β* 3.17 s
COOMe			3.79 s	3.77 s	

Table 2-10-45: ^{1}H NMR spectroscopic data of aporphine-type isoquinoline alkaloids **2-10-110**~**2-10-114**.

H	2-10-110	2-10-111	2-10-112	2-10-113	2-10-114
3	6.48 s	6.61 s	6.70 s	6.52 s	6.53 s
4	*α* 2.63 m	2.56 m,	2.62 m,	*α* 2.87 ddd(15.4,	*α* 2.87 ddd(15.4,
		2.76 m	2.81 m	3.3, 1.6)	2.2, 1.3)
	β 3.17 m			*β* 3.03 ddd(15.4,	*β* 3.03 ddd(15.4,
				12.5, 4.5)	12.3, 4.5)
5	*α* 3.45 m	2.76 m,	2.78 m,	*α* 5.08 ddd(12.5,	*α* 5.08 ddd(12.3,
		4.82 m	4.86 m	4.5, 1.6)	4.5, 1.3)
	β 3.11 m			*β* 3.95 td(12.5,	*β* 3.96
				12.5, 3.3)	dt(12.3,12.3,2.2)
6a	3.48 m	4.83 d(12.3)	4.71 d(11.9)	5.37 dd(14.0, 4.3)	5.33 dd(14.0, 4.3)
7	*α* 2.96 m	6.25 d(12.3)	6.18 d(11.9)	*α* 2.59 br t(14.0)	*α* 2.59 br t(14.0)
	β 2.45 t(11.6)			*β* 3.12 dd(14.0, 4.3)	*β* 3.09 dd(14.0, 4.3)
8	6.49 d(7.1)	7.32~7.43 m	7.34~7.45 m	7.17~7.31 m	6.77 br d(2.6)
9	6.67 d(7.5)	7.32~7.43 m	7.34~7.45 m	7.17~7.31 m	3.83 s(OMe)
10	3.81 s(OMe)	7.32~7.43 m	7.34~7.45 m	7.17~7.31 m	6.83 dd(8.3, 2.6)
11		8.09 d(7.6)	8.45 d(7.9)	8.01 dd(7.6, 2.0)	7.99 d(8.3)
NMe	*α* 3.23 s, *β*				
	2.79 s				
NAc		2.18 s	2.17 s		
OMe	3.76 s(2-OMe)		3.67 s(1-OMe)		
			3.90 s(2-OMe)		
OAc		2.26 s	2.26 s		
OCH_2O		6.12 d(1.2)		6.00 d(1.8)	5.95 d(1.2)
		6.01 d(1.2)		5.90 d(1.8)	6.05 d(1.2)

Table 2-10-46: ^{1}H NMR spectroscopic data of aporphine-type isoquinoline alkaloids **2-10-115**~**2-10-119**.

H	2-10-115	2-10-116	2-10-117	2-10-118	2-10-119
3	6.60 s	7.39 s	6.60 s	7.01 s	6.79 s
4	2.51 ddd (16.2, 3.7, 1.2) 3.12 ddd (16.2, 10.7, 5.5)	2.50 dd (17.7, 3.4) 3.11 ddd (17.7, 11.9, 5.5)	2.52 m 3.12 m	2.57 dd (17.1, 3.4) 3.14 ddd (17.1, 11.9, 4.9)	3.85 m 2.78 m
5	2.39 ddd (11.6, 10.7, 3.7) 2.94 ddd (11.6, 5.5, 1.2)	2.23 ddd (11.9, 11.6, 3.4) 2.84 dd (11.6, 5.5)	2.40 ddd (12.1, 11.9,4.0) 2.94 ddd (11.9, 5.8, 1.2)	2.36 ddd (11.9, 11.6, 3.4) 2.90 dd (11.6, 4.9)	3.80 m 3.53 m
6a	3.08 dd(13.7, 4.3)	2.96 dd(13.7, 4.0)	3.11 dd(13.7, 4.3)	3.01 dd(13.7, 3.7)	4.08 d(12.0)
7	2.48 dd(13.7, 13.7) 3.91 dd(13.7, 4.3)	2.75 dd(13.7, 13.7) 3.21 dd(13.7, 4.0)	2.54 dd(14.3, 13.7) 3.91 dd(13.4, 4.3)	2.78 dd(13.7, 13.7) 3.22 dd(13.7, 3.7)	3.22 t(13.2) 3.10 dd(13.6, 2.8)
8	4.01 s(OMe)	7.38 d(7.6)	3.89 s(OMe)	7.40 d(7.3)	6.89 d(8.4)
9		7.31 dd(7.6, 7.6)		7.32 dd(7.6,7.3)	6.87 d(8.0)
10	7.71 d(8.6)	7.42 dd(7.9, 7.6)	7.30 d(8.6)	7.43 dd(8.2, 7.6)	3.92 s(OMe)
11	8.01 d(8.6)	8.70 d(7.9)	8.07 d(8.6)	8.72 d(8.2)	
1′	5.71 d(7.3)	5.64 d(7.0)			
2′	4.38 m	4.38 m			
3′	4.38 m	4.37 m			
4′	4.35 m	4.31 m			
5′	4.19 ddd(8.9, 5.5, 2.1)	4.16 ddd(7.6, 5.2, 2.1)			
6′	4.42 dd(11.9, 5.5) 4.60 dd(11.9, 2.1)	4.40 dd(11.9, 5.5) 4.62 dd(11.9, 2.1)			
NMe	2.48 s	2.41 s	2.50 s	2.43 s	3.48 s
OMe		3.93 s(1-OMe)		3.73 s(1-OMe)	3.68 s(1-OMe) 3.92 s(2-OMe)
OCH_2O	5.98 d(1.2) 6.11 d(1.2)		6.00 d(1.2) 6.11 d(1.2)		
OH			11.69 br s(9-OH)	11.18 br s(2-OH)	8.79 s(11-OH)

Table 2-10-47: ^{1}H NMR spectroscopic data of aporphine-type isoquinoline alkaloids **2-10-120**~**2-10-124**.

H	2-10-120	2-10-121	2-10-122	2-10-123	2-10-124
1	8.74 s(OH)	3.65 s(OMe)			7.08 d(2.4)
3	6.76 s	6.58 s	6.55 s	4.00 s(OMe)	6.61 d(2.4)

Table 2-10-47 (continued)

H	2-10-120	2-10-121	2-10-122	2-10-123	2-10-124
4	3.82 m, 2.75 m	2.52~3.19 m	2.81 m, 2.60 m	2.62 m, 2.46 m	–
5	3.78 br s, 3.57 m	2.52~3.19 m	4.40 m, 2.97 m	4.40 m, 2.90 m	–
6a	4.14 d(10.0)	2.52~3.19 m	4.82 m	4.79 m	–
7	3.28 t(13.2)	2.52~3.19 m	2.81 m	2.90 m	–
	3.14 dd(13.2, 2.4)		2.97 m	2.62 m	
8	6.92 d(8.4)	6.81 s	6.83 s	6.82 s	7.16 d(8.2)
9	7.17 d(8.0)				6.79 dd(2.6, 8.2)
11	3.74 s(OMe)	8.06 s	7.67 s	7.62 s	7.21 d(2.6)
NMe	3.47 s	2.55 s			2.53 s
OMe	3.93 s(2-OMe)	3.88 s(2-OMe)	3.94 s(10-OMe)	3.93 s(10-OMe)	3.83 s(2-OMe)
	3.93 s(10-OMe)	3.89 s(10-OMe)			3.84 s(10-OMe)
OCH_2O			6.09 d(1.6)	6.09 d(1.6)	
			5.96 d(1.6)	5.94 d(1.6)	
NCOOMe			3.76 s	3.76 s	
OH			5.47 br	5.66 br	

Table 2-10-48: ^{1}H NMR spectroscopic data of aporphine-type isoquinoline alkaloids **2-10-125**~**2-10-127**.

H	2-10-125	2-10-126	2-10-127
3		3.90 s(OMe)	6.71 s
4	–	–	2.70 m, 3.00 m
5	4.46 m	4.45 m	3.00 m, 3.36 m
6a	4.75 dd(14.0, 4.3)	4.72 dd(13.6, 4.2)	3.76 br dd(13.5, 4.0)
7	–	–	2.52 br t(13.5), 2.81 dd(13.5, 4.0)
8	6.79 s	6.78 s	7.02 dd(8.0, 0.5)
9	3.93 s(OMe)	3.93 s(OMe)	6.92 d(8.0)
11	8.07 s	8.03 s	
OMe	3.73 s(1-OMe)	3.72 s(1-OMe)	3.69 s(1-OMe)
	3.96 s(2-OMe)	3.97 s(2-OMe)	3.92 s(2-OMe)
	3.94 s(10-OMe)	3.94 s(10-OMe)	
NCOOMe	3.77 s	3.77 s	

Table 2-10-49: ^{1}H NMR spectroscopic data of aporphine-type isoquinoline alkaloids **2-10-128**~**2-10-130**.

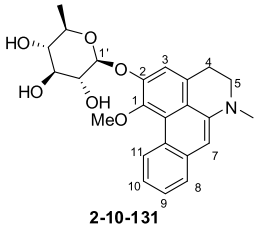
H	2-10-128	2-10-129	2-10-130
3	4.05 s(OMe)	6.73 s	4.03 s(OMe)
4	2.79 ddd(16.0, 2.6, 1.3)	2.84 ddd(16.0, 2.7, 1.5)	2.40 m
	3.12 ddd(16.0, 12.5, 4.5)	2.81 ddd(16.0, 12.5, 4.6)	2.38 m

Table 2-10-49 (continued)

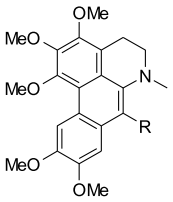
H	2-10-128	2-10-129	2-10-130
5	3.66 ddd(12.5, 4.5, 1.3)	2.90 ddd(12.5, 12.5, 2.7)	3.66 m
	3.50 ddd(12.5, 12.5, 2.6)	3.73 ddd(12.5, 4.6, 1.5)	3.41 m
6a	4.10 d(3.2)	4.72 d(7.9)	4.66 d(7.6)
7	4.66 d(3.2)	5.61 d(7.9)	5.60 d(7.6)
8	7.37~7.42 m	7.49~7.55 m	7.45~7.51 m
9	7.27 td(8.0, 1.2)	7.37 td(8.1, 1.2)	7.34 td(8.1, 1.2)
10	7.37~7.42 m	7.49~7.55 m	7.45~7.51 m
11	8.11 dd(8.0, 1.2)	8.18 dd(8.2, 1.2)	8.24 dd(8.2, 1.2)
OCH_2O	6.11 d(1.6), 5.98 d(1.6)	6.14 d(1.6), 6.04 d(1.6)	6.13 d(1.6), 6.01 d(1.6)

Table 2-10-50: Cos, MFs, and TSs of aporphine-type isoquinoline alkaloids **2-10-131**~**2-10-140**.

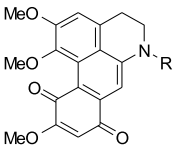
No.	Compounds	MFs	Test solvents	References
2-10-131	(–)-6a,7-dehydrofloripavidine	$C_{24}H_{27}NO_6$	$CDCl_3$	[278]
2-10-132	7-hydroxy-dehydrothalicsimidine	$C_{22}H_{25}NO_6$	$CDCl_3$	[279]
2-10-133	7-formyl-dehydrothalicsimidine	$C_{23}H_{25}NO_6$	$CDCl_3$	[279]
2-10-134	isocorydione	$C_{20}H_{19}NO_5$	$CDCl_3$	[274]
2-10-135	norisocorydione	$C_{19}H_{17}NO_5$	$CDCl_3$	[274]
2-10-136	7-formyldehydrohernangerine	$C_{19}H_{15}NO_5$	$CDCl_3$	[280]
2-10-137	laurodionine	$C_{20}H_{15}NO_6$	DMSO-d_6	[281]
2-10-138	fissilandione	$C_{19}H_{15}NO_5$	$CDCl_3$	[282]
2-10-139	norfissilandione	$C_{18}H_{13}NO_5$	$CDCl_3$	[282]
2-10-140	bulbodione	$C_{19}H_{15}NO_5$	$CDCl_3$	[282]



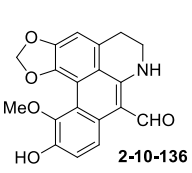
2-10-131



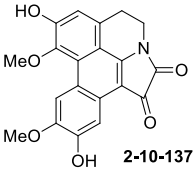
2-10-132 R = OH
2-10-133 R = CHO



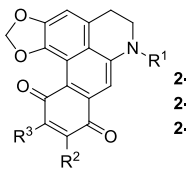
2-10-134 R = Me
2-10-135 R = H



2-10-136



2-10-137



	R^1	R^2	R^3
2-10-138	Me	OMe	H
2-10-139	H	OMe	H
2-10-140	Me	H	OMe

Table 2-10-51: ^{1}H NMR spectroscopic data of aporphine-type isoquinoline alkaloids **2-10-131**~**2-10-135**.

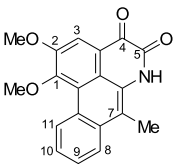
H	2-10-131	2-10-132	2-10-133	2-10-134	2-10-135
3	7.17 s	3.96 s(OMe)	3.94 s(OMe)	6.89 s	6.94 s
4	3.10 m	3.25 t(6.8)	3.16 t(6.8)	3.08 t(6.6)	3.12 t(6.2)
5	3.22 m	3.29 t(6.8)	3.52 t(6.8)	3.43 t(6.6)	3.53 m
7	6.58 s		10.26 s(CHO)	6.91 s	6.97 s
8	7.60 m	7.07 s	8.68 s		
9	7.40 m	4.02 s(OMe)	4.07 s(OMe)	5.87 s	5.89 s
10	7.24 m	4.04 s(OMe)	4.03 s(OMe)	3.85 s(OMe)	3.88 s(OMe)
11	9.32 d(8.6)	8.99 s	8.89 s		
1′	5.69 br s				
2′	4.41 br s				
3′	4.25 dd (9.3, 3.2)				
4′	3.76 t (9.3)				
5′	4.00 m				
6′	1.35 d(6.20)				
OMe	3.86 s(1-OMe)	3.95 s(1-OMe)	3.87 s(1-OMe)	3.94 s(1-OMe)	3.98 s(1-OMe)
		4.08 s(2-OMe)	4.10 s(2-OMe)	3.90 s(2-OMe)	3.95 s(2-OMe)
NMe	3.01 s	3.06 s	3.36 s	3.13 s	

Table 2-10-52: ^{1}H NMR spectroscopic data of aporphine-type isoquinoline alkaloids **2-10-136**~**2-10-140**.

H	2-10-136	2-10-137	2-10-138	2-10-139	2-10-140
3	6.97 s	7.09 s	6.94 s	6.96 s	6.91 s
4	3.15 t(6.8)	3.19 t(6.4)	3.18 t(6.4)	3.15 t(6.5)	3.17 t(6.4)
5	3.63 dt(6.8, 2.8)	3.76 t(6.4)	3.52 t(6.4)	3.55 t(6.4)	3.55 t(6.4)
7	10.46 s(CHO)		7.00 s	7.00 s	7.02 s
8	7.82 d(9.0)	7.84 s			
9	7.21 d(9.0)		3.85 s(OMe)	3.85 s(OMe)	5.95 s
10	5.95 s(OH)	3.89 s(OMe)	6.05 s	6.05 s	3.89 s(OMe)
11	3.61 s(OMe)	8.84 s			
OMe		3.83 s(1-OMe)			
NMe			3.23 s		3.26 s
OCH_2O	6.17 s		6.14 s	6.14 s	6.13 s
NH	10.97 br s				

Table 2-10-53: Cos, MFs, and TSs of aporphine-type isoquinoline alkaloids **2-10-141** and **2-10-142**.

No.	Compounds	MFs	Test solvents	References
2-10-141	griffithdione	$C_{19}H_{15}NO_4$	$CDCl_3$	[283]
2-10-142	aristoliukine B	$C_{17}H_{12}NO_5$	DMSO-d_6	[284]



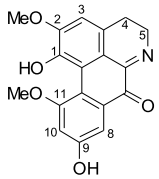
2-10-141 2-10-142

Table 2-10-54: ^{1}H NMR spectroscopic data of aporphine-type isoquinoline alkaloids **2-10-141** and **2-10-142**.

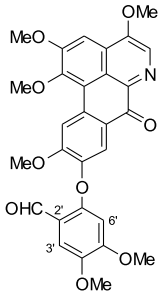
H	2-10-141	2-10-142	H	2-10-141	2-10-142
3	8.29 s	8.06 s	10	7.68 t(7.0)	
7	2.73 s(Me)	7.41 s	11	9.64 d(7.0)	8.89 d(2.0)
8	8.12 d(7.0)	7.76 d(8.8)	1-OMe	4.09 s	4.08 s
9	7.73 t(7.0)	7.15 dd(8.8, 2.0)	2-OMe	4.13 s	
NH	11.22 s	11.93 s	OH		10.62 s,9.94 s

Table 2-10-55: Cos, MFs, and TSs of aporphine-type isoquinoline alkaloids **2-10-143**~**2-10-153**.

No.	Compounds	MFs	Test solvents	References
2-10-143	1,9-dihydroxy-2,11-dimethoxy-4,5-dihydro-7-oxoaporphine	$C_{18}H_{15}NO_5$	DMSO-d_6	[285]
2-10-144	4-methoxyoxohernandaline	$C_{29}H_{25}NO_9$	$CDCl_3$	[280]
2-10-145	teliglazine	$C_{19}H_{15}NO_4$	$CDCl_3$	[286]
2-10-146	artabonatine C	$C_{19}H_{15}NO_4$	$CDCl_3$	[277]
1-20-147	artabonatine D	$C_{18}H_{13}NO_4$	$CDCl_3$	[277]
2-10-148	oxophoebine	$C_{20}H_{15}NO_6$	$CDCl_3$	[287]
2-10-149	oxoglaucine	$C_{20}H_{17}NO_5$	$CDCl_3$	[287]
2-10-150	*O*-methyl-moschatoline	$C_{19}H_{15}NO_4$	$CDCl_3$	[287]
2-10-151	lysicaumine	$C_{18}H_{13}NO_3$	$CDCl_3$	[287]
2-10-152	lauterine	$C_{18}H_{11}NO_4$	CF_3COOD	[287]
2-10-153	10-hydroxyliriodenine	$C_{17}H_9NO_4$	CF_3COOD	[287]



2-10-143



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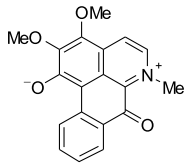
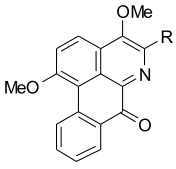
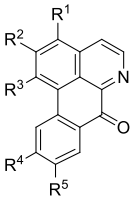


2-10-145



2-10-146 R = OMe
2-10-147 R = OH



	R^1	R^2	R^3	R^4	R^5
2-10-148	OMe	OMe	OMe	OCH_2O	
2-10-149	H	OMe	OMe	OMe	OMe
2-10-150	OMe	OMe	OMe	H	H
2-10-151	H	OMe	OMe	H	H
2-10-152	H	OCH_2O		OMe	H
2-10-153	H	OCH_2O		OH	H

Table 2-10-56: ^{1}H NMR spectroscopic data of aporphine-type isoquinoline alkaloids **2-10-143**~**2-10-147**.

H	2-10-143	2-10-144	2-10-145	2-10-146	2-10-147
2	3.86 s(OMe)	3.85 s(OMe)	4.34 s(OMe)	7.36 d(7.2)	7.42 d(7.0)
3	7.07 s	6.58 s	4.11 s(OMe)	7.55 d(7.2)	7.56 d(7.0)
4	3.07 t (7.0)	4.16 s(OMe)	8.14 br d(4.6)	4.02 s(OMe)	4.02 s(OMe)
5	3.55 t (7.0)	8.32 s	7.88 br d(4.6)	4.36 s(OMe)	4.36 s(OMe)
8	7.23 s	7.16 s	8.48 dd(1.5, 8.3)	8.24 dd(8.4, 1.7)	8.31 dd(8.0, 1.6)
9			7.42 ddd(1.5, 6.9, 8.3)	7.45 td(8.4, 1.1)	7.40 td(8.0, 1.6)
10	6.07 s	4.13 s(OMe)	7.79 ddd(1.5, 6.9, 8.3)	7.71 td(8.4, 1.7)	7.70 td(8.0, 1.6)
11	3.94 s(OMe)	9.30 s	10.03 dm(8.3)	8.97 dd(8.4, 1.1)	8.31 dd(8.0, 1.6)
2′		10.31 s(CHO)			
3′		7.48 s			
6′		7.32 s			
NMe			4.87 s		
OMe		4.16 s(1- OMe)		4.31 s(1- OMe)	4.31 s(1- OMe)
		3.99 s(4′-OMe)			
		3.82 s(5′-OMe)			

Table 2-10-57: ^{1}H NMR spectroscopic data of aporphine-type isoquinoline alkaloids **2-10-148**~**2-10-151**.

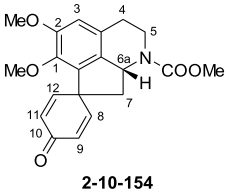
H	2-10-148	2-10-149	2-10-150	2-10-151
3		7.21 s		7.23 s
4	8.19 d(5.0)	7.79 d(5.3)	8.22 d(5.4)	7.81 d(5.1)
5	8.96 d(5.0)	8.92 d(5.3)	8.97 d(5.4)	8.91 d(5.1)
8	7.99 s	8.03 s	8.58 dd(7.9, 1.6)	8.59 dd(7.8, 1.5)
9			7.56 dt(7.9, 0.7)	7.61 dt(7.8, 0.9)
10			7.74 dt(7.9, 1.6)	7.78 dt(7.8, 1.5)
11	8.61 s	8.83 s	9.11 dd(7.9, 0.7)	9.18 dd(7.8, 0.9)
OMe	4.18 s, 4.09 s	4.03 s, 4.08 s	4.17 s, 4.11 s	4.11 s,4.02 s
	4.07 s	4.08 s, 4.11 s	4.08 s	
OCH_2O	6.14 s			

Table 2-10-58: ^{1}H NMR spectroscopic data of aporphine-type isoquinoline alkaloids **2-10-152** and **2-10-153**.

H	2-10-152	2-10-153	H	2-10-152	2-10-153
3	7.63 s	7.61 s	9	7.45 dd(8.5, 2.5)	7.45 dd(8.0, 3.0)
4	8.47 d(6.5)	8.45 d(6.0)	11	8.10 d(2.5)	8.08 d(3.0)
5	8.78 d(6.5)	8.79 d(6.0)	OMe	4.20 s	
8	8.65 d(8.5)	8.60 d(8.0)	OCH_2O	6.70 s	6.69 s

Table 2-10-59: Cos, MFs, and TSs of aporphine-type isoquinoline alkaloids **2-10-154**~**2-10-158**.

No.	Compounds	MFs	Test solvents	References
2-10-154	promucosine	$C_{20}H_{21}NO_5$	$CDCl_3$	[275]
2-10-155	isocryprochine	$C_{19}H_{25}NO_3$	$CDCl_3$	[288]
2-10-156	prooxocryptochine	$C_{17}H_{15}NO_4$	$CDCl_3$	[288]
2-10-157	isoamuronine	$C_{19}H_{23}NO_3$	$CDCl_3$	[288]
2-10-158	(+)-8,9-dihydrostepharine	$C_{18}H_{21}NO_3$	$CDCl_3$	[288]



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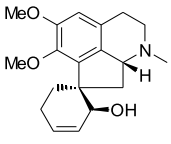

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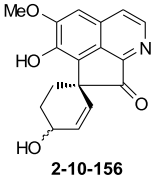
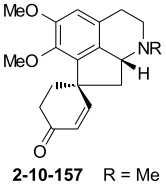


2-10-156



2-10-157 R = Me
2-10-158 R = H

Table 2-10-60: ^{1}H NMR spectroscopic data of aporphine-type isoquinoline alkaloids **2-10-154**~**2-10-158**.

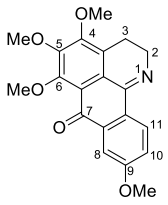
H	2-10-154	2-10-155	2-10-156	2-10-157	2-10-158
3	6.71 s	6.58 s	7.15 s	6.61 s	6.68 s
4	–	2.95 m 2.77 dd(17.2, 5.6)	7.67 d(5.6)	2.76 dd(12.5, 7)	2.77 br s
5	4.38 m	3.08 dd(11.6, 6.8) 2.45 ddd (11.6, 11.6, 5.6)	8.77 d(5.6)	3.07 m, 3.72 m	3.07 m, 3.62 m
6a	4.86 m	3.39 t(8)		3.72 m	4.14 m
7	–	2.59 dd(7.6, 4.8) 1.63 dd(19.2, 12)		2.09 m,2.52 m	2.20 m, 2.66 m
8	6.85 dd(10.0, 2.8)	4.71 s	5.49 dd(10, 3.2)	6.92 d(10.1)	6.76 d(10.4)
9	6.40 dd(10.0, 2.0)	5.66 d(8)	6.25 dd(10, 3.2)	5.97 d(10.1)	6.01 d(10.4)
10		5.83 d(8)	4.44 m		
11	6.31 dd(10.0, 2.0)	2.09 m	2.29 m	3.31 m, 2.52 m	2.85 m, 2.74 m
12	7.02 dd(10.0, 2.8)	2.63 dd(10.8, 6.8) 1.57 dd(8.8, 4.8)	2.06 m,2.29 m	2.09 m, 3.07 m	2.03 m, 3.07 m

Table 2-10-60 (continued)

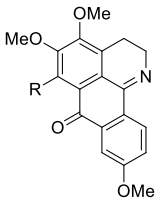
H	2-10-154	2-10-155	2-10-156	2-10-157	2-10-158
1-OMe	3.63 s	3.82 s	3.72 s	3.71 s	3.77 s
2-OMe	3.84 s	3.80 s	4.02 s	3.85 s	3.85 s
COOMe	3.77 s				
NMe		2.36 s		2.37 s	

Table 2-10-61: Cos, MFs, and TSs of aporphine-type isoquinoline alkaloids **2-10-159**~**2-10-165**.

No.	Compounds	MFs	Test solvents	References
2-10-159	2,3-dihydrodauriporphine	$C_{20}H_{19}NO_5$	DMSO-d_6	[289]
2-10-160	tyraminoporphine	$C_{27}H_{24}N_2O_5$	DMSO-d_6	[289]
2-10-161	dauriporphine	$C_{20}H_{17}NO_5$	$CDCl_3$	[289]
2-10-162	daurioxoisoporphine A	$C_{26}H_{22}N_2O_4$	$CDCl_3$	[290]
2-10-163	daurioxoisoporphine B	$C_{19}H_{16}N_2O_4$	$CDCl_3$	[290]
2-10-164	daurioxoisoporphine C	$C_{19}H_{16}N_2O_3$	$CDCl_3$	[290]
2-10-165	daurioxoisoporphine D	$C_{18}H_{13}NO_4$	$CDCl_3$	[290]

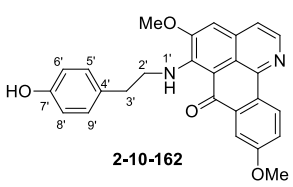


2-10-159

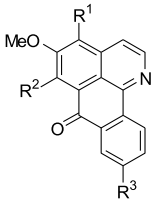


2-10-160 R = NHCH$_2$CH$_2$-(4-hydroxyphenyl)

2-10-161 R = OMe



2-10-162



	R^1	R^2	R^3
2-10-163	OMe	NH_2	OMe
2-10-164	H	NHMe	OMe
2-10-165	H	OMe	OH

Table 2-10-62: ^{1}H NMR spectroscopic data of aporphine-type isoquinoline alkaloids **2-10-159**~**2-10-162**.

H	2-10-159	2-10-160	2-10-161	2-10-162
2	3.96 t(7.9)	8.66 d(4.9)	8.68 d(5.6)	8.86 d(5.1)
3	2.77 t(7.9)	7.88 d(4.9)	7.94 d(5.6)	7.63 d(5.1)
4	3.92 s(OMe)	4.12 s(OMe)	4.27 s(OMe)	7.33 s
8	7.52 d(2.7)	7.89 d(2.7)	7.87 d(2.7)	8.44 d(2.7)
10	7.30 dd(2.7, 8.8)	7.44 dd(2.7, 8.8)	7.32 dd(2.7, 8.8)	7.56 dd(2.7, 8.8)
11	8.19 d(8.8)	8.88 d(8.8)	8.80 d(8.8)	9.43 d(8.8)

Table 2-10-62 (continued)

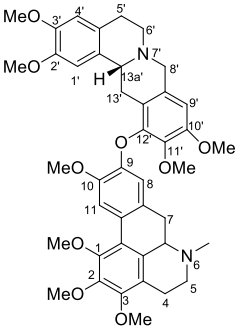
H	2-10-159	2-10-160	2-10-161	2-10-162
5-OMe	3.91 s	3.87 s	4.05 s	3.82 s
6-OMe	3.84 s		4.17 s	
9-OMe	3.89 s	3.96 s	3.98 s	3.84 s
1′		12.51 br s		13.30 br
2′		4.08 m		4.25 dt(7.2, 12.7)
3′		2.94 t(7.3)		3.10 t(7.2)
5′		7.14 d(8.7)		7.42 d(8.4)
6′		6.69 d(8.7)		7.21 d(8.4)
8′		6.69 d(8.7)		7.21 d(8.4)
9′		7.14 d(8.7)		7.42 d(8.4)

Table 2-10-63: ^{1}H NMR spectroscopic data of aporphine-type isoquinoline alkaloids **2-10-163**~**2-10-165**.

H	2-10-163	2-10-164	2-10-165	H	2-10-163	2-10-164	2-10-165
2	8.90 d(5.2)	8.11 d(5.1)	8.82 d(5.5)	11	9.39 d(8.8)	8.91 d(8.8)	9.21 d(8.8)
3	7.59 d(5.2)	7.39 d(5.1)	7.67 d(5.5)	1′	6.35 br 10.35 br	12.50 br	
4	4.08 s(OMe)	7.02 s	7.57 s	OMe	3.90 s(5-OMe) 3.83 s(9-OMe)	3.95 s(5-OMe) 3.95 s(9-OMe)	3.90 s(5-OMe) 4.20 s(6-OMe)
8	8.42 d(2.7)	7.32 d(2.7)	8.34 d(2.6)	NMe		3.52 s	
10	7.54 dd (2.7, 8.8)	7.96 dd (2.7, 8.8)	7.64 dd (2.6, 8.8)				

Table 2-10-64: Co, MF, and TS of aporphine-type isoquinoline alkaloid **2-10-166**.

No.	Compound	MF	Test solvent	Reference
2-10-166	(–)-thalibealine	$C_{42}H_{48}N_2O_9$	$CDCl_3$	[291]



2-10-166

Table 2-10-65: ^{1}H NMR spectroscopic data of aporphine-type isoquinoline alkaloid **2-10-166**.

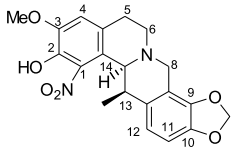
H	2-10-166	H	2-10-166	H	2-10-166
4	2.80, 2.88	3-OMe	3.87	9′	6.59
5	2.39, 3.07	6-NMe	2.48	13′	2.48, 3.51
6a	2.93	10-OMe	4.01	13a’	3.55
7	2.40, 2.78	1′	6.74	2′-OMe	3.84
8	6.40	4′	6.58	3′-OMe	3.83
11	8.02	5′	2.66, 3.13	10′-OMe	3.88
1-OMe	3.74	6′	2.64, 3.18	11′-OMe	3.80
2-OMe	3.94	8′	3.73, 4.04		

2.10.5 Protoberberine-type, dihydroberberine-type, berberine-type isoquinoline alkaloids

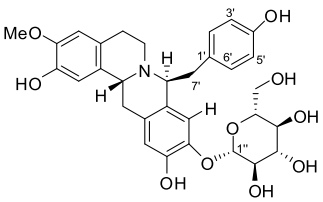
Table 2-10-66: Cos, MFs, and TSs of protoberberine-type isoquinoline alkaloids **2-10-167**~**2-10-183**.

No.	Compounds	MFs	Test solvents	References
2-10-167	1-nitro-apocavidine	$C_{20}H_{20}N_2O_6$	$CDCl_3$	[292]
2-10-168	(–)-8-benzylberbine A	$C_{31}H_{35}NO_{10}$	CD_3CN-D_2O(7:3)	[293]
2-10-169	(–)-*N*-oxide-8-benzylberbine B	$C_{31}H_{35}NO_{11}$	CD_3CN-D_2O(7:3)	[293]
2-10-170	tetrahydroberberrubine	$C_{19}H_{19}NO_4$	DMSO-d_6	[294]
2-10-171	escholidine perchlorate	$C_{20}H_{21}ClNO_8$	DMSO-d_6	[294]
2-10-172	(±)-8-(*p*-hydroxybenzyl)-2,3,10,11-tetrahydroxyprotoberberine pentaacetate	$C_{34}H_{33}NO_{10}$	CD_3COCD_3	[235]
2-10-173	(–)-tetrahydropalmatine	$C_{21}H_{25}NO_4$	$CDCl_3$	[229]
2-10-174	(–)-corydalmine	$C_{20}H_{23}NO_4$	$CDCl_3$	[229]
2-10-175	(–)-8-oxocanadine	$C_{20}H_{19}NO_5$	$CDCl_3$	[295]
2-10-176	(–)-8-oxotetrahydrothalifendine	$C_{19}H_{17}NO_5$	$CDCl_3$	[295]
2-10-177	(–)-oxoisocorypalmine	$C_{20}H_{21}NO_5$	$CDCl_3$	[295]
2-10-178	(–)-8-oxo-3-hydroxy-2,4,9,10-tetra-methoxyberbine①	$C_{21}H_{23}NO_6$	$CDCl_3$	[295]
2-10-179	gusanlung D	$C_{18}H_{15}NO_3$	$CDCl_3$	[296]
2-10-180	stecepharine	$C_{21}H_{26}NO_5$	CD_3OD	[243]
2-10-181	9-*O*-methylstecepharine	$C_{22}H_{28}NO_5$	CD_3OD	[243]
2-10-182	(–)-coulteroberbinone	$C_{21}H_{20}NO_6$	$CDCl_3$-CD_3OD	[297]
2-10-183	(–)-8-oxopolyalthiaine	$C_{19}H_{19}NO_6$	C_5D_5N	[298]

① The substitution sites of OMe and OH at 3-and 4-positions were not assigned in the original literature. In Table 2-10-70, the data of **2-10-178** may be corresponding to the isomer.



2-10-167



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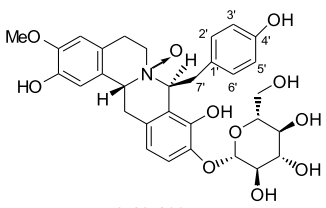


2-10-169

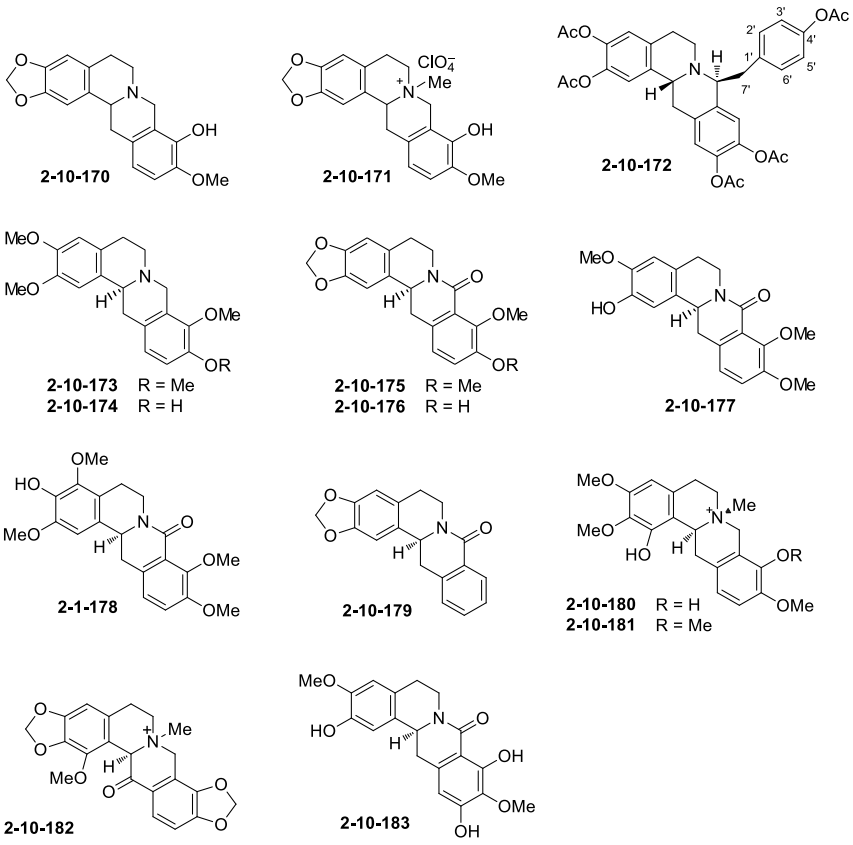


Table 2-10-67: ^{1}H NMR spectroscopic data of protoberberine-type isoquinoline alkaloids **2-10-167**~**2-10-169** and **2-10-172**.

H	2-10-167	2-10-168	2-10-169	2-10-172
1		6.71 s	6.75 s	7.16 s
4	6.74 s	6.75 s	6.80 s	6.98 s
5	*α* 3.12 m	ax 3.02 ddd (18.0, 11.7, 5.0)	ax 2.77 dd(18.0, 5.0)	2.90 m
	β 2.62 m	eq 3.4~3.5 m	eq 3.38 m	2.77 m
6	*α* 2.67 m	ax 3.27 m	ax 3.70 m	3.12 m
	β 3.09 m	eq 3.4~3.5 m	eq 3.43~3.65 m	2.77 m
8	*α* 3.63 d(15)	4.37 dd(10.1, 3.6)	5.20 dd(8.7, 4.0)	4.12 dd(8.5, 5.5)
	β 4.03 d(15)			
9		6.25 s		6.82 s
11	6.67 d(8)		7.20 d(7.8)	
12	6.57 d(8)	6.69 s	6.83 d(7.8)	7.00 s
13	2.83 qd(7, 3)	ax 2.80 dd(17.8, 11.4)	ax 3.07 dd(16.5, 12.0)	3.02dd(17.1, 5.0)
		eq 3.30 dd(17.8, 5.5)	eq 3.43~3.65 m	2.90 m

Table 2-10-67 (continued)

H	2-10-167	2-10-168	2-10-169	2-10-172
14	4.29 d(3)	4.68 dd(11.4, 5.5)	4.94 dd(12.0, 4.0)	4.58 dd(11.5, 5.0)
13-Me	0.89 d(7)			
3-OMe	3.92 s	3.81 s	3.85 s	
OCH_2O	5.94 d(2), 5.91 d(2)			
Ac				2.03~2.08 s
2′, 6′		6.95 d(8.3)	7.08 d(7.8)	7.29 d(8.0)
3′, 5′		6.74 d(8.3)	6.77 d(7.8)	6.98 d(8.0)
7′		2.92 dd(13.7, 10.1)	3.36 dd(16.0, 4.0)	*α* 3.28 dd(14.0, 8.5)
		3.32 dd(13.7,3.6)	3.32 dd(16.0, 8.7)	*β* 2.95 dd(14.0, 5.5)
1″		4.38 d(7.4)	4.85 d(7.5)	
2″-5″		3.3~3.5 m	3.43~3.65 m	
6″		3.71 dd(12.5, 5.5)	3.75 dd(12.0, 5.2)	
		3.75 dd(12.5, 5.5)	3.88 dd(12.5, 2.5)	

Table 2-10-68: ^{1}H NMR spectroscopic data of protoberberine-type isoquinoline alkaloids **2-10-170**~**2-10-171**.

H	2-10-170	2-10-171	H	2-10-170	2-10-171
1	6.90	6.93	12	6.57	6.69
4	6.66	6.87	13	2.53, 3.27	3.10, 3.35
5	2.60, 2.91	3.17	14	3.37	4.74
6	2.43, 3.07	3.61, 3.70	10-OMe	3.76	3.80
8	3.27, 4.01	4.62, 4.74	OCH_2O	5.94	6.01, 6.03
11	6.78	6.98	NMe		3.20
			OH	8.57	

Table 2-10-69: ^{1}H NMR spectroscopic data of protoberberine-type isoquinoline alkaloids **2-10-173**~**2-10-175**.

H	2-10-173	2-10-174	2-10-175
1	6.7 s	6.69 s	6.66 s
4	6.58 s	6.59 s	6.67 s
5ax	3.10 m	3.15 m	2.85
5eq	2.65 m	2.70 d	2.85
6ax	2.59 m	2.69 m	2.85
6eq	3.16 m	3.21 m	4.97 m
8ax	3.49 d(15)	3.59 d(15.4)	
8eq	4.20 d(5.5)	4.23 d(15.4)	
11	6.74 d(8.3)	6.78 d(8.2)	7.01 d(8.3)
12	6.83 d(8.4)	6.70 d(8.2)	6.93 d(8.3)

Table 2-10-69 (continued)

H	2-10-173	2-10-174	2-10-175
13ax	2.79 dd(11.4, 15.6)	2.82 dd(15.3, 15.0)	3.02 dd(15.3, 3.3)
13eq	3.23 dd(3.7, 15.6)	3.23 d(11.8)	2.85
14	3.49 dd	3.64 m	4.69 dd(12.8, 3.3)
OMe	3.84 s(2-OMe)	3.85 s(2-OMe)	4.01 s(9-OMe)
	3.81 s(3-OMe)	3.83 s(3-OMe)	3.89 s(10-OMe)
	3.82 s(9-OMe)	3.76 s(9-OMe)	
	3.80 s(10-OMe)		
OCH_2O			5.95 s

Table 2-10-70: ^{1}H NMR spectroscopic data of protoberberine-type isoquinoline alkaloids **2-10-176**~**2-10-178**.

H	2-10-176	2-10-177	2-10-178
1	6.67 s	6.77 s	6.31 s
4	6.68 s	6.67 s	
5ax	2.89 ddd(15, 11, 3)	2.88 br dd(15.5, 11, 3)	2.63 ddd(13, 12.5, 3)
5eq	2.75 ddd(15, 3, 2)	2.75 ddd(15.5, 3, 2)	3.02 ddd(16, 3, 7.5)
6ax	2.95 ddd(12.5, 11, 2.5)	2.95 ddd(12.5, 12, 3)	2.83 ddd(13, 12.5, 3)
6eq	4.97 m	4.97 m	5.07 ddd(13, 5, 2.5)
11	7.07 d(8)	7.00 d(8)	7.00 d(8.5)
12	6.90 br d(8)	6.93 dd(8, 1)	6.94 d(8.5)
13	3.02 dd(15.5, 3)	3.03 dd(15.5, 3)	3.01 dd(15.5, 3)
	2.84 br dd(15.5, 13.5)	2.81 ddd(15.5, 13.5, 1)	2.85 br dd(16, 13.5)
14	4.73 dd(13.5, 3)	4.69 dd(13, 3)	4.71 dd(13, 3)
OMe	4.01 s(9-OMe)	3.90 s(3-OMe)	3.88 s
		4.01 s(9-OMe)	3.91 s
		3.88 s(10-OMe)	4.02 s
			3.89 s
OCH_2O	5.96 s		
OH	6.03 br	5.55 br s	5.91 s

Table 2-10-71: ^{1}H NMR spectroscopic data of protoberberine-type isoquinoline alkaloids **2-10-179**~**2-10-183**.

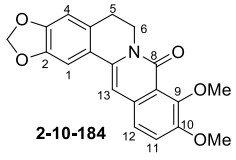
H	2-10-179	2-10-180	2-10-181	2-10-182	2-10-183
1	7.35 s			3.94 s(OMe)	7.12 s
4	6.80 s	6.50 s	6.50 s	6.29 s	6.78 s
5	2.70~3.40 m	3.20 dd(18.5, 7.0)	3.20 dd(18.5, 7.0)	3.20 m	α 2.64 ddd(13.0, 2.0, 3.0)
		3.30 dt(18.5, 8.0)	3.33 dt(18.5, 8.0)	2.84 dd(18.3, 6.3)	β 2.86 ddd(13.0, 11.0, 2.6)

Table 2-10-71 (continued)

H	2-10-179	2-10-180	2-10-181	2-10-182	2-10-183
6	4.80 m	3.48 m	3.76 m	3.8 m	*α* 2.90 ddd(12.5, 11.0, 2.0)
		3.79 m	3.50 m	3.5 m	*β* 4.98 ddd(12.5, 3.0, 2.6)
8		4.72 d(16.0)	4.82 d(16.0)	5.26 d(15.7)	
		4.92 d(16.0)	4.93 d(16.0)	5.15 d(15.7)	
9	8.07 d(8.0)		3.89 s(OMe)		13.97 s(OH)
10	7.29~7.41 m	3.86 s(OMe)	3.86 s(OMe)		3.99 s(OMe)
11	7.29~7.41 m	6.95 d(8.5)	7.06 d(8.5)	6.86 d(8.3)	
12	7.29~7.41 m	6.68 d(8.5)	6.94 d(8.5)	7.60 d(8.3)	6.62 s
13	2.70~3.40 m	2.95 dd(18.5, 11.5)	2.97 dd(18.0, 11.5)		*α* 3.25 dd(15.6, 4.0)
		3.48 m	3.52 dd(18.0, 6.0)		*β* 2.81 dd(15.6, 13.6)
14	3.95 m	4.97 ddd (11.5, 5.5, 1.5)	4.98 ddd (11.5, 6.0, 1.5)	5.64 s	4.77 dd(13.6, 4.0)
OMe		3.80 s(2-OMe)	3.80 s(2-OMe)		3.78 s(3-OMe)
		3.85 s(3-OMe)	3.85 s(3-OMe)		
2,3-OCH_2O	6.20 s, 6.06 s			5.88 d(1.2), 5.87 d(1.2)	
9,10-OCH_2O				6.11 d(1.2), 6.09 d(1.2)	
NMe		3.25 s	3.26 s	3.36 s	

Table 2-10-72: Cos, MFs, and TSs of dihydroberberine-type isoquinoline alkaloids **2-10-184**~**2-10-191**.

No.	Compounds	MFs	Test solvents	References
2-10-184	oxyberberine	$C_{20}H_{17}NO_5$	$CDCl_3$	[295]
2-10-185	oxypalmatine	$C_{21}H_{21}NO_5$	$CDCl_3$	[295]
2-10-186	yuanamide	$C_{22}H_{23}NO_5$	$CDCl_3$	[299]
2-10-187	8-oxythalifendine	$C_{19}H_{15}NO_5$	$CDCl_3$	[296]
2-10-188	acetyl-8-oxythalifendine	$C_{21}H_{17}NO_6$	$CDCl_3$	[296]
2-10-189	8-oxyberberrubine	$C_{19}H_{15}NO_5$	$CDCl_3$	[296]
2-10-190	8-oxocoptisine	$C_{19}H_{13}NO_5$	$CDCl_3$	[300]
2-10-191	7,8-dihydro-8-hydroxypalmatine	$C_{21}H_{23}NO_5$	$CDCl_3$	[301]



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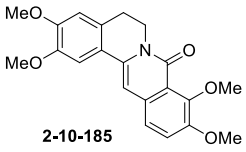

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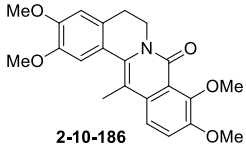

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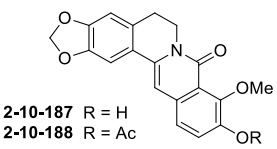
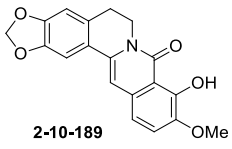
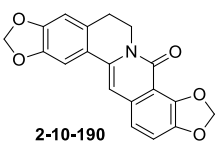






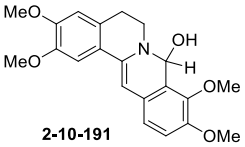


Table 2-10-73: ^{1}H NMR spectroscopic data of dihydroberberine-type isoquinoline alkaloids **2-10-184**~**2-10-187**.

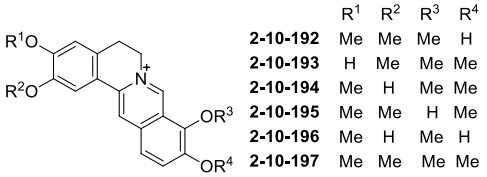
H	2-10-184	2-10-185	2-10-186	2-10-187
1	6.70 s	6.77 s	7.08 s	6.74 s
4	6.68 s	6.73 s	6.79 s	6.72 s
5	2.87 t(6)	2.93 t(6.3)	2.86 t(5.7)	2.90 t(7.2)
6	4.28 t(6)	4.32 t(6.3)	4.23 t(5.7)	4.30 t(7.2)
11	7.31 d(8.7)	7.35 d(8.5)	7.51 d(8.7)	7.33 ABq(7.0)
12	7.25 d(8.7)	7.30 d(8.5)	7.39 d(8.7)	7.24 ABq(7.0)
13	7.19 s	7.23 s		7.20 s
OMe	4.01 s, 4.02 s	4.02 s(9-OMe)	3.91 s, 3.96 s	4.04 s(9-OMe)
		3.96 s, 3.99 s, 3.94 s	3.97 s, 4.02 s	
13-Me			2.57 s	
10-OAc				2.37 s
OCH_2O	5.99 s			6.00 s

Table 2-10-74: ^{1}H NMR spectroscopic data of dihydroberberine-type isoquinoline alkaloids **2-10-188**~**2-10-191**.

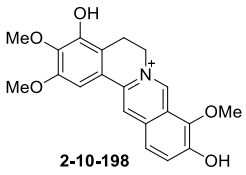
H	2-10-188	2-10-189	2-10-190	2-10-191
1	6.78 s	6.83 s	7.22 s	7.20 s
4	6.74 s	6.72 s	6.73 s	6.65 s
5	2.90 t(7.2)	2.91 t(7.2)	2.89 t(4.8)	2.76 dt(4.4, 15.5)
				3.42 ddd(4.4, 9.9, 15.5)
6	4.30 t(7.2)	4.27 t(7.2)	4.28 t(4.8)	3.57 ddd(3.8, 9.9, 13.7), 3.87 (ov)
8				5.65 s
11	7.32 ABq(7.0)	7.30 ABq(8.0)	7.03 d(8.3)	7.00 d(8.4)
12	7.26 ABq(7.0)	7.00 ABq(8.0)	7.16 d(8.3)	6.90 d(8.4)
13	7.27 s	7.21 s	6.69 s	–
OMe	3.96 s(9-OMe)	3.96 s(10-OMe)		3.95 s(2-OMe), 3.90 s(3-OMe)
				3.96 s(9-OMe), 3.89 s(10-OMe)
OCH_2O	6.10 s	6.02 s	6.21 s, 5.99 s	

Table 2-10-75: Cos, MFs, and TSs of berberine-type isoquinoline alkaloids **2-10-192**~**2-10-203**.

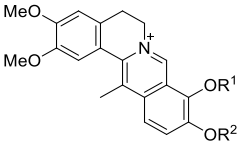
No.	Compounds	MFs	Test solvents	References
2-10-192	dehydrocorydalmine	$C_{20}H_{20}NO_4$	CD_3OD	[302]
2-10-193	jatrorrhizine	$C_{20}H_{20}NO_4$	DMSO-d_6	[302]
2-10-194	columbamine	$C_{20}H_{20}NO_4$	DMSO-d_6	[302]
2-10-195	palmatrubine	$C_{20}H_{20}NO_4$	DMSO-d_6	[302]
2-10-196	stephanine	$C_{19}H_{18}NO_4$	DMSO-d_6	[302]
2-10-197	gindarinine	$C_{21}H_{21}NO_4$	DMSO-d_6	[302]
2-10-198	fissisaine	$C_{20}H_{20}NO_5$	DMSO-d_6	[302]
2-10-199	13-methyl-palmatrubine	$C_{21}H_{22}NO_4$	CD_3OD	[303]
2-10-200	13-methyl-dehydrocorydalmine	$C_{21}H_{22}NO_4$	CD_3OD	[303]
2-10-201	dehydrocorydaline	$C_{22}H_{24}NO_4$	$CDCl_3$	[303]
2-10-202	thalifendine cloride	$C_{19}H_{16}ClNO_4$	DMSO-d_6	[294]
2-10-203	2,10-dimethoxy-3,11-dihydroxy-5,6-dihydroprotoberberine	$C_{19}H_{17}NO_4$	DMSO-d_6-CF_3COOD	[285]



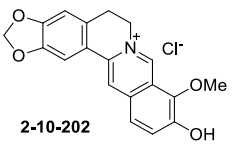
	R^1	R^2	R^3	R^4
2-10-192	Me	Me	Me	H
2-10-193	H	Me	Me	Me
2-10-194	Me	H	Me	Me
2-10-195	Me	Me	H	Me
2-10-196	Me	H	Me	H
2-10-197	Me	Me	Me	Me



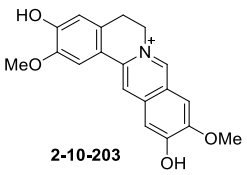
2-10-198



	R^1	R^2
2-10-199	H	Me
2-10-200	Me	H
2-10-201	Me	Me



2-10-202



2-10-203

Table 2-10-76: ^{1}H NMR spectroscopic data of berberine-type isoquinoline alkaloids **2-10-192**~**2-10-195**.

H	2-10-192	2-10-193	2-10-194	2-10-195
1	7.55 s	7.69 s	7.55 s	7.57 s
4	6.98 s	6.85 s	7.06 s	7.01 s
5	3.21 t(6.4)	3.15 t(6.1)	3.18 t(5.7)	3.15 t(5.6)
6	4.81 t(6.4)	4.92 t(6.1)	4.93 t(5.7)	4.82 t(5.6)
8	9.26 s	9.85 s	9.86 s	9.30 s
11	7.59 d(9.0)	8.19 d(9.1)	8.19 dd(9.2, 2.5)	7.65 d(8.8)
12	7.72 d(9.0)	8.01 d(9.1)	8.06 dd(9.2, 2.5)	7.56 d(8.8)
13	8.49 s	8.98 s	8.81 br s	8.63 s
OMe	3.96 s(2-OMe)	3.94 s(2-OMe)	3.89 s(3-OMe)	3.85 s(2-OMe)
	3.91 s(3-OMe)	4.06 s(9-OMe)	4.06 s(9-OMe)	3.89 s(3-OMe)
	4.04 s(9-OMe)	4.09 s(10-OMe)	4.09 s(10-OMe)	4.03 s(10-OMe)

Table 2-10-77: ^{1}H NMR spectroscopic data of berberine-type isoquinoline alkaloids **2-10-196**~**2-10-199**.

H	2-10-196	2-10-197	2-10-198	2-10-199
1	7.52 s	7.68 s	7.33 s	7.28 s
4	7.04 s	7.07 s		7.03 s
5	3.17 t(5.7)	3.22 t(6.2)	3.11 t(6.3)	3.08 t
6	4.91 t(5.8)	4.99 t(6.2)	4.88 t(6.3)	4.53 t
8	9.67 s	9.84 s	9.17 s	9.35 s
11	7.82 d(9.0)	8.18 d(9.2)	7.87 d(2.5)	7.58 d(8.5)
12	7.86 d(9.0)	8.00 d(9.2)	7.87 d(2.5)	7.02 d(8.5)
13	8.67 s	8.96 s	8.94 s	–
OMe	3.88 s(3-OMe)	3.36 s(2-OMe)	3.97 s(2-OMe)	3.89 s(2-OMe)
	4.07 s(9-OMe)	3.92 s(3-OMe)	3.77 s(3-OMe)	3.91 s(3-OMe)
		4.10 s(9-OMe)	4.06 s(9-OMe)	3.89 s(10-OMe)
		4.06 s(10-OMe)		
13-Me				2.78 s

Table 2-10-78: ^{1}H NMR spectroscopic data of berberine-type isoquinoline alkaloids **2-10-200**~**2-10-203**.

H	2-10-200	2-10-201	2-10-202	2-10-203
1	7.38 s	7.16 s	7.76	7.60 s
4	7.12 s	6.93 s	7.07	6.81 s
5	3.19 t	3.25 t	3.19	3.09 t(5.7)
6	4.83 t	5.30 br s	4.89	4.68 t(5.7)
8	9.68 s	10.68 s	9.69	9.37 s
9				7.65 s
11	8.05 d(8.5)	7.87 d(9.1)	7.84	
12	7.84 d(8.5)	7.92 d(9.1)	7.84	7.40 s
13			8.83	8.62 s
OMe	3.91 s(2-OMe)	3.94 s(2-OMe)	4.06 s(9-OMe)	3.91 s(2-OMe)
	3.95 s(3-OMe)	4.00 s(3-OMe)		3.97 s(10-OMe)
	4.16 s(9-OMe)	4.08 s(9-OMe)		
		4.35 s(10-OMe)		
13-Me	3.01 s	2.97 s		
OCH_2O			6.16	

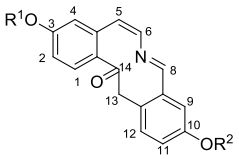
2.10.6 Protopine-type isoquinoline alkaloids

Table 2-10-79: Cos, MFs, and TSs of protopine-type isoquinoline alkaloids **2-10-204**~**2-10-210**.

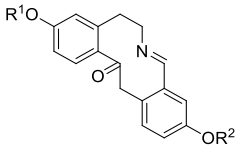
No.	Compounds	MFs	Test solvents	References
2-10-204	3,5-di-*O*-methylconstrictosine	$C_{19}H_{17}NO_3$	CD_3OD	[304]
2-10-205	constrictosine	$C_{17}H_{13}NO_3$	CD_3OD	[304]

Table 2-10-79 (continued)

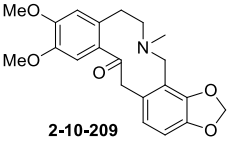
No.	Compounds	MFs	Test solvents	References
2-10-206	3-*O*-methylconstrictosine	$C_{18}H_{15}NO_3$	CD_3OD	[304]
2-10-207	5,6-dihydro-3,5-di-*O*-methylconstrictosine	$C_{19}H_{19}NO_3$	CD_3OD	[304]
2-10-208	5,6-dihydroconstrictosine	$C_{17}H_{15}NO_3$	CD_3OD	[304]
2-10-209	cryptopine	$C_{21}H_{23}NO_5$	$CDCl_3$	[300]
2-10-210	hunnemanine	$C_{20}H_{21}NO_5$	CD_3OD	[305]



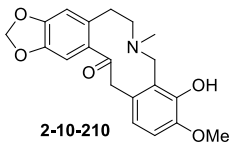
	R^1	R^2
2-10-204	Me	Me
2-10-205	H	H
2-10-206	Me	H



	R^1	R^2
2-10-207	Me	Me
2-10-208	H	H



2-10-209



2-10-210

Table 2-10-80: ^{1}H NMR spectroscopic data of protopine-type isoquinoline alkaloids **2-10-204**~**2-10-207**.

H	2-10-204	2-10-205	2-10-206	2-10-207
1	7.28 d(8.6)	7.25 d(8.6)	7.28 d(8.6)	7.22 d(8.7)
2	6.90 dd(8.6, 2.5)	6.67 dd(8.6, 2.4)	6.90 dd(8.6, 2.5)	6.88 dd(8.7, 2.5)
4	8.05 d(2.5)	7.93 d(2.4)	8.05 d(2.5)	7.91 d(2.5)
5	8.04 d(5.1)	8.06 d(5.2)	8.01 d(5.1)	2.86 t(8.4)
6	8.30 d(5.1)	8.80 d(5.2)	8.28 d(5.1)	3.95 t(8.4)
8	8.77 s	8.78 s	8.73 s	8.10 s
9	7.16 d(2.5)	7.45 d(2.4)	7.45 d(2.5)	7.12 d(2.5)
11	6.86 dd(2.5, 8.7)	7.44 dd(2.4, 8.8)	7.42 dd(2.5, 8.7)	6.83 dd(2.5, 8.7)
12	7.15 d(8.7)	7.48 d(8.8)	7.48 d(8.7)	7.15 d(8.7)
13	4.10 br s	4.10 br s	4.10 br s	4.08 br s
3-OMe	3.85 s		3.83 s	3.85 s
10-OMe	3.87 s			3.88 s

Table 2-10-81: ^{1}H NMR spectroscopic data of protopine-type isoquinoline alkaloids **2-10-208**~**2-10-210**.

H	2-10-208	2-10-209	2-10-210
1	7.21 d(8.8)	7.02 s	7.03 s
2	6.73 dd(8.8, 2.4)		

Table 2-10-81 (continued)

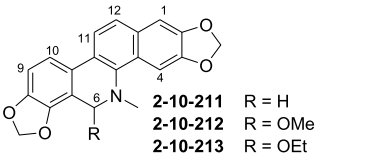
H	2-10-208	2-10-209	2-10-210
4	7.75 d(2.4)	6.71 s	6.70 s
5	2.86 t(8.4)	2.11 m	3.00~3.50 m
6	3.90 t(8.4)	2.83 m	3.00~3.50 m
8	8.06 s	3.09 m	2.79 br s
9	6.94 d(2.5)		–
11	6.75 dd(2.5, 8.7)	6.71 m	6.87 d(8)
12	7.17 d(8.7)	6.71 m	6.67 d(8)
13	4.08 br s	3.75 m	4.17 br s
OMe		3.92 s(2-OMe) 3.92 s(3-OMe)	3.85 s(10-OMe)
OCH_2O		5.95 s	5.95 s
NMe		2.21 s	2.50 s

2.10.7 Benzophenanthridine-type isoquinoline alkaloids

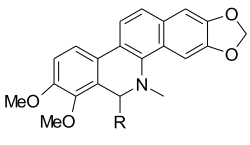
Table 2-10-82: Cos, MFs, and TSs of benzophenanthridine-type isoquinoline alkaloids **2-10-211**~**2-10-226**.

No.	Compounds	MFs	Test solvents	References
2-10-211	dihydrosanguinarine	$C_{20}H_{15}NO_4$	$CDCl_3$	[306]
2-10-212	8-methoxyldihydrosanguinarine[①]	$C_{21}H_{17}NO_5$	$CDCl_3$	[307]
2-10-213	ethoxydihydrosanguinarine	$C_{22}H_{19}NO_5$	$CDCl_3$	[308]
2-10-214	8-hydroxymethyldihydrosanguinarine	$C_{21}H_{17}NO_5$	$CDCl_3$	[308]
2-10-215	spallidamine	$C_{22}H_{17}NO_6$	$CDCl_3$	[299]
2-10-216	6-(2-hydroxyrthyl)-5,6-dihydrosanguinarine	$C_{22}H_{19}NO_5$	DMSO-d_6	[309]
2-10-217	6-acetonyl-5,6-dihydrosanguinarine	$C_{23}H_{19}NO_5$	$CDCl_3$	[309]
2-10-218	dihydrochelerythrine	$C_{21}H_{19}NO_4$	$CDCl_3$	[306]
2-10-219	*N*-methyl-2,3,7,8-tetramethoxy-5,6-dihydrobenzo-phenanthridine-6-ethanoic acid	$C_{24}H_{25}NO_6$	DMSO-d_6	[309]
2-10-220	*N*-methyl-2,3,7,8-tetramethoxy-6-oxo-5,6-dihydro-benzophenanthridine	$C_{22}H_{21}NO_5$	$CDCl_3$	[309]
2-10-221	(*R*)-8-[(*R*)-1-hydroxyethyl]dihydrochelerythrine	$C_{23}H_{23}NO_5$	CD_3OD	[310]
2-10-222	zanthomuurolanine	$C_{37}H_{45}NO_5$	C_6D_6	[311]
2-10-223	*epi*-zanthomuurolanine	$C_{37}H_{45}NO_5$	C_6D_6	[311]
2-10-224	zanthocadinanine A	$C_{37}H_{45}NO_5$	C_6D_6	[311]
2-10-225	zanthocadinanine B	$C_{37}H_{45}NO_5$	C_6D_6	[311]
2-10-226	*epi*-zanthocadinanine B	$C_{37}H_{45}NO_5$	C_6D_6	[311]

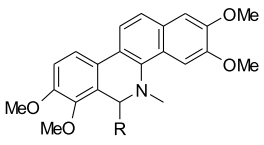
[①] In the literature, the numbering system of this compound is different from that of popular numbering system of this class of compounds. Compound **2-10-212** is 8-methoxyldihydrosanguinarine.



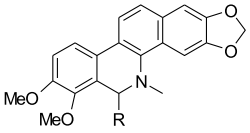
2-10-211 R = H
2-10-212 R = OMe
2-10-213 R = OEt
2-10-214 R = CH_2OH
2-10-215 R = CH_2COOH
2-10-216 R = β-CH_2CH_2OH
2-10-217 R = β-CH_2COMe



2-10-218 R = H
2-10-221 R = CHOHMe

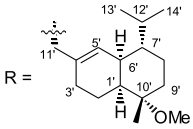


2-10-219 R = β-CH_2COOH
2-10-220 R = = O

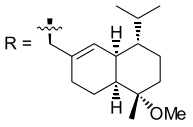


2-10-222~2-10-226

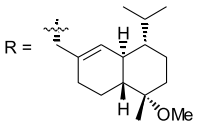
2-10-222 R =



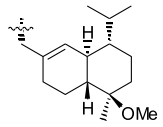
2-10-223 R =



2-10-224 R =



2-10-225 R=



2-10-226 R =

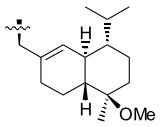


Table 2-10-83: ^{1}H NMR spectroscopic data of benzophenanthridine-type isoquinoline alkaloids **2-10-211**~**2-10-216**.

H	2-10-211	2-10-212	2-10-213	2-10-214	2-10-215	2-10-216
1	7.11 s	7.13 s	7.08 s	7.01 s	7.14 s	7.28 s
4	7.68 s	7.70 s	7.67 s	7.53 s	7.41 s	7.41 s
6	4.20 s	5.40 s	5.47 s	4.41 q	4.69 t(7.7)	4.60 dd(10.5, 3.5)
		3.46 s(OMe)				
9	6.85 d(8.1)	6.94 d(8)	6.95 d(9)	6.78 d(9)	6.91 d(8.4)	7.70 d(8.5)
10	7.30 d(8.1)	7.42 d	7.55 d(9)	7.38 d(9)	7.35 d(8.4)	7.84 d(8.5)
11	7.69 d(8.5)	7.77 d(9)	7.84 d(9)	7.58 d(8)	7.70 d(8.7)	7.46 d(8.2)
12	7.49 d(8.5)	7.49 d(9)	7.35 d(9)	7.24 d(8)	7.58 d(8.7)	6.95 d(8.2)
NMe	2.62 s	2.70 s	2.73 s	2.71 s	2.79 s	2.49 s
OCH_2O	6.05 s(C-2,3)	6.13, 6.07 d(1)	6.05 s(C-2,3)	5.97 s	6.07 s	6.10 s
	6.03 s(C-7,8)	6.06, 6.04 d(1)	6.03 s(C-7,8)	5.97 s	6.07 s	6.10 s
CH_2			3.70 q(7)		2.45 d(7.7)	2.16 m, 1.96 m
Me			1.08 t(7)			
CH_2OH				3.12 t, 3.48 q		3.69 m 3.64 br s(OH)

Table 2-10-84: ^{1}H NMR spectroscopic data of benzophenanthridine-type isoquinoline alkaloids **2-10-217**~**2-10-221**.

H	2-10-217	2-10-218	2-10-219	2-10-220	2-10-221
1	7.13 s	7.11 s	7.30 s	7.36 s	7.12 s
2			3.80 s(OMe)	3.89 s(OMe)	
3			3.80 s(OMe)	3.89 s(OMe)	
4	7.58 s	7.67 s	7.36 s	7.44 s	7.63 s
6	4.88 dd(10.5, 3.5)	4.29 s	4.67 dd(10.5, 3.5)		4.21 d(10.0)
7		3.87 s(OMe)	3.81 s(OMe)	3.91 s(OMe)	3.94 s(OMe)
8		3.92 s(OMe)	3.80 s(OMe)	3.91 s(OMe)	3.94 s(OMe)
9	6.81 d(8.5)	6.94 d(8.5)	7.86 d(8.5)	8.00 d(8.5)	6.99 d(8.6)
10	7.71 d(8.5)	7.51 d(8.5)	7.90 d(8.5)	8.20 d(8.5)	7.53 d(8.6)
11	7.42 d(8.2)	7.70 d(8.6)	7.45 d(8.2)	7.67 d(8.2)	7.70 d(8.6)
12	6.85 d(8.2)	7.48 d(8.6)	6.98 d(8.2)	7.32 d(8.2)	7.50 d(8.6)
NMe	2.65 s	2.59 s	2.59 s	2.80 s	2.70 s
OCH_2O	6.04 s, 6.04 s	6.04 s			6.07 s
CH_2	2.62 dd(15.5, 3.5) 2.38 dd(15.5, 10.5)		2.12 dd(15.5, 3.5) 2.36 dd(15.5, 10.5)		
CH					3.21 dq(10.0, 6.0)
Me	2.07 s				1.10 d(6.0)
OH			11.2 s(OH)		

Table 2-10-85: ^{1}H NMR spectroscopic data of benzophenanthridine-type isoquinoline alkaloids **2-10-222**~**2-10-226**.

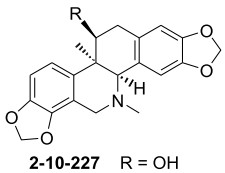
H	2-10-222	2-10-223	2-10-224	2-10-225	2-10-226
1	7.06 s	7.05 s	7.09 s	7.08 s	7.07 s
4	7.97 s	7.95 s	7.93 s	7.87 s	7.95 s
6	4.81 dd(9.3, 4.2)	4.85 dd(10.5, 5.5)	4.92 dd(11.0, 5.0)	4.86 dd(11.0, 4.5)	4.85 dd(9.5, 5.5)
9	6.65 d(8.5)	6.65 d(8.5)	6.64 d(8.5)	6.64 d(8.5)	6.64 d(8.5)
10	7.50 d(8.5)	7.51 d(8.5)	7.50 d(8.5)	7.50 d(8.5)	7.51 d(8.5)
11	7.78 d(8.5)	7.78 d(8.5)	7.77 d(8.5)	7.76 d(8.5)	7.78 d(8.5)
12	7.38 d(8.5)	7.38 d(8.5)	7.39 d(8.5)	7.38 d(8.5)	7.38 d(8.5)
NMe	2.61 s	2.59 s	2.67 s	2.50 s	2.56 s
1′	1.94 br d(12.5)	2.05 d(13.0)	1.39 m	1.79 br t(12.5)	1.56 td(14.0, 7.8)
2′α	1.57 m	1.65 m	2.09 dd(12.9, 6.0)	2.43 dd(12.5, 5.4)	1.44 m
2′β	1.49 m	1.50 m	1.89 dddd (12.9, 12.9, 12.9, 6.0)	1.29 dddd (12.5, 12.5, 12.5, 5.8)	2.38 m
3′α	2.23 m	2.21 dd(15.0, 5.5)	2.50 m	2.52 m	2.42 dd(17.0, 5.0)
3′β	2.33 dd(17.0, 5.8)	2.29 m	2.23 dd(17.0, 6.0)	2.17 dd(16.8, 5.4)	2.18 m

Table 2-10-85 (continued)

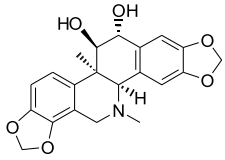
H	2-10-222	2-10-223	2-10-224	2-10-225	2-10-226
5′	5.63 d(6.0)	5.50 br d(5.5)	5.23 s	5.08 s	5.21 br s
6′	2.45 m	2.50 m	2.39 m	1.69 m	1.86 m
7′	1.27 m	1.36 m	1.03 m	0.96 m	0.84 tt(11.3, 3.0)
8′α	1.35 m	1.28 m	1.40 m	1.54 m	1.00 qd(15.5, 4.9)
8′β	1.52 m	1.56 m	1.31 dd(13.8, 2.5)	0.97 m	1.48 m
9′α	1.71 br d(13.5)	1.68 m	1.20 ddd(14.0, 10.5, 3.5)	1.64 m	1.41 q(4.0)
9′β	1.24 td(13.5, 4.0)	1.24 m	2.00 dt(14.0, 3.5)	1.81 m	1.70 m
11′	2.26 dd(14.0, 9.3)	2.27 dd(13.5, 10.5)	2.30 dd(13.5, 11.0)	2.29 dd(13.8, 11.0)	2.32 dd(14.5, 9.5)
	2.43 dd(14.0, 4.2)	2.35 dd(13.5, 5.5)	2.34 dd(13.5, 5.0)	2.38 dd(13.8, 4.5)	2.32 dd(14.5, 5.5)
12′	2.01 br sept(7.0)	1.81 br sept(7.0)	1.60 br sept(7.0)	1.48 br sept(7.0)	1.72 br sept(7.0)
13′	0.85 d(7.0)	0.75 d(7.0)	0.62 d(7.0)	0.50 d(7.0)	0.67 d(7.0)
14′	1.01 d(7.0)	0.88 d(7.0)	0.83 d(7.0)	0.77 d(7.0)	0.74 d(7.0)
10′-Me	1.04 s	1.07 s	1.13 s	1.06 s	1.17 s
10′-OMe	3.05 s	3.16 s	3.00 s	3.21 s	3.13 s
7-OMe	3.86 s	3.84 s	3.85 s	3.83 s	3.83 s
8-OMe	3.39 s	3.40 s	3.39 s	3.39 s	3.40 s
OCH_2O	5.35, 5.41d(0.1)	5.35, 5.36 s	5.38, 5.52 d(1.0)	5.39, 5.52 d(0.5)	5.38, 5.45 s

Table 2-10-86: Cos, MFs, and TSs of benzophenanthridine-type isoquinoline alkaloids **2-10-227**~**2-10-231**.

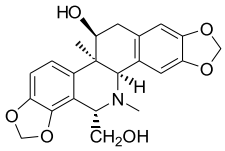
No.	Compounds	MFs	Test solvents	References
2-10-227	(±)-corynoline	$C_{21}H_{21}NO_5$	$CDCl_3$	[308]
2-10-228	(±)-acetylcorynoline	$C_{23}H_{23}NO_6$	$CDCl_3$	[308]
2-10-229	(±)-5-hydroxycorynoline	$C_{21}H_{21}NO_6$	$CDCl_3$	[308]
2-10-230	(±)-corynolamine	$C_{22}H_{23}NO_6$	$CDCl_3$	[308]
2-10-231	8-oxocorynoline	$C_{21}H_{19}NO_6$	$CDCl_3$	[308]



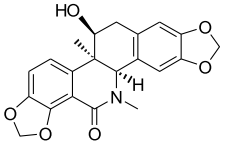
2-10-227 R = OH
2-10-228 R = OAc



2-10-229



2-10-230



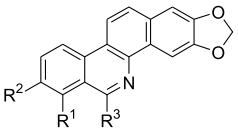
2-10-231

Table 2-10-87: ^{1}H NMR spectroscopic data of benzophenanthridine-type isoquinoline alkaloids **2-10-227**~**2-10-231**.

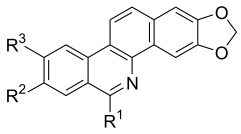
H	2-10-227	2-10-228	2-10-229	2-10-230	2-10-231
1	6.69 s	6.57 s	6.65 s	6.68 s	6.43 s
4	6.71 s	6.92 s	7.02 s	6.79 s	6.63 s
4b	3.33 s	3.56 s	3.34 s	4.29 s	4.05 s
6	3.49 d(16) 4.04 d(16)	3.58 d(16) 3.91 d(16)	3.44 q(15) 4.07 q(15)	4.18 s	
9	6.83 q(8.5)	6.76 q(8.5)	6.77 d(8.5)	7.01 q(8.5)	7.55 d(7)
10	6.86 q(8.5)	6.98 q(8.5)	6.98 d(8.5)	6.89 q(8.5)	6.76 d(7)
10b-Me	1.13 s	1.27 s	1.26 s	1.11 s	1.41 s
11	3.99 m	5.25 dd(8, 7.5)	3.92 br s	3.99 s	4.16 t(9)
12	3.14 d(2.9)	2.91 dd(16, 7.5) 2.96 dd(16, 8)	4.94 br s	3.09 d(2.8)	3.02 dd(9, 1)
NMe	2.23 s	2.49 s	2.17 s	2.31 s	3.47 s
2,3-OCH_2O	5.94 m	5.94 s	5.96 s	6.0 m	5.85~6.09 m
7,8-OCH_2O	5.94 m	5.94 s	5.98 s	6.0 m	5.85~6.09 m
Ac		1.87 s			
CH_2OH				4.81 s	

Table 2-10-88: Cos, MFs, and TSs of benzophenanthridine-type isoquinoline alkaloids **2-10-232**~**2-10-241**.

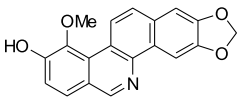
No.	Compounds	MFs	Test solvents	References
2-10-232	isodecarine	$C_{19}H_{13}NO_4$	$CDCl_3$	[312]
2-10-233	norchelerythrine	$C_{20}H_{15}NO_4$	$CDCl_3$	[308]
2-10-234	8-methoxyisodecarine	$C_{20}H_{15}NO_5$	CD_3OD	[313]
2-10-235	norsanguinarine	$C_{19}H_{11}NO_4$	CF_3COOD	[308]
2-10-236	decarine	$C_{19}H_{13}NO_4$	CF_3COOD	[308]
2-10-237	des-*N*-methylavicine	$C_{19}H_{11}NO_4$	CF_3COOD	[308]
2-10-238	*N*-nornitidine	$C_{20}H_{15}NO_4$	CD_3COCD_3	[308]
2-10-239	rhoifoline A	$C_{20}H_{13}NO_5$	$CDCl_3$	[314]
2-10-240	rhoifoline B	$C_{21}H_{17}NO_5$	$CDCl_3$	[314]
2-10-241	zanthoxyline	$C_{19}H_{13}NO_4$	DMSO-d_6	[315]



	R^1	R^2	R^3
2-10-232	OH	OMe	H
2-10-233	OMe	OMe	H
2-10-234	OH	OMe	OMe
2-10-235	OCH_2O		H
2-10-236	OMe	OH	H



	R^1	R^2	R^3
2-10-237	H	OCH_2O	
2-10-238	H	OMe	OMe
2-10-239	OMe	OCH_2O	
2-10-240	OMe	OMe	OMe



2-10-241

Table 2-10-89: ^{1}H NMR spectroscopic data of benzophenanthridine-type isoquinoline alkaloids **2-10-232**~**2-10-236**.

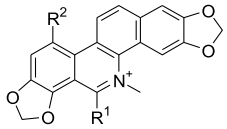
H	2-10-232	2-10-233	2-10-234	2-10-235	2-10-236
1	7.28 s	7.24 s	7.52 s	7.42 s	7.44 s
4	8.72 s	8.75 s	8.55 s	7.94 s	8.04 s
6	9.71 s	9.78 s	3.75 s(OMe)	9.51 s	9.73 d(7.5)
7	5.95 br s(OH)	4.10 s(OMe)			4.34 s(OMe)
8	4.06 s(OMe)	4.17 s(OMe)	3.93 s(OMe)		
9	7.59 d(9.0)	7.58 d(9.2)	7.60 d(9.0)	7.92 d(8)	8.09 d(9.5)
10	8.36 d(9.0)	8.36 d(9.2)	8.48 d(9.0)	8.45 d(8)	8.50 d(9.5)
11	8.37 d(9.0)	8.36 d(9)	8.53 d(9.0)	8.15 d(10)	8.59 d(9.5)
12	7.86 d(9.0)	7.86 d(9)	7.98 d(9.0)	8.47 d(10)	8.17 d(9.5)
OCH_2O	6.14 s	6.16 s	6.22 s	6.25 s(C-2,3) 6.50 s(C-7,8)	6.20 s

Table 2-10-90: ^{1}H NMR spectroscopic data of benzophenanthridine-type isoquinoline alkaloids **2-10-237**~**2-10-241**.

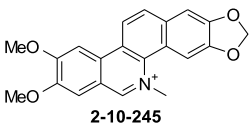
H	2-10-237	2-10-238	2-10-239	2-10-240	2-10-241
1	7.43 s	7.63 s	7.07 s	7.19 s	7.46
4	8.14 s	8.65 s	7.50 s	7.65 s	8.57
6	9.20 d(8)	9.25 s	3.88 s(OMe)	3.99 s(OMe)	9.61
7	7.64 s	–	7.81 s	7.94 s	8.42d
8		4.02 s(OMe)		4.06 s(OMe)	7.61d
9		4.13 s(OMe)		4.11 s(OMe)	
10	7.99 s	8.13 s	7.53 s	7.60 s	4.09 s(OMe)
11	8.37 d(9)	8.50 d(10)	7.82 d(8.8)	8.00 d(8.9)	8.46 d
12	8.10 d(9)	7.90 d(10)	7.44 d(8.8)	7.57 d(8.9)	7.92 d
OCH_2O	6.23 s(C-2,3) 6.38 s(C-7,8)	6.15 s	6.00 s(C-2,3) 6.03 s(C-8,9)	6.11 s(C-2,3)	6.09 s

Table 2-10-91: Cos, MFs, and TSs of benzophenanthridine-type isoquinoline alkaloids **2-10-242**~**2-10-245**.

No.	Compounds	MFs	Test solvents	References
2-10-242	8-methoxysanguinarine	$C_{21}H_{16}NO_5$	CD_3OD	[313]
2-10-243	sanguinarine	$C_{20}H_{14}NO_4$	CF_3COOD	[308]
2-10-244	chelirubine	$C_{21}H_{16}NO_5$	CF_3COOD	[308]
2-10-245	nitidine	$C_{21}H_{18}NO_4$	CF_3COOD	[308]



	R^1	R^2
2-10-242	OMe	H
2-10-243	H	H
2-10-244	H	OMe



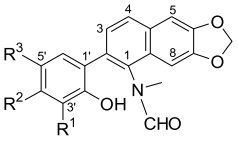
2-10-245

Table 2-10-92: ^{1}H NMR spectroscopic data of benzophenanthridine-type isoquinoline alkaloids **2-10-242**~**2-10-245**.

H	2-10-242	2-10-243	2-10-244	2-10-245
1	7.39 s	7.52 s	7.48 s	7.55 s
4	8.03 s	8.05 s	7.87 s	8.14 s
6	3.85 s(OMe)	9.62 s	9.49 s	9.42 s
7				7.80 s
8				4.28 s(OMe)
9	8.03 d(9.0)	8.57 d(9)	7.61 s	4.32 s(OMe)
10	8.46 d(9.0)	7.94 d(9)	4.27 s(OMe)	8.26 s
11	8.49 d(9.0)	8.47 d(9)	9.53 d(8.5)	8.57 d(9)
12	8.07 d(9.0)	8.20 d(9)	8.13 d(8.5)	8.21 d(9)
NMe	4.83 s	5.05 s	4.96 s	5.05 s
2,3-OCH_2O	6.19 s	6.28 s	6.24 s	6.30 s
7,8-OCH_2O	6.22 s	6.52 s	6.44 s	

Table 2-10-93: Cos, MFs, and TSs of benzophenanthridine-type isoquinoline alkaloids **2-10-246**~**2-10-249**.

No.	Compounds	MFs	Test solvents	References
2-10-246	arnottianamide	$C_{21}H_{19}NO_6$	DMSO-d_6	[316]
2-10-247	iwamide	$C_{20}H_{17}NO_6$	$CDCl_3$-CD_3OD	[308]
2-10-248	integriamide	$C_{20}H_{15}NO_6$	$CDCl_3$-CD_3OD	[308]
2-10-249	isoarnottianamide	$C_{21}H_{19}NO_6$	CF_3COOD	[308]



	R^1	R^2	R^3
2-10-246	OMe	OMe	H
2-10-247	OMe	OH	H
2-10-248	H	OCH_2O	
2-10-249	H	OMe	OMe

Table 2-10-94: ^{1}H NMR spectroscopic data of benzophenanthridine-type isoquinoline alkaloids **2-10-246**~**2-10-249**.

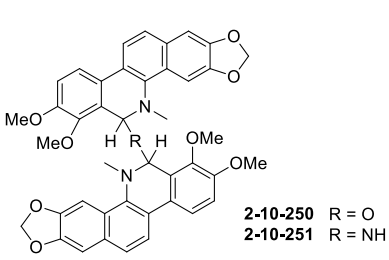
H	2-10-246	2-10-247	2-10-248	2-10-249
3	7.23 d(8.3)	7.71 d(9)	7.74 d(8.5)	7.88 d(9)
4	7.80 d(8.3)	7.27 d(9)	7.28 d(8.5)	7.35 d(9)
5	7.45 s	7.00 s	7.02 s	7.02 s
8	7.01 s	7.18 s	7.20 s	7.27 s
2′	8.90 br s(OH)			
3′	3.35 s(OMe)	3.82 s(OMe)	6.53 s	6.88 s
4′	3.69 s(OMe)			3.97 s(OMe)
5′	6.56 d(8.8)	6.43 d(9)		3.97 s(OMe)
6′	6.73 d(8.8)	6.66 d(9)	6.48 s	6.78 s

Table 2-10-94 (continued)

H	2-10-246	2-10-247	2-10-248	2-10-249
NMe	2.89 s	2.97 s	3.01 s	3.26 s
CHO	7.96 s	8.07 s	8.10 s	8.50 s
6,7-OCH_2O	6.18 s	6.05 s	5.91 s	6.08 s
4′,5′-OCH_2O			6.07 s	

Table 2-10-95: Cos, MFs, and TSs of benzophenanthridine-type isoquinoline alkaloids **2-10-250**~**2-10-252**.

No.	Compounds	MFs	Test solvents	References
2-10-250	bis[6-(5,6-dihydrochelerythrinyl)]ether	$C_{42}H_{36}N_2O_9$	$CDCl_3$	[317]
2-10-251	bis[6-(5,6-dihydrochelerythrinyl)]amine	$C_{42}H_{37}N_3O_8$	$CDCl_3$	[317]
2-10-252	caymandimerine	$C_{44}H_{38}N_2O_9$	$CDCl_3$	[318]



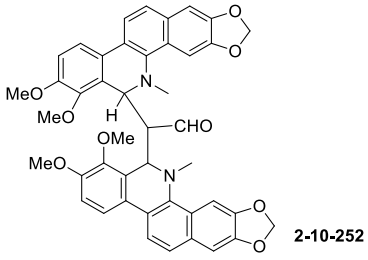


Table 2-10-96: ^1H NMR spectroscopic data of benzophenanthridine-type isoquinoline alkaloids **2-10-250**~**2-10-252**.

H	2-10-250	2-10-251	2-10-252
1,1′	7.16 s	7.15 s	7.05 s
4,4′	7.93 s	7.99 s	7.55 s
6,6′	6.61 s	5.98 s	5.06 d(5.7)
9,9′	6.84 d(8.7)	6.79 d(8.7)	7.49 or 6.93 q(8.7)
10,10′	7.48 d(8.7)	7.42 d(8.7)	7.49 or 6.93 q(8.7)
11,11′	7.68 d(8.5)	7.65 d(8.5)	7.59 or 7.41 q(8.6)
12,12′	7.44 d(8.5)	7.42 d(8.5)	7.59 or 7.41 q(8.6)
OCH_2O	6.11 s	6.10 s	6.02, 6.01 q(1.1)
NMe	3.05 s	2.91 s	2.69
7,7′-OMe	2.41 s	2.58 s	3.88 or 3.74 s
8,8′-OMe	3.72 s	3.72 s	3.88 or 3.74 s
CH			2.02 dd(6.8, 5.7)
CHO			8.78 d(6.8)

Bibliography

[226] Singh S, Singh TD, Singh VP, et al. Nat Prod Res, 2010, 24: 63.
[227] Liu C, Beecher CWW, Zhao SX. J Nat Prod, 2005, 58: 1100.
[228] Chen J, Chen B, Tian J, et al. Chin Chem Lett, 2002, 13: 426.
[229] Blanchfield JT, Sands DPA, Kennard CHL, et al. Phytochemistry, 2003, 63: 711.
[230] Lee SS, Lin YJ, Chen CK, et al. J Nat Prod, 1993, 56: 1971.
[231] Nishiyama Y, Moriyasu M, Ichimaru M, et al. Phytochemistry, 2006, 67: 2671.
[232] Nishiyama Y, Moriyasu M, Ichimaru M, et al. Phytochemistry, 2004, 65: 939.
[233] Lee SS, Lai YC, Chen CK, et al. J Nat Prod, 2007, 70: 637.
[234] Moriyasu M, Ichimaru M, Nishiyama Y, et al. J Nat Prod, 1997, 60: 299.
[235] Xu Q, Lin M. J Nat Prod, 1999, 62: 1025.
[236] Zhou Y, Zhang GL, Li BG. Phytochemistry, 1999, 50: 339.
[237] Carroll AR, Davis RA, Forster PI, et al. J Nat Prod, 2001, 64: 1572.
[238] Buchanan MS, Davis RA, Duffy S, et al. J Nat Prod, 2009, 72: 1541.
[239] Zhang XQ, Ye WC, Zhao SX, et al. Phytochemistry, 2004, 65: 929.
[240] Al-Rehaily AJ, Sharaf MHM, Zemaitis MA, et al. J Nat Prod, 1999, 62: 146.
[241] Chen CY, Chang FR, Pan WB, et al. Phytochemistry, 2001, 56: 753.
[242] Chang FR, Hsieh TJ, Huang TL, et al. J Nat Prod, 2002, 65: 255.
[243] Tanahashi T, Su Y, Nagakura N, et al. Chem Pharm Bull, 2000, 48: 370.
[244] Táborská E, Bochořáková H, Soušek J, et al. Heterocycles, 1997, 45: 817.
[245] Böhlke M, Guinaudeau H, Angerhofer CK, et al. J Nat Prod, 1996, 59: 576.
[246] Lin LZ, Hu SF, Chu M, et al. Phytochemistry, 1999, 50: 829.
[247] Lee SS, Doskotch RW. J Nat Prod, 1999, 62: 803.
[248] Philipov SA, Istatkova RS. Phytochemistry, 1999, 51: 1161.
[249] Mahiou V, Roblot F, Fournet A, et al. Phytochemistry, 2000, 54: 709.
[250] Istatkova RS, Philipov SA. Phytochemistry, 2000, 54: 959.
[251] Lee SS, Wu WN, Wilton JH, et al. J Nat Prod, 1999, 62: 1410.
[252] Kanyinda B, Vanhaelen-Fastré R, Vanhaelen M, et al. J Nat Prod, 1997, 60: 1121.
[253] Kanyinda B, Vanhaelen-Fastre R, Vanhaelen M. J Nat Prod, 1995, 58: 1587.
[254] Wang JZ, Chen QH, Wang FP. J Nat Prod, 2010, 73: 1288.
[255] Menachery MD, Stern EW, Steinbeiser RJ, et al. Tetrahedron, 2000, 41: 2843.
[256] Kanyinda B, Vanhaelen-Fastre R, Vanhaelen M, et al. J Nat Prod, 1993, 56: 957.
[257] Atta-Ur-Rahman, Atia-Tul-Wahab, Nawaz SA, et al. Chem Pharm Bull, 2004, 52: 802.
[258] Kashiwaba N, Morooka S, Kimura M, et al. J Nat Prod, 1996, 59: 476.
[259] Carroll AR, Arumugan T, Redburn J, et al. J Nat Prod, 2010, 73: 988.
[260] Sugimoto Y, Inanaga S, Kato M, et al. Phytochemistry, 1998, 49: 1293.
[261] Sugimoto Y, Babiker HAA, Saisho T, et al. J Org Chem, 2001, 66: 3299.
[262] Yu BW, Chen JY, Wang YP, et al. Phytochemistry, 2002, 61: 439.
[263] Guinaudeau H, Lin LZ, Cordell GA, et al. Phytochemistry, 1998, 49: 2561.
[264] Morimoto S, Suemori K, Taura F, et al. J Nat Prod, 2003, 66: 987.
[265] Jin HZ, Wang XL, Wang HB, et al. J Nat Prod, 2008, 71: 127.
[266] Yang JH, Li L, Wang YS, et al. Helv Chim Acta, 2005, 88: 2523.
[267] Deng Y, Chen J, Wu FE. Chin Chem Lett, 2002, 13: 862.
[268] Chen JH, Du ZZ, Shen YM, et al. Arch Pharm Res, 2009, 32: 3.
[269] Takatsu H, Yamadaya T, Furihata K, et al. J Nat Prod, 2005, 68: 430.
[270] Carollo CA, Siqueira JMD, Garcez WS, et al. J Nat Prod, 2006, 69: 1222.
[271] Kashiwaba N, Ono M, Toda J, et al. J Nat Prod, 2000, 63: 477.
[272] Zhang MS, Liang GY, Yu JP, et al. Nat Prod Res, 2010, 24: 1243.
[273] Wu YC, Chao YC, Chang FR, et al. Phytochemistry, 1997, 46: 181.

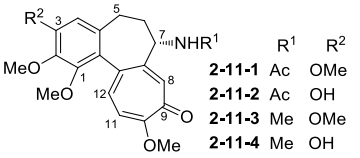
[274] Lee SS, Chen CK, Chen IS, et al. J Nat Prod, 1996, 59: 55.
[275] Chang FR, Chen CY, Wu PH, et al. J Nat Prod, 2000, 63: 746.
[276] Hsieh TJ, Chen CY, Kuo RY, et al. J Nat Prod, 1999, 62: 1192.
[277] Hsieh TJ, Chang FR, Chia YC, et al. J Nat Prod, 2001, 64: 1157.
[278] Sari A, Gary AI, Sariyar G. Nat Prod Res, 2004, 18: 265.
[279] Chang FR, Wei JL, Teng CM, et al. Phytochemistry, 1998, 49: 2015.
[280] Chen JJ, Tsai IL, Ishikawa T, et al. Phytochemistry, 1996, 42: 1479.
[281] Chen CC, Huang YL, Lee SS, et al. J Nat Prod, 1997, 60: 826.
[282] Chia YC, Chang FR, Wu YC. J Nat Prod, 1998, 61: 1430.
[283] Zhang YJ, Kong M, Chen RY, et al. J Nat Prod, 1999, 62: 1050.
[284] Wu TS, Leu YL, Chan YY. Chem Pharm Bull, 1998, 46: 1624.
[285] Chen B, Feng C, Li BG, et al. Nat Prod Res, 2003, 17: 397.
[286] Menachery MD, Mandell HM, DeSaw SA, et al. J Nat Prod, 1997, 60: 1328.
[287] Harrigan GG, Gunatilaka AAL, Kingston DGI, et al. J Nat Prod, 1994, 57: 68.
[288] Wu TS, Lin FW. J Nat Prod, 2001, 64: 1404.
[289] Sugimoto Y, Babiker HAA, Inanaga S, et al. Phytochemistry, 1999, 52: 1431.
[290] Yu BW, Meng LH, Chen JY, et al. J Nat Prod, 2001, 64: 968.
[291] Al-Howiriny TA, Zemaitis MA, Gao CY, et al. J Nat Prod, 2001, 64: 819.
[292] Li HL, Zhang WD, Zhang W, et al. Chin Chem Lett, 2005, 16: 367.
[293] Lopes LMX, Humpfer E. Phytochemistry, 1997, 45: 431.
[294] Chudík S, Marek R, Sečkářová P, et al. J Nat Prod, 2006, 69: 954.
[295] Pinho PMM, Pinto MMM, Kijjoa A, et al. Phytochemistry, 1992, 31: 1043.
[296] Zhang JS, Men-Olivier LL, Massiot G. Phytochemistry, 1995, 39: 439.
[297] Valpuesta M, Díaz A, Suau R. Phytochemistry, 1999, 51: 1157.
[298] Chen CY, Chang FR, Shih YC, et al. J Nat Prod, 2000, 63: 1475.
[299] Ito C, Mizuno T, Wu TS, et al. Phytochemistry, 1990, 29: 2044.
[300] Atta-UR-Rahman, Ahmad AS, Bhatti MK, et al. Phytochemistry, 1995, 40: 593.
[301] Wafo P, Nyasse B, Fontaine C. Phytochemistry, 1999, 50: 279.
[302] Thuy TT, Franke K, Porzel A, et al. J Chem, 2006, 44: 259.
[303] Cheng XY, Shi Y, Zheng SL, et al. J Asian Nat Prod Res, 2008, 10: 1117.
[304] Rastrelli L, Capasso A, Pizza C, et al. J Nat Prod, 1997, 60: 1065.
[305] Rücker G, Breitmaier E, Zhang GL, et al. Phytochemistry, 1994, 36: 519.
[306] Oechslin SM, König GM, Oechslin-Merkel K, et al. J Nat Prod, 1991, 54: 519.
[307] Zhang GL, Rücker G, Breitmaier E, et al. Phytochemistry, 1995, 40: 1813.
[308] Krane BD, Fagbule MO, Shamma M. J Nat Prod, 1984, 47: 1.
[309] Koul S, Razdan TK, Andotra CS, et al. Planta Med, 2002, 68: 262.
[310] Hu J, Zhang WD, Liu RH, et al. Chem Biodivers, 2006, 3: 990.
[311] Yang CH, Cheng MJ, Chiang MY, et al. J Nat Prod, 2008, 71: 669.
[312] Chen JJ, Fang HY, Duh CY, et al. Planta Med, 2005, 71: 470.
[313] Cui XG, Zhao QJ, Chen QL, et al. Helv Chim Acta, 2008, 91: 155.
[314] Gonzaga WDA, Weber AD, Giacomelli SR, et al. Planta Med, 2003, 69: 371.
[315] Moura NFD, Ribeiro HB, Machado ECS, et al. Phytochemistry, 1997, 46: 1443.
[316] Hsiao JJ, Chiang HC. Phytochemistry, 1995, 39: 899.
[317] Dostál J, Táborská E, Slavík J, et al. J Nat Prod, 1995, 58: 723.
[318] Ng KM, Gray AI, Waterman PG. Phytochemistry, 1987, 26: 3251.

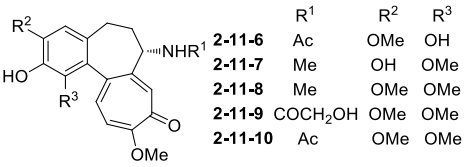
2.11 Phenylethylated tetrahydroisoquinoline alkaloids

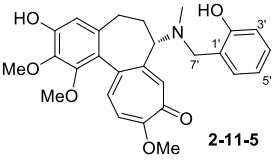
2.11.1 Colchicine-type phenylethylated tetrahydroisoquinoline alkaloids

Table 2-11-1: Cos, MFs, and TSs of colchicine-type alkaloids **2-11-1**~**2-11-11**.

No.	Compounds	MFs	Test solvents	References
2-11-1	(–)-colchicine	$C_{22}H_{25}NO_6$	$CDCl_3$	[319]
2-11-2	(–)-3-demethylcolchicine	$C_{21}H_{23}NO_6$	$CDCl_3$	[319, 321]
2-11-3	(–)-demecolcine	$C_{21}H_{25}NO_5$	$CDCl_3$	[319, 321]
2-11-4	(–)-3-demethyldemecolcine	$C_{20}H_{23}NO_5$	$CDCl_3$	[319, 321]
2-11-5	(–)-specioritchine	$C_{27}H_{29}NO_6$	$CDCl_3$	[319]
2-11-6	1,2-didemethylcolchicine	$C_{20}H_{21}NO_6$	CD_3OD	[320]
2-11-7	(–)-2,3-didemethyldemecolcine	$C_{19}H_{21}NO_5$	$CDCl_3$	[321]
2-11-8	2-demethyldemecolcine	$C_{20}H_{23}NO_5$	$CDCl_3$	[322]
2-11-9	2-demethylcolchifoline	$C_{21}H_{23}NO_7$	$CDCl_3$	[322]
2-11-10	2-demethylcolchicine	$C_{21}H_{23}NO_6$	$CDCl_3$	[323]
2-11-11	(+)-demecolcinone	$C_{19}H_{19}NO_5$	$CDCl_3$	[321]







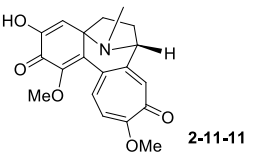


Table 2-11-2: ^{1}H NMR spectroscopic data of colchicine-type alkaloids **2-11-1**~**2-11-6**.

H	2-11-1	2-11-2	2-11-3	2-11-4	2-11-5	2-11-6
1	3.63 s(OMe)	3.64 s(OMe)	3.57 s(OMe)	3.59 s(OMe)	3.52 s(OMe)	
2	3.92 s(OMe)	4.00 s(OMe)	3.89 s(OMe)	3.99 s(OMe)	3.99 s(OMe)	
3	3.88 s(OMe)		3.87 s(OMe)			3.86 s(OMe)
4	6.52	6.59	6.51	6.59	6.59	6.43
5α	2.34 m	2.30	2.34	2.26	2.35 m	2.28
5β	2.50 m	2.49	2.40	2.43	2.51 m	2.45
6α	1.98 m	1.93	1.62	1.75	1.96 m	1.94
6β	2.34 m	2.30	2.12	2.26	2.35 m	2.18
7	4.63 m	4.65	3.23	3.36	3.16 m	4.39 dd(6, 11.5)
8	7.64	7.56	7.66	7.70	7.62 m	7.15

Table 2-11-2 (continued)

H	2-11-1	2-11-2	2-11-3	2-11-4	2-11-5	2-11-6
10	4.00 s(OMe)	4.01 s(OMe)	3.97 s(OMe)	4.01 s(OMe)	3.99 s(OMe)	–
11	6.89 d(10.5)	6.90 d(10.5)	6.78 d(10.5)	6.82 d(10.5)	6.77 d(10.5)	6.98 d(10.5)
12	7.33 d(10.5)	7.34 d(10.5)	7.20 d(10.5)	7.24 d(10.5)	7.25 d(10.5)	7.23 d(10.5)
NH	8.53 d(6.3)	7.78 d(6.3)				–
NMe			2.18	2.30	2.24	
Ac	1.93 s	1.99				1.88
3′					6.83 m	
4′					7.14 m	
5′					6.73 m	
6′					6.85 m	
7′					3.92 d, 3.52 d	

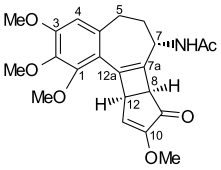
Table 2-11-3: ^{1}H NMR spectroscopic data of colchicine-type alkaloids **2-11-7**~**2-11-11**.

H	2-11-7	2-11-8	2-11-9	2-11-10	2-11-11
1	3.47 s(OMe)	3.57 s(OMe)	3.58 s(OMe)	3.62 s(OMe)	4.14 s(OMe)
3		3.93 s(OMe)	3.89 s(OMe)	3.89 s(OMe)	
4	6.58 s	6.53 s	6.49 s	6.48 s	5.25 s
5	2.31 m, 2.40 m	1.71~2.47 m	1.90~2.50 m	1.85~2.50 m	1.69 m, 2.43 m
6	1.63 m, 2.15 m	1.71~2.47 m	1.90~2.50 m	1.85~2.50 m	1.85 m, 2.04 m
7	3.31 dd(6, 6)	3.37 m	4.67 m	4.61 m	4.31 dd(2, 2)
8	7.72 s	7.66 s	7.57 s	7.54 s	7.16 s
10	4.00 s(OMe)	4.01 s(OMe)	3.95 s(OMe)	3.97 s(OMe)	3.93 s(OMe)
11	6.79 d(11)	6.84 d(10.7)	6.84 d(10.8)	6.83 d(10.8)	6.66 d(11)
12	7.24 d(11)	7.29 d(10.7)	7.31 d(10.8)	7.30 d(10.8)	7.11 d(11)
NH	4.15 br s		7.74 d	7.75 d	
NMe	2.21 s	2.28 s			3.03 s
Ac				1.93 s	
$COCH_2OH$			4.12, 3.98 5.92(OH)		

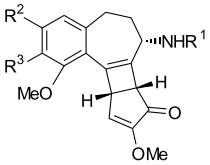
Table 2-11-4: Cos, MFs, and TSs of colchicine-type alkaloids **2-11-12**~**2-11-18**.

No.	Compounds	MFs	Test solvents	References
2-11-12	*γ*-lumicolchicine	$C_{22}H_{25}NO_6$	$CDCl_3$	[324]
2-11-13	*β*-lumicolchicine	$C_{22}H_{25}NO_6$	$CDCl_3$	[324]
2-11-14	3-*O*-demethyl-*N*-formyldesacetyl-*β*-lumicolchicine	$C_{20}H_{21}NO_6$	$CDCl_3$	[325]
2-11-15	2-demethyl-*β*-lumicolchicine①	$C_{21}H_{23}NO_6$	$CDCl_3$	[322]
2-11-16	(–)-jerusalemine	$C_{19}H_{23}NO_5$	$CDCl_3$	[326]
2-11-17	(–)-salimine	$C_{22}H_{25}NO_7$	$CDCl_3$	[326]
2-11-18	(–)-suhailamine	$C_{22}H_{25}NO_6$	$CDCl_3$	[326]

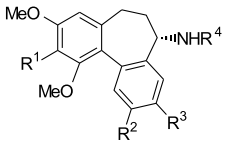
① Typographic errors exist in the literature, with the double bond of C7a-C8a being drown as single bond.



2-11-12



	R^1	R^2	R^3
2-11-13	Ac	OMe	OMe
2-11-14	CHO	OH	OMe
2-11-15	Ac	OMe	OH



	R^1	R^2	R^3	R^4
2-11-16	OH	OH	OMe	Me
2-11-17	OMe	COOH	OMe	Ac
2-11-18	OMe	H	COOMe	Ac

Table 2-11-5: ^{1}H NMR spectroscopic data of colchicine-type alkaloids **2-11-12**~**2-11-15**.

H	2-11-12	2-11-13	2-11-14	2-11-15
1	3.96 s(OMe)	3.98 s(OMe)	3.94 s(OMe)	3.94 s(OMe)
2	3.87 s(OMe)	3.88 s(OMe)	3.91 s(OMe)	
3	3.86 s(OMe)	3.86 s(OMe)		3.89 s(OMe)
4	6.48 s	6.50 s	6.54 s	6.50 s
5	2.66 m	2.77 dd(15.3, 8.7)	α 2.53 dm(16)	2.76 dd(15.8, 7.9)
			β 2.73 dm(16)	2.56 dd(15.8, 9.0)
6	1.96 m	2.60 dd(15.3, 8.8)	α 2.01 dm(12)	2.00 m
		2.00 m	β 2.04 dm(12)	
7	4.66 dt(7.6, 6.5, 6.5)	4.82 m	4.90 dm(13.3)	4.83 m
8	3.63 dd(2.8, 1.0)	3.61 dd(2.6, 1.8)	3.63 dd(1.7, 2.8)	3.63 dd(2.8, 2.0)
10	3.69 s(OMe)	3.68 s(OMe)	3.70 s(OMe)	3.67 s(OMe)
11	6.62 d(3.8)	6.67 d(3.1)	6.62 d(3.3)	6.71 d(3.2)
12	4.04 dd(3.8, 2.8)	4.11 dd(3.1, 2.6)	4.09 dd(2.9, 6.0)	4.13 dd(3.2, 2.8)
NH	6.02 d(7.6)	6.17 d(7.2)	6.02 br d(6.3)	6.71 d(7.1)
Ac	2.02 s	2.06 s		2.04 s
CHO			8.20 t(1.3)	

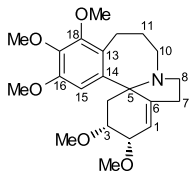
Table 2-11-6: ^{1}H NMR spectroscopic data of colchicine-type alkaloids **2-11-16**~**2-11-18**.

H	2-11-16	2-11-17	2-11-18
1	3.50 s(OMe)	3.55 s(OMe)	3.52 s(OMe)
2		3.92 s(OMe)	3.92 s(OMe)
3	3.91 s(OMe)	3.89 s(OMe)	3.89 s(OMe)
4	6.55	6.55	6.55
5	α 2.36 m, β 2.45 m	α 2.35 m, β 2.45 m	α 2.41 m, β 2.45 m
6	α 1.77 m, β 2.29 m	α 1.80 m, β 2.39 m	α 1.77 m, β 2.35 m
7	3.41 dd(6.4, 12.0)	4.80 m	4.78 m
8	7.05	6.90	6.80 d(2.8)
9	3.87 s(OMe)	3.93 s(OMe)	3.05 (COOMe)
10		10.82 (COOH)	6.84 dd(2.8, 8.4)
11	7.14	7.96	7.42 d(8.4)
NH	2.40 (NMe)	5.75 br d	5.71 br d
Ac		2.04	2.06

2.11.2 Homoerythrine-type phenylethylated tetrahydroisoquinoline alkaloids

Table 2-11-7: Cos, MFs, and TSs of homoerythrine-type alkaloids **2-11-19**~**2-11-27**.

No.	Compounds	MFs	Test solvents	References
2-11-19	2-*α*-methoxycomosivine	$C_{22}H_{31}NO_5$	$CDCl_3$	[327]
2-11-20	cephalezomine M	$C_{18}H_{23}NO_3$	CD_3OD	[328]
2-11-21	1,2-didehydro-6,7-epoxy-3*α*,16,17-trimethoxy-erythrinan-8-one	$C_{20}H_{23}NO_5$	$CDCl_3$	[329]
2-11-22	robustimine	$C_{18}H_{21}NO_4$	$CDCl_3$	[330]
2-11-23	*O*-methylrobustimine	$C_{19}H_{23}NO_4$	$CDCl_3$	[330]
2-11-24	1,6-epoxy-comoswine	$C_{21}H_{29}NO_5$	$CDCl_3$	[331]
2-11-25	2*α*-methoxylenticellarine	$C_{20}H_{27}NO_6$	$CDCl_3$	[327]
2-11-26	2*α*-hydroxylenticellarine	$C_{19}H_{25}NO_6$	$CDCl_3$	[327]
2-11-27	lenticellarine	$C_{19}H_{25}NO_5$	CD_2Cl_2, $CDCl_3$	[332]



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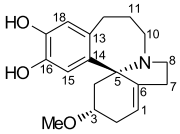

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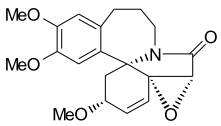

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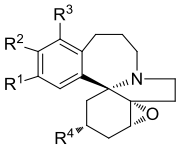
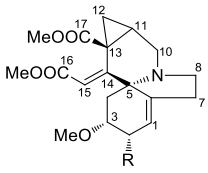


	R^1	R^2	R^3	R^4
2-11-22	OCH_2O		H	OH
2-11-23	OCH_2O		H	OMe
2-11-24	OMe	OMe	OMe	OMe



2-11-25 R = OMe
2-11-26 R = OH
2-11-27 R = H

Table 2-11-8: ^{1}H NMR spectroscopic data of homoerythrine-type alkaloids **2-11-19**~**2-11-23**.

H	2-11-19	2-11-20	2-11-21	2-11-22	2-11-23
1	5.70 br s	6.05 s	5.76 dd(10.4, 2.4)	3.78	3.79
2	4.40 dd(6, 2) 3.36 s(OMe)	2.08 m,2.72 m	6.26 td(10.4, 1.5)	1.98,2.41	2.01,2.35
3	3.98 m 3.30 s(OMe)	3.28 m 3.23 s(OMe)	3.47 m 3.31 s(OMe)	3.43	3.00 3.18 (OMe)
4	ax 1.78 m eq 2.60 m	1.77 t(11.4) 2.84 m		ax 1.85 eq 2.46	ax 1.80 eq 2.50
7	2.00~1.23 m	2.72 m	3.83 br s	2.28, 1.8	2.26, 1.84
8	3.42 m	2.72 m, 3.35 s		3.01, 2.82	3.00, 2.82

Table 2-11-8 (continued)

H	2-11-19	2-11-20	2-11-21	2-11-22	2-11-23
10	ax 3.74 dd(9, 15) eq 3.46 t(5)	3.51 d(14.2) 3.77 m	α 3.21 dd(12.4, 2.0) β 4.46 td(12.4, 3.2)	3.30,3.43	3.32,3.47
11	2.00~1.23 m	1.94 m, 2.08 m	α 1.64 m, β 1.99 m	1.8, 1.53	1.84, 1.51
12	2.00~1.23 m	3.17 t(13.0) 3.28 m	α 2.86 dd(15.6, 6.8) β 3.18 dd(15.6, 2.4)	3.12 2.70	3.15 2.71
15	6.62 s	6.75 s	7.07 s	6.68	6.71
16	3.84 s(OMe)		3.83 s(OMe)		
17	3.76 s(OMe)		3.88 s(OMe)		
18	3.86 s(OMe)	6.71 s	6.64 s	6.63	6.66
OCH_2O				5.93	5.96

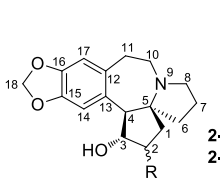
Table 2-11-9: ^{1}H NMR spectroscopic data of homoerythrine-type alkaloids **2-11-24**~**2-11-27**.

H	2-11-24	2-11-25	2-11-26	2-11-27(CD_2Cl_2)	2-11-27($CDCl_3$)
1	3.79 d	5.72 br s	5.72 br s	5.52 br s	5.52 br s
2	2.29 m 2.05 dd(15.5, 6.7)	4.42 d(3, 8) 3.82 s(OMe)	4.43 dd(3, 8)	ax 1.94 m(16.4, 8.6) eq 2.65 br d(16.4, <2)	ax 2.00 br eq 2.67 br d(15)
3	3.01 or 2.96 m 3.16 s(OMe)	3.67 m 3.33 s(OMe)	3.68 m 3.34 s(OMe)	3.6 m(ov) 3.28 s(OMe)	3.66 m
4	2.54 dd 1.83 dd(11.3, 11.3)	ax 1.90 t(12) eq 2.62 dd(4, 12)	ax 1.92 dd(4, 12) eq 2.62 t(4)	ax 1.37 br t(12.3, 11.7) eq 2.03 dd(12.3, 4)	ax 1.37 t(12) eq 2.06 dd(12, 4)
7	2.29 m 1.83 m	2.48 m	2.48 m	2.38 m(15.3) 2.40 m(15.3)	2.49 m 2.47 m
8	2.82 m 3.01 m or 2.96 m	2.96 m	2.96 m	2.95 m	2.97 m 2.95 m
10	3.48 m 3.32 br d(13.5)	3.04 dd(5, 15) 3.44 dd(9, 15)	3.04 dd(5, 15) 3.44 dd(9, 15)	3.02 dd(15.2, 5.2) 3.41 dd(15.2, 8.6)	3.04 dd(15, 5) 3.44 dd(15, 9)
11	1.78 m 1.58 m	1.60 m	1.62 br m	1.58 dddd (9, 8.6, 6.8, 5.2)	1.60 m
12	3.48 m 2.59 m	0.92 dd(5, 9) 1.07 dd(5.0, 5.8)	0.94 dd(5, 9) 1.09 dd(5, 5.8)	1.01 dd(6.8, 4.9) 2.29 dd(9, 4.9)	1.08 dd(6.8, 5) 2.37 dd(9, 5)
15	6.55 s	6.90 s	6.88 s	6.04 s	6.07 s
16		3.92 s(OMe)	3.92 s(OMe)	3.64 s(OMe)	
17		3.46 s(OMe)	3.47 s(OMe)	3.54 s(OMe)	
OMe	3.89 s, 3.82 s, 3.78 s				3.69 s, 3.62 s, 3.33 s

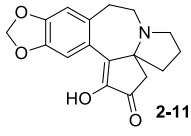
2.11.3 Cephalotaxine-type phenylethylated tetrahydroisoquinoline alkaloids

Table 2-11-10: Cos, MFs, and TSs of cephalotaxine-type alkaloids **2-11-28**~**2-11-43**.

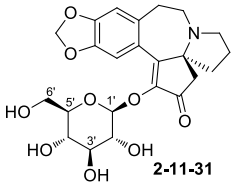
No.	Compounds	MFs	Test solvents	References
2-11-28	cephalezomine G	$C_{17}H_{21}NO_4$	CD_3OD	[328]
2-11-29	cephalezomine H	$C_{17}H_{21}NO_4$	CD_3OD	[328]
2-11-30	5,6,8,9-tetrahydro-1-hydro-4*H*-cyclopenta[*a*]-[1,3]-dioxolo[4,5-*h*]-pyrrolo[2,1-*b*][3]benzaepin-2(3*H*)-one	$C_{17}H_{17}NO_4$	C_5D_5N	[329]
2-11-31	cephalezomine J	$C_{23}H_{27}NO_9$	CD_3OD	[328]
2-11-32	deoxyharringtonine	$C_{28}H_{37}NO_8$	$CDCl_3$	[333]
2-11-33	nordeoxyharringtonine	$C_{27}H_{35}NO_8$	$CDCl_3$	[333]
2-11-34	homodeoxyharringtonine	$C_{29}H_{39}NO_8$	$CDCl_3$	[333]
2-11-35	bishomodeoxyharringtonine	$C_{30}H_{41}NO_8$	$CDCl_3$	[333]
2-11-36	5′-des-*O*-methylharringtonine	$C_{27}H_{35}NO_9$	DMSO-d_6	[334]
2-11-37	3′*S*-hydroxy-5′-des-*O*-methylharringtonine	$C_{27}H_{35}NO_{10}$	DMSO-d_6	[334]
2-11-38	5′-des-*O*-methylisoharringtonine	$C_{27}H_{35}NO_9$	DMSO-d_6	[334]
2-11-39	5′-des-*O*-methylhomoharringtonine	$C_{28}H_{37}NO_9$	DMSO-d_6	[334]
2-11-40	cephalezomine C	$C_{28}H_{37}NO_{10}$	$CDCl_3$	[335]
2-11-41	cephalezomine D	$C_{28}H_{37}NO_{10}$	$CDCl_3$	[335]
2-11-42	cephalezomine E	$C_{29}H_{39}NO_{10}$	$CDCl_3$	[335]
2-11-43	cephalezomine F	$C_{29}H_{39}NO_9$	$CDCl_3$	[335]



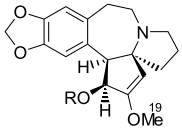
2-11-28 R = α-OH
2-11-29 R = β-OH

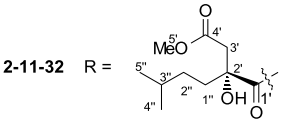


2-11-30

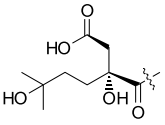


2-11-31

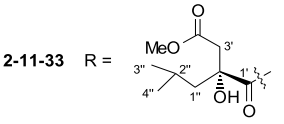




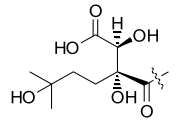
2-11-32 R =



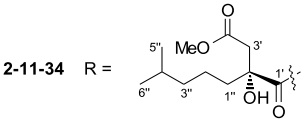
2-11-36 R =



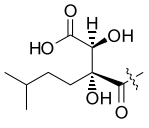
2-11-33 R =



2-11-37 R =



2-11-34 R =



2-11-38 R =

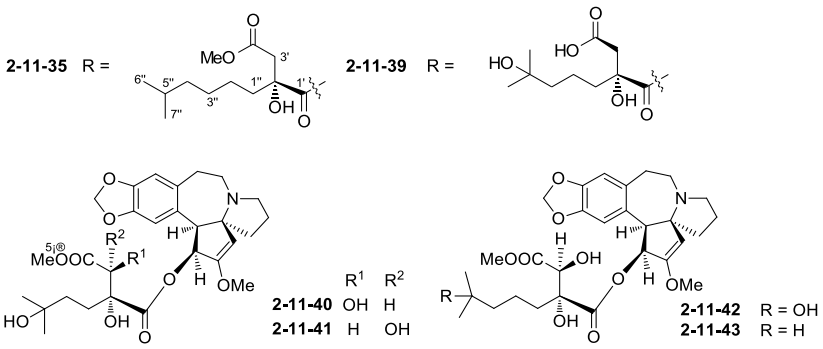


Table 2-11-11: ^{1}H NMR spectroscopic data of cephalotaxine-type alkaloids **2-11-28**~**2-11-31**.

H	2-11-28	2-11-29	2-11-30	2-11-31
1	1.93 m	2.01 m	*α* 2.83 d(18.2)	3.07 s
		2.39 m	*β* 3.80 d(18.2)	
2	4.29 d(4.2)	4.23 m		
3	4.23 d(6.6)	4.16 dd(5.6, 4.9)		
4	3.69 d(6.6)	3.48 d(5.6)		
6	1.93 m, 2.81 m	1.92 m, 2.39 m	*α* 1.82 m, *β* 2.02 m	2.28 m
7	1.93 m, 2.10 m	1.92 m, 2.11 m	*α* 1.96 m, *β* 2.00 m	2.41 m
8	3.19 m, 3.52 m	3.20 m, 3.54 m	*α* 3.20 m, *β* 3.69 m	3.57 m, 4.01 m
10	3.31 m	3.35 m, 3.41 m	*α* 3.17 m, *β* 3.67 m	3.52 m, 3.83 m
11	2.52 m, 4.21 m	2.58 m, 4.31 m	*α* 3.13 m, *β* 3.20 m	3.30 t(3.8)
14	6.82 s	6.81 s	7.32 s	7.26 s
17	6.80 s	6.80 s	6.71 s	6.98 s
18	5.94 br s	5.94 br s	6.00 s, 6.07 s	6.18 br s
1′				5.87 d(7.8)
2′				3.43 m
3′				3.52 m
4′				3.47 m
5′				3.39 m
6′				3.81 m, 4.08 m

Table 2-11-12: ^{1}H NMR spectroscopic data of cephalotaxine-type alkaloids **2-11-32**~**2-11-35**.

H	2-11-32	2-11-33	2-11-34	2-11-35
1	5.06 s	5.04 s	5.04 s	5.04 s
3	5.99 d(9.5)	5.97 d(9.8)	6.01 d(9.5)	5.99 d(9.5)
4	3.78 d(9.5)	3.78 d(9.8)	3.78 d(9.5)	3.78 d(9.5)
6*α*	2.05 m	2.03 dt(12.0, 10.0)	2.05 m	2.04 m
6*β*	1.95 m	1.90 ddd(12.0, 8.0, 4.0)	1.94 m	1.91 m

Table 2-11-12 (continued)

H	2-11-32	2-11-33	2-11-34	2-11-35
7	1.79 m	1.76 m	1.77 m	1.76 m
8α	3.15 m	3.10 m	3.10 m	3.10 m
8β	2.64 m	2.58 dd(9.0, 7.0)	2.59 m	2.60 m
10α	2.98 td(11.0, 7.0)	2.94 td(11.0, 7.0)	2.95 td(11.0, 7.0)	2.96 td(11.4, 7.0)
10β	2.64 m	2.60 dd(11.0, 8.0)	2.60 m	2.60 m
11α	2.40 dd(14.0, 6.8)	2.38 dd(14.0, 7.0)	2.38 dd(14.0, 7.0)	2.39 dd(13.9, 7.0)
11β	3.15 m	3.14 m	3.12 m	3.11 m
14	6.55 s	6.53 s	6.54 s	6.54 s
17	6.63 s	6.62 s	6.63 s	6.62 s
18	5.86 d(1.5)	5.85 d(1.6)	5.85 d(1.4)	5.86 d(1.2)
	5.88 d(1.5)	5.88 d(1.6)	5.87 d(1.4)	5.87 d(1.2)
19	3.69 s	3.67 s	3.67 s	3.67 s
3′	1.90 d(16.5)	1.83 d(16.3)	1.90 d(16.5)	1.93 d(16.5)
	2.28 d(16.5)	2.24 d(16.3)	2.26 d(16.5)	2.27 d(16.5)
5′	3.57 s	3.57 s	3.57 s	3.57 s
1″	1.41 m	1.34 dd(14.0, 6.0)	1.12 m	1.10~1.40 m
		1.36 dd(14.0, 6.0)	1.40 m	
2″	0.97 m, 1.28 m	1.65 m	1.38 m	1.10~1.40 m
3″	1.41 m	0.82 d(6.7)	1.20 m	1.10~1.40 m
4″	0.83 d(7.5)	0.91 d(6.7)	1.49 m	1.10~1.40 m
5″	0.84 d(7.5)		0.84 d(6.4)	1.50 m
6″			0.85 d(6.4)	0.85 d(6.9)
7″				0.87 d(6.9)

Table 2-11-13: ^{1}H NMR spectroscopic data of cephalotaxine-type alkaloids **2-11-36**~**2-11-39**.

H	2-11-36	2-11-37	2-11-38	2-11-39
1	5.13 s	5.17 s	5.20 s	5.14 s
3	5.77 d(9.7)	5.76 d(9.5)	5.76 d(9.5)	5.70 d(9.5)
4	3.80 d(9.7)	3.86 d(9.5)	3.88 d(9.5)	3.82 d(9.5)
6	α 1.82 m, β 1.81 m	α 1.92 m, β 1.90 m	α 1.92 m, β 1.91 m	α 1.87 m, β 1.85 m
7	α 1.69 m, β 1.57 m	α 1.76 m, β 1.63 m	α 1.78 m, β 1.64 m	α 1.70 m, β 1.58 m
8α	2.55 m	2.70 m	2.71 m	2.57 m
8β	2.83 ddd(8.8, 8.6, 3.9)	2.95 m	2.99 m	2.85 ddd(9.3, 9.1, 3.7)
10α	2.71 ddd(13.4, 11.6, 7.0)	2.81 m	2.83 m	2.73 ddd(12.9, 11.3, 7.0)
10β	2.54 m	2.69 m	2.70 m	2.57 m
11α	2.35 dd(14.2, 7.0)	2.38 m	2.40 dd(14.2, 7.0)	2.37 dd(13.4, 7.0)
11β	3.02 ddd(14.2, 13.4, 7.0)	3.15 m	3.14 m	3.01 ddd(13.4, 12.9, 7.0)
14	6.53 s	6.57 s	6.59 s	6.54 s

Table 2-11-13 (continued)

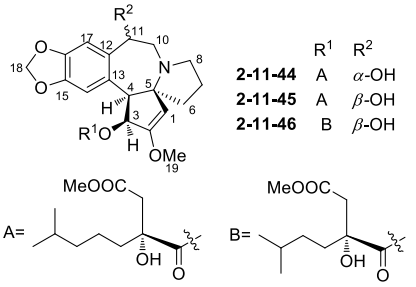
H	2-11-36	2-11-37	2-11-38	2-11-39
17	6.66 s	6.66 s	6.67 s	6.65 s
18	5.92 d(1.4), 5.76 d(1.4)	5.89 s, 5.79 s	5.89 s, 5.80 s	5.92 d(1.1), 5.78 d(1.1)
19	3.58 s	3.62 s	3.63 s	3.59 s
3′	1.88 d(15.3), 1.61 d(15.3)	3.06 s	3.12 s	1.89 d(15.8), 1.67 d(15.8)
1″	1.45 ddd(12.4, 12.1, 8.7) 1.09 dd(12.4, 8.7)	1.39 m, 1.51 m	1.43 ddd(13.4, 13.1, 4.3) 1.27 m	1.26 m
2″	1.35 m	1.24 m, 0.99 m	1.04 m, 0.71 m	1.02 m, 0.96 m
3″				1.18 m
4″	0.96 s	0.96 s	0.76 d(6.7)	
5″	1.01 s	1.00 s	0.78 d(6.7)	1.01 s
6″				1.02 s

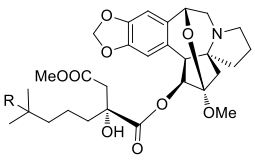
Table 2-11-14: ^{1}H NMR spectroscopic data of cephalotaxine-type alkaloids **2-11-40**~**2-11-43**.

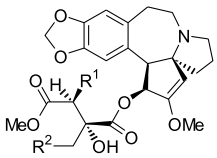
H	2-11-40	2-11-41	2-11-42	2-11-43
1	5.10 s	5.09 s	5.08 s	5.07 s
3	6.00 d(9.7)	6.04 d(9.8)	5.99 d(9.7)	5.99 d(9.8)
4	3.82 d(9.7)	3.79 d(9.8)	3.80 d(9.7)	3.80 d(9.8)
6	1.93 br m, 2.06 br m	1.94 br m, 2.06 br m	1.90 br m, 2.04 m	1.90 br m, 2.03 m
7	1.78 br m	1.79 br m	1.77 br m	1.76 br m
8	2.62 br m, 3.14 br m	2.66 br m, 3.14 br m	2.61 br m, 3.09 m	2.60 br m, 3.10 m
10	2.62 br m, 2.98 br m	2.66 br m, 2.98 br m	2.61 br m, 2.96 m	2.62 br m, 2.97 m
11	2.44 br m	2.43 dd(6.6, 14.2)	2.42 dd(6.9, 14.2)	2.42 dd(6.7, 14.3)
	3.14 br m	3.14 br m	3.13 m	3.14 br m
14	6.61 s	6.55 s	6.61 s	6.61 s
17	6.64 s	6.65 s	6.65 s	6.65 s
18	5.85 s, 5.86 s	5.85 s, 5.81 s	5.84 s, 5.86 s	5.84 s, 5.87 s
19	3.71 s	3.70 s	3.68 s	3.67 s
3′	3.48br s	3.39 s	3.41br s	3.40br s
5′	3.76 s	3.61 s	3.74 s	3.74 s
1″	1.19 m	1.28 m	1.51 m	1.47 m
	1.55 dt(5.1, 13.0)	1.48 m	1.57 m	1.52 m
2″	1.67 dt(5.1, 13.0)	1.62 m	1.18 m	1.34 m
	1.77 m	1.99 m	1.41 m	1.10 m
3″			1.41 m	1.10 m
4″	1.14 s	1.16 s		1.45 m
5″	1.17 s	1.17 s	1.18 s	0.84 d(6.5)
6″			1.19 s	0.85 d(6.5)

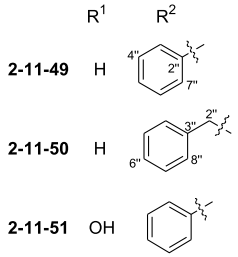
Table 2-11-15: Cos, MFs, and TSs of cephalotaxine-type alkaloids **2-11-44**~**2-11-56**.

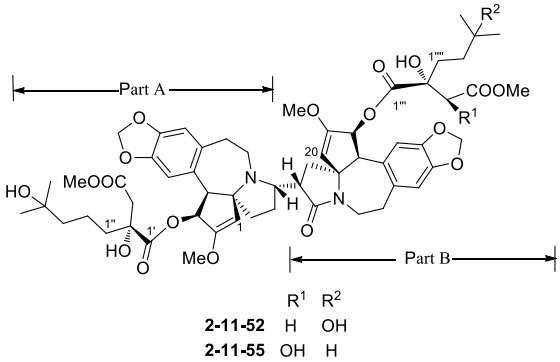
No.	Compounds	MFs	Test solvents	References
2-11-44	11α-hydroxyhomodeoxyharringtonine	$C_{29}H_{39}NO_9$	$CDCl_3$	[336]
2-11-45	11β-hydroxyhomodeoxyharringtonine	$C_{29}H_{39}NO_9$	$CDCl_3$	[336]
2-11-46	11β-hydroxydeoxyharringtonine	$C_{28}H_{37}NO_9$	$CDCl_3$	[336]
2-11-47	cephalezomine A	$C_{29}H_{39}NO_{10}$	$CDCl_3$	[335]
2-11-48	cephalezomine B	$C_{29}H_{39}NO_9$	$CDCl_3$	[335]
2-11-49	neoharringtonine	$C_{30}H_{33}NO_8$	$CDCl_3$	[337]
2-11-50	homoneoharringtonine	$C_{31}H_{35}NO_8$	$CDCl_3$	[337]
2-11-51	3′*S*-hydroxyneoharringtonine	$C_{30}H_{33}NO_9$	$CDCl_3$	[337]
2-11-52	bis-cephalezomine A	$C_{57}H_{72}N_2O_{19}$	$CDCl_3$	[338]
2-11-53	bis-cephalezomine B	$C_{57}H_{72}N_2O_{19}$	$CDCl_3$	[338]
2-11-54	bis-cephalezomine C	$C_{56}H_{70}N_2O_{19}$	$CDCl_3$	[338]
2-11-55	bis-cephalezomine D	$C_{57}H_{72}N_2O_{19}$	$CDCl_3$	[338]
2-11-56	bis-cephalezomine E	$C_{57}H_{72}N_2O_{18}$	$CDCl_3$	[338]

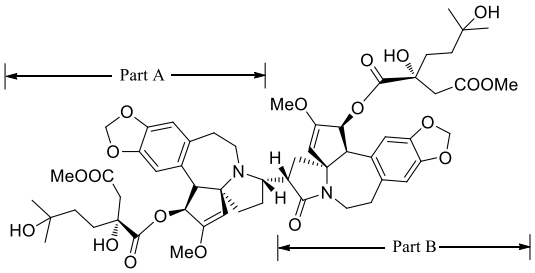




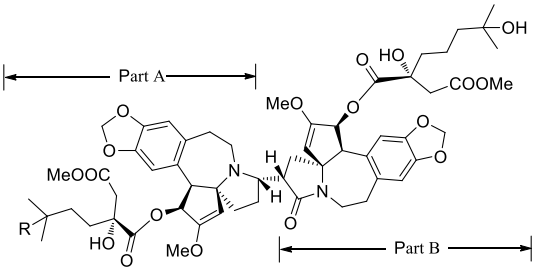








2-11-54



2-11-53 R = OH
2-11-56 R = H

Table 2-11-16: ^{1}H NMR spectroscopic data of cephalotaxine-type alkaloids **2-11-44**~**2-11-46**.

H	2-11-44	2-11-45	2-11-46
1	5.02 s	4.75 s	4.75 s
3	5.95 d(9.8)	5.77 d(8.1)	5.78 d(8.1)
4	3.79 d(9.8)	3.57 d(8.1)	3.57 d(8.1)
6	*α* 2.01 m, *β* 1.90 m	*α* 1.86 m, *β* 2.03 m	*α* 1.87 m, *β* 2.03 m
7	1.76 m	*α* 1.76 m, *β* 1.68 m	*α* 1.77 m, *β* 1.68 m
8	*α* 3.07 m, *β* 2.64 m	*α* 2.86 m, *β* 2.90 m	*α* 2.87 m, *β* 2.91 m
10*α*	2.64 dd(10.4, 10.2)	3.24 dd(14.5, 7.5)	3.25 dd(14.5, 7.5)
10*β*	2.91 dd(10.4, 7.0)	3.32 dd(14.5, 10.5)	3.32 dd(14.5, 10.5)
11	5.22 br dd(10.2, 7.0)	*α* 4.88 ddd(10.8, 10.5, 7.5)	*α* 4.88 ddd(11.5, 10.5, 7.5)
	2.03 br s(OH)	4.20 br d(10.8, OH)	4.16 br d(11.5, OH)
14	6.55 s	6.51 s	6.52 s
17	7.12 s	7.08 s	7.07 s
18	5.90 br d(1.4)	5.87 d(1.5), 5.91 d(1.5)	5.92 d(1.5), 5.86 d(1.5)
19	3.69 s	3.68 s	3.68 s
3′	2.31 d(16.7), 2.15 d(16.7)	2.53 d(16.5), 2.92 d(16.5)	2.53 d(16.5), 2.91 d(16.5)
5′	3.56 s	3.68 s	3.68 s
1″	1.29 m	0.85~0.95 m	1.21 m
2″	1.08 m	0.85~0.95 m	0.73 m, 0.87 m
3″	1.29 m	0.85~0.95 m	1.27 m
4″	1.48 m	1.36 m	0.75 d(6.5)
5″	0.84 d(6.5)	0.81 d(6.5)	0.75 d(6.5)
6″	0.84 d(6.5)	0.79 d(6.5)	

Table 2-11-17: ^{1}H NMR spectroscopic data of cephalotaxine-type alkaloids **2-11-47** and **2-11-48**.

H	2-11-47	2-11-48	H	2-11-47	2-11-48
1	1.55 d(14.0)	1.53 d(14.0)	7	1.77 m	1.76 m
	2.67 d(14.0)	2.66 d(14.0)			
3	5.23 d(9.5)	5.16 d(9.4)	8	3.05 m	3.05 m
				2.41 dd(8.6, 17.5)	2.40 dd(8.6, 17.5)
4	3.56 d(9.5)	3.57 d(9.4)	10	3.11 dd(4.9, 13.1)	3.10 dd(4.9, 12.9)
				2.97 d(13.1)	2.97 d(12.9)
6	2.19 m, 2.04 m	2.19 m, 2.03 m	11	4.86 d(4.9)	4.85 d(4.9)
14	6.45 s	6.44 s	1″	1.45 m	1.40 m
17	6.78 s	6.64 s	2″	1.45 m	1.31 m
				1.15 m	1.08 m
18	5.87 s	5.78 s	3″	1.37 m	1.07 m
	5.91 s	5.91 s			
19	3.41 s	3.39 s	4″		1.47 m
3′	1.96 d(16.5)	1.97 d(16.5)	5″	1.18 s	0.83 d(6.5)
	2.29 d(16.5)	2.30 d(16.5)			
5′	3.67 s	3.66 s	6″	1.17 s	0.82 d(6.5)

Table 2-11-18: ^{1}H NMR spectroscopic data of cephalotaxine-type alkaloids **2-11-49**~**2-11-51**.

H	2-11-49	2-11-50	2-11-51
1	5.07 s	5.09 s	5.11 s
3	5.86 d(9.8)	6.07 d(9.8)	5.93 d(9.8)
4	3.77 d(9.8)	3.81 d(9.8)	3.77 d(9.8)
6α	2.01 td(12.0, 9.5)	2.05 td(12.0, 9.7)	2.03 td(12.5, 9.8)
6β	1.89 ddd(12.0, 8.0, 4.0)	1.93 ddd(12.4, 8.3, 4.4)	1.90 ddd(12.0, 8.0, 4.2)
7	1.74 m	1.75 m	1.77 m
8α	3.09 td(12.0, 7.0)	3.12 td(8.5, 5.0)	3.12 m
8β	2.59 m	2.62 m	2.61 m
10α	2.93 td(12.0, 7.0)	2.95 td(12.0, 7.0)	2.97 td(12.0, 7.0)
10β	2.59 m	2.62 m	2.61 m
11α	2.40 dd(14.0, 7.0)	2.37 m	2.41dd(14.0, 7.0)
11β	3.17 ddd(14.0, 12.0, 8.0)	3.13 m	3.12 m
14	6.52 s	6.56 s	6.51 s
17	6.64 s	6.59 s	6.68 s
18	5.80 d(1.5), 5.86 d(1.5)	5.73 d(1.5), 5.84 d(1.5)	5.81 d(1.5), 5.88 d(1.5)
19	3.71 s	3.70 s	3.74 s
3′	1.90 d(16.5), 2.23 d(16.5)	1.97 d(16.5), 2.30 d(16.5)	3.34 br d(7.0)
5′	3.55 s	3.59 s	3.64 s
1″	2.70 d(13.5), 2.71 d(13.5)	1.75 m	2.84 d(13.9), 3.17 d(13.9)
2″		2.37 m, 2.62 m	

Table 2-11-18 (continued)

H	2-11-49	2-11-50	2-11-51
3″	7.14 m		7.21 m
4″	7.21 m	7.11 br d(8.0)	7.21 m
5″	7.23 m	7.27 br t(8.0)	7.21 m
6″	7.21 m	7.18 br t(7.5)	7.21 m
7″	7.14 m	7.27 br t(8.0)	7.21 m
8″		7.11 br d(8.0)	

Table 2-11-19(a): ^{1}H NMR spectroscopic data of cephalotaxine-type alkaloids **2-11-52**~**2-11-54**.

H	2-11-52		2-11-53		2-11-54	
	Part A	Part B	Part A	Part B	Part A	Part B
1(20)	4.96 s	4.69 s	4.97 s	4.68 s	4.95 s	4.70 s
3(22)	5.95 d(10.0)	5.89 d(9.2)	5.94 d(10.1)	5.90 d(9.4)	5.96 d(10.0)	5.91 d(10.6)
4(23)	3.68 m	3.52 m	3.71 d(10.1)	3.51 m	3.71 d(10.0)	3.51 d(10.6)
6(25)	1.70 m	1.46 m, 1.60 m	1.70 m	1.46 m, 1.60 m	1.72 m	1.56 m, 1.62 m
7(26)	0.79 m, 1.54 m	2.68 m	0.79 m, 1.53 m	2.68 m	0.88 m, 1.60 m	2.61 m
8(27)	3.29 m		3.29 m		3.31 m	
10(29)	2.60 m, 2.68 m	2.83 m, 3.04 m	2.59 m, 2.68 m	3.05 m, 3.83 m	2.61 m, 2.70 m	3.84 ddd(12.7, 12.7, 6.6) 3.10 m
11(30)	2.31 m, 3.06 m	2.54 m, 3.17 m	2.33 m, 3.05 m	2.54 m, 3.17 m	2.34 m, 3.07 m	2.56 ddd(12.2, 6.6, 2.5) 3.18 ddd(12.7, 12.2, 6.6)
14(33)	6.54 s	6.51 s	6.54 s	6.51 s	6.56 s	6.53 s
17(36)	6.56 s	6.56 s	6.56 s	6.56 s	6.57 s	6.57 s
18(37)	5.86 d(1.3) 5.97 d(1.3)	5.86 d(1.4) 5.89 d(1.4)	5.86 d(1.3) 5.97 d(1.3)	5.86 d(1.4) 5.88 d(1.4)	5.88 d(1.1) 6.00 d(1.2)	5.88 d(1.1) 5.90 d(1.3)
19(38)	3.66 s	3.62 s	3.65 s	3.63 s	3.71 s	3.67 s
3′(3‴)	1.90 d(16.4) 2.24 d(16.4)	2.05 d(16.4) 2.34 d(16.4)	1.89 d(16.4) 2.26 d(16.4)	2.04 d(16.5) 2.31 d(16.5)	1.91 d(16.5) 2.28 d(16.5)	2.07 d(16.5) 2.35 d(16.5)
5′(5‴)	3.55 s	3.56 s	3.53 s	3.57 s	3.56 s	3.59 s
1″(1⁗)	1.38 m	1.57 m	1.56 m	1.38 m	1.58 m	1.58 m
2″(2⁗)	1.17 m, 1.37 m	1.52 m	1.56 m	1.11 m, 1.35 m	1.60 m	1.60 m
3″(3⁗)	1.38 m			1.32 m		
4″(4⁗)		1.13 s	1.14 s		1.15 s	1.16 s
5″(5⁗)	1.16 s	1.12 s	1.12 s	1.15 s	1.14 s	1.14 s
6″(6⁗)	1.16 s			1.15 s		

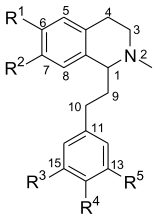
Table 2-11-19(b): ^{1}H NMR spectroscopic data of cephalotaxine-type alkaloids **2-11-55** and **2-11-56**.

H	2-11-55		2-11-56	
	Part A	Part B	Part A	Part B
1(20)	4.97 s	4.71 s	4.95 s	4.68 s
3(22)	5.96 d(9.8)	5.93 d(9.3)	5.95 d(9.8)	5.92 d(9.5)
4(23)	3.69 d(10.0)	3.53 m	3.69 m	3.52 m
6(25)	1.70 m	1.50 m, 1.57 m	1.71 m	1.49 m, 1.60 m
7(26)	0.82 m, 1.55 m	2.70 m	0.81 m, 1.56 m	2.69 m
8(27)	3.32 m		3.30 m	
10(29)	2.60 m, 2.70 m	3.07 m, 3.86 m	2.69 m	3.84 ddd
			2.60 m	(12.6, 12.6, 6.6)
				3.06 m
11(30)	2.33 m, 3.07 m	2.63 m, 3.19 m	2.32 m, 3.09 m	2.55 m, 3.18 m
14(33)	6.55 s	6.52 s	6.54 s	6.52 s
17(36)	6.60 s	6.58 s	6.57 s	6.57 s
18(37)	5.88 s	5.83 s	5.88 d(1.2)	5.86 s
	5.99 s	5.90 s	5.99 d(1.2)	5.90 d(1.1)
19(38)	3.67 s	3.64 s	3.66 s	3.63 s
3′(3‴)	1.92 d(16.4)	3.48 d(8.2)	1.89 d(16.4)	2.06 d(16.4)
	2.26 d(16.4)		2.26 d(16.4)	2.30 d(16.4)
5′(5‴)	3.58 s	3.57 s	3.54 s	3.57 s
1″(1⁗)	1.42 m	1.49 m	1.41 m	1.41 m
		1.85 ddd(13.4, 13.4, 4.0)		
2″(2⁗)	1.14 m, 1.37 m	0.88 m, 1.21 m	0.95 m	1.13 m, 1.41 m
3″(3⁗)	1.37 m	1.37 m	1.27 m	1.35 m
4″(4⁗)		0.84 d(6.6)	0.73 d(6.6)	
5″(5⁗)	1.18 s	0.82 d(6.6)	0.75 d(6.6)	1.16 s
6″(6⁗)	1.18 s			1.17 s

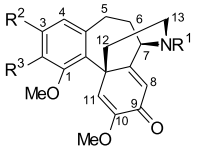
2.11.4 1-phenylethyl-tetrahydroisoquinoline-type alkaloids

Table 2-11-20: Cos, MFs, and TSs of 1-phenylethyl-tetrahydroisoquinoline-type alkaloids **2-11-57**~**2-11-63**.

No.	Compounds	MFs	Test solvents	References
2-11-57	(+)-colchiethanamine	$C_{19}H_{23}NO_3$	$CDCl_3$	[339]
2-11-58	(+)-colchiethine	$C_{20}H_{25}NO_3$	$CDCl_3$	[339]
2-11-59	1,2,3,4-tetrahydro-7-hydroxy-1-(4′-methoxyphenethyl)-6-methoxy-2-methylisoquinoline	$C_{20}H_{25}NO_3$	$CDCl_3$	[339]
2-11-60	(–)-autumnaline	$C_{21}H_{27}NO_5$	$CDCl_3$	[319]
2-11-61	(–)-isoautumnaline	$C_{21}H_{27}NO_5$	$CDCl_3$	[319]
2-11-62	(+)-androcymbine	$C_{21}H_{25}NO_5$	$CDCl_3$	[319]
2-11-63	(+)-colchiritchine	$C_{20}H_{21}NO_5$	$CDCl_3$	[319]



	R^1	R^2	R^3	R^4	R^5
2-11-57	OH	OMe	H	OH	H
2-11-58	OH	OMe	H	OMe	H
2-11-59	OMe	OH	H	OMe	H
2-11-60	OMe	OH	OMe	OMe	OH
2-11-61	OH	OMe	OMe	OMe	OH



	R^1	R^2	R^3
2-11-62	Me	OH	OMe
2-11-63	H	OCH_2O	

Table 2-11-21: ^{1}H NMR spectroscopic data of 1-phenylethyl-tetrahydroisoquinoline-type alkaloids **2-11-57**~**2-11-61**.

H	2-11-57	2-11-58	2-11-59	2-11-60	2-11-61
1	3.47 t(5.2)	3.48 t(5.1)	3.59 t(5.4)	3.45 m	3.41 m
3	3.20 m, 2.76 m	3.19 m, 2.77 m	3.31 m, 2.92 m	3.17 m, 2.70 m	2.64 m, 3.13 m
4	2.76 m, 2.65 m	2.77 m, 2.68 m	2.83 m, 2.76 m	2.70 m	2.68 m
5	6.63	6.65	6.58	6.56 s	6.63
6			3.86 s(OMe)	3.87 s(OMe)	
7	3.83 s(OMe)	3.84 s(OMe)			3.84 s(OMe)
8	6.49	6.49	6.64	6.67 s	6.52
9	2.49 m, 2.01 m	2.10 m, 2.01 m	2.23 m, 2.02 m	2.04 m	2.03 m
10	2.54 m, 2.65 m	2.57 m, 2.68 m	2.83 m, 2.63 m	2.43 m, 2.70 m	2.68 m, 2.52 m
12	7.01 d(8.2)	7.12 d(8.2)	7.14 d(8.2)	6.45 d(2)	6.44 d(2)
13	6.72 d(8.2)	6.83 d(8.2)	6.81 d(8.2)		
14				3.86 s(OMe)	3.86 s(OMe)
15	6.72 d(8.2)	6.83 d(8.2)	6.81 d(8.2)	3.84 s(OMe)	3.83 s(OMe)
16	7.01 d(8.2)	7.12 d(8.2)	7.14 d(8.2)	6.32 d(2)	6.29 d(2)
NMe	2.48 s	2.58 s	2.58 s	2.49 s	2.48 s

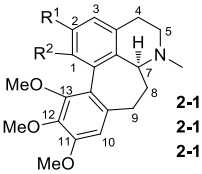
Table 2-11-22: ^{1}H NMR spectroscopic data of 1-phenylethyl-tetrahydroisoquinoline-type alkaloids **2-11-62** and **2-11-63**.

H	2-11-62	2-11-63	H	2-11-62	2-11-63
1	4.00 s(OMe)	4.08 s(OMe)	10	3.64 s(OMe)	3.67 s(OMe)
2	3.85 s(OMe)		11	6.80	6.81
4	6.42	6.27	12	α 2.92, β 1.70	α 3.27, β 1.40
5	α 2.88, β 2.36	α 2.82, β 2.34	13	α 2.78, β 2.58	α 2.85, β 2.85
6	α 2.14, β 1.79	α 2.42, β 1.70	NMe	2.39	
7	3.91	4.11 m	OCH_2O		5.93 d(1.4)
					5.97 d(1.4)
8	6.30	6.31			

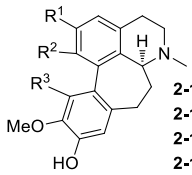
2.11.5 Homoaporphine-type alkaloids

Table 2-11-23: Cos, MFs, and TSs of homoaporphine-type alkaloids **2-11-64**~**2-11-70**.

No.	Compounds	MFs	Test solvents	References
2-11-64	(+)-*O*-methylkreysigine	$C_{23}H_{29}NO_5$	$CDCl_3$	[340]
2-11-65	(+)-kreysigine	$C_{22}H_{27}NO_5$	$CDCl_3$	[340]
2-11-66	(+)-androcine	$C_{22}H_{27}NO_5$	$CDCl_3$	[340]
2-11-67	(+)-androcimine	$C_{22}H_{27}NO_5$	$CDCl_3$	[340]
2-11-68	(+)-androbine	$C_{21}H_{25}NO_5$	$CDCl_3$	[340]
2-11-69	baytopine	$C_{21}H_{25}NO_5$	$CDCl_3$	[341]
2-11-70	bechuanine	$C_{21}H_{25}NO_5$	–	[342]



	R^1	R^2
2-11-64	OMe	OMe
2-11-65	OMe	OH
2-11-66	OH	OMe



	R^1	R^2	R^3
2-11-67	OMe	OMe	OMe
2-11-68	OH	OMe	OMe
2-11-69	OMe	OMe	OH
2-11-70	OMe	OH	OMe

Table 2-11-24: ^{1}H NMR spectroscopic data of homoaporphine-type alkaloids **2-11-64**~**2-11-67**.

H	2-11-64	2-11-65	2-11-66	2-11-67
1	3.54 s(OMe)		3.31 s(OMe)	3.51 s(OMe)
2	3.88 s(OMe)	3.92 s(OMe)		3.88 s(OMe)
3	6.70	6.68	6.74	6.69
4	*α* 3.03 m, *β* 2.67 m	*α* 3.09 m, *β* 2.78 m	*α* 2.99 m, *β* 2.60 m	*α* 3.03 m, *β* 2.68 m
5	*α* 2.85 m, *β* 3.19 m	*α* 3.02 m, *β* 3.31 m	*α* 2.80 m, *β* 3.13 m	*α* 2.85 m, *β* 3.16 m
7	3.32 dd(6.4, 11.0)	3.49 dd(5.8, 11.2)	3.29 m	3.37 dd(5.9, 10.3)
8	*α* 2.28 m, *β* 1.99 m	*α* 2.30 m, *β* 2.09 m	*α* 2.20 m, *β* 2.03 m	*α* 2.22 m, *β* 1.98 m
9	*α* 2.45 m, *β* 2.28 m	*α* 2.53 m, *β* 2.30 m	*α* 2.51 m, *β* 2.33 m	*α* 2.45 m, *β* 2.45 m
10	6.56	6.63	6.60	6.60
11	3.91 s(OMe)	3.92 s(OMe)	3.92 s(OMe)	
12	3.90 s(OMe)	3.92 s(OMe)	3.91 s(OMe)	3.94 s(OMe)
13	3.66 s(OMe)	3.69 s(OMe)	3.56 s(OMe)	3.59 s(OMe)
NMe	2.45	2.53	2.41	2.45

Table 2-11-25: ^{1}H NMR spectroscopic data of homoaporphine-type alkaloids **2-11-68**~**2-11-70**.

H	2-11-68	2-11-69	2-11-70
1	3.32 s(OMe)	3.64 s(OMe)	
2		3.92 s(OMe)	3.90 s(OMe)

Table 2-11-25 (continued)

H	2-11-68	2-11-69	2-11-70
3	6.77	6.68 s	6.66 s
4	α 3.04 m, β 2.74 m	3.15 m, 3.35 m	3.02 m, 3.14 m
5	α 2.91 m, β 3.12 m	2.83 m, 3.11 m	2.63 m, 2.79 m
7	3.44 m	3.63 dd(11.7, 6.3)	3.34 dd(11.1, 6.8)
8	α 2.28 m, β 2.03 m	2.07 m, 2.39 m	2.00 m, 2.24 m
9	α 2.50 m, β 2.50 m	2.26 m, 2.48 m	2.11 m, 2.42 m
10	6.63	6.65 s	6.64 s
12	3.97 s(OMe)	3.95 s(OMe)	3.95 s(OMe)
13	3.53 s(OMe)		3.59 s(OMe)
NMe	2.50	2.59 s	2.40 s

Bibliography

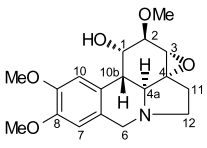
[319] Freyer AJ, Zarga MHA, Firdous S, et al. J Nat Prod, 1987, 50: 684.
[320] Chaudhuri PK, Thakur RS. J Nat Prod, 1993, 56: 1174.
[321] Alali FQ, El-Elimat T, Chen L, et al. J Nat Prod, 2005, 68: 173.
[322] He HP, Hu L, Liu FC. Chem Res Appl, 1999;11(5):509.
[323] He HP, Liu FC, Hu L, et al. Acta Bot Yunnanica, 1999;21(3):364.
[324] Meksuriyen D, Lin LJ, Cordell GA. J Nat Prod, 1988, 51: 88.
[325] Al-Tel TH, Zarga MHA, Sabri SS, et al. Phytochemistry, 1991, 30: 3081.
[326] Zarga MHA, Sabri SS, Al-Tel TH, et al. J Nat Prod, 1991, 54: 936.
[327] Aladesanmi AJ, Hoffmann JJ. Phytochemistry, 1994, 35: 1361.
[328] Morita H, Yoshinaga M, Kobayashi J. Tetrahedron, 2002, 58: 5489.
[329] Wang LW, Su HJ, Yang SZ, et al. J Nat Prod, 2004, 67: 1182.
[330] Langiois N. Heterocycles, 1990, 30: 659.
[331] Pusset J, Barre SL, Langlois N, et al. Phytochemistry, 1989, 28: 1298.
[332] Aladesanmi AJ, Adewunmi CO, Kelley CJ, et al. Phytochemistry, 1988, 27: 3789.
[333] Takano I, Yasuda I, Nishijima M, et al. J Nat Prod, 1996, 59: 965.
[334] Takano I, Yasuda I, Nishijima M, et al. Phytochemistry, 1996, 43: 299.
[335] Morita H, Arisaka M, Yoshida N, et al. Tetrahedron, 2000, 56: 2929.
[336] Takano I, Yasuda I, Nishijima M, et al. J Nat Prod, 1996, 59: 1192.
[337] Takano I, Yasuda I, Nishijima M, et al. Phytochemistry, 1997, 44: 735.
[338] Yoshiaga M, Morita H, Dota T, et al. Tetrahedron, 2004, 60: 7861.
[339] Tojo E, Önür MA, Freyer AJ, et al. J Nat Prod, 1990, 53: 634.
[340] Tojo E, Zarga MHA, Sabri SS, et al. J Nat Prod, 1989, 52: 1055.
[341] Husek A, Sütlüpinar N, Potěšilová H, et al. Phytochemistry, 1989, 28: 3217.
[342] Husek A, Sütlüpinar N, Sedmera P, et al. Heterocycles, 1999, 28: 79.

2.12 Benzylated phenethylamine alkaloids

2.12.1 Lycorine-type benzylated phenethylamine alkaloids

Table 2-12-1: Cos, MFs, and TSs of lycorine-type alkaloids **2-12-1**~**2-12-11**.

No.	Compounds	MFs	Test solvents	References
2-12-1	incartine	$C_{18}H_{23}NO_5$	$CDCl_3$	[343]
2-12-2	narcissidine	$C_{18}H_{23}NO_5$	$CDCl_3$	[344]
2-12-3	3-*O*-acetylnarcissidine	$C_{20}H_{25}NO_6$	$CDCl_3$	[344]
2-12-4	2-*O*-(3′-acetoxyhydroxybutanoyl)lycorine	$C_{22}H_{25}NO_7$	$CDCl_3$	[345]
2-12-5	galanthine	$C_{18}H_{23}NO_4$	$CDCl_3$	[346]
2-12-6	(–)-lycorine	$C_{16}H_{17}NO_4$	DMSO-d_6	[347]
2-12-7	(+)-kirkine	$C_{16}H_{19}NO_3$	CD_3OD	[348]
2-12-8	1-*O*-acetylnorpluviine	$C_{18}H_{21}NO_4$	$CDCl_3$	[349]
2-12-9	(–)-amarbellisine	$C_{17}H_{19}NO_4$	$CDCl_3$	[350]
2-12-10	1,2-di-*O*-acetyllycorine	$C_{20}H_{21}NO_6$	$CDCl_3$	[351]
2-12-11	2-*O*-acetyllycorine	$C_{18}H_{19}NO_5$	$CDCl_3$	[352]



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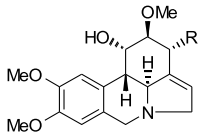

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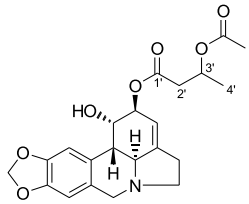
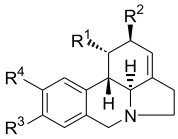
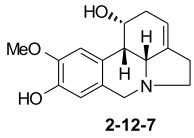
2-12-3 R = OAc



2-12-4



	R^1	R^2	R^3	R^4
2-12-5	OH	OMe	OMe	OMe
2-12-6	OH	OH	OCH_2O	
2-12-8	OAc	H	OH	OMe
2-12-11	OH	OAc	OCH_2O	



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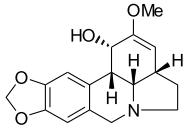
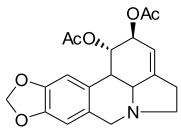


2-12-9



2-12-10

Table 2-12-2: ^{1}H NMR spectroscopic data of lycorine-type alkaloids **2-12-1**~**2-12-3**.

H	2-12-1	2-12-2	2-12-3
1	4.55 br s(2.9, 3.0, 0.8)	4.66 m	4.70 br s
2	3.91 br s(2.9, 1.2)	4.18~3.36 m	4.30~3.50 m

Table 2-12-2 (continued)

H	2-12-1	2-12-2	2-12-3
2-OMe	3.57 s	3.44 s	3.50 s
3	3.44 br s(1.2, 0.8)	4.66 m	5.90 br s
3-OAc			2.03 s
4a	3.52 d(13.4)	4.18~3.36 m	4.30~3.50 m
6	*α* 4.31 d(13.0), *β* 4.71 d(13.0)	*α* 3.54 d(13.0), *β* 4.09 d(13.0)	*α* 3.66 d(13.0), *β* 4.19 d(13.0)
7	6.97 s	6.68 s	6.74 s
8-OMe	3.90 s	3.82 s	3.85 s
9-OMe	3.92 s	3.86 s	3.88 s
10	6.95 s	6.88 s	6.94 s
10b	2.60 dd(13.4, 3.0)	2.70 d(11.0)	2.85 d(11.0)
11	*α* 2.04 dd(13.1, 5.3) *β* 3.38 ddd(13.1, 14.2, 7.2)	5.56 br s	5.71 br s
12	*α* 3.76 ddd(10.8, 14.2, 5.3) *β* 4.08 dd(10.8, 7.2)	4.18~3.36 m	4.30~3.50 m

Table 2-12-3: ^{1}H NMR spectroscopic data of lycorine-type alkaloids **2-12-4**~**2-12-7**.

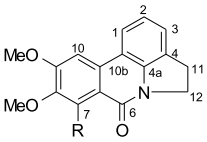
H	2-12-4	2-12-5	2-12-6	2-12-7
1	4.51 s	4.55 s	4.27 br s 4.79 br d(2.9, OH)	4.37 q(3)
2	5.31 dt(3.0, 1.5)	3.72 m	3.97 br s 4.90 br s(OH)	*α* 2.46 ddd(16.5, 7.5, 3) *β* 2.34 dddd(16.5, 3, 2.5, 2.5)
3	5.44 br t(2.5)	5.55 br s	5.37 br s	5.87 br dt (7.5, 2.5)
4a	2.82 br d(10.5)	2.65 s	2.60 d(10.6)	4.24 br d(8)
6	*α* 3.53 dd(14.0, 1.0) *β* 4.19 d(14.0)	*α* 3.40 br d(14.0) *β* 4.05 d(14.0)	*α* 3.32 d(14.4) *β* 4.02 d(14.4)	*α* 4.40 d(14) *β* 4.61 dt(14, 1)
7	6.58 s	6.52 s	6.68 s	6.60 s
9				3.87 s(OMe)
10	6.79 s	6.78 s	6.81 s	7.08 s
10b	2.68 br d(10.5)	2.65 s	2.50 m	3.52 dd(8, 3)
11	2.63 m	2.45~2.60 m	2.44 m	*α* 2.87 ddd(16, 8.5, 2.5) *β* 2.81 dddd(16, 10.5, 3, 2.5)
12	*α* 2.40 dd(9.0, 8.5) *β* 3.35 ddd (9.5, 9.2, 5.0)	*α* 2.25 dd(8.2, 16.4) *β* 3.25 ddd (6.0, 10.0, 16.4)	*α* 2.19 ddd (14.4, 8.6, 1.5) *β* 3.19 dd(14.4, 7.5)	*α* 3.71 ddd(11, 8.5, 3) *β* 3.83 ddd(11, 10.5, 2.5)
2′	2.51 dd(15.5, 5.5) 2.63 dd(15.5, 7.5)			
3′	5.25 m			
4′	1.29 d(6.0)			
OMe		3.78 s, 3.74 s, 3.40 s		
OAc	2.00 s			
OCH_2O	5.90~5.92 d(1.5)		5.94 s, 5.96 s	

Table 2-12-4: ^{1}H NMR spectroscopic data of lycorine-type alkaloids **2-12-8**~**2-12-11**.

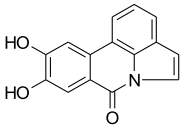
H	2-12-8	2-12-9	2-12-10	2-12-11
1	5.97 dd(5.9, 1.0) 1.90 s(OAc)	3.48 br s(2.5, 2.3, 1.8)	5.72 br s	4.52 s
2	*α* 2.33 m *β* 2.62 m	3.43 s(OMe)	5.24 br s	5.32 br s 2.08 s(OAc)
3	5.39 d(2.2)	5.56 br s(2.3, 2.3)	5.51 br s	5.47 br s
4		3.41 br ddd(11.8, 5.4, 2.3)		
4a	2.76 d(9.9)	4.08 br s(3.7, 3.4, 2.5)	2.76 d(10.7)	2.80 d(10.4)
6	*α* 3.50 d(14.5) *β* 4.13 d(14.5)	4.33 d(16.7) 3.79 d(16.7)	*α* 3.52 d(14.1) *β* 4.15 d(14.1)	3.53 d(14.0) 4.15 d(14.0)
7	6.64 s	6.45 s	5.65 s	6.59 s
9	3.83 s(OMe)			
10	6.74 s	6.54 s	6.74 s	6.81 s
10b	2.66 d(9.9)	3.28 br s(1.8)	2.86 br d(10.7)	2.71 d(10.4)
11	2.59 m	2.14 ddd(12.9, 5.4, 3.4) 1.56 ddd(12.9, 11.8, 3.7)	2.64 m	2.65 m
12	*α* 2.37 m *β* 3.34 ddd (11.3, 8.4, 4.5)	3.07 dd(11.2, 2.2) 3.02 d(11.2)	*α* 2.39 dd(15.1, 9.0) *β* 3.36 ddd (15.1, 8.6, 2.2)	2.39 br q(8.9) 3.36 ddd (8.9, 4.6, 4.6)
OAc			1.94 s, 2.07 s	
OCH_2O		5.88 d(1.1) 5.86 d(1.1)	5.90 s	5.91 d(1.2) 5.94 d(1.2)

Table 2-12-5: Cos, MFs, and TSs of lycorine-type alkaloids **2-12-12**~**2-12-24**.

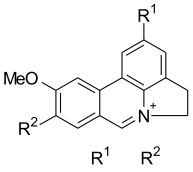
No.	Compounds	MFs	Test solvents	References
2-12-12	7-methoxyoxoassoanine	$C_{18}H_{17}NO_4$	CD_3OD	[353]
2-12-13	oxoassoanine	$C_{17}H_{15}NO_3$	$CDCl_3$	[354]
2-12-14	hippacine	$C_{15}H_9NO_3$	DMSO-d_6	[355]
2-12-15	vasconine	$C_{17}H_{16}NO_2$	–	[356]
2-12-16	8-*O*-demethylvasconine	$C_{16}H_{14}NO_2$	CD_3OD	[348]
2-12-17	tortuosine	$C_{18}H_{18}NO_3$	CD_3OD	[357]
2-12-18	roserine	$C_{18}H_{22}NO_3$	$CDCl_3$	[358]
2-12-19	mooreine	$C_{17}H_{18}NO_4$	CD_3OD	[359]
2-12-20	ungeremine	$C_{16}H_{11}NO_3$	CD_3OD	[360]
2-12-21	zefbetaine	$C_{16}H_{13}NO_3$	CD_3OD	[360]
			CD_3OD-CD_3COOD(3:1)	[360]
2-12-22	*iso*-zefbetaine	$C_{16}H_{13}NO_3$	CD_3OD-CD_3COOD(3:1)	[360]
2-12-23	oxoassoanine-*N*-oxide	$C_{17}H_{15}NO_4$	CD_3OD	[354]
2-12-24	3-*O*-acetylnarcissidine *N*-oxide	$C_{20}H_{25}NO_7$	CD_3OD	[344]



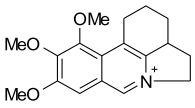
2-12-12 R = OMe
2-12-13 R = H



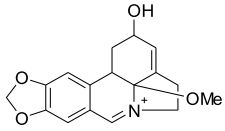
2-12-14



	R^1	R^2
2-12-15	H	OMe
2-12-16	H	OH
2-12-17	OMe	OMe



2-12-18



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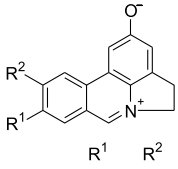
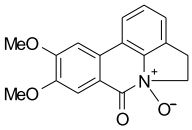
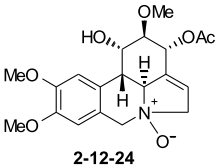


	R^1	R^2
2-12-20	OCH_2O	
2-12-21	OMe	OH
2-12-22	OH	OMe



2-12-23



2-12-24

Table 2-12-6: ^{1}H NMR spectroscopic data of lycorine-type alkaloids **2-12-12**~**2-12-17**.

H	2-12-12	2-12-13	2-12-14	2-12-15	2-12-16	2-12-17
1	7.81 d(8.0)	7.84 d(7.5)	8.02 dd	8.32 d(7)	7.95 d(7.5)	8.13 d(1.4)
2	7.18 t(8.0)	7.26 t(7.5)	7.46 t(7.6)	7.84 t(7)	7.67 t(7.5)	4.20 s(OMe)
3	7.31 d(7.5)	7.33 d(7.5)	7.78 dd(1.0)	7.70 d(7)	7.52 d(7.5)	7.59 br s
6				10.42 s	8.85 s	9.55 s
7	4.06 s(OMe)	7.57 s	7.79 s	8.09 s	6.92 s	7.92 s
8	4.02 s(OMe)			4.08 s(OMe)		4.17 s(OMe)
9	3.97 s(OMe)			4.19 s(OMe)	3.85 s(OMe)	4.33 s(OMe)
10	7.44 s	7.81 s	7.61 s	7.86 s	7.21 s	8.34 s
11	3.40 t(8.0)	3.45 t(8)	7.01 d(3.4)	3.76 t(7)	3.64 t(6.5)	3.88 t(7.2)
12	4.45 t(8.0)	4.45 t(8)	8.05 d(3.4)	5.42 t(7)	4.96 t(6.5)	5.35 t(7.2)
OMe		4.09 s, 4.03 s				

Table 2-12-7: ^{1}H NMR spectroscopic data of lycorine-type alkaloids **2-12-18**, **2-12-19**, and **2-12-24**.

H	2-12-18	2-12-19	2-12-24
1	α 2.85 ddd(16.0, 6.0, 1.5)	5.49 ddd(0.7, 3.8, 5.3)	4.76 br s
	β 3.13 dd(16.0, 6.0)	5.48 ddd(0.5, 1.1, 5.3)	
2	α 1.80~1.95 m	4.61 m	3.70 dd(3, 2)
	β 2.25~2.40 m		3.51 s(OMe)
3	α 1.38 qd(11.0, 2.0)	6.45 m	5.67 br s
	β 2.25~2.40 m		2.03 s(OAc)
4	3.40~3.50 m		
4a			4.39 d(12.5)
6	9.50 s	9.36 s	α 4.06 d(13), β 4.52 d(13)

Table 2-12-7 (continued)

H	2-12-18	2-12-19	2-12-24
7	6.95 s	7.63 s	7.02 s
8			3.86 s(OMe)
9			3.88 s(OMe)
10		7.77 s	7.13 s
10b		5.31 m	2.90 ddd(12.5, 2, 1)
11	*α* 2.05 ddd(11.0, 10.0, 6.0) *β* 2.70 dt(11.0, 6.0)	3.32 m	6.01 dd(3.5, 2)
12	4.75~4.95 m	5.00 m	*α* 4.60 ddd(17, 2, 1) *β* 4.87 ddd(17, 2.5, 2)
OMe	3.90 s, 4.05 s, 4.15 s	3.32 s	
OCH_2O		6.38	

Table 2-12-8: ^{1}H NMR spectroscopic data of lycorine-type alkaloids **2-12-20**~**2-12-23**.

H	2-12-20	2-12-21	2-12-21	2-12-22	2-12-23
		(CD_3OD)	(CD_3OD-CD_3COOD 3:1)		
1	7.41 d(1.8)	7.43 d(1.8)	7.64 d(1.8)	7.79 d(1.8)	8.52 d(8)
2					7.97 t(8)
3	7.22 dt(1.8, 1.8)	7.15 dt(1.8, 1.8)	7.34 dt(1.8, 1.8)	7.35 dt(1.8, 1.8)	7.86 d(8)
6	9.13 br s	9.00 br s	9.30 br s	9.24 br s	
7	7.60 s	7.32 s	7.78 s	7.67 s	8.01 s
8		3.90 s(OMe)	4.10 s(OMe)		
9				4.23 s(OMe)	
10	7.95 s	7.90 s	7.94 s	8.07 s	8.05 s
11	3.68 td(6.8)	3.64 td(6.8)	3.75 td(6.8)	3.76 td(6.8)	3.85 t(7)
12	5.15 t(6.8)	5.00 t(6.8)	5.21 t(6.8)	5.22 t(6.8)	5.39 t(7)
OMe					4.28 s, 4.26 s
OCH_2O	6.34 s				

2.12.2 Crinine-type benzylated phenethylamine alkaloids

Table 2-12-9: Cos, MFs, and TSs of crinine-type alkaloids **2-12-25**~**2-12-32**.

No.	Compounds	MFs	Test solvents	References
2-12-25	(–)-8-demethylmaritidine	$C_{16}H_{19}NO_3$	$CDCl_3$	[361]
2-12-26	maritidine	$C_{17}H_{21}NO_3$	CD_3OD	[362]
2-12-27	oxomaritidine	$C_{17}H_{19}NO_3$	$CDCl_3$	[363]
2-12-28	(–)-*O*-methylpapyramine	$C_{19}H_{25}NO_4$	$CDCl_3$	[364]
2-12-29	(+)-*O*-methylmaritidine	$C_{18}H_{23}NO_3$	$CDCl_3$	[364]
2-12-30	papyramine	$C_{18}H_{23}NO_4$	$CDCl_3$	[346]
2-12-31	6-*epi*-papyramine	$C_{18}H_{23}NO_4$	$CDCl_3$	[346]
2-12-32	cantabricine	$C_{18}H_{23}NO_4$	$CDCl_3$	[365]

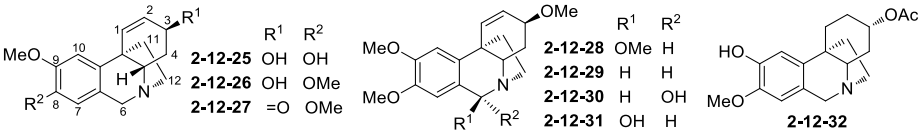


Table 2-12-10: ^{1}H NMR spectroscopic data of crinine-type alkaloids **2-12-25**~**2-12-28**.

H	2-12-25	2-12-26	2-12-27	2-12-28
1	6.71 d(10.2)	6.71 d(10.0)	7.68 d(10.0)	6.59 d(10)
2	5.92 dd(10.2, 5.2)	5.93 dd(10.0, 5.5)	6.10 dd(10.0, 1.0)	5.90 dd(10, 4.6)
3	4.27 m	4.27 ddd(4.2, 5.6, 1.7)		3.75 m
4	*α* 1.79 ddd(4.1, 13.2, 13.2)	*α* 1.95 m	*α* 2.48 dd(17.0, 13.0)	ax 1.52 ddd(4.1, 13.5, 13.5)
		β 1.81 ddd	*β* 2.69 ddd(13.5, 13.5, 4.2)	
	β 1.92 m		(17.0, 5.5, 1.0)	eq 1.85 m
4a	3.42 dd(4.2, 13.2)	3.48 dd(13.4, 4.2)	3.66 dd(13.0, 5.5)	3.47 dd(4.4, 13.5)
6	*α* 4.34 d(16.4)	4.39 d(16.6)	*α* 3.82 d(16.0)	4.43 s
	β 3.79 d(16.4)	3.84 d(16.6)	*β* 4.43 d(16.0)	
7	6.51 s	6.66 s	6.54 s	6.71 s
8		3.77 s(OMe)	3.82 s(OMe)	
9	3.83 s(OMe)	3.81 s(OMe)	3.89 s(OMe)	
10	6.94 s	6.97 s	6.89 s	6.73 s
11	*α* 2.19 ddd(12.6, 9, 4)	2.17 m	en 2.39 ddd(12.5, 9.0, 3.5)	1.85 m
	β 1.98 m	1.95 m	ex 2.18 ddd(12.5, 10.5, 6.5)	
12	*α* 3.37 m	3.34	en 3.01 ddd(13.0, 9.0, 6.5)	3.28 ddd(4.8, 9.7, 13.5)
	β 2.93 m	2.93 ddd(13.5, 9.3, 6.6)	ex 3.55 ddd(13.0, 10.5, 3.5)	2.65 ddd(5.7, 8.3, 13.5)
OMe				3.79 s, 3.77 s, 3.52 s 3.26 s

Table 2-12-11: ^{1}H NMR spectroscopic data of crinine-type alkaloids **2-12-29**~**2-12-32**.

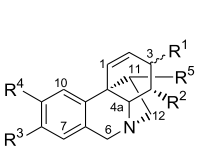
H	2-12-29	2-12-30	2-12-31	2-12-32
1	6.49 d(10.1)	6.65 d(10.0)	6.66 d(10.0)	ax 1.77 ddd(13.5, 13.5, 4.5)
				eq 2.39 dt(13.5, 3.5)
2	6.08 dd(10.1, 4.8)	6.00 dd(5.0, 10.0)	6.00 dd(5.0, 10.0)	ax 1.61 dddd(13.5, 13.5, 11.5, 3.5)
				eq 2.06 m
3	3.85 m	3.88 m	3.85 m	4.67 tt(11.5, 4.5)
				2.02 s(OAc)

Table 2-12-11 (continued)

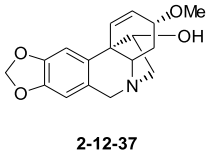
H	2-12-29	2-12-30	2-12-31	2-12-32
4	ax 1.73 ddd(13.5, 13.5, 3.9) eq 2.95 ddd(13.6, 3.8, 2)	1.77 ddd(4.0, 13.4, 13.5) 2.21 br d(13.5)	1.60 ddd(4.0, 13.8, 13.9) 2.09 m	ax 1.44 ddd(12.5, 12, 11.5) eq 2.23 m
4a	3.9 dd(13.5, 3.8)	3.59 dd(4.0, 13.4)	3.90 m	3.18 dd(12, 5)
6	4.80 d(15.8) 4.25 d(15.8)	5.86 s	5.16 s	*α* 3.91 d(16.5) *β* 4.50 d(16.5)
7	6.57 s	7.03 s	6.89 s	6.48 s
8				3.82 s(Ome)
10	6.81 s	6.80 s	6.83 s	6.74 s
11	2.37 ddd(4.3, 8.8, 12.5) 2.22 ddd(7.1, 12.0, 12.5)	2.00 m	2.00 m	ex 2.30 ddd(12, 11.5, 7) en 1.83 ddd(12, 9, 5)
12	4.08 ddd(4.3, 12.0, 12.6) 3.27 ddd(7.1, 8.8, 12.6)	en 3.02 ddd(4.0, 9.5, 13.5) ex 3.73 ddd(4.5, 8.0, 13.5)	en 2.84ddd(6.5, 8.0, 14.0) ex 3.39 m	ex 3.60 m en 2.94 m
OMe	3.86 s, 3.82 s, 3.34 s	3.88 s, 3.88 s, 3.37 s	3.88 s, 3.88 s, 3.31 s	

Table 2-12-12: Cos, MFs, and TSs of crinine-type alkaloids **2-12-33**~**2-12-46**.

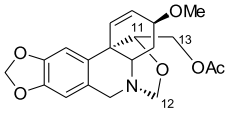
No.	Compounds	MFs	Test solvents	References
2-12-33	albiflomanthine	$C_{17}H_{19}NO_5$	$CDCl_3$	[366]
2-12-34	(+)-narcidine	$C_{17}H_{21}NO_4$	$CDCl_3$-D_2O(99:1)	[367]
2-12-35	yemenine A	$C_{18}H_{19}NO_5$	$CDCl_3$	[368]
2-12-36	hamayne	$C_{16}H_{17}NO_4$	$CDCl_3$	[369]
2-12-37	(+)-crinamine	$C_{17}H_{19}NO_4$	$CDCl_3$	[347]
2-12-38	11-*O*-acetylhaemanthamine	$C_{19}H_{21}NO_5$	$CDCl_3$	[370]
2-12-39	bujeine	$C_{20}H_{23}NO_6$	$CDCl_3$	[370]
2-12-40	delagoensine	$C_{18}H_{21}NO_5$	$CDCl_3$	[371]
2-12-41	delagoenine	$C_{19}H_{25}NO_5$	$CDCl_3$	[371]
2-12-42	3-[4′-(8′-aminoethyl)phenoxy]-bulbispermine	$C_{24}H_{26}N_2O_4$	CD_3OD	[359]
2-12-43	11-*O*-(3′-hydroxybutanoyl)hamayne	$C_{20}H_{23}NO_6$	$CDCl_3$	[345]
2-12-44	3,11-*O*-(3′,3″-dihydroxybutanoyl)-hamayne	$C_{24}H_{29}NO_8$	$CDCl_3$	[345]
2-12-45	3-*O*-(2″-butenoyl)-11-*O*-(3′-hydroxybutanoyl)hamayne	$C_{24}H_{27}NO_7$	$CDCl_3$	[345]
2-12-46	3,11,3″-*O*-(3′,3″,3‴-trihydroxybutanoyl)hamayne	$C_{28}H_{35}NO_{10}$	$CDCl_3$	[345]



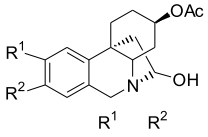
	R^1	R^2	R^3	R^4	R^5
2-12-33	β-OMe	OH	OCH_2O		OH
2-12-34	β-OMe	H	OH	OMe	OH
2-12-35	α-OAc	H	OCH_2O		OH
2-12-36	α-OH	H	OCH_2O		OH
2-12-38	β-OMe	H	OCH_2O		OAc



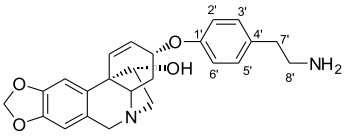
2-12-37



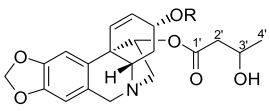
2-12-39



2-12-40 OCH_2O
2-12-41 OMe OMe



2-12-42



2-12-43 R = H

2-12-44 R =

2-12-45 R =

2-12-46 R =

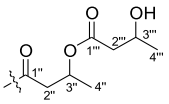


Table 2-12-13: ^{1}H NMR spectroscopic data of crinine-type alkaloids **2-12-33**~**2-12-35**.

H	2-12-33	2-12-34	2-12-35
1	6.58 d(10)	6.48 d(10.1)	6.33 dd(2.1, 10.2)
2	6.23 ddd(10, 5, 1)	6.35 dd(10.1, 5.0)	6.10 dd(1.1, 10.2)
3	3.71 dd(5, 1.5), 3.40 s(OMe)	3.86 m(ov), 3.35 s(OMe)	5.46 m, 2.09 s(OAc)
4	4.24 ddd(4, 1.5, 1)	α 2.11 ddd(4.2, 13.7)	α 2.24 ddd(10.0, 13.0, 13.4)
		β 2.04 dd(14.2, 6.3, <1)	β 2.13 ddd(3.6, 5.1, 13.4)
4a	3.39 d(4)	3.38 m	3.28 dd(3.6, 13.0)
6	3.67 d(17), 4.34 d(17)	α 3.70 d(16.8), β 4.31 d(16.8)	3.72 d(17.5), 4.32 d(17.5)
7	6.46 s	6.53 s	6.48 br s
9		3.86 s(OMe)	
10	6.88 s	6.78 s	6.79 br s
11	4.05 dd(6.5, 4)	3.99 ddd(6.6, 3.3, 1.1)	4.01 m
12	3.29 m	3.26 dd(14.0, 3.3), 3.39 m	3.37 m
OCH_2O	5.90 d(1.5), 5.92 d(1.5)		5.90 br s

Table 2-12-14: ^{1}H NMR spectroscopic data of crinine-type alkaloids **2-12-36**~**2-12-39**.

H	2-12-36	2-12-37	2-12-38	2-12-39
1	6.19 s	6.23 s	6.35 d(10.0)	6.38 d(10.0)
2	6.19 s	6.23 s	6.16 ddd(10.0, 5.0, 1.0)	6.08 dd(10.0, 4.8)
3	4.35 m	3.38 s(OMe)	3.85 ddd(5.0, 4.5, 1.5)	3.89 ddd(4.8, 4.6, 1.5)
			3.36 s(OMe)	3.36 s(OMe)

Table 2-12-14 (continued)

H	2-12-36	2-12-37	2-12-38	2-12-39
4	2.10 m	–	*α* 1.93 ddd (13.5, 13.5, 4.5) *β* 2.01 dddd (13.5, 5.0, 1.5, 1.0)	*α* 2.43 ddd (13.5, 13.5, 4.6) *β* 2.06 ddd (13.5, 4.0, 1.5)
4a	3.25 dd(13.5, 4.5)	–	3.35 dd(13.5, 5.0)	3.29 dd(13.5, 4.0)
6	*α* 3.65 d(16) *β* 4.30 d(16)	*α* 4.29 d(16.9) *β* 3.67 d(16.9)	*α* 3.71 d(17.0) *β* 4.34 d(17.0)	*α* 4.04 d(17.0) *β* 4.45 d(17.0)
7	6.47 s	6.46 s	6.46 s	6.57 s
10	6.81 s	6.78 s	6.90 s	6.81 s
11	4.00 m	–	4.96 ddd(7.0, 3.5, 1.0) 1.97 s(OAc)	3.93 dd(8.3, 3.0)
12	3.35 m	–	en 3.40 dd(14.0, 7.0) ex 3.31 dd(14.0, 3.5)	en 4.51 d(11.0) ex 4.82 d(11.0)
13				4.27 dd(11.6, 3.0) 3.58 dd(11.6, 8.3) 2.00 s(OAc)
OCH_2O	5.90 s	5.87 s	5.89 s	5.91 s

Table 2-12-15: ^{1}H NMR spectroscopic data of crinine-type alkaloids **2-12-40**, **2-12-41**, and **2-12-43**.

H	2-12-40	2-12-41	2-12-43
1	ax 1.88 ddd(13.5, 13.0, 4.0) eq 2.16 dddd(13.5, 3.5, 2.6, 1.0)	ax 1.97 ddd(13.4, 13.0, 4.5) eq 2.27 dddd(13.4, 3.7, 2.8, 1.0)	6.15 dd(10.2, 2.0)
2	ax 1.82 dddd (13.0, 12.5, 2.6, 2.5) eq 1.98 ddddd (12.5, 4.0, 3.5, 3.5, 1.0)	ax 1.86 dddd (13.0, 12.5, 2.8, 2.5) eq 2.05 ddddd (12.5, 4.5, 3.7, 3.5, 1.0)	5.96 dt(10.5, 1.5)
3	5.20 ddddd (4.0, 3.5, 2.5, 2.5, 1.0) 1.97 s(OAc)	5.23 ddddd (4.0, 3.5, 2.5, 2.5, 1.0) 2.01 s(OAc)	4.37 ddt(10.2, 6.0, 2.0)
4	ax 2.24 ddd(13.0, 12.2, 2.5) eq 2.05 dddd(13.0, 5.5, 4.0, 1.0)	ax 2.34 ddd(12.5, 12.2, 2.5) eq 2.20 dddd(12.5, 5.8, 4.0, 1.0)	*α* 1.90 ddd(13.5, 12.0, 10.0) *β* 2.11 dddd(12.0, 6.0, 4.0, 1.0)
4a	3.22 ddd(12.2, 5.5, 1.0)	3.38 ddd(12.2, 5.8, 1.0)	3.22 dd(13.5, 4.0)
6	*α* 3.90 d(16.7) *β* 4.36 d(16.7)	*α* 4.10 d(16.5) *β* 4.42 d(16.5)	*α* 3.69 d(17.0) *β* 4.32 d(16.5)
7	6.46 s	6.52 s	6.46 s
8		3.85 s(OMe)	
9		3.88 s(OMe)	
10	6.73 s	6.73 s	6.87 s
11	en 2.27 ddd(13.5, 6.5, 1.0) ex 2.30 dd(13.5, 5.0)	en 2.34 ddd(13.5, 6.2, 1.0) ex 2.37 dd(13.5, 5.0)	4.95 dt(5.5, 1.0)

Table 2-12-15 (continued)

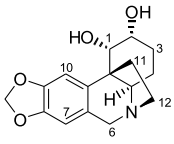
H	2-12-40	2-12-41	2-12-43
12	en 5.04 dd(6.5, 5.0)	en 5.20 dd(6.2, 5.0)	3.41 d(5.5)
2′			2.45 dd(16.0, 3.5)
			2.39 dd(16.0, 8.5)
3′			4.17 ddq(8.5, 6.2, 3.2)
4′			1.22 d(6.0)
OCH_2O	5.90 d(1.5), 5.92 d(1.5)		5.89 d(1.0)

Table 2-12-16: ^{1}H NMR spectroscopic data of crinine-type alkaloids **2-12-42** and **2-12-44**~**2-12-46**.

H	2-12-42	2-12-44	2-12-45	2-12-46
1	6.05 dd(1.2, 10.3)	6.27 dd(10.4, 2.4)	6.22 dd(10.0, 2.5)	6.23 dd(10.5, 2.0)
2	6.24 dd(2.3, 10.3)	5.92 (ov)	5.89 (ov)	5.84 dt(10.0, 1.5)
3	4.40 m	5.52 ddt(10.0, 6.0, 2.0)	5.51 ddt(10.0, 6.5, 2.5)	5.47 ddt(10.5, 6.5, 2.0)
4	2.02 m	*α* 2.01 ddd	*α* 1.99 ddd	*α* 1.95 ddd
		(13.6, 12.0, 10.4)	(13.7, 12.0, 10.5)	(13.5, 12.0, 10.5)
		β 2.16 dddd	*β* 2.13 dddd	*β* 2.11 ddd
		(12.0, 6.4, 4.4, 1.5)	(12.0, 6.0, 4.5, 1.5)	(12.0, 6.0, 4.5)
4a	3.32 m	3.30 dd(13.6, 4.4)	3.28 dd(13.5, 5.0)	3.26 dd(13.5, 4.0)
6	4.27 d(16.9)	*α* 3.72 d(17.2)	*α* 3.69 d(17.0)	*α* 3.70 d(17.0)
	3.72 d(16.9)	*β* 4.34 d(16.8)	*β* 4.32 d(17.0)	*β* 4.32 d(17.0)
7	6.86 s	6.49 s	6.46 s	6.46 s
10	6.53 s	6.87 s	6.85 s	6.84 s
11	3.95 dd(3.6, 7)	5.01 dd(6.4, 4.0)	4.99 dd(6.5, 4.0)	4.99 ddd(6.5, 4.0, 1.0)
12	3.32 m	3.39 dd(14.4, 4.4)	3.37 dd(14.0, 3.5)	3.37 dd(14.5, 4.5)
		3.45 dd(14.4, 6.4)	3.41 dd(14.0, 6.5)	3.42 dd(14.5, 6.5)
2′	7.05 d(8.5)	2.49 dd(16.4, 3.6)	2.49 dd(16.5, 3.5)	2.46 dd(15.5, 4.0)
		2.42 dd(16.0, 8.4)	2.41 dd(16.5, 9.0)	2.39 dd(16.0, 8.5)
3′	6.73 d(8.6)	4.17 m	4.19 m	4.18 m
4′		1.24 d(6.0)	1.22 d(6.5)	1.21 d(6.0)
5′	6.73 d(8.6)			
6′	7.05 d(8.5)			
7′	2.70 t(7)			
8′	2.90 t(7)			
2″		2.54 dd(16.4, 3.6)	5.85 dq(15.5, 1.5)	2.65 dd(15.5, 8.0)
		2.47 dd(16.0, 8.4)		2.58 dd(15.0, 5.0)
3″		4.23 m	7.02 dq(15.5, 7.0)	5.34 m
4″		1.26 d(6.0)	1.88 dd(7.0, 1.5)	1.32 d(6.5)
2‴				2.49 dd(16.0, 4.0)
				2.42 dd(16.0, 8.5)
3‴				4.18 m
4‴				1.23 d(6.5)
OCH_2O	5.87 s	5.92 d(3.6)	5.89 d(1.5)	5.89~5.90 d(1.5)

Table 2-12-17: Cos, MFs, and TSs of crinine-type alkaloids **2-12-47**~**2-12-58**.

No.	Compounds	MFs	Test solvents	References
2-12-47	(–)-amabiline	$C_{16}H_{19}NO_4$	DMSO-d_6	[347]
2-12-48	4*a*-dehydroxycrinamabine	$C_{16}H_{19}NO_4$	$CDCl_3$	[372]
2-12-49	crinamabine	$C_{16}H_{19}NO_5$	CD_3OD	[373]
2-12-50	josephinine	$C_{18}H_{21}NO_5$	$CDCl_3$	[369]
2-12-51	1-epibowdensine	$C_{21}H_{25}NO_7$	$CDCl_3$	[374]
2-12-52	1-epidemethoxybowdensine	$C_{20}H_{23}NO_6$	$CDCl_3$	[374]
2-12-53	1-epideacteylbowdensine	$C_{17}H_{21}NO_5$	$CDCl_3$	[374]
2-12-54	flexinine	$C_{16}H_{17}NO_4$	$CDCl_3$	[373]
2-12-55	augustine	$C_{17}H_{19}NO_4$	$CDCl_3$	[373]
2-12-56	crinamidine	$C_{17}H_{19}NO_5$	$CDCl_3$	[374]
2-12-57	1,2β-epoxyambelline	$C_{18}H_{21}NO_6$	$CDCl_3$	[375]
2-12-58	6α-hydroxycrinamidine	$C_{17}H_{19}NO_6$	$CDCl_3$	[375]



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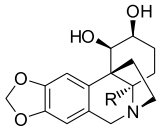
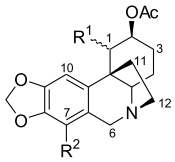
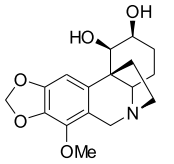


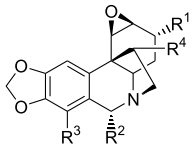
2-12-48 R = H
2-12-49 R = OH



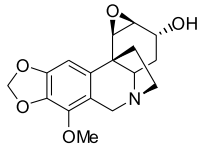
	R^1	R^2
2-12-50	α-OH	H
2-12-51	β-OAc	OMe
2-12-52	β-OAc	H



2-12-53



	R^1	R^2	R^3	R^4
2-12-54	OH	H	H	H
2-12-55	OMe	H	H	H
2-12-57	OMe	H	OMe	OH
2-12-58	OH	OH	OMe	H



2-12-56

Table 2-12-18: ^{1}H NMR spectroscopic data of crinine-type alkaloids **2-12-47**~**2-12-49**.

H	2-12-47	2-12-48	2-12-49
1	4.32 br s	4.05 d(4.5)	3.98 d(4.2)
2	3.88 br d(11.2)	4.15 ddd(4.5, 3.5, 2.5)	4.08 ddd(4.2, 3.2, 2.7)
3	α 1.48 m	ax 1.53 dddd(14.0, 12.5, 3.5, 2.5)	α 2.02 dddd(10.1, 10.7, 3.6,2.7)
	β 1.61 m	eq 2.02 dddd(14.0, 3.5, 3.5, 3.0)	β 2.01 dddd(10.1, 6.9, 3.2, 3.0)
4	α 1.56 m	ax 1.75 dddd(14.0, 12.5, 11.5, 3.5)	α 1.83 ddd(13.4, 3.6, 3.0)
	β 1.25 dddd(12.7, 12.1, 12.1, 0.8)	eq 1.58 dddd(14.0, 5.0, 3.5, 3.0)	β 2.42 ddd(13.4, 10.7, 6.9)

Table 2-12-18 (continued)

H	2-12-47	2-12-48	2-12-49
4a	3.05 dd(12.1, 5.7)	2.98 dd(11.5, 5.0)	
6*α*	4.13 d(16.6)	4.37 d(16.5)	4.69 d(15.2)
6*β*	3.59 d(16.6)	3.75 d(16.5)	4.18 d(15.2)
7	6.51 s	6.42 s	6.63 s
10	6.79 s	7.47 s	7.64 s
11	*α* 2.06 ddd(16.4, 5.9, 5.6)	en 1.97 ddd(12.0, 8.5, 4.0)	*α* 3.29 ddd(13.5, 11.5, 6.6)
	β 1.67 m	ex 2.75 ddd(12.0, 9.0, 6.0)	*β* 2.05 ddd(13.5, 9.5, 4.0)
12	*α* 3.13 m	en 2.80 ddd(12.5, 8.5, 6.0)	*α* 3.75 ddd(12.5, 11.5, 4.0)
	β 2.62 ddd(14.2, 6.1, 5.9)	ex 3.43 ddd(12.5, 9.0, 4.0)	*β* 3.34 ddd(12.5, 9.5, 6.6)
OCH_2O	5.89 s, 5.91 s	5.88 s	5.93, 5.92 AB(1.4)

Table 2-12-19: ^{1}H NMR spectroscopic data of crinine-type alkaloids **2-12-50**~**2-12-52**.

H	2-12-50	2-12-51	2-12-52
1	4.52 d(3)	5.30 d(4.0), 2.08 s(OAc)	5.32 d(4.5), 2.09 s(OAc)
2	5.10 dd(5, 3)	5.55 ddd(4.0, 3.5, 2.0)	5.56 ddd(4.5, 3.5, 2.5)
2-OAc	2.12 s	2.08 s	2.09 s
3	*α* 2.07 tt(13, 4)	ax 1.57 dddd(14.0, 13.5, 3.5, 2.0)	ax 1.56 dddd(14.0, 13.5, 3.5, 2.5)
	β 1.79~1.85 m	eq 1.92 dddd(14.0, 3.5, 3.0, 2.5)	eq 1.93 dddd(14.0, 3.5, 3.0, 3.0)
4	*α* 1.73 m	ax 1.67 dddd(14.0, 13.5, 11.5, 2.5)	ax 1.68 dddd(14.0, 13.5, 12.0, 3.0)
	β 1.59 dddd(13, 13, 13, 3.5)	eq 1.60 dddd(14.0, 5.5, 3.5, 3.0)	eq 1.60 dddd(14.0, 5.5, 3.5, 3.0)
4a	3.35 dd(13, 5)	3.01 dd(11.5, 5.5)	3.06 dd(12.0, 5.5)
6	*α* 4.35 d(16.5)	*α* 4.16 d(17.5)	*α* 4.33 d(17.0)
	β 3.72 d(16.5)	*β* 3.74 d(17.5)	*β* 3.74 d(17.0)
7	6.48 s	3.95 s(OMe)	6.42 s
10	6.77 s	6.16 s	6.43 s
11	en 1.79~1.85 m	en 2.01 ddd(12.5, 9.0, 4.5)	en 2.03 ddd(12.5, 9.0, 4.5)
	ex 2.65 ddd(12, 11, 6.5)	ex 2.73 ddd(12.5, 10.5, 5.5)	ex 2.75 ddd(12.5, 10.5, 5.5)
12	en 2.80 ddd(13, 8.5, 6.5)	en 2.80 ddd(12.5, 9.0, 5.5)	en 2.82 ddd(12.5, 9.0, 5.5)
	ex 3.38 m	ex 3.40 ddd(12.5, 10.5, 4.5)	ex 3.41 ddd(12.5, 10.5, 4.5)
OCH_2O	5.92 s	5.80 d~5.82 d(1.5)	5.84 d~5.85 d(1.5)

Table 2-12-20: ^{1}H NMR spectroscopic data of crinine-type alkaloids **2-12-53**~**2-12-55**.

H	2-12-53	2-12-54	2-12-55
1	4.08 d(4.5)	3.76 d(3.4)	3.77 d(3.5)
2	4.17 ddd(4.5, 3.5, 2.5)	3.32 dd(3.4, 2.1)	3.32 dd(3.5, 2.5)

Table 2-12-20 (continued)

H	2-12-53	2-12-54	2-12-55
3	ax 1.56 dddd(14.0, 12.5, 3.5, 2.5) eq 2.06 dddd(14.0, 3.5, 3.5, 3.0)	4.55 ddd(3.1, 2.4, 2.1)	3.92 ddd(3.0, 2.5, 2.4) 3.47 s(OMe)
4	ax 1.79 dddd(14.0, 12.5, 11.5, 3.5) eq 1.59 dddd(14.0, 5.0, 3.5, 3.0)	*α* 1.89 ddd(13.8, 3.4, 2.4) *β* 1.61 ddd(13.8, 13.3, 3.1)	*α* 1.70 ddd(13.9, 3.6, 2.4) *β* 1.40 ddd(13.9, 13.3, 3.0)
4a	2.95 dd(11.5, 5.0)	3.41 dd(13.3, 3.4)	3.09 dd(13.3, 3.6)
6	*α* 4.22 d(17.5) *β* 3.80 d(17.5)	*α* 4.48 d(16.5) *β* 3.82 d(16.5)	*α* 4.37 d(16.8) *β* 3.69 d(16.8)
7	4.00 s(OMe)	6.49 s	6.47 s
10	7.24 s	6.92 s	6.88 s
11	en 2.01 ddd(12.5, 8.5, 4.5) ex 2.78 ddd(12.5, 9.5, 6.0)	*α* 2.49 ddd(12.5, 11.0, 6.0) *β* 2.09 ddd(12.5, 9.1, 4.5)	*α* 2.38 ddd(13.0, 10.8, 5.7) *β* 2.0 ddd(13.0, 9.2, 4.6)
12	en 2.80 ddd(12.5, 8.5, 6.0) ex 3.42 ddd(12.5, 9.5, 4.5)	*α* 3.39 ddd(13.0, 11.0, 4.5) *β* 2.92 ddd(13.0, 9.1, 6.0)	*α* 3.18 ddd(12.9, 10.8, 4.6) *β* 2.80 ddd(12.9, 9.2, 5.7)
OCH_2O	5.88~5.90 d(1.5)	5.93, 5.92 AB(1.4)	5.90, 5.89 AB(1.4)

Note: ex denotes *exo* and en *endo*. The same bellow.

Table 2-12-21: ^{1}H NMR spectroscopic data of crinine-type alkaloids **2-12-56**~**2-12-58**.

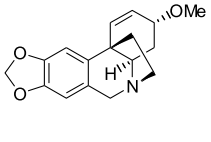
H	2-12-56	2-12-57	2-12-58
1	3.75 d(4.0)	3.69 d(3.5)	3.65 d(3.5)
2	3.26 ddd(4.0, 2.5, 1.5)	3.29 ddd(3.5, 2.5, <1.0)	3.20 br s
3	4.48 ddd(3.0, 2.5, 2.0)	3.95 dt(3.5, 2.5)	4.44 br s
3-OMe		3.39 s	
4	*α* 1.61 dddd(13.5, 5.5, 2.0. 1.5) *β* 1.56 ddd(13.5, 12.5, 3.0)	*α* 1.76 dddd(14.0, 3.5, 3.0, <1.0) *β* 1.48 ddd(14.0, 13.5, 3.5)	*α* 1.72 m *β* 1.45 ddd(14.0, 13.5, 3.0)
4a	3.17 dd(12.5, 5.5)	3.12 dd(13.5, 3.0)	3.93 dd(13.5, 3.0)
6	*α* 4.19 d(17.5) *β* 3.71 d(17.5)	*α* 4.23 d(17.5) *β* 3.76 d(17.5)	5.33 s
7	3.95 s(OMe)	3.98 s(OMe)	4.01 s(OMe)
10	6.61 s	6.69 s	6.61 s
11	en 2.0 ddd(12.5, 9.0, 5.0) ex 2.37 ddd(12.5, 10.5, 5.5)	4.86 br dd(7.5, 4.0)	en 1.75 ddd(12.0, 9.0, 4.0) ex 2.33 ddd(12.0, 11.5, 6.5)
12	en 2.77 ddd(12.5, 9.0, 5.5) ex 3.17 ddd(12.5, 10.5, 5.0)	en 2.38 ddd(14.0, 4.0, 1.5) ex 3.50 dd(14.0, 7.5)	en 2.70 ddd(13.0, 9.0, 6.5) ex 3.18 ddd(13.0, 11.5, 4.0)
OCH_2O	5.85~5.86 d(1.5)	5.91 s	5.88~5.89 d(1.5)

Table 2-12-22: Cos, MFs, and TSs of crinine-type alkaloids **2-12-59**~**2-12-68**.

No.	Compounds	MFs	Test solvents	References
2-12-59	(–)-buphanisine	$C_{17}H_{19}NO_3$	$CDCl_3$	[347]
2-12-60	crinine	$C_{16}H_{17}NO_3$	$CDCl_3$	[376]

Table 2-12-22 (continued)

No.	Compounds	MFs	Test solvents	References
2-12-61	buphanidrine	$C_{18}H_{21}NO_4$	$CDCl_3$	[376]
2-12-62	ambelline	$C_{18}H_{21}NO_5$	$CDCl_3$	[376]
2-12-63	11-*O*-acetylambelline	$C_{20}H_{23}NO_6$	$CDCl_3$	[376]
2-12-64	6*α*-hydroxybuphanidrine	$C_{18}H_{21}NO_5$	$CDCl_3$	[377]
2-12-65	6*α*-methoxybuphanidrine	$C_{19}H_{23}NO_5$	$CDCl_3$	[377]
2-12-66	macowine	$C_{16}H_{19}NO_3$	$CDCl_3$	[372]
2-12-67	brunsbelline	$C_{18}H_{21}NO_5$	$CDCl_3$	[376]
2-12-68	filifoline	$C_{24}H_{24}N_2O_6$	$CDCl_3$	[377]



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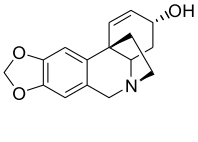

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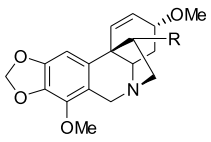
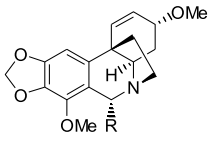
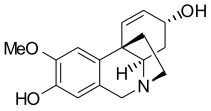


2-12-61 R = H
2-12-62 R = OH
2-12-63 R = OAc



2-12-64 R = OH
2-12-65 R = OMe



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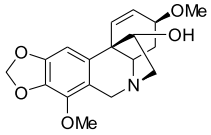

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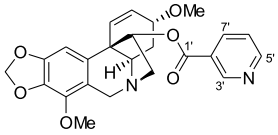


2-12-68

Table 2-12-23: ^{1}H NMR spectroscopic data of crinine-type alkaloids **2-12-59**~**2-12-62**.

H	2-12-59	2-12-60	2-12-61	2-12-62
1	6.58 d(10.0)	6.59 d(10.0)	6.42 d(10.0)	6.57 d(10.0)
2	5.95 dd(10.0, 5.3)	5.96 dd(5.4, 10.0)	5.95 dd(5.0, 10.0)	6.01 dd(5.0, 10.0)
3	3.80 m	4.33 m	3.77 m	3.84 m
3-OMe	3.34 s		3.29 s	3.35 s
4	–	*α* 2.06 br d(13.5) *β* 1.74 ddd(4.2, 13.5, 13.5)	*α* 2.46 br d(13.5) *β* 1.60 ddd(4.0, 13.5, 13.5)	*α* 2.11 dd(4.5, 14.0) *β* 1.67 ddd(4.0, 14.0, 14.0)
4a	–	3.42 dd(3.9, 13.5)	3.50 dd(3.5, 13.5)	3.36 dd(4.5, 14.0)
6*α*	4.38 d(16.8)	4.41 d(16.8)	4.35 d(17.0)	4.27 dd(17.0)
6*β*	3.76 d(16.8)	3.78 d(16.8)	3.95 d(17.0)	3.86 d(17.0)
7	6.45 br s	6.48 s	3.92 s(OMe)	3.99 s(OMe)
10	6.81 s	6.85 s	6.50 s	6.59 s
11	–	en 2.19 ddd(3.9, 8.7, 12.5) ex 1.94 ddd(6.3, 10.8, 12.5)	en 2.20 ddd(4.0, 9.0, 12.5) ex 1.99 ddd(6.0, 10.5, 12.5)	ex 4.39 dd(4.5, 8.5)

Table 2-12-23 (continued)

H	2-12-59	2-12-60	2-12-61	2-12-62
12	–	en 2.91 ddd(6.3, 8.7, 12.8) ex 3.36 br d(12.8)	en 2.99 ddd(6.0, 9.0, 13.0) ex 3.64 m	en 2.52 ddd(1.5, 4.5, 13.5) ex 3.63 dd(8.5, 13.5)
OCH_2O	5.86 s, 5.85 s	5.89~5.91 d(1.2)	5.81~5.82 d(1.5)	5.87~5.88 d(1.5)

Table 2-12-24: ^{1}H NMR spectroscopic data of crinine-type alkaloids **2-12-63**~**2-12-65**.

H	2-12-63	2-12-64	2-12-65
1	6.51 d(10.0)	6.52 d(10.0)	6.53 d(10.0)
2	6.02 ddd(1.0, 5.5, 10.0)	5.94 ddd(10.0, 5.0, 0.5)	5.94 ddd(10.0, 5.0, 1.0)
3	3.83 ddd(2.0, 4.0, 5.5) 3.32 s(OMe)	3.79 ddd(5.0, 4.0, 1.5) 3.32 s(OMe)	3.81 ddd(5.0, 4.0, 1.5) 3.32 s(OMe)
4	*α* 2.16 br d(13.5)	*α* 2.08 dddd(14.0, 4.0, 1.5, 0.5)	*α* 1.94 dddd(14.0, 4.0, 1.5, 1.0)
	β 1.70 ddd(4.0, 13.5, 13.5)	*β* 1.54 ddd(14.0, 13.5, 4.0)	*β* 1.57 ddd(14.0, 13.5, 4.0)
4a	3.43 dd(3.0, 13.5)	3.84 ddd(13.5, 4.0, 0.5)	3.65 ddd(13.5, 4.0, 0.5)
6	*α* 4.33 d(17.5) *β* 3.86 d(17.5)	5.24 s	4.59 s 3.56 s(OMe)
7	3.97 s(OMe)	4.01 s(OMe)	3.95 s(OMe)
10	6.43 s	6.55 s	6.54 s
11	ex 5.11 dd(4.0, 8.0) 1.81 s(OAc)	en 1.90 dddd(12.5, 9.0, 4.5, 0.5) ex 1.84 ddd(12.5, 10.5, 6.0)	en 1.87 dddd(12.5, 9.0, 4.5, 0.5) ex 1.81 ddd(12.5, 10.0, 6.5)
12	en 2.72 ddd(1.5, 4.0, 14.0) ex 3.78 dd(8.0, 14.0)	en 2.79 ddd(13.0, 9.0, 6.0) ex 3.31 ddd(13.0, 10.5, 4.5)	en 2.67 ddd(13.0, 9.0, 6.5) ex 3.27 ddd(13.0, 10.0, 4.5)
OCH_2O	5.83~5.87 d(1.5)	5.84~5.87 d(1.5)	5.83~5.87 d(1.5)

Table 2-12-25: ^{1}H NMR spectroscopic data of crinine-type alkaloids **2-12-66**~**2-12-68**.

H	2-12-66	2-12-67	2-12-68
1	6.61 d(10.0)	6.35 d(10.0)	6.58 d(10.0)
2	5.96 ddd(10.0, 5.0, <1)	6.32 dd(1.8, 10.0)	6.07 ddd(10.0, 5.0, 1.0)
3	4.33 ddd(5.0, 4.0, 1.5)	3.84 m 3.34 s(OMe)	3.87 ddd(5.0, 4.0, 2.0) 3.35 s(OMe)
4	*α* 2.01 dddd(13.5, 4.0, 1.5, <1) *β* 1.72 ddd(13.5, 13.5, 4.0)	2.14 m	*α* 2.15 dddd(14.0, 4.5, 2.0, 1.0) *β* 1.76 ddd(14.0, 13.5, 4.0)
4a	3.41 dd(13.5, 4.0)	3.39 m	3.48 ddd(13.5, 4.5, 1.0)
6	*α* 4.37 d(16.5) *β* 3.76 d(16.5)	*α* 4.22 d(17.0) *β* 3.80 d(17.0)	*α* 4.34 d(17.5) *β* 3.90 d(17.5)
7	6.54 s	3.95 s(OMe)	3.97 s(OMe)
9	3.86 s(OMe)		

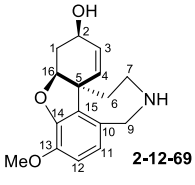
Table 2-12-25 (continued)

H	2-12-66	2-12-67	2-12-68
10	6.79 s	6.54 s	6.48 s
11	en 2.17 ddd(12.5, 9.0, 4.5) ex 1.92 ddd(12.5, 10.5, 6.0)	en 4.02 dd(3.5, 7.0)	ex 5.36 dd(8.0, 4.0)
12	en 2.89 ddd(13.0, 9.0, 6.0) ex 3.37 ddd(13.0, 10.5, 4.5)	3.39 m	en 2.83 ddd(14.0, 4.0, 1.0) ex 3.88 dd(14.0, 8.0)
OCH_2O		5.84~5.85 d(1.5)	5.77~5.81 d(1.5)
3′			8.74 ddd(2.0, 1.0, 0.5)
5′			8.69 ddd(5.0, 2.0, 0.5)
6′			7.27 ddd(8.0, 5.0, 1.0)
7′			7.93 ddd(8.0, 2.0, 2.0)

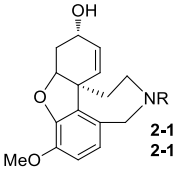
2.12.3 Galanthamine-type benzylated phenethylamine alkaloids

Table 2-12-26: Cos, MFs, and TSs of galanthamine-type alkaloids **2-12-69**~**2-12-77**.

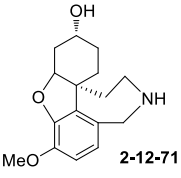
No.	Compounds	MFs	Test solvents	References
2-12-69	*N*-demethylgalanthamine	$C_{16}H_{19}NO_3$	CD_3OD	[378]
2-12-70	epinorgalanthamine	$C_{16}H_{19}NO_3$	$CDCl_3$	[379]
2-12-71	epinorlycoramine	$C_{16}H_{21}NO_3$	$CDCl_3$	[379]
2-12-72	narcisine	$C_{18}H_{21}NO_4$	$CDCl_3$	[380]
2-12-73	leucovernine	$C_{20}H_{25}NO_5$	$CDCl_3$	[381]
2-12-74	acetylleucovernine	$C_{22}H_{27}NO_6$	$CDCl_3$	[381]
2-12-75	galanthamine *N*-oxide	$C_{17}H_{21}NO_4$	C_5D_5N-CD_3OD(3: 1)	[382]
2-12-76	sanguinine *N*-oxide	$C_{16}H_{19}NO_4$	C_5D_5N-CD_3OD(3: 1)	[382]
2-12-77	lycoramine *N*-oxide	$C_{17}H_{23}NO_4$	C_5D_5N-CD_3OD(3: 1)	[382]



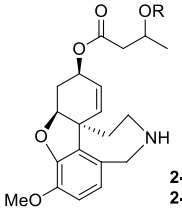
2-12-69



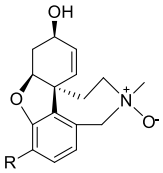
2-12-70 R = H
2-12-72 R = Ac



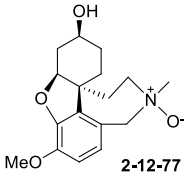
2-12-71



2-12-73 R = H
2-12-74 R = Ac



2-12-75 R = OMe
2-12-76 R = OH



2-12-77

Table 2-12-27: ^{1}H NMR spectroscopic data of galanthamine-type alkaloids **2-12-69**~**2-12-72**.

H	2-12-69	2-12-70	2-12-71	2-12-72
1	*α* 2.24 ddd(15.5, 5.4, 3.3) *β* 2.60 dm(15.5)	*α* 2.04 ddd(15.7, 5, 2.4) *β* 2.68 dd(15.7, 3.5)	*α* 1.88 m *β* 2.50 ddd(15.9, 1.5, <1)	1.95 m, 2.67 m
2	4.28 m	4.15 m	4.09 m	–
3	6.06 ddd(10.5, 4.8, 1.4)	5.98 d(10.4)	1.54~1.70 m	6.08 ddd(10, 4.4, 0.5)
4	6.23 dt (10.5, 1.2)	6.05 d(10.4)	1.73 m, 195 m	6.68 d(10)
6	2.08 m	*α* 1.88 dt(13.5, 3.2) *β* 1.80 td(13.5, 13.5, 3.6)	1.70~1.87 m	1.7 m,2.05 m
7	*α* 3.42 m *β* 3.57 m	*α* 3.22 m *β* 3.38 dt(14.7, 3.6)	*α* 3.19 dt(13, <1) *β* 3.43 m	2.71 m,3.18 m
9	*α* 4.21 d(15) *β* 4.44 d(15)	*α* 3.93 d(15.6) *β* 4.04 d(15.6)	3.98 s	*α* 3.68 d(16) *β* 4.48 d(16)
11	6.84 d(8.4)	6.62 d(8)	6.65 d(8.1)	6.84 d(8.5)
12	6.90 d(8.4)	6.68 d(8)	6.62 d(8.1)	8.12 d(8.5)
13	3.91 s(OMe)	3.84 s(OMe)	3.85 s(OMe)	3.86 s(OMe)
16	4.67 m	4.62 br s	4.37 t(3.0)	5.22 m
Ac				2.01 s

Table 2-12-28: ^{1}H NMR spectroscopic data of galanthamine-type alkaloids **2-12-73** and **2-12-74**.

H	2-12-73	2-12-74	H	2-12-73	2-12-74
1	*α* 2.05 ddd(16.2, 4.8, 3.2) *β* 2.70 ddd(16.2, 3.2, 1.4)	*α* 2.09 ddd(16.3, 5.5, 3.0) *β* 2.67 ddd(16.7, 3.0, 1.2)	11	6.58 d(8.2)	6.58 d(8.0)
2	5.38 t(5.0, 4.8)	5.33 t(5.0, 5.0)	12	6.64 d(8.2)	6.64 d(8.0)
3	5.87 ddd(10.3, 5.0, 1.2)	5.89 ddd(10.1, 4.8, 1.0)	13	3.83 s(OMe)	3.84 s(OMe)
4	6.31 d(10.3)	6.27 d(10.3)	16	4.56 t(3.2, 3.2)	4.55 t(3.2, 3.1)
6	*α* 1.79 ddd(13.7, 12.0, 3.0)	*α* 1.80 ddd(13.5, 12.4, 3.2)	2′	2.48 dd(16.3, 3.2)	2.49 dd(15.8, 6.0)
	β 1.85 dt(13.8, 3.5, 3.0)	*β* 1.86 dt(13.5, 2.5, 2.5)		2.39 dd(16.3, 9.4)	2.61 dd(15.7, 6.9)
7	*α* 3.35 dt(13.6, 3.5, 3.0) *β* 3.24 ddd(13.5, 12.1, 3.0)	*α* 3.36 dt(13.5, 3.6, 3.4) *β* 3.24 ddd(13.3, 13.4, 2.3)	3′	4.19 ddq (9.4, 6.4, 3.2)	5.24 ddq (9.5, 6.9, 3.0)
9	*α* 3.93 d(15.4) *β* 4.02 d(15.4)	*α* 3.95 d(15.3) *β* 4.04 d(15.5)	4′	1.18 d(6.4)	1.27 d(6.2)
Ac		1.95 s			

Table 2-12-29: ^{1}H NMR spectroscopic data of galanthamine-type alkaloids **2-12-75**~**2-12-77**.

H	2-12-75	2-12-76	2-12-77
1*α*	2.73 d(16)	2.70 d(15)	2.57 d(16)
1*β*	2.12 d-like(16)	2.16 d-like(15)	2.09 m

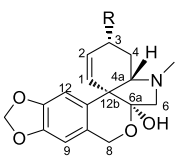
Table 2-12-29 (continued)

H	2-12-75	2-12-76	2-12-77
2	4.46 br s	4.46 t(5)	–
3	6.28 dd(10, 4)	6.22 dd(10, 5)	–
4	6.43 d(10)	6.39 d(10)	–
6*α*	2.24 t(14)	2.23 t(16)	–
6*β*	1.90 d(14)	1.90 d(16)	1.73 m
7*α*	3.96 d(14)	3.89 d(14)	3.86 d(16)
7*β*	4.35 t(14)	4.26 t(14)	4.30 (16)
9*α*	4.66 d(14)	4.59 d(14)	4.57 d(14)
9*β*	5.32 d(14)	5.21 d(14)	5.07 d(14)
11	6.71 d(8)	6.78 d(8)	6.83 s
12	6.82 d(8)	6.95 d(8)	6.83 s
13	3.71 s(OMe)		3.80 s(OMe)
16	4.69 br s	4.67 br s	4.45 br s
NMe	3.20 s	3.16 s	3.12 s

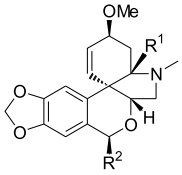
2.12.4 Tazettine-type benzylated phenethylamine alkaloids

Table 2-12-30: Cos, MFs, and TSs of tazettine-type alkaloids **2-12-78**~**2-12-90**.

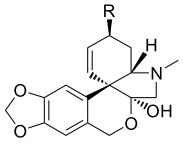
No.	Compounds	MFs	Test solvents	References
2-12-78	(+)-isotazettinol	$C_{17}H_{19}NO_5$	CD_3OD	[383]
2-12-79	criwelline	$C_{18}H_{21}NO_5$	$CDCl_3$	[384]
2-12-80	6-*O*-methylpretazettine	$C_{19}H_{23}NO_5$	CD_3OD	[353]
2-12-81	littoraline	$C_{18}H_{21}NO_5$	$CDCl_3$	[385]
2-12-82	tazettine	$C_{18}H_{21}NO_5$	$CDCl_3$	[386]
2-12-83	3-*O*-demethyltazettine	$C_{17}H_{19}NO_5$	$CDCl_3$	[386]
2-12-84	8*α*-ethoxy precriwelline	$C_{20}H_{25}NO_5$	$CDCl_3$	[387]
2-12-85	*N*-desmethyl-8*α*-ethoxy pretazettine	$C_{19}H_{23}NO_5$	$CDCl_3$	[387]
2-12-86	*N*-desmethyl-8*β*-ethoxy pretazettine	$C_{19}H_{23}NO_5$	$CDCl_3$	[387]
2-12-87	pretazettine	$C_{18}H_{21}NO_5$	$CDCl_3$	[387]
2-12-88	(+)-3-*O*-demethylmacronine	$C_{17}H_{17}NO_5$	CD_3OD	[383]
2-12-89	(+)-3-*O*-demethyl-3-epimacronine	$C_{17}H_{17}NO_5$	CD_3OD	[383]
2-12-90	egonine	$C_{18}H_{23}NO_5$	$CDCl_3$	[386]



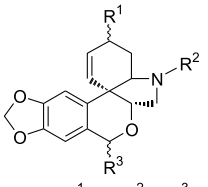
2-12-78 R = OH
2-12-79 R = OMe



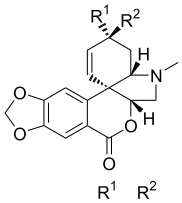
2-12-80 R^1 = H R^2 = OMe
2-12-81 R^1 = OH R^2 = H



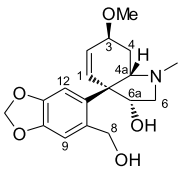
2-12-82 R = OMe
2-12-83 R = OH



	R^1	R^2	R^3
2-12-84	α-OMe	Me	α-OEt
2-12-85	β-OMe	H	α-OEt
2-12-86	β-OMe	H	β-OEt
2-12-87	β-OMe	Me	β-OH



	R^1	R^2
2-12-88	OH	H
2-12-89	H	OH



2-12-90

Table 2-12-31: ^{1}H NMR spectroscopic data of tazettine-type alkaloids **2-12-78**~**2-12-81**.

H	2-12-78	2-12-79	2-12-80	2-12-81
1	5.78 dd(10.2, 1.1)	5.78 d(10)	5.51 dt(10.0, 2.0)	5.89 br d(10.0)
2	6.34 ddd(10.2, 5.1, 0.9)	6.20 dd(10, 3.5)	5.86 br d(10.5)	6.12 td(10.0, 2.0)
3	4.16 m	3.89 ddd(7, 4, 3.5)	4.14 m	3.94 m
		3.45 s(OMe)	3.41 s(OMe)	3.38 s(OMe)
4	α 1.88 ddd(15.0, 4.1, 1.9)	1.93 ddd(15, 7, 3)	α 2.49 m	1.86 dd(9.5, 13.0)
	β 2.14 dt(15.0, 1.9)	2.09 ddd(15, 4, 3)	β 1.74 ddd(13.5, 9.5, 2.0)	2.20 ddd(1.5, 6.5, 12.5)
4a	3.05 t(1.7)	2.95 t(3)	2.91 br s	
6	3.38 d(11.2)	2.83 d(10.5)	α 2.98 t(10.5)	2.78 dd(8.0, 10.0)
	2.74 d(11.2)	3.30 d(10.5)	β 2.64 dd(10.0, 7.5)	3.25 dd(7.5, 9.5)
6a			4.23 dd(11.0, 7.5)	4.33 dd(9.5, 7.5)
8	5.01 d(14.9)	4.68 d(15)	5.56 s	4.63
	4.67 d(14.9)	4.94 d(15)	3.53 s(OMe)	
9	6.59 s	6.55 s	6.75 s	6.44 s
12	6.60 s	6.52 s	6.73 s	6.90 s
NMe	2.48 s	2.38 s	2.47 s	2.43 s
OCH_2O	5.92 d(1.1), 5.92 d(1.1)	5.92 s	5.89 d(1.5), 5.88 d(1.5)	5.89 d(2.0), 5.92 d(2.0)

Table 2-12-32: ^{1}H NMR spectroscopic data of tazettine-type alkaloids **2-12-82**~**2-12-86**.

H	2-12-82	2-12-83	2-12-84	2-12-85	2-12-86
1	5.54 ddd	5.52 ddd(10.4, 2.1, 1.6)	5.87 d(10.3)	5.73~5.90 m	6.22 s
2	6.08 ddd	5.99 ddd(10.4, 1.8, 1.3)	5.99~6.06 m	5.99 m	6.22 s
3	4.06 dddd	4.53 dddd(10.1, 6.1, 2.1, 1.8)	3.87 m	3.80 m	4.04 m
	3.40 s(OMe)	1.5~2.8 (OH)	3.42 s(OMe)	3.40 s(OMe)	3.4 s(OMe)

Table 2-12-32 (continued)

H	2-12-82	2-12-83	2-12-84	2-12-85	2-12-86
4	*α* 2.23 dddd	*α* 2.18 dddd(13.7, 6.1, 3.8, 1.3)	2.32 m	2.10~2.38 m	2.03~2.09 m
	β 1.56 ddd	*β* 1.57 ddd(13.7, 10.1, 2.4)			
4a	2.79 m	2.80 m(3.8, 2.4, 1.6)	2.87 br s	3.46 br s	3.52 br s
6	*α* 3.24 d	*α* 3.26 d(10.6)	*α* 3.09 dd(9.8, 11.2)	2.83~2.98 m	3.20 d(4.9)
	β 2.62 d	*β* 2.62 d(10.6)	*β* 2.66 dd(8.0, 9.8)		
6a	2.6~2.9(OH)	1.5~2.8(OH)	4.35 dd(8.0, 11.2)	4.46 dd(6.7, 11.2)	3.87 m
8	*α* 4.89 d	*α* 4.89 d(14.7)	5.68 s	5.70 s	4.50 s
	β 4.58 d	*β* 4.58 d(14.7)			
9	6.44 s	6.45 s	6.61 s	6.56 s	6.72 s
12	6.79 s	6.78 s	6.76 s	6.78 s	6.73 s
NMe	2.43 s	2.34 s	2.50 s		
OCH_2O	5.84 AB(1.8)	5.85 AB(1.8)	5.80 s	5.89 d(1.4)	5.9 d(1.4)
	5.85 AB(1.8)	5.86 AB(1.8)		5.9 d(1.4)	5.8 d(1.4)
$O\underline{CH_2}CH_3$			3.94 dd(7.0, 9.7)	3.92 dd(7.0, 9.7)	4.03 dd(7.0, 9.7)
			3.70 dd(7.0, 9.7)	3.70 dd(7.0, 9.7)	3.66 dd(7.0, 9.7)
$OCH_2\underline{CH_3}$			1.30 t(7.0)	1.29 t(7.0)	1.26 t(7.0)

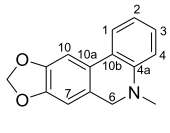
Table 2-12-33: ^{1}H NMR spectroscopic data of tazettine-type alkaloids **2-12-87**~**2-12-90**.

H	2-12-87	2-12-88	2-12-89	2-12-90
1	6.17 m	5.57 dd(10.1, 1.4)	5.39 d(10.3)	5.73 ddd(10.6, 2.1, 1.8)
2	5.84 br	6.14 dd(10.1, 5.2)	5.90 d(10.3)	5.92 m
3	–	4.11 m	4.49 dd(9.2, 7.0)	3.94 dddd (10.6, 5.6, 1.8, 1.6)
	3.4 s(OMe)			3.33 s(OMe)
4	–	ax 2.23 ddd(15.2, 4.6, 1.7)	ax 1.85 t(11.8)	*α* 2.32 m
		eq 2.44 dddd (15.2, 3.8, 1.6, 1.6)	eq 2.53 m	*β* 1.69 ddd (13.2, 10.6, 2.6)
4a	–	3.36 dd(3.3, 1.6)	3.3 (ov)	2.93 m
6	–	*α* 3.24 dd(11.3, 10.2)	*α* 3.17 t(10.8)	*α* 3.07 d(10.6)
		β 2.89 dd(10.1, 7.8)	*β* 2.93 m	*β* 2.49 m
6a	–	4.71 dd(11.3, 7.8)	4.65 t(9.1)	4.45 d(5.5), 2.3~3.1 (OH)
8	6.08 s			4.69, 4.79 d(11.9)
				2.3~3.1 (OH)
9	6.51 s	7.43 s	7.44 s	6.91 s
12	6.82 s	6.84 s	6.90 s	6.67 s
NMe	2.42 s	2.58 s	2.60 s	2.38 s
OCH_2O	5.92 s	6.08 d(1.0)	6.09 d(0.7)	5.87 d(1.8)
		6.07 d(1.0)	6.08 d(0.7)	5.86 d(1.8)

2.12.5 Narciclasine-type benzylated phenethylamine alkaloids

Table 2-12-34: Cos, MFs, and TSs of narciclasine-type alkaloids **2-12-91**~**2-12-98**.

No.	Compounds	MFs	Test solvents	References
2-12-91	5,6-dihydrobicolorine	$C_{15}H_{13}NO_2$	$CDCl_3$	[354]
2-12-92	3-hydroxy-8,9-methylenedioxyphenanthridine	$C_{14}H_9NO_3$	$CDCl_3$	[384]
2-12-93	8,9-methylenedioxophenantridine	$C_{14}H_9NO_2$	$CDCl_3$	[388]
2-12-94	*N*-methyl-8,9-methylenedioxy-6-phenantridone	$C_{15}H_{11}NO_3$	$CDCl_3$	[388]
2-12-95	bicolorine	$C_{15}H_{12}NO_2$	$CDCl_3$-CD_3OD	[354]
2-12-96	1-*O*-(3-hydroxybutyryl)pancratistatin	$C_{18}H_{21}NO_{10}$	C_5D_5N	[389]
2-12-97	1-*O*-(3-*O*-*β*-D-glucopyranosylbutyryl)-pancratistatin	$C_{24}H_{31}NO_{15}$	C_5D_5N	[389]
2-12-98	narciclasine-4-*O*-*β*-D-glucopyranoside	$C_{20}H_{23}NO_{12}$	DMSO-d_6	[390]



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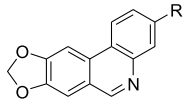
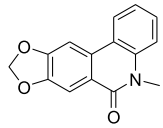
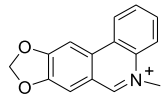


2-12-92 R = OH
2-12-93 R = H



2-12-94



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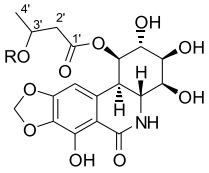
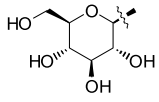
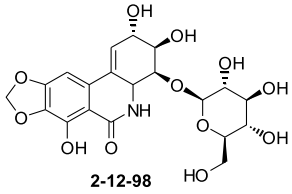


2-12-96 R = H

2-12-97 R =





2-12-98

Table 2-12-35: ^{1}H NMR spectroscopic data of narciclasine-type alkaloids **2-12-91**~**2-12-94**.

H	2-12-91	2-12-92	2-12-93	2-12-94
1	7.00 d(7.3, 1.5)	8.05 d(9)	8.36 dddd(8.0, 1.8, 1.0, 1.0)	8.07 dd(8.1, 1.4)
2	6.83 ddd(8.5, 7.3, 1.1)	7.01 dd(9, 2)	7.61 ddd(8.0, 7.0, 1.7)	7.28 ddd(8.1, 7.1, 1.3)
3	7.30 ddd(8.5, 8.2, 1.5)		7.67 ddd(7.0, 7.0, 1.8)	7.50 ddd(8.4, 7.1, 1.4)
4	6.75 dd(8.2, 1.1)	7.13 d(2)	8.11 ddd(7.0, 1.7, 1.0)	7.38 dd(8.4, 1.3)
6	*α* 4.27 d(12) *β* 4.19 d(12)	8.68 s	9.06 s	
7	6.68 s	7.09 s	7.32 s	7.90 s
10	7.02 s	7.60 s	7.89 s	7.61 s
OCH_2O	6.00 s	5.97 s	6.15 s	6.20 s
NMe	2.74 s			3.70 s

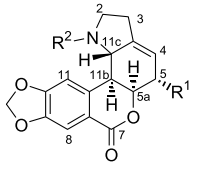
Table 2-12-36: ^{1}H NMR spectroscopic data of narciclasine-type alkaloids **2-12-95**~**2-12-98**.

H	2-12-95	2-12-96	2-12-97	2-12-98
1	8.73 dd(7.8, 2)	6.22 br s	6.16 br s	6.17 t(3.0)
2	8.02 td(7.8, 1.6)	4.94 br s	5.05 br s	4.10 br s
3	8.09 td(7.8, 2)	4.76 br s	4.79 br s	3.87 br s
4	8.31 dd(7.8, 1.6)	4.67 dd(3, 10)	4.65 dd(3, 10)	3.92 dd(9.7, 2.4)
4a		4.72 (ov)	4.72 (ov)	4.30 br d(9.7)
6	10.24 s			
7	7.97 s			
10	8.16 s	6.61 br s	6.57 br s	6.86 s
10b		3.96 br d(15)	3.89 (ov)	
2′		2.66 dd(9, 15)	2.75 dd(5, 15)	
		2.74 dd(4, 15)	2.91 dd(6, 15)	
3′		4.44 (ov)	4.72 (ov)	
4′		1.22 d(7)	1.40 d(6)	
OCH_2O	6.41 s	5.95 br s	5.99 br s	6.06 s
NH		9.23 s	9.22 s	
NMe	4.73 s			
Glu				
1			5.02 d(8)	4.32 d(7.9)
2			3.96 (ov)	3.10 t(7.9)
3			4.18 (ov)	3.22 t(7.9)
4			4.20 (ov)	3.20 t(7.9)
5			3.90 (ov)	3.30 m
6			4.31 dd(5, 12)	3.57 dd(11.6, 5.6)
			4.47 br d(12)	3.79 dd(11.6, 2.2)

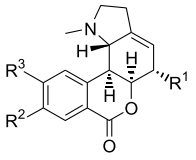
2.12.6 Lycorenine-type benzylated phenethylamine alkaloids

Table 2-12-37: Cos, MFs, and TSs of lycorenine-type alkaloids **2-12-99**~**2-12-110**.

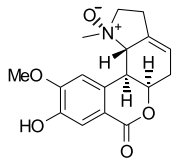
No.	Compounds	MFs	Test solvents	References
2-12-99	hippeastrine	$C_{17}H_{17}NO_5$	$CDCl_3$	[391]
2-12-100	*N*-demethylmasonine	$C_{16}H_{15}NO_4$	$CDCl_3$	[392]
2-12-101	masonine	$C_{17}H_{17}NO_4$	$CDCl_3$	[392]
2-12-102	*O*-demethylhomolycorine	$C_{17}H_{19}NO_4$	$CDCl_3$	[391]
2-12-103	(+)-8-*O*-acetylhomolycorine	$C_{19}H_{21}NO_5$	$CDCl_3$-CD_3OD	[393]
2-12-104	9-*O*-demethyl-2*α*-hydroxyhomolycorine	$C_{17}H_{19}NO_5$	$CDCl_3$-CD_3OD	[394]
2-12-105	(+)-5-methoxy-9-*O*-demethylhomolycorine	$C_{18}H_{21}NO_4$	$CDCl_3$	[395]
2-12-106	(+)-9-*O*-demethylhomolycorine *N*-oxide	$C_{17}H_{19}NO_5$	$CDCl_3$-CD_3OD	[364]
2-12-107	*O*-methyloduline	$C_{18}H_{21}NO_4$	$CDCl_3$	[392]
2-12-108	oduline	$C_{17}H_{19}NO_4$	$CDCl_3$	[392]
2-12-109	2*α*-hydroxy-6-*O*-methyloduline	$C_{18}H_{21}NO_5$	$CDCl_3$	[356]
2-12-110	9-*O*-demethyl-7-*O*-methyllycorenine	$C_{18}H_{23}NO_4$	$CDCl_3$	[396]



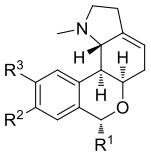
	R^1	R^2
2-12-99	OH	Me
2-12-100	H	H
2-12-101	H	Me



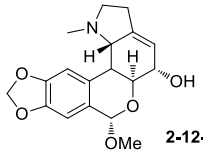
	R^1	R^2	R^3
2-12-102	H	OH	OMe
2-12-103	H	OAc	OMe
2-12-104	OH	OMe	OH
2-12-105	OMe	OH	OMe



2-12-106



	R^1	R^2	R^3
2-12-107	OMe	OCH_2O	
2-12-108	OH	OCH_2O	
2-12-110	OMe	OH	OMe



2-12-109

Table 2-12-38: ^{1}H NMR spectroscopic data of lycorenine-type alkaloids **2-12-99**~**2-12-102**.

H	2-12-99	2-12-100	2-12-101	2-12-102
2	3.16 m	3.15 ddd(10.8, 6.8, 4.3)	3.18 ddd(7.4, 6.3, 4.2)	2.40~2.80 m
	2.25 m	2.89 dd(18.6, 8.6)	2.27 dd(18.7, 9.2)	
3	2.53 m	2.52 m(7.6)	2.48~2.52 br m	2.40~2.80 m
4	5.67 m	5.64 m(<1)	5.50 br d(2.2)	5.51 br s
5	4.42 m	2.62 m	2.58~2.62 m(2.9)	2.40~2.80 m
5a	4.61 br s	4.85 m(<1)	4.75 m(2.2, 2.1)	4.79 br s
8	7.49 s	7.52 s	7.49 s	7.61 s
10				3.96 s(OMe)
11	6.94 s	7.11 s	6.96 s	6.98 s
11b	2.86 dd(9.3, 2.2)	2.66 d(9.7)	2.72~2.76 m(5.2, 1.6)	2.40~2.80 m
11c	2.65 m	5.50 br d(9.3)	2.72~2.76 m(5.2, 1.6)	3.15 m
NMe	2.06 s		2.06 s	2.00 s
OCH_2O	6.09, 6.07 d(1.2)	6.05 d(5.1)	6.07 dd(4.7, 0.9)	

Table 2-12-39: ^{1}H NMR spectroscopic data of lycorenine-type alkaloids **2-12-103**~**2-12-106**.

H	2-12-103	2-12-104	2-12-105	2-12-106
2	*α* 3.31 ddd(9.3, 6.0, 4.2)	*α* 3.17 ddd(9.7, 7.4, 2.5)	3.19 m	3.57 m
	β 2.45 dd(18.0, 9.3)	*β* 2.31 dd(18.5, 9.7)	2.28 dd(18.6, 9.5)	
3	2.5~2.7 m	2.5~2.6 m	2.55 m	2.88 m, 2.72 m
4	5.63 br d(2.0)	5.68 br s	5.67 br s	5.76 br d(2.2)
5	2.5~2.7 m	4.28 br t(1.0)	3.93 br s	2.64 m
5-OMe			3.45 s	
5a	4.86 br d(4.7)	4.59 br d(1.0)	4.68 br s	4.83 m
8	7.47 s	7.47 s	7.61 s	7.44 s
9-OMe	2.00 s	3.95 s		
10-OMe	3.97 s		3.97 s	3.96 s

Table 2-12-39 (continued)

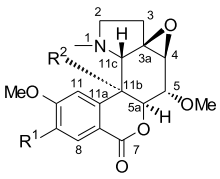
H	2-12-103	2-12-104	2-12-105	2-12-106
11	7.13 s	6.95 s	7.02 s	7.24 s
11b	2.86 dd(10.0, 2.0)	2.83 dd(9.7, 1.0)	2.77 d(7.8)	3.5 dd(9.9, 2.0)
11c	2.96 br d(10.0)	2.66 br d(9.7)	2.70 d(9.9)	3.87 br d(9.9)
NMe	2.16 s	2.08 s	2.01 s	2.94 s

Table 2-12-40: ^{1}H NMR spectroscopic data of lycorenine-type alkaloids **2-12-107**~**2-12-110**.

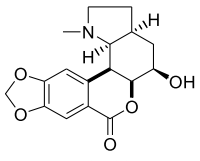
H	2-12-107	2-12-108	2-12-109	2-12-110
2	3.08 d(8.0, 4.4)	3.14 ddd(9.2, 6.3, 3.8)	*α* 3.33 m	3.29 m
	2.17 dd(18.5, 9.4)	2.25 dd(18.7, 9.5)	*β* 2.38 dt(9.5, 9.5)	2.34 m
3	2.33~2.43 m	2.43~2.50 m	2.54 m	2.52 m
4	5.39 br s	5.46 br d(2.9)	5.69 br s	5.52 br d(2.0)
5	2.53 dm(19.2, 3.1)	2.62 dm(19.3)	4.21 br s	2.65 m
	2.26 dd(19.1, 2.6)	2.31 dm(19.0, 2.8)		2.34 m
5a	4.17 d(5.5)	4.35 d(6.0)	4.15 br s	4.29 br d(5.5)
7	5.39 br s	5.99 s	5.43 s	5.47 s
7-OMe	3.43 s		3.51 s	3.51 s
8	6.67 s	6.85 s	6.73 s	6.83 s
10				3.88 s(OMe)
11	6.77 s	6.90 s	6.94 s	7.01 s
11b	2.35 dd(10.9, 1.1)	2.43~2.50 m	2.85 d(10.0)	2.59 m
11c	2.69 br d(8.7)	2.72 br d(9.5)	2.87 d(10.0)	2.90 m
NMe	2.05 s	2.11 s	2.22 s	2.17 s
OCH_2O	5.83 d(20.0)	5.97 d(14.7)	5.91 d(2.0)	

Table 2-12-41: Cos, MFs, and TSs of lycorenine-type alkaloids **2-12-111**~**2-12-124**.

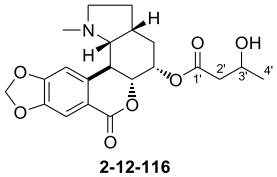
No.	Compounds	MFs	Test solvents	References
2-12-111	(+)-16-hydroxygalwesine	$C_{19}H_{23}NO_7$	$CDCl_3$	[395]
2-12-112	(+)-16-hydroxy-9-*O*-demethylgalwesine	$C_{18}H_{21}NO_7$	$CDCl_3$-D_2O	[395]
2-12-113	(+)-galwesine	$C_{19}H_{23}NO_6$	CD_3OD	[395]
2-12-114	(+)-9-*O*-demethylgalwesine	$C_{18}H_{21}NO_6$	$CDCl_3$	[395]
2-12-115	nobilisitine A	$C_{17}H_{19}NO_5$	$CDCl_3$	[397]
2-12-116	nobilisitine B	$C_{21}H_{25}NO_7$	$CDCl_3$	[397]
2-12-117	galasine	$C_{19}H_{23}NO_7$	$CDCl_3$	[395]
2-12-118	hostasine	$C_{18}H_{21}NO_7$	CD_3OD	[396]
2-12-119	8-demethoxyhostasine	$C_{17}H_{19}NO_6$	CD_3OD	[396]
2-12-120	8-demethoxy-10-*O*-methylhostasine	$C_{18}H_{21}NO_6$	CD_3OD	[396]
2-12-121	10-*O*-methylhostasine	$C_{19}H_{23}NO_7$	CD_3OD	[396]
2-12-122	lycosinine A	$C_{18}H_{21}NO_3$	$CDCl_3$	[398]
2-12-123	lycosinine B	$C_{18}H_{19}NO_3$	$CDCl_3$	[398]
2-12-124	galanthindole	$C_{17}H_{15}NO_3$	$CDCl_3$	[399]



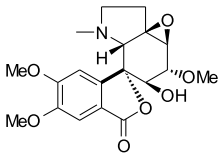
	R^1	R^2
2-12-111	OMe	OH
2-12-112	OH	OH
2-12-113	OMe	H
2-12-114	OH	H



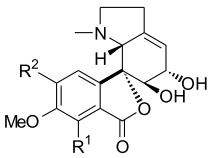
2-12-115



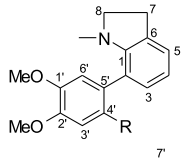
2-12-116



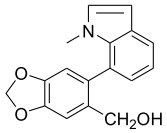
2-12-117



	R^1	R^2
2-12-118	OMe	OH
2-12-119	H	OH
2-12-120	H	OMe
2-12-121	OMe	OMe



2-12-122 R = CH_2OH
2-12-123 R = CHO



2-12-124

Table 2-12-42: ^{1}H NMR spectroscopic data of lycorenine-type alkaloids **2-12-111**~**2-12-114**.

H	2-12-111	2-12-112	2-12-113	2-12-114
2*α*	3.18 t(8.5)	3.18 t(8.6)	3.14 t(8.5)	3.10 t(8.4)
2*β*	2.64 m	2.63 m	2.52 m	2.51 m
3	2.32 m	*α* 2.33 m	*α* 2.35 m	*α* 2.35 d
	1.88 dd(14.4, 6.2)	*β* 1.88 ddd(14.2, 6.5, 1.1)	*β* 1.81 ddd(14.1, 6.2, 0.9)	*β* 1.83 dd(14.0, 6.3, 0.9)
4	3.41 s	3.41 s	3.39 s	3.33 s
5	3.90 s	3.90 s	3.84 d(0.9)	3.88 s
5-OMe	3.58 s	3.75 s	3.53 s	3.53 s
5a	4.58 s	4.56 s	4.62 br s	4.52 br s
8	7.48 s	7.54 s	7.47 s	7.60 s
10		4.00 s(OMe)		3.98 s(OMe)
11	7.13 s	7.13 s	7.14 s	6.96 s
11b			2.81 dd(10.5, 2.4)	2.74 dd(10.2, 2.4)
11c	2.69 s	2.69 s	2.38 d(10.5)	2.45 d(10.2)
NMe	2.01 s	2.01 s	2.03 s	2.03 s
OMe	3.99 s, 3.93 s		3.93 s, 3.86 s	

Table 2-12-43: ^{1}H NMR spectroscopic data of lycorenine-type alkaloids **2-12-115**~**2-12-117**.

H	2-12-115	2-12-116	2-12-117
2	3.24 ddd(13.6, 8.0, 4.2)	3.28 ddd(13.4, 9.0, 5.0)	*α* 3.07 dt(8.4, 1.6)
	2.30 m	2.56 m	*β* 2.50 m
3	1.62 m,2.00 m	2.11 dddd(12.9, 9.0, 4.6, 4.6)	*α* 2.37 m
		1.93 m	*β* 1.84 ddd(13.9, 6.7, 1.8)
3a	2.27 m	2.52 m	
4	1.62 m,2.02 m	2.26 ddd(15.9, 2.8, 2.8)	3.55 s
		1.99 ddd(15.9, 7.1, 4.1)	
5	3.96 ddd(9.9, 6.6, 5.0)	5.40 ddd(4.1, 4.1, 2.8)	3.80 s, 3.49 s(OMe)
5a	4.65 dd(6.6, 5.0)	4.21 dd(12.6, 4.1)	3.74 d(9.0), 2.27 d(9.0, OH)①
8	7.53 s	7.50 s	7.29 s
11	7.05 s	7.70 s	7.16 s
11b	3.34 dd(5.0, 5.0)	3.25 dd(12.6, 9.0)	
11c	2.67 dd(5.8, 5.0)	2.94 dd(9.0, 6.5)	3.04 s
2′		2.51 d(6.2)	
3′		4.21 m	
4′		1.24 d(6.3)	
NMe	2.24 s	2.55 s	1.66 s
OMe			3.97 s, 3.93 s
OCH_2O	6.05 br s	6.05 d(1.4), 6.04 d(1.4)	

① After the adding of D_2O, the signal of *δ* 2.27 d(9.0) disappeared, while the signal of *δ* 3.74 s changed into *δ* 3.74 d.

Table 2-12-44: ^{1}H NMR spectroscopic data of lycorenine-type alkaloids **2-12-118**~**2-12-121**.

H	2-12-118	2-12-119	2-12-120	2-12-121
2	3.04 m	3.02 m	3.05 m	3.06 m
	2.30 dd(18.4, 9.2)	2.28 dd(19.0, 9.0)	2.36 dd(18.4, 9.2)	2.36 dd(18.5, 9.2)
3	2.53 m, 2.48 m	2.54 m, 2.47 m	2.58 m, 2.48 m	2.59 m, 2.48 m
4	5.66 br s	5.67 br s	5.70 br s	5.69 br s
5	4.05 br s	4.06 br s	4.10 br s	4.09 br s
5a	3.58 s	3.57 s	3.60 s	3.61 s
8	4.05 s(OMe)	7.26 s	7.34 s	4.07 s(OMe)
9	3.84 s(OMe)	3.91 s(OMe)	3.92 s(OMe)	3.84 s(OMe)
10			3.96 s(OMe)	3.96 s(OMe)
11	6.94 s	7.10 s	7.35 s	7.17 s
11c	3.36 s	3.38 br s	3.49 s	3.49 s
NMe	1.75 s	1.68 s	1.66 s	1.72 s

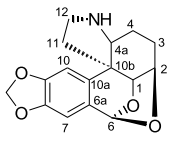
Table 2-12-45: ^{1}H NMR spectroscopic data of lycorenine-type alkaloids **2-12-122**~**2-12-124**.

H	2-12-122	2-12-123	2-12-124
3	6.93 d(7.2)	6.86 d(7.5)	6.93 dd(7.1, 1.2)
4	6.90 t(7.2)	6.72 t(7.4)	7.09 t(7.8)
5	7.14 d(7.2)	7.09 d(7.4)	7.62 dd(7.9, 1.0)
7	3.09 dd(9.9, 5.5) 2.96~3.02 m	2.94~2.98 m	6.52 d(3.1)
8	3.60 dd(14.3, 9.9) 2.92~2.99 m	3.32 dd(16.0, 8.3) 3.21 dd(17.1, 8.8)	6.95 d(3.1)
1′	3.93 s(OMe)	3.94 s(OMe)	
2′	3.87 s(OMe)	3.91 s(OMe)	
3′	6.99 s	7.44 s	7.04 s
6′	6.83 s	6.83 s	6.82 s
7′	4.20 s	9.58 s	4.30 s
NMe	2.19 s	2.21 s	3.29 s
OCH_2O			6.04 d(1.2), 6.03 d(1.2)

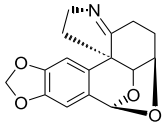
2.12.7 Other benzylated phenethylamine alkaloids

Table 2-12-46: Cos, MFs, and TSs of other benzylated phenethylamine alkaloids **2-12-125**~**2-12-138**.

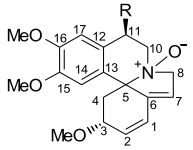
No.	Compounds	MFs	Test solvents	References
2-12-125	noraugustamine	$C_{16}H_{17}NO_4$	$CDCl_3$	[400]
2-12-126	4*a*,*N*-dedihydronoraugustamine	$C_{16}H_{15}NO_4$	$CDCl_3$	[400]
2-12-127	erythristemine-*N*-oxide	$C_{20}H_{25}NO_5$	$CDCl_3$	[401]
2-12-128	erysotrine-*N*-oxide	$C_{19}H_{23}NO_4$	$CDCl_3$	[401]
2-12-129	erythrartine	$C_{19}H_{23}NO_4$	$CDCl_3$	[401]
2-12-130	(+)-graciline	$C_{17}H_{17}NO_3$	$CDCl_3$	[402]
2-12-131	(+)-11-acetoxygraciline	$C_{19}H_{19}NO_5$	$CDCl_3$, C_6D_6	[402]
2-12-132	(+)-3,4-dihydro-3-hydroxygraciline	$C_{17}H_{19}NO_4$	CD_3OD	[402]
2-12-133	nangustine	$C_{16}H_{17}NO_4$	CD_3OD	[403]
2-12-134	pancracine	$C_{16}H_{19}NO_4$	DMSO-d_6	[404]
2-12-135	cripowellin A	$C_{25}H_{31}NO_{12}$	$CDCl_3$	[405]
2-12-136	(+)-plicamine	$C_{26}H_{26}N_2O_6$	$CDCl_3$	[406]
2-12-137	(–)-secoplicamine	$C_{26}H_{28}N_2O_6$	$CDCl_3$	[406]
2-12-138	obliquine	$C_{26}H_{28}N_2O_5$	$CDCl_3$	[407]



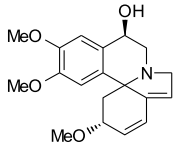
2-12-125



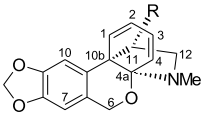
2-12-126



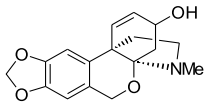
2-12-127 R = OMe
2-12-128 R = H



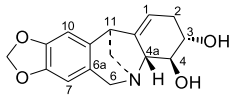
2-12-129



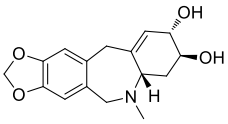
2-12-130 R = H
2-12-131 R = OAc



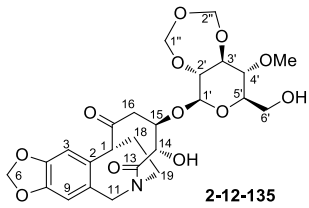
2-12-132



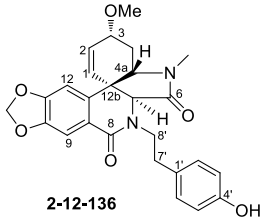
2-12-133



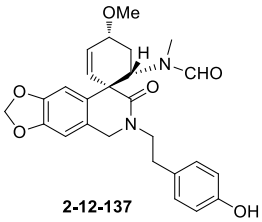
2-12-134



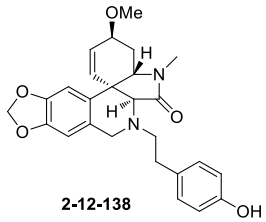
2-12-135



2-12-136



2-12-137



2-12-138

Table 2-12-47: ^{1}H NMR spectroscopic data of other benzylated phenethylamine alkaloids **2-12-125**, **2-12-126** and **2-12-130**.

H	2-12-125	2-12-126	2-12-130
1	4.24 d(4.5)	4.35 d(4.5)	6.09 d(9.0)
2	4.19 dt(4.5, 4.0)	4.22 dt(4.5, 3.5)	5.78 dd(9.3, 5.0)
3	ax 1.99 dddd(15.0, 10.0, 4.5, 4.0)	ax 1.78 dddd(14.0, 13.0, 5.0, 3.0)	6.10 dd(9.8, 5.5)
	eq 1.73 dddd(15.0, 4.5, 4.0, 4.0)	eq 2.29 dddd(14.0, 5.0, 5.0, 3.5)	
4	ax 1.38 dddd(14.5, 10.0, 4.5, 4.5)	ax 2.26 ddd(13.0, 13.0, 5.0)	5.90 (ov)
	eq 1.44 dddd(14.5, 4.0, 4.0, 4.0)	eq 1.98 ddd(13.0, 5.0, 5.0, 2.0)	
4a	3.36 dd(4.5, 4.0)		
6	5.84 s	5.94 s	α 4.50 d(14.5)
			β 4.67 d(14.5)
7	6.60 s	6.62 s	6.41 s
10	6.72 s	6.40 s	6.73 s
11	en 2.05 ddd(13.0, 10.0, 7.5)	en 2.34 ddd(14.0, 9.0, 5.0)	α 2.34 m
	ex 2.36 ddd(13.0, 8.5, 4.0)	ex 2.44 ddd(14.0, 9.0, 7.0)	β 2.30 m
12	en 3.30 ddd(10.5, 10.0, 4.0)	en 4.12 dddd(16.0, 9.0, 5.0, 2.5)	α 2.86 m
	ex 3.21 ddd(10.5, 8.5, 7.5)	ex 4.07 dddd(16.0, 9.0, 7.0, 2.5)	β 2.92 dt(8.4, 6.8)
OCH_2O	5.91~5.90 d(1.5)	5.92~5.91 d(1.5)	5.91 d(1.4)
			5.88 d(1.4)
NMe			2.54 s

Table 2-12-48: ^{1}H NMR spectroscopic data of other benzylated phenethylamine alkaloids **2-12-127**~**2-12-129**.

H	2-12-127	2-12-128	2-12-129
1	6.17 d(10.0)	6.18 d(10.0)	6.02 d(10.0)
2	6.68 dd(10.0, 2.5)	6.78 dd(10.0, 2.5)	6.61 dd(10.0, 2.5)
3	4.34 m	4.24 m	4.06 m
	3.36 s(OMe)	3.36 s(OMe)	3.32 s(OMe)
4	ax 3.23 t(11.0, 11.0)	ax 3.24 t(11.0,11.0)	ax 1.81 t(11.0, 11.0)
	eq 2.02 dd(6.0, 11.0)	eq 2.14 dd(5.5, 11.0)	eq 2.42 dd(5.5, 11.0)
7	5.78 br s	5.80 br s	5.75 br s
8	5.10 d(15.0, 2.0)	4.43 br s	3.97 d(14.5)
	4.34 m		3.87 dd(14.5, 3.5)
10	ax 4.34 m	ax 4.07 m	ax 3.59 dd(14.5, 4.0)
	eq 3.84 dd(15.0, 2.5)	eq 3.92 m	eq 3.10 dd(14.5, 2.5)
11	4.48 d(2.5)	3.66 m	4.70 t(4.5, 2.5)
	3.62 s(OMe)		
14	6.70 s	6.62 s	6.85 s
15	3.77 s(OMe)	3.76 s(OMe)	3.78 s(OMe)
16	3.92 s(OMe)	3.88 s(OMe)	3.90 s(OMe)
17	6.80 s	6.68 s	6.99 s

Table 2-12-49: ^{1}H NMR spectroscopic data of other benzylated phenethylamine alkaloids **2-12-131** and **2-12-132**.

H	2-12-131 ($CDCl_3$)	2-12-131 (C_6D_6)	2-12-132
1	6.06 dd(11.2, 1.0)	6.10 dd(9.7, 1.0)	6.35 br d(9.7)
2	5.94~5.88 m	5.69 dd(9.7, 5.2)	6.02 br m
3	6.14 ddd(1.1, 5.1, 9.8)	5.86 ddd(9.7, 5.2, 1.0)	4.40 br s
4	5.94~5.88 m	5.77 d(9.7)	α 2.24 br d(15.0)
			β 2.30 br d(15.5)
6α	4.50 d(14.6)	4.37 d(14.4)	4.97 d(15.7)
6β	4.61 d(14.6)	4.43 d(14.4)	4.93 br d(15.6)
7	6.41 s	6.10 s	6.63 s
10	7.04 s	7.36 s	6.96 br s
11	5.42 dd(2.8, 6.7)	5.59 dd(7.1, 3.4)	α 2.35 br m
	2.13 s(OAc)	1.65 s(OAc)	β 2.44 m
12α	2.73 dd(10.0, 2.8)	2.67 dd(9.6, 3.0)	3.40 br dd(10.3, 16.4)
12β	3.34 dd(10.0, 6.7)	3.31 dd(9.7, 7.1)	3.83 br m
NMe	2.53 s	2.40 s	2.93 br s
OCH_2O	5.92 d(1.2), 5.90 d(1.2)	5.27 d(1.3), 5.26 d(1.3)	5.95 br s, 5.94 br s

Table 2-12-50: ^{1}H NMR spectroscopic data of other benzylated phenethylamine alkaloids **2-12-133** and **2-12-134**.

H	2-12-133	2-12-134	H	2-12-133	2-12-134
1	5.52 dt(3.5, 2.5)	5.37 dddd(1.8, 1.8, 1.0, 1.8)	7	6.51 s	6.60 br s
2	*α* 2.05 ddt(18.0, 9.0, 3.5) *β* 2.57 dddd(18.0, 7.0, 3.5, 2.0)	3.73 dddd(1.6, 1.8, 6.0, 1.0) 4.76 d(6.0, OH)	10	6.56 s	6.67 br s
3	3.62 ddd(9.0, 9.0, 7.0)	3.65 ddddd(2.3, 3.8, 1.6, 1.8, 3.0) 4.71 d(3.0, OH)	11	3.33 br d(2.5)	3.25 br s
4	3.31 t(9.0)	ax 1.36 ddd(11.5, 2.3, 12.0) eq 1.83 ddd(4.5, 3.8, 1.0)	12	ax 3.03 d(11.0) eq 2.94 dd(11.0, 2.0)	
4a	3.16 br d(9.0)	3.20 ddd(11.5, 4.5, 1.8)	NMe		2.86 s
6	*α* 4.32 d(16.5) *β* 3.83 d(16.5)	*α* 3.63 br d(16.5) *β* 4.16 br d(16.5)	OCH_2O	5.86~5.85 d(1.5)	5.88 d(1.0) 5.93 d(1.0)

Table 2-12-51: ^{1}H NMR spectroscopic data of other benzylated phenethylamine alkaloids **2-12-135**.

H	2-12-135	H	2-12-135	H	2-12-135
1	3.27 dd(4.8, 2.9)	15	4.11 ddd(12.1, 7.3, 4.0)	3′	3.58 dd(8.6, 9.0)
3	6.55 s	16	3.09 dd(14.6, 12.1) 2.23 dd(14.6, 4.0)	4′	3.23 dd(9.0, 9.6) 3.52 s(OMe)
6	6.00 m	18	3.13 m, 2.21 m	5′	3.37 ddd(9.6, 6.2, 2.4)
9	6.67 s	19	4.35 m 2.77 ddd(13.7, 8.6, 8.6)	6′	3.90 dd(11.7, 2.4) 3.71 dd(11.7, 6.2)
11	5.28 d(17.8) 4.49 d(17.8)	1′	4.54 d(8.0)	1″	4.94 d(6.0) 4.81 d(6.0)
14	4.66 d(7.3)	2′	3.33 dd(8.0, 8.6)	2″	5.02 d(5.8), 4.88 d(5.8)

Table 2-12-52: ^{1}H NMR spectroscopic data of other benzylated phenethylamine alkaloids **2-12-136**~**2-12-138**.

H	2-12-136	2-12-137	2-12-138
1	5.54 d(9.8)	5.73 d(9.9)	5.89 d(9.5)
2	6.00 dd(9.8, 5.2)	6.33 dd(9.9, 4.9)	6.05 dd(9.5, 5.3)
3	3.91 dt(5.1, 3.2), 3.46 s(OMe)	4.09 m, 3.44 s(OMe)	3.89 m, 3.45 s(OMe)
4	*α* 1.48 m *β* 2.44 m	2.77~2.83 m 1.94 br d(13.2)	*α* 1.49 ddd(13.5, 11.5, 3.0) *β* 2.36 dt(13.5, 4.0)

Table 2-12-52 (continued)

H	2-12-136	2-12-137	2-12-138
4a	4.03 dd(11.9, 4.5)	3.88 dd(13.1, 2.8)	3.75 dd(11.5, 4.5)
6		7.23 s	α 3.67 d(15.3),β 3.92 d(15.3)
6a	3.87 s		
7			6.46 s
8		α 4.12 d(16.2),β 4.30 d(16.2)	
9	7.60 s	6.46 s	
10			6.75 s
11			3.64 s
12	6.74 s	6.72 s	
2′, 6′	7.10 d(8.4)	7.03 d(8.4)	7.01 d(8.7)
3′, 5′	6.74 d(8.4)	6.75 d(8.4)	6.73 d(8.7)
7′	2.98~2.89 m	2.83~2.77 m	2.75 t(8.0)
8′	4.50 ddd(14.0, 8.4, 5.4)	3.96 m	3.29 dt(12.5, 8.0)
	3.53 m	3.17 m	3.23 dt(12.5, 8.0)
NMe	2.80 s	2.64 s	2.71 s
OCH_2O	6.02 d(1.3), 6.01 d(1.3)	6.00 d(1.3), 5.96 d(1.3)	5.88 s

Bibliography

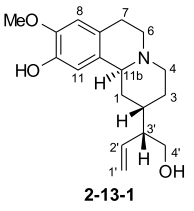
[343] Kihara M, Xu L, Konishi K, et al. Chem Pharm Bull, 1994, 42: 289.
[344] Kihara M, Ozaki T, Kobayashi S, et al. Chem Pharm Bull, 1995, 43: 318.
[345] Berkov S, Codina C, Viladomat F, et al. Phytochemistry, 2007, 68: 1791.
[346] Bastida J, Codina C, Viladomat F, et al. J Nat Prod, 1990, 53: 1456.
[347] Likhitwitayawuid K, Angerhofer CK, Chai H, et al. J Nat Prod, 1993, 56: 1331.
[348] Bastida J, Codina C, Peeters P, et al. Phytochemistry, 1995, 40: 1291.
[349] Campbell WE, Nair JJ, Gammon DW, et al. Phytochemistry, 2000, 53: 587.
[350] Evidente A, Andolfi A, Abou-Donia AH, et al. Phytochemistry, 2004, 65: 2113.
[351] Campbell WE, Nair JJ, Gammon DW, et al. Planta Med, 1998, 64: 91.
[352] Szlávik L, Gyuris A, Minárovits J, et al. Planta Med, 2004, 70: 871.
[353] Cabezas F, Ramírez A, Viladomat F, et al. Chem Pharm Bull, 2003, 51: 315.
[354] Viladomat F, Bastida J, Tribo G, et al. Phytochemistry, 1990, 29: 1307.
[355] Ramadan MA, Kamel MS, Ohtani K, et al. Phytochemistry, 2000, 54: 891.
[356] Almanza GR, Fernández JM, Wakori EWT, et al. Phytochemistry, 1996, 43: 1375.
[357] Bastida J, Fernández JM, Viladomat F, et al. Phytochemistry, 1995, 38: 549.
[358] Bastida J, Codina C, Viladomat F, et al. J Nat Prod, 1992, 55: 134.
[359] Elgorashi EE, Drewes SE, Staden JV. Phytochemistry, 2001, 56: 637.
[360] Abou-Donia AH, Abib AA, Din ASE, et al. Phytochemistry, 1992, 31: 2139.
[361] Abou-Donia AH, Amer ME, Darwish FA, et al. Planta Med, 2002, 68: 379.
[362] Youssef DTA, Frahm AW. Planta Med, 1998, 64: 669.
[363] Herrera MR, Machocho AK, Brun R, et al. Planta Med, 2001, 67: 191.
[364] Suau R, Rico R, García AI, et al. Heterocycles, 1990, 31: 517.
[365] Bastida J, Contreras JL, Codina C, et al. Phytochemistry, 1995, 40: 1549.
[366] Baudouin G, Tillequin F, Koch M. Heterocycles, 1994, 38: 965.
[367] Tojo E. J Nat Prod, 1991, 54: 1387.

[368] Abdel-Halim OB, Morikawa T, Ando S, et al. J Nat Prod, 2004, 67: 1119.
[369] Viladomat F, Bastida J, Codina C, et al. Phytochemistry, 1994, 35: 809.
[370] Labraña J, Choy G, Solans X, et al. Phytochemistry, 1999, 50: 183.
[371] Nair JJ, Campbell WE, Gammon DW, et al. Phytochemistry, 1998, 49: 2539.
[372] Nair JJ, Machocho AK, Campbell WE, et al. Phytochemistry, 2000, 54: 945.
[373] Pham LH, Döpke W, Wagner J, et al. Phytochemistry, 1998, 48: 371.
[374] Viladomat F, Almanza GR, Codina C, et al. Phytochemistry, 1996, 43: 1379.
[375] Machocho A, Chhabra SC, Viladomat F, et al. Phytochemistry, 1999, 51: 1185.
[376] Viladomat F, Codina C, Bastida J, et al. Phytochemistry, 1995, 40: 961.
[377] Nair JJ, Campbell WE, Brun R, et al. Phytochemistry, 2005, 66: 373.
[378] Bastida J, Viladomat F, Llabrés JM, et al. Planta Med, 1990, 56: 123.
[379] Bastida J, Viladomat F, Bergoñon S, et al. Phytochemistry, 1993, 34: 1656.
[380] Abdallah OM. Phytochemistry, 1993, 34: 1447.
[381] Forgo P, Hohmann J. J Nat Prod, 2005, 68: 1588.
[382] Kobayashi S, Satoh K, Numata A, et al. Phytochemistry, 1991, 30: 675.
[383] Ünver N, Noyan S, Gözler T, et al. Planta Med, 1999, 65: 347.
[384] Razafimbelo J, Andriantsiferana M, Baudouin G, et al. Phytochemistry, 1996, 41: 323.
[385] Lin LZ, Hu SF, Chai HB, et al. Phytochemistry, 1995, 40: 1295.
[386] Pham LH, Gründemann E, Wagner J, et al. Phytochemistry, 1999, 51: 327.
[387] Elgorashi EE, Drewes SE, Staden JV. Phytochemistry, 1999, 52: 533.
[388] Suau R, Gómez AI, Rico R. Phytochemistry, 1990, 29: 1710.
[389] Kojima K, Mutsuga M, Inoue M, et al. Phytochemistry, 1998, 48: 1199.
[390] Abou-Donia AH, Giulio AD, Evidente A, et al. Phytochemistry, 1991, 30: 3445.
[391] Kihara M, Konishi K, Xu L, et al. Chem Pharm Bull, 1991, 39: 1849.
[392] Kreh M, Matusch R. Phytochemistry, 1995, 38: 1533.
[393] Bastida J, Codina C, Viladomat F, et al. J Nat Prod, 1992, 55: 122.
[394] Bastida J, Codina C, Viladomat F, et al. Phytochemistry, 1990, 29: 2683.
[395] Latvala A, Önür MA, Gözler T, et al. Phytochemistry, 1995, 39: 1229.
[396] Wang YH, Zhang ZK, Yang FM, et al. J Nat Prod, 2007, 70: 1458.
[397] Evidente A, Abou-Donia AH, Darwish FA, et al. Phytochemistry, 1999, 51: 1151.
[398] Yang Y, Huang SX, Zhao YM, et al. Helv Chim Acta, 2005, 88: 2550.
[399] Unver N, Kaya GI, Werner C, et al. Planta Med, 2003, 69: 869.
[400] Machocho AK, Bastida J, Codina C, et al. Phytochemistry, 2004, 65: 3143.
[401] Chawla AS, Sood A, Kumar M, et al. Phytochemistry, 1992, 31: 372.
[402] Noyan S, Rentsch GH, Önür MA, et al. Heterocycles, 1998, 48: 1777.
[403] Labraña J, Machocho AK, Kricsfalusy V, et al. Phytochemistry, 2002, 60: 847.
[404] Ail AA, Mesbah MK, Frahm AW. Planta Med, 1984, 50: 188.
[405] Velten R, Erdelen C, Gehling M, et al. Tetrahedron Lett, 1998, 39: 1737.
[406] Ünver N, Gözler T, Walch N, et al. Phytochemistry, 1999, 50: 1255.
[407] Brine ND, Campbell WE, Bastida J, et al. Phytochemistry, 2002, 61: 443.

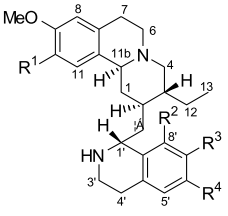
2.13 Emetine-type alkaloids

Table 2-13-1: Cos, MFs, and TSs of emetine-type alkaloids **2-13-1**~**2-13-12**.

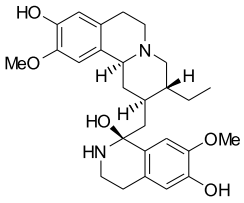
No.	Compounds	MFs	Test solvents	References
2-13-1	alangine	$C_{18}H_{25}NO_3$	$CDCl_3$	[408]
2-13-2	emetine	$C_{29}H_{40}N_2O_4$	$CDCl_3$	[409]
2-13-3	cephaeline	$C_{28}H_{38}N_2O_4$	$CDCl_3$	[409]
2-13-4	neocephaeline	$C_{28}H_{38}N_2O_4$	$CDCl_3$	[409]
2-13-5	7′-*O*-demethylcephaeline	$C_{27}H_{36}N_2O_4$	CD_3OD	[409]
2-13-6	10-*O*-demethylcephaeline	$C_{27}H_{36}N_2O_4$	CD_3OD	[409]
2-13-7	(–)-klugine	$C_{27}H_{36}N_2O_5$	$CDCl_3$, C_5D_5N	[410]
2-13-8	isocephaeline	$C_{28}H_{38}N_2O_4$	$CDCl_3$	[409]
2-13-9	(–)-7′-demethylisocephaeline	$C_{27}H_{36}N_2O_4$	$CDCl_3$, C_5D_5N	[410]
2-13-10	tubulosine	$C_{29}H_{37}N_3O_3$	DMSO-d_6	[411]
2-13-11	1′,2′-dehydrotubulosine	$C_{29}H_{35}N_3O_3$	CD_3OD	[408]
2-13-12	1′,2′,3′,4′-tetradehydrotubulosine	$C_{29}H_{33}N_3O_3$	CD_3OD	[412]



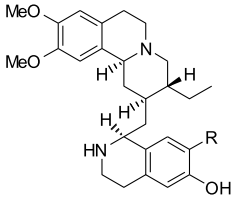
2-13-1



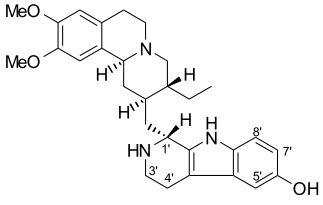
	R^1	R^2	R^3	R^4
2-13-2	OMe	H	OMe	OMe
2-13-3	OMe	H	OMe	OH
2-13-4	OMe	OH	OMe	H
2-13-5	OMe	H	OH	OH
2-13-6	OH	H	OMe	OH



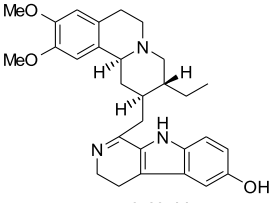
2-13-7



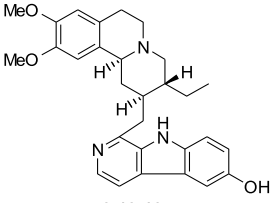
2-13-8 R = OMe
2-13-9 R =OH



2-13-10



2-13-11



2-13-12

Table 2-13-2: ^{1}H NMR spectroscopic data of emetine-type alkaloids **2-13-1**~**2-13-3**.

H	2-13-1	2-13-2	2-13-3
1	2.14 m	1.26 br q(13.0) 2.62 dt(13.0, 3.0)	1.25 br q(13.0) 2.63 dt(13.0, 3.0)
2	1.68 m	1.61~1.70 m	1.62~1.71 m
3	1.71~1.81 m	1.40~1.49 m	1.39~1.47 m
4	2.84 m	2.13 br t(11.0) 3.10 dd(11.0, 4.0)	2.11 br t(11.5) 3.10 dd(11.5, 4.0)
6	3.07 m,3.19 m	2.53 td(11.5, 4.0) 3.02 ddd(11.5, 6.0, 3.0)	2.52 td(11.0, 4.0) 3.00 m
7	2.71 m,3.09 m	2.65 br d(15.0) 3.14 ddd(15.0, 11.5, 6.0)	2.63~2.70 m 3.07~3.20 m
8	6.57 s	6.60 s	6.59 s
9	3.86 s(OMe)		
11	6.77 s	6.77 s	6.77 s
11b	4.07 m	3.20 br d(11.0)	3.18 br d(11.0)
12		1.14 dq(14.0, 7.5) 1.65 dqd(14.0, 7.5, 3.0)	1.15 dq(14.0, 7.5) 1.66 dqd(14.0, 7.5, 3.0)
13		0.91 t(7.5)	0.91 t(7.5)
α		1.44 ddd(14.0, 11.0, 2.5) 2.10 ddd(14.0, 11.0, 2.5)	1.42 ddd(15.0, 11.0, 3.5) 2.10 ddd(15.0, 11.0, 2.5)
1′	5.17 ddd(17.0, 1.5, 0.5) 5.23 dd(10.0, 1.5)	4.13 br d(11.0)	4.11 br d(11.0)
2′	5.60 ddd(17.0, 10.0, 9.5)		
3′	2.35 m	3.04 dt(13.0, 6.0) 3.24 dt(13.0, 6.0)	3.01 dt(13.0, 6.0) 3.20 dt(13.0, 6.0)
4′	3.57 dd(10.5, 7.5) 3.79 dd(10.5, 4.5)	2.73 t(6.0)	2.69 t(6.0)
5′		6.57 s	6.63 s
8′		6.52 s	6.50 s
OMe		3.81 s, 3.84 s, 3.84 s, 3.85 s	3.81 s, 3.84 s, 3.85 s

Table 2-13-3: ^{1}H NMR spectroscopic data of emetine-type alkaloids **2-13-4**~**2-13-6**.

H	2-13-4	2-13-5	2-13-6
1	1.36 br q(12.0) 2.68 dt(12.0, 4.0)	1.17 br q(13.0) 2.62 dt(13.0, 3.5)	1.10~1.25 m 2.64 dt(13.0, 3.0)
2	1.57~1.70 m	1.51~1.63 m	1.51~1.60 m
3	1.39~1.48 m	1.42 br q(10.0)	1.39~1.48 m
4	2.04 br t(11.0) 3.08 dd(11.0, 4.0)	2.16 br t(12.0) 3.11 dd(12.0, 4.0)	2.11 t(11.5) 3.03~3.13 m
6	2.51 td(11.5, 4.5) 3.00 ddd(11.5, 6.0, 2.5)	2.54 td(11.0, 6.0) 3.00~3.08 m	2.48~2.55 m 3.03~3.13 m
7	3.03~3.10 m 2.61~2.71 m	2.68~2.77 m 3.08~3.15 m	2.67~2.73 m 3.03~3.13 m
8	6.58 s	6.69 s	6.66 s
11	6.80 s	6.84 s	6.88 s
11b	3.15 dd(11.0, 4.0)	3.21 br d(11.0)	3.03~3.13 m

Table 2-13-3 (continued)

H	2-13-4	2-13-5	2-13-6
12	1.14 dq(14.0, 7.0)	1.12~1.22 m	1.10~1.25 m
	1.66 dqd(14.0, 7.0, 3.0)	1.73 dqd(14.0, 7.5, 3.0)	1.71 dqd(14.0, 7.5, 3.0)
13	0.90 t(7.0)	0.95 t(7.5)	0.94 t(7.5)
α	1.57~1.70 m	1.51~1.63 m	1.51~1.60 m
	2.11 br t(11.0)	2.10 br t(11.0)	2.07~2.20 m
1′	4.42 br d(11.0)	4.20 br d(11.0)	4.38 br d(10.5)
3′	3.04 ddd(13.0, 6.0, 3.0)	3.00~3.08 m	3.03~3.13 m
	3.09-3.20 m	3.28 dt(13.0, 7.0)	3.10~3.20 m
4′	2.61~2.71 m	2.68~2.77 m①	2.76~2.84 m
	2.79 ddd(16.5, 10.0, 6.0)	2.81 ddd(16.0, 7.0, 6.0)①	2.84~2.92 m
5′	6.60 d(8.0)	6.53 s	6.59 s
6′	6.70 d(8.0)		
8′		6.57 s	6.70 s
OMe	3.84 s, 3.85 s, 3.85 s	3.78 s, 3.79 s	3.80 s, 3.82 s

① Typographic error exists in the literature, also assigning an addition signal at *δ* 2.83 dt(15.5, 7.0) to H-4′.

Table 2-13-4: ^{1}H NMR spectroscopic data of emetine-type alkaloids **2-13-7**~**2-13-9**.

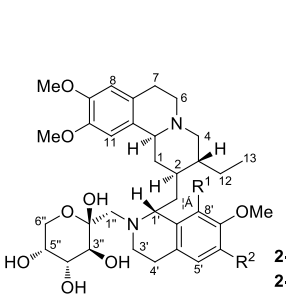
H	2-13-7	2-13-8	2-13-9
1	1.57 m, 2.07 m	1.14~1.22 m,2.34 dt(13.0, 3.0)	1.30 m, 2.71 m
2	1.59 m	1.59~1.68 m	1.68 m
3	1.43 m	1.52 m	1.53 m
4	2.12 m,3.08 m	2.05 br t(11.5), 3.08 dd(11.5, 4.0)	2.32 m, 3.18 m
6	2.99 m, 3.09 m	2.47 td(11.5, 4.0), 2.98 dd(11.5, 6.5)	2.69 m, 3.20 m
7	2.65 m, 3.07 m	2.61 dd(15.5, 4.0)	2.95 m, 3.07 m
		3.11 ddd(15.5, 11.5, 6.5)	
8	6.52 s	6.55 s	6.77 s
11	6.79 s	6.52 s	6.76 s
11b	3.17 br d(10.6)	3.05 br d(11.0)	3.47 br d(10.9)
12	1.13 m,1.69 m	1.14~1.22 m	1.10 m, 1.67 m
		1.77 dqd(15.0, 7.5, 2.5)	
13	0.95 t(7.7)	0.95 t(7.5)	0.87 t(7.5)
α	2.60 dd(3.1, 14.6)	1.59~1.68 m,2.15 m	1.65 m, 2.35 m
	1.15 dd(8.9, 14.6)		
1′		4.09 br t(5.5)	4.52 m
3′	3.03 m,3.27 m	3.00 ddd(12.5, 7.5, 5.5)	3.32 m, 3.52 m
		3.27 dt(12.5, 5.5)	
4′	2.74 m,2.78 m	2.64 dt(16.0, 5.5)	2.67 m, 3.04 m
		2.77 ddd(16.0, 7.5, 5.5)	
5′	6.55 s	6.61 s	6.44 s
8′	6.68 s	6.63 s	6.76 s
OMe	3.79 s, 3.78 s	3.79 s, 3.82 s, 3.83 s	3.82 s, 3.80 s
NH	7.86 s		

Table 2-13-5: ^{1}H NMR spectroscopic data of emetine-type alkaloids **2-13-10**~**2-13-12**.

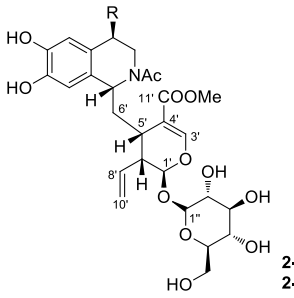
H	2-13-10	2-13-11	2-13-12
1	1.04 (12.2, 12.2, 12.2)	1.17 dt(13.5, 11.5)	1.90~2.00 m(ov)
	2.61 m	2.03 ddd(13.5, 4.0, 3.0)	1.28~1.41 m(ov)
2	1.66 m	1.81 m	1.90~2.00 m(ov)
3	1.25 m	1.55 m	1.68 m
4	1.99 (11.2, 11.2)	2.10 t(11.5)	3.00~3.14 m(ov)
	2.96 m	3.11 dd(11.5, 4.0)	2.20 dd(11.6, 11.6)
6	2.37 (11.2, 10.6, 6.1)	2.50 m	3.00~3.14 m(ov)
	2.90 m	3.18 m	2.56 m
7	2.58 m, 2.93 m	2.65 dt(14.0, 4.0), 3.18 m	3.00~3.14 m(ov), 2.67 m
8	6.60 s	6.61 s	6.59 s
11	6.27 s	6.19 s	6.06 s
11b	2.99 m	3.18 m	3.00~3.14 m(ov)
12	1.10 (14.2, 7.6, 7.6)	1.24~1.32 m	1.90~2.00 m(ov)
	1.57 m	1.85 dqd(13.5, 7.5, 3.0)	1.28~1.41 m(ov)
13	0.86 (7.6)	1.00 t(7.5)	1.01 t(7.4)
α	1.54 (12.0, 12.0)	1.24~1.32 m	3.49 dd(13.3, 3.8)
	1.53 m		2.93 dd(13.3, 9.6)
1′	4.11 (12.0)		
3′	2.90 m	3.78 m	8.20 d(5.4)
	3.11 m	3.92 dt(15.0, 7.0)	
4′	2.52 m	2.96 m	7.87 d(5.4)
5′	6.66 (2.4)	6.92 dd(2.5, 0.5)	7.51 d(2.3)
7′	6.77 (2.4, 8.6)	6.90 dd(8.5, 2.5)	7.10 dd(8.8, 2.3)
8′	7.01 (8.6)	7.28 dd(8.5, 0.5)	7.41 d(8.8)
OMe	3.70 s(10-OMe)	3.27 s(10-OMe)	3.71 s(10-OMe)
	3.70 s(9-OMe)	3.72 s(9-OMe)	3.17 s(9-OMe)

Table 2-13-6: Cos, MFs, and TSs of emetine-type alkaloids **2-13-13**~**2-13-22**.

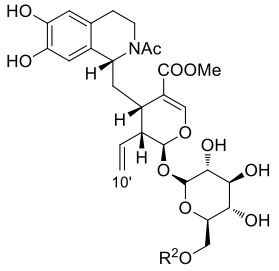
No.	Compounds	MFs	Test solvents	References
2-13-13	2′-*N*-(1″-deoxy-1″-*β*-D-fructopyranosyl)cephaeline	$C_{34}H_{48}N_2O_9$	$CDCl_3$	[409]
2-13-14	2′-*N*-(1″-deoxy-1″-*β*-D-fructopyranosyl)neocephaeline	$C_{34}H_{48}N_2O_9$	$CDCl_3$	[409]
2-13-15	(4*R*)-4-hydroxyipecoside	$C_{27}H_{35}NO_{13}$	CD_3OD	[413]
2-13-16	ipecoside	$C_{27}H_{35}NO_{12}$	CD_3OD	[413]
2-13-17	6″-*O*-*β*-D-glucopyranosylipecoside	$C_{33}H_{45}NO_{17}$	CD_3OD	[413]
2-13-18	6″-*O*-*α*-D-glucopyranosylipecoside	$C_{33}H_{45}NO_{17}$	CD_3OD	[413]
2-13-19	*trans*-cephaeloside	$C_{35}H_{41}NO_{14}$	CD_3OD	[414]
2-13-20	6-*O*-methyl-*trans*-cephaeloside	$C_{36}H_{43}NO_{14}$	CD_3OD	[414]
2-13-21	*cis*-cephaeloside	$C_{35}H_{41}NO_{14}$	CD_3OD	[414]
2-13-22	6-*O*-methyl-*cis*-cephaeloside	$C_{36}H_{43}NO_{14}$	CD_3OD	[414]



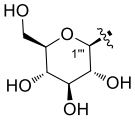
	R^1	R^2
2-13-13	H	OH
2-13-14	OH	H



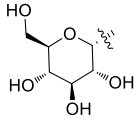
2-13-15 R = OH
2-13-16 R = H

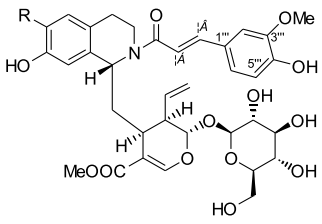


2-13-17 R =

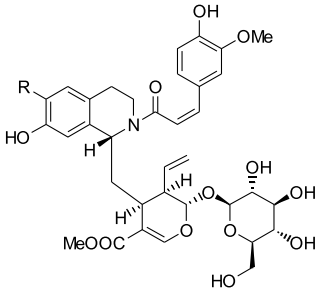


2-13-18 R =





2-13-19 R = OH
2-13-20 R = OMe



2-13-21 R = OH
2-13-22 R = OMe

Table 2-13-7: ^{1}H NMR spectroscopic data of emetine-type alkaloids **2-13-13** and **2-13-14**.

H	2-13-13	2-13-14	H	2-13-13	2-13-14
1	1.23 br q(14.0)	1.23~1.30 m	1′	3.62 br d(11.0)	3.64~3.68 m
	2.66 dt(14.0, 3.0)	2.61 dt(13.0, 3.5)			
2	1.60 br q(11.0)	1.60 br q(11.0)	3′	3.12 dt(10.5, 4.5)	3.10 dt(11.0, 4.0)
				3.20~3.26 m	3.18~3.22 m
3	1.43 br q(10.0)	1.45 br q(11.0)	4′	2.36 dd(16.0, 5.0)	2.42 dd(16.0, 5.5)
				3.07~3.16 m	3.13~3.22 m
4	2.13 br t(11.5)	2.14 br t(12.0)	5′	6.63 s	6.62 d(8.0)
	3.10 dd(11.5, 4.0)	3.02 dd(12.0, 4.5)			

Table 2-13-7 (continued)

H	2-13-13	2-13-14	H	2-13-13	2-13-14
6	2.53 td(12.0, 4.0) 3.02 ddd(12.0, 6.0, 4.0)	2.54 td(11.5, 4.5) 2.99~3.09 m	6′		6.71 d(8.0)
7	2.75 br d(14.0) 3.11~3.20 m	2.74 br d(14.0) 3.07-3.14 m	8′	6.40 s	
8	6.59 s	6.59 s	1″	2.75 br d(13.5) 3.03 d(13.5)	2.76 br d(13.5) 3.03 d(13.5)
11	6.80 s	6.81 s	3″	3.44 br d(9.5)	3.43 br d(9.0)
11b	3.18 br d(12.0)	3.17 br d(10.0)	4″	3.81 dd(9.5, 3.1)	3.83 dd(9.0, 3.5)
12	1.13 dq(14.0, 7.5) 1.62 dqd (14.0, 7.5, 3.0)	1.12 dq(14.0, 7.5) 1.63 dqd (14.0, 7.5, 3.0)	5″	3.95 dt(3.1, 1.5)	3.98 dt(3.5, 1.5)
13	0.90 t(7.5)	0.90 t(7.5)	6″	3.70 dd(13.0, 1.5) 4.00 dd(13.0, 1.5)	3.73 dd(12.0, 1.5) 4.02 dd(12.0, 1.5)
α	1.23 ddd(15.0, 10.0, 3.5) 2.23 ddd(15.0, 11.0, 2.5)	1.23~1.30 m 2.25 m	OMe	3.82 s, 3.85 s 3.87 s	3.84 s, 3.85 s 3.88 s

Table 2-13-8: ^{1}H NMR spectroscopic data of emetine-type alkaloids **2-13-15** and **2-13-16**.

H	2-13-15	2-13-16	H	2-13-15	2-13-16
1	5.64 dd(12.0, 3.0)	5.55 dd(12.0, 3.5)	1′	5.41 d(3.0)	5.41 d(3.0)
3	3.74 dd(14.5, 2.5) 4.02 br d(14.5)	3.64 ddd(14.0, 12.0, 4.5) 3.92 ddd(14.0, 6.0, 1.5)	3′	7.38 d(2.0)	7.38[①], 7.44 d(1.5)
4	4.51 br t(2.0)	2.65 ddd(16.0, 4.5, 1.5) 2.84 ddd(16.0, 12.0, 6.0)	5′	2.71 dddd(12.0, 6.0, 2.5, 2.0)	2.71 dddd(11.5, 6.0, 2.5, 1.5)
5	6.72 s	6.50[①], 6.61 s	6′	1.45 ddd(14.5, 12.0, 3.0) 2.45 ddd(14.5, 12.0, 2.5)	1.51 ddd(14.5, 11.5, 3.5) 2.51 ddd(14.5, 12.0, 2.5)
8	6.46 s	6.45[①], 6.52 s	8′	5.78 dt(17.0, 10.0)	5.76 dt(17.0, 10.0)
9′	3.28 ddd (10.0, 6.0, 3.0)	3.23 ddd (10.0, 6.0, 3.0)	4″	3.28~3.34 m	3.27~3.36 m
10′	5.45 dd(10.0, 2.0) 5.58 dd(17.0, 2.0)	5.43 dd(10.0, 2.0) 5.54 dd(17.0, 2.0)	5″	3.28~3.34 m	3.27~3.36 m
1″	4.61 d(8.0)	4.61[①], 4.66d(8.0)	6″	3.68 dd(12.0, 5.0) 3.89 dd(12.0, 2.0)	3.67 dd(12.0, 5.0) 3.89 dd(12.0, 2.0)
2″	3.16 dd(9.0, 8.0)	3.16 dd(9.0, 8.0)	NAc	2.25 s	2.17, 2.19[①] s
3″	3.28~3.34 m	3.27~3.36 m	COOMe	3.64 s	3.65[①] s, 3.68 s

[①] Signals of the major rotamer.

Table 2-13-9: ^{1}H NMR spectroscopic data of emetine-type alkaloids **2-13-17**~**2-13-19**.

H	2-13-17	2-13-18	2-13-19
1	5.55 dd(11.5, 3.5)	5.56 dd(11.5, 3.5)	5.69 dd(11.5, 3.2)
3	3.62~3.69 m	3.62~3.72 m	4.30 dd(14.0, 5.3)
	3.90 ddd(13.5, 6.0, 2.0)	3.89 ddd(14.0, 6.0, 2.0)	
4	2.65 ddd(16.0, 4.0, 2.0)	2.66 ddd(16.0, 4.0, 2.0)	2.85 ddd(16.2, 12.0, 5.5)
	2.83 ddd(16.0, 12.0, 6.0)	2.83 ddd(16.0, 11.5, 6.0)	
5	6.50[①], 6.64 s	6.52[①], 6.66 s	6.49[①], 6.51[①], 6.56, 6.68 s
8	6.48[①], 6.53 s	6.51[①], 6.54 s	6.49[①], 6.51[①], 6.56, 6.68 s
α			6.98, 7.06[①] d(15.5)
β			7.51[①], 7.52 d(15.5)
1′	5.43 d(3.5)	5.40 d(3.5)	5.42[①], 5.43 d(2.8)
3′	7.38 d(1.5)	7.37[①], 7.44 d(1.5)	7.39[①], 7.41, 7.43 d(1.8)
5′	2.72 m	2.75 m	–
6′	1.54 ddd(14.5, 11.0, 3.5)	1.57 ddd(14.5, 11.0, 3.5)	1.57 ddd(14.5, 11.7, 3.2)
	2.44 ddd(14.5, 11.5, 2.0)	2.42 ddd(14.5, 11.5, 3.0)	2.60 ddd(14.5, 11.5, 2.2)
8′	5.77 dt(17.0, 10.0)	5.78 dt(17.0, 10.0)	5.79 dt(17.2, 10.0)
9′	3.15 ddd(10.0, 6.0, 3.5)	3.13 ddd(10.0, 5.5, 3.5)	–
10′	5.43 dd(10.0, 2.0)	5.41 dd(10.0, 2.0)	5.34, 5.46[①] dd(10.0, 1.8)
	5.56 dd(17.0, 2.0)	5.54 dd(17.0, 2.0)	5.60 dd(17.2, 1.8)
1″	4.63[①], 4.67 d(8.0)	4.65[①], 4.69 d(8.0)	4.55, 4.61[①], 4.63 d(8.0)
2″	3.18 dd(9.0, 8.0)	3.21 dd(9.0, 8.0)	3.12 dd(9.0, 8.0)
3″	3.26~3.36 m	3.36 t(9.0)	–
4″	3.26~3.36 m	3.41 t(9.0)	–
5″	3.48 ddd(9.5, 6.0, 2.0)	3.49 ddd(9.0, 5.0, 2.0)	–
6″	3.78 dd(11.5, 6.0)	3.80~3.85 m	3.86, 3.88[①] dd(12.0, 2.0)
	4.17 dd(11.5, 2.0)	3.94 dd(11.0, 5.0)	–
1‴	4.35, 4.39[①] d(8.0)	4.87, 4.89[①] d(3.5)	
2‴	3.22 dd(9.0, 8.0)	3.40 dd(9.5, 3.5)	7.23, 7.24[①] d(1.8)
3‴	3.26~3.36 m	3.62~3.72 m	
4‴	3.26~3.36 m	3.31 m	
5‴	3.26~3.36 m	3.62~3.72 m	6.81, 6.83[①] d(8.0)
6‴	3.62~3.69 m	3.62~3.72 m	7.12[①], 7.13 dd(8.0, 1.8)
	3.88 dd(12.0, 1.5)	3.80~3.85 m	
NAc	2.17, 2.19[①] s	2.17, 2.19[①] s	
OMe			3.93 s
COOMe	3.66[①], 3.69 s	3.67[①], 3.70 s	3.64, 3.66[①], 3.67 s

[①] Signals of the major rotamer.

Table 2-13-10: ^{1}H NMR spectroscopic data of emetine-type alkaloids **2-13-20**~**2-13-22**.

H	2-13-20	2-13-21	2-13-22
1	5.71 dd(11.5, 2.8)	5.67 dd(11.5, 3.0)	5.69 dd(11.5, 3.0)
3	4.33 dd(14.0, 5.5)	4.09 dd(13.7, 6.5)	4.11 dd(14.0, 6.5)
4	–	2.36 ddd(16.0, 12.2, 6.5)	2.36 ddd(16.0, 12.3, 6.5)
		2.46 dd(16.0, 4.5)	2.52 dd(16.0, 4.5)

Table 2-13-10 (continued)

H	2-13-20	2-13-21	2-13-22
5	6.48, 6.50[①], 6.52, 6.65[①], 6.70, 6.71 s	6.24, 6.36[①], 6.45, 6.48[①], 6.50 s	6.43, 6.45, 6.48[①], 6.50[①], 6.53 s
8	6.48, 6.50[①], 6.52, 6.65[①], 6.70, 6.71 s	6.24, 6.36[①], 6.45, 6.48[①], 6.50 s	6.43, 6.45, 6.48[①], 6.50[①], 6.53 s
α	6.99, 7.08[①] d(15.2)	6.02, 6.06[①] d(12.8)	6.04, 6.06[①] d(12.5)
β	7.49, 7.52[①], 7.53 d(15.2)	6.61[①], 6.68 d(12.8)	6.63[①], 6.70 d(12.5)
1′	5.42[①], 5.43 d(2.7)	5.42, 5.43[①] d(3.0)	5.44[①], 5.45 d(2.8)
3′	7.39[①], 7.40, 7.42 d(1.8)	7.38, 7.39, 7.40[①] d(1.7)	7.40, 7.41[①] d(1.8)
6′	1.57 ddd(14.3, 12.0, 3.5)	1.57 ddd(15.0, 12.0, 3.0)	1.57 ddd(14.0, 12.0, 3.0)
	2.62 ddd(14.3, 11.5, 2.2)	2.58 ddd(15.0, 11.5, 2.0)	2.59 ddd(14.0, 11.5, 2.0)
8′	5.80 dt(17.2, 10.5)	5.80 dt(17.2, 10.0)	5.80 dt(17.2, 10.0)
10′	5.47 dd(10.5, 1.8)	5.47 dd(10.0, 1.8)	5.47 dd(10.0, 1.8)
	5.61 dd(17.2, 1.8)	5.60 dd(17.2, 1.8)	5.61 dd(17.2, 1.8)
1″	4.53, 4.61[①], 4.62 d(8.0)	4.61, 4.63[①], 4.64 d(8.0)	4.63[①], 4.64 d(8.0)
2″	3.12 dd(9.0, 8.0)	3.19 dd(9.0, 8.0)	3.19 dd(9.0, 8.0)
6″	3.87 dd(12.0, 2.0)	3.88[①], 3.93 dd(12.0, 2.0)	3.88 dd(12.0, 2.0)
	–	–	–
2‴	7.23, 7.24, 7.25[①] d(1.7)	6.86 d(1.8)	6.86 d(1.8)
5‴	6.80, 6.81[①] d(8.0)	6.69[①], 6.70 d(8.0)	6.68 d(8.0)
6‴	7.11 dd(8.0, 1.7)	6.80 dd(8.0, 1.8)	6.80 dd(8.0, 1.8)
OMe	3.80[①], 3.83 s, 3.93 s	3.22[①], 3.92 s	3.22[①], 3.93 s, 3.77[①], 3.79 s
COOMe	3.64, 3.65[①], 3.83 s	3.60, 3.64[①], 3.68 s	3.64[①], 3.67 s

[①] Signals of the major rotamer.

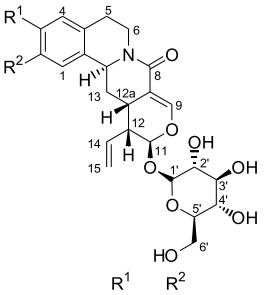
Table 2-13-11: Cos, MFs, and TSs of emetine-type alkaloids **2-13-23**~**2-13-43**.

No.	Compounds	MFs	Test solvents	References
2-13-23	methylisoalangiside	$C_{26}H_{33}NO_{10}$	CD_3OD	[415]
2-13-24	isoalangiside	$C_{25}H_{31}NO_{10}$	CD_3OD	[415]
2-13-25	3-*O*-demethyl-2-*O*-methylisoalangiside	$C_{25}H_{31}NO_{10}$	CD_3OD	[415]
2-13-26	alangiside	$C_{25}H_{31}NO_{10}$	DMSO-d_6	[416]
			CD_3OD	[416]
2-13-27	3-*O*-demethyl-2-*O*-methylalangiside	$C_{25}H_{31}NO_{10}$	DMSO-d_6	[416]
			CD_3OD	[416]
2-13-28	2-*O*-methylalangiside	$C_{26}H_{33}NO_{10}$	CD_3OD	[416]
2-13-29	demethylneoalangiside	$C_{24}H_{29}NO_{10}$	CD_3OD	[415]
2-13-30	neoalangiside	$C_{25}H_{31}NO_{10}$	CD_3OD	[415]
2-13-31	–	$C_{26}H_{31}NO_{11}$	CD_3OD	[417]
2-13-32	–	$C_{26}H_{31}NO_{11}$	CD_3OD	[417]
2-13-33	2′-*O*-*trans*-feruloylalangiside	$C_{35}H_{39}NO_{13}$	CD_3OD	[418]

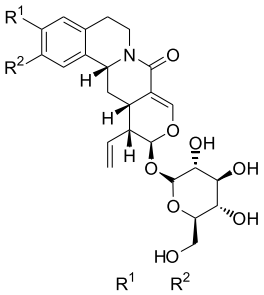
Table 2-13-11 (continued)

No.	Compounds	MFs	Test solvents	References
2-13-34	2′-*O*-*trans*-feruloyl-3-*O*-demethyl-2-*O*-methylalangiside	$C_{35}H_{39}NO_{13}$	CD_3OD	[418]
2-13-35	2′-*O*-*trans*-sinapoyldemethylalangiside	$C_{35}H_{39}NO_{14}$	CD_3OD	[418]
2-13-36	2′-*O*-*trans*-sinapoylalangiside	$C_{36}H_{41}NO_{14}$	CD_3OD	[418]
2-13-37	2′-*O*-*trans*-[4-(1,3-dihydroxypropoxy)-3-methoxycin-namoyl]alangiside	$C_{38}H_{45}NO_{15}$	CD_3OD	[418]
2-13-38	6′-*O*-*β*-D-glucopyranosylalangiside	$C_{31}H_{41}NO_{15}$	CD_3OD	[419]
2-13-39	3′-*O*-*β*-D-glucopyranosylalangiside	$C_{31}H_{41}NO_{15}$	CD_3OD	[419]
2-13-40	6′-*O*-*α*-D-glucopyranosylalangiside	$C_{31}H_{41}NO_{15}$	CD_3OD ①	[419]
2-13-41	6′-*O*-*α*-D-glucopyranosyl-3-*O*-demethyl-2-*O*-methyl-alangiside	$C_{31}H_{41}NO_{15}$	CD_3OD ①	[419]
2-13-42	6′-*O*-*α*-D-xylopyranosylalangiside	$C_{30}H_{39}NO_{14}$	CD_3OD	[419]
2-13-43	2-*O*-*β*-D-glucopyranosyldemethylalangiside	$C_{30}H_{39}NO_{15}$	CD_3OD	[413]

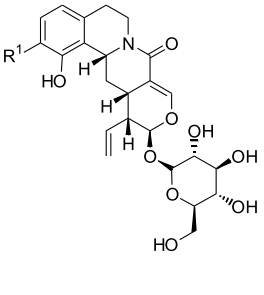
① Recorded at 50 °C.



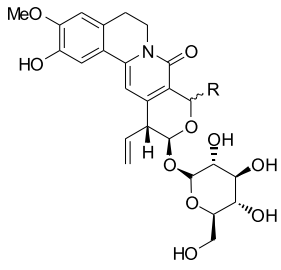
	R^1	R^2
2-13-23	OMe	OMe
2-13-24	OMe	OH
2-13-25	OH	OMe



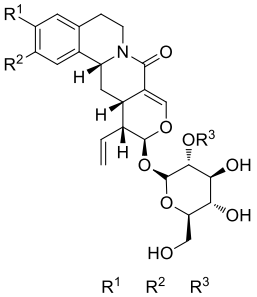
	R^1	R^2
2-13-26	OMe	OH
2-13-27	OH	OMe
2-13-28	OMe	OMe



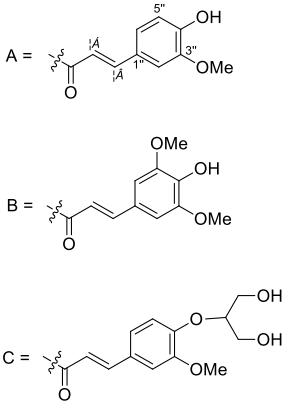
2-13-29 R = OH
2-13-30 R = OMe

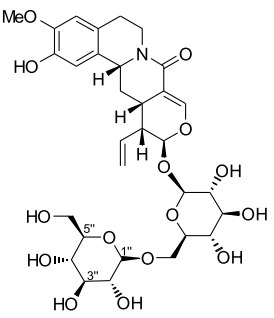


2-13-31 R = *α*-OMe
2-13-32 R = *β*-OMe

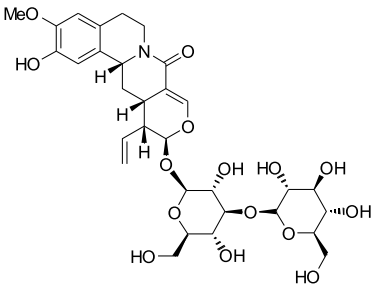


	R^1	R^2	R^3
2-13-33	OMe	OH	A
2-13-34	OH	OMe	A
2-13-35	OH	OH	B
2-13-36	OMe	OH	B
2-13-37	OMe	OH	C

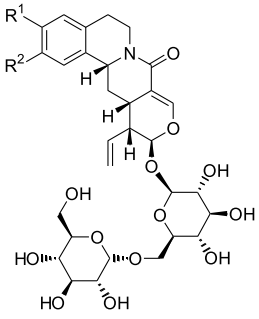




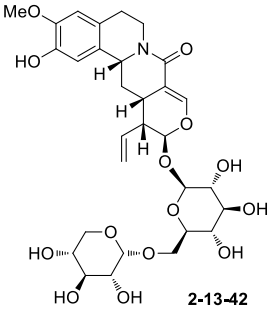
2-13-38



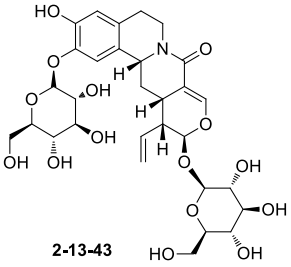
2-13-39



	R^1	R^2
2-13-40	OMe	OH
2-13-41	OH	OMe



2-13-42



2-13-43

Table 2-13-12: ^{1}H NMR spectroscopic data of emetine-type alkaloids **2-13-23**~**2-13-25**.

H	2-13-23	2-13-24	2-13-25
1	6.83 s	6.72 s	6.80 s
4	6.74 s	6.71 s	6.59 s
5	2.70 m	2.68 ddd(16.0, 4.5, 2.0)	2.62 ddd(16.0, 4.5, 2.0)
	2.99 ddd(15.5, 11.5, 5.5)	2.96 ddd(16.0, 11.0, 6.0)	2.92 ddd(16.0, 12.0, 6.0)
6	3.05 m	3.06 ddd(12.5, 11.0, 4.5)	3.04 td(12.0, 4.5)
	4.68 ddd(12.0, 5.5, 2.0)	4.64 ddd(12.5, 6.0, 2.0)	4.65 ddd(12.0, 6.0, 2.0)
9	7.33 d(2.5)	7.32 d(2.5)	7.33 d(2.5)
11	5.42 d(1.5)	5.41 d(2.5)	5.42 d(1.5)
12	2.70 m	2.67 ddd(10.5, 5.5, 1.5)	2.70 ddd(10.0, 5.5, 1.5)
12a	2.85 dddd(12.5, 5.5, 4.5, 2.5)	2.87 dddd(13.0, 5.5, 4.5, 2.5)	2.87 dddd(12.5, 5.5, 4.5, 2.5)
13	2.00 ddd(14.0, 12.5, 5.5)	1.96 ddd(14.0, 13.0, 5.5)	1.99 ddd(14.0, 12.5, 5.0)
	2.46 ddd(14.0, 4.5, 3.0)	2.35 ddd(14.0, 4.5, 3.0)	2.45 ddd(14.0, 4.5, 3.0)
13a	4.78 br t(4.5)	4.72 br t(4.5)	4.76 br t(4.0)
14	5.76 dt(17.0, 10.0)	5.66 dt(17.0, 10.5)	5.67 dt(17.0, 10.0)
15	5.32 dd(10.0, 2.0)	5.31 dd(10.5, 2.0)	5.32 dd(10.0, 2.0)
	5.39 dd(17.0, 2.0)	5.38 dd(17.0, 2.0)	5.39 dd(17.0, 2.0)
1′	4.61 d(8.0)	4.61 d(8.0)	4.62 d(8.0)
2′	3.06 dd(9.0, 8.0)	3.06 dd(9.0, 8.0)	3.06 dd(9.0, 8.0)

Table 2-13-12 (continued)

H	2-13-23	2-13-24	2-13-25
3′	3.30 t(9.0)	3.30 t(9.0)	3.29 t(9.0)
4′	3.23 t(9.0)	3.23 t(9.0)	3.23 t(9.5, 9.0)
5′	3.28 ddd(9.0, 6.0, 2.0)	3.28 ddd(9.0, 5.5, 2.0)	3.28 ddd(9.5, 5.5, 2.0)
6′	3.64 dd(12.0, 6.0)	3.65 dd(12.0, 5.5)	3.65 dd(12.0, 5.5)
	3.87 dd(12.0, 2.0)	3.87 dd(12.0, 2.0)	3.87 dd(12.0, 2.0)
OMe	3.80 s, 3.83 s	3.82 s	3.85 s

Table 2-13-13: ^{1}H NMR spectroscopic data of emetine-type alkaloids **2-13-26** and **2-13-27**.

H	2-13-26 (DMSO-d_6)	2-13-26 (CD_3OD)	2-13-27(DMSO-d_6)	2-13-27 (CD_3OD)
1	6.65 s	6.69 s	6.83 s	6.78 s
4	6.67 s	6.69 s	6.53 s	6.57 s
5	2.59 m	2.66 dt(15.5, 3.0)	2.52~2.60 m	2.60 dt(15.5, 3.0)
		2.76 ddd(15.5, 11.5, 4.0)		2.71 ddd(15.5, 12.5, 4.5)
6	2.72 dt(12.5, 7.5)	2.88 td(11.5, 3.0)	2.61~2.70 m	2.84 td(12.5, 3.0)
	4.63~4.68 m	4.71 ddd(11.5, 4.0, 3.0)	4.68~4.72 m	4.73 ddd(12.5, 4.5, 3.0)
9	7.26 d(2.5)	7.41 d(2.5)	7.28 d(2.5)	7.41 d(2.5)
11	5.37 d(1.5)	5.49 d(1.5)	5.39 d(1.5)	5.49 d(1.5)
12	2.65 ddd(9.5, 5.5, 1.5)	2.70 ddd(10.0, 5.5, 1.5)	2.61~2.70 m	2.71 ddd(10.0, 5.5, 1.5)
12a	3.06 m	3.19 dddd(13.0, 5.5, 3.5, 2.5)	3.10 m	3.21 dddd(13.0, 5.5, 3.5, 2.5)
13	1.16 td(13.0, 11.5)	1.35 td(13.0, 11.0)	1.18 td(13.0, 11.5)	1.36 td(13.0, 11.5)
	2.25 dt(13.0, 3.5)	2.30 dt(13.0, 3.5)	2.39 dt(13.0, 3.5)	2.37 dt(13.0, 3.5)
13a	4.63~4.68 m	4.72 dd(11.0, 3.5)	4.68~4.72 m	4.76 dd(11.5, 3.5)
14	5.43 dt(17.0, 10.0)	5.52 dt(17.0, 10.0)	5.46 dt(17.0, 10.0)	5.53 dt(17.0, 10.0)
15	5.15 dd(10.0, 2.0)	5.19 dd(10.0, 2.0)	5.17 dd(10.0, 2.0)	5.19 dd(10.0, 2.0)
	5.27 dd(17.0, 2.0)	5.28 dd(17.0, 2.0)	5.28 dd(17.0, 2.0)	5.28 dd(17.0, 2.0)
1′	4.50 d(7.8)	4.69 d(8.0)	4.52 d(8.0)	4.69 d(8.0)
2′	2.98 m	3.20 dd(9.0, 8.0)	2.98 m	3.19 dd(9.0, 8.0)
3′	3.13~3.19 m	3.38 t(9.0)	3.14~3.20 m	3.38 t(9.0)
4′	3.03 m	3.29 dd(9.5, 9.0)	3.04 m	3.28 dd(9.5, 9.0)
5′	3.13~3.19 m	3.32 ddd(9.5, 5.5, 2.0)	3.14~3.20 m	3.33 ddd(9.5, 5.5, 2.0)
6′	3.42 m	3.68 dd(12.0, 5.5)	3.44 m	3.67 dd(12.0, 5.5)
	3.68 m	3.90 dd(12.0, 2.0)	3.69 m	3.89 dd(12.0, 2.0)
OMe	3.72 s	3.83 s	3.74 s	3.82 s

Table 2-13-14: ^{1}H NMR spectroscopic data of emetine-type alkaloids **2-13-28**~**2-13-32**.

H	2-13-28	2-13-29	2-13-30	2-13-31	2-13-32
1	6.73 s			7.23 s	7.14 s
3		6.65 d(8.0)	6.81 d(8.0)		

Table 2-13-14 (continued)

H	2-13-28	2-13-29	2-13-30	2-13-31	2-13-32
4	6.82 s	6.48 d(8.0)	6.61 d(8.0)	6.89 s	6.88 s
5	2.68 dt(15.0, 2.5) 2.78 ddd(15.0, 11.5, 3.5)	2.58~2.71 m	2.61~2.73 m	2.92 t(6.5)	2.90 t(6.5)
6	2.85 td(11.5, 2.5) –	2.58~2.71 m –	2.61~2.73 m –	4.17 dt(13.0, 6.5) 4.24 dt(13.0, 6.5)	4.17 dt(13.0, 6.5) 4.21 dt(13.0, 6.5)
9	7.42 d(2.5)	7.45 d(2.5)	7.45 d(2.5)	5.46 s	5.63 s
11	5.50 d(1.5)	5.50 d(1.5)	5.50 d(2.0)	5.53 d(1.0)	5.49 d(7.5)
12	2.72 ddd(10.0, 5.5, 1.5)	2.58~2.71 m	2.61~2.73 m	3.49 br d(8.0)	3.41 br t(8.5)
12a	3.23 dddd(13.0, 5.5, 3.5, 2.5)	3.24 m	3.24 m		
13	1.36 td(13.0, 11.5) 2.39 dt(13.0, 3.5)	1.14 td(13.0, 11.0) 2.77 ddd(13.0, 3.5, 2.0)	1.14 td(13.0, 11.0) 2.76 ddd(13.0, 4.0, 2.5)	6.61 s	6.57 s
13a	–	4.98 dd(11.0, 2.0)	4.99 dd(11.0, 3.5)		
14	5.53 dt(17.0, 10.0)	5.50 dt(17.0, 10.5)	5.50 dt(17.0, 10.0)	5.83 ddd(17.0, 10.5, 8.0)	5.78 dt(17.5, 9.5)
15	5.19 dd(10.0, 2.0) 5.29 dd(17.0, 2.0)	5.15 dd(10.5, 1.5) 5.22 dd(17.0, 1.5)	5.15 dd(10.0, 2.0) 5.22 dd(17.0, 2.0)	5.20 br d(10.5) 5.24 dt(17.0, 1.0)	5.39~5.44 m
1′	4.70 d(8.0)	4.71 d(7.5)	4.70 d(8.0)	4.78 d(8.0)	4.80 d(8.0)
2′	3.19 dd(9.0, 8.0)	3.23 dd(9.0, 7.5)	3.22 dd(9.0, 8.0)	3.17 dd(9.0, 8.0)	3.23 dd(9.0, 8.0)
3′	3.38 t(9.0)	3.39 t(9.0)	3.38 t(9.0)	3.42 t(9.0)	3.41 t(9.0)
4′	3.28 dd(9.5, 9.0)	–	–	3.26 dd(9.5, 9.0)	–
5′	3.33 ddd(9.5, 5.5, 2.0)	–	–	3.37 ddd(9.5, 6.0, 2.0)	–
6′	3.67 dd(12.0, 5.5) 3.90 dd(12.0, 2.0)	3.69 dd(12.0, 5.5) 3.90 dd(12.0, 1.5)	3.68 dd(12.0, 5.5) 3.90 dd(12.0, 2.0)	3.67 dd(12.0, 6.0) 3.91 dd(12.0, 2.0)	3.68 dd(12.0, 5.5) 3.87 dd(12.0, 2.0)
OMe	3.80 s, 3.81 s		3.84 s	3.66 s, 3.92 s	3.57 s, 3.91 s

Table 2-13-15: ^{1}H NMR spectroscopic data of emetine-type alkaloids **2-13-33**~**2-13-35**.

H	2-13-33	2-13-34	2-13-35
1	6.63 s	6.75 s	6.43 s or 6.60 s
4	6.58 s	6.46 s	6.43 s or 6.60 s
5	2.29 dt(15.5, 3.0) 2.47 ddd(15.5, 11.5, 4.5)	2.24 dt(15.5, 3.0) 2.43 ddd(15.5, 11.5, 4.5)	2.21 dt(15.5, 3.5) 2.40 ddd(15.5, 11.5, 3.5)
6	1.80 ddd(12.5, 11.5, 3.0) 4.08 ddd(12.5, 4.5, 3.0)	1.74 ddd(12.5, 11.5, 3.0) 4.12 ddd(12.5, 4.5, 3.0)	1.75 ddd(12.5, 11.5, 3.5) 4.07 dt(12.5, 3.5)
9	7.37 d(2.5)	7.38 d(2.5)	7.37 d(2.5)
11	5.44 d(1.5)	5.45 d(2.0)	5.45 d(1.5)
12	2.63 ddd(10.0, 5.5, 1.5)	2.64 ddd(10.0, 5.5, 2.0)	2.62 ddd(10.0, 5.5, 1.5)

Table 2-13-15 (continued)

H	2-13-33	2-13-34	2-13-35
12a	2.86 dddd(13.0, 5.5, 3.5, 2.5)	2.88 dddd(13.0, 5.5, 3.5, 2.5)	2.85 dddd(13.0, 5.5, 3.5, 2.5)
13	1.20 td(13.0, 11.5)	1.22 td(13.0, 11.5)	1.22 td(13.0, 11.5)
	2.18 dt(13.0, 3.5)	2.26 dt(13.0, 3.5)	2.16 dt(13.0, 3.5)
13a	4.38 dd(11.5, 3.5)	4.44 br d(11.5)	4.35 dd(11.5, 3.5)
14	5.45 dt(17.0, 10.0)	5.46 dt(17.0, 10.0)	5.46 dt(17.0, 10.0)
15	5.16 dd(10.0, 2.0)	5.16 dd(10.0, 2.0)	5.16 dd(10.0, 2.0)
	5.25 dd(17.0, 2.0)	5.26 dd(17.0, 2.0)	5.25 dd(17.0, 2.0)
α	6.30 d(15.5)	6.30 d(16.0)	6.33 d(15.5)
β	7.60 d(15.5)	7.61 d(16.0)	7.60 d(15.5)
$6'$	3.93 br d(12.0)	3.93 dd(12.0, 1.5)	3.93 br d(12.0)
$2''$	7.16 d(2.0)	7.15 d(2.0)	6.85 s
$5''$	6.76 d(8.0)	6.75 d(8.0)	
$6''$	7.02 dd(8.0, 2.0)	7.01 dd(8.0, 2.0)	6.85 s
OMe	3.79 s, 3.86 s	3.83 s, 3.85 s	3.79 s, 3.79 s

Table 2-13-16: ^{1}H NMR spectroscopic data of emetine-type alkaloids **2-13-36** and **2-13-37**.

H	2-13-36	2-13-37	H	2-13-36	2-13-37
1	6.64 s	6.63 s	α	6.33 d(15.5)	6.37 d(16.0)
4	6.58 s	6.58 s	β	7.61 d(15.5)	7.63 d(16.0)
5	2.30 dt(15.5, 3.0)	2.31 dt(15.5, 3.0)	$1'$	4.89 d(8.0)	–
	2.47 ddd(15.5, 11.5, 4.5)	2.47 ddd(15.5, 12.0, 4.5)			
6	1.73 ddd(13.0, 11.5, 3.0)	1.75 ddd(12.5, 12.0, 3.0)	$2'$	4.92 t(8.0)	–
	4.12 ddd(13.0, 4.5, 3.0)	4.09 ddd(12.5, 4.5, 3.0)			
9	7.38 d(2.5)	7.38 d(2.5)	$6'$	3.93 br d(12.0)	3.93 dd(12.0, 1.0)
11	5.45 d(1.5)	5.45 d(1.5)	$2''$	6.86 s	7.22 d(2.0)
12	2.63 ddd(10.0, 5.5, 1.5)	2.63 ddd(10.0, 5.5, 1.5)	$5''$		7.04 d(8.5)
12a	2.85 dddd	2.85 m	$6''$	6.86 s	7.10 dd(8.5, 2.0)
	(13.0, 5.5, 3.5, 2.5)				
13	1.21 td(13.0, 11.5)	1.21 td(13.0, 11.5)	$1'''$, $3'''$		3.71, 3.75 dd(17.0, 5.0)
	2.17 dt(13.0, 3.5)	2.18 dt(13.0, 3.0)			3.74, 3.77 dd(17.0, 5.0)
13a	4.37 dd(11.5, 3.5)	4.37 dd(11.5, 3.0)	$2'''$		4.33 q(5.0)
14	5.45 dt(17.0, 10.0)	5.45 dt(17.0, 10.0)	OMe	3.79 s, 3.79 s, 3.79 s	3.79 s, 3.86 s
15	5.16 dd(10.0, 2.0)	5.16 dd(10.0, 2.0)			
	5.25 dd(17.0, 2.0)	5.25 dd(17.0, 2.0)			

Table 2-13-17: ^{1}H NMR spectroscopic data of emetine-type alkaloids **2-13-38**~**2-13-40**.

H	2-13-38	2-13-39	2-13-40
1	6.69 s	6.69 s	6.69 s
4	6.69 s	6.70 s	6.70 s
5	2.66 dt(15.5, 3.0)	2.67 dt(15.5, 3.5)	2.66 dt(15.5, 3.5)
	2.76 ddd(15.5, 11.0, 4.0)	2.76 ddd(15.5, 11.0, 4.5)	2.77 ddd(15.5, 11.0, 4.5)
6	2.89 ddd(12.5, 11.0, 3.0)	2.89 ddd(12.0, 11.0, 3.5)	2.90 ddd(12.5, 11.0, 3.5)
	4.71 m	4.68~4.75 m	4.69 ddd(12.5, 4.5, 3.5)
9	7.41 d(2.5)	7.41 d(2.5)	7.41 d(2.5)
11	5.45 d(1.5)	5.49 d(2.0)	5.42 d(1.5)
12	2.71 br dd(10.0, 5.5)	2.71 ddd(10.0, 5.5, 1.5)	2.71 ddd(10.0, 5.5, 1.5)
12a	3.18 dddd(13.0, 5.5, 3.5, 2.5)	3.17 m	3.18 dddd(13.0, 5.5, 3.5, 2.5)
13	1.35 td(13.0, 12.0)	1.35 td(13.0, 11.5)	1.38 td(13.0, 11.5)
	2.31 dt(13.0, 3.5)	2.31 dt(13.0, 3.5)	2.31 dt(13.0, 3.5)
13a	4.72 br d(12.0)	4.68~4.75 m	4.72 br d(11.5)
14	5.51dt(17.0, 10.0)	5.52 dt(17.0, 10.0)	5.52 dt(17.0, 10.0)
15	5.20 dd(10.0, 1.5)	5.19 dd(10.0, 2.0)	5.19 dd(10.0, 2.0)
	5.30 dd(17.0, 1.5)	5.28 dd(17.0, 2.0)	5.30 dd(17.0, 2.0)
1′	4.70 d(8.0)	4.74 d(8.0)	4.72 d(8.0)
2′	3.21 br t(8.5)	3.41 dd(9.0, 8.0)	3.24 dd(9.0, 8.0)
3′	3.25~3.41 m	3.59 t(9.0)	3.40 t(9.0)
4′	3.25~3.41 m	3.42 dd(10.0, 9.0)	3.43 t(9.0)
5′	3.52 ddd(9.5, 5.5, 2.0)	3.37 ddd(10.0, 5.5, 2.0)	3.54 ddd(9.0, 5.0, 2.0)
6′	3.80 dd(11.5, 5.5)	3.70 dd(12.0, 5.5)	3.80 dd(11.0, 2.0)
	4.18 dd(11.5, 2.0)	3.91 dd(12.0, 2.0)	3.97 dd(11.0, 5.0)
1″	4.37 d(7.5)	4.57 d(8.0)	4.87 d(3.5)
2″	3.23 br t(8.5)	3.26 dd(9.0, 8.0)	3.40 dd(9.5, 3.5)
3″	3.25~3.41 m	3.38 t(9.0)	3.66 t(9.5)
4″	3.25~3.41 m	3.27 dd(10.0, 9.0)	3.33 br t(9.5)
5″	3.25~3.41 m	3.31 m	3.68 ddd(9.0, 5.0, 2.0)
6″	3.67 dd(12.0, 5.5)	3.63 dd(12.0, 6.0)	3.70 dd(11.0, 5.0)
	3.87 dd(12.0, 2.0)	3.88 dd(12.0, 2.0)	3.82 dd(11.0, 2.0)
OMe	3.83 s	3.83 s	3.83 s

Table 2-13-18: ^{1}H NMR spectroscopic data of emetine-type alkaloids **2-13-41**~**2-13-43**.

H	2-13-41	2-13-42	2-13-43
1	6.79 s	6.69 s	7.10 s
4	6.59 s	6.69 s	6.63 s
5	2.60 dt(15.5, 3.0)	2.66 dt(15.5, 3.0)	2.64 dt(16.0, 3.5)
	2.73 ddd(15.5, 11.5, 4.5)	2.76 ddd(15.5, 11.0, 4.5)	2.73 ddd(16.0, 11.0, 3.5)
6	2.87 ddd(12.5, 11.5, 3.0)	2.89 ddd(12.5, 11.0, 3.0)	2.88 ddd(12.5, 11.0, 3.5)
	4.72 ddd(12.5, 4.5, 3.0)	4.70 ddd(12.5, 4.5, 3.0)	4.71 dt(12.5, 3.5)
9	7.41 d(2.5)	7.41 d(2.5)	7.41 d(2.5)
11	5.42 d(1.5)	5.42 d(1.5)	5.49 d(2.0)

Table 2-13-18 (continued)

H	2-13-41	2-13-42	2-13-43
12	2.73 ddd(10.0, 5.5, 1.5)	2.71 ddd(10.0, 5.5, 1.5)	2.71 ddd(10.0, 5.5, 2.0)
12a	3.20 dddd(13.0, 5.5, 3.5, 2.5)	3.18 dddd(13.0, 5.5, 3.5, 2.5)	3.20 dddd(13.0, 5.5, 3.5, 2.5)
13	1.39 td(13.0, 11.5)	1.36 td(13.0, 11.5)	1.36 td(13.0, 11.5)
	2.38 dt(13.0, 3.5)	2.31 dt(13.0, 3.5)	2.41 dt(13.0, 3.5)
13a	4.77 dd(11.5, 3.5)	4.72 dd(11.5, 3.5)	4.75 dd(11.5, 3.5)
14	5.53 dt(17.0, 10.0)	5.51 dt(17.0, 10.0)	5.52 dt(17.0, 10.0)
15	5.20 dd(10.0, 1.5)	5.19 dd(10.0, 2.0)	5.19 dd(10.0, 2.0)
	5.31 dd(17.0, 1.5)	5.30 dd(17.0, 2.0)	5.30 dd(17.0, 2.0)
1′	4.73 d(8.0)	4.71 d(8.0)	4.69 d(8.0)
2′	3.23 dd(9.0, 8.0)	3.23 dd(9.0, 8.0)	3.19 dd(9.0, 8.0)
3′	3.40 t(9.0)	3.39 t(9.0)	3.38 t(9.0)
4′	3.43 t(9.0)	3.41 t(9.0)	3.28 dd(10.0, 9.0)
5′	3.54 ddd(9.0, 4.5, 2.0)	3.53 ddd(9.5, 5.5, 2.0)	3.33 or 3.44 ddd(10.0, 6.0, 2.0)
6′	3.80 dd(11.0, 2.0)	3.77 dd(11.0, 2.0)	3.67 or 3.69 dd(12.0, 6.0)
	3.97 dd(11.0, 4.5)	3.91 dd(11.0, 5.0)	3.90 or 3.92 dd(12.0, 2.0)
1″	4.87 d(3.5)	4.80 d(3.5)	4.72 d(7.5)
2″	3.40 dd(9.5, 3.5)	3.36 dd(9.5, 3.5)	3.49 dd(9.0, 7.5)
3″	3.66 t(9.5)	3.59 dd(9.5, 9.0)	3.46 t(9.0)
4″	3.33 t(9.5)	3.47 ddd(10.5, 9.0, 5.5)	3.37 dd(10.0, 9.0)
5″	3.69 ddd(9.5, 5.0, 2.0)	3.54 dd(10.5, 5.5)	3.33 or 3.44 ddd(10.0, 6.0, 2.0)
		3.58 t(10.5)	
6″	3.70 dd(11.5, 5.0)		3.67 or 3.69 dd(12.0, 6.0)
	3.81 dd(11.5, 2.0)		3.90 or 3.92 dd(12.0, 2.0)
OMe	3.84 s	3.83 s	

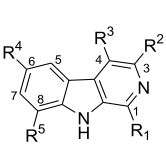
Bibliography

[408] Itoh A, Ikuta Y, Tanahashi T, et al. J Nat Prod, 2000, 63: 723.
[409] Itoh A, Ikuta Y, Baba Y, et al. Phytochemistry, 1999, 52: 1169.
[410] Muhammad I, Dunbar DC, Khan SI, et al. J Nat Prod, 2003, 66: 962.
[411] Ma WW, Anderson JE, Mckenzie AT, et al. J Nat Prod, 1990, 53: 1009.
[412] Ito A, Lee YH, Chai HB, et al. J Nat Prod, 1999, 62: 1346.
[413] Itoh A, Baba Y, Tanahashi T, et al. Phytochemistry, 2002, 59: 91.
[414] Nagakura N, Itoh A, Tanahashi T. Phytochemistry, 1993, 32: 761.
[415] Itoh A, Tanahashi T, Nagakura N. J Nat Prod, 1995, 58: 1228.
[416] Itoh A, Tanahashi T, Nagakura N, et al. Phytochemistry, 1994, 36: 383.
[417] Itoh A, Tanahashi T, Nagakura N. Heterocycles, 1998, 48: 499.
[418] Itoh A, Tanahashi T, Nagakura N. Phytochemistry, 1996, 41: 651.
[419] Itoh A, Tanahashi T, Nagakura N. Phytochemistry, 1997, 46: 1225.

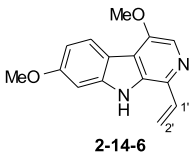
2.14 Carboline alkaloids

Table 2-14-1: Cos, MFs, and TSs of carboline alkaloids **2-14-1**~**2-14-16**.

No.	Compounds	MFs	Test solvents	References
2-14-1	taraxacine A	$C_{12}H_{10}N_2O$	$CDCl_3$	[420]
2-14-2	taraxacine B	$C_{13}H_{10}N_2O_3$	$CDCl_3$	[420]
2-14-3	picrasidine P	$C_{13}H_{12}N_2O_2$	$CDCl_3$	[421]
2-14-4	1-acetyl-8-hydroxyl-*β*-carboline	$C_{13}H_{10}N_2O_2$	$CDCl_3$-DMSO-d_6	[422]
2-14-5	6-methoxy-1-methyl-9*H*-pyrido[3,4-*b*]indole	$C_{13}H_{12}N_2O$	DMSO-d_6	[423]
2-14-6	4,7-dimethoxy-1-vinyl-*β*-carboline	$C_{15}H_{14}N_2O_2$	$CDCl_3$	[424]
2-14-7	7-hydroxy-*β*-carboline 1-propionic acid	$C_{14}H_{12}N_2O_3$	DMSO-d_6	[425]
2-14-8	dichotomine A	$C_{14}H_{12}N_2O_3$	DMSO-d_6	[426]
2-14-9	dichotomine C	$C_{15}H_{14}N_2O_4$	DMSO-d_6	[426]
2-14-10	dichotomine D	$C_{18}H_{20}N_2O_4$	CD_3OD	[426]
2-14-11	4-(9*H*-*β*-carbolin-1-yl)-4-oxobutyric acid	$C_{15}H_{12}N_2O_3$	DMSO-d_6	[427]
2-14-12	*n*-pentyl *β*-carboline-1-propionate	$C_{19}H_{22}N_2O_2$	CD_3COCD_3	[428]
2-14-13	dichotomideI	$C_{18}H_{17}N_3O_4$	$CDCl_3$	[426]
2-14-14	dichotomideII	$C_{18}H_{17}N_3O_4$	$CDCl_3$	[426]
2-14-15	1,2,3,4-tetrahydro-1,3,4-trioxo-*β*-carboline	$C_{11}H_6N_2O_3$	DMSO-d_6	[421]
2-14-16	picrasidine V	$C_{12}H_8N_2O_4$	DMSO-d_6	[421]



	R^1	R^2	R^3	R^4	R^5
2-14-1	OMe	H	H	H	H
2-14-2	OMe	COOH	H	H	H
2-14-3	H	H	OMe	H	OMe
2-14-4	Ac	H	H	H	OH
2-14-5	Me	H	H	OMe	H



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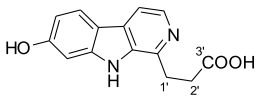

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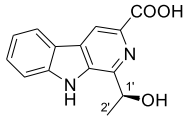

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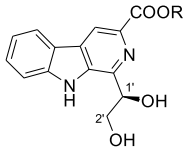
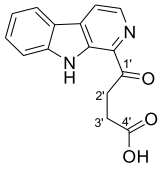
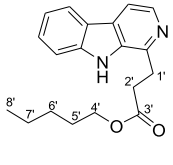


2-14-9 R = Me
2-14-10 R = $(CH_2)_3CH_3$



2-14-11



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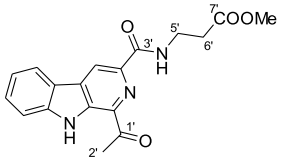

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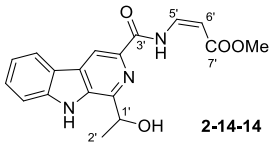
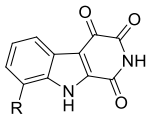


2-14-14



2-14-15 R = H
2-14-16 R = OMe

Table 2-14-2: ^{1}H NMR spectroscopic data of carboline alkaloids **2-14-1**~**2-14-5**.

H	2-14-1	2-14-2	2-14-3	2-14-4	2-14-5
1	4.30 s(OMe)	4.24 s(OMe)	8.61 s	2.85 s(Ac)	2.5 (Me)
3	8.90 d(5.6)	13.46 br s(COOH)	8.07 s	8.47 d(4.8)	6.9 d(6)
4	8.54 d(5.6)	9.07 s	4.15 s(OMe)	8.17 d(4.8)	7.2 d(6)
5	8.31 d(8.0)	8.32 d(8.0)	7.91 dd(7.8, 1.0)	7.65 d(7.8)	5.8 d(1)
6	7.56 d(8.0)	7.54 t(8.0)	7.22 t(7.8)	7.15 dd(7.8, 7.8)	3.6 s(OMe)
7	7.85 t(8.0)	7.85 t(8.0)	6.99 dd(7.8, 1.0)	7.02 d(7.8)	6.1 dd(1)
8	7.72 d(8.0)	7.97 d(8.0)	4.03 s(OMe)	11.31 br s	6.7 d(8)
9-NH	10.29 s	10.14 s	8.49 s	9.71 br s	

Table 2-14-3: ^{1}H NMR spectroscopic data of carboline alkaloids **2-14-6**~**2-14-10**.

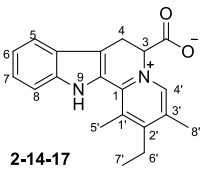
H	2-14-6	2-14-7	2-14-8	2-14-9	2-14-10
3	8.02 s	8.13 d(5.3)		3.97 s(COOMe)	
4		7.74 d(5.3)	8.81 s	9.09 s	9.20 s
5	8.18 d(9.5)	7.93 d(8.6)	8.36 or 7.73 d(7.0)	8.34 or 7.75 d(7.3)	8.34 or 7.73 d(7.8)
6	6.88 dd(9.5, 2.0)	6.69 dd(8.6, 2.0)	7.29 or 7.58 m	7.39 or 7.60 m	7.39 or 7.63 m
7			7.29 or 7.58 m	7.39 or 7.60 m	7.39 or 7.63 m
8	6.85 d(2.0)	6.90 d(2.0)	8.36 or 7.73 d(7.0)	8.34 or 7.75 d(7.3)	8.34 or 7.73 d(7.8)
1′	7.17 dd(17.2, 11.0)	2.82 t(7.3)	5.26 m	5.94 m	5.94 t(5.4)
2′	5.46 dd(11.0, 1.5) 6.23 dd(17.6, 1.5)	3.27 t(7.3)	1.57 d(7.4)	4.61 m	4.66 m
NH	9.92 s	11.28 br s	11.70 br s	12.60 br s	12.60 br s
OMe	4.08 s, 3.80 s				
OC_4H_9					0.86 t(7.3) 1.43 m, 1.73 m 4.51 t(6.8)

Table 2-14-4: ^{1}H NMR spectroscopic data of carboline alkaloids **2-14-11**~**2-14-16**.

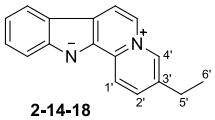
H	2-14-11	2-14-12	2-14-13	2-14-14	2-14-15	2-14-16
3	8.49 d(4.5)	7.97 d(5.3)				
4	8.43 d(4.5)	8.29 d(5.3)	9.10 s	8.92 s		
5	8.28 d(8.0)	8.21 d(7.4)	8.22 or 7.62 d(7.8)	8.40 or 7.78 d(7.8)	8.36 ddd(7.7, 1.3, 0.9)	7.62 dd(8.1, 0.7)
6	7.28 t(8.0)	7.26 dd(7.4, 7.0)	7.40 or 7.65 m	7.30 or 7.59 m	7.37 ddd(7.7, 1.3, 0.9)	7.27 t(8.1)
7	7.57 t(8.0)	7.54 dd(8.3, 7.0)	7.40 or 7.65 m	7.30 or 7.59 m	7.44 ddd(8.1, 7.1, 1.3)	6.95 dd(8.1, 0.7)
8	7.76 d(8.0)	7.63 d(8.3)	8.22 or 7.62 d(7.8)	8.40 or 7.78 d(7.8)	7.63 ddd(8.1, 1.1, 0.9)	3.97 s(OMe)
1′		3.45 t(7.3)		5.31 m		
2′	3.56 d(6.5)	2.98 t(7.3)	2.94 s	1.72 d(6.5)		
3′	2.68 d(6.5)					
4′		4.02 t(6.5)	8.63 br s(NH)	12.40 br d(1.0)		
5′		1.53 m	3.86 dt(1.0, 6.2)	7.78 d(8.9)		
6′		1.22~1.35 m	2.75 t(6.2)	5.31 d(8.9)		
7′		1.22~1.35 m				
8′		0.84 t(7.4)				
9-NH		10.85 br s	10.40 br s	11.80 br s	11.87 br s	11.71 br s
2-NH					13.35 br s	13.54 br s
COOMe			3.76 s	3.75 s		

Table 2-14-5: Cos, MFs, and TSs of carboline alkaloids **2-14-17**~**2-14-26**.

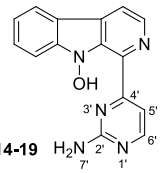
No.	Compounds	MFs	Test solvents	References
2-14-17	javacarboline	$C_{20}H_{20}N_2O_2$	DMSO-d_6	[429]
2-14-18	flavopereirine	$C_{17}H_{14}N_2$	DMSO-d_6	[430]
2-14-19	*N*-hydroxyannomontine	$C_{15}H_{11}N_5O$	CD_3COCD_3	[431]
2-14-20	(–)-isocyclocapitelline	$C_{20}H_{24}N_2O_2$	$CDCl_3$	[432]
2-14-21	(+)-cyclocapitelline	$C_{20}H_{24}N_2O_2$	$CDCl_3$	[432]
2-14-22	chrysotricine	$C_{21}H_{26}N_2O_2$	$CDCl_3$	[433]
			CD_3OD	[433]
2-14-23	hedyocapitine	$C_{20}H_{18}N_2O$	CD_3OD	[434]
2-14-24	hedyocapitelline	$C_{22}H_{30}N_2O_2$	$CDCl_3$	[434]
2-14-25	tangutorine	$C_{20}H_{24}N_2O$	$CDCl_3$-CD_3OD	[435]
2-14-26	kirondrine	$C_{30}H_{28}N_4O_4$	$CDCl_3$	[424]



2-14-17



2-14-18



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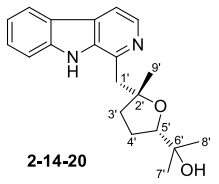
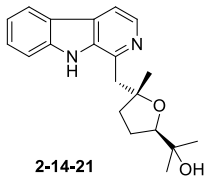
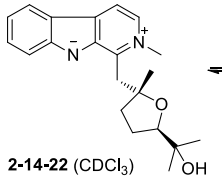
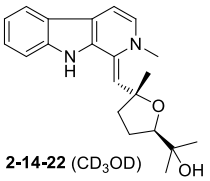
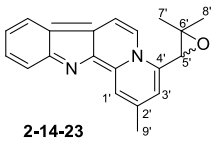
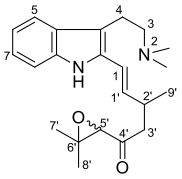


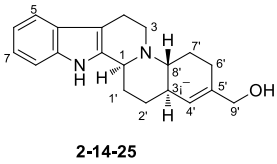












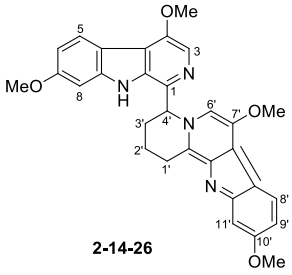


Table 2-14-6: ^{1}H NMR spectroscopic data of carboline alkaloids **2-14-17**~**2-14-20**.

H	2-14-17	2-14-18	2-14-19	2-14-20
3	5.89 dd(6.1, 1.2)	8.93 d(6.7)	8.53 d(5.0)	8.35 d(5.3)
4	3.84 dd(16.8, 1.2) 3.55 dd(16.8, 6.1)	8.75 d(6.7)	8.31 d(5.0)	7.84 d(5.3)
5	7.74 br d(8.0)	8.40 d(8.1)	8.31 ddd(7.9, 1.1, 0.8)	8.10 d(7.9)
6	7.17 ddd(8.0, 7.1, 0.9)	7.40 t(7.7)	7.34 ddd(7.9, 6.5, 1.7)	7.25
7	7.34 ddd(8.2, 7.1, 0.7)	7.67 td(8.1, 1.0)	7.70 ddd(8.3, 6.5, 1.1)	7.51
8	7.64 dt(8.2, 0.9)	7.86 d(8.1)	7.67 ddd(8.3, 1.7, 0.8)	7.51
1′		9.02 d(8.7)		3.49 d(13.7) 3.22 d(13.7)
2′		8.32 d(8.7)		
3′				2.10, 1.95
4′	8.84 s	9.30 s		2.10, 1.98
5′	2.84 s	2.93 q(7.5)	8.02 d(5.2)	3.91 m
6′	2.96 q(7.6)	1.37 t(7.5)	8.57 d(5.2)	
7′	1.21 t(7.6)		6.83 br s	1.31 s
8′	2.47 s			1.20 s
9′				1.25 d(0.6)
9-NH	11.75 s			9.91 s
9-NOH			15.08 br s	

Table 2-14-7: ^{1}H NMR spectroscopic data of carboline alkaloids **2-14-21** and **2-14-22**.

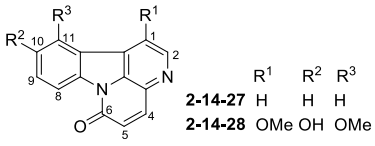
H	2-14-21	2-14-22 ($CDCl_3$)	2-14-22 (CD_3OD)
3	8.37 d(5.4)	8.02 d	7.82 d(6.4)
4	7.87 d(5.4)	8.09 d	8.13 d(6.4)
5	8.12 d(7.9)	8.13 d(8.1)	8.18 dt(1.2, 8.1)
6	7.27 dd(7.9, 7.3)	7.21 t	7.14 ddd(1.0, 6.8, 8.1)
7	7.52 dd(8.2, 7.3)	7.59 t	7.52 ddd(1.2, 6.8, 8.5)
8	7.45 d(8.2)	7.61 d	7.79 dt(1.0, 8.5)
1′	3.54 d(14.4), 3.42 d(14.4)	3.18 br, 3.88 br	3.78 s
3′	2.02, 1.95	2.24 m, 2.15 m	2.19 m, 1.96 m
4′	1.95, 1.74	1.74 m, 1.49 m	1.72 m, 1.47 m
5′	3.59 dd(10.0, 5.7)	4.27 m	3.20 m
7′	1.32 s	1.12 s	1.12 s
8′	1.11 s	1.02 s	1.07 s
9′	1.28 s	1.40 s	1.38 s
9-NH	9.84 s		
NMe		4.42 s	4.44 s

Table 2-14-8: ^{1}H NMR spectroscopic data of carboline alkaloids **2-14-23**~**2-14-26**.

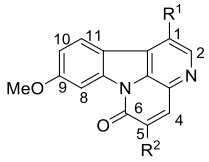
H	2-14-23	2-14-24	2-14-25	2-14-26
1		6.54 dd(16.1, 1.2)	3.54 d(11.3)	
3	8.22 d(6.9)	2.52 m	2.45 m, 3.61 m	7.80 s
4	8.37 d(6.9)	2.94 m	2.84 m, 2.94 m	4.02 s(OMe)
5	8.18 d(8.0)	7.53 d(7.9)	7.14 d(7.8)	8.06 d(8.6)
6	7.26 dd(8.0, 6.9)	7.07 dd(7.9, 7.1)	7.03 t(7.3)	6.92 br d(8.6)
7	7.53 dd(8.3, 6.9)	7.15 dd(7.8, 7.1)	7.10 t(7.3)	3.90 s(OMe)
8	7.77 d(8.3)	7.27 d(7.8)	7.33 t(8.3)	7.17 br s
1′	8.57 br s	5.92 dd(16.1, 7.3)	1.77 m	3.56 ddd(17.0, 9.0, 8.0)
			2.29 m	3.74 dd(17.0, 4.2)
2′		3.04 m	1.39 m, 1.97 m	1.89 m, 2.01 m
3′	7.46 br s	2.65 d(7.0)	2.23 m	2.66 m, 2.64 m
4′			5.41 br s	6.95 t
5′	4.18 s	3.40 s		
6′			2.21 m, 2.91 m	8.15 s
7′	1.08 s	1.26 s	1.55 m, 2.37 m	3.97 s(OMe)
8′	1.72 s	1.44 s	2.32 m	8.13 d(8.8)
9′	2.65 s	1.18 d(6.8)	3.97 br s	7.04 br d(8.8)
10′				3.95 s(OMe)
11′				7.25 br s
NMe		2.36 s		
NH		8.05 d		12.22

Table 2-14-9: Cos, MFs, and TSs of carboline alkaloids **2-14-27**~**2-14-32**.

No.	Compounds	MFs	Test solvents	References
2-14-27	canthin-6-one	$C_{14}H_8N_2O$	$CDCl_3$	[436]
2-14-28	bruceolline C	$C_{16}H_{12}N_2O_4$	DMSO-d_6	[437]
2-14-29	5-hydroxymethyl-9-methoxycanthin-6-one	$C_{16}H_{12}N_2O_3$	DMSO-d_6	[428]
2-14-30	1-hydroxy-9-methoxycanthin-6-one	$C_{15}H_{10}N_2O_3$	DMSO-d_6	[428]
2-14-31	luotonin C	$C_{16}H_{12}N_2O_2$	$CDCl_3$	[438]
2-14-32	luotonin D	$C_{17}H_{14}N_2O_2$	$CDCl_3$	[438]



	R^1	R^2	R^3
2-14-27	H	H	H
2-14-28	OMe	OH	OMe



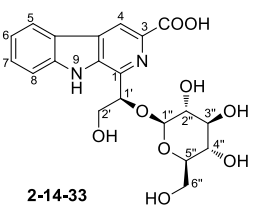
	R^1	R^2
2-14-29	H	CH_2OH
2-14-30	OH	H
2-14-31	H	Me
2-14-32	H	Et

Table 2-14-10: ^{1}H NMR spectroscopic data of carboline alkaloids **2-14-27**~**2-14-32**.

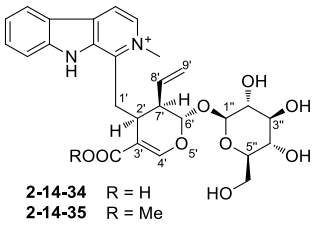
H	2-14-27	2-14-28	2-14-29	2-14-30	2-14-31	2-14-32
1	7.59 d(5.0)	4.25 s(OMe)	8.78 d(5.0)		7.81 d(5.0)	7.80 d(5.0)
2	8.56 d(5.0)	8.65 s	8.17 d(5.0)	8.38 s	8.75 d(5.0)	8.73 d(5.0)
4	7.77 d(9.7)	8.05 d(9.9)	8.02 br s	8.04 d(9.6)	7.89 q(1.4)	7.87 t(1.4)
5	6.75 d(9.7)	6.79 d(9.9)		6.74 d(9.6)		
8	8.28 d(7.7)	8.20 d(8.8)	8.09 d(2.3)	8.10 d(2.3)	8.27 d(2.4)	8.24 d(2.4)
9	7.45 t(7.7)	7.25 d(8.8)	3.93 s(OMe)	3.92 s(OMe)	3.99 s(OMe)	3.98 s(OMe)
10	7.28 t(7.7)		7.20 dd (8.7, 2.3)	7.17 dd (8.6, 2.3)	7.08 dd (2.4, 8.6)	7.08 dd (2.4, 8.6)
11	7.73 d(7.7)	3.90 s(OMe)	8.29 d(8.7)	8.09 d(8.6)	7.97 d(8.6)	7.94 d(8.6)
CH_2OH			4.60 br d(5.4) 5.55 t(5.4, OH)			
CH_2						2.83 dq(1.3, 7.4)
Me					2.44 d(1.4)	1.37 t(7.4)

Table 2-14-11: Cos, MFs, and TSs of carboline alkaloids **2-14-33**~**2-14-39**.

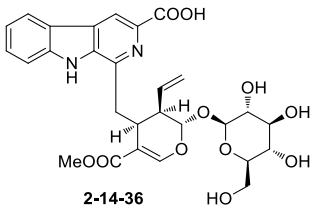
No.	Compounds	MFs	Test solvents	References
2-14-33	glusodichotomine B	$C_{20}H_{30}N_2O_9$	DMSO-d_6	[439]
2-14-34	3,4,5,6-tetradehydropalicoside	$C_{27}H_{31}N_2O_9$	CD_3OD	[440]
2-14-35	3,4,5,6-tetradehydrodolichantoside	$C_{28}H_{33}N_2O_9$	CD_3OD	[440]
2-14-36	desoxycordifoline	$C_{28}H_{30}N_2O_{11}$	CD_3OD	[440]
2-14-37	bruceolline G	$C_{20}H_{18}N_2O_7$	DMSO-d_6	[437]
2-14-38	canthin-6-one 9-*O*-β-glucopyranoside	$C_{20}H_{18}N_2O_7$	DMSO-d_6	[425]
2-14-39	bruceacanthinoside	$C_{26}H_{28}N_2O_{12}$	DMSO-d_6	[441]



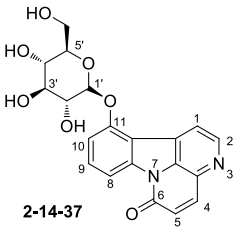
2-14-33



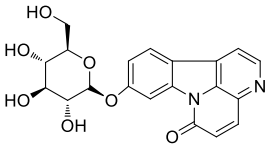
2-14-34 R = H
2-14-35 R = Me



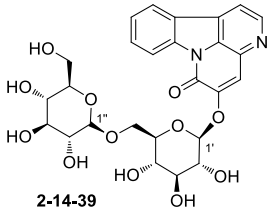
2-14-36



2-14-37



2-14-38



2-14-39

Table 2-14-12: ^{1}H NMR spectroscopic data of carboline alkaloids **2-14-33** and **2-14-37**~**2-14-39**.

H	2-14-33	2-14-37	2-14-38	2-14-39
1		8.32 d(4.9)	8.15 d(5.0)	8.19 d(5.3)
2		8.80 d(4.9)	8.75 d(5.0)	8.77 d(5.3)
4	8.88 s	8.10 d(9.8)	8.08 d(9.8)	7.76 s
5	7.70 or 8.40 d(7.5)	6.97 d(9.8)	6.93 d(9.8)	
6	7.32 or 7.62 m			
7	7.32 or 7.62 m			
8	7.70 or 8.40 d(7.5)	8.19 d(8.0)	8.12 d(2.0)	8.53 d(8.0)
9		7.70 t(8.0)		7.77 m
10		7.42 d(8.0)	7.27 dd(8.6, 2.0)	7.61 dd(8.0, 8.0)
11			8.23 d(8.6)	8.39 d(8.0)
1′	5.42 t(7.0)	5.20 d(7.7)	5.02 d(8.0)	5.22 d(7.6)
2′	4.07 m	–	–	3.42 m
3′		–	–	3.01~3.20 m
4′		–	–	3.26 m
5′		–	–	3.70~3.82 m
6′		–	–	4.01~4.09 m
1″	4.46 d(7.6)			4.21 d(7.3)
2″-3″	–			3.01~3.20 m
4″	–			3.70~3.82 m
5″	–			4.01~4.09 m
6″	–			3.48 m, 3.67 m
9-NH	11.80 br s			

Table 2-14-13: ^{1}H NMR spectroscopic data of carboline alkaloids **2-14-34**~**2-14-36**.

H	2-14-34	2-14-35	2-14-36
3	8.34 d	8.28 d(6.3)	11.5 s①
4	8.34 d	8.36 d(6.3)	8.69 s
5	8.24 d(8.0)	8.31 d(8.0)	8.19 d(7.8)
6	7.33 t(8.1)	7.36 t(8.0, 7.3)	7.28 t(7.8, 7.3)
7	7.66	7.71 t(7.3, 8.3)	7.56 t(7.3, 7.7)
8	7.66	7.75 d(8.3)	7.59 d(7.7)
1′	3.6 m, 3.5 m	3.75 m, 3.65 m	3.43 m, 3.27 m
2′	3.45 m	3.50 m(3.4)	3.67 m
4′	7.44 s	7.51 s	7.59 s
6′	5.95 d(8.9)	6.15 d(9.4)	5.86 d(7.3)
7′	2.72 d(8.9)	2.83 ddd(9.4, 8.0, 3.4)	2.62 dd(7.3, 7.2)
8′	5.97 dd(10.8, 17.9)	6.04 ddd(10.2, 17.1, 8.0)	5.67 ddd(10.7, 17.3, 7.2)
9′	*trans* 5.23 d(17.9)	*trans* 5.46 d(17.1)	*trans* 4.67 d(17.3)
	cis 5.31 d(10.8)	*cis* 5.35 d(10.2)	*cis* 4.93 d(10.7)
1″	4.78 d(7.9)	4.91 d(7.9)	4.78 d(7.8)
2″	3.15 d(7.9)	3.27 d(7.9)	3.19 d(7.8)
3″	3.35 m	3.30 m	3.39 m
4″	3.22 m	3.32 m	3.22 m
5″	3.35 m	3.45 m	3.39 m
6″	3.92 d(10.8), 3.60 d(10.8)	4.01 d(11.9), 3.76 d(11.9)	3.99 d(10.7), 3.67 d(10.7)
NMe	4.35 s	4.44 s	
COOMe		2.90 s	3.54 s

① Data redorded in DMSO-d_6.

Bibliography

[420] Leu YL, Shi LS, Damu AG. Chem Pharm Bull, 2003, 51: 599.
[421] Koike K, Ohmoto T, Ikeda K. Phytochemistry, 1990, 29: 3060.
[422] Zhou TS, Ye WC, Wang ZT, et al. Phytochemistry, 1998, 49: 1807.
[423] Ayoub MT, Rashan LJ. Phytochemistry, 1991, 30: 1046.
[424] Krebs HC, Rakotoarimanga JV, Rasoanaivo P, et al. J Nat Prod, 1997, 60: 1183.
[425] Kanchanapoom T, Kasai R, Chumsri P, et al. Phytochemistry, 2001, 56: 383.
[426] Sun B, Morikawa T, Matsuda H, et al. J Nat Prod, 2004, 67: 1464.
[427] Zou ZJ, Dong YS, Yang JS. Chin Chem Lett, 2005, 16: 1484.
[428] Kuo PC, Shi LS, Damu AG, et al. J Nat Prod, 2003, 66: 1324.
[429] Koike K, Ohmoto T, Uchida A, et al. Heterocycles, 1994, 38: 1413.
[430] Steele JCP, Veitch NC, Kite GC, et al. J Nat Prod, 2002, 65: 85.
[431] Costa EV, Pinheiro MLB, Xavier CM, et al. J Nat Prod, 2006, 69: 292.
[432] Phuong NM, Sung TV, Porzel A, et al. Phytochemistry, 1999, 52: 1725.
[433] Peng JN, Feng XZ, Zheng QT, et al. Phytochemistry, 1997, 46: 1119.
[434] Phuong NM, Sung TV, Porzel A, et al. Planta Med, 1999, 65: 761.
[435] Duan JA, Williams ID, Che CT, et al. Tetrahedron Lett, 1999, 40: 2593.
[436] Aono H, Koike K, Kaneko J, et al. Phytochemistry, 1994, 37: 579.
[437] Ouyang Y, Mitsunaga K, Koike K, et al. Phytochemistry, 1995, 39: 911.

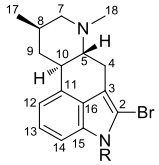
[438] Ma ZZ, Hano Y, Nomura T, et al. Phytochemistry, 2000, 53: 1075.
[439] Morikawa T, Sun B, Matsuda H, et al. Chem Pharm Bull, 2004, 52: 1194.
[440] Brandt V, Tits M, Geerlings A, et al. Phytochemistry, 1999, 51: 1171.
[441] Kitagawa I, Mahmud T, Simanjuntak P, et al. Chem Pharm Bull, 1994, 42: 1416.

2.15 Semiterpenoid indole (or ergoline) alkaloids

2.15.1 Clavine-type semiterpenoid indole alkaloids

Table 2-15-1: Cos, MFs, and TSs of semiterpenoid indole alkaloids **2-15-1** and **2-15-2**.

No.	Compounds	MFs	Test solvents	References
2-15-1	pibocin	$C_{16}H_{19}BrN_2$	C_6D_6	[442]
2-15-2	pibocin B	$C_{17}H_{22}BrN_2O$	C_5D_5N	[443]



2-15-1 R=H
2-15-2 R=OMe

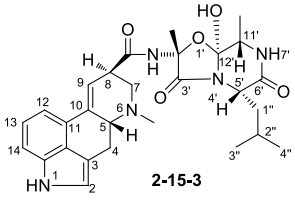
Table 2-15-2: ^{1}H NMR spectroscopic data of semiterpenoid indole alkaloids **2-15-1** and **2-15-2**.

H	2-15-1	2-15-2	H	2-15-1	2-15-2
4*α*	2.57 dd(15.0, 11.0)	2.94 dd(15.0, 11.0)	10	2.86 dddd(4.0, 12.5, 9.5, 1.8)	3.29 ddd(12.5, 10.5, 3.8)
4*β*	3.24 dd(15.0, 4.5)	3.32 dd(15.0, 4.5)	12	6.82 dt(0.9, 6.7, 1.8)	6.99 dt(6.4,1.5)
5	1.88 ddd(11.0, 4.5, 9.5)	2.38 ddd(10.5, 11.0, 4.5)	13	7.19 dd(6.7, 8.0)	7.33 dd(7.9, 6.4)
7*α*	2.70 ddd(11.0, 4.0, 1.8)	3.10 dq(11.5, 1.9)	14	6.93 dd(8.0, 1.8)	7.37 dd(7.9, 1.5)
7*β*	1.59 t(11.0, 11.0)	2.06 t(11.5)	8-OMe	0.80 d(6.4)	0.86 d(6.5)
8	1.83 m	2.24 m	NMe	2.17 s	2.59 s
9*α*	2.38 ddt(12.5, 4.0, 4.0, 1.8)	2.50 ddd(12.5, 3.8, 5.7)	NOMe		4.02 s
9*β*	0.92 q(12.5)	1.03 q(12.5)	NH	6.77 brs	

2.15.2 Lysergic acid-type semiterpenoid indole alkaloids

Table 2-15-3: Cos, MFs, and TSs of semiterpenoid indole alkaloids **2-15-3**~**2-15-7**.

No.	Compounds	MFs	Test solvents	References
2-15-3	ergobalansine	$C_{28}H_{35}N_5O_5$	$CDCl_3$	[444]
2-15-4	ergobalansinine	$C_{28}H_{35}N_5O_5$	$CDCl_3$	[444]
2-15-5	ergobine	$C_{28}H_{33}N_5O_5$	$CDCl_3$	[445]
2-15-6	10-hydroxypaspalic acid amide	$C_{16}H_{17}N_3O_2$	CD_3OD	[446]
2-15-7	10-hydroxy-*cis*-paspalic acid amide	$C_{16}H_{17}N_3O_2$	CD_3OD	[446]



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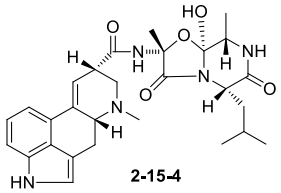

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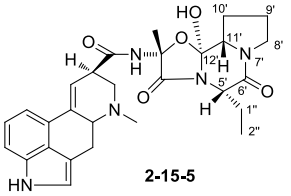

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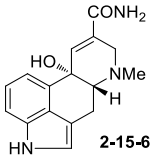

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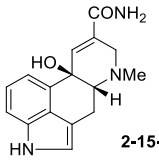


2-15-7

Table 2-15-4: ^{1}H NMR spectroscopic data of semiterpenoid indole alkaloids **2-15-3**~**2-15-7**.

H	2-15-3	2-15-4	2-15-5	2-15-6	2-15-7
1	7.99 s	8.05 s			
2	6.93 m	6.90 m	6.94 m	7.03 dd	6.99 d
4	2.82 m(14.3, 12.2)	3.59 m(14.4, 5.3)	ax 2.85 ddd(2.0, 14.5, 14.5)	ax 2.98 ddd(1.7, 14.1)	ax 3.06 mt
	3.32 m(14.3, 5.0)	2.64 m(14.4, 12.0)	eq 3.34 dd(5.3, 14.5)	eq 3.26 ddd(0.4)	eq 3.22 ddd(1.1, 15.7)
5	3.79 m(12.2, 5.0)	3.20 m(12.0, 5.3)	ax 3.70 m	2.58 dd(4.5, 11.9)	3.16 dd(4.6, 10.1)
7	2.98 dd(12.0, 3.7)	3.12 m(11.9)	ax 2.91 dd(3.5, 12.0)	ax 3.76 dd(1.3)	ax 3.46 dmt
	2.88 dd(12.0, 2.8)	2.75 (11.9, 3.5)	eq 3.04 m	eq 3.00 dd(17.0, 2.4)	eq 3.40 br mt(17.1)
8	3.18 m(5.6, 3.7, 2.8)	3.06 m(6.3)	3.24 m		
9	6.35 dd(5.6)	6.49 dd(6.3)	6.34 dd(1.5, 5.5)	7.51 dd	7.02 br s
12	7.18 m	7.15 m	7.1~7.3 m	7.28 dd(0.7)	7.23 dd(1.0)
13	7.18 m	7.15 m	7.1~7.3 m	7.09 dd(7.2, 8.1)	7.17 dd(7.2, 7.9)
14	7.18 m	7.15 m	7.1~7.3 m	7.30 dd	7.26 dd
5′	4.52 dd	4.42 dd	4.42 dd(5.0, 7.6)		
8′			3.5~3.6 m		
9′			2.0~2.2 m		
10′			2.0~2.2 m		
11′	3.56 m	3.51	3.5~3.6 m		
1″	1.94 m	1.90 m	2.0~2.3 m		
2″	1.94 m	1.90 m	1.12 t(7.6)		
3″	1.05 d	0.96 d			
4″	1.00 d	0.95 d			
6-NMe	2.64 s	2.61 s	2.68 s	2.55 s	2.52 s
2′-Me	1.52 s	1.50 s	1.32 s		
11′-Me	1.34 d	1.32 d			

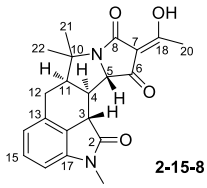
Table 2-15-4 (continued)

H	2-15-3	2-15-4	2-15-5	2-15-6	2-15-7
1-NH			7.96 br s		
7′-NH	6.78 br s	6.77 br s			
NHCO			9.23 br s		
8-NH	9.26 s	9.85 s			
OH	5.84 s	6.01 s	6.76 br s(12′OH)		

2.15.3 Secoergoline-type semiterpenoid indole alkaloids

Table 2-15-5: Co, MF, and TS of semiterpenoid indole alkaloid **2-15-8**.

No.	Compound	MF	Test solvent	References
2-15-8	speradine A	$C_{21}H_{22}N_2O_4$	$CDCl_3$	[447]



2-15-8

Table 2-15-6: ^{1}H NMR spectroscopic data of semiterpenoid indole alkaloid **2-15-8**.

H	2-15-8	H	2-15-8
3	3.48 d(6.8)	15	7.20 t(7.5)
4	2.54 m	16	6.67 d(7.5)
5	4.27 d(9.3)	20	2.43
11	2.17 m	21	1.50 s
12	2.76 dd(5.6,14.1), 2.63 br t(12.6)	22	1.61 s
14	6.86 d(7.6)	23	3.18 s

Bibliography

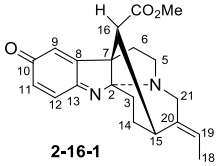
[442] Makarieva TN, Ilyin SG, Stonik VA, et al. Tetrahedron Lett, 1999, 40: 1591.
[443] Makarieva TN, Dmitrenok AS, Dmitrenok PS, et al. J Nat Prod, 2001, 64: 1559.
[444] Powell RG, Plattner RD, Yates SG. J Nat Prod, 1990, 53: 1272.
[445] Perellino NC, Malyszko J, Ballabio M, et al. J Nat Prod, 1993, 56: 489.
[446] Flieger M, Sedmera P, Havlíček V. J Nat Prod, 1993, 56: 810.
[447] Tsuda M, Mugishima T, Komatsu K, et al. Tetrahedron, 2003, 59: 3227.

2.16 Monoterpenoid indole alkaloids

2.16.1 Monomeric monoterpenoid indole alkaloids

Table 2-16-1: Cos, MFs, and TSs of monoterpenoid indole alkaloids **2-16-1**~**2-16-28**.

No.	Compounds	MFs	Test solvents	References
2-16-1	alstomaline	$C_{20}H_{22}N_2O_3$	$CDCl_3$	[448]
2-16-2	alstohentine	$C_{21}H_{28}N_2O_3$	$CDCl_3$	[448]
2-16-3	alstomicine	$C_{20}H_{26}N_2O_2$	$CDCl_3$	[448]
2-16-4	16-hydroxyalstonisine	$C_{20}H_{22}N_2O_4$	$CDCl_3$	[448]
2-16-5	16-hydroxyalstonal	$C_{20}H_{22}N_2O_4$	$CDCl_3$	[448]
2-16-6	16-hydroxy-*N*(4)-demethylalstophyllal oxindole	$C_{21}H_{24}N_2O_5$	$CDCl_3$	[448]
2-16-7	alstophyllal	$C_{22}H_{26}N_2O_3$	$CDCl_3$	[448]
2-16-8	6-oxoalstophylline	$C_{23}H_{26}N_2O_4$	$CDCl_3$	[448]
2-16-9	6-oxoalstophyllal	$C_{22}H_{24}N_2O_4$	$CDCl_3$	[448]
2-16-10	alstonoxine A	$C_{19}H_{24}N_2O_3$	$CDCl_3$	[449]
2-16-11	alstonoxine B	$C_{19}H_{26}N_2O_3$	$CDCl_3$	[449]
2-16-12	*N*(1)-demethylalsonisine	$C_{19}H_{20}N_2O_3$	$CDCl_3$	[449]
2-16-13	*N*(1)-demethylalstonal	$C_{19}H_{20}N_2O_3$	$CDCl_3$	[449]
2-16-14	akuammiline *N*(4)-oxide	$C_{23}H_{26}N_2O_5$	$CDCl_3$	[450]
2-16-15	16-*epi*-deacetylakuammiline *N*(4)-oxide	$C_{21}H_{24}N_2O_4$	$CDCl_3$	[450]
2-16-16	9-methoxy-N_b-methylgeissoschizol	$C_{21}H_{29}N_2O_2$	CD_3OD	[451]
2-16-17	*C*-alkaloid O	$C_{20}H_{25}N_2O$	CD_3OD	[451]
2-16-18	10-methoxyaffinisine	$C_{21}H_{26}N_2O_2$	$CDCl_3$	[452]
2-16-19	10-methoxycathafoline	$C_{22}H_{28}N_2O_3$	$CDCl_3$	[452]
2-16-20	alstonerinal	$C_{21}H_{24}N_2O_2$	$CDCl_3$	[452]
2-16-21	nauclealine A	$C_{20}H_{14}N_2O_3$	DMSO-d_6	[453]
2-16-22	nauclealine B	$C_{17}H_{16}N_2O_3$	DMSO-d_6	[453]
2-16-23	naucleoside A	$C_{26}H_{28}N_2O_9$	DMSO-d_6	[453]
2-16-24	naucleoside B	$C_{26}H_{28}N_2O_9$	DMSO-d_6	[453]
2-16-25	javaniside	$C_{26}H_{30}N_2O_9$	CD_3OD	[454]
2-16-26	19(*S*),20(*R*)-dihydroperaksine	$C_{19}H_{24}N_2O_2$	C_5D_5N	[455]
2-16-27	19(*S*),20(*R*)-dihydroperaksine-17-al	$C_{19}H_{22}N_2O_2$	C_5D_5N	[455]
2-16-28	10-hydroxy-19(*S*),20(*R*)-dihydroperaksine	$C_{19}H_{24}N_2O_3$	C_5D_5N	[455]



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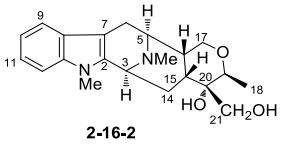

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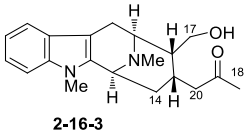

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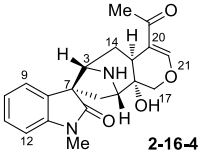

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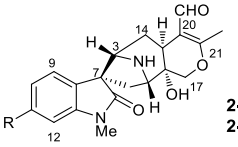

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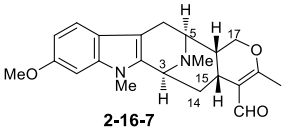
2-16-6 R = OMe



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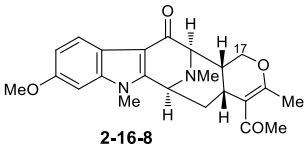

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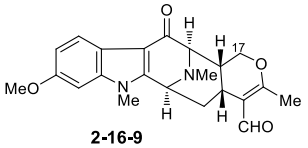

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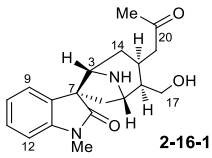

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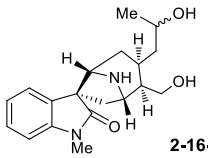

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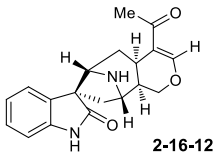

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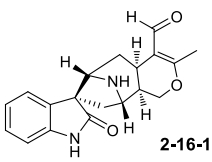

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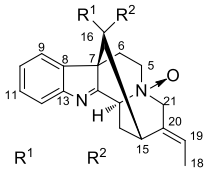
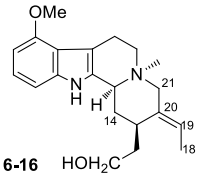


2-16-14 CH_2OAc CO_2Me
2-16-15 CO_2Me CH_2OH



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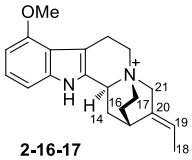

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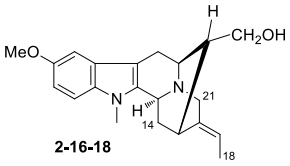

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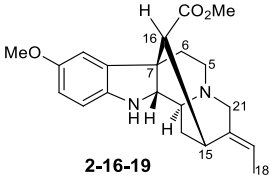

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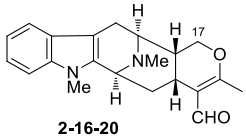

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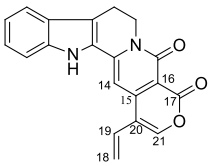

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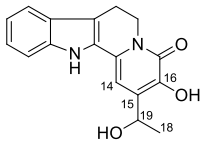

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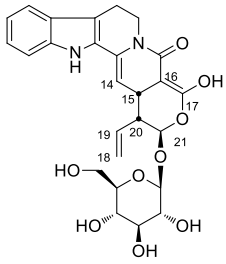

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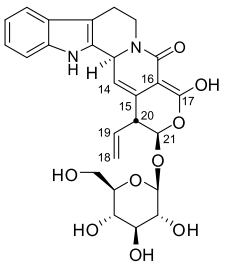

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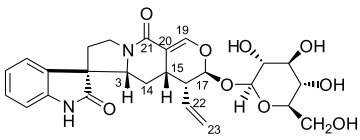

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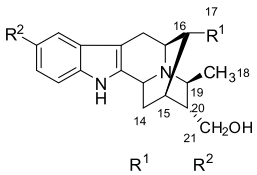


	R^1	R^2
2-16-26	CH_2OH	H
2-16-27	CHO	H
2-16-28	CH_2OH	OH

Table 2-16-2: ^{1}H NMR spectroscopic data of monoterpenoid indole alkaloids **2-16-1**~**2-16-5**.

H	2-16-1	2-16-2	2-16-3	2-16-4	2-16-5
3	1.28 ddd(16, 10, 8)	3.83 d(10)	4.01 br s	3.16 t(3)	3.16 br s
	2.75 m				
5	2.75 m, 2.75 m	3.31 m	3.49 d(7)	3.53 d(7)	3.52 d(7)
6	1.87 m	2.37 d(16)	2.56 d(17)	2.35 m	2.35 dd(14, 7)
	2.52 ddd(14, 11, 8)	2.95 dd(16, 4)	3.31 dd (17, 7)	2.51 dd(14, 1)	2.49 dd(14, 1)
9	6.54 d(2)	7.41 br d(8)	7.50 d(8)	8.17 dd(8, 1)	8.16 dd(8, 1)
10		7.04 td(8, 1)	7.11 t(8)	7.29 td(8, 1)	7.28 td(8, 1)
11	6.60 dd(10, 2)	7.14 td(8, 1)	7.20 t(8)	7.33 td(8, 1)	7.33 td(8, 1)
12	7.38 d(10)	7.24 br d(8)	7.31 d(8)	6.87 dd(8, 1)	6.87 dd(8,1)
14	1.87 m	1.77 dd (14, 10)	1.61 m, 2.27 m	1.39 ddd (15, 12, 3)	1.38 ddd(15, 11, 3)
		2.73 dd (14, 10)		2.35 m	2.40 ddd(15, 7,3)
15	3.79 m	1.90 dd(10, 6)	2.27 m	3.10 ddd(12, 7, 2)	3.06 m
16	2.75 m	1.61 td(6, 3)	1.69 br s		
17		3.44 dd(12, 3)	3.90 br d(11)	4.01 dd(12, 2)	4.03 dd(12, 2)
		3.70 br d(12)	3.98 br d(11)	4.70 d(12)	4.76 d(12)
18	1.64 dd(7, 2)	1.21 d(6)	2.04 s	2.26 s	2.30 s
19	5.44 q(7)	3.32 q(6)			
20			2.43 dd (17, 7)		
			2.64 dd (17, 7)		
21	3.02 d(16)	3.34 s		7.66 s	9.89 s
	4.02 d(16)	3.34 s			
N(1)-Me		3.57 s	3.63 s	3.20 s	3.20 s
N(4)-Me		2.37 s	2.37 s		
16-COOMe	3.79 s				
20-OH		9.01 br s			

Table 2-16-3: ^{1}H NMR spectroscopic data of monoterpenoid indole alkaloids **2-16-6**~**2-16-10**.

H	2-16-6	2-16-7	2-16-8	2-16-9	2-16-10
3	3.11 t(3)	3.84 t(3)	4.06 br s	4.06 br s	3.25 br s
5	3.49 br d(7)	3.06 d(7)	3.20 br s	3.20 br s	3.90 br d(8)
6	2.37 m	2.45 d(16)			2.15 dd(13, 2)
	2.44 br d(14)	3.29 dd(16, 7)			2.43 dd(13, 8)
9	8.07 d(8)	7.34 d(8)	8.04 d(8)	8.04 d(8)	7.84 br d(8)
10	6.78 dd(8, 2)	6.76 dd(8, 2)	6.96 dd(8, 2)	6.96 dd(8, 2)	7.20 td(8, 1)
11		–	–	–	7.32 td(8, 1)
12	6.45 d(2)	6.81 d(2)	6.82 d(2)	6.82 d(2)	6.87 br d(8)
14	1.36 ddd(14, 11, 3)	1.77 td (12, 4)	2.00 m	2.00 m	1.71 m
	2.37 m	2.14 m	2.18 m	2.18 m	1.87 ddd(14, 6, 2)

Table 2-16-3 (continued)

H	2-16-6	2-16-7	2-16-8	2-16-9	2-16-10
15	3.03 m	2.61 m	2.69 dt(11, 6)	2.69 dt(11, 6)	3.05 m
16		1.89 m	2.18 m	2.18 m	1.71 m
17	4.03 dd(11, 2)	4.19 ddd(11, 4, 2)	4.25 ddd(11, 4, 2)	4.27 ddd (11, 4, 2)	3.80 dd(12, 2)
	4.76 d(11)	4.46 t(11)	4.46 t (11)	4.51 t(11)	4.02 dd(12, 1)
18	2.30 s	2.17 s	2.11 s	2.19 s	2.21 s
20					2.72 dd(18, 6)
					2.79 dd(18, 7)
21	9.89 s	9.66 s	7.56 s	9.67 s	
N(1)-Me	3.17 s	3.60 s	3.69 s	3.70 s	3.20 s
N(4)-Me		2.32 s	2.44 s	2.44 s	
11-OMe	3.85 s	3.89 s	3.91 s	3.91 s	

Table 2-16-4: ^{1}H NMR spectroscopic data of monoterpenoid indole alkaloids **2-16-11**~**2-16-15**.

H	2-16-11	2-16-12	2-16-13	2-16-14	2-16-15
3	3.25 br s	3.27 br s	3.27 br s	5.05 d(4.5)	5.07 br s
5	3.91 m	3.69 br d(7)	3.69 br d(7)	3.17 m	3.13 m
				3.42 m	3.58 br dd(13, 5)
6	2.13 dd(14, 1)	2.20 br d(13)	2.19 br d(13)	2.12 br dd(15, 5)	1.93 br dd(15, 5)
	2.41 dd(14, 8)	2.57 dd(13, 7)	2.56 dd(13,7)	3.42 m	2.86 m
9	7.52 br d(8)	8.22 br d(8)	8.22 br d(8)	7.62 dd(7.5, 1)	7.76 br d(7.5)
10	7.10 td(8, 1)	7.25 m	7.25 m	7.24 td(7.5, 1)	7.22 td(7.5, 1)
11	7.31 td(8, 1)	7.20 m	7.20 m	7.37 td(7.5, 1)	7.35 td(7.5, 1)
12	6.88 br d(8)	6.91 br d(8)	6.91 br d(8)	7.66 dd(7.5, 1)	7.64 br d(7.5)
14	1.53 ddd(14, 9, 5)	1.57 ddd(14, 12, 2)	1.55 ddd(14, 12, 2)	2.17 m	2.81 m
	1.85 ddd(14, 9, 3)	2.26 ddd(14, 6, 2)	2.30 ddd(14, 6, 2)	2.91 ddd(15, 4.5, 2.5)	2.81 m
15	2.72 m	3.39 dt(12, 6)	3.35 dt(12, 6)	3.57 br s	3.38 br s
16	1.77 m	1.98 m	1.98 m		
17	4.01 dd(11, 1)	4.26 ddd(11, 4, 2)	4.28 ddd(11, 4, 2)	3.59 m	4.26 d(12)
	3.91 m	4.46 t(11)	4.52 t(11)	3.59 m	4.30 d(12)
18	1.30 d(6)	2.24 s	2.24 s	1.72 dd(7.2)	1.85 dd(7.2)
19	3.91 m	7.63 s	9.86 s	5.73 q (7)	5.88 q(7)
20	1.77 m,1.77 m				
21	–	–		4.07 d(16)	4.60 dd(17, 2)
				4.53 dd(16, 2)	4.28 d(17)
16-CO_2Me				3.79 s	3.18 s
16-OAc				1.59 s	
N(1)-Me	3.20 s				
NH		8.54 s	8.57 s		

Table 2-16-5: ^{1}H NMR spectroscopic data of monoterpenoid indole alkaloids **2-16-16**~**2-16-20**.

H	2-16-16	2-16-17	2-16-18	2-16-19	2-16-20
2				2.44 s	
3	4.63 br s	4.93 t	4.20 dd(10, 2)	4.12 d(5)	3.86 t(3)
5	3.82 m	3.7 m	2.79 m	2.64 m	3.06 br d(7)
	3.77 m	3.6 m		3.88 td(14, 5)	
6	3.42 m	3.35 m	2.59 dd(15, 1)	1.45 dd(15, 5)	2.48 d(16)
	3.32 m		3.04 dd(15, 5)	3.07 ddd(15, 14, 5)	3.31 dd(16, 7)
9			6.92 d(1)	6.61 d(1)	7.44 br d(8)
10	6.54 d(8)	6.54 d			7.08 td(8, 1)
11	7.09 t(8)	7.08 t	6.83 dd(8, 1)	6.63 dd(8, 1)	7.19 td(8, 1)
12	6.98 d(8)	6.98 d	7.17 d(8)	6.53 d(8)	7.31 br d(8)
14	2.65 m	2.65 m	1.67 ddd(12, 4, 2)	1.61 dd(14, 3)	1.79 td(12,3)
	2.3 m	2.2 m	2.01 ddd(12, 10, 2)	2.37 ddd(14, 5, 3)	2.12 ddd(12, 5, 3)
15	3.23 m	3.29 m	1.79 m	3.60 br s	2.61 dt(12,5)
16	1.62 m, 1.44 m	2.05 m	1.81 tdd(8, 6, 1)	2.94 d(4)	1.89 m
17	3.51 m	3.8 m	3.54 m		4.18 ddd(11, 4, 2)
		3.46 m	3.54 m		4.46 t(11)
18	1.84 d(5.6)	1.8 d	1.63 dt(7, 2)	1.50 dd(7, 3)	2.15 s
19	5.99 q	5.6 q	5.40 br q(7)	5.41 br q(7)	
21	4.35 d(14)	4.41 d	3.54 m	2.94 d(16)	9.65 s
	3.71 d(14)	4.16 d	3.54 m	3.95 br d(16)	
9-OMe	3.9 s	4.86 s			
10-OMe			3.85 s	3.73 s	
16-CO_2Me				3.77 s	
N(1)-Me			3.59 s	2.65 s	3.63 s
N(4)-Me	3.19 s				2.31 s

Table 2-16-6: ^{1}H NMR spectroscopic data of monoterpenoid indole alkaloids **2-16-21**~**2-16-25**.

H	2-16-21	2-16-22	2-16-23	2-16-24	2-16-25
1	11.98 s	11.86 s	11.73 s	11.73 s	–
3				5.06 br s	4.09 dd(11.6, 3.0)
5	4.31 t(7.0)	4.37 m	4.02 m	4.07 m	α 4.04 ddd(11.4, 10.5, 7.4)
			4.53 m	4.44 m	β 3.76 br t(11.4, 11.2)
6	3.11 t(7.0)	3.10 t(6.5)	3.07 m	2.95~3.07 m	α 2.23 dd(13.2, 7.4)
					β 2.40 ddd(13.2, 11.2, 10.5)
9	7.66 d(8.0)	7.62 d(7.9)	7.59 d(8.0)	7.59 d(7.8)	7.30 br d(7.5)
10	7.11 t(8.0)	7.09 t(7.9)	7.06 t(8.0)	7.05 t(7.8)	7.07 td(7.5, 1.2)
11	7.30 t(8.0)	7.24 t(7.9)	7.21 t(8.0)	7.21 t(7.8)	7.25 td(7.5, 1.2)
12	7.48 d(8.0)	7.47 d(7.9)	7.41 d(8.0)	7.41 d(7.8)	6.90 br d(7.5)
14	6.92 s	7.23 s	6.57 s	6.57 s	α 1.27 td(12.2, 12.4)
					β 1.37 dt(12.2, 3.8, 3.8)
15			3.34 m		2.94 dddd(12.4, 5.5, 3.8, 2.4)

Table 2-16-6 (continued)

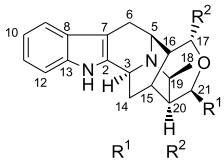
H	2-16-21	2-16-22	2-16-23	2-16-24	2-16-25
16					2.56 ddd(9.8, 5.5, 1.8)
17		–			5.42 d(1.8)
18	5.43 dd(10.6, 1.8)	1.50 d(6.3)	5.24 d(10.0)	5.25 d(10.9)	
	5.69 dd(17, 1.8)		5.26 d(17.2)	5.27 d(18.6)	
19	6.70 dd(17, 10.6)	5.31 q(6.3)	5.73 m	5.73 m	7.38 d(2.4)
20			3.34 m	3.38 m	
21	7.94 s		5.41 br s	5.41 br s	
22					5.49 dt(16.8, 10.0)
23					5.16 dd(10.0, 1.8)
					5.19 dd(16.8, 1.8)
1′			4.86 d(7.2)	4.63 d(8.1)	4.64 d(8.0)
2′			3.04 m	2.95~3.07 m	3.14 dd(9.0, 8.0)
3′			3.04 m	2.95~3.07 m	3.36 m
4′			3.04 m	2.95~3.07 m	3.26 m
5′			3.17 m	3.20 m	3.28 m
6′			3.34 m	3.20 m	3.63 dd(12.0, 5.6)
			3.62 m	3.68 m	3.83 dd(12.0, 2.0)

Table 2-16-7: ^{1}H NMR spectroscopic data of monoterpenoid indole alkaloids **2-16-26**~**2-16-28**.

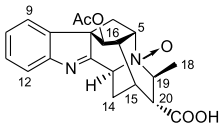
H	2-16-26	2-16-27	2-16-28
3	4.51 d(9.6)	4.28 d(9.4)	4.41 d(9.4)
5	3.58 m	4.17 dd(8.2, 4.7)	3.47 m
6*α*	3.39 dd(15.1, 4.8)	3.23 dd(15.3, 4.7)	3.30 dd(15, 4.4)
6*β*	3.11 d(15.1)	2.75 d(15.3)	3.05 d(15.1)
9	7.66 dd(7.2, 1.4)	7.68 dd(7.6, 1.2)	7.49 d(2.4)
10	7.23 ddd(7.2, 7.2, 1.4)	7.25 ddd(7.2, 7.2, 1.2)	
11	7.27 ddd(7.2, 7.2, 1.4)	7.29 ddd(7.2, 7.2, 1.2)	7.24 dd(8.5, 2.4)
12	7.58 dd(7.2, 1.4)	7.57 d(7.6, 1.2)	7.50 d(8.5)
14*α*	2.33 ddd(13, 9.6, 1.4)	2.22 ddd(12.9, 9.4, 1.2)	2.29 dd(12.9, 9.4)
14*β*	1.54 dd(13, 3.1)	1.47 dd(12.9, 2.4)	1.53 d(12.9)
15	2.48 m	2.73 m	2.51 m
16	2.12 m	2.32 d(8.2)	2.19 m
17	4.01 m, 4.01 m	9.87 s	4.01 m, 4.01 m
18	1.52 d(6.5)	1.32 d(6.5)	1.47 d(6.5)
19	2.81 ddd(14.7, 6.5, 6.5)	2.51 ddd(14.7, 6.5, 6.5)	2.74 ddd(15.3, 6.5, 6.5)
20	2.07 ddd(14.7, 7.9, 5.8)	1.57 ddd(14.7, 8.8, 5.3)	2.04 dd(15.3, 8.2, 5.9)
21	3.81 dd(10.9, 7.9)	3.75 dd(11.4, 8.8)	3.82 dd(10.9, 8.2)
	3.87 dd(10.6, 5.8)	3.82 dd(11.4, 5.3)	3.90 dd(10.9, 5.9)
10-OH			10.72 br s
17-OH	5.58 br s		5.28 br s
21-OH	6.16 br s	6.18 br s	6.17 br s
NH	12.02 br s	11.90 br s	11.64 br s

Table 2-16-8: Cos, MFs, and TSs of monoterpenoid indole alkaloids **2-16-29**~**2-16-43**.

No.	Compounds	MFs	Test solvents	References
2-16-29	alstoyunine A	$C_{20}H_{24}N_2O_3$	CD_3OD	[456]
2-16-30	alstoyunine B	$C_{20}H_{24}N_2O_3$	CD_3OD	[456]
2-16-31	alstoyunine C	$C_{21}H_{22}N_2O_5$	CD_3OD	[456]
2-16-32	alstoyunine D	$C_{21}H_{22}N_2O_6$	CD_3OD	[456]
2-16-33	melodinine E	$C_{19}H_{20}N_2O_2$	$CDCl_3$	[457]
2-16-34	melodinine F	$C_{20}H_{26}N_2O_2$	$CDCl_3$	[457]
2-16-35	melodinine G	$C_{19}H_{22}N_2O$	$CDCl_3$	[457]
2-16-36	11,12-methylenedioxykopsinaline *N*(4)-oxide	$C_{22}H_{26}N_2O_6$	$CDCl_3$	[450]
2-16-37	jerantinine A	$C_{22}H_{26}N_2O_4$	$CDCl_3$	[458]
2-16-38	jerantinine C	$C_{22}H_{24}N_2O_5$	$CDCl_3$	[458]
2-16-39	jerantinine E	$C_{22}H_{28}N_2O_4$	$CDCl_3$	[458]
2-16-40	jerantinine B	$C_{22}H_{26}N_2O_5$	$CDCl_3$	[458]
2-16-41	jerantinine D	$C_{22}H_{24}N_2O_6$	$CDCl_3$	[458]
2-16-42	jerantinine F	$C_{22}H_{26}N_2O_5$	$CDCl_3$	[458]
2-16-43	jerantinine G	$C_{22}H_{24}N_2O_5$	$CDCl_3$	[458]



2-16-29 OMe OH
2-16-30 OH OMe



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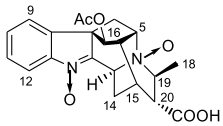

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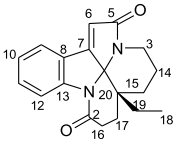

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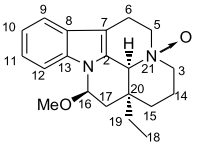

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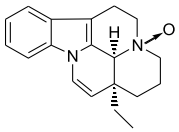

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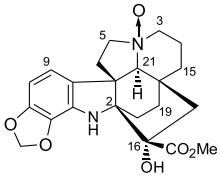

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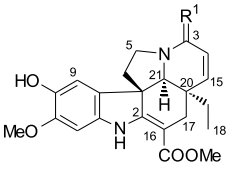
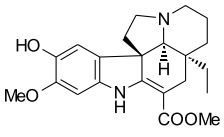
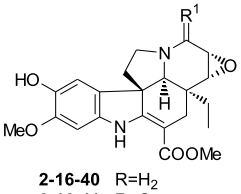


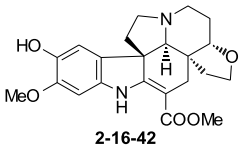
2-16-37 R=H_2
2-16-38 R=O



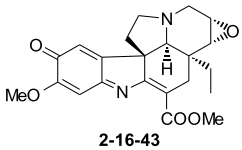
2-16-39



2-16-40 R=H_2
2-16-41 R=O



2-16-42



2-16-43

Table 2-16-9: ^{1}H NMR spectroscopic data of monoterpenoid indole alkaloids **2-16-29**~**2-16-32**.

H	2-16-29	2-16-30	2-16-31	2-16-32
3	4.07 d(9.0)	4.68 d(9.5)	4.52 d(9.8)	5.16 d(9.2)
5	3.81 dd(5.5, 5.2)	4.20 dd(5.5, 5.2)	4.30 dd(6.0, 5.0)	4.27 dd(6.0, 5.0)
6	2.97 dd(15.5, 5.5)	3.18 dd(16.5, 5.5)	2.91 m	2.90 dd(13.0, 4.3)
	2.62 d(15.5)	2.80 d(16.5)	2.46 d(13.0)	2.60 d(13.0)
9	7.35 d(8.0)	7.43 d(8.0)	7.62 d(7.5)	7.74 d(7.8)
10	6.94 t(8.0)	7.03 t(8.0)	7.31 t(7.5)	7.58 t(7.8)
11	7.01 t(8.0)	7.13 t(8.0)	7.43 t(7.5)	7.62 t(7.8)
12	7.25 d(8.0)	7.32 d(8.0)	7.61 d(7.5)	7.79 d(7.8)
14	1.99 dd(13.8, 9.0)	2.34 dd(14.0, 9.5)	2.60 dd(14.5, 9.8)	2.64 dd(14.4, 9.2)
	1.59 dd(13.8, 4.2)	1.88 m	2.07 dd(14.5, 5.0)	2.14 dd(14.4, 4.0)
15	2.11 br s	2.49 br s	2.83 m	2.85 m
16	1.53 d(5.0)	1.93 br s	3.08 m	3.07 m
17	5.05 d(1.1)	4.98 d(1.3)	4.99 s	5.11 d(1.0)
18	1.37 d(7.2)	1.57 d(7.1)	1.54 d(6.5)	1.39 d(6.3)
19	3.25 m	3.85 m	4.08 m	4.15 m
20	1.75 dd(10.0, 3.5)	2.08 m	2.88 m	2.79 d(9.5)
21	4.81 br s	5.39 br s		
17-OAc			2.19 s	2.19 s
17-OMe		3.48 s		
21-OMe	3.40 s			

Table 2-16-10: ^{1}H NMR spectroscopic data of monoterpenoid indole alkaloids **2-16-33**~**2-16-36**.

H	2-16-33	2-16-34	2-16-35	2-16-36
3	4.42 m, 3.21 m	3.32 m, 3.09 m	3.48 m, 3.16 br d(11.5)	3.57 m, 3.93 m
5		3.93 m, 3.82 m	3.98 m, 3.86 m	3.57 m, 3.93 m
6	6.20 s	3.04 m, 3.06 m	3.11 m, 3.04 m	2.35 m, 2.47 br dd(15, 8)
9	7.44 d(7.5)	7.45 d(8.0)	7.45 d(7.8)	7.50 d(8)
10	7.10 t(7.5)	7.14 t(8.0)	7.24 t(7.8)	6.15 d(8)
11	7.31 t(7.5)	7.23 t(8.0)	7.14 t(7.8)	
12	8.14 d(7.5)	7.25 d(8.0)	7.33 d(7.8)	
14	2.00 m, 1.79 m	2.59 m, 1.39 br d(13.6)	2.66 m, 1.46 br d(14.2)	1.82 m, 2.94 m
15	1.62 m, 1.05 m	1.93 m, 1.47 br d(14.0)	1.59 br d(14.0), 1.24 dd(14.0, 3.5)	1.75 m, 2.04 m
16	3.04 m, 2.59 m	5.42 d(3.4)	6.93 d(7.8)	
17	2.09 m	2.18 br d(15.2)	5.03 d(7.8)	1.26 d(15)
	1.66 m	1.96 dd(15.2, 4.4)		3.16 dd(15, 2)
18	0.73 t(7.4)	0.99 t(7.3)	1.07 t(7.4)	1.45 m, 1.67 m
19	1.42 q(7.4)	2.60 q(7.3), 2.25 q(7.3)	2.78 q(7.4), 1.88 q(7.4)	1.33 m, 1.82 m
	1.32 q(7.4)			
21		4.43 s	4.78 s	3.62 br s

Table 2-16-10 (continued)

H	2-16-33	2-16-34	2-16-35	2-16-36
16-OMe		3.50 s		
16-OAc				3.83 s
OCH_2O				5.51 d(1.5), 5.71 d(1.5)
NH				5.29 br s

Table 2-16-11: ^{1}H NMR spectroscopic data of monoterpenoid indole alkaloids **2-16-37**~**2-16-40**.

H	2-16-37	2-16-38	2-16-39	2-16-40
3	3.16 br d(16)		2.38 td(10.8, 3.2)	2.86 m
	3.45 ddd(16, 4.6, 1.3)		3.10 m	3.51 m
5	2.68 ddd(11.5, 7, 4.3)	3.35 td(12, 5)	2.52 ddd(11.5, 8, 4.5)	2.43 m
	3.02 t(7)	4.29 dd(12, 7)	2.89 dd(8, 6.7)	2.86 m
6	1.76 dd(11.5, 4.3)	1.86 dd(12, 5)	1.66 dd(11.5, 4.5)	1.67 dd(11.5, 4.3)
	2.06 td(11.5, 7)	1.94 td(12, 7)	2.03 td(11.5, 6.7)	1.93 td(11.5, 6.2)
9	6.89 s	6.87 s	6.84 s	6.79 s
12	6.45 s	6.52 s	6.43 s	6.45 s
14	5.78 ddd(10.0, 4.6, 1.5)	5.95 d(10.0)	1.53 m, 1.81 m	3.50 m
15	5.70 dt(10.0, 1.3)	6.44 d(10.0)	1.25 m, 1.81 m	3.10 d(3.5)
17	2.42 d(15)	2.04 dd(15.5, 1.5)	2.27 dd(15.1, 1.5)	2.47 d(15)
	2.53 dd(15, 1.5)	2.59 d(15.5)	2.70 d(15.1)	2.57 d(15)
18	0.64 t(7.3)	0.71 t(7)	0.57 t(7)	0.74 t(7.2)
19	0.86 dq(14, 7.3)	1.00 dq(14, 7)	0.63 dq(14, 7)	0.89 dq(14, 7.2)
	1.00 dq(14, 7.3)	1.08 dq(14, 7)	0.98 dq(14, 7)	1.10 dq(14, 7.2)
21	2.60 s	3.92 s	2.36 d(1.5)	2.38 s
NH	8.87 br s	8.91 br s	8.77 br s	8.82 br s
10-OH	5.36 br s	5.59 br s	5.37 br s	5.50 br s
11-OMe	3.87 s	3.89 s	3.86 s	3.85 s
16-COOMe	3.76 s	3.77 s	3.75 s	3.78 s

Table 2-16-12: ^{1}H NMR spectroscopic data of monoterpenoid indole alkaloids **2-16-41**~**2-16-43**.

H	2-16-41	2-16-42	2-16-43
3		2.70 m, 2.93 m	2.80 m, 3.50 m
5	3.19 td(12, 5.8)	2.63 ddd(11.5, 8, 4,5)	2.31 ddd(11.8, 8.8, 4.5)
	4.40 dd(12, 7)	2.93 m	2.95 dd(8.8, 6.2)
6	1.73 m	1.74 dd(11.5, 4.5)	1.77 dd(11.8, 4.5)
	1.73 m	2.02 td(11.5, 6)	2.03 td(11.8, 6.2)
9	6.80 s	6.88 s	6.22 s
12	6.48 s	6.45 s	6.69 s
14	3.57 m	1.95 m, 1.95 m	3.50 m

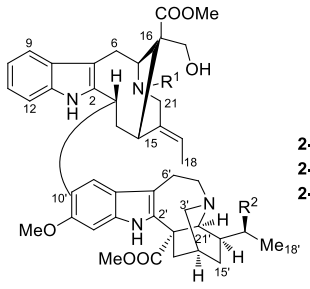
Table 2-16-12 (continued)

H	2-16-41	2-16-42	2-16-43
15	3.43 d(3.6)	3.68 m	3.09 d(3.4)
17	1.82 d(15.4), 2.63 d(15.4)	2.28 dd(14.5, 1.7), 2.73 d(14.5)	2.79 m, 2.79 m
18	0.77 t(7.4)	3.69 m, 3.78 m	0.88 t(7)
19	1.02 dq(14, 7.4)	1.30 ddd(12.5, 8, 4.5)	0.73 dq(14, 7)
	1.23 dq(14, 7.4)	1.44 ddd(12.5, 10, 7.5)	1.25 dq(14, 7)
21	3.57 s	2.74 s	2.25 s
NH	8.83 br s	8.76 br s	
10-OH	5.49 br s	5.38 br s	
11-OMe	3.86 s	3.87 s	3.89 s
16-COOMe	3.76 s	3.77 s	3.91 s

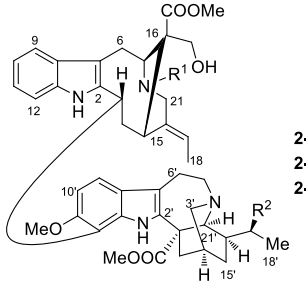
2.16.2 Dimeric monoterpenoid indole alkaloids

Table 2-16-13: Cos, MFs, and TSs of monoterpenoid indole alkaloids **2-16-44**~**2-16-58**.

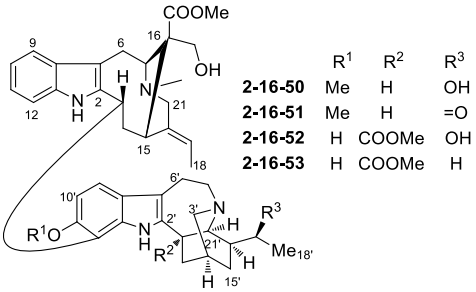
No.	Compounds	MFs	Test solvents	References
2-16-44	conodiparine A	$C_{44}H_{54}N_4O_7$	$CDCl_3$	[459]
2-16-45	conodiparine C	$C_{44}H_{52}N_4O_7$	$CDCl_3$	[459]
2-16-46	conodiparine E	$C_{43}H_{50}N_4O_7$	$CDCl_3$	[459]
2-16-47	conodiparine B	$C_{44}H_{54}N_4O_7$	$CDCl_3$	[459]
2-16-48	conodiparine D	$C_{44}H_{52}N_4O_7$	$CDCl_3$	[459]
2-16-49	conodiparine F	$C_{43}H_{52}N_4O_7$	$CDCl_3$	[459]
2-16-50	conodutarine A	$C_{42}H_{52}N_4O_5$	$CDCl_3$	[459]
2-16-51	conodutarine B	$C_{42}H_{50}N_4O_5$	$CDCl_3$	[459]
2-16-52	cononitarine A	$C_{43}H_{52}N_4O_7$	$CDCl_3$	[459]
2-16-53	cononitarine B	$C_{43}H_{52}N_4O_6$	$CDCl_3$	[459]
2-16-54	strychnochrysine	$C_{38}H_{39}N_4O_2$	$CDCl_3$	[460]
2-16-55	melodinine H	$C_{40}H_{44}N_4O_2$	CD_3COCD_3	[461]
2-16-56	melodinine I	$C_{40}H_{44}N_4O_3$	CD_3COCD_3	[461]
2-16-57	melodinine J	$C_{40}H_{44}N_4O_3$	DMSO-d_6	[461]
2-16-58	melodinine K	$C_{42}H_{46}N_4O_6$	$CDCl_3$	[461]



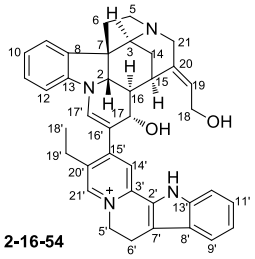
	R^1	R^2
2-16-44	Me	OH
2-16-45	Me	=O
2-16-46	H	OH



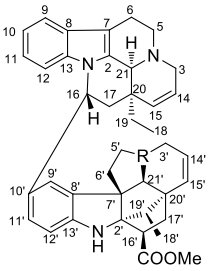
	R^1	R^2
2-16-47	Me	OH
2-16-48	Me	=O
2-16-49	H	OH



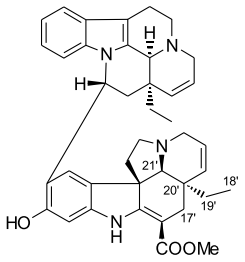
	R^1	R^2	R^3
2-16-50	Me	H	OH
2-16-51	Me	H	=O
2-16-52	H	COOMe	OH
2-16-53	H	COOMe	H



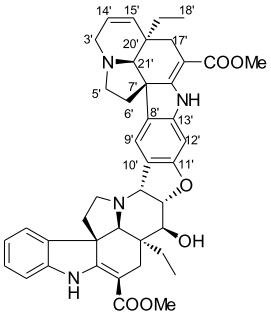
2-16-54



2-16-55 R = N
2-16-56 R = N→O



2-16-57



2-16-58

Table 2-16-14: ^{1}H NMR spectroscopic data of monoterpenoid indole alkaloids **2-16-44**~**2-16-48**.

H	2-16-44	2-16-45	2-16-46	2-16-47	2-16-48
3	5.14 br d(13)	5.14 br d(13)	5.16 br d(13)	5.32 dd(13, 3)	5.32 dd(13, 3)
5	3.92 t(9)	3.95 m	4.00 t(9)	3.94 m	3.96 m
6	3.28 m	3.28 dd(15, 8)	3.38 dd (4, 9)	3.49 m	3.44 m
	3.61 m	3.56 m	3.78 m	3.49 m	3.44 m
9	7.55 d(7)	7.55 d(6.9)	7.57 br d(7.5)	7.68 dd(7.4, 1)	7.67 br d(7.7)
10	7.05 m	7.06 m	7.06 m	7.14 td(7.4, 1)	7.13 td(7.7, 1)
11	7.05 m	7.06 m	7.06 m	7.08 td(7.4, 1)	7.08 td(7.7, 1)
12	7.05 m	7.06 m	7.06 m	7.02 dd(7.4, 1)	7.02 br d(7.7)
14	2.00 m	2.02 m	2.03 m	1.95 ddd(15, 7, 3)	1.97 m
	2.63 m	2.59 m	2.76 m	2.68 m	2.70 m
15	3.52 m	3.50 m	3.54 m	3.59 dd(11, 7)	3.59 m
17	3.70	3.72	3.61 d(10.5)	3.75	3.75
	3.70	3.72	3.81 d(10.5)	3.75	3.75
18	1.65 d(6)	1.64 d(6)	1.61 d(6.6)	1.66 dd(6.8, 1.6)	1.66 dd(6.6, 1)
19	5.37 q(6)	5.38 q(6)	5.31 q(6.6)	5.37 q(6.8)	5.37 q(6.6)
21	2.96 d(14)	2.98 d(14)	3.11 d(15)	2.95 d(10)	2.96 d(14)
	3.59 d(14)	3.62 d(14)	4.02 br d(15)	3.51 dd(10, 1.6)	3.53 br d(14)
NH	7.59 br s	7.60 br s	7.81 br s	7.69 br s	7.77 br s
16-COOMe	2.38 s	2.38 s	2.38 s	2.46 s	2.45 s
NMe	2.56 s	2.58 s		2.59 s	2.60 s
3′	2.73 br d(9)	2.72 m	2.76 m	2.43 m	2,42 br d(8)
	2.91 m	2.91 m	2.91 m	2.75 ddd(9, 4, 2)	2.74 dt(8, 3)
5′	2.91 m	2.91 m	2.99 m	2.97 m	2.97 m
	3.28 m	3.20 m	3.27 dt(13.5, 7)	3.31 m	3.25 m

Table 2-16-14 (continued)

H	2-16-44	2-16-45	2-16-46	2-16-47	2-16-48
6′	2.80 m	2.72 m	2.99 m	2.97 m	2.97 m
	3.02 m	2.91 m	2.99 m	2.97 m	2.97 m
9′	6.85 s	6.86 s	6.88 s	7.25 d(8.7)	7.25 d(8.8)
10′				6.83 d(8.7)	6.83 d(8.8)
12′	6.81 s	6.82 s	6.81 s		
14′	1.98 m	1.97 m	1.87 m	1.57 m	1.57 m
15′	1.51 m	1.54 m	1.87 m	1.30 m	1.37 dddd(14, 10, 4, 2)
	1.87 m	2.20 m	1.51 m	1.72 m	1.97 m
17′	1.87 m	1.87 dt(13, 3)	1.87 m	0.69 ddd(14, 4, 2)	0.77 dt(14, 3)
	2.52 m	2.57 m	2.55 br d(13.5)	1.72 m	1.76 dt(14, 2)
18′	1.06 d(6)	2.19 s	1.06 d(6)	1.00 d(6)	2.15 s
19′	4.10 br q(6)		4.10 q(6)	4.04 qd(6, 1)	
20′	1.40 m	2.42 m	1.40 m	1.24 m	2.23 ddd(10, 6.5, 1)
21′	3.73 br s	4.14 d(1)	3.73 br s	3.65 br s	4.07 d(1)
NH’	7.72 br s	7.72 br s	7.96 br s	7.50 br s	7.51 br s
11′-OMe	3.96 s	3.96 s	3.95 s	3.97 s	3.97 s
16′-COOMe	3.68 s	3.75 s	3.68 s	3.71 s	3.74 s
19′-OH			6.45 br s		

Table 2-16-15: ^{1}H NMR spectroscopic data of monoterpenoid indole alkaloids **2-16-49**~**2-16-53**.

H	2-16-49	2-16-50	2-16-51	2-16-52	2-16-53
3	5.34 dd(13, 3)	5.31 dd(13, 3)	5.33 dd(13, 3)	5.29 dd (13, 3)	5.29 dd (13, 3)
5	4.16 t (9)	3.94 m	3.99 m	3.97 t (9)	3.98 t (9)
6	3.62 m	3.36 m	3.39 dd (15, 8)	3.51 m	3.54 m
	3.76 m	3.59 m	3.63 m	3.51 m	3.54 m
9	7.73 br d(7.4)	7.60 br d(7.8)	7.62 br d(7.8)	7.69 br d(7.7)	7.69 br d(7.5)
10	7.15 td(7.4, 1)	7.11 m	7.11 m	7.14 td(7.7, 1)	7.14 td(7.5, 1)
11	7.10 td(7.4, 1)	7.11 m	7.11 m	7.09 td(7.7, 1)	7.08 td(7.5, 1)
12	7.03 br d(7.4)	7.04 br d(7.8)	7.07 br d(7.8)	7.03 br d(7.7)	7.02 br d(7.5)
14	2.01 ddd(15, 7, 3)	2.02 ddd(15, 7, 3)	2.02 ddd(15, 7, 3)	2.04 ddd(15, 7, 3)	2.01 ddd(15, 7, 3)
	2.81 m	2.69 m	2.74 m	2.68 m	2.73 m
15	3.62 m	3.59 m	3.63 m	3.65 m	3.62 dd (11, 5, 7)
17	3.72	3.75	3.76	3.79	3.76
	3.89 d(11.5)	3.75	3.76	3.79	3.76
18	1.65 d(6.5)	1.67 dd(6.8, 1.5)	1.68 dd(6.8, 1.6)	1.63 dd(6.7, 1.5)	1.61 dd(6.8, 1.5)
19	5.39 q(6.5)	5.38 q(6.8)	5.39 q(6.8)	5.38 q(6.7)	5.38 q(6.8)
21	3.23 d(15)	2.97 d(11)	2.97 d(13.6)	2.96 d(13)	2.97 dd(11.8, 1.5)
	3.98 m	3.57 d(11)	3.59 dd(13.6, 1.6)	3.53 dd(13, 1.5)	3.50 d(11.8)

Table 2-16-15 (continued)

H	2-16-49	2-16-50	2-16-51	2-16-52	2-16-53
NH	7.74 br s	7.59 br s	7.60 br s	7.89 br s	7.91 br s
16-COOMe	2.49 s	2.44 s	2.43 s	2.48 s	2.48 s
NMe		2.57 s	2.59 s	2.60 s	2.61 s
3′	2.44 br d(9)	2.42 m	2.45 m	2.46 br d(9.8)	2.46 m
	2.75 m	2.81 dt(8, 3)	2.84 dt(9, 2.5)	2.75 m	2.67 m
5′	2.95 m	3.11 m	3.06 m	2.91 m	3.09 m
	3.31 m	3.21 m	3.15 m	3.30 dt(14, 7)	3.24 dt(14, 7.5)
6′	2.95 m	2.61 m	2.53 m	2.99 m	2.83 m
	2.95 m	3.11 m	3.06 m	2.99 m	3.09 m
9′	7.25 d(8.7)	7.23 d(8.6)	7.24 d(8.7)	7.13 d(8.4)	7.13 d(8.5)
10′	6.84 d(8.7)	6.82 d(8.6)	6.83 d(8.7)	6.67 d(8.4)	6.66 d(8.5)
14′	1.55 m	1.69 m	1.71 m	1.58 m	1.51 m
15′	1.30 m	1.37 m	1.37 m	1.31 m	0.93 m
	1.69 m	1.80 br dd(13, 6)	2.24 m	1.73 m	1.45 m
16′		2.37 m	2.48 m		
17′	0.68 br d(14)	0.84 br d(14)	0.99 br d(14)	0.71 br d(13.5)	0.68 br d(14)
	1.69 m	1.13 m	1.26 m	1.70 m	1.75 br d(14)
18′	0.99 d(6)	1.04 d(6.3)	2.15 s	1.01 d(6.3)	0.81 t(7)
19′	4.04 qd(6, 1)	4.07 qd(6.3, 1.3)		4.06 qd(6.3, 2)	1.34 m, 1.45 m
20′	1.24 m	1.49 br dd(11, 6)	2.48 m	1.24 m	1.10 m
21′	3.64 br s	2.95 br s	3.44 t(2)	3.76 br s	3.34 br s
NH’	7.43 br s	7.82 br s	7.79 br s	7.52 br s	7.52 br s
11′-OMe	3.98 s	3.96 s	3.97 s		
16′-COOMe	3.70 s			3.72 s	3.70 s

Table 2-16-16: ^{1}H NMR spectroscopic data of monoterpenoid indole alkaloids **2-16-54**~**2-16-58**.

H	2-16-54	2-16-55	2-16-56	2-16-57	2-16-58
1					8.91 s
2	3.87 d(11.3)				
3	3.70	2.80 m, 2.97 m	2.80 m, 3.01 m	2.73 m, 2.98 m	4.84 d(8.0)
5	3.25, 2.90	3.36 m, 3.69 m	3.32 m, 3.34 m	3.26 m, 3.28 m	2.90 m, 2.97 m
6	2.60	2.46 dd(15.6, 6.8)	2.47 dd(16.0, 6.0)	2.43 (ov)	1.64 m
	1.95	3.15 m	3.11 m	3.06 m	1.99 m
9	7.24 d	7.31 d(8.0)	7.32 d(7.5)	7.27 d(8.0)	6.02 d(7.3)
10	6.99 t	6.83 t(8.0)	6.84 t(7.5)	6.79 t(8.0)	6.39 t(7.3)
11	7.23 t	6.70 t(8.0)	6.67 t(7.5)	6.57 t(8.0)	6.97 t(7.3)
12	6.77 d	6.39 d(8.0)	6.47 t(7.5)	6.10 d(8.0)	6.69 d(7.3)

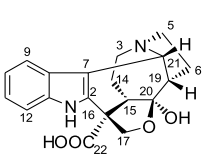
Table 2-16-16 (continued)

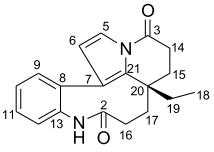
H	2-16-54	2-16-55	2-16-56	2-16-57	2-16-58
14	2.30 (13.7) 1.80 (13.7)	5.55 m	5.90 br s	5.56 (ov)	5.09 dd(8.0, 3.5)
15	3.24	5.64 d(10.4)	5.56 (ov)	5.56 (ov)	4.14 d(3.5)
16	2.10 (11.3, 10.2)	4.66 dd(11.6, 4.0)	4.61 dd(9.8, 3.6)	5.04 br d(10.6)	
17	5.15 d(10.2)	1.94 dd(14.6, 11.6)	1.95 (ov)	1.88 dd(13.8, 10.6)	2.40 d(15.5)
		2.17 dd(14.0, 4.0)	2.13 dd(13.0, 3.5)	2.13 br d(13.8)	2.76 d(15.5)
18	4.25 (12.6, 6.4) 4.10 (12.6, 6.4)	0.92 t(7.6)	0.88 t(7.5)	0.88 t(7.5)	0.68 t(7.0)
19	6.14 t(6.4)	1.52 dq(15.2, 7.6)	1.51 dq(13.0, 7.5)	1.61 m	0.85 qd(14.8, 7.0)
		1.86 dq(15.2, 7.6)	1.84 dq(13.0, 7.5)	1.82 m	1.14 qd(14.8, 7.0)
21	3.75 (16.0) 3.25 (16.0)	4.08 s	4.06 s	4.09 s	2.58 s
1′				9.61 s	8.99 s
3′		3.31 m	4.31 m	2.82 m	3.20 d(16.0)
		3.33 m	4.50 m	3.21 m	3.48 dd(16.0, 3.3)
5′	4.55	3.24 m	4.13 (ov)	2.42 m	2.73 m
		3.26 m	4.13 (ov)	2.85 m	3.06 dd(15.0, 6.5)
6′	3.30	1.74 (ov)	2.29 m	1.58 m	1.78 dd(11.5, 4.5)
		2.21 m	2.34 m	1.77 m	2.07 m
9′	7.46 d	7.11 br s	7.55 s	6.87 s	7.20 s
10′	7.09 t				
11′	7.32 t	7.00 d(8.0)	7.03 d(7.7)	–	–
12′	7.70 d	6.66 d(8.0)	6.70 d(7.7)	6.72 s	6.33 s
14′	8.58 s	5.67 m	5.56 (ov)	5.56 (ov)	5.80 (ov)
15′		6.06 d(9.6)	6.25 br s	5.48 d(10.0)	5.80 (ov)
16′		3.07 dd(12.0, 6.4)	3.26 dd(12.4, 5.5)		
17′	7.20 s	1.74 (ov)	1.95 (ov)	2.29 (ov)	2.46 d(16.0)
		2.35 dd(13.6, 6.4)	2.40 m	2.29 (ov)	2.62 d(16.0)
18′	1.40 t	0.99 d(6.8)	1.00 br s	0.36 t(7.4)	0.80 t(7.5)
19′	2.90	1.98 q(6.8)	2.03 m	0.53 m	0.99 dq(15.3, 7.5)
	2.80			0.78 m	1.24 dq(15.3, 7.5)
21′	7.83 s	3.18 s	3.78 br s	2.15 s	2.69 s
16-COOMe					3.77 s
16′-COOMe		3.64 s	3.69 s	3.66 s	3.78 s
OH				9.67 s	

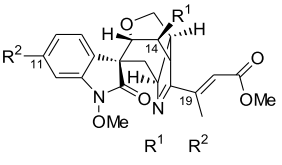
2.16.3 Other monoterpenoid indole alkaloids

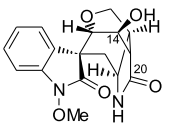
Table 2-16-17: Cos, MFs, and TSs of monoterpenoid indole alkaloids **2-16-59**~**2-16-67**.

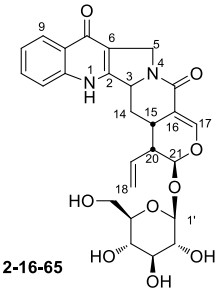
No.	Compounds	MFs	Test solvents	References
2-16-59	actinophyllic acid	$C_{19}H_{20}N_2O_4$	DMSO-d_6	[462]
2-16-60	3-oxo-rhazinilam	$C_{19}H_{20}N_2O_2$	C_5D_5N	[463]
2-16-61	gelsecrotonidine	$C_{22}H_{24}N_2O_5$	$CDCl_3$	[464]
2-16-62	14-hydroxygelsecrotonidine	$C_{22}H_{24}N_2O_6$	$CDCl_3$	[464]
2-16-63	11-methoxygelsecrotonidine	$C_{23}H_{26}N_2O_6$	$CDCl_3$	[464]
2-16-64	14-hydroxygelsedilam	$C_{17}H_{18}N_2O_5$	CD_3OD	[464]
2-16-65	pumiloside	$C_{26}H_{28}N_2O_9$	DMSO-d_6	[453]
2-16-66	croceaine A	$C_{27}H_{32}N_2O_9$	CD_3OD	[465]
2-16-67	croceaine B	$C_{27}H_{28}N_2O_9$	CD_3OD	[465]

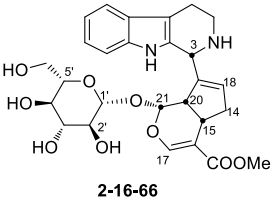












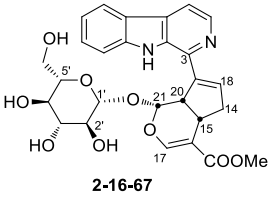


Table 2-16-18: ^{1}H NMR spectroscopic data of monoterpenoid indole alkaloids **2-16-59**~**2-16-63**.

H	2-16-59	2-16-60	2-16-61	2-16-62	2-16-63
1	11.70 s				
3	2.84 m		3.74 dd(4.8, 2.1)	3.68 br dd(1.9, 1.9)	3.71 dd(4.3, 1.7)

Table 2-16-18 (continued)

H	2-16-59	2-16-60	2-16-61	2-16-62	2-16-63
5	3.41 ddd(3.5, 9.0, 12.0)	7.55 d(3.2)	4.67 br ddd(7.7, 5.0, 2.4)	4.67 br ddd(7.6, 5.0, 2.9)	4.65 ddd(7.9, 5.0, 2.4)
	3.54 ddd(7.2, 12.0, 12.0)				
6	2.26 ddd(3.5, 12.0, 12.0)	5.94 d(3.2)	2.51 dd(15.5, 5.0)	2.54 dd(15.8, 5.0)	2.47 dd(15.4, 5.0)
			2.35 dd(15.5, 2.4)	2.38 dd(15.8)	2.32 dd(15.4, 2.4)
	2.50 m				
9	7.51 d(7.8)	7.50 d(7.3)	7.54 d(7.6)	7.53 d(7.5)	7.41 d(8.3)
10	6.91 t(7.8)	7.33~7.47 m	7.08 ddd(7.6, 7.6, 1.0)	7.10 ddd(7.5, 7.5, 1.1)	6.57 dd(8.3, 2.4)
11	7.01 t(7.8)	7.33~7.47 m	7.27 ddd(7.6, 7.6, 1.0)	7.29 ddd(7.5, 7.5, 1.1)	3.81 s(OMe)
12	7.44 d(7.8)	7.33~7.47 m	6.89 d(7.6)	6.89 d(7.5)	6.47 d(2.4)
14	2.09 br ddd(3.6, 12.0, 12.0)	*α* 2.88 ddd(18.2, 13.5, 5.3)	2.41 dd(14.9, 2.1)	4.49 d(1.9)	2.38 dd(15.0, 1.7)
			2.24 ddd(14.9, 9.5, 4.8)		2.23 ddd(15.0, 9.5, 4.3)
	2.20 br dd(2.1, 12.0)	*β* 2.60 ddd(18.2, 4.7, 3.2)			
15	2.89 d(2.0)	*α* 1.91 ddd(14.1, 13.5, 4.7)	3.33 dd(9.5, 9.5)	3.33 br d(8.5)	3.32 dd(9.5, 9.5)
		β 1.50 ddd(14.1, 5.3, 3.2)			
16		2.51 dd(14.1, 12.3)	2.64 m	2.67 br ddd(8.5, 8.5, 3.4)	2.63 m
		2.19 dd(14.1, 7.9)			
17	3.36 d(7.2)	2.65 dd(13.2, 12.3)	4.34 dd(11.3, 2.8)	4.50 dd(10.9, 3.4)	4.33 dd(11.2, 3.0)
	3.94 d(7.2)	1.46 dd(13.2, 7.9)	4.30 dd(11.3, 1.8)	4.37 d(10.9)	4.29 dd(11.2, 1.5)
18		0.62 t(7.3)	2.59 br d(1.3)	2.58 br d(1.4)	2.58 d(0.7)
19	2.94 dd(9, 6.6)	1.47 m			
		1.29 m			
21	5.26 d(6.6)		6.20 br d(1.3)	6.41 br d(1.4)	6.20 br d(0.7)
23			3.77 s	3.78 s	3.77 s
20-OH	5.90 br s				
NOMe			3.92 s	3.92 s	3.91 s
NH			10.00 s		

Table 2-16-19: ^{1}H NMR spectroscopic data of monoterpenoid indole alkaloids **2-16-64**~**2-16-67**.

H	2-16-64	2-16-65	2-16-66	2-16-67
3	3.56 s	4.72 d(11.8)	4.94 s	
5	3.99 m	4.31 d(14.1)	3.08~3.14 m	8.24 d(5.2)
		4.46 d(14.1)		
6	2.39 dd(15.7, 3.8)		2.78~2.84 m	7.96 d(5.2)
	1.91 br dd(15.7, 2.5)			
9	7.47 d(7.7)	8.10 d(8.0)	7.40 d(7.5)	8.16 d(8.0)
10	7.04 ddd(7.7, 7.7, 1.1)	7.31 t(8.0)	6.97 td(7.5, 1.2)	7.24 t(8.0)
11	7.24 ddd(7.7, 7.7, 1.1)	7.64 t(8.0)	7.05 td(7.5, 1.2)	7.50 t(8.0)
12	6.90 d(7.7)	7.60 d(8.0)	7.28 d(7.5)	7.61 d(8.0)
14	4.41 br d(2.2)	1.98 q(11.8)	α 2.14 ddd(15.9, 9.3, 1.0)	α 2.64 m
		2.50 (ov)	β 2.92 ddd(15.9, 8.0, 2.7)	β 3.12 m
15	2.44 d(8.4)	3.28 m	3.30 m	3.50 m
16	2.79 m			
17	4.27 dd(10.9, 3.0)	7.04 d(2.3)	7.55 s	7.51 s
	4.20 d(10.9)			
18		5.32 d(10.9)	5.85 br s	6.59 br s
		5.44 d(17.2)		
19		5.74 m		
20		2.62 m	2.82 m	3.71 m
21		5.39 br s	5.16 d(9.0)	5.50 d(6.3)
1′		4.54 d(7.9)	4.80 d(7.8)	4.43 d(7.8)
2′		2.98 m	3.16 dd(9.0, 7.8)	2.47 dd(9.0, 7.8)
3′		3.18 m	3.36 t(9.0)	3.12 t(9.0)
4′		3.04 m	3.07 t(9.0)	2.64 t(9.0)
5′		3.18 m	3.27 m	3.24 m
6′		3.42 m	3.58 dd(12.0, 6.6)	3.67 dd(11.7, 6.6)
		3.69 d(11.3)	3.84 dd(12.0, 2.4)	3.86 m
16-COOMe			3.71 s	3.75 s
NOMe	3.87 s			

Bibliography

[448] Kam TS, Choo YM. J Nat Prod, 2004, 67: 547.
[449] Kam TS, Choo YM. Tetrahedron, 2000, 56: 6143.
[450] Kam TS, Sim KW, Koyano T, et al. Phytochemistry, 1999, 50: 75.
[451] Penelle J, Tits M, Christen P, et al. Phytochemistry, 2000, 53: 1057.
[452] Kam TS, Iek IH, Choo YM. Phytochemistry, 1999, 51: 839.
[453] Zhang ZZ, Elsohly HN, Jacob MR, et al. J Nat Prod, 2001, 64: 1001.
[454] Pham VC, Ma J, Thomas SJ, et al. J Nat Prod, 2005, 68: 1147.
[455] Sheludko Y, Gerasimenko I, Kolshorn H, et al. J Nat Prod, 2002, 65: 1006.
[456] Feng T, Li Y, Cai XH, et al. J Nat Prod, 2009, 72: 1836.
[457] Feng T, Cai XH, Liu YP, et al. J Nat Prod, 2010, 73: 22.
[458] Lim KH, Hiraku O, Komiyama K, et al. J Nat Prod, 2008, 71: 1591.
[459] Kam TS, Sim KM, Pang HS. J Nat Prod, 2003, 66: 11.

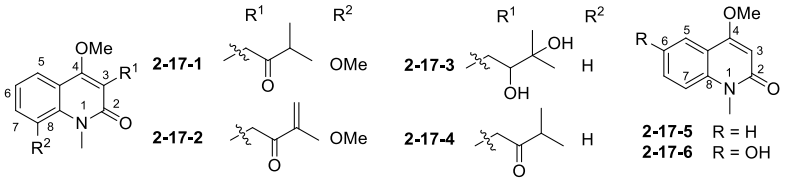
[460] Biala RG, Tits M, Penelle J, et al. J Nat Prod, 1998, 61: 139.
[461] Feng T, Li Y, Wang YY, et al. J Nat Prod, 2010, 73: 1075.
[462] Carroll AR, Hyde E, Smith J, et al. J Org Chem, 2005, 70: 1096.
[463] Gerasimenko I, Sheludko Y, Stöckigt J. J Nat Prod, 2001, 64: 114.
[464] Yamada Y, Kitajima M, Kogure N, et al. Tetrahedron, 2008, 64: 7690.
[465] Dusman LT, Jorge TCM, Souza MCD, et al. J Nat Prod, 2004, 67: 1886.

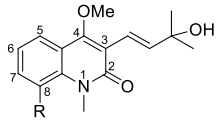
2.17 Quinoline alkaloids

2.17.1 2-oxo-type quinoline alkaloids

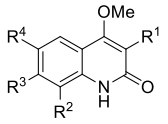
Table 2-17-1: Cos, MFs, and TSs of 2-oxo-type quinoline alkaloids **2-17-1**~**2-17-27**.

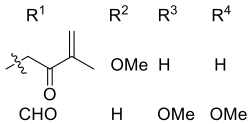
No.	Compounds	MFs	Test solvents	References
2-17-1	orixalone A	$C_{17}H_{21}NO_4$	–	[466]
2-17-2	orixalone B	$C_{17}H_{19}NO_4$	–	[466]
2-17-3	edulinine	$C_{16}H_{21}NO_4$	$CDCl_3$	[467]
2-17-4	orijanone	$C_{16}H_{19}NO_3$	$CDCl_3$	[468]
2-17-5	4-methoxy-1-methyl-2-quinolone	$C_{11}H_{11}NO_2$	$CDCl_3$	[469]
2-17-6	4-methoxy-6-hydroxy-l-methyl-2-quinolone	$C_{11}H_{11}NO_3$	C_5D_5N	[470]
2-17-7	glycocitlone A	$C_{16}H_{19}NO_3$	$CDCl_3$	[471]
2-17-8	glycocitlone B	$C_{16}H_{19}NO_4$	$CDCl_3$	[471]
2-17-9	glycocitlone C	$C_{17}H_{21}NO_4$	$CDCl_3$	[471]
2-17-10	orixalone C	$C_{16}H_{17}NO_4$	–	[466]
2-17-11	melisemine	$C_{13}H_{13}NO_5$	$CDCl_3$	[472]
2-17-12	semecarpifoline	$C_{13}H_{15}NO_4$	$CDCl_3$	[473]
2-17-13	4-carbomethoxy-6-hydroxy-2-quinolone	$C_{11}H_9NO_4$	DMSO-d_6	[474]
2-17-14	7-hydroxy-4-(5′-hydroxymethylfuran-2′-yl)-2-quinolone	$C_{14}H_{11}NO_4$	DMSO-d_6	[475]
2-17-15	6-bromo-4-hydroxy-2-quinolone	$C_9H_6BrNO_2$	DMSO-d_6	[476]
2-17-16	6,7-dibromo-4-hydroxy-2-quinolone	$C_9H_5Br_2NO_2$	DMSO-d_6	[476]
2-17-17	severibuxine	$C_{29}H_{39}NO_3$	$CDCl_3$	[477]
2-17-18	pinolinone	$C_{15}H_{19}NO_3$	DMSO-d_6	[478]
2-17-19	haplotubinone	$C_{19}H_{23}NO_4$	CD_3OD	[479]
2-17-20	penicillazine	$C_{20}H_{20}N_2O_9$	DMSO-d_6	[480]
2-17-21	peniprequinolone	$C_{22}H_{25}NO_5$	$CDCl_3$	[481]
2-17-22	3-methoxy-4-hydroxy-4-(4′-methoxyphenyl)qui-nolinone	$C_{17}H_{17}NO_4$	$CDCl_3$	[481]
2-17-23	3-methoxy-4,6-dihydroxy-4-(4′-methoxyphenyl)-quinolinone	$C_{17}H_{17}NO_5$	$CDCl_3$	[481]
2-17-24	*N*-mercapto-4-formylcarbostyril	$C_{10}H_7NO_2S$	CD_3COCD_3	[482]
2-17-25	isaindigotidione	$C_{23}H_{22}N_2O_5$	DMSO-d_6	[483]
2-17-26	megistoquinone Π	$C_{14}H_{13}NO_6$	$CDCl_3$	[484]
2-17-27	simulenoline	$C_{20}H_{23}NO_3$	$CDCl_3$	[485]



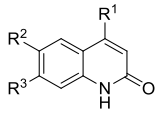


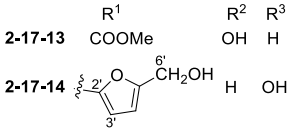
2-17-7 R = H
2-17-8 R = OH
2-17-9 R = OMe





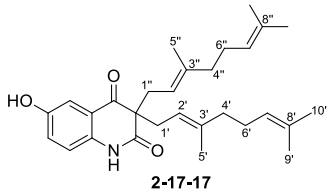
	R1	R2	R3	R4
2-17-10		OMe	H	H
2-17-11	CHO	H	OMe	OMe
2-17-12	CH_2OMe	H	OMe	H



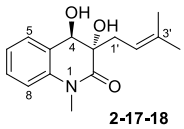


	R1	R2	R3
2-17-13	COOMe	OH	H
2-17-14		H	OH

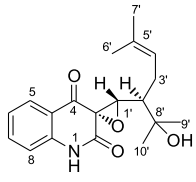
	R1	R2	R3
2-17-15	OH	Br	H
2-17-16	OH	Br	Br



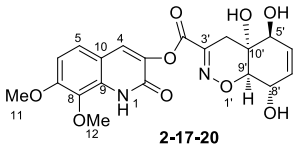
2-17-17



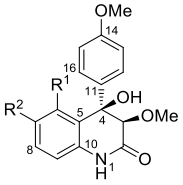
2-17-18



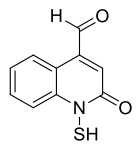
2-17-19



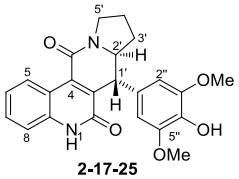
2-17-20



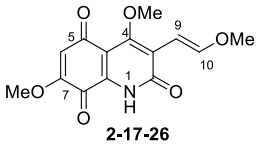
	R1	R2
2-17-21	OH	
2-17-22	H	H
2-17-23	OH	H



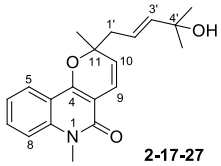
2-17-24



2-17-25



2-17-26



2-17-27

Table 2-17-2: ^{1}H NMR spectroscopic data of 2-oxo-type quinoline alkaloids **2-17-1**~**2-17-5**.

H	2-17-1	2-17-2	2-17-3	2-17-4	2-17-5
3					6.05 s
4	3.86 s(OMe)	3.86 s(OMe)	3.97 s(OMe)	3.90 s(OMe)	3.95 s(OMe)
5	7.44 br d(8.1)	7.45 br d(8.1)	7.86 dd(8, 1.5)	7.84 dd(8.3, 1.5)	7.34 d(8.1)
6	7.18 t(8.1)	7.19 t(8.1)	7.32 td (7.5, 1)	7.27 ddd(8.3, 7.3, 1.5)	7.22 dd(1.0, 8.1)
7	7.07 br d(8.1)	7.07 br d(8.1)	7.61 dddd(8, 7.5, 1.5)	7.58 ddd(8.3, 7.3, 1.5)	7.59 dtd(1.5, 1.6, 7.9)

Table 2-17-2 (continued)

H	2-17-1	2-17-2	2-17-3	2-17-4	2-17-5
8	3.90 s(OMe)	3.90 s(OMe)	7.44 dd(8, 1)	7.39 br d(8.3)	7.99 dd(1.6, 8.0)
1′	3.82 s	4.08 s	3.13 dd(14, 2) 2.74 dd(14, 10)	3.85 s	
2′			3.60 dd(10, 2)		
3′	2.89 sept(7.0)			2.90 sep(6.8)	
4′	1.22 d(7.0)	1.94 s	1.32 s	1.23 d(6.8)	
5′	1.22 d(7.0)	5.85 br s 6.19 br s	1.33 s	1.23 d(6.8)	
NMe	3.94 s	3.94 s	3.76 s	3.71 s	3.65 s

Table 2-17-3: ^{1}H NMR spectroscopic data of 2-oxo-type quinoline alkaloids **2-17-6**~**2-17-10**.

H	2-17-6	2-17-7	2-17-8	2-17-9	2-17-10
3	6.12 s				
4	3.69 s(OMe)	3.92 s(OMe)	3.85 s(OMe)	3.87 s(OMe)	3.89 s(OMe)
5	7.77 d(2.4)	7.90 dd(1.5, 8.1)	7.44 br d(8.1)	7.51 dd(1.5, 8.1)	7.35 br d(8.1)
6		7.27 br t(8.1)	7.05 t(8.1)	7.18 t(8.1)	7.16 t(8.1)
7	7.44 dd(9.0, 2.4)	7.57 br t(8.1)	6.96 br d(8.1)	7.06 dd(1.5, 8.1)	6.97 br d(8.1)
8	7.21 d(9.0)	7.37 br d(8.1)		3.90 s(OMe)	3.98 s(OMe)
1′		6.85 d(16.1)	6.89 d(16.1)	6.83 d(16.1)	4.09 s
2′		7.26 d(16.1)	7.14 d(16.1)	7.24 d(16.1)	
3′		1.71 s(OH)		1.99 s(OH)	
4′		1.46 s	1.44 s	1.45 s	1.94 s
5′		1.46 s	1.44 s	1.45 s	5.84 br s, 6.17 br s
NMe	3.63 s	3.74 s	4.00 s	3.94 s	
NH					9.13 br s

Table 2-17-4: ^{1}H NMR spectroscopic data of 2-oxo-type quinoline alkaloids **2-17-11**~**2-17-15**.

H	2-17-11	2-17-12	2-17-13	2-17-14	2-17-15
1					11.98 s
2			3.92 s(OMe)		
3			6.88 s	7.22 s	8.01 d(2.7)
3	10.49 s(CHO)				
4	4.19 s (OMe)	4.10 s(OMe)			3.37 s
5	7.33 s	7.73 d(8.8)	7.49 d(2.6)	6.65 d(8.0)	8.07 s
6	3.96 s(OMe)	6.81 dd(8.8, 2.4)	9.54 s(OH)	6.30 dd(2.4, 8.0)	
7	4.02 s(OMe)	3.89 s(OMe)	7.08 dd(2.6, 8.9)	8.99 br s(OH)	7.39 d(8.5)
8	6.71 s	6.61 d(2.4)	7.24 d(8.9)	7.79 d(2.4)	7.26 d(8.8)

Table 2-17-4 (continued)

H	2-17-11	2-17-12	2-17-13	2-17-14	2-17-15
1′		4.54 s			
3′		3.47 s		7.16 d(3.4)	
4′				6.59 d(3.4)	
6′				4.64 s	
				5.47 br s(OH)	
NH	11.20 br s	10.25 br s	11.95 s	10.2 br s	

Table 2-17-5: ^{1}H NMR spectroscopic data of 2-oxo-type quinoline alkaloids **2-17-16**~**2-17-20**.

H	2-17-16	2-17-17	2-17-18	2-17-19	2-17-20
1					
3	8.08 s		5.15 br s(OH)		
4	3.36 s		4.54 d(4.8)		8.59 s
			5.65 d(4.8, OH)		
5	8.28 s	7.40 d(2.4)	7.38 d(7.8)	7.89 d(7.4)	7.30 d(9.0)
6		6.32 br s(OH)	7.05 t(7.8)	7.22 t(7.6)	7.08 d(9.0)
7		7.07 dd(8.4, 2.4)	7.28 t(7.8)	7.63 dd(6.9, 7.2)	
8	7.86 s	6.82 d(8.4)	7.06 d(7.8)	7.16 d(8.1)	
11					3.97 s
12					3.92 s
NH	12.07 s	8.97 br s	3.30 s		9.40 br s
1′		2.78 dd(13.2, 7.6)	2.30 dd(15.0, 7.7)	3.42 d(9.5)	
		2.72 dd(13.2, 7.6)	2.15 dd(15.0, 7.7)		
2′		4.91 t(7.6)	5.04 br t(7.7)	1.82 ddd(11.1, 3.4, 3.4)	
3′				2.22 d(15.5)	
				2.07~2.14 m	
4′		1.77 br s	1.55 s	4.52 d(7.3)	2.28 d(19.5)
					2.74 dd(19.5, 2.5)
5′		1.57 s	1.37 s		4.39 dd(5.5, 2.0)
6′		1.77 br s		0.85 s	5.62 ddd(10.0, 2.0, 2.0)
7′		4.88 m		1.42 s	5.55 ddd(10.0, 2.0,2.0)
8′		1.45 s, 1.52 s		1.38 s, 1.39 s	4.15 ddd(5.5, 7.5, 2.0)
9′					4.12 dd(7.5, 2.5)
1″		2.78 dd(13.2, 7.6)			
		2.72 dd(13.2, 7.6)			
2″		4.91 t(7.6)			
4″		1.77 br s			
5″		1.57 s			
6″		1.77 br s			
7″		4.88 m			
8″		1.45 s, 1.52 s			
OH					4.48 s, 4.67 s

Table 2-17-6: 2-17-6 ^{1}H NMR spectroscopic data of 2-oxo-type quinoline alkaloids **2-17-21**~**2-17-25**.

H	2-17-21	2-17-22	2-17-23	2-17-24	2-17-25
3	3.67 s 3.60 s(OMe)	3.76 s 3.55 s(OMe)	3.76 s 3.61 s(OMe)	8.65 s	
4	4.55 s(OH)		4.52 s(OH)	10.1 s(CHO)	
5					8.76 d(7.7)
6	8.90 s(OH)	7.40 dd(7.6, 1.5)	8.73 s(OH)		7.19 t(7.7)
7		7.08 m	6.64 dd(8.3, 1.0)	7.95 dd(8.35, 1.16)	7.47 t(7.7)
8	7.02 d(8.0)	7.28 m	7.17 dd(8.3, 8.1)	7.48 t-like(8.43, 7.14, 1.29)	7.31 d(7.7)
9	6.29 d(8.0)	6.84 dd(7.8, 1.5)	6.34 dd(8.3, 1.0)	7.05 t(7.33, 8.15)①	
10				7.12 d(8.36)	
12,16	7.16 d(8.5)	7.19 d(9.0)	7.16 d(9.0)		
13,15	6.81 d(8.5)	6.82 d(9.0)	6.82 d(9.0)		
14	3.75 s(OMe)	3.76 s(OMe)	3.70 s(OMe)		
17	3.23 m				
18	5.28 dt(7.0, 1.0)				
20	1.73 s				
21	1.67 s				
NH	8.21 s	8.39 s	7.78 s	11.55 br s(NSH)	11.7 s
1′					3.89 d(12.2)
2′					3.83 m
3′					1.86 m, 1.79 m
4′					1.94 m, 1.72 m
5′					3.68 m, 3.56 m
2″					6.44 s
3″					3.67 s(OMe)
4″					8.10 s(OH)
5″					3.67 s(OMe)
6″					6.44 s

① Typographic error exists in the literature, giving one more coupling constant.

Table 2-17-7: ^{1}H NMR spectroscopic data of 2-oxo-type quinoline alkaloids **2-17-26** and **2-17-27**.

H	2-17-26	2-17-27	H	2-17-26	2-17-27
4	3.85 s(OMe)		1′		2.47 d(6.4)
5		7.94 dd(7.8, 1.4)	2′		5.69 dt(16.0, 6.4)
6	5.94 s	7.23 t(7.8)	3′		5.63 d(16.0)
7	3.89 s(OMe)	7.55 ddd(7.8, 7.8, 1.4)	5′		1.18 s
8		7.32 br d(7.8)	6′		1.18 s
9	6.19 d(12.0)	6.80 d(10.0)	NMe		3.69 s
10	8.42 d(12.0) 3.80 s(OMe)	5.49 d(10.0)	NH	9.53 br s	
11		1.51 s			

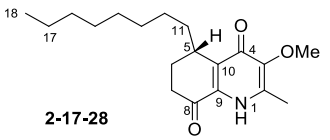
2.17.2 4-oxo-type quinoline alkaloids

Table 2-17-8: Cos, MFs, and TSs of 4-oxo-type quinoline alkaloids **2-17-28**~**2-17-59**.

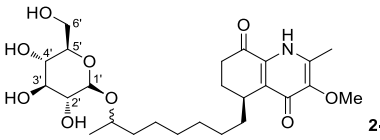
No.	Compounds	MFs	Test solvents	References
2-17-28	(*S*)-antidesmone	$C_{19}H_{29}NO_3$	$CDCl_3$	[486]
2-17-29	(17*RS*)-17-(*β*-D-glucopyranosyloxy)antidesmone	$C_{26}H_{41}NO_9$	CD_3OD	[487]
2-17-30	(17*RS*)-8-deoxo-17-(*β*-D-glucopyranosyloxy)antidesmone	$C_{26}H_{43}NO_8$	CD_3OD	[487]
2-17-31	5,6-dimethoxy-2-(3-methoxyphenyl)-1*H*-quinolin-4-one	$C_{18}H_{17}NO_4$	CD_3OD	[488]
2-17-32	5,6-dimethoxy-2-(3,4-dimethoxyphenyl)-1*H*-quinolin-4-one	$C_{19}H_{19}NO_5$	CD_3OD	[488]
2-17-33	5,6-dimethoxy-2-(2,5,6-trimethoxyphenyl)-1*H*-quinolin-4-one	$C_{20}H_{21}NO_6$	CD_3OD	[488]
2-17-34	quinolactacin A1	$C_{16}H_{18}N_2O_2$	$CDCl_3$	[489]
2-17-35	quinolactacin A2	$C_{16}H_{18}N_2O_2$	$CDCl_3$	[489]
2-17-36	cyclomegistine	$C_{18}H_{21}NO_7$	$CDCl_3$	[490]
2-17-37	eduline	$C_{17}H_{15}NO_2$	$CDCl_3$	[469]
2-17-38	2-(12-oxo-tridecanyl)-3-methoxy-4-quinolone	$C_{23}H_{33}NO_3$	$CDCl_3$	[491]
2-17-39	2-(10-hydroxy-10-methyldodecanyl)-3-methoxy-4-quinolone	$C_{23}H_{35}NO_3$	$CDCl_3$	[491]
2-17-40	2-(11-hydroxy-11-methyldodecanyl)-3-methoxy-4-quinolone	$C_{23}H_{35}NO_3$	$CDCl_3$	[491]
2-17-41	2-(12-hydroxytridecanyl)-3-methoxy-4-quinolone	$C_{23}H_{35}NO_3$	$CDCl_3$	[491]
2-17-42	2-(14′-hydroxy-14′,15′-dimethylhexadecanyl)-3-methoxy-4-quinolone	$C_{28}H_{45}NO_3$	$CDCl_3$	[492]
2-17-43	2-(12′-hydroxy-12′-methyltridecanyl)-3-methoxy-4-quinolone	$C_{24}H_{37}NO_3$	$CDCl_3$	[492]
2-17-44	2-(14′-hydroxy-14′,15′-dimethylhexadecanyl)-4-quinolone	$C_{27}H_{43}NO_2$	$CDCl_3$	[492]
2-17-45	2-(12′-hydroxy-12′-methyltridecanyl)-4-quinolone	$C_{23}H_{35}NO_2$	$CDCl_3$	[492]
2-17-46	2-{6′-(2*H*-benzo[*d*]1″,3″-dioxolen-5″-yl)hexyl}-hydroquino-lin-4-one	$C_{22}H_{23}NO_3$	$CDCl_3$	[493]
2-17-47	2-(nonan-8-one)-(1*H*)-4-quinoline	$C_{18}H_{23}NO_2$	$CDCl_3$	[494]
2-17-48	2-[4′-(3′,4′-methylenedioxyphenyl)butyl]-4-quinolone	$C_{20}H_{19}NO_3$	$CDCl_3$	[495]
2-17-49	7-hydroxy-2-(3-hydroxy-3-methylbutyl)-4-quinolone	$C_{14}H_{17}NO_3$	DMSO-d_6	[491]
2-17-50	6-hydroxy-2-(3-hydroxy-3-methylbutyl)-4-quinolone	$C_{14}H_{17}NO_3$	DMSO-d_6	[491]
2-17-51	6-methoxydictyolomide A	$C_{20}H_{25}NO_2$	$CDCl_3$	[492]

Table 2-17-8 (continued)

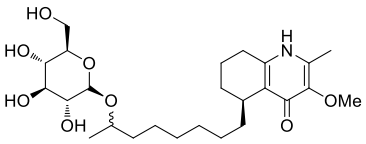
No.	Compounds	MFs	Test solvents	References
2-17-52	chestnutamide	$C_{14}H_{12}N_2O_2$	$CDCl_3$	[496]
2-17-53	megistonine II	$C_{14}H_{15}NO_6$	$CDCl_3$	[497]
2-17-54	megistonine I	$C_{18}H_{21}NO_5$	$CDCl_3$	[497]
2-17-55	–	$C_{24}H_{22}Br_2N_6O_8$	DMSO-d_6	[498]
2-17-56	sarcomejine	$C_{16}H_{19}NO_6$	$CDCl_3$	[499]
2-17-57	1-methyl-2-[6′-(3′,4′-methylenedioxyphenyl)hexyl]-4-quinolone	$C_{23}H_{25}NO_3$	$CDCl_3$	[495]
2-17-58	graveoline	$C_{17}H_{13}NO_3$	$CDCl_3$	[500]
2-17-59	1-methyl-2-tetredecyl-4(1*H*)-quinolone	$C_{24}H_{37}NO$	$CDCl_3$	[501]



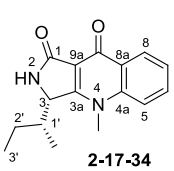
2-17-28



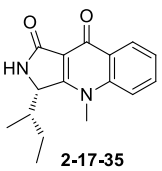
2-17-29



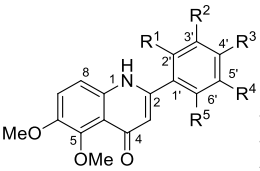
2-17-30



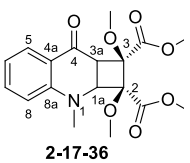
2-17-34



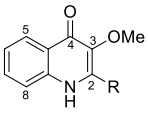
2-17-35



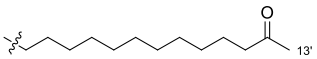
	R^1	R^2	R^3	R^4	R^5
2-17-31	H	OMe	H	H	H
2-17-32	H	OMe	OMe	H	H
2-17-33	OMe	H	H	OMe	OMe



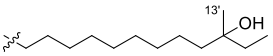
2-17-36



2-17-38 R =



2-17-39 R =



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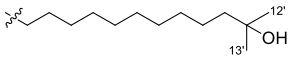
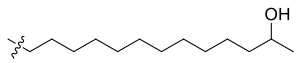
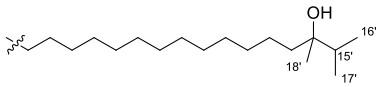


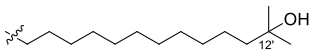
2-17-41 R =



2-17-42 R =



2-17-43 R =



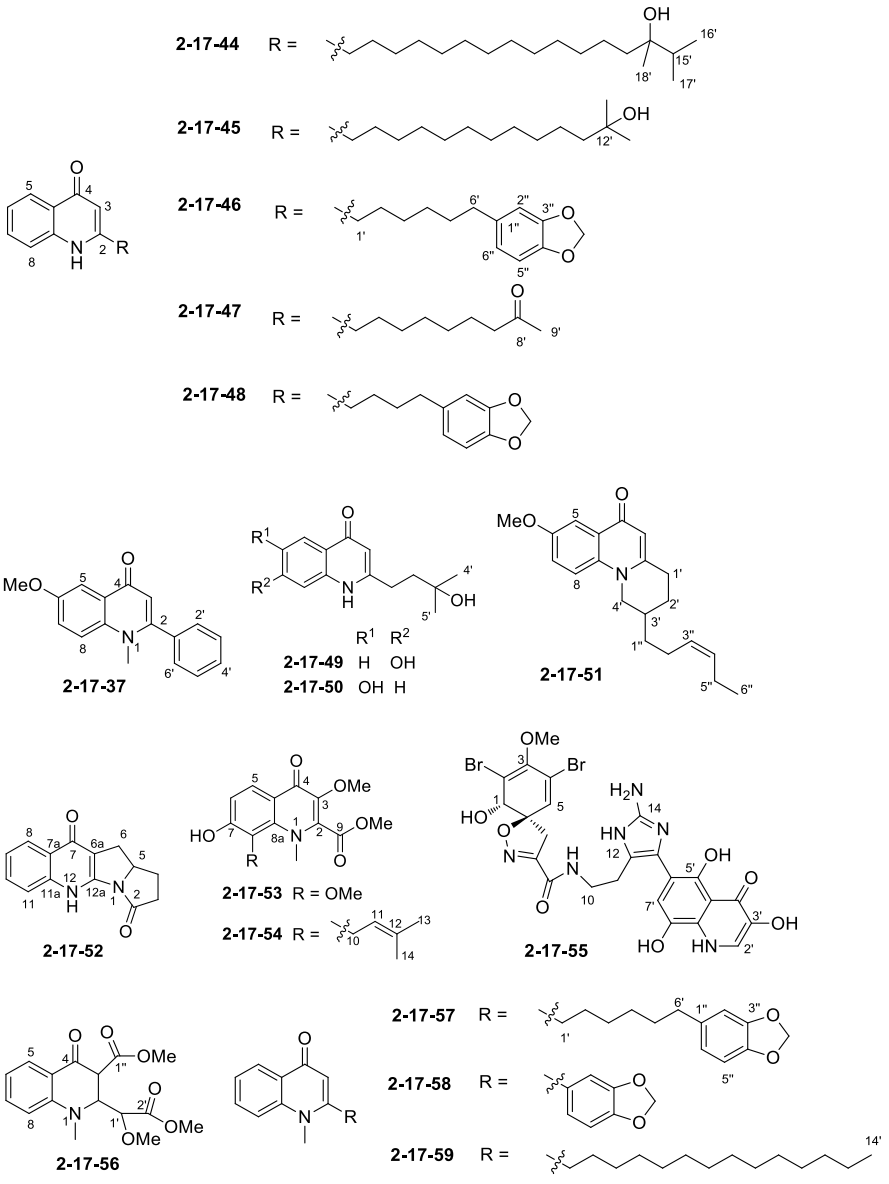


Table 2-17-9: ^{1}H NMR spectroscopic data of 4-oxo-type quinoline alkaloids **2-17-28**~**2-17-32**.

H	2-17-28	2-17-29	2-17-30	2-17-31	2-17-32
2	2.37 s(Me)	2.39 s(Me)	2.29 s(Me)		
3	3.94 s(OMe)	3.83 s(OMe)	3.75 s(OMe)	6.74 s	6.55 s
5	3.27 br s	3.19 s	2.90 s		

Table 2-17-9 (continued)

H	2-17-28	2-17-29	2-17-30	2-17-31	2-17-32
6	ax 2.08 dddd(14.7, 14.0, 4.7, 4.4) eq 2.20 dddd(14.0, 5.3, 2.4, 2.4)	2.21 dddd(14.5, 4.5, 2.4, 2.4) 2.09 dddd(14.5, 14.5, 4.5, 4.5)	1.91 d(13.0) 1.52 dddd(13.0, 13.0, 4.0, 4.0)		
7	ax 2.75 ddd(18.2, 14.7, 5.3) eq 2.58 ddd(18.2, 4.4, 2.4)	2.54 ddd(18.2, 4.5, 2.4) 2.88 ddd(18.2, 14.5, 4.5)	1.82 m 1.75 m	7.49 d(9.2)	7.37 d(9.2)
8				7.56 d(9.2)	7.47 d(9.2)
11	1.41 (ov) 1.77 m	1.52 m, 1.69 m	1.22, 1.71①		
12	1.46 (ov)	1.40~1.60 m	1.40~1.60①		
13	1.2~1.4 (ov)	1.40~1.60 m	1.40~1.60①		
14	1.2~1.4 (ov)	1.40~1.60 m	1.40~1.60①		
15	1.2~1.4 (ov)	1.38, 1.28①	1.20~1.40①		
16	1.25 (ov)	1.45, 1.61①	1.40, 1.62①		
17	1.26 (ov)	3.88 ddd(6.0, 6.0, 6.0)	3.88(3.82)①		
18	0.87 t(7.0)	1.16(1.22) d(6.2)②	1.16(1.22) d(6.0)③		
1′		4.32 d(7.8)	4.32 d(7.8)		
2′		3.14 dd(9.0, 7.8)	3.14 dd(9.0, 7.8)	7.44~7.57 m	7.18 d(1.8)
3′		3.35 dd(9.0, 9.0)	3.35 dd(9.0, 9.0)	–	–
4′		3.28 dd(9.0, 9.0)	3.28 dd(9.0, 9.0)	7.13 s	–
5′		3.25 ddd(9.0, 5.5, 2.5)	3.24 m	7.44~7.57 m	7.20 d(7.6)
6′		3.66 dd(12.0, 2.5)	3.85 dd(12.5, 2.0)	7.44~7.57 m	7.33 dd(7.6, 1.8)
OMe				3.93 s 3.88 s (2×OMe)	3.93 s 3.91 s 3.85 s (2×OMe)

① The peaktype was not given in the literature.
② (17*R*): 2.2 H; (17*S*): 0.8 H.
③ (17*R*): 1.7 H; (17*S*): 1.3 H.

Table 2-17-10: ^{1}H NMR spectroscopic data of 4-oxo-type quinoline alkaloids **2-17-33**~**2-17-37**.

H	2-17-33	2-17-34	2-17-35	2-17-36	2-17-37
1				3.74 s(NMe)	3.60 s(NMe)
2				3.69 s(OMe) 3.75 s(COOMe)	
3	6.20 s	4.80 d(2.5)	4.70 d(2.6)	3.70 s(OMe) 3.77 s(COOMe)	6.32 s
4		3.80 s	3.82 s		
5		7.47 d(8.2)	7.51 d(8.2)	8.49 dd(8.8, 1.5)	6.89 d(2.1)
6		7.66 ddd(8.2, 7.5, 1.4)	7.62 ddd(8.2, 7.5, 1.6)	7.45 td(8.8, 1.5)	3.90 s(OMe)

Table 2-17-10 (continued)

H	2-17-33	2-17-34	2-17-35	2-17-36	2-17-37
7	7.39 d(9.2)	7.29 dd(7.6, 7.5)	7.36 dd(7.5, 7.6)	7.73 td(8.8, 1.5)	7.03 dd(2.1, 8.9)
8	7.54 d(9.2)	8.23 dd(7.6, 1.4)	8.30 dd(7.6, 1.6)	7.48 dd(8.8, 1.5)	8.42 d(8.9)
1′		2.13 m	2.17 m		
		0.51 d(6.7, Me)	1.21 d(6.9, Me)		
2′		1.62 m	0.98 m		7.40 m
		1.48 m	0.84 m		
3′	6.81 d(9.2)	1.09 t(7.4)	0.69 t(7.4)		7.51 m
4′	7.14 d(9.2)				7.51 m
5′					7.51 m
6′					7.40 m
NH		7.37 br s	7.44 br s		
OMe	3.92 s				
	3.88 s				
	3.85 s				
	3.81 s				
	3.75 s (2×OMe)				

Table 2-17-11: ^{1}H NMR spectroscopic data of 4-oxo-type quinoline alkaloids **2-17-38**~**2-17-42**.

H	2-17-38	2-17-39	2-17-40	2-17-41	2-17-42
3	3.92 s(OMe)	3.91 s(OMe)	3.90 s(OMe)	3.91 s(OMe)	3.90 s(OMe)
5	8.40 br d(7.5)	8.38 dd(7.2, 1.0)	8.38 dd(8.0, 1.2)	8.37 dd(8.0, 1.1)	8.40 dd(8.0, 1.0)
6	7.28 br t(7.5)	7.27 br t(7.2)	7.27 br t(8.0)	7.28 br t(8.0)	7.28 t(8.0)
7	7.54 br t(7.5)	7.54 br t(7.2)	7.53 br t(8.0)	7.54 br t(8.0)	7.55 t(8.0)
8	7.73 d(7.5)	7.82 d(7.2)	7.82 d(8.0)	7.61 d(8.0)	7.78 d(8.0)
1′	2.84 t(7.6)	2.83 t(7.6)	2.84 t(7.7)	2.81 t(7.8)	2.86 t(7.2)
2′	1.70 m	1.67 m	1.68 m	1.70 m	1.68~1.72 m
3′	1.30 m	1.25 m	1.28 m	1.19~1.25 m	1.68~1.72 m
4′	1.21~1.28 m	1.25 m	1.25 m	1.19~1.25 m	1.68~1.72 m
5′	1.21~1.28 m	1.25 m	1.25 m	1.19~1.25 m	1.68~1.72 m
6′	1.21~1.28 m	1.25 m	1.25 m	1.19~1.25 m	1.68~1.72 m
7′	1.21~1.28 m	1.25 m	1.25 m	1.19~1.25 m	1.68~1.72 m
8′	1.21~1.28 m	1.35 m	1.25 m	1.19~1.25 m	1.68~1.72 m
9′	1.26 m	1.37 m	1.25 m	1.19~1.25 m	1.68~1.72 m
10′	1.54 m		1.43 m	1.30 m	1.68~1.72 m
11′	2.43 t(7.3)	1.45 q(7.4)		1.45 m	1.68~1.72 m
12′		0.87 t(7.4)	1.20 s	3.80 sext(6.2)	1.68~1.72 m
13′	2.14 s	1.12 s	1.20 s	1.19 d(6.2)	1.68~1.72 m
15′					2.44 sept(7.0)
16′					1.10 d(7.0)
17′					1.10 d(7.0)
18′					1.40 s
NH	11.12 br s	11.75 br s	12.00 br s	10.22 br s	11.60 br s

Table 2-17-12: ^{1}H NMR spectroscopic data of 4-oxo-type quinoline alkaloids **2-17-43**~**2-17-47**.

H	2-17-43	2-17-44	2-17-45	2-17-46	2-17-47
3	3.92 s(OMe)	6.22 s	6.17 s	5.93 s	6.21 s
5	8.40 br d(8.0)	8.35 d(8.0)	8.30 br d(8.0)	8.05 d(8.0)	8.35 br d(8)
6	7.31 dt(8.0, 1.5)	7.32 dt(8.0, 1.8)	7.26 t(8.0)	7.28 dd(7.7, 7.3)	7.32 br t(8)
7	7.51 dt(8.0, 1.2)	7.56 dt(8.0, 1.5)	7.52 t(8.0)	7.61 dd(8.1, 7.1)	7.58 dt(8)
8	7.59 dd(8.0, 1.5)	7.60 dd(8.0, 1.8)	7.71 d(8.0)	7.53 d(8.3)	7.72 br d(8)
1′	2.80 t(8.0)	2.64 t(7.8)	2.60 br t	2.57 dd(7.7, 7.6)	2.63 t(7)
2′	1.28~1.71 m	1.70~1.80 m	1.10~1.80 m	1.66 m	1.70 br s
3′	1.28~1.71 m	1.20~1.30 m	1.10~1.80 m	1.32 m	1.50 br s
4′	1.28~1.71 m	1.20~1.30 m	1.10~1.80 m	1.32 m	1.20 br s
5′	1.28~1.71 m	1.20~1.30 m	1.10~1.80 m	1.52 m	1.20 br s
6′	1.28~1.71 m	1.20~1.30 m	1.10~1.80 m	2.47 dd(7.6, 7.6)	1.20 br s
7′	1.28~1.71 m	1.20~1.30 m	1.10~1.80 m		2.38 t(7)
8′	1.28~1.71 m	1.20~1.30 m	1.10~1.80 m		
9′	1.28~1.71 m	1.20~1.30 m	1.10~1.80 m		2.1 s
10′	1.28~1.71 m	1.20~1.30 m	1.10~1.80 m		
11′	1.28~1.71 m	1.20~1.30 m	1.10~1.80 m		
12′		1.20~1.30 m			
13′	1.20 s	1.20~1.30 m	1.20 s		
14′	1.20 s		1.20 s		
15′		2.45 sept(7.0)			
16′		1.20 d(7.0)			
17′		1.12 d(7.0)			
18′		1.40 s			
2″				6.75 br s	
5″				6.77 d(7.9)	
6″				6.61 d(7.8)	
NMe					3.70 s
NH	10.10 br s	10.50 br s	12.20 br s	11.50 s	
OCH_2O				5.94 s	

Table 2-17-13: ^{1}H NMR spectroscopic data of 4-oxo-type quinoline alkaloids **2-17-48**~**2-17-52**.

H	2-17-48	2-17-49	2-17-50	2-17-51	2-17-52
3	5.92 s	6.68 s	5.82 s	6.20 s	3.27 dd(17.6, 8.5) 3.46 dd(17.6, 8.5)
4					2.26 m
5	7.92 dd(8.2, 1.0)	7.90 d(9.0)	7.37 d(2.7)	7.87 d(3.3)	4.63 m
6	7.07 ddd(8.2, 6.9, 1.0)	7.43 dd(9.0, 2.5)	9.65 s(OH)		2.31 dd(15.3, 8.6) 1.13 dd(15.3, 3.5)
7	7.34 ddd(8.1, 6.9, 1.4)		7.12 dd(8.8, 2.7)	7.26 dd(10.0, 3.3)	

Table 2-17-13 (continued)

H	2-17-48	2-17-49	2-17-50	2-17-51	2-17-52
8	7.21 br d(8.1)	7.47 d(2.5)	7.43 d(8.8)	7.52 d(10.0)	8.15 dd(7.6, 1.4)
9					7.37 m
10					7.76 m
11					7.64 dd(7.5, 1.4)
1′	2.40 t(7.6)	2.91 t(8.3)	2.61 t(8.3)	2.97 dt(10.0, 5.0) 2.94 dt(10.0, 5.0)	
2′	1.35~1.50 m	1.80 t(8.3)	1.72 t(8.3)	2.07 m 1.48 m	
3′	1.35~1.50 m	4.01 br s(OH)	4.42 br s(OH)	2.07 m	
4′	2.29 t(7.0)	1.17 s	1.15 s	3.55 dd(12.0, 10.2) 4.26 dd(12.0, 6.3)	
5′		1.17 s	1.15 s		
1″				1.57 dt(10.0, 7.0)	
2″	6.35 d(1.5)			2.21 q(7.2)	
3″				5.33 dt(10.6, 7.2)	
4″				5.45 dt(10.6, 7.2)	
5″	6.39 d(7.8)			2.07 m	
6″	6.30 dd(7.8, 1.5)			0.98 t(7.5)	
NH		10.50 br s	11.45 br s		12.61
OCH_2O	5.59 s				

Table 2-17-14: ^{1}H NMR spectroscopic data of 4-oxo-type quinoline alkaloids **2-17-53**~**2-17-57**.

H	2-17-53	2-17-54	2-17-55	2-17-56	2-17-57
1			3.89 d(0.6) 6.42 br s(OH)		
2					1.65 m
3	3.93 s(OMe)	3.92 s(OMe)			6.20 s
5	8.16 d(8.8)	8.17 d(8.8)	6.56 d(0.6)	8.45 dd(8.8, 1.5)	8.43 dd(7.9, 1.6)
6	7.92 d(8.8)	6.98 d(8.8)		7.42 td(8.8, 1.5)	7.35 t(7.7)
7	10.6 br s(OH)	10.5 br s(OH)	3.12 d(18.0) 3.60 (ov)	7.71 td(8.8, 1.5)	7.65 ddd (8.6, 7.8, 1.6)
8	3.77 s(OMe)			7.53 dd(8.8, 1.5)	7.49 d(8.6)
9	4.02 s(OMe)	3.99 s(OMe)	8.62 br t(5.7, NH)		
10		3.53 d(8.3)	3.40 (ov)		
11		5.27 t(8.3)	2.75 m		
12			11.95 br s(NH)		
13		1.76 s			
14		1.76 s	7.27 br s(NH_2)		
15			3.69 s		
1′			9.00 br s(NH)	5.28 s 3.55 s(OMe)	2.66 m

Table 2-17-14 (continued)

H	2-17-53	2-17-54	2-17-55	2-17-56	2-17-57
2′			7.70 s	3.94 s(OMe)	
3′			11.81 br s(OH)		1.45 m
4′					1.37 m
5′			14.42 br s(OH)		1.58 m
6′					2.51 t(7.25)
7′			6.96 s		
8′			10.36 br s(OH)		
1″				3.78 s(OMe)	
2″					6.64 d(1.4)
5″					6.70 d(7.8)
6″					6.59 dd(7.8, 1.4)
NMe	3.84 s	3.65 s		3.78 s	3.70 s
OCH_2O					5.89 s

Table 2-17-15: ^{1}H NMR spectroscopic data of 4-oxo-type quinoline alkaloids **2-17-58** and **2-17-59**.

H	2-17-58	2-17-59	H	2-17-58	2-17-59
2		1.64 m	2′	6.92 d(1.7)	
3	6.49 s	6.47 br s	5′	6.89 dd(8.3, 1.7)	
5	8.48 dd(8.2, 1.7)	8.40 dd(1.5, 7.8)	6′	6.89 d(8.3)	
6	7.46 ddd(8.5, 8.5, 0.9)	7.36 t(7.4)	14′		0.81 t(7.5)
7	7.76 ddd(8.5, 8.5, 1.6)	7.65 dt(1.6, 8.0)	OCH_2O	6.08 s	
8	7.60 d(8.5)	7.51 d(8.0)	NMe	3.70 s	3.75 s
1′		2.71 t(7.5)	Other		1.10~1.42 m

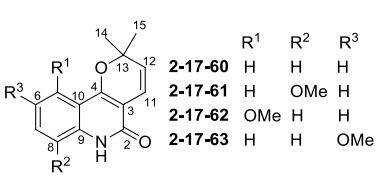
2.17.3 Quinolinopyran-type quinoline alkaloids

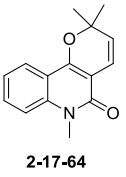
Table 2-17-16: Cos, MFs, and TSs of quinolinopyran-type quinoline alkaloids **2-17-60**~**2-17-74**.

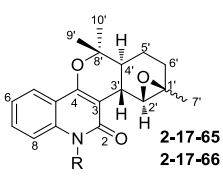
No.	Compounds	MFs	Test solvents	References
2-17-60	flindersine	$C_{14}H_{13}NO_2$	$CDCl_3$	[502]
2-17-61	8-methoxyflindersine	$C_{15}H_{15}NO_3$	$CDCl_3$	[502]
2-17-62	haplophytin A	$C_{15}H_{15}NO_3$	$CDCl_3$	[503]
2-17-63	haplamine	$C_{15}H_{15}NO_3$	$CDCl_3$	[504]
2-17-64	*N*-methylflindersine	$C_{15}H_{15}NO_2$	$CDCl_3$	[469]
2-17-65	*trans*-erioaustrasine	$C_{22}H_{25}NO_5$	$CDCl_3$	[505]
2-17-66	*trans*-deacetoxyarioaustrasine	$C_{20}H_{23}NO_3$	$CDCl_3$	[505]
2-17-67	*trans*-deacetoxyaustralasine hydrate	$C_{20}H_{25}NO_4$	$CDCl_3$	[505]

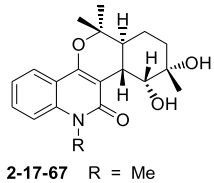
Table 2-17-16 (continued)

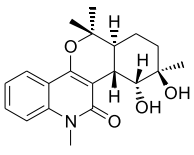
No.	Compounds	MFs	Test solvents	References
2-17-68	*trans*-australasine hydrate	$C_{22}H_{27}NO_6$	$CDCl_3$	[505]
2-17-69	*trans*-1′-*epi*deacetoxyaustralasine hydrate	$C_{20}H_{25}NO_4$	$CDCl_3$	[505]
2-17-70	(–)-(*R*)-geilbalansine	$C_{15}H_{16}NO_3$	$CDCl_3$	[506]
2-17-71	orixalone D	$C_{15}H_{17}NO_4$	–	[466]
2-17-72	helietidine	$C_{19}H_{21}NO_2$	$CDCl_3$	[507]
2-17-73	(–)-tabouensinium chloride	$C_{16}H_{20}NO_3$	DMSO-d_6	[508]
2-17-74	(–)-*cis*-1,2-dihydroxy-1,2-dihydromedicosmine	$C_{17}H_{17}NO_5$	$CDCl_3$	[509]

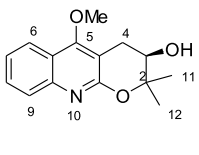


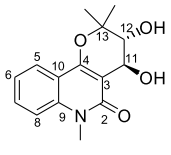


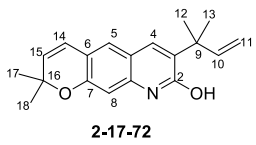


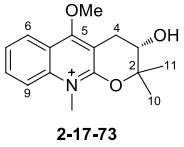












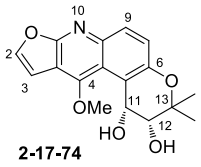


Table 2-17-17: ^{1}H NMR spectroscopic data of quinolinopyran-type quinoline alkaloids **2-17-60**~**2-17-64**.

H	2-17-60	2-17-61	2-17-62	2-17-63	2-17-64
5	7.89 d(7.5)	7.46 dd(8.0, 1.0)	3.90 s(OMe)	7.23 d(2.0)	7.97 dd(1.3, 1.6)
6	7.19 t(7.5)	7.10 t(8.0)	7.26 dd(8.8, 2.8)	3.86 s(OMe)	7.23 ddd(1.0, 1.0)
7	7.48 t(7.5)	6.94 dd(8.0, 1.0)	7.10 d(8.8)	7.11 d(8.4)	7.56 ddd(1.3, 1.6, 1.6)
8	7.33 d(7.5)	3.95 s(OMe)	7.25 dd(8.8, 2.8)	7.39 d(8.8)	7.32 d(1.0)
11	6.77 d(9.9)	6.71 d(9.9)	5.54 d(9.9)	6.80 d(10)	5.54 d(9.9)
12	5.56 d(9.9)	5.52 d(9.9)	6.76 d(9.9)	5.54 d(10)	6.76 d(9.9)
14,15	1.54 s	1.52 s	1.51 s	1.54 s	1.52 s
NMe					3.70 s
NH	11.5 s	8.87 s		12.78 br s	

Table 2-17-18: ^{1}H NMR spectroscopic data of quinolinopyran-type quinoline alkaloids **2-17-65**~**2-17-69**.

H	2-17-65	2-17-66	2-17-67	2-17-68	2-17-69
5	7.96 dd(8.0, 1.5)	7.96 dd(8.0, 1.5)	7.98 dd(8.0, 1.4)	7.98 dd(8.0, 1.4)	7.96 dd(8.0, 1.5)
6	7.24 td(7.2, small)	7.22 ddd(8.0, 7.2, 0.8)	7.21 ddd(8.0, 7.1, 0.9)	7.24 t(7.5)	7.21 ddd(8.0, 7.2, 0.9)
7	7.55 ddd(8.4, 7.2, 1.4)	7.55 ddd(8.6, 7.2, 1.6)	7.54 ddd(8.6, 7.1, 1.6)	7.54 ddd(8.4, 7.4, 1.5)	7.54 ddd(8.8, 7.2, 1.6)
8	7.33 d(8.4)	7.33 d(8.4)	7.24 d(8.4)	7.34 d(8.4)	7.24 d(8.8)
2′	3.79 s	3.79 s	5.78 t(1.9)	5.65 s	5.12 s
3′	2.79 d(12.2)	2.79 d(12.0)	3.57 dd(11.4, 2.6)	3.53 dd(11.5, 2.6)	3.20 dd(11.8, 1.8)
4′	1.50 td(12.0, sm)	1.52 td(12.0, sm)	2.18 ddd(11.7, 11.6, 3.7)	2.18 td(12.0, 3.7)	2.08 td(12.0, 3.0)
5′	1.60 m, 1.22 m	1.60 ddt(13.6, 6.5, sm) 1.24 tdd(12.1, 12.0, 5.2)	1.61 dd(12.4, 3.6) 1.26 m	1.63 m, 1.25 m	1.61 m, 1.26 m
6′	2.26 dd(14.9, 5.2) 1.79 dddd(12.1, 11.9, 6.4, 2.8)	2.27 dd(14.9, 5.1) 1.79 dddd(14.9, 11.9, 6.4, 2.8)	1.94 td(13.0, 5.0) 1.73 m	1.94 td(13.2, 4.5) 1.73 m	1.81 ddd(13.5, 13.2, 4.5) 1.68 m
7′	1.32 s 1.47 s	1.32 s 1.46 s	1.36 s		
9′	1.13 s	1.12 s	1.22 s	1.23 s	1.19 s
10′	1.55 s	1.54 s	1.57 s	1.57 s	1.54 s
NMe	3.72 s	3.60 s	3.60 s		
NCH_2	6.40 d(9.2) 6.31 d(9.2)			6.35 d(9.2) 6.31 d(9.2)	
Ac	2.11 s			2.11 s	

Table 2-17-19: ^{1}H NMR spectroscopic data of quinolinopyran-type quinoline alkaloids **2-17-70**~**2-17-74**.

H	2-17-70	2-17-71	2-17-72	2-17-73	2-17-74
2					7.65 d(3)
3	4.64 dd(8.4, 8.0)			3.60 dd(2.3, 10.1)	7.03 d(3)
4	3.56 m, 3.64 m		7.47 s	α 2.69 dd(10.1, 13.1) β 2.84 dd(2.3, 13.1)	4.42 s(OMe)
5	4.23 s(OMe)	8.00 br d(8.1)	7.03 s	3.92 s(OMe)	
6	7.98 d(8.0)	7.27 dd(8.1, 8.1)		7.81 d(8.0)	
7	7.29 dd(8.0, 8.0)	7.60 dd(8.1, 8.1)		7.32 dd(7.1, 8.5)	
8	7.54 dd(6.8, 6.8)	7.35 br d(8.1)	6.67 s	7.57 dd(7.1, 8.0)	7.24 d(8)
9	7.72 d(8.0)			7.62 d(8.5)	7.88 d(8)

Table 2-17-19 (continued)

H	2-17-70	2-17-71	2-17-72	2-17-73	2-17-74
10			6.17 dd(10.7, 16.7)	1.40 s/1.48 s	
11	1.28 s	4.75 d(7.7)	5.05 ddd (0.8, 10.7, 16. 7)	1.48 s/1.40 s	5.40 dd(5, 3) 3.77 d(3, OH)
12	1.43 s	3.84 d(7.7)	1.46 s		3.90 dd(9, 5) 3.35 d(9, OH)
13		1.63 s 1.32 s	1.46 s		1.39 s 1.49 s
14			6.34 d(9.2)		
15			5.67 d(9.2)		
17			1.45 s		
18			1.45 s		
NMe		3.74 s		3.62 s	
Others		5.56 br s 2.82 br s			

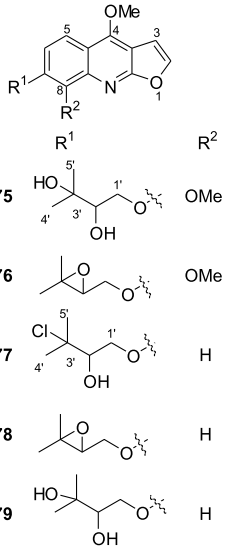
2.17.4 Quinolinofuran-type quinoline alkaloids

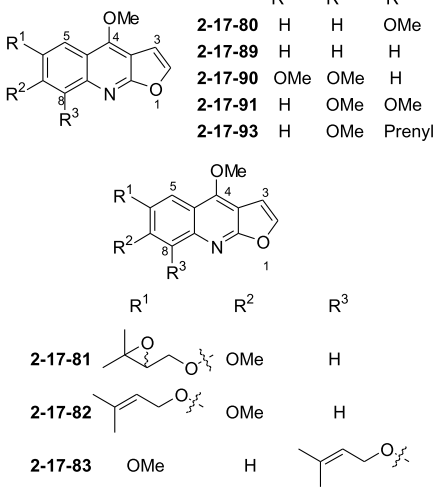
Table 2-17-20: Cos, MFs, and TSs of quinolinofuran-type quinoline alkaloids **2-17-75**~**2-17-115**.

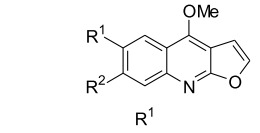
No.	Compounds	MFs	Test solvents	References
2-17-75	haplophytin B	$C_{18}H_{21}NO_6$	CD_3OD	[503]
2-17-76	anhydroevoxine	$C_{18}H_{19}NO_5$	$CDCl_3$	[504]
2-17-77	7-(2′-hydroxy-3′-chloroprenyloxy)-4-methoxyfuro-quinoline	$C_{17}H_{18}ClNO_4$	CD_3OD	[510]
2-17-78	7-(2′,3′-epoxyprenyloxy)-4-methoxyfuroquinoline	$C_{17}H_{17}NO_4$	$CDCl_3$	[510]
2-17-79	evellerine	$C_{17}H_{19}NO_5$	$CDCl_3$	[510]
2-17-80	γ-fagarine	$C_{13}H_{11}NO_3$	$CDCl_3$	[469]
2-17-81	tecleanatalensine A	$C_{18}H_{19}NO_5$	$CDCl_3$	[511]
2-17-82	tecleanatalensine B	$C_{18}H_{19}NO_4$	$CDCl_3$	[511]
2-17-83	tecleabine	$C_{18}H_{19}NO_4$	$CDCl_3$	[512]
2-17-84	tecleoxine	$C_{18}H_{19}NO_5$	$CDCl_3$	[512]
2-17-85	isotecleoxine	$C_{18}H_{19}NO_5$	$CDCl_3$	[512]
2-17-86	methylnkolbisine	$C_{19}H_{23}NO_6$	$CDCl_3$	[512]
2-17-87	chlorodesnkolbisine	$C_{18}H_{20}ClNO_5$	$CDCl_3$	[512]
2-17-88	confusadine	$C_{17}H_{17}NO_4$	$CDCl_3$	[472]
2-17-89	dictamine	$C_{12}H_9NO_2$	$CDCl_3$	[500]
2-17-90	kokusaginine	$C_{14}H_{13}NO_4$	$CDCl_3$	[500]
2-17-91	skimmianine	$C_{14}H_{13}NO_4$	$CDCl_3$	[500]
2-17-92	haplotubine	$C_{23}H_{29}NO_6$	$CDCl_3$	[479]
2-17-93	7-methoxy-8-(3,3-dimethylallyl)-dictamnine	$C_{18}H_{19}NO_3$	$CDCl_3$	[518]

Table 2-17-20 (continued)

No.	Compounds	MFs	Test solvents	References
2-17-94	melicarpinone	$C_{13}H_{11}NO_3$	$CDCl_3$	[472]
2-17-95	5-methoxymaculine	$C_{14}H_{11}NO_5$	$CDCl_3$	[513]
2-17-96	5,8-dimethoxymaculine	$C_{15}H_{13}NO_6$	$CDCl_3$	[513]
2-17-97	flindersiamine	$C_{14}H_{11}NO_5$	$CDCl_3$	[469]
2-17-98	4,5,6,7,8-pentamethoxyfuroquinoline	$C_{16}H_{17}NO_6$	$CDCl_3$	[513]
2-17-99	melicarpine	$C_{15}H_{15}NO_6$	$CDCl_3$	[514]
2-17-100	semecarpine	$C_{17}H_{17}NO_3$	CD_3COCD_3	[514]
2-17-101	(±)-8-methoxyplatydesmine	$C_{16}H_{19}NO_4$	$CDCl_3$	[514]
2-17-102	2-acetylevolitrine	$C_{15}H_{13}NO_4$	$CDCl_3$	[473]
2-17-103	2-acetylpteleine	$C_{15}H_{13}NO_4$	$CDCl_3$	[473]
2-17-104	isopteleflorine	$C_{16}H_{17}NO_5$	$CDCl_3$	[468]
2-17-105	hyemaline	$C_{16}H_{15}NO_3$	$CDCl_3$	[506]
2-17-106	(2*S*)-1-[(6,7-dimethoxyfuro[2,3-*b*]quinolin-4-yl)oxy]-3-methylbutane-2,3-diol	$C_{18}H_{21}NO_6$	C_5D_5N	[515]
2-17-107	rel-(7*R*,8*R*)-8-[(*E*)-3-hydroxy-3-methyl-1-butenyl]-4,8-dimethoxy-5,6,7,8-tetrahydrofuro[2,3-*b*]quinoline-7-yl acetate	$C_{20}H_{25}NO_6$	$CDCl_3$	[516]
2-17-108	sarcodifurine A	$C_{23}H_{27}NO_5$	$CDCl_3$	[517]
2-17-109	sarcodifurine B	$C_{23}H_{27}NO_5$	$CDCl_3$	[517]
2-17-110	megistoquinone I	$C_{13}H_9NO_5$	$CDCl_3$	[484]
2-17-111	7-*O*-acetyhaplophyllidine	$C_{20}H_{25}NO_5$	$CDCl_3$	[518]
2-17-112	megistosarcimine	$C_{18}H_{20}N_2O_4$	$CDCl_3$	[519]
2-17-113	megistosarconine	$C_{18}H_{19}NO_5$	$CDCl_3$	[519]
2-17-114	dictangustine A	$C_{12}H_9NO_3$	CD_3COCD_3	[520]
2-17-115	*iso*-γ-fagarine	$C_{13}H_{11}NO_3$	CD_3COCD_3	[520]







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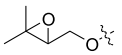
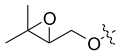
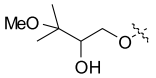
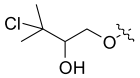
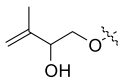
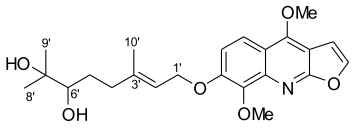
2-17-84		OMe
2-17-85	OMe	
2-17-86		OMe
2-17-87		OMe
2-17-88	H	



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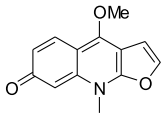

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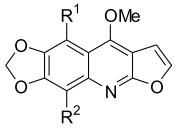
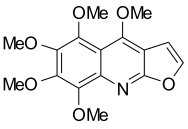
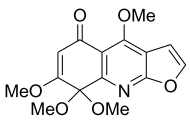


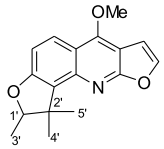
	R^1	R^2
2-17-95	OMe	H
2-17-96	OMe	OMe
2-17-97	H	OMe



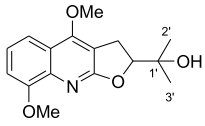
2-17-98



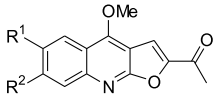
2-17-99



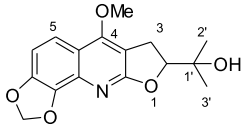
2-17-100



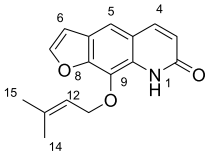
2-17-101



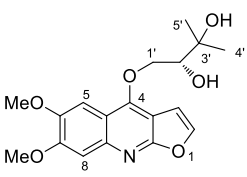
	R^1	R^2
2-17-102	H	OMe
2-17-103	OMe	H



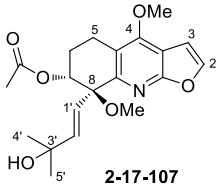
2-17-104



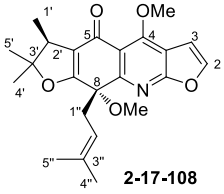
2-17-105



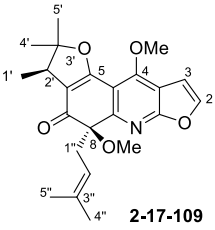
2-17-106



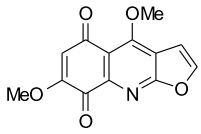
2-17-107



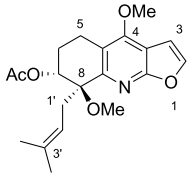
2-17-108



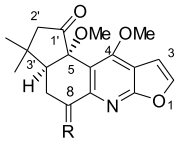
2-17-109



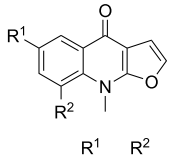
2-17-110



2-17-111



2-17-112 R = NH
2-17-113 R = O



	R^1	R^2
2-17-114	OH	H
2-17-115	H	OMe

Table 2-17-21: ^{1}H NMR spectroscopic data of quinolinofuran-type quinoline alkaloids **2-17-75**~**2-17-79**.

H	2-17-75	2-17-76	2-17-77	2-17-78	2-17-79
2	7.73 d(2.8)	7.59 d(2.4)	7.90 d(2.8)	7.55 d(2.8)	7.56 d(2.8)
3	7.30 d(2.8)	7.04 d(2.4)	7.51 d(2.8)	7.03 d(2.8)	7.04 d(2.7)
4	4.47 s(OMe)	4.43 s(OMe)	4.59 s(OMe)	4.41 s(OMe)	4.42 s(OMe)
5	8.01 d(9.4)	7.98 d(9.2)	8.28 d(9.2)	8.14 d(9.2)	8.15 d(9.2)
6	7.34 d(9.4)	7.25 d(9.2)	7.29 dd(9.2, 2.3)	7.13 dd(9.2, 2.8)	7.09 dd(9.2, 2.8)
8	4.10 s(OMe)	4.13 s(OMe)	7.26 d(2.3)	7.30 d(1.8)	7.34 d(2.8)
1′	4.13 ds(9.9, 8.0)	4.39 dd(6.0, 11.2)	4.57 dd(9.2, 2.8)	4.28 dd(4.6, 11)	4.29 dd(2.7, 5.2)
	3.81 ds(8.0, 2.5)	4.30 dd(6.0, 11.2)	4.21 dd(7.8, 9.2)	4.16 m	4.15 t(7.4)
2′	4.49 dd(9.9, 2.5)	3.25 t(4.8)	4.02 dd(7.8, 2.8)	3.22 dd(4.6)	3.89 dd(2.8, 7.3)
4′	1.29 s	1.39 s	1.68 s	1.40 s	1.33 s
5′	1.25 s	1.36 s	1.65 s	1.38 s	1.29 s

Table 2-17-22: ^{1}H NMR spectroscopic data of quinolinofuran-type quinoline alkaloids **2-17-80**~**2-17-84**.

H	2-17-80	2-17-81	2-17-82	2-17-83	2-17-84
2	7.75 d(2.8)	7.54 d(2.8)	7.56 d(2.6)	7.61 d(2.8)	7.45 d(2.7)
3	7.05 d(2.6)	7.02 d(2.8)	7.03 d(2.6)	7.03 d(2.8)	6.89 d(2.7)
4	4.50 s(OMe)	4.41 s(OMe)	4.43 s(OMe)	4.43 s(OMe)	4.28 s(OMe)
5	7.90 dd(1.3, 1.3)	7.53 s	7.48 s	7.08 d(2.8)	7.39 s
6	7.40 t(7.6, 8.5)			3.92 s(OMe)	
7	7.05 d(2.8)	3.99 s(OMe)	3.98 s(OMe)	6.74 d(2.8)	3.94 s(OMe)
8	4.10 s(OMe)	7.32 s	7.36 s		7.23 s
1′		4.27 m	4.70 d(6.8)	4.79 d(6.4)	4.23 m
					4.17 dd(4.7, 11.1)
2′		3.25 t(5.1)	5.58 br t(6.8)	5.65 t(6.4)	3.21 dd(4.7, 7.5)
4′		1.39 s	1.79 s	1.79 s	1.35 s
5′		1.39 s	1.79 s	1.78 s	1.35 s

Table 2-17-23: ^{1}H NMR spectroscopic data of quinolinofuran-type quinoline alkaloids **2-17-85**~**2-17-89**.

H	2-17-85	2-17-86	2-17-87	2-17-88	2-17-89
2	7.51 d(2.7)	7.49 d(2.7)	7.93 d(2.7)	7.58 d(2.8)	
3	6.97 d(2.7)	6.97 d(2.7)	6.38 d(2.7)	7.06 d(2.8)	
4	4.36 s(OMe)	4.36 s(OMe)	4.41 s(OMe)	4.44 s(OMe)	4.44 s(OMe)
5	7.41 s	7.46 s	7.45 s	8.17 d(9.2)	8.02 dd(8.0, 1.2)
6	3.97 s(OMe)			7.13 dd(9.2, 2.4)	7.68 ddd(8.0, 6.8, 1.3)
7		3.92 s(OMe)	3.93 s(OMe)		7.44 ddd(8.3, 6.8, 1.0)

Table 2-17-23 (continued)

H	2-17-85	2-17-86	2-17-87	2-17-88	2-17-89
8	7.32 s	7.25 s	7.27 s	7.33 d(2.4)	8.27 dd(8.3, 1.3)
1′	4.36 m	4.26 dd(3.1, 9.5)	4.33 dd(1.6, 9.8)	4.09 dd(9.6, 8.0)	7.07 d(2.7)
	4.21 dd(5.9, 11.1)	4.05 t(9.5)	3.96 m	4.22 dd(9.6, 3.2)	
2′	3.28 dd(4.5, 5.9)	3.99 dd(2.8, 8.9)	3.67 dd(1.6, 7.7)	4.57 br d(8.0)	7.62 d(2.7)
				2.42 br s(OH)	
3′		3.23 s(OMe)		1.86 s(Me)	
4′	1.38 s	1.22 s	1.15 s	5.04 s	
				5.19 s	
5′	1.38 s	1.24 s	1.20 s		

Table 2-17-24: ^{1}H NMR spectroscopic data of quinolinofuran-type quinoline alkaloids **2-17-90**~**2-17-94**.

H	2-17-90	2-17-91	2-17-92	2-17-93	2-17-94
2			7.58 d(2.8)	7.52 d(3.0)	7.48 d(2.4)
3			7.04 d(2.8)	7.00 d(3.0)	7.12 d(2.4)
4	4.45 s(OMe)	4.43 s(OMe)	4.42 s(OMe)	4.38 s(OMe)	4.45 s(OMe)
5	7.48 s	8.01 d(9.4)	7.97 d(9.6)	8.12 d(9.0)	7.97 d(9.6)
6	4.02 s(OMe)	7.23 d(9.4)	7.21 d(9.2)	7.15 d(9.0)	6.96 dd(9.6, 2.0)
7	4.03 s(OMe)	4.03 s(OMe)		3.92 s(OMe)	
8	7.34 s	4.11 s(OMe)	4.11 s(OMe)		6.58 d(2.0)
1′	7.05 d(2.8)	7.04 d(2.9)	4.79 d(6.8)		
2′	7.58 d(2.8)	7.58 d(2.9)	5.61 ddd(1.2, 6.6, 6.6)	5.32 br t(6.0)	
4′			2.29~2.32 m	1.90 s	
			2.12~2.20 m		
5′			1.58~1.62 m	1.65 s	
			1.43~1.48 m		
6′			3.31 dd(1.6, 10.6)		
8′			1.15 s		
9′			1.13 s		
10′			1.75 s		
NMe					3.95 s

Table 2-17-25: ^{1}H NMR spectroscopic data of quinolinofuran-type quinoline alkaloids **2-17-95**~**2-17-99**.

H	2-17-95	2-17-96	2-17-97	2-17-98	2-17-99
2	7.62 d(2.7)	7.57 d(2.7)	7.58 d(2.8)	7.60 d(2.7)	7.69 d(2.6)
3	7.04 d(2.7)	7.01 d(2.7)	7.02 d(2.8)	7.05 d(2.7)	7.12 d(2.6)
4	4.40 s(OMe)	4.39 s(OMe)	4.50 s(OMe)	4.44 s(OMe)	4.40 s(OMe)

Table 2-17-25 (continued)

H	2-17-95	2-17-96	2-17-97	2-17-98	2-17-99
5	4.17 s(OMe)	4.18 s(OMe)	7.30 s	4.15 s(OMe)	
6				3.99 s(OMe)	5.85 s
7				3.91 s(OMe)	3.90 s(OMe)
8	7.15 s	4.25 s(OMe)	4.30 s(OMe)	4.26 s(OMe)	3.28 s(OMe)
					3.28 s(OMe)
OCH_2O	6.05 s	6.05 s	6.06 s		

Table 2-17-26: ^{1}H NMR spectroscopic data of quinolinofuran-type quinoline alkaloids **2-17-100**~**2-17-104**.

H	2-17-100	2-17-101	2-17-102	2-17-103	2-17-104
2	7.79 d(2.4)	4.63 t(8.6)	2.67 s(Ac)	2.68 s(Ac)	4.63 t(8.6)
3	7.49 d(2.4)	3.60 d(8.6)	7.82 s	7.83 s	3.57 d(8.6)
4	4.53 s(OMe)	4.24 s(OMe)	4.49 s(OMe)	4.52 s(OMe)	4.23 s(OMe)
5	7.56 d(9.4)	7.62 dd(8.4, 1.2)	8.17 d(9.4)	7.53 d(3.0)	7.59 d(8.6)
6	6.70 d(9.4)	7.26 dd(8.4, 8.0)	7.11 dd(9.4, 2.6)	3.95 s(OMe)	6.95 d(8.6)
7		6.98 dd(8.0, 1.2)	3.96 s(OMe)	7.42 dd(9.5, 3.0)	
8		4.00 s(OMe)	7.32 d(2.6)	7.93 d(9.5)	
1′	4.78 q(6.8)	1.95 br s(OH)			
2′		1.26 s			1.41 s
3′	1.70 d(6.8)	1.42 s			1.26 s
4′	1.48 s				
5′	1.54 s				
OCH_2O					6.15 d(6.9)
					6.15 d(6.9)

Table 2-17-27: ^{1}H NMR spectroscopic data of quinolinofuran-type quinoline alkaloids **2-17-105**~**2-17-109**.

H	2-17-105	2-17-106	2-17-107	2-17-108	2-17-109
2		7.79 d(2.6)	7.58 d(2.6)	7.58 d(2.3)	7.59 (2.3)①
3	6.35 d(9.0)	7.40 d(2.6)	6.97 d(2.6)	7.04 d(2.3)	6.99 d(2.3)
4	7.77 d(9.0)		4.30 s(OMe)	4.33 s(OMe)	4.26 s(OMe)
5	7.35 s	7.86 s	2.78 m		
6	6.80 d(2.8)	3.70 s(OMe)	2.20 m, 2.01 m		
7	7.71 d(2.8)	3.83 s(OMe)	5.43 dd(8.5, 2.9)		
			2.03 s(OAc)		
8		7.64 s	3.14 s(OMe)	2.94 s(OMe)	2.97 s(OMe)
11	5.01 d(7.2)				

Table 2-17-27 (continued)

H	2-17-105	2-17-106	2-17-107	2-17-108	2-17-109
12	5.60 m				
14	1.72 s				
15	1.74 s				
1′		5.11 dd(9.8, 8.0) 5.45 dd(9.8, 2.7)	6.06 d(16.1)	1.11 d(7.2)	1.14 d(7.2)
2′		4.51 dd(8.0, 2.7)	5.69 d(16.1)	3.04 q(7.2)	2.93 q(7.2)
4′		1.63 s	1.34 s	1.37	1.44 s
5′		1.68 s	1.31 s	1.40	1.34 s
1″				3.02 t(7.5)	2.78 t(7.5)
2″				4.39 t(7.5)	4.55 dd(7.5)
4″				1.40 s	1.39 s
5″				1.40 s	1.26 s

① The peaktype was not given in the literature.

Table 2-17-28: ^{1}H NMR spectroscopic data of quinolinofuran-type quinoline alkaloids **2-17-110**~**2-17-114**.

H	2-17-110	2-17-111	2-17-112	2-17-113	2-17-114
2	7.79 d(2.6)	7.55 d(3)	7.65 d(3)	7.67 d(3)	7.53 d(2.4)
3	7.16 d(2.6)	6.95 d(3)	7.05 d(3)	7.11 d(3)	6.95 d(2.4)
4	4.41 s(OMe)	4.28 s(OMe)	4.45 s(OMe)	4.45 s(OMe)	
5			3.32 s(OMe)	3.38 s(OMe)	7.88 d(3.2)
6	6.10 s		2.94 td(3.5, 1.7)	2.80 td(3.2, 1.7)	8.76 s(OH)
7	3.86 s(OMe)	5.20 m	2.83 dd(14, 3.5)	2.91 dd(13.5, 3.2)	7.31 dd(8.8, 3.2)
		1.98 s(OAc)	2.55 dd(14, 3.5)	2.60 dd(13.5, 3.2)	
8		3.13 s(OMe)			7.65 d(8.8)
2′		5.20 m	2.22 d(15)	2.38 d(15)	
			2.10 d(15, 1.7)①	2.20 d(15, 1.7)①	
3′		1.66 s	1.19 s	1.21 s	
		1.60 s	1.10 s	1.10 s	
NMe					3.96 s
NH			3.89 br s		

① Typographic error exists in the literature, giving one more *J* value.

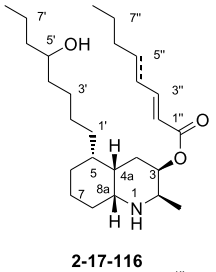
Table 2-17-29: ^{1}H NMR spectroscopic data of quinolinofuran-type quinoline alkaloid **2-17-115**.

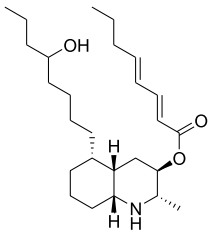
H	2-17-115	H	2-17-115
2	7.56 d(2.2)	7	7.35 dd(7.9, 1.9)
3	6.94 d(2.2)	8	4.01 s(OMe)
5	8.02 dd(7.9, 1.9)	NMe	4.20 s
6	7.28 t(7.9)		

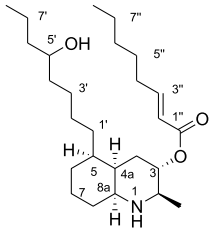
2.17.5 Decahydroquiniline-type quinoline alkaloids

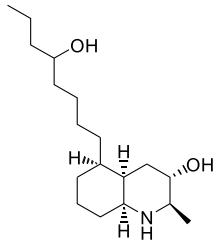
Table 2-17-30: Cos, MFs, and TSs of decahydroquiniline-type quinoline alkaloids **2-17-116**~**2-17-124**.

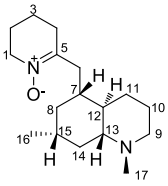
No.	Compounds	MFs	Test solvents	References
2-17-116	lepadin F	$C_{26}H_{47}NO_3$	C_6D_6	[521]
2-17-117	lepadin G	$C_{26}H_{45}NO_3$	C_6D_6	[521]
2-17-118	lepadin H	$C_{26}H_{45}NO_3$	C_6D_6	[521]
2-17-119	lepadin E	$C_{26}H_{47}NO_3$	$CDCl_3$	[522]
2-17-120	lepadin D	$C_{18}H_{35}NO_2$	CD_3OD	[522]
2-17-121	huperzine J	$C_{17}H_{30}N_2O$	$CDCl_3$	[523]
2-17-122	huperzine K	$C_{16}H_{28}N_2O$	$CDCl_3$	[523]
2-17-123	huperzine L	$C_{17}H_{30}N_2O_2$	$CDCl_3$	[523]
2-17-124	senepodine A	$C_{23}H_{40}N_2$	CD_3OD	[524]

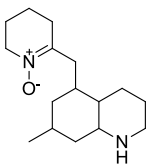


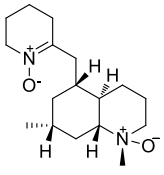












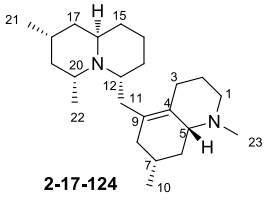


Table 2-17-31: ^{1}H NMR spectroscopic data of decahydroquiniline-type quinoline alkaloids **2-17-116**~**2-17-119**.

H	2-17-116	2-17-117	2-17-118	2-17-119
2	2.84 qd(6.6, 1.8)	2.86 qd(6.4, 1.0)	3.19 qd(6.4, 4.8)	3.08 m
3	5.00 br s	5.02 ddd(3.7, 3.7, 1.0)	4.96 ddd(4.6, 4.6, 4.8)	4.78 m

Table 2-17-31 (continued)

H	2-17-116	2-17-117	2-17-118	2-17-119
4				1.52 m 1.89 ddd(4.5, 11.7, 14.3)
4*α*	1.35 m	1.35 m	1.66 ddd(14.9, 10.8, 4.6)	
4*β*	1.82 ddd(13.8, 4.0, 3.0)	1.83 ddd(14.5, 3.7, 3.7)	1.58 ddd(14.9, 4.6, 4.6)	
4a	2.17 dddd(13.8, 4.0, 4.0, 4.6)	2.19 dddd(13.2, 4.8, 4.4, 4.4)	2.28 dddd(10.8, 4.6, 4.8, 5.4)	2.17 m
5	1.35 m	1.32 m	1.37 m	1.47 m
6				1.09 m, 1.38 m
6*α*	0.80 dddd(13.2, 13.2, 13.2, 3.6)	0.80 dddd(12.9, 12.9, 12.9, 3.7)	0.96 m	
6*β*	12.4 m	1.26 m	1.19 m	
7				1.13 m, 1.66 m
7*α*	1.65 m	1.66 ddddd(12.9, 4.1, 3.7, 3.7, 3.7)	1.52 m	
7*β*	1.13 m	1.15 m	1.02 m	
8				1.55 m, 1.68 m
8*α*	1.58 dddd(13.2, 13.2, 12.6, 4.2)	1.58 dddd(12.9, 12.9, 12.9, 4.1)	1.36 m	
8*β*	1.48 m	1.48 dddd(12.9, 4.4, 3.7, 3.7)	1.72 dddd(12.6, 4.8, 4.6, 4.8)	
8a	2.81 ddd(12.6, 4.6, 4.2)	2.82 ddd(12.9, 4.8, 3.7)	3.04 ddd(10.2, 4.8, 4.8)	2.95 m
9	1.08 d(6.6)	1.08 d(6.4)	1.13 d(6.4)	1.19 d(6.8)
1′	1.10 m	1.10 m	1.32 m	1.26 m
2′	1.21 m	1.28 m	1.25 m	1.26 m
3′	1.38 m	1.31 m	1.41 m	1.46 m
4′	1.32 m	1.32 m	1.40 m	1.40 m
5′	3.42 tt(6.5, 5.4)	3.42 tt(6.1, 6.1)	3.49 tt(6.1, 6.1)	3.56 m
6′	1.30 m	1.32 m	1.35 m	1.40 m
7′	1.30 m, 1.43 m	1.30 m, 1.44 m	1.34 m, 1.47 m	1.39 m
8′	0.90 t(6.6)	0.90 t(7.0)	0.92 t(7.0)	0.90 t(7.0)
2″	6.00 dt(15.6, 1.2)	6.00 d(15.6)	6.02 d(15.6)	5.83 d(15.4)
3″	7.19 dt(15.6, 7.2)	7.59 dd(15.6, 10.8)	7.61 dd(15.6, 10.8)	6.99 ddd(6.8, 6.8, 15.4)
4″	1.89 ddt(1.2, 7.2, 7.2)	5.97 dd(15.0, 10.8)	6.00 dd(15.0, 10.8)	2.20 m
5″	1.18 m	5.75 dt(15.0, 7.2)	5.86 dt(15.0, 7.2)	1.26 m, 1.40 m
6″	1.08 m	1.80 dt(7.2, 7.0)	1.82 dt(7.2, 7.2)	1.29 m
7″	1.13 m	1.18 qt(7.0, 7.0)	1.20 qt(7.2, 7.2)	1.31 m
8″	0.81 t(6.5)	0.74 t(7.0)	0.76 t(7.2)	0.90 t(6.8)

Table 2-17-32: ^{1}H NMR spectroscopic data of decahydroquiniline-type quinoline alkaloids **2-17-120**~**2-17-123**.

H	2-17-120	2-17-121	2-17-122	2-17-123
1		3.75 t(5.5)	3.78 t(5.5)	3.76 t(6.1)
2	2.99 dq(4.1, 7.0)	1.88 m	1.90 m	1.90 m
3	3.70 ddd(4.1, 4.3, 4.4)	1.70 m	1.71 m	1.74 m
4	1.50 m	2.36 t(6.2)	2.36 t(5.8)	2.40 t(5.5)
	1.87 ddd(4.4, 14.0, 14.0)			
4a	2.38 m			
5	1.53 m			
6	1.12 dddd(3.3, 12.5, 12.5, 12.5)			
	1.38 m			
6a		2.84 dd(10.4, 4.1)	2.74 dd(13.0, 3.4)	3.03 br d(13.2)
6b		2.08 d(10.4)	2.29 d(13.0)	1.82 dd(13.2, 3.2)
7	1.79 m	1.72 m	1.90 m	2.08 m
8	1.66 dddd(3.8, 12.2, 12.5, 12.5)			
	1.72 m			
8a	3.03 ddd(4.9, 6.0, 12.2)	1.44 m	1.84 m	1.43 m
8b		0.75 m	1.41 m	0.80 m
9	1.28 d(7.0)			
9a		2.81 m	2.89 br d(11.3)	3.34 d(12.0)
9b		2.04 m	2.09 m	3.14 dd(12.0, 3.0)
10a		1.62 m	1.66 m	2.36 m
11a		1.98 m	2.00 br d(14.5)	2.14 m
11b		1.05 m	1.03 m	1.12 m
12		0.94 m	1.03 m	1.78 m
13		1.57 m	1.88 br d(13.0)	2.68 ddd(11.0, 10.6, 3.4)
14a		2.01 dd(12.2, 4.1)	1.32 m	1.94 m
14b		0.78 m	1.30 m	1.59 m
15		1.40 m	2.11 m	1.46 m
16		0.86 d(6.4)	0.99 d(7.3)	0.93 d(6.3)
17		2.22 s		3.10 s
1′	1.34 m			
2′	1.50 m			
3′	1.47 m			
4′	1.49 m			
5′	3.56 m			
6′	1.44 m			
7′	1.51 m, 1.40 m			
8′	0.97 t(7.0)			

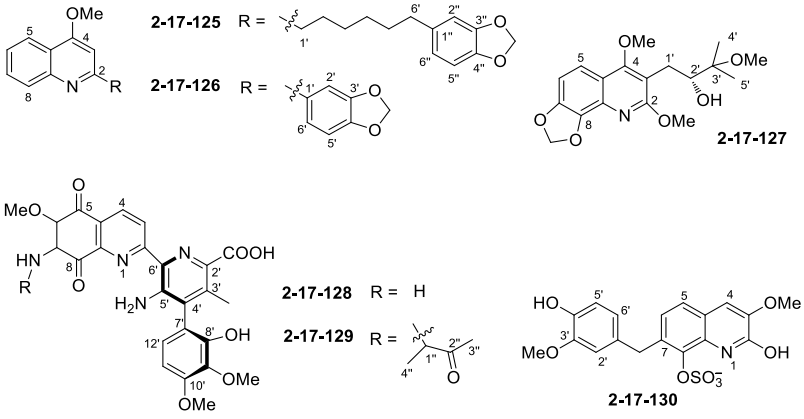
Table 2-17-33: ^{1}H NMR spectroscopic data of decahydroquiniline-type quinoline alkaloid **2-17-124**.

H	2-17-124	H	2-17-124	H	2-17-124
1	2.92 br d(11.8), 2.31 m	10	0.99 d(6.6)	17	1.47 dt(4.9, 13.1) 1.59 br d(12.4)
2	1.54 m, 1.73 m	11	2.13 br d(12.8) 3.03 t(12.2)	18	1.82 m
3	2.86 br d(13.6), 1.75 m	12	3.48 m	19	1.13 ddd(12.5, 12.5, 12.5) 1.72 m
5	2.63 br t(8.0)	13	1.34 br d(12.6), 1.70 m	20	3.44 m
6	1.04 dt(10.3, 12.5), 2.09 m	14	1.77 m, 1.68 m	21	0.90 d(6.5)
7	1.58 br d(14.0)	15	1.28 br d(17.9) 2.04 ddd(4.1, 13.1, 13.1)	22	1.18 d(6.1)
8	1.79 m, 1.92 br d(16.1)	16	3.46 m	23	2.30 s

2.17.6 Other quinoline alkaloids

Table 2-17-34: Cos, MFs, and TSs of other quinoline alkaloids **2-17-125**~**2-17-133**.

No.	Compounds	MFs	Test solvents	References
2-17-125	2-{6′-(2*H*-benzo[*d*]1″,3″-dioxolen-5″-yl)hexyl}-4-methoxy-quinoline	$C_{23}H_{25}NO_3$	$CDCl_3$	[493]
2-17-126	graveolinine	$C_{17}H_{13}NO_3$	$CDCl_3$	[500]
2-17-127	3′-*O*-methylorixine	$C_{18}H_{23}NO_6$	$CDCl_3$	[468]
2-17-128	streptonigrin	$C_{25}H_{22}N_4O_8$	$CDCl_3$	[525]
2-17-129	7-(1-methyl-2-oxopropyl)streptonigrin	$C_{29}H_{28}N_4O_9$	$CDCl_3$	[525]
2-17-130	scolopendrine	$C_{18}H_{16}NO_8S$	CD_3OD	[526]
2-17-131	1,2,3,4-tetrahydro-2(*n*-pentyl)-1-methylquinoline	$C_{15}H_{23}N$	$CDCl_3$	[527]
2-17-132	2-[(3′-hydroxy-4′-methoxy-phenylethyl)]-2,3,4-tetrahydro-1-methylquinoline	$C_{19}H_{23}NO_2$	$CDCl_3$	[527]
2-17-133	2,3-methylenedioxy-4,7-dimethoxyquinoline	$C_{12}H_{11}NO_4$	$CDCl_3$	[528]



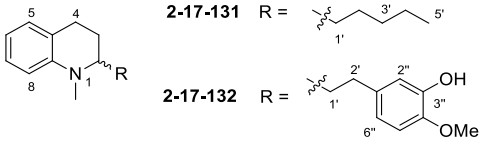


Table 2-17-35: ^{1}H NMR spectroscopic data of other quinoline alkaloids **2-17-125**~**2-17-129**.

H	2-17-125	2-17-126	2-17-127	2-17-128	2-17-129
2			4.10 s(OMe)		
3	6.64 s	7.07 s		8.48 d(8.4)	8.47 d(8.4)
4	4.05 s(OMe)	4.09 s(OMe)	4.00 s(OMe)	8.69 d(8.4)	8.70 d(8.4)
5	8.16 dd(7.5, 0.8)	7.71 dd(8.2, 1.2)	7.49 d(8)		
6	7.46 ddd(7.9, 7.2, 0.8)	7.45 ddd(8.8, 8.2, 1.5)	7.04 d(8)	4.10 s(OMe)	4.00 s(OMe)
7	7.68 ddd(8.3, 7.0, 1.4)	8.13 ddd(8.8, 8.8, 1.2)		5.12 br s(NH_2)	5.97 d(6.9, NH)
8	8.00 d(7.4)	7.67 dd(8.8, 1.5)			
1′	2.93 dd(8.0, 7.9)		2.88 d(6)		
2′	1.84 m	7.67 d(1.7)	3.80 t(6)	12.39 br s(COOH)	
			2.54 br s(OH)		
3′	1.48 m		3.29 s(OMe)	2.50 s(Me)	2.50 s(Me)
4′	1.41 m		1.27 s		
5′	1.61 m	6.93 d(8.1)	1.27 s		
6′	2.54 dd(7.7, 7.6)	7.61 dd(8.1, 1.7)			
8′				5.95 br s(OH)	5.94 br s(OH)
9′				3.96 s(OMe)	3.95 s(OMe)
10′				4.00 s(OMe)	3.96 s(OMe)
11′				6.68 d(8.6)	6.69 d(8.6)
12′				6.80 d(8.5)	6.81 d(8.6)
2″	6.68 d(1.4)				4.74 pent(7.1)
3″					2.18 s
4″					1.49 d(7.2)
5″	6.73 d(7.9)				
6″	6.62 dd(8.0, 1.3)				
OCH_2O	5.93 s	6.03 s	6.19 s		

Table 2-17-36: ^{1}H NMR spectroscopic data of other quinoline alkaloids **2-17-130**~**2-17-133**.

H	2-17-130	2-17-131	2-17-132	2-17-133
2		3.35 m	3.26 m	
3	3.90 s(OMe)	2.01 m	1.92 m	
4	7.22 s	2.94 m, 2.78 m	2.82 m, 2.68 m	4.25 s(OMe)
5	7.31 d(8.1)	6.65 d(8.2)	6.51 d(8.2)	7.85 d(9.1)

Table 2-17-36 (continued)

H	2-17-130	2-17-131	2-17-132	2-17-133
6	6.96 d(8.1)	7.21 t(8.2)	7.06 t(8.2)	6.99 dd(9.1, 2.5)
7	4.14 s	6.71 td(7.3, 1)	6.57 td(7.3, 0.9)	3.88 s(OMe)
8		7.09 d(7.2)	6.96 d(7.3)	7.16 d(2.5)
1′		1.73 m	1.87①	
2′	6.91 d(1.2)	1.42~1.55①	1.70①	
3′	3.79 s(OMe)	1.42~1.55①		
4′		1.42~1.55①		
5′	6.70 (8.1)①	1.42~1.55①		
6′	6.71 (1.2, 8.1)①			
2″			6.65 s	
4″			3.85 s(OMe)	
5″			6.80 d(7.2)	
6″			6.65 dd(7.1, 1.9)	
N-Me		3.04 s	2.89 s	
OCH_2O				6.00 s

① The peaktypes were not given in the literature.

Bibliography

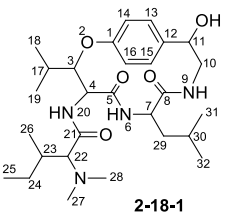
[466] Ito C, Itoigawa M, Furukama A, et al. J Nat Prod, 2004, 67: 1800.
[467] Puricelli L, Innocenti G, Monache GD, et al. Nat Prod Lett, 2002, 16: 95.
[468] Noshita T, Tando M, Suzuki K, et al. Biosci Biotechnol Biochem, 2001, 65: 710.
[469] Simpson DS, Jacobs H. Biochem Syst Ecol, 2005, 33: 841.
[470] Niu XM, Li SH, Peng LY, et al. J Asian Nat Prod Res, 2001, 3: 299.
[471] Ito C, Kondo Y, Wu TS, et al. Chem Pharm Bull, 2000, 48: 65.
[472] Chen JJ, Chang YL, Teng CM, et al. Planta Med, 2002, 68: 790.
[473] Chen IS, Chen HF, Cheng MJ, et al. J Nat Prod, 2001, 64: 1143.
[474] Chung HS, Woo SW. J Nat Prod, 2001, 64: 1579.
[475] Chen SB, Gao GY, Li YS, et al. Planta Med, 2002, 68: 554.
[476] Aoki S, Ye Y, Higuchi K, et al. Chem Pharm Bull, 2001, 49: 1372.
[477] Wu TS, Leu YL, Chan YY, et al. Phytochemistry, 1998, 49: 1467.
[478] Ito C, Itoigawa M, Otsuka T, et al. J Nat Prod, 2000, 63: 1344.
[479] Al-Rehaily AJ, Al-Howiriny TA, Ahmad MS, et al. Phytochemistry, 2001, 57: 597.
[480] Lin YC, Shao ZY, Jiang G, et al. Tetrahedron, 2000, 56: 9607.
[481] Kusano M, Koshino H, Uzawa J, et al. Biosci Biotechnol Biochem, 2000, 64: 2559.
[482] Fakhouri W, Walker F, Vogler B, et al. Phytochemistry, 2001, 58: 1297.
[483] Wu XY, Qin GW. Tetrahedron, 1997, 53: 13323.
[484] Fokialakis N, Magiatis P, Chinou I, et al. Chem Pharm Bull, 2002, 50: 413.
[485] Chen IS, Tsai IW, Teng CM. Phytochemistry, 1997, 46: 525.
[486] Bringmann G, Schlauer J, Rischer H, et al. Tetrahedron, 2000, 56: 3691.
[487] Buske A, Schmidt J, Porzel A, et al. Eur J Org Chem, 2001, 3537.
[488] Khaleel AEM. Monatsh Chem, 2002, 133: 183.
[489] Kim WG, Song NK, Yoo ID. J Antibiotics, 2001, 54: 831.
[490] Fokialakis N, Magiatis P, Terzis A, et al. Tetrahedron Lett, 2001, 42: 5323.

[491] Linma MP, Rosas LV, Silva MFGF, et al. Phytochemistry, 2005, 66: 1560.
[492] Sartor CFP, Silva MFGF, Fernandes JB, et al. Phytochemistry, 2003, 63: 185.
[493] Sayed KE, Al-Said MS, El-Feraly FS, et al. J Nat Prod, 2000, 63: 995.
[494] Touati D, Rahman A, Ulubelen A. Phytochemistry, 2000, 53: 277.
[495] Oliva A, Meepagala KM, Wedge DE, et al. J Agric Food Chem, 2003, 51: 890.
[496] Wang S, Ding XB, Chen YF, et al. J Asian Nat Prod Res, 2001, 3: 89.
[497] Fokialakis N, Magiatis P, Mitaku S, et al. Chem Pharm Bull, 2000, 48: 2009.
[498] Nicholas GM, Newton GL, Fahey RC, et al. Org Lett, 2001, 3: 1543.
[499] Fokialakis N, Magiatis P, Skaltsounis AL, et al. J Nat Prod, 2000, 63: 1004.
[500] Wu TS, Shi LS, Wang JJ, et al. J Chin Chem Soc, 2003, 50: 171.
[501] Ko JS, Rho MC, Chung MY, et al. Planta Med, 2002, 68: 1131.
[502] Moraes VRS, Tomazela DM, Ferracin RJ, et al. J Braz Chem Soc, 2003, 14: 380.
[503] Ali MS, Pervez MK, Saleem M, et al. Phytochemistry, 2001, 57: 1277.
[504] Cantrell CL, Schrader KK, Mamonov LK, et al. J Agric Food Chem, 2005, 53: 7741.
[505] Sultana N, Waterman PG. Phytochemistry, 2001, 58: 329.
[506] Moura NF, Morel AF, Dessoy EC, et al. Planta Med, 2002, 68: 534.
[507] Moura NF, Simionatto E, Porto C, et al. Planta Med, 2002, 68: 631.
[508] Wabo HK, Tane P, Connolly JD, et al. Nat Prod Res, 2005, 19: 591.
[509] Grougnet R, Magiatis P, Fokialakis N, et al. J Nat Prod, 2005, 68: 1083.
[510] Komala I, Rahmani M, Sukari MA, et al. Nat Prod Res, 2006, 20: 355.
[511] Tarus PK, Coombes PH, Crouch NR, et al. Phytochemistry, 2005, 66: 703.
[512] Al-Rehaily AJ, Ahmad MS, Muhammad I, et al. Phytochemistry, 2003, 64: 1405.
[513] Chaturvedula VSP, Schilling JK, Miller JS, et al. J Nat Prod, 2003, 66: 532.
[514] Chen JJ, Duh CY, Huang HY, et al. Planta Med, 2003, 69: 542.
[515] He HP, Shen YM, Zuo GY, et al. Helv Chim Acta, 2003, 86: 3187.
[516] Ambrozin ARP, Mafezoli J, Vieira PC, et al. J Braz Chem Soc, 2005, 16: 434.
[517] Chosson E, Hay AE, Chiaroni A, et al. Heterocycles, 2004, 63: 2043.
[518] Santo CS, Januario AH, Vieira PC, et al. J Braz Chem Soc, 1998, 9: 39.
[519] Fokialakis N, Mitaku S, Mikros E, et al. Phytochemistry, 1999, 52: 1745.
[520] Wu TS, Li CY, Leu YL, et al. Phytochemistry, 1999, 50: 509.
[521] Davis RA, Carroll AR, Quinn RJ. J Nat Prod, 2002, 65: 454.
[522] Wright AD, Goclik E, Konig GM, et al. J Med Chem, 2002, 45: 3067.
[523] Gao WY, Li YM, Jiang SH, et al. Planta Med, 2000, 66: 664.
[524] Morita H, Hirasawa Y, Yoshida N, et al. Tetrahedron Lett, 2001, 42: 4199.
[525] Wang HS, Yeo SL, Xu J, et al. J Nat Prod, 2002, 65: 721.
[526] Noda N, Yashiki Y, Nakatani T, et al. Chem Pharm Bull, 2001, 49: 930.
[527] Collet IJ, Hannedouche S, Fabre N, et al. Phytochemistry, 1999, 51: 1167.
[528] Cui B, Chai H, Dong Y, et al. Phytochemistry, 1999, 52: 95.

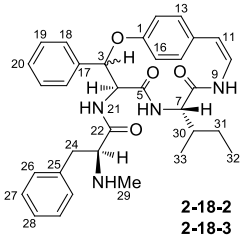
2.18 Peptide alkaloids

Table 2-18-1: Cos, MFs, and TSs of peptide alkaloids **2-18-1**~**2-18-15**.

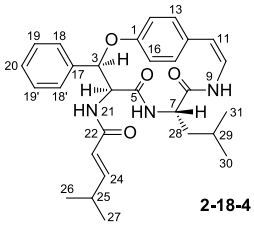
No.	Compounds	MFs	Test solvents	References
2-18-1	discarine L	$C_{28}H_{46}N_4O_5$	DMSO-d_6	[529]
2-18-2	condaline A	$C_{33}H_{38}N_4O_4$	DMSO-d_6	[530]
2-18-3	scutianine M	$C_{33}H_{38}N_4O_4$	DMSO-d_6	[531]
2-18-4	discarene C	$C_{29}H_{35}N_3O_4$	$CDCl_3$	[532]
2-18-5	discarene D	$C_{32}H_{33}N_3O_4$	$CDCl_3$	[532]
2-18-6	scutianene E	$C_{32}H_{33}N_3O_5$	DMSO-d_6	[533]
2-18-7	3,4,28-tris-*epi*-scutianene E	$C_{32}H_{33}N_3O_5$	DMSO-d_6	[533]
2-18-8	28-*epi*-scutianene E	$C_{32}H_{33}N_3O_5$	DMSO-d_6	[533]
2-18-9	mauritine L	$C_{30}H_{40}N_4O_4$	$CDCl_3$	[534]
2-18-10	lotusine A	$C_{30}H_{38}N_4O_4$	$CDCl_3$	[535]
2-18-11	lotusine D	$C_{29}H_{36}N_4O_4$	$CDCl_3$	[535]
2-18-12	lotusine F	$C_{29}H_{36}N_4O_5$	$CDCl_3$	[536]
2-18-13	lotusine E	$C_{36}H_{49}N_5O_6$	$CDCl_3$	[536]
2-18-14	lotusine C	$C_{35}H_{47}N_5O_5$	$CDCl_3$	[536]
2-18-15	lotusine B	$C_{36}H_{49}N_5O_5$	$CDCl_3$	[536]



2-18-1



2-18-2 R = α-H
2-18-3 R = β-H



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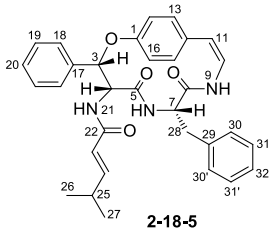

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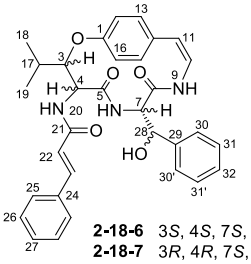
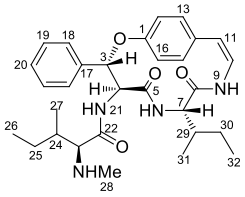
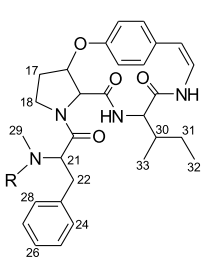


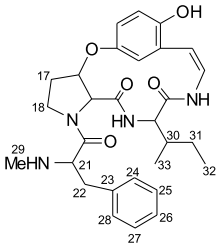
2-18-6 3*S*, 4*S*, 7*S*, 28*R*
2-18-7 3*R*, 4*R*, 7*S*, 28*S*
2-18-8 3*S*, 4*S*, 7*S*, 28*S*



2-18-9



2-18-10 R = Me
2-18-11 R = H



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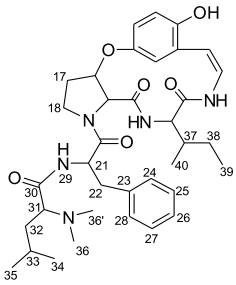
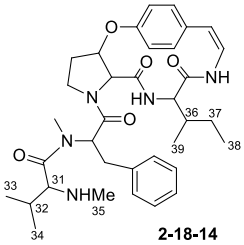
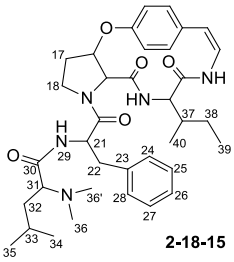


2-18-13



2-18-14



2-18-15

Table 2-18-2: ^{1}H NMR spectroscopic data of peptide alkaloids **2-18-1**~**2-18-5**.

H	2-18-1	2-18-2	2-18-3	2-18-4	2-18-5
3	4.69 dd(2.0, 9.0)	5.81 br s	6.16 (7.7)	6.14 d(6.2)	5.80 d(7.6)
4	4.35 dd(9.0, 10.0)	4.58 d(9.2)	4.70 dd(7.7, 9.0)	4.86 dd(6.2, 8.8)	4.88 dd(7.6, 10.0)
6	6.72 d(9.0)	8.03 d(9.0)	6.36 d(8.2)	6.38 (NH)	8.03 d(8.2, NH)
7	4.20 m	3.90 dd(9.0, 4.4)	4.08 dd(8.2, 3.8)	4.13 m	4.06 m
9	7.54 d(10.0)	–	6.60 d(10.0)	6.64 d(10.2, NH)	–
10	4.02 m, 2.80 m	6.03 d(7.6, 9.0)	6.72 d(7.0, 10.0)	6.74 dd(7.6, 10.2)	6.51
11	4.90 d(4.0)	6.98 d(7.6)	6.41 d(7.0)	6.37 d(7.6)	6.51
13	6.85 dd(8, 2)	6.99	–	7.17	6.97~7.28
14	7.24 dd(8, 2)	6.93	–	7.33	6.97~7.28
15	6.85 dd(8, 2)	7.15	–	7.41	6.97~7.28
16	6.78 dd(8, 2)	6.81	7.00~7.60	7.15	6.97~7.28
17	2.20 m				
18	1.02 d(7)	7.58	–	7.52	7.42
19	0.89 d(7)	7.36	–	7.47	6.97~7.28
20	8.17 d(10)	7.25	–	7.43	6.97~7.28
21		8.03 d(9.2)	7.40 (ov)	5.12 d(8.8, NH)	7.59 d(10.0, NH)
22	2.68 d				
23	1.71 m	3.02 dd(5.6, 13.6)	2.90 dd(9.4, 3.6)	5.23 d(14.0)	5.32 d(14.0)
24	1.52 m, 1.05 m	2.52 dd(13.6, 8.4)	2.78 dd(9.4, 14.0)	6.36 dd(6.8, 14.0)	6.29 dd(6.6, 14.0)
		2.38 dd(5.6, 8.4)	1.58 dd(3.6, 14.0)		
25	0.78 t(7)			2.27 m	2.25 m
26	0.60 d(7)	7.0~7.5	7.0~7.6	0.92 d(6.7)	0.90 d(6.0)

Table 2-18-2 (continued)

H	2-18-1	2-18-2	2-18-3	2-18-4	2-18-5
27	2.16 s	7.0~7.5	7.0~7.6	0.91 d(6.7)	0.92 d(6.0)
28	2.16 s	7.0~7.5	7.0~7.6	1.79 m	3.01 dd(6.4, 14)
				1.29 m	2.60 dd(6.4, 14)
29	1.25 m, 1.71 m	2.08 s	1.90 s	1.18 m	
30	1.24 m	1.40 m	2.18 m	0.75 d(6.5)	6.67~7.28
31	0.71 d(7)	1.29 m, 0.86 m	1.24 m, 1.0 m	0.64 d(6.5)	6.67~7.28
32	0.68 d(7)	0.83 t(6.5)	0.83 t(7.0)		6.67~7.28
33		0.84 d(6.5)	0.70 d(7.0)		
OH	5.30				

Table 2-18-3: ^{1}H NMR spectroscopic data of peptide alkaloids **2-18-6**~**2-18-9**.

H	2-18-6	2-18-7	2-18-8	2-18-9
3	4.81 dd(7.1, 1.8)	4.16 dd(7.2, 2.0)	4.76 dd(8.1, 1.8)	6.17 d(6.3)
4	4.67 dd(7.1, 9.8)	4.19 dd(7.2, 8.8)	4.34 dd(8.1, 10.0)	4.64 dd(8.4, 6.3)
6	7.10 d(9.9)	7.08 d(9.9)	6.66 d(10.0)	6.42 d(7.8)
7	4.03 dd(1.8, 9.9)	4.06 dd(7.2, 9.9)	4.05 dd(8.8, 10.0)	4.03 dd(7.8, 3.1)
9	6.90 d(6.8)	8.30 ls	8.06 d(2.5)	6.57 br d(9.7)
10	6.45 dd(8.8, 6.8)	5.96 dd(7.0, 1.0)	6.09 dd(7.4, 2.5)	6.71 dd(9.7, 7.3)
11	6.63 d(8.8)	6.75 d(7.0)	6.65 d(7.4)	6.36 d(7.3)
13	6.68~7.19	6.68~7.64	7.56 d(7.0)	7.34 m
14	6.68~7.19	6.68~7.64	7.47 d(7.0)	7.12 br t(7.8)
15	7.57 d(7.2)	–	7.13 d(7.5)	7.12 br t(7.8)
16	6.68~7.19	–	6.99 d(7.5)	7.34 m
17	2.05 m	1.86 m	2.04 m	
18	1.12 d(6.4)	1.15 d(6.8)	1.06 d(6.8)	7.50 br d(7.4)
19	0.82 d(6.4)	0.89 d(6.8)	0.84 d(6.8)	7.40 m
20	8.33 d(9.8)	7.96 d(8.8)	8.00 d(10.0)	7.40 m
21				7.43
22	6.42 d(16.3)	6.42 d(15.7)	6.50 d(15.8)	
23	7.48 d(16.3)	7.50 d(15.7)	7.46 d(15.8)	2.52 d(4.1)
24				1.36 m
25	6.68~7.19	6.66~7.64	6.83~7.47	0.75 m, 0.51 m
26	6.68~7.19	6.66~7.64	6.83~7.47	0.65 t(6.4)
27	6.68~7.19	6.66~7.64	6.83~7.47	0.62 d(6.8)
28	5.15 (1.8, 1)	4.26 dd(7.2, 4.6)	4.41 dd(8.8, 4.4)	2.05 s
29				2.15 m
30	6.68~7.19	6.66~7.34	6.83~7.47	1.61 m, 0.95 m
31	6.68~7.19	6.66~7.34	6.83~7.47	0.81 t(7.2)
32	6.68~7.19	6.66~7.34	6.83~7.47	0.66 d(6.4)
33				
OH	5.63	5.30 d(4.6)	5.33 d(4.4)	

Table 2-18-4: ^{1}H NMR spectroscopic data of peptide alkaloids **2-18-10**~**2-18-13**.

H	2-18-10	2-18-11	2-18-12	2-18-13
3	5.52 ddd(9.8, 7.2, 5.3)	5.44 ddd(9.8, 7.1, 5.3)	5.33 dt(7.0, 2.8)	5.34 dt(7.0, 2.9)
4	4.26 d(5.3)	4.32 d(5.3)	4.41 d(2.8)	4.40 d(2.9)
6	6.70 d(8.6)	6.75 d(8.5)	7.39 d(5.1)	7.27 m
7	4.14 dd(8.6, 3.0)	4.21 dd(8.5, 3.2)	4.36 dd(4.7, 4.5)	4.35 dd(5.0, 4.5)
9	6.51 d(10.6)	6.57 d(10.5)	8.47 d(11.3)	8.46 d(11.2)
10	6.75 dd(10.6, 7.8)	6.75 dd(10.5, 7.7)	6.96 dd(11.3, 9.0)	6.94 dd(11.2, 9.0)
11	6.29 d(7.8)	6.34 d(7.7)	5.85 d(9.0)	5.87 d(9.0)
13	7.10 m	7.14 m	6.57 d(2.9)	6.58 d(2.8)
14	7.28 m	7.27 m	6.68 dd(8.8, 2.9)	6.67 dd(8.8, 2.8)
15	7.11 m	7.11 m	6.80 d(8.8)	6.80 d(8.8)
16	7.08 m	7.07 m		
17	2.48 ddd(12.2, 7.2, 5.1)	2.44 ddd(12.2, 7.1, 5.2)	2.40~2.31 m	2.42 m
	2.13~2.0 m	2.05 m	2.16~2.06 m	2.20 m
18	4.21 dd(10.8, 8.0)	3.75 dd(11.3, 8.2)	3.67 m	4.03 ddd(11.1, 8.4, 3.0)
	3.07~2.99 m	2.59 m	2.51 dt(6.5, 8.5)	2.88 m
21	3.51 dd(9.1, 4.0)	3.56 t(7.0)	3.60 dd(9.3, 4.9)	4.96 q(7.7)
22	3.07~2.99 m	2.90 dd(13.4, 7.0)	3.05 dd(13.0, 4.9)	2.96 br d(7.4)
	2.90 dd(13.8, 4.0)	2.67 dd(13.4, 7.0)	2.74 dd(13.0, 9.3)	2.96 br d(7.4)
24	7.19 m	7.25 m	7.02 dd(7.7, 2.0)	7.08 dd(7.6, 1.9)
25	7.07 m	7.06 m	7.25 m	7.26 m
26	7.15 m	7.23 m	7.21 m	7.25 m
27	7.07 m	7.06 m	7.25 m	7.26 m
28	7.19 m	7.25 m	7.02 dd(7.7, 2.0)	7.08 dd(7.6, 1.9)
29	2.39 br s, 2.39 br s	2.34 br s	2.38 br s	7.59 br d(7.7)
30	2.13~2.0 m	2.20 m	2.16~2.06 m	
31	1.28 m, 1.13 m	1.31 m, 1.15 m	1.44 ddd(13.2, 7.3, 3.8) 1.28~1.15 m	2.90 m
32	0.88 t(7.4)	0.90 t(7.3)	0.92 t(7.3)	1.48 m
33	0.62 d(7.0)	0.86 d(7.0)	1.06 d(6.9)	1.61 m
34				0.88 d(6.4)
35				0.89 d(6.4)
36				2.21 br s, 2.21 br s
37				2.09 m
38				1.39 m, 1.20 m
39				0.92 t(7.0)
40				1.03 d(6.9)

Table 2-18-5: ^{1}H NMR spectroscopic data of peptide alkaloids **2-18-14** and **2-18-15**.

H	2-18-14	2-18-15	H	2-18-14	2-18-15
3	5.55 m	5.54 m	24/28	7.26 m	7.22 m
4	4.23 d(5.4)	4.29 d(5.7)	25/27	7.23 m	7.15 m

Table 2-18-5 (continued)

H	2-18-14	2-18-15	H	2-18-14	2-18-15
6	6.46 br d(8.6)	6.56 br d(8.6)	26	7.22 m	7.19 m
7	4.17 dd(8.6, 3.0)	4.20 dd(8.6, 3.0)	29	2.36 br s	–
9	6.47 br d(10.1)	6.50 br d(10.5)	31	4.43 t(8.5)	–
10	6.74 dd(10.1, 7.7)	6.75 dd(10.5, 7.7)	32	1.81~2.01 m	–
11	6.32 d(7.7)	6.32 d(7.7)	33	0.79~0.93 m	
13	7.09 m	7.10 m	34	0.79~0.93 m	0.87 d(6.4)
14	7.14 m	7.12 m	35	2.34 br s	0.85 d(6.4)
15	7.28 m	7.28 m	36	2.10~2.20 m	2.22 br s, 2.22 br s
16	7.12 m	7.07 m	37	1.19~1.29 m	2.17~2.30 m
				1.01~1.13 m	
17	2.53~2.61 m	2.45~2.60 m	38	0.86 t(6.5)	1.20~1.35 m
	2.13~2.34 m	2.10~2.25 m			1.05~1.20 m
18	4.32 dd(10.6, 8.3)	4.18~4.20 m	39	0.75 d(6.9)	0.91 t(7.2)
	3.43 m	2.90~3.05 m			
21	3.36 t(6.4)	4.89 q(7.0)	40		0.82 d(6.9)
22	3.21 dd(14.0, 6.4)	2.86 br d(7.0)	NH	7.36 d(8.5)	
	2.92 dd(14.0, 6.4)	2.86 br d(7.0)			

Bibliography

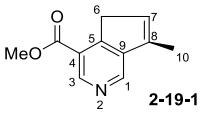
[529] Morel AF, Machado EC, Wessjohann LA. Phytochemistry, 1995, 39: 431.
[530] Morel AF, Araujo CA, Silva UFD, et al. Phytochemistry, 2002, 61: 561.
[531] Morel AF, Maldaner G, Ilha V, et al. Phytochemistry, 2005, 66: 2571.
[532] Giacomelli SR, Missau FC, Mostardeiro MA, et al. J Nat Prod, 2001, 64: 997.
[533] Maldaner G, Marangon P, Ilha V, et al. Phytochemistry, 2011, 72: 804.
[534] Panseeta P, Lomchoey K, Prabpai S, et al. Phytochemistry, 2011, 72: 909.
[535] Ghedira K, Chemli R, Richard B, et al. Phytochemistry, 1993, 32: 1591.
[536] Ghedira K, Chemli R, Caron C, et al. Phytochemistry, 1995, 38: 767.

2.19 Monoterpenoid alkaloids

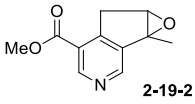
2.19.1 Pyridine monoterpenoid alkaloids

Table 2-19-1: Cos, MFs, and TSs of pyridine monoterpenoid alkaloids **2-19-1**~**2-19-11**.

No.	Compounds	MFs	Test solvents	References
2-19-1	racemigerine	$C_{11}H_{11}NO_2$	$CDCl_3$	[537]
2-19-2	6,7-epoxyracemigerine	$C_{11}H_{11}NO_3$	$CDCl_3$	[537]
2-19-3	scaevoline	$C_{11}H_{11}NO_3$	$CDCl_3$	[537]
2-19-4	7-*O*-thiocarbonylimidazoylcantleyine	$C_{15}H_{15}N_3O_3S$	$CDCl_3$	[538]
2-19-5	(*S*)-(–)-deoxyrhexifoline	$C_{11}H_{13}NO_2$	$CDCl_3$	[538]
2-19-6	(*S*)-(–)-tecostidine	$C_{10}H_{13}NO$	$CDCl_3$	[538]
2-19-7	(*S*)-(–)-acetyltecostidine	$C_{12}H_{15}NO_2$	$CDCl_3$	[538]
2-19-8	coumarate *trans* de l'hydroxy-9 cantleyine	$C_{20}H_{19}NO_6$	$CDCl_3$	[539]
2-19-9	coumarate *cis* de l'hydroxy-9 cantleyine	$C_{20}H_{19}NO_6$	$CDCl_3$	[539]
2-19-10	argutine A	$C_{13}H_{15}NO$	$CDCl_3$	[540]
2-19-11	argutine B	$C_{10}H_{12}N_2O$	$CDCl_3$	[540]



2-19-1



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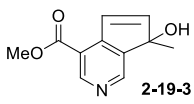

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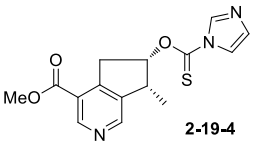

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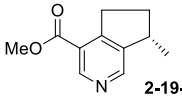
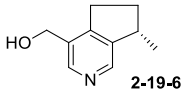
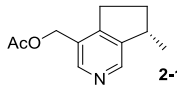


2-19-5



2-19-6



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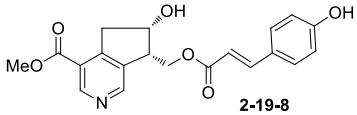
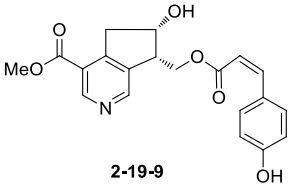
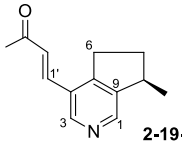


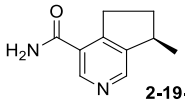
2-19-8



2-19-9



2-19-10



2-19-11

Table 2-19-2: ^{1}H NMR spectroscopic data of pyridine monoterpenoid alkaloids **2-19-1**~**2-19-5**.

H	2-19-1	2-19-2	2-19-3	2-19-4	2-19-5
1	8.79 s	9.08 s	8.77 s	8.66 s	8.54 s
3	8.40 s	8.75 s	8.46 s	9.12 s	8.99 s
6	3.50 m	3.36 dd(19, 5)	7.16 d(16)	3.73 m	3.40 ddd(18, 8, 4)
		3.58 dd(19, 3)			3.14 ddd(18, 8, 3)
7	6.15 m	3.77 dd(5, 3)	6.65 d(6)	6.28 ddd(7, 5, 3)	2.40 m, 1.68 m
8				3.73 m	3.31 dqd(8, 7, 1)
10	2.15 d(2)	1.52 s	1.62 s	1.51 d(8)	1.35 d(7)
OMe	3.87 s	3.94 s	3.93 s	4.00 s	3.94 s
2′				8.26 s	
4′				7.02 d(3)	
5′				7.51 d(3)	

Table 2-19-3: ^{1}H NMR spectroscopic data of pyridine monoterpenoid alkaloids **2-19-6**~**2-19-8**.

H	2-19-6	2-19-7	2-19-8	2-19-8 (diethyl ester)
1	8.36 br s	8.37 s	8.82 s	8.68 s
3	8.36 br s	8.40 s	9.01 s	9.01 s
5			3.37 dd(18.7, 4.0)	3.44 dd(18.9, 3.8)
			3.47 dd(18.7, 6.1)	3.58 dd(18.9, 6.0)
6	3.02 ddd(17, 9, 4)	3.00 ddd(16, 8, 4)	4.78 m	5.75 ddd(6.7, 6.0, 3.8)
	2.91 ddd(17, 9, 1)	2.88 dd(16, 9)		
7	2.38 m, 1.69 m	2.38 m, 1.67 m	3.65 ddd(6.7, 6.5, 6.0)	3.84 ddd(6.7, 6.5, 6.0)
8	3.30 qdd(7, 6, 1)	3.30 qdd(7, 6, 1)		
9			4.59 dd(12, 6.0)	4.54 dd(13, 6.0)
			4.68 dd(12, 6.5)	
10	1.34 d(7)	1.35 d(7)		
11	4.72 s	5.12 s		
OMe			4.00 s	3.90 s
OAc		2.11 s		2.03 s(6-OAc)
				2.28 s(7′-OAc)
2′			6.60 d(16.0)	6.36 d(16.0)
3′			7.72 d(16.0)	7.61 d(16.0)
5′			7.71 d(8.0)	7.49 d(8.6)
6′			6.94 d(8.0)	7.09 d(8.6)
8′			6.94 d(8.0)	7.09 d(8.6)
9′			7.71 d(8.0)	7.49 d(8.6)

Table 2-19-4: ^{1}H NMR spectroscopic data of pyridine monoterpenoid alkaloids **2-19-9**~**2-19-11**.

H	2-19-9 (diethyl ester)	2-19-10	2-19-11
1	8.68 s	8.41 s	8.50 s
3	9.01 s	8.59 s	8.69 s
5	3.42 dd(18.9, 3.9)		
	3.56 dd(18.9, 6.0)		
6	5.70 ddd(6.7, 6.0, 3.9)	3.11 m, 2.97 m	3.27 m, 3.10 m
7	3.83 ddd(6.7, 6.5, 6.0)	2.42 m, 1.63 m	2.42 m, 1.72 m
8		3.32 q	3.30 q
9	4.40 dd(13, 6.0)		
10		1.30 d(7.0)	3.32 d(7.0)
OMe	3.89 s		
6-OAc	1.99 s		
7′-OAc	2.26 s		
1′		7.57 d(16.5)	
2′	5.95 d(12.5)	6.75 d(16.5)	
3′	6.90 d(12.5)		
4′		2.39 s	
5′	7.63 d(8.7)		
6′	7.07 d(8.7)		
8′	7.07 d(8.7)		
9′	7.63 d(8.7)		
NH_2			6.04 br

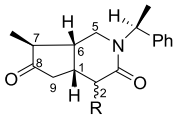
2.19.2 Piperidine monoterpenoid alkaloids

Table 2-19-5: Cos, MFs, and TSs of piperidine monoterpenoid alkaloids **2-19-12**~**2-19-26**.

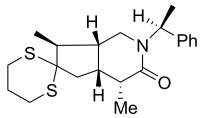
No.	Compounds	MFs	Test solvents	References
2-19-12	(–)-(1*R*,5*R*,6*R*,9*S*,l′*S*)-5,9-dimethyl-3-(1′-phenylethyl)-3-azabicyclo[4.3.0]nonane-4,8-dione	$C_{18}H_{23}NO_2$	$CDCl_3$, $CDCl_3$-C_6D_6	[541]
2-19-13	(–)-(1*R*,5*S*,6*R*,9*S*,l′*S*)-5,9-dimethyl-3-(1′-phenylethyl)-3-azabicyclo[4.3.0]nonane-4,8-dione	$C_{18}H_{23}NO_2$	$CDCl_3$	[541]
2-19-14	(–)-(1*R*,5*R*,6*R*.9*S*,1′*S*)-5,9-dimethyl-3-(1′-phenylethyl)-spiro{[3]azabicyclo[4.3.0]-nonane-8,2″-[l,3]dithiane}-4-one	$C_{21}H_{29}NOS_2$	$CDCl_3$	[541]
2-19-15	(–)-(1*R*,5*S*,6*R*,9*S*,l′*S*)-5,9-dimethyl-3-(1′-phenylethyl)-spiro{[3]azabicyclo[4.3.0]-nonane-8,2″-[l,3]dithiolane}-4-one	$C_{20}H_{27}NOS_2$	$CDCl_3$	[541]

Table 2-19-5 (continued)

No.	Compounds	MFs	Test solvents	References
2-19-16	(–)-(1*R*,5*S*,6*R*,9*S*,1′*S*)-5,9-dimethyl-3-(1′-phenylethyl)-spiro{[3]azabicyclo[4.3.0]-nonane-8,2″-[1,3]dithiane}-4-one	$C_{21}H_{29}NOS_2$	$CDCl_3$	[541]
2-19-17	(–)-(1*S*,5*S*,6*S*,9*R*)-3.5,9-trimethyl-3-azabicyclo[4.3.0]-nonane	$C_{11}H_{21}N$	$CDCl_3$	[541]
2-19-18	(1*R*,5*S*,6*S*,9*S*,1′*S*)-5.9-dimethyl-3-(l'-phenylethyl)spiro{[3]azabicyclo[4.3.0]-nonane-8,2″-[1,3]dithiolane}	$C_{20}H_{29}NS_2$	$CDCl_3$	[541]
2-19-19	(–)-(1*R*,5*S*,6*S*,9*S*,1′*S*)-5,9-dimethyl-3-(1′-phenylethyl)spiro{[3]azabicyclo[4.3.0]-nonane-8,2″-[1,3]dithiane}	$C_{21}H_{31}NS_2$	$CDCl_3$	[541]
2-19-20	(–)-(1*R*,5*S*,6*R*,9*S*,1′*S*)-5.9-dimethyl-3-(1′-phenylethyl)spiro{[3]azabicyclo[4.3.0]-nonane-8,2″-[1,3]dioxolane}-4-one	$C_{20}H_{27}NO_3$	$CDCl_3$	[541]
2-19-21	(–)-(1*R*,5*S*,6*S*,9*S*)-5,9-dimethylspiro{[3]-azabicyclo[4.3.0]nonane-8,2″-[l,3]dioxolane}	$C_{12}H_{21}NO_2$	$CDCl_3$	[541]
2-19-22	(+)-(1*R*,5*S*,6*S*,9*S*)-3,5.9-trimethyl-3-azabicyclo[4.3.0]nonan-8-one	$C_{11}H_{19}NO$	$CDCl_3$	[541]
2-19-23	(–)-(1*S*,5*S*,6*R*,9*R*,1′*S*)-5,9-dimethyl-3-(1′-phenylethyl)-3-azabicyclo[4.3.0]nonan-4-one	$C_{18}H_{25}NO$	$CDCl_3$	[541]
2-19-24	(+)-(l*S*,5*S*,6*R*,9*R*)-5,9-dimethyl-3-azabicyclo[4.3.0]nonan-4-one	$C_{10}H_{17}NO$	$CDCl_3$	[541]
2-19-25	(–)-(1*S*,5*R*,6*S*,9*R*)-5,9-dimethyl-3-azabicyclo[4.3.0]nonane	$C_{10}H_{19}N$	$CDCl_3$	[541]
2-19-26	incarvilline	$C_{11}H_{21}NO$	$CDCl_3$	[542]



2-19-12 R = α-Me
2-19-13 R = β-Me



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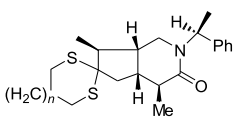
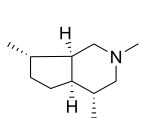


2-19-15 n = 0
2-19-16 n = 1



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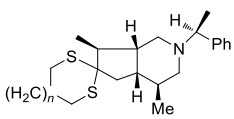
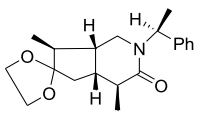


2-19-18 n = 0
2-19-19 n = 1



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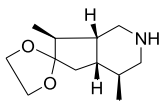

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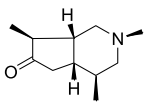

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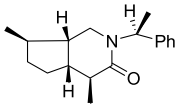

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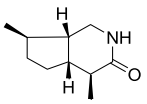
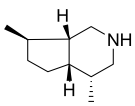
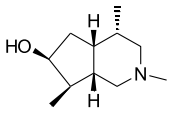


2-19-24



2-19-25



2-19-26

Table 2-19-6: ^{1}H NMR spectroscopic data of piperidine monoterpenoid alkaloids **2-19-12**~**2-19-15**.

H	2-19-12	2-19-12 ($CDCl_3$)	2-19-13 ($CDCl_3$-C_6D_6, 3:2)	2-19-14	2-19-15
1	2.20 m	1.87 dddd(10.0, 7.0, 5.2, 2.0)	1.80 m(9.5, 9.5, 9.5, 6.0)	2.11 m	1.40 m
2ax	2.95 dd(18.0, 2.0)	2.71 dd(13.5, 2.0)	3.01 dd(13.0, 9.5)	2.80 m	2.85 dd(13.7, 10.6)
				2.92 dd(13.8, 4.3)①	
2eq	3.15 dd(18.0, 6.0)	2.82 dd(13.5, 5.2)	3.25 dd(13.0, 6.0)		3.10 dd(13.7, 6.0)
5	2.65 quint(6.0)	2.34 quint(6.5)	2.18~2.30 m	2.50 quint(7.0)	2.19 m
6	2.81 dddd(10.0, 10.0, 9.0, 6.5)	2.44 ddt(10.0, 9.0, 6.5)	2.18~2.30 m	2.66~2.77 m	2.00 m
7	2.07 ddd(17.0, 10.0, 2.0)	1.96 ddd(17.8, 10.0, 2.0)	2.52 dd(18.0, 7.9)	1.49 dd(12.8, 11.0)	1.92 dd(12.5, 10.2)
				2.80 m	2.57 dd(12.5, 7.3)
	2.32 dd(17.0, 9.0)	2.14 dd(17.8, 9.0)			
9	2.20 m	2.04 dq(7.0, 2.0)	2.02 m(9.5, 7.2)	1.77 m	1.84 dq(10.0, 6.6)
1′	6.08 q(6.3)	5.72 q(7.0)	6.03 q(7.2)	6.05 q(6.9)	5.95 q(7.3)
2′	1.48 d(6.3)	1.29 d(7.0)	1.50 d(7.2)	1.49 d(6.9)	1.43 d(7.3)
4″				2.11 m	
3″ax				2.66~2.77 m	
3″eq				3.08 ddd(14.0, 12.0, 3.0)	
5″ax				2.99 ddd(14.1, 12.1, 2.9)	
5″eq				2.66~2.77 m	
5-Me	1.21 d(6.0)	1.09 d(7.0)	1.38 d(6.1)	1.17 d(6.5)	1.19 d(6.8)
9-Me	1.10 d(6.0)	0.96 d(7.1)	0.96 d(7.0)	1.14 d(7.0)	0.90 d(6.6)
Ar-H	7.22~7.35 m	6.80~7.10 m	7.26~7.38 m	727~7.45 m	7.27~7.45 m
SCH_2CH_2S					3.20~3.30 m

Table 2-19-7: ^{1}H NMR spectroscopic data of piperidine monoterpenoid alkaloids **2-19-16**~**2-19-20**.

H	2-19-16	2-19-17	2-19-18	2-19-19	2-19-20
1	1.75 m	1.38~1.51 m	2.15 m	1.83~1.97 m	1.55 m
2ax	2.86 dd(12.8, 9.5)	1.38~1.51 m	2.00 dd(12.1, 4.0)	2.02 dd(12.1, 3.9)	2.89 dd(13.0, 11.0)
2eq	3.06~3.15 m	2.65 dd(9.7, 2.0)	2.73 dm(12.1)	2.71 dm(12.1)	3.11 dd(13.0, 7.0)
4ax		2.22 dd(11.6, 4.0)	1.48~1.60 m	1.55 m	
4eq		2.79 dt(11.6, 1.1)	2.91 dm(11.5)	2.91 ddd(11.0, 4.0, 2.0)	
5	2.23 dq(10.1, 6.9)	2.07 m	1.48~1.60 m	1.72 m	2.20 dq(11.0, 6.5)
6	2.18 m	1.38~1.51 m	1.48~1.60 m	1.55 m	1.94 tt(11.0, 8.0)
7	1.68 dd(12.6, 8.7)	1.38~1.51 m	2.30 dd(13.6, 2.8)	2.33 dd(14.1, 2.0)	1.48 m
	3.06~3.15 m	1.70 m	2.61 dd(13.6, 8.0)	2.80 m	2.10 dd(12.5, 8.0)
8		1.15 m			
		1.93 m			
9	1.70 m	1.38~1.51 m	2.45 dq(11.5, 6.7)	2.45 dq(14.0, 6.7)	1.72 dq(9.8, 7.2)
1′	5.95 q(7.3)		3.10 q(7.3)	3.31 q(7.3)	5.96 q(7.3)
2′	1.46 d(7.3)		1.46 d(7.3)	1.32 d(7.3)	1.48 d(7.3)
4″	1.84 m			1.83~1.97 m	
	2.11 m			2.12 m	
3″ax	3.00 ddd(14.2, 12.0, 3.8) or 3.06~3.15 m			3.15 ddd(14.1, 12.1, 3.8)	
3″eq	2.74 m			2.80 m	
5″ax	3.00 ddd(14.2, 12.0, 3.8) or 3.06~3.15 m			2.98 ddd(14.0, 12.1, 3.8)	
5″eq	2.74 m			2.80 m	
5-Me	1.21 d(6.9)	0.96 d(6.8)	0.80 d(6.6)	0.81 d(6.5)	1.19 d(6.5)
9-Me	0.95 d(6.5)	0.81 d(6.2)	0.90 d(6.8)	1.00 d(7.0)	0.70 d(7.2)
NMe		2.21 s			
Ar-H	7.27~7.35 m		7.27~7.45 m	7.27~7.35 m	7.29~7.35 m
SCH_2CH_2S			3.15~3.46 m		
OCH_2CH_2O					3.90 s

Table 2-19-8: ^{1}H NMR spectroscopic data of piperidine monoterpenoid alkaloids **2-19-21**~**2-19-23**.

H	2-19-21	2-19-22	2-19-23
1	1.56 m	1.85 m	1.34 m
2ax	2.79 dd(13.0, 4.2)	2.13 dd(12.2, 4.0)	2.83 dd(13.5, 11.0)
2eq	2.98 d(13.0)	2.90 dt(12.2, 2.0)	3.45 dd(13.5, 6.0)
4ax	2.11 dd(12.5, 11.0)	1.59 t(11.0)	
4eq	2.90 ddd(12.5, 3.8, 1.9)	2.73 ddd(11.0, 3.5, 2.0)	
5	1.47 m	1.48 m	2.13 dq(10.5, 6.9)
6	1.51 m	1.66 m	1.92 m
7	1.78 dd(14.0, 2.2)	2.268 m	1.22 m
	1.96 dd(14.0, 7.9)		2.00 m
8			1.10 m
			1.76 d quint(6.5, 2.8)
9	2.20 quint(6.8)	2.49 sext(7.5)	1.47 m
1′			5.99 q(7.3)
2′			1.46 d(7.3)
5-Me	0.79 d(6.5)	0.88 d(6.8)	1.20 d(6.9)
9-Me	0.90 d(6.8)	1.05 d(7.5)	0.80 d(6.6)
NMe		2.26 s	
Ar-H			7.27~7.35 m
OCH_2CH_2O	3.80~3.97 m		

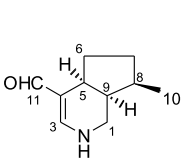
Table 2-19-9: ^{1}H NMR spectroscopic data of piperidine monoterpenoid alkaloids **2-19-24**~**2-19-26**.

H	2-19-24	2-19-25	2-19-26
1	1.85 m	1.60 m	ax 2.68 ddd(2, 6, 12)
			eq 1.56 t(12)
2ax	3.02 ddd(12.0, 10.0, 2.5)	2.24 t(12.8)	
2eq	3.38 dt(12.2, 6.0)	2.76 dd(12.8, 6.8)	
3ax			1.67 t(12)
3eq			2.51 ddd(2, 5, 11.5)
4ax		2.35 t(12.4)	2.08 m
4eq		2.69 dd(12.4, 4.1)	
5	1.99~2.13 m	1.88 m	2.41 ddd(2, 6, 12)
6	1.99~2.13 m	2.17 m	ax 1.50 br q(5, 7, 13)
			eq 1.80 m
7	1.85 m, 1.99~2.13 m	1.54 m, 1.95 m	4.31 td(2, 6.5)
8	1.19~1.31 m	1.13 m, 1.54 m	1.82 m
9	1.65 m	1.60 m	1.93 pent(6, 6, 12)
Me	1.16 d(7.2) (5-Me)	0.85 d(7.1) (5-Me)	0.86 d(4-Me)
	1.01 d(7.0) (9-Me)	0.94 d(7.2) (9-Me)	1.02 d(8-Me)
NH	5.90 br s	1.70 br s	
NMe			2.27 s

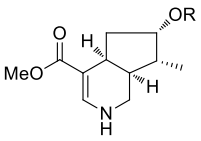
2.19.3 Tetrahydropyridine monoterpenoid alkaloids

Table 2-19-10: Cos, MFs, and TSs of tetrahydropyridine monoterpenoid alkaloids **2-19-27**~**2-19-37**.

No.	Compounds	MFs	Test solvents	References
2-19-27	lindenialine	$C_{10}H_{15}NO$	$CDCl_3$	[543]
2-19-28	tetrahydrocantleyine	$C_{11}H_{17}NO_3$	$CDCl_3$	[537]
2-19-29	6-*O*-nicotinoyltetrahydrocantleyine	$C_{17}H_{20}N_2O_4$	$CDCl_3$	[537]
2-19-30	6-*O*-(5-vinylnicotinoyl)tetrahydrocantleyine	$C_{19}H_{22}N_2O_4$	$CDCl_3$	[537]
2-19-31	6-*O*-nicotinoylstrychnovoline	$C_{17}H_{18}N_2O_5$	$CDCl_3$	[537]
2-19-32	dinklageine	$C_{19}H_{23}NO_5$	DMSO-d_6	[544]
2-19-33	strychnovoline	$C_{11}H_{15}NO_4$	$CDCl_3$	[544]
2-19-34	*O*-acetyl-7-strychnovoline	$C_{13}H_{17}NO_5$	$CDCl_3$	[544]
2-19-35	–	$C_{20}H_{24}N_2O_3$	$CDCl_3$	[545]
2-19-36	–	$C_{16}H_{24}N_2O_3$	$CDCl_3$	[545]
2-19-37	–	$C_{15}H_{22}N_2O_3$	$CDCl_3$	[545]

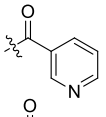


2-19-27

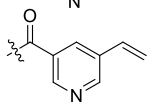


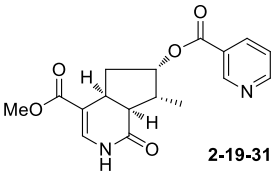
2-19-28 R = H

2-19-29 R =



2-19-30 R =





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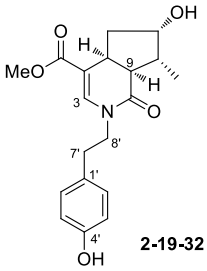
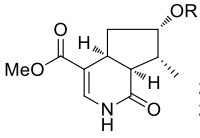
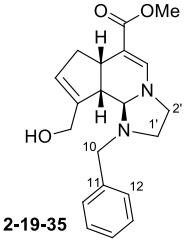


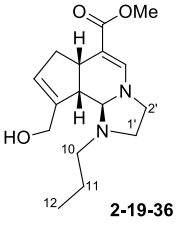
2-19-32



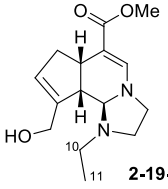
2-19-33 R = H
2-19-34 R = Ac



2-19-35



2-19-36



2-19-37

Table 2-19-11: ^{1}H NMR spectroscopic data of tetrahydropyridine monoterpenoid alkaloids **2-19-27**~**2-19-31**.

H	2-19-27	2-19-28	2-19-29	2-19-30	2-19-31
1ax	2.88 dd(12.5, 12.0)	2.74 ddd(12, 7, 2)	2.93 ddd(12, 7, 2)	2.93 ddd(12, 7, 2)	
1eq	3.23 dt(12.5, 5)	3.18 ddd(12, 5, 2)	3.33 ddd(12, 5, 2)	3.28 ddd(12, 5, 2)	
2	5.48 br s	4.49 s	4.69 s	4.66 s	7.54 d(6)
3	7.10 d(6.5)	7.42 d(6)	7.57 d(6)	7.49 d(6)	7.23 d(6)
5	2.93 td(9, 6)	3.05 td(9, 7)	3.20 td(9, 7)	3.20 td(9, 7)	3.55 ddd(11, 9, 7)
6	1.3 m	1.47 ddd(15, 9, 5)	1.82 ddd(15, 9, 5)	1.98 ddd(15, 9, 5)	1.83 ddd(14, 9, 4)
		2.17 ddd(15, 7, 2)	2.47 ddd(15, 7, 2)	2.44 ddd(15, 7, 2)	2.58 ddd(14, 7, 2)
7	1.93 m	4.06 ddd(5, 4, 2)	5.51 ddd(5, 4, 2)	5.49 ddd(5, 4, 2)	5.46 td(4, 4, 2)
	2.30 m				
8	2.30 m	1.71 dqd(8, 7, 4)	2.13 m	2.11 m	2.65 dqd(8, 7, 4)
9	1.93 m	1.88 dddd(9, 8, 7, 5)	2.13 m	2.11 m	2.82 dd(11, 8)
10	1.0 d(7.0)	1.00 d(7)	1.26 d(7)	1.10 d(7)	1.30 d(7)
11	8.95 s				
OMe		3.60 s	3.69 s	3.66 s	3.70 s
2′			9.24 d(1)	9.08 d(1)	9.03 d(1)
4′			8.33 dt(8, 1)	8.29 t(1)	8.29 dt(8, 1)
5′			7.44 dd(8, 5)		7.42 dd(8, 5)
6′			8.80 dd(5, 1)	8.74 d(1)	8.80 dd(5, 1)
1″				6.73 dd(18, 12)	
2″				5.91 d(18)	
				5.47 d(12)	

Table 2-19-12: ^{1}H NMR spectroscopic data of tetrahydropyridine monoterpenoid alkaloids **2-19-32**~**2-19-34**.

H	2-19-32	2-19-33	2-19-34
2		7.52	7.46
3	7.15 s	7.22 d(6)	7.17 d(6)
5	3.26 ddd(11, 9, 7)	3.56 ddd(11, 9, 7)	3.48 ddd(11, 9, 7)
6α	2.11 ddd(14, 7, 2)	2.44 ddd(14, 7, 2)	2.49 ddd(14, 7, 1)
6β	1.38 ddd(14, 9, 4)	1.62 ddd(14, 9, 4)	1.71 ddd(14, 9, 4)
7	3.78 td(4, 4, 2)	4.14 td(4, 4, 2)	5.19 td(4, 4, 1)
8	1.98 dqd(8, 7, 4)	2.30 dqd(8, 7, 4)	2.40 dqd(8, 7, 4)
9	2.49 dd(11, 8)	2.71 dd(11, 8)	2.68 dd(11, 8)
10	1.05 d(7)	1.31 d(7)	1.19 d(7)
OMe	3.60 s	3.77 s	3.73 s
OAc			2.07 s
2′	6.95 d(8)		
3′	6.64 d(8)		
5′	6.64 d(8)		
6′	6.95 d(8)		
4′-OH	9.15 d		
7′	2.64 t(7)		
8′	3.55 t(7)		

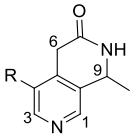
Table 2-19-13: ^{1}H NMR spectroscopic data of tetrahydropyridine monoterpenoid alkaloids **2-19-35**~**2-19-37**.

H	2-19-35	2-19-36	2-19-37	H	2-19-35	2-19-36	2-19-37
1	3.80 m	3.67 m	3.65 s	12	3.45 dd, 3.43 dd	2.06 t	
3	7.62 s	7.59 s	7.59 s	13		1.54 m	1.15 t(7)
5	3.29 m	3.25 m	2.78 m	14		0.89 t	
6	2.97 m			Ar-H	7.25~7.45 m		
7	5.71 s	5.72 m	5.73 m	OMe	3.66 s	3.66 s	3.67 s
9	3.36 m	3.51 m	3.53 m	1′	2.82 m	3.02 m	2.67 m
10	4.26 dd	4.21 dd	4.46 dd, 4.30 dd	2′	3.33 m	3.33 t	3.36 m

2.19.4 Pyridinolactam monoterpenoid alkaloids

Table 2-19-14: Cos, MFs, and TSs of pyridinolactam monoterpenoid alkaloids **2-19-38** and **2-19-39**.

No.	Compounds	MFs	Test solvents	References
2-19-38	jasminidin	$C_9H_{10}N_2O$	$CDCl_3$	[546]
2-19-39	jasminin	$C_{11}H_{12}N_2O_3$	$CDCl_3$	[546]



2-19-38 R = H
2-19-39 R = COOMe

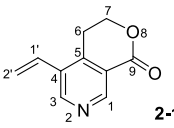
Table 2-19-15: ^{1}H NMR spectroscopic data of pyridinolactam monoterpenoid alkaloids **2-19-38** and **2-19-39**.

H	2-19-38	2-19-39	H	2-19-38	2-19-39
1	8.45 m	8.59 s	9	4.72 q(7)	4.78 q(7)
3	8.45 m	9.04 s	9-Me	1.58 d(6.7)	1.56 d(7)
4	7.07 d(4.9)		COOMe		3.95 s
6	3.58 s	4.05 m	NH	7.60 br s	7.70 br s

2.19.5 Pyridino-*δ*-lactone monoterpenoid alkaloids

Table 2-19-16: Co, MF, and TS of pyridino-*δ*-lactone monoterpenoid alkaloid **2-19-40**.

No.	Compound	MF	Test solvent	Reference
2-19-40	gentianine	$C_{10}H_9NO_2$	$CDCl_3$	[547]



2-19-40

Table 2-19-17: ^{1}H NMR spectroscopic data of pyridino-*δ*-lactone monoterpenoid alkaloid **2-19-40**.

H	2-19-40	H	2-19-40
1	8.89 s	7	4.60 t(6.0)
3	9.22 s	1′	5.63 d(11.2)
6	3.12 t(6.0)	2′	5.85 d(17.5), 6.82 dd(11.2, 17.5)

Bibliography

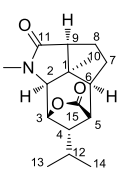
[537] Skaltsounis A-L, Michel S, Tilleqnin F, et al. Helv Chim Acta, 1985, 68: 1679.
[538] Ranarivelo Y, Hotellier F, Skaltsounis A-L, et al. Heterocycles, 1990, 31: 1727.
[539] Lopez JL, Pusset J. J Nat Prod, 1988, 51: 829.
[540] Ji TF, Feng XZ. Chin Tradit Herb Drugs, 2002, 33: 967.
[541] Cid MM, Pombo-Villar E. Helv Chim Acta, 1993, 76: 1591.
[542] Chi YM, Yan WM, Chen DC, et al. Phytochemistry, 1992, 31: 2930.
[543] Saad HEA, Anton R, Quirion JC, et al. Tetrahedron Lett, 1988, 29: 615.
[544] Michel S, Skaltsounis AL, Tillequin F, et al. J Nat Prod, 1985, 48: 86.
[545] Shao RF, Sun K, Zou C, et al. J Yunnan Univ Chin Med, 2009, 32: 36.
[546] Ripperger H. Phytochemistry, 1978, 17: 1069.
[547] Yang J, Ma J, Zhou DX, et al. Chin Tradit Herb Drugs, 2006, 37: 187.

2.20 Sesquiterpenoid alkaloids

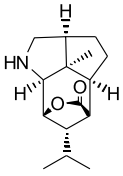
2.20.1 Dendrobine-type sesquiterpenoid alkaloids

Table 2-20-1: Cos, MFs, and TSs of dendrobine-type sesquiterpenoid alkaloids **2-20-1**~**2-20-5**.

No.	Compounds	MFs	Test solvents	References
2-20-1	mubironine A	$C_{16}H_{23}NO_3$	$CDCl_3$-CD_3OD(9:1)	[548]
2-20-2	mubironine B	$C_{15}H_{23}NO_2$	$CDCl_3$-CD_3OD(9:1)	[548]
2-20-3	mubironine C	$C_{17}H_{29}NO_3$	$CDCl_3$-CD_3OD(9:1)	[548]
2-20-4	dendrobine	$C_{16}H_{25}NO_2$	$CDCl_3$	[549]
2-20-5	3-hydroxy-2-oxodendrobine	$C_{16}H_{23}NO_4$	$CDCl_3$	[549]



2-20-1



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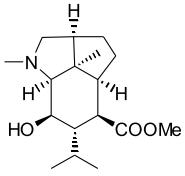

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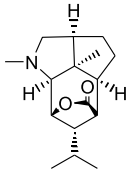
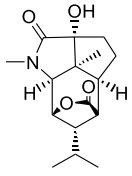


2-20-4



2-20-5

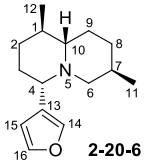
Table 2-20-2: ^{1}H NMR spectroscopic data of dendrobine-type sesquiterpenoid alkaloids **2-20-1**~**2-20-5**.

H	2-20-1	2-20-2	2-20-3	2-20-4	2-20-5
2	3.24 d(3.4)	3.47 s	3.35 s	2.68 d(3)	3.18 d(3.5)
3	4.72 dd(5.4, 3.4)	4.77 d(2.4)	3.92 m	4.83 dd(3, 5.5)	4.73 dd(3, 5.5)
4	2.28 m	2.10 m	2.17 m	2.11	2.25
5	2.52 m	2.45 t(5.0)	2.50 m	2.45 dd(4, 9.5)	2.53 dd(4.5, 5.5)
6	2.17 t(7.0)	2.00 m	1.88 m	2.01	2.27
7	2.06 m, 2.01 m	2.15 m, 2.03 m	1.97 m, 1.43 m	2.06, 2.15	1.84, 2.05
8	1.76 m, 2.10 m	1.90 m, 1.42 m	1.70 m, 1.64 m	1.54, 1.85	2.02, 2.25
9	2.55 m	2.49 m	2.49 m	2.35 quint(9)	
10	1.43 s	1.32 s	1.33 s	1.38 s	1.37 s
11		3.15 t(10.0)	3.45 m	3.22 t(9)	
		2.93 t(10.0)	3.40 m	2.70 t(9)	
12	1.88 m	1.58 m	1.78 m	1.75 d sept (6, 6.5)	2.07
13	1.01 d(6.5)	0.87 d(6.4)	0.87 d(7.0)	0.95 d(6.5)	1.03 d(6.5)
14	1.03 d(6.5)	0.89 d(6.4)	1.01 d(7.0)	0.96 d(6.5)	1.04 d(6.5)
NMe	2.87 s		3.03 s	2.50 s	2.88 s
OMe			3.63 s		
OH					3.43 br s

2.20.2 Nupharidine-type sesquiterpenoid alkaloids

Table 2-20-3: Cos, MFs, and TSs of nupharidine-type sesquiterpenoid alkaloids **2-20-6**~**2-20-9**.

No.	Compounds	MFs	Test solvents	References
2-20-6	(–)-7-*epi*-deoxynupharidine	$C_{15}H_{23}NO$	$CDCl_3$	[550]
2-20-7	(–)-castoramine	$C_{15}H_{23}NO_2$	$CDCl_3$	[550]
2-20-8	(–)-nupharolutine	$C_{15}H_{23}NO_2$	$CDCl_3$	[550]
2-20-9	(–)-nupharmine	$C_{15}H_{23}NO_2$	$CDCl_3$	[550]



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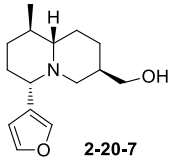
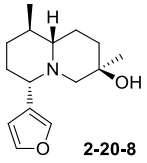
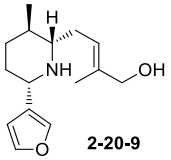


2-20-7



2-20-8



2-20-9

Table 2-20-4: ^{1}H NMR spectroscopic data of nupharidine-type sesquiterpenoid alkaloids **2-20-6**~**2-20-9**.

H	2-20-6	2-20-7	2-20-8	2-20-9
4ax	2.78~2.95 m	2.95 dd(6.4, 6.9)	3.08~3.00 m	–
6eq	2.78~2.95 m	3.07 dd(11.3, 2.1)	2.68 dd(11.0, 2.3)	1.69 s
8	–	–	–	5.43 br t
10	–	–	–	3.57 dd(11.5, 2.6)
11	0.73 d(6.0)	3.75 d(2.4)	1.21 s	3.99 s
12	0.91 d(5.8)	0.92 d(5.9)	0.90 d(6.7)	0.92 d(6.4)
14,16	7.35 m, 7.31 br s	7.35 m	7.33 m	7.34 m
15	6.45 br s	6.45 br s	6.35 br s	6.39 br s
Other	1.1~2.1 (12H)	1.1~2.5 (12H)	1.0~2.0 (10H)	1.0~2.5 (8H)

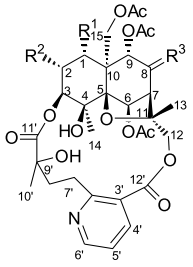
2.20.3 Dihydro-*β*-agarofuran-type sesquiterpenoid alkaloids

Table 2-20-5: Cos, MFs, and TSs of dihydro-*β*-agarofuran-type sesquiterpenoid alkaloids **2-20-10**~**2-20-21**.

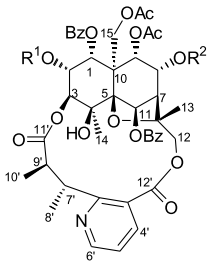
No.	Compounds	MFs	Test solvents	References
2-20-10	chiapenine ES-I	$C_{48}H_{51}NO_{19}$	$CDCl_3$	[551]
2-20-11	chiapenine ES-II	$C_{46}H_{47}NO_{18}$	$CDCl_3$	[551]
2-20-12	chiapenine ES-III	$C_{39}H_{43}NO_{17}$	$CDCl_3$	[551]

Table 2-20-5 (continued)

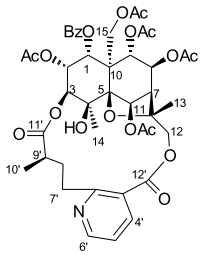
No.	Compounds	MFs	Test solvents	References
2-20-13	chiapenine ES-IV	$C_{34}H_{41}NO_{17}$	$CDCl_3$	[551]
2-20-14	euoverrine A	$C_{48}H_{51}NO_{18}$	$CDCl_3$	[552]
2-20-15	euoverrine B	$C_{43}H_{49}NO_{18}$	$CDCl_3$	[552]
2-20-16	euophelline	$C_{47}H_{51}NO_{17}$	$CDCl_3$	[552]
2-20-17	euojaponine C	$C_{46}H_{49}NO_{17}$	$CDCl_3$	[552]
2-20-18	euonymine	$C_{38}H_{42}NO_{18}$	$CDCl_3$	[553]
2-20-19	wilfordsine	$C_{43}H_{49}NO_{19}$	$CDCl_3$	[553]
2-20-20	euonine	$C_{43}H_{49}NO_{18}$	$CDCl_3$	[554]
2-20-21	wilfordcoine	$C_{39}H_{45}NO_{19}$	$CDCl_3$	[554]



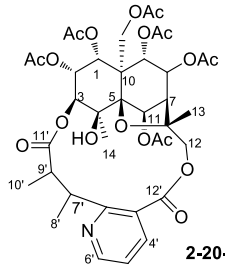
	R^1	R^2	R^3
2-20-10	OBz	OBz	α-OAc, β-H
2-20-11	OBz	OBz	O
2-20-12	OBz	OH	O
2-20-13	OAc	OH	O



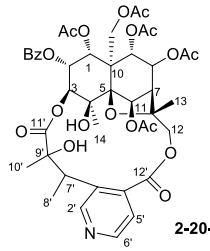
	R^1	R^2
2-20-14	Ac	Ac
2-20-16	H	Pro
2-20-17	H	Ac



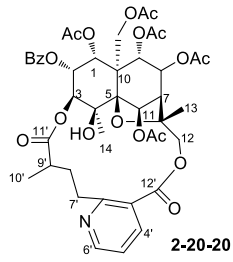
2-20-15



2-20-18



2-20-19



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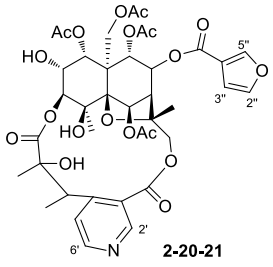


2-20-21

Table 2-20-6: ^{1}H NMR spectroscopic data of dihydro-*β*-agarofuran-type sesquiterpenoid alkaloids **2-20-10**~**2-20-13**.

H	2-20-10	2-20-11	2-20-12	2-20-13
1	6.09 d(3.6)	6.21 d(3.3)	5.94 d(3.0)	5.62 d(2.7)
2	5.53 (ov)	5.49 dd(3.3, 2.3)	4.09 br s	3.99 br s
3	5.16 d(2.7)	5.25 d(2.3)	5.14 d(3.3)	5.08 d(3.3)
6	6.92 s	6.79 s	6.74 s	6.77 s
7	2.41 d(3.8)	3.07 s	3.04 s	3.04 s
8	5.53 (ov)			
9	5.46 d(5.8)	5.70 s	5.62 s	5.55 s
12	3.75 d(12.0)	3.78 d(12.0)	3.76 d(11.3)	3.75 d(11.9)
	5.87 d(12.0)	5.95 d(12.0)	5.93 d(11.3)	5.97 d(11.9)
13	1.69 s	1.61 s	1.56 s	1.53 s
14	1.74 s	1.75 s	1.74 s	1.67 s
15	4.61 d(13.2)	4.96 d(13.0)	4.69 d(13.4)	4.99 d(13.3)
	5.72 d(13.2)	5.11 d(13.0)	5.26 d(13.4)	4.62 d(13.3)
4′	8.16 dd(7.8, 1.6)	8.15 dd(8.0, 1.6)	8.15 dd(7.9, 1.9)	8.31 dd(7.9, 1.8)
5′	7.23 dd(7.8, 4.5)	7.23 dd(8.0, 4.8)	7.25 dd(7.9, 4.8)	7.38 dd(7.9, 4.7)
6′	8.71 dd(4.5, 1.6)	8.72 dd(4.8, 1.6)	8.70 dd(4.8, 1.9)	8.74 dd(4.7, 1.8)
7′	4.11 m, 2.90 m	4.08 m, 2.88 m	4.06 m, 2.90 m	4.09 m, 2.94 m
8′	2.55 m, 2.28 m	2.50 m, 2.26 m	2.45 m, 2.25 m	2.54 m, 2.23 m
10′	1.57 s	1.56 s	1.47 s	1.43 s
1-OAc				2.01 s
6-OAc	2.22 s	2.24 s	2.02 s	2.22 s
8-OAc	2.18 s			
9-OAc	1.39 s	1.53 s	1.56 s	2.13 s
15-OAc	2.25 s	2.08 s	2.23 s	2.03 s

Table 2-20-7: ^{1}H NMR spectroscopic data of dihydro-*β*-agarofuran-type sesquiterpenoid alkaloids **2-20-14**~**2-20-17**.

H	2-20-14	2-20-15	2-20-16	2-20-17
1	5.80 d(3.8)	5.95 d(3.4)	5.79 d(3.9)	5.78 d(3.9)
2	5.45 dd(3.8, 2.5)	5.29 dd(3.4, 2.5)	4.16 dd(3.9, 2.5)	4.16 dd(3.8, 2.5)
3	4.86 d(2.5)	5.04 d(2.5)	4.85 d(2.5)	4.86 d(2.5)
6	7.22 s	6.60 s	7.21 s	7.22 s
7	2.53 d(4.2)	2.48 d(3.3)	2.53 d(4.2)	2.53 d(4.2)
8	5.57 dd(4.2, 6.0)	5.65 dd(3.3, 9.6)	5.59 dd(4.2, 6.0)	5.57 dd(4.2, 5.9)
9	5.46 d(6.0)	5.80 d(9.6)	5.45 d(6.0)	5.46 d(5.9)
12	3.61 d(11.4)	3.86 d(11.5)	3.61 d(11.4)	3.61 d(11.4)
	6.04 d(11.4)	5.67 d(11.5)	6.06 d(11.4)	6.05 d(11.4)
13	1.73 s	1.74 s	1.73 s	1.72 s
14	1.61 s	1.62 s	1.63 s	1.63 s
15	4.90 d(13.8)	4.83 d(12.4)	4.88 d(13.7)	4.90 d(13.8)
	5.53 d(13.8)	5.01 d(12.4)	5.54 d(13.7)	5.53 d(13.8)

Table 2-20-7 (continued)

H	2-20-14	2-20-15	2-20-16	2-20-17
4′	8.04 dd(7.8, 1.8)	8.29 dd(7.8, 1.8)	8.04 dd(7.8, 1.8)	8.04 dd(7.8, 1.8)
5′	7.62 m	7.28 m	7.56 m	7.56 m
6′	8.70 dd(4.8, 1.8)	8.75 dd(4.8, 1.8)	8.71 dd(4.8, 1.8)	8.70 dd(4.8, 1.8)
7′	4.75 m	3.81 m, 3.01 m	4.74 m	4.75 m
8′	1.42 d(7.2)	2.18 m, 2.26 m	1.42 d(7.2)	1.41 d(7.2)
9′	2.58 m	2.56 m	2.58 m	2.58 m
10′	1.17 d(7.2)	1.27 d(6.5)	1.16 d(7.2)	1.17 d(7.2)
Ac	1.42 s, 2.16 s	1.40 s, 1.96 s, 2.15 s	1.41 s, 2.34 s	1.42 s, 2.19 s
	2.19 s, 2.35 s	2.20 s, 2.33 s		2.35 s
Bz	8.34 dd, 7.96 dd	8.34 dd, 7.96 dd	8.34 dd, 7.95 dd	8.34 dd, 7.96 dd
	7.57 m, 7.27 m	7.57 m, 7.27 m	7.59 m, 7.26 m	7.57 m, 7.26 m
	7.52 dd, 7.42 dd	7.52 dd, 7.42 dd	7.54 dd, 7.43 dd	7.53 dd, 7.42 dd
Pro			1.18 t(7.0)	
			2.45 q(7.0)	

Table 2-20-8: ^{1}H NMR spectroscopic data of dihydro-β-agarofuran-type sesquiterpenoid alkaloids **2-20-18**~**2-20-21**.

H	2-20-18	2-20-19	2-20-20	2-20-21
1	5.50 d(3.4)	5.78 d(3.3)	5.60 d(4)	5.58 d(4)
2	3.97 t(3.0)	5.45 t(3.0)	5.18 t(3.4)	5.20 t(3.3)
3	5.01 d(2.6)	5.05 d(2.6)	4.92 d(3.5)	4.95 d(3.6)
6	6.91 s	6.88 s	6.90 s	7.18 s
7	2.40 d(3.8)	2.40 d(4.0)	2.35 d(3.8)	2.82 d(3.8)
8	5.50 dd(4.0, 5.9)	5.55 dd(4.0, 6.0)	5.54 dd(4.6)	5.68 dd(4.6)
9	5.39 d(5.9)	5.40 d(6.0)	5.34 d(5.8)	5.48 d(5.9)
12	3.80 ABq(11.9)	3.76 ABq(12)	3.76 ABq(11.7)	5.01 ABq(12.0)
	5.75 ABq(11.9)	5.80 ABq(12)	5.74 ABq(11.7)	5.05 ABq(12.0)
13	1.62 s	1.76 s	1.56 s	1.61 s
14	1.52 s	1.56 s	1.65 s	1.65 s
15	5.42 ABq(13.5)	5.56 ABq(13.2)	4.45 ABq(13.5)	4.24 ABq(11.7)
	4.60 ABq(13.5)	4.40 ABq(13.2)	5.23 ABq(13.5)	5.20 ABq(13.4)
2′		9.01 s		8.98 s
3′				
4′	8.10 dd(7.9, 1.8)		8.33 dd(7.9, 4.6)	
5′	7.20 dd(7.9, 4.6)	7.80 d(5.5)	7.41 dd(7.9, 1.8)	7.82 d(5.5)
6′	8.76 dd(4.6, 1.8)	8.70 d(5.5)	8.72 dd(4.6, 1.8)	8.69 d(5.5)
7′	4.74 q(7.0)	4.25 q(7.3)	4.23 m	4.23 q(7.3)
8′	1.15 d(7.0)	1.18 s	2.35 m, 2.47 m	1.20 d(7.3)
9′	2.42 d(7.1)		2.48 m	
10′	1.36 d(7.0)	1.38 s	1.18 d(6.8)	1.36 s
4-OH	5.08 s	5.08 s	5.06 s	3.40 s
9′-OH		3.08 s		3.43 s

Table 2-20-8 (continued)

H	2-20-18	2-20-19	2-20-20	2-20-21
Ac	1.68, 1.83, 1.98, 2.15 2.20, 2.32	1.82, 1.96, 2.08, 2.15 2.20	1.68, 1.82, 1.98, 2.13 2.21, 2.30	1.78, 1.90, 1.93, 2.15 2.28
Bz		8.07 d(7.3), 7.63 t(7.3) 7.51 t(7.3)		
2″				7.50 d(5.5)
3″				6.99 d(1.8)
5″				8.53 s

Bibliography

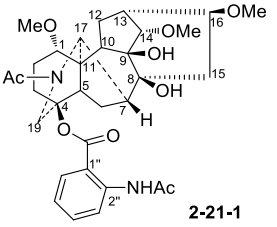
[548] Morita H, Fujiwara M, Yoshida N, et al. Tetrahedron, 2000, 56: 5801.
[549] Wang XK, Zhao TF, Che CT. J Nat Prod, 1985, 48: 796.
[550] Miyazawa M, Yoshio K, Ishikawa Y, et al. J Agric Food Chem, 1998, 46: 1059.
[551] Núñez MJ, Guadaño A, Jiménez IA, et al. J Nat Prod, 2004, 67: 14.
[552] Zhu JB, Wang MG, Wu WJ, et al. Phytochemistry, 2002, 61: 699.
[553] Lin S, Li YC, Sakurai N, et al. Acta Pharm Sin, 1995, 30: 513.
[554] Lin S, Li YC, Sakurai N, et al. Acta Pharm Sin, 2001, 36: 116.

2.21 Diterpenoid alkaloids[1]

2.21.1 A1-type diterpenoid alkaloids

Table 2-21-1: Cos, MFs, and TSs of A1-type diterpenoid alkaloids **2-21-1**~**2-21-14**.

No.	Compounds	MFs	Test solvents	References
2-21-1	sinaconitine B	$C_{32}H_{42}N_2O_9$	$CDCl_3$	[555]
2-21-2	acotoxicine	$C_{22}H_{35}NO_5$	$CDCl_3$	[556]
2-21-3	kiridine	$C_{22}H_{31}NO_6$	$CDCl_3$	[557]
2-21-4	delphicrispuline	$C_{30}H_{42}N_2O_6$	$CDCl_3$	[558]
2-21-5	sinomontanine B	$C_{31}H_{42}N_2O_8$	$CDCl_3$	[559]
2-21-6	(+)-lappaconitine	$C_{32}H_{44}N_2O_8$	$CDCl_3$	[560]
2-21-7	(+)-*N*-deacetyllappaconitine	$C_{30}H_{42}N_2O_7$	$CDCl_3$	[560]
2-21-8	*N*-acetylsepaconitine	$C_{32}H_{44}N_2O_9$	$CDCl_3$	[561]
2-21-9	8-acetyldolaconine	$C_{26}H_{39}NO_6$	$CDCl_3$	[562]
2-21-10	piepunendine B	$C_{30}H_{43}NO_5$	$CDCl_3$	[563]
2-21-11	piepunendine A	$C_{20}H_{29}NO_5$	$CDCl_3$	[563]
2-21-12	sinomontanine A	$C_{30}H_{40}N_2O_8$	$CDCl_3$	[559]
2-21-13	puberanidine	$C_{30}H_{42}N_2O_7$	$CDCl_3$	[564]
2-21-14	8,9-methylenedioxylappaconitine	$C_{33}H_{44}N_2O_8$	$CDCl_3$, C_6D_6	[565]



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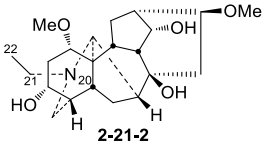
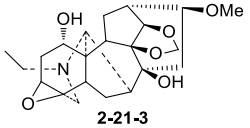


2-21-2



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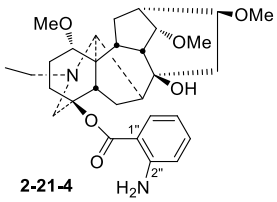

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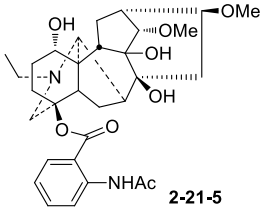

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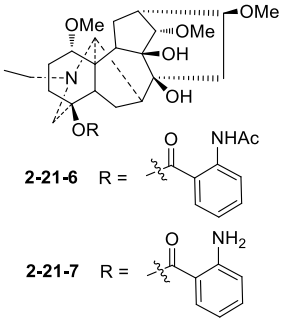
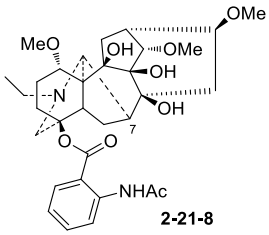


2-21-6 R =

2-21-7 R =

1 The classification of diterpenoid alkaloids is with reference to the literature 'Wang FP, Chen QH, Liu XY. Nat Prod Rep, 1999, 16: 619'.



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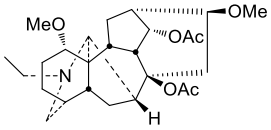
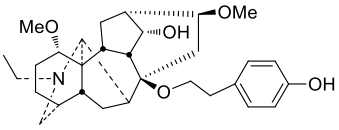


2-21-9



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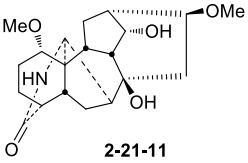
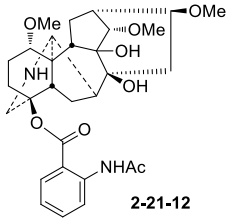
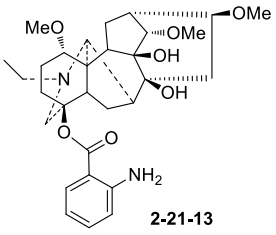


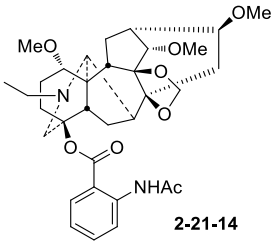
2-21-11



2-21-12



2-21-13



2-21-14

Table 2-21-2: ^{1}H NMR spectroscopic data of A1-type diterpenoid alkaloids **2-21-1**~**2-21-4**.

H	2-21-1	2-21-2	2-21-3	2-21-4
1	3.24 dd(11.8, 6.4)	3.15 dt (9.9, 6.9)	4.00 br t	3.20 d(9)
2	*α* 1.50 m	2.22 m	*α* 2.26 m	*α* 1.50 m
	β 2.29 m		*β* 1.34 ddd(14.3, 6.4, 2.4)	*β* 1.65 m
3	*α* 2.59 m, *β* 1.88 m	3.70 br d(10.7)	3.09 t(6.4, 6.4)	1.62 m
4		1.74 br s		
5	2.59 m	1.79 m	2.34 d(9.0)	1.75 d(8)
6	*α* 1.88 m	*α* 2.21 m	*α* 2.15 m	1.60 m
	β 2.81 m	*β* 1.41 dd(14.3, 7.6)	*β* 1.98 dd(14.9, 9.0)	1.90 m
7	1.99 m	2.20 m	1.56 d(6.3)	2.45 d(7.5)
9		2.28 m		2.0 dd(4, 10)
10	2.21 m	1.67 dd(13.8, 8.3)	2.55~2.65 m	1.85 m
12	*α* 2.43 m	1.83 m	*α* 1.72 dd(12.2, 4.4)	1.60 m
	β 1.99 m		*β* 2.55~2.65 m	2.05 dd(4,8)
13	2.43 m	2.34 br s	2.15 m	2.30 m
14	3.48 d(4.5)	4.15 t(4.0)	4.03 br s	3.45 t(4.5)
15	*α* 2.48 dd(14.1, 7.3)	*α* 2.43 dd(17.1, 8.6)	1.88 dd(15.1, 7.8)	2.05 m
15	*β* 2.15 dd(14.1, 8.5)	*β* 2.07 d(17.1)	2.20 m	
16	3.29 m	3.40 d(8.6)	3.47 t(7.8, 7.8)	3.17 d(8)

Table 2-21-2 (continued)

H	2-21-1	2-21-2	2-21-3	2-21-4
17	3.93 br s	3.08 s	2.88 br s	3.02 s
19	*α* 4.92 d(14.5)	2.99 d(12.0)	2.99 d(10.0)	2.65 d(11)
	β 3.16 d(14.5)	2.30 m	2.37 d(10.0)	3.60 d(11)
NAc	2.18 s			
21		2.48 br m	2.55~2.65 m	2.45 m, 2.74 m
22		1.08 t(6.9)	1.11 t(7.3)	1.07 t(7)
1-OMe	3.26 s	3.27 s		3.32 s
14-OMe	3.43 s			3.30 s
16-OMe	3.31 s	3.34 s	3.32 s	3.40 s
NHAc	2.23 s			
3″	8.68 d(8.5)			6.67 d(8)
4″	7.50 dd(8.5, 7.7)			7.28 dd(1.5, 8)
5″	7.02 dd(8.0, 7.7)			6.63 dd(1.5, 8)
6″	7.91 d(8.0)			7.80 dd(1.5, 8)
OCH_2O			5.28 s	
NH	11.0 br s			5.72 br s

Table 2-21-3: ^{1}H NMR spectroscopic data of A1-type diterpenoid alkaloids **2-21-5**~**2-21-8**.

H	2-21-5	2-21-6	2-21-7	2-21-8
14*β*	–	–		3.78 d(4.5)
22	1.14 t(7.1)	1.11 t(7)	1.13 t(6.5)	1.13 t(7)
OMe	3.33(3.32) s	3.27 s(6H)	3.28 s, 3.32 s, 3.42 s	3.32 s, 3.33 s, 3.43 s
	3.36 s	3.38 s(3H)		
Ac	2.25(2.23) s	2.18 s		2.24 s①
3′	7.92 d(7.9)			7.92 dd(1.3, 8)
4′	7.03 t(7.2)			7.30 dt(1.3, 8)
5′				7.49 dt(1.3, 8)
6′	7.69 d(8.3)			8.68 dd(1.3, 8)
Ar-H		6.93 t(8), 7.35 t(8)	6.49~6.70 m(2H)	
		7.85 d(8), 8.58 d(8)	7.27 t(8), 7.81 d(8)	
NH	11.07 s	10.90 br s		11.05 br s

① Typographic error exists in the literature, NAc being printed as OAc.

Table 2-21-4: ^{1}H NMR spectroscopic data of of A1-type diterpenoid alkaloids **2-21-9**~**2-21-11**.

H	2-21-9	2-21-10	2-21-11
1*α*		3.08 m	
1*β*	3.06 dd(10, 6.5)		3.30 m
2*α*	1.85 m	1.80 m	1.45 m
2*β*	1.16 m	2.20 m	2.09 m
3*α*	2.73 dd(15, 9)	1.92 m	1.36 m

Table 2-21-4 (continued)

H	2-21-9	2-21-10	2-21-11
3*β*	2.04 dd(15, 7)	2.10 m	2.21 m
4	1.55 br s	2.46 m	2.52 br s
5	1.85 br s	1.82 m	1.83 m
6*α*	1.69 d(12)	1.13 m	1.82 m
6*β*	1.38 dd(12)	1.85 m	2.20 m
7	1.68 br s	2.19 m	2.08 m
9	2.54 br s	2.28 t(6.0)	2.36 m
10	2.29 br s	1.69 m	2.11 m
12*α*	2.13 d(9.5)	2.17 m	1.88 m
12*β*	1.93 d(9.5)	1.92 m	1.67 m
13	3.20 br s	2.06 m	2.45 m
14*β*	4.74 t(4.6)	3.97 t(4.4)	4.17 dd(4.8, 4.8)
15*α*	1.84 m	1.70 m	2.36 m
15*β*	2.30 m	1.64 m	2.12 m
16	3.19 m	3.30 m	3.46 m
17	2.77 s	2.85 br s	3.64 d(4.4)
19	*α* 2.44 dd(11.4, 7)	2.43 ABq(11.2)	
	β 2.49 dd(11.4, 5)	2.62 ABq(11.2)	
21	2.35 q(7.0)	2.40 m, 2.46 m	
22	1.00 t(7.0)	1.05 t(7.2)	
OMe	3.22 s,3.26 s		
1-OMe		3.26 s	3.28 s
16-OMe		3.25 s	3.36 s
3″, 5″		6.78 AA′BB′(11.2)	
2″, 6″		7.04 AA′BB′ (11.2)	
7″		2.68 m,2.70 m	
8″		3.47 m, 3.51 m	
OAc	1.88 s, 1.98 s		

Table 2-21-5: ^{1}H NMR spectroscopic data of A1-type diterpenoid alkaloids **2-21-12**∼**2-21-14**.

H	2-21-12	2-21-13	2-21-14 ($CDCl_3$)	2-21-14 (C_6D_6)
1	–	*β* 3.20 dd(9, 6)	*β* 3.17 dd(6.8, 9.9)	*β* 2.82 dd(5.4, 9.9)
2*α*	–	–	2.24 m	2.34 m
2*β*	–	–	2.13 m	1.93 m(5.4, 13.0, 2.5, 5.0)
3*α*	–	–	2.56 m	2.69 m
3*β*	–	–	1.84 br t(13.3, 4.9, 11.3)	1.75 m
5	–	–	2.30 m	2.45 d(7.8)
6*α*	–	–	2.60 m	1.77 m
6*β*	–	–	1.99 dd(14.9, 7.6)	2.36 m
7	–	–	2.17 m	2.32 m
10	–	–	2.27 m	2.11 dd(4.3, 12.0)
12*α*	–	–	1.62 m	2.69 m(4.3, 14.9)
12*β*	–	–	1.98 dd(15.7, 8.4)	1.69 m

Table 2-21-5 (continued)

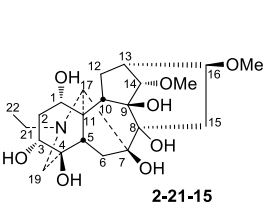
H	2-21-12	2-21-13	2-21-14 ($CDCl_3$)	2-21-14 (C_6D_6)
13	–	–	2.29 m	2.24 dd(4.5, 7.5)
14	–	β 3.46 d(5)	3.56 d(4.5)	3.55 d(4.5)
15	–	–	α 2.21 m	α 2.29 m(8.3)
			β 2.40 dd(13.6, 8.7)	β 2.90 dd(13.7, 8.8)
16	–	α 3.20 dd(9, 6)	3.32 m	3.33 br t(8.8, 8.3)
17	–	3.00 s	2.98 s($W_{1/2}$ = 4.2)	3.08 s
19	–	3.62 d(11)	α 3.50 d(11.2)	α 3.64 d(11.2)
		2.55 d(11)	β 2.50 d(11.2)	β 2.56 d(11.2)
20	11.0 br s			
21		–	2.50 m	2.32 m
22		1.12 t(7)	1.10 t(7.3)	1.02 t(7.2)
OMe	3.27 s(6H)	3.30, 3.32, 3.42	3.28 s(1-OMe)	2.99 s(1-OMe)
	3.38 s(3H)		3.33 s(14-OMe)	3.27 s(14-OMe)
			3.30 s(16-OMe)	3.04 s(16-OMe)
OCH_2O			α 5.46 dd(2.2)	α 5.78 d(2.1)
			β 5.12 dd(2.2)	β 5.19 d(2.1)
3′			8.65 dd(7.2, 1.4)	9.27 dd(8.5, 1.1)
4′			7.49 ddd(7.7, 7.2, 1.7)	7.22 dt(7.2, 8.5, 1.7)①
5′			7.02 ddd(8.2, 7.7, 1.4)	6.75 dt(7.2, 8.0, 1.1)①
6′			7.89 dd(8.2, 1.7)	8.05 dd(8.0, 1.7)
Ar-H	6.90~8.70 m(4H)	6.65 d(7), 7.25 t(7)		
		6.62 t(7), 7.78 d(7)		
NH		5.68 br	11.04 s	11.17 br s
Ac	2.20 s		2.21 s	1.77 s

① Typographic error exists in the literature, giving *J* values more.

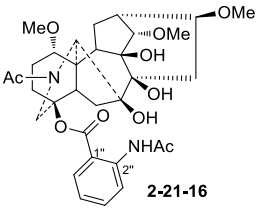
2.21.2 A2-type diterpenoid alkaloids

Table 2-21-6: Cos, MFs, and TSs of A2-type diterpenoid alkaloids **2-21-15**~**2-21-28**.

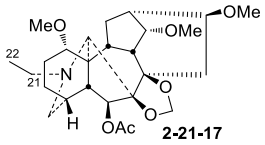
No.	Compounds	MFs	Test solvents	References
2-21-15	sinomontanine D	$C_{22}H_{35}NO_8$	$CDCl_3$	[566]
2-21-16	sinaconitine A	$C_{32}H_{42}N_2O_{10}$	$CDCl_3$	[555]
2-21-17	anthriscifolcine A	$C_{26}H_{39}NO_7$	$CDCl_3$	[567]
2-21-18	anthriscifolcine C	$C_{25}H_{37}NO_8$	$CDCl_3$	[567]
2-21-19	anthriscifolcine D	$C_{26}H_{39}NO_8$	$CDCl_3$	[567]
2-21-20	linearilin	$C_{24}H_{39}NO_8$	$CDCl_3$	[568]
2-21-21	lamarckinine	$C_{22}H_{33}NO_6$	$CDCl_3$	[569]
2-21-22	hispaconitine	$C_{26}H_{41}NO_8$	C_5D_5N	[570]
2-21-23	14-*O*-demethyldelboxine	$C_{23}H_{35}NO_7$	$CDCl_3$	[571]
2-21-24	puberanine	$C_{32}H_{44}N_2O_9$	$CDCl_3$	[564]
2-21-25	leucostine	$C_{23}H_{35}NO_6$	$CDCl_3$	[572]
2-21-26	hohenackeridine	$C_{22}H_{31}NO_7$	$CDCl_3$	[573]
2-21-27	anthriscifolcine E	$C_{24}H_{37}NO_7$	$CDCl_3$	[567]
2-21-28	tiantaishansine	$C_{22}H_{33}NO_7$	–	[574]



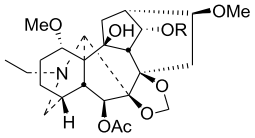
2-21-15



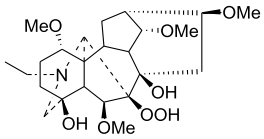
2-21-16



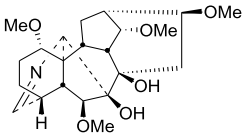
2-21-17



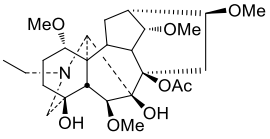
2-21-18 R = H
2-21-19 R = Me



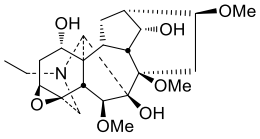
2-21-20



2-21-21



2-21-22



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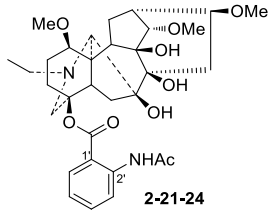
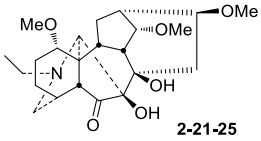
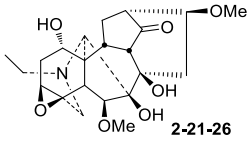


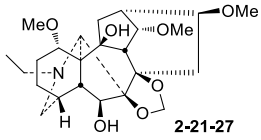
2-21-24



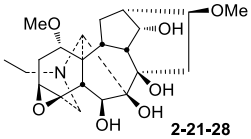
2-21-25



2-21-26



2-21-27



2-21-28

Table 2-21-7: ^{1}H NMR spectroscopic data of A2-type diterpenoid alkaloids **2-21-15**~**2-21-19**.

H	2-21-15	2-21-16	2-21-17	2-21-18	2-21-19
1	3.65 t(8.0)	3.10 dd(9.0, 8.2)	3.01 t(9.6)	3.62 t(8.0)	3.50 t(8.8)
2	α 2.36 m, β 1.98 m	α 1.55 m, β 2.25 m	2.08 m	α 2.06 m, β 2.12 m	α 2.06 m, β 2.12 m
3	4.05 t(3.5)	α 2.55 m, β 1.84 m	1.36 m, 1.74 m	α 1.35 m, β 1.78 m	α 1.37 m, β 1.80 m
4			2.31 t(5.6)	2.10 m	2.09 m
5	26.1 d(8.0)①	2.51 d(7.4)	1.46 s	1.83 m	1.84 m

Table 2-21-7 (continued)

H	2-21-15	2-21-16	2-21-17	2-21-18	2-21-19
6	*α* 3.15 dd(15.2, 8.0) *β* 1.68 dd(14.8, 7.5)	*α* 1.59 d(15.1) *β* 3.16 dd(7.4, 15.1)	5.21 s	5.33 s	5.27 s
9			2.10 m	3.32 m	3.29 m
10	2.09 dd (12.4, 4.4)	2.18 m	3.47 m		
12	*α* 2.42 dd(10.8, 4.4) *β* 2.05 m (ov)	*α* 2.47 m *β* 1.99 m	2.58 m 1.77 m	2.55 m	2.48 m
13	2.39 m (ov)	2.41 m	2.11 m	1.72 m	2.51 m
14	3.48 d(4.6)	3.50 d(4.3)	3.66 t(4.8)	4.64 dd(10.0, 4.8)	4.10 t(4.4)
15	*α* 2.99 (ov) *β* 1.74 dd(14.0, 8.0)	*α* 3.04 dd(8.4, 15.1) *β* 1.80 dd(7.8, 15.1)	2.44 m 1.80 m (ov)	*α* 1.83 m *β* 2.64 m	*α* 1.85 m *β* 2.69 m
16	3.28 d(8.0)	3.22 m	3.20 m	3.46 d(8.4)	3.18 d(8.0)
17	2.78 s	3.72 s	3.12 s	3.30 br s	3.05 br s
19	*α* 3.26 d(8.0) *β* 2.97 (ov)	*α* 4.88 d(14.4) *β* 3.33 d(14.4)	2.70 m 2.78 m	2.80 m	2.75 m
1-OMe		3.24 s	3.27 s	3.26 s	3.25 s
14-OMe	3.39 s	3.43 s	3.34 s		3.43 s
16-OMe	3.31 s	3.32 s	3.45 s	3.35 s	3.31 s
NHAc		11.0 br s(NH) 2.20 s(Ac)			
3″		8.66 d(8.5)			
4″		7.49 dd(7.5, 8.5)			
5″		7.01 dd(7.5, 7.8)			
6″		7.90 d(7.8)			
21	2.93 m, 3.03 m		2.73 m, 2.75 m	2.90 m	2.85 m
22	1.09 t(7.2)	2.15 s	1.03 t(7.2)	1.07 t(7.2)	1.04 t(7.2)
OAc			2.04 s	2.10 s	2.06 s
OCH_2O			4.91 s	4.99 s, 5.01 s	4.92 s, 4.94 s

① Typographic error exists in the original literature.

Table 2-21-8: ^{1}H NMR spectroscopic data of A2-type diterpenoid alkaloids **2-21-20**~**2-21-24**.

H	2-21-20	2-21-21	2-21-22	2-21-23	2-21-24
1	3.24 dd(10.5, 6.8)	–	2.92 dd(9.9, 7.3)	3.93 br s ($W_{1/2}$ = 7.0)	*α* 3.1 m
2	1.88 m, 2.34 m	–		*α* 2.22 ddd(14.0, 5.5, 2.4) *β* 1.26 ddd(14.0, 7.1, 2.4)	
3	1.92 m, 1.62 m	–	2.72 br d(9.5)	*α* 3.11 dd(7.1, 5.5)	
4		2.59 m($W_{1/2}$ = 12.5)			
5	2.40 s	–	2.04 br s	1.42 d(2.9)	
6	3.97 s	*α* 3.52 s	4.25 br s	4.24 s	
9	2.20 br t(6.3)	2.77 t(6.1)		3.32 t(5.9)	
10	1.85 m	–		2.10 m	

Table 2-21-8 (continued)

H	2-21-20	2-21-21	2-21-22	2-21-23	2-21-24
12	1.65 m, 1.95 m	–	2.57 dd(13.8, 4.4)	*α* 1.58 t(8.9) *β* 2.09 m	
13	2.28 m	2.38 dd(4.4, 6.8)	2.47 br t(4.4)	2.32 t(6.1)	
14	3.56 t(4.8)	*β* 3.65 t(4.4)	3.68 t(4.4)	*β* 4.05 ddd(4.6, 4.6, 3.4)	*β* 3.50 d(5)
15	2.54 m	*β* 2.87 dd(14.9, 8.7)		*α* 2.70 dd(16.2, 8.9) *β* 1.84 dd(16.2, 6.0)	
16	3.35 dd (10, 4.6)	–		*α* 3.42 m	*α* 3.10 m
17	2.96 s	3.82 br s($W_{1/2}$ = 6.7)		2.93 d(2.9)	2.85 s
19	2.80 d(12) 3.50 d(12)	7.66 br s($W_{1/2}$ = 6.5)	3.84 d(11.7)	*α* 2.53 d(9.6) *β* 3.46 d(9.6)	3.65 d(12) 3.20 d(12)
21	2.88 m 2.60 m			3.03 dq(13.8, 7.4) 3.02 dq(13.8, 7.4)	
22	1.05 t(4.5)		1.10 t(7.0)	1.10 t(7.4)	1.15 t(7)
OMe	3.27 s(1-OMe) 3.29 s(6-OMe) 3.29 s(14-OMe) 3.35 s(16-OMe)	3.17 s(1-OMe) 3.38 s(6-OMe) 3.43 s(14-OMe) 3.35 s(16-OMe)	3.20 s 3.24 s 3.32 s 3.47 s	3.48 s(6-OMe) 3.50 s(8-OMe) 3.42 s(16-OMe)	3.28 s 3.34 s 3.44 s
3′					7.90 d(7)
4′					7.50 t(7)
5′					7.05 t(7)
6′					8.70 d(7)
Ac			1.98 s(OAc)		2.25 s(NAc)
OH	3.57 br s, 3.94 br s		4.36 br s, 5.52 br s		
NH					11.0 s

Table 2-21-9: ^{1}H NMR spectroscopic data of A2-type diterpenoid alkaloids **2-21-25**~**2-21-28**.

H	2-21-25	2-21-26	2-21-27	2-21-28
1	3.14 dd(9.2, 6.2)	*β* 4.04 br t(2.5)		3.90 s
2	*α* 2.30~2.50 m①	*α*2.27 ddd(14.5, 6.0, 3.0)		1.26 m, 2.18 m
	β 2.07~2.28 m	*β*1.26 ddd(14.5, 7.5, 2.5)		
3	*α* 1.53 m	3.12 dd(7.0, 5.5)		3.05 m
	β 1.8~2.05 m			
4	2.07~2.28 m			
5	2.07~2.28 m	1.44 d(2.0)		1.56 s
6		*α* 4.41 s	4.28 s	4.82 s
7				
9	1.8~2.05 m	2.84 dd(8.0, 1.0)		3.12 m(5.6)
10	1.8~2.05 m	2.18 ddd(12.0, 8.0, 5.0)		2.12 m
12		*α* 1.90 dd(14.0, 5.0)		1.58 m, 2.12 m
		β 2.34 ddd(14.0, 11.5, 8.0)		
13	2.30~2.50 m	2.44 br d(6.5)		2.26 m
14*β*	3.68 t(4.7)		4.15 t(4.8)	4.03 m(4.4)
15	*α* 2.55~2.90 m	*α* 2.70 dd(16.0, 7.5)		1.97 m, 2.59 m
	β 1.67 dd(14.3, 5.4)	*β* 1.30 ddd(16.0, 9.0, 1.5)		

Table 2-21-9 (continued)

H	2-21-25	2-21-26	2-21-27	2-21-28
16	3.20~3.48 m	*α* 3.61 t(7.5)		3.39 s
17	3.20~3.48 m	3.15 d(3.0)		2.91 d(1.5)
19	2.55~2.90 m	*α* 2.55 dd(10.0, 0.5)		2.49 (ov)
		β 3.44 d(10.0)		3.36 (ov)
21	2.07~2.28 m(2H)	3.04 dq(14.0, 7.0)		2.98 m, 3.39 m
	2.55~2.90 m(2H)②	3.01 dq(14.0, 7.0)		
22	1.03 t(7.1)	1.09 t(7.5)	1.06 t(7.2)	1.07 t(7.2)
OMe	3.27 s(1-OMe)	3.38 s(6-OMe)	3.26 s	3.51 s(1-OMe)
	3.32 s(14-OMe)	3.32 s(16-OMe)	3.35 s	3.41 s(14-OMe)
	3.37 s(16-OMe)		3.45 s	
OCH_2O			5.07 s, 5.13 s	
7-OH	2.85 br s			
8-OH				

① Assigning this signal to H-1 may be a typographic error in the literature.
② Typographic error exists in the literature.

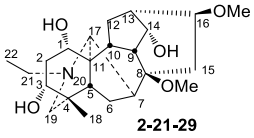
2.21.3 B1-type diterpenoid alkaloids

Table 2-21-10: Cos, MFs, and TSs of B1-type diterpenoid alkaloids **2-21-29**~**2-21-98**.

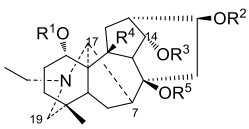
No.	Compounds	MFs	Test solvents	References
2-21-29	crispulidine	$C_{23}H_{37}NO_5$	$CDCl_3$	[558]
2-21-30	16*β*-hydroxycardiopetaline	$C_{21}H_{33}NO_4$	$CDCl_3$	[575]
2-21-31	8-ethoxysachaconitine	$C_{23}H_{41}NO_4$	$CDCl_3$	[575]
2-21-32	genicunin B	$C_{23}H_{37}NO_5$	$CDCl_3$	[575]
2-21-33	14-acetylgenicunin B	$C_{25}H_{39}NO_6$	$CDCl_3$	[575]
2-21-34	14-dehydrogenicunin B	$C_{23}H_{35}NO_5$	$CDCl_3$	[575]
2-21-35	8-*O*-methylcolumbianine	$C_{23}H_{37}NO_5$	$CDCl_3$	[576]
2-21-36	piepunensine A	$C_{22}H_{33}NO_6$	$CDCl_3$	[577]
2-21-37	18-acetylcammaconine	$C_{25}H_{39}NO_6$	$CDCl_3$	[577]
2-21-38	8-acetylcondelphine	$C_{27}H_{41}NO_7$	$CDCl_3$	[578]
2-21-39	*N*-deethyltalatisamine	$C_{22}H_{35}NO_5$	$CDCl_3$	[579]
2-21-40	liljestrandisine	$C_{23}H_{37}NO_5$	$CDCl_3$	[580]
2-21-41	leucanthumsine A	$C_{36}H_{49}NO_8$	$CDCl_3$	[581]
2-21-42	leucanthumsine D	$C_{23}H_{35}NO_6$	$CDCl_3$	[581]
2-21-43	leucanthumsine E	$C_{33}H_{45}NO_{10}$	$CDCl_3$	[581]
2-21-44	acotoxinine	$C_{33}H_{47}NO_9$	$CDCl_3$	[582]
2-21-45	aconitilearine	$C_{25}H_{41}NO_7$	$CDCl_3$	[583]
2-21-46	ouvrardiantine	$C_{35}H_{49}NO_{11}$	$CDCl_3$	[584]
2-21-47	royleinine	$C_{24}H_{39}NO_5$	$CDCl_3$	[585]
2-21-48	lasiansine	$C_{24}H_{39}NO_7$	$CDCl_3$	[587]
2-21-49	1-*epi*-deacetylaconitine	$C_{32}H_{45}NO_{10}$	$CDCl_3$	[586]
2-21-50	spicatine A	$C_{34}H_{49}NO_{10}$	$CDCl_3$	[588]
2-21-51	spicatine B	$C_{31}H_{41}NO_{10}$	$CDCl_3$	[588]

Table 2-21-10 (continued)

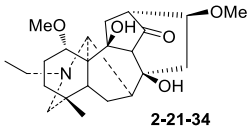
No.	Compounds	MFs	Test solvents	References
2-21-52	13-hydroxyfranchetine	$C_{31}H_{41}NO_7$	$CDCl_3$	[589]
2-21-53	10-dehydroxyflavaconitine	$C_{31}H_{41}NO_{10}$	$CDCl_3$	[589]
2-21-54	manshuritine	$C_{38}H_{47}NO_{11}$	$CDCl_3$	[590]
2-21-55	lasianine	$C_{25}H_{42}N_2O_8$	CD_3OD	[591]
2-21-56	francheline	$C_{24}H_{37}NO_6$	$CDCl_3$	[591]
2-21-57	pengshenine A	$C_{24}H_{37}NO_6$	–	[592]
2-21-58	pengshenine B	$C_{22}H_{33}NO_5$	$CDCl_3$	[592]
2-21-59	macrorhynine A	$C_{33}H_{43}NO_9$	$CDCl_3$	[593]
2-21-60	macrorhynine B	$C_{33}H_{43}NO_{10}$	$CDCl_3$	[593]
2-21-61	sinomontanitine A	$C_{35}H_{44}N_2O_9$	$CDCl_3$	[559]
2-21-62	8-*O*-ethylbenzoylaconine	$C_{39}H_{49}NO_{10}$	$CDCl_3$	[594]
2-21-63	10-hydroxyneoline	$C_{24}H_{39}NO_7$	$CDCl_3$	[595]
2-21-64	hokbusine A	$C_{32}H_{45}NO_{10}$	$CDCl_3$	[596]
2-21-65	linearilobin	$C_{37}H_{46}N_2O_9$	$CDCl_3$	[568]
2-21-66	karaconitine	$C_{34}H_{47}NO_{11}$	$CDCl_3$	[597]
2-21-67	raveyine	$C_{23}H_{37}NO_5$	$CDCl_3$	[598]
2-21-68	karakoline	$C_{22}H_{35}NO_4$	$CDCl_3$	[599]
2-21-69	aconitine-*N*-oxide	$C_{34}H_{47}NO_{12}$	$CDCl_3$	[600]
2-21-70	delphinine-*N*-oxide	$C_{33}H_{45}NO_{10}$	$CDCl_3$	[600]
2-21-71	delphisine-*N*-oxide	$C_{28}H_{43}NO_9$	$CDCl_3$	[600]
2-21-72	14-*O*-acetylneoline	$C_{26}H_{41}NO_7$	$CDCl_3$	[601]
2-21-73	brachyaconitine	$C_{38}H_{53}NO_{11}$	$CDCl_3$	[601]
2-21-74	8-*O*-acetyl-15α-hydroxyneoline	$C_{26}H_{41}NO_8$	$CDCl_3$	[601]
2-21-75	8-*O*-acetyl karasamine	$C_{25}H_{39}NO_5$	$CDCl_3$	[602]
2-21-76	12β-hydroxykarasamine 8-*O*-acetate	$C_{25}H_{39}NO_6$	$CDCl_3$	[602]
2-21-77	l-*epi*-12β-hydroxykarasamine	$C_{23}H_{37}NO_5$	$CDCl_3$	[602]
2-21-78	12β-hydroxykarasamine	$C_{23}H_{37}NO_5$	$CDCl_3$	[602]
2-21-79	peregrinine	$C_{24}H_{35}NO_6$	$CDCl_3$	[603]
2-21-80	lactam	$C_{26}H_{39}NO_7$	$CDCl_3$	[603]
2-21-81	14-*O*-benzoylperegrine	$C_{33}H_{45}NO_7$	$CDCl_3$	[604]
2-21-82	14-*O*-acetylperegrine	$C_{28}H_{43}NO_7$	$CDCl_3$	[604]
2-21-83	14-*O*-methylperegrine	$C_{27}H_{43}NO_6$	$CDCl_3$	[604]
2-21-84	munzianone	$C_{24}H_{37}NO_5$	$CDCl_3$	[604]
2-21-85	munzianine	$C_{23}H_{37}NO_5$	$CDCl_3$	[604]
2-21-86	10-hydroxyperegrine	$C_{26}H_{41}NO_7$	$CDCl_3$	[604]
2-21-87	bikhaconitine	$C_{36}H_{51}NO_{11}$	$CDCl_3$	[605]
2-21-88	pseudaconitine	$C_{36}H_{51}NO_{12}$	$CDCl_3$	[605]
2-21-89	veratroylbikhaconine	$C_{34}H_{49}NO_{10}$	$CDCl_3$	[605]
2-21-90	veratroylpseudaconine	$C_{34}H_{49}NO_{11}$	$CDCl_3$	[605]
2-21-91	aconitine	$C_{34}H_{47}NO_{11}$	$CDCl_3$	[605]
2-21-92	benzoylaconine	$C_{32}H_{45}NO_{10}$	$CDCl_3$	[605]
2-21-93	3-*O*-acetylbeiwutine	$C_{35}H_{47}NO_{13}$	$CDCl_3$	[606]
2-21-94	beiwutine	$C_{33}H_{45}NO_{12}$	$CDCl_3$	[606]
2-21-95	flavaconidine	$C_{32}H_{41}NO_{12}$	C_5D_5N	[607]
2-21-96	*N*-acetylflavaconitine	$C_{33}H_{43}NO_{12}$	C_5D_5N	[607]
2-21-97	flavaconijine	$C_{33}H_{43}NO_{11}$	C_5D_5N	[607]
2-21-98	flavaconitine	$C_{31}H_{41}NO_{11}$	C_5D_5N	[607]



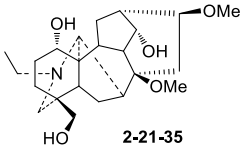
2-21-29



	R^1	R^2	R^3	R^4	R^5
2-21-30	H	H	H	H	H
2-21-31	Me	Me	H	H	Et
2-21-32	Me	Me	H	OH	H
2-21-33	Me	Me	Ac	OH	H



2-21-34



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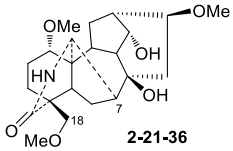

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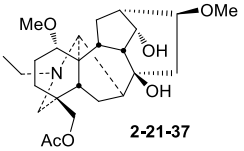
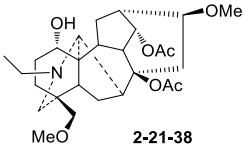
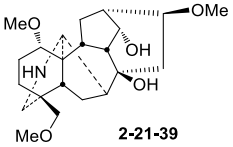


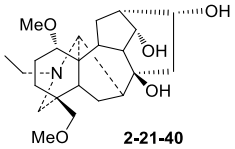
2-21-37



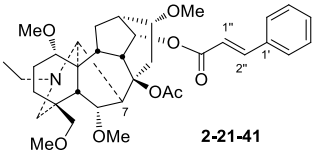
2-21-38



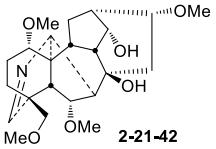
2-21-39



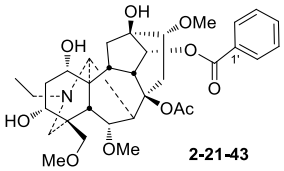
2-21-40



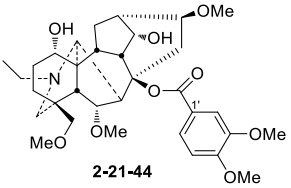
2-21-41



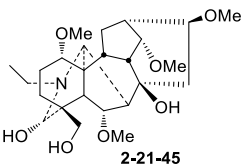
2-21-42



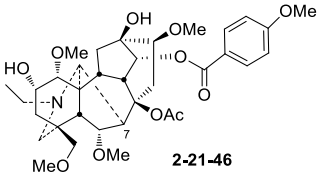
2-21-43



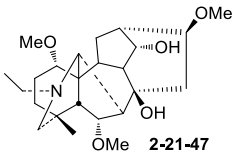
2-21-44



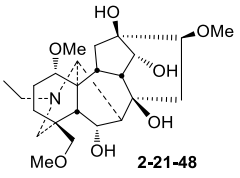
2-21-45



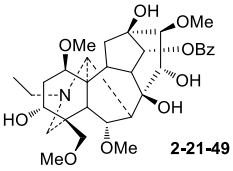
2-21-46



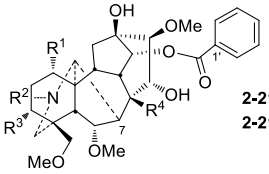
2-21-47



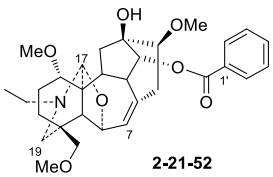
2-21-48



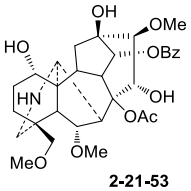
2-21-49



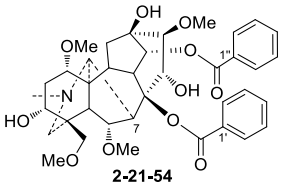
	R^1	R^2	R^3	R^4
2-21-50	OMe	Et	OH	OEt
2-21-51	OH	H	H	OAc



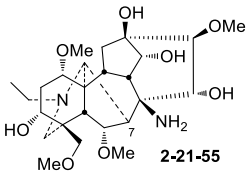
2-21-52



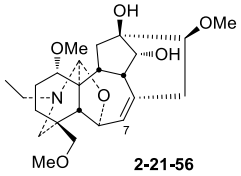
2-21-53



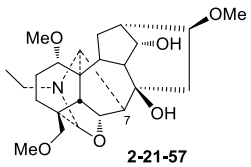
2-21-54



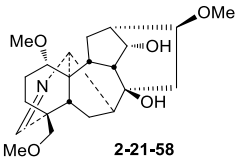
2-21-55



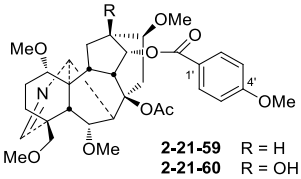
2-21-56



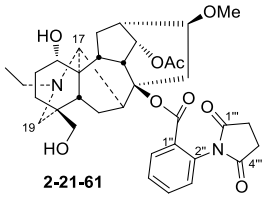
2-21-57



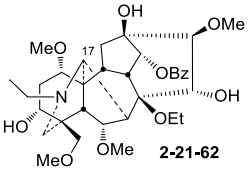
2-21-58



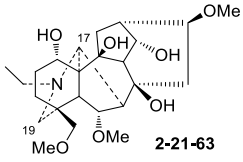
2-21-59 R = H
2-21-60 R = OH



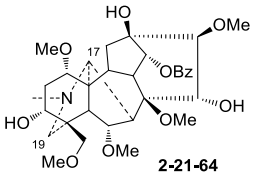
2-21-61



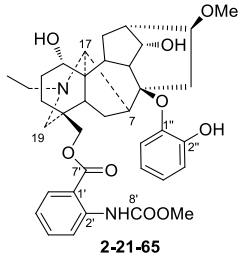
2-21-62



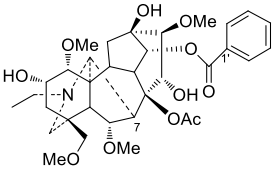
2-21-63



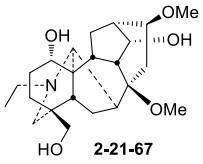
2-21-64



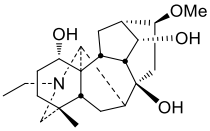
2-21-65



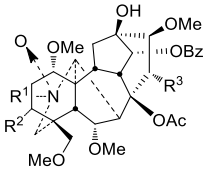
2-21-66



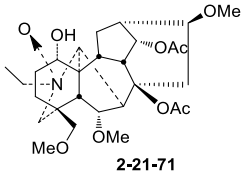
2-21-67



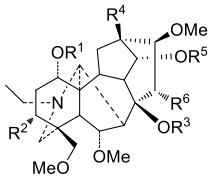
2-21-68



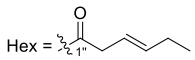
	R^1	R^2	R^3
2-21-69	Et	OH	OH
2-21-70	Me	H	H

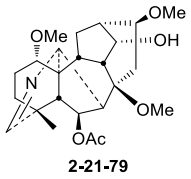


2-21-71

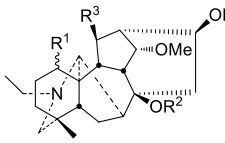


	R^1	R^2	R^3	R^4	R^5	R^6
2-21-72	H	H	H	H	Ac	H
2-21-73	Me	OH	Hex	OH	Bz	OH
2-21-74	H	H	Ac	H	H	OH

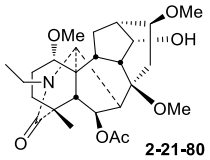




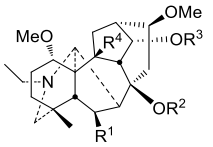
2-21-79



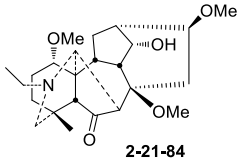
	R^1	R^2	R^3
2-21-75	α-OH	Ac	H
2-21-76	α-OH	Ac	OH
2-21-77	β-OH	H	OH
2-21-78	α-OH	H	OH



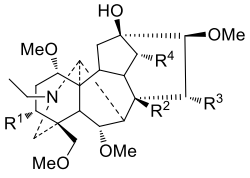
2-21-80



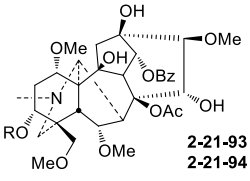
	R^1	R^2	R^3	R^4
2-21-81	OAc	Me	Bz	H
2-21-82	OAc	Me	Ac	H
2-21-83	OAc	Me	Me	H
2-21-85	OH	H	H	H
2-21-86	OAc	Me	H	OH



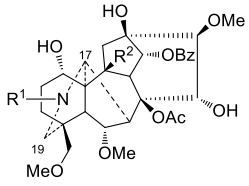
2-21-84



	R^1	R^2	R^3	R^4
2-21-87	H	OAc	H	OVr
2-21-88	OH	OAc	H	OVr
2-21-89	H	OH	H	OVr
2-21-90	OH	OH	H	OVr
2-21-91	OH	OAc	OH	OBz
2-21-92	OH	OH	OH	OBz



2-21-93 R = Ac
2-21-94 R = H



	R^1	R^2
2-21-95	CHO	OH
2-21-96	Ac	OH
2-21-97	Ac	H
2-21-98	H	OH

Table 2-21-11: ^{1}H NMR spectroscopic data of B1-type diterpenoid alkaloids **2-21-29**~**2-21-33**.

H	2-21-29	2-21-30	2-21-31	2-21-32	2-21-33
1	3.72 t(3.5)	3.70 t(3)	3.07 dd(10.7, 6.6)	3.76 dd(10.5, 6.9)	3.74 dd(10.3, 6.9)
2	*α* 1.74 m	1.58 m	2.28 dddd(14.5, 13, 10.5, 3)	2.32 ddd(14.5, 13, 10.5)	2.35 m
	β 1.80 m		1.91 m	1.95 m	1.98 m

Table 2-21-11 (continued)

H	2-21-29	2-21-30	2-21-31	2-21-32	2-21-33
3	*β* 3.68 dd(4, 11)	1.72 dtd(14.5, 12.6, 6.3)	1.57 m	1.57 m	1.60 m
		1.49 m	1.21 m	1.19 ddd d(14.5, 13.5, 4.5, 2.5)	1.22 ddd d(14.5, 13.5, 4.5, 2.5)
5	1.90 d(8)	1.61 d(7.8)	1.41 d(7.3)	1.59 d(7.4)	1.63 d(6.5)
6	*α* 1.5 dd(8, 14)	1.88 dd(14.8, 7.8)	1.91 dd(14.8, 7.3)	1.95 dd(15.1, 7.4)	1.89 dd(15.6, 6.5)
	β 1.8 m	1.58 m	1.33 dd(14.8, 8)	1.51 dd(15.1, 7.8)	1.56 dd(15.6, 8)
7	2.4 d(8)	2.04 br d(8)	2.35 br d(8)	2.09 d(8)	2.08 d(8)
9	2.10 dd(5.11)	2.20 br t(*ca*.6)	2.21 br t(*ca*. 6)	2.14 br d(4.8)	2.18 d(5)
10	1.76 m	1.82 ddd(11.6, 6.2, 6)	1.76 ddd(11.6, 6.3, 6)		
12	1.60 m	1.98 m	2.06 dd(14.8, 6.3)	2.46 m	2.71 d(16)
	2.0 d(9)	1.63 m	1.84 ddd (14.8, 11.6, 8)	2.07 dd(15.7, 12.7)	1.74 dd(16, 8)
13	2.32 m	2.25 m	2.32 br t(5.5)	2.41 s	2.78 br dd(8, 5)
14	4.22 t(4.5)	4.30 t(5)	4.01 br q(5.3)	4.72 t(5.2)	5.32 t(5)
15	2.25 m	2.54 m	2.18 dd(15.7, 4.8)	2.48 m	2.41 dd(16.5, 9.5)
		2.00 m	3.33 br d(15.7, 9)	1.70 dd(16.2, 8.4)	1.91 dd(16.5, 4.5)
16	3.25 t(9.0)	3.83 dd(9.2, 4.3)		3.40 br d(9)	3.17 dd(9.5, 4.5)
17	2.20 s	2.79 br s	2.91 br s[①]	3.00 br s	2.85 br s
18	0.90 s	0.87 s	0.77 br s[①]	0.78 s	0.80 s
19	2.30 m	2.28 d(11.1)	2.46 d(11.6)[①]	2.50 d(11.6)	2.50 d(11.1)
	1.95 d(11)	2.06 br d(11.1)	2.00 dd(11.6, 1.7)[①]	2.07 d(11.6)	2.04 br d(11)
21	2.50 m	2.53 m	2.50 m[①]	2.48 m	2.48 m
	2.58 m	2.47 m	2.39 m[①]	2.38 m	2.41 m
22	1.12 t(7)	1.10 t(7.2)	1.11 t(7)[①]	1.05 t(7.1)	1.06 t(7)
OMe	3.35 s(8-OMe)		3.25 s[①]	3.26 s, 3.33 s	3.28 s, 3.22 s
	3.38 s(16-OMe)		3.26 s[①]		
8-O$\underline{CH_2}CH_3$			3.38 m, 3.37 m		
8-O$CH_2\underline{CH_3}$			1.12 t(7)		
OAc					2.05 s
OH			3.60 d(6.5)		

[①] Typographic error exists in the literature, some data being printed in the wrong lines.

Table 2-21-12: ^{1}H NMR spectroscopic data of B1-type diterpenoid alkaloids **2-21-34**~**2-21-38**.

H	2-21-34	2-21-35	2-21-36	2-21-37	2-21-38
1	3.90 dd(10.6, 7)	*β* 3.75 t(3.5)	3.25 m	3.10 m	3.72 br s
2	2.33 m	*α* 1.59 m	*α* 1.80 m	*α* 1.82 m	*α* 2.55 m
	2.00 m	*β* 1.57 m	*β* 2.22 m	*β* 2.09 m	*β* 1.28 dd(5, 12)
3	1.60 m	1.92 m	*α* 1.70 m	*α* 1.63 m	*α* 1.60 dd(4, 12)
	1.21 dddd (14.5,13.5, 4.5, 2.5)		*β* 2.04 m	*β* 1.82 m (ov)	*β* 2.20 m
5	1.64 d(7.2)	1.87 d(6.9)	2.32 m	2.38 m (ov)	1.85 m
6	2.33 m	*α* 1.81 m	*α* 1.67 m (ov)	*α* 1.28 m	*α* 1.68 dd(4, 14)
	1.54 dd(14.8, 7.8)	*β* 1.51 m	*β* 2.09 m	*β* 1.44 m	*β* 1.95 m
7	2.21 d(7.5)	2.44 d(7.6)	2.14 m	1.69 m	*β* 2.07 m
9	2.24 br s	2.11 t(5.7)	2.34 m	2.30 m	2.62 t(6)
10		1.91 m	2.06 m	1.82 m	2.15 dd(3, 13)
12	2.58 br d(15.6)	*α* 1.65 m	*α* 1.89 m	*α* 1.92 m	*α* 1.58 dd(4, 11)
	1.92 dd(15.6, 8)	*β* 2.04 ddd (14.1, 11.6, 7.7)	*β* 1.85 m (ov)	*β* 1.72 m (ov)	*β* 1.94 m
13	2.74 br t(6.4)	*α* 2.37 (ov)	1.86 m (ov)	2.20 m	1.97 m
14		*β* 4.12 t(4.6)	4.14 t(4.8)	4.14 t(4.4)	4.84 t(4.5)
15	2.50 dd(16.9, 6)	2.17 m	*α* 2.18 m	*α* 2.12 m	*α* 1.92 m
	1.81 d(16.9)		*β* 2.38 m	*β* 2.56 m	*β* 2.25 dd(5, 10)
16	3.88 t(6)	3.38 t(8.9)	3.47 m	3.43 m	3.25 dd(4, 11)
17	3.46 br s	2.73 s	3.63 br s	3.18 br s	2.74 br s
18	0.80 s	3.48 d(10.5)	3.57 ABq(10.0)	3.77 ABq(11.2)	*α* 3.00 d(9)
		3.32 d(10.5)	3.64 ABq(10.0)	3.81 ABq(11.2)	*β* 3.15 d(9)
19	2.50 d(11)	2.37 d(11.2)		2.07 ABq(11.2)	*α* 2.32 m
	2.10 br d(11)	2.09 d(11.2)		2.53 ABq(11.2)	*β* 1.98 m
21	2.52 m, 2.41 m	2.55 m, 2.49 m		2.36 m, 2.46 m	2.48 m
22	1.09 t (7)	1.14 t		1.06 t (7.2)	1.10 t (7)
OMe	3.31 s, 3.34 s	3.19 s(8-OMe)	3.28 s(1-OMe)	3.28 s(1-OMe)	3.27 s(16-OMe)
		3.39 s(16-OMe)	3.36 s(16-OMe)	3.34 s(16-OMe)	3.30 s(18-OMe)
			3.35 s(18-OMe)		
OAc				2.06 s	2.05 s, 2.03 s

Table 2-21-13: ^{1}H NMR spectroscopic data of B1-type diterpenoid alkaloids **2-21-39**~**2-21-43**.

H	2-21-39	2-21-40	2-21-41	2-21-42	2-21-43
1	3.25 m	3.07 dd(6.4, 10.4)	3.04 t	3.14~3.17 m	3.75 br s
2	*α* 1.88 m	1.98 m	*α* 1.91~1.94 m	*α* 1.48~1.51 m	*α* 1.81~1.83 m
	β 1.94 m	2.19 m	*β* 2.35 t(5.2)	*β* 1.81~1.83 m	*β* 1.95 br d(8.0)
3	*α* 1.67 m	1.38 dd(11.6)	1.64 (ov)	*α* 1.54 t(2.0)	4.12 br d(4.8)
	β 1.56 m	1.74 m		*β* 1.84 (ov)	
5	1.78 m	1.64 d(7.2)	2.08 d(6.8)	2.18 d(1.6)	2.10 (ov)
6	*α* 1.76 m	1.47 dd(7.6, 14.4)	4.06 d(6.8)	4.14 (ov)	4.0 br d(6.8)
	β 2.00 t(7.6)	1.89 d(7.6)			
7	2.09 m	2.07 d(8.0)	3.04 t	2.04 t(5.2)	3.09 s
9	2.23 t(5.2)	2.25 m	2.72 t	2.12 t(7.6)	2.84 (ov)
10	1.72 m	1.70 m	1.96~1.98 m	2.35~2.39 m	2.13 (ov)
12	*α* 1.61 m	1.78 m	*α* 2.57~2.59 m	*α* 1.85~1.86 m	*α* 2.25 br s
	β 1.82 m		*β* 1.96~1.98 m	*β* 1.71~1.75 m	*β* 2.06 d(4.4)

Table 2-21-13 (continued)

H	2-21-39	2-21-40	2-21-41	2-21-42	2-21-43
13	2.34 t(6.8)	2.23 m	2.36 t(5.2)	1.76~1.78 m	
14	4.16 t(5.2)	4.22 t(5.0)	4.94 t(5.2)	4.15 t(4.8)	4.92 d(5.4)
15	*α* 2.06 m	1.95 m	*α* 2.16~2.18 m	2.14 t(7.6)	*α* 2.42 d(8.8)
	β 2.55 dd(17.2, 8.4)	2.58 m	*β* 2.85~2.91 m	2.57~2.64 m	*β* 3.07 d(8.8)
16	3.39 d(8.0)	3.82 d(8.0)	3.32 (ov)	3.47 br d(8.8)	3.50 (ov)
17	3.24 s	3.18 s	2.84 s	4.03 s	2.84 (ov)
18	2.97 ABq(8.8)	2.99 ABq(9.2)	3.14 ABq(8.4)	3.67 ABq(8.8)	3.45 (ov) ABq
	3.10 ABq(8.8)	3.11 ABq(8.4)	3.64 ABq(8.4)	3.82 ABq(8.8)	3.48 (ov) ABq
19	2.52 ABq(13.6)	2.02 d(11.6)	2.42 ~2.47 m	7.45 s	2.40 s
	2.80 ABq(13.6)	2.51 m			3.15 s
21		2.39 m	2.53~2.56 m		2.58~2.63 m
		2.56 m	3.20 (ov)		3.09 br s
22		1.05 t(7.2)	1.07 t(6.8)		1.18 t(7.2)
1-OMe	3.27 s	3.26 s	3.24 s	3.36 s	
6-OMe			3.21 s	3.25 s	3.18 s
16-OMe	3.34 s		3.37 s	3.20 s	3.57 s
18-OMe	3.29 s	3.29 s	3.29 s	3.34 s	3.30 s
OAc			1.80 s		1.32
2′, 6′			7.49~7.52 m		8.06 d(6.8)
3′, 5′			7.37~7.39 m		7.45 t(8.0)
4′			7.37~7.39 m		7.56 t(7.6)
1″			6.42 d(16)		
2″			7.68 d(16)		

Table 2-21-14: ^{1}H NMR spectroscopic data of B1-type diterpenoid alkaloids **2-21-44**~**2-21-48**.

H	2-21-44	2-21-45	2-21-46	2-21-47	2-21-48
1	*β* 3.71 br s	*β* 3.20 dd(9.6)	3.12 d(4.8)	*β* 3.80 m	3.00 dd(10.8, 6.4)
2	*α* 1.65 m	*α* 1.70~1.73 m	4.01 m	*α* 1.80 m	1.94 m
	β 1.53 m	*β* 1.66~1.68 m		*β* 2.10 m	2.27 m
3	*α* 1.90 dd(13.7, 5.6)	*α* 1.74~1.77 m	*α* 1.74 dd(14.8, 3.6)	*α* 1.60 m	1.50 td(11.6, 3.6)
	β 1.70 dd(13.7, 4.6)	*β* 2.43~2.47 m	*β* 1.96 dd(15.0, 2.2)	*β* 2.40 m	1.68 dt(12.8, 3.6)
5	2.28 d(6.4)	1.89~1.91 m	2.23 d(6.4)	2.00 m	2.00 (ov)
6	4.20 m	4.10 dd(1.6)	4.01 d(6.0)	4.05 dd(1, 7)	4.71 d(6.8)
7	3.31 s	2.25 d(1)	3.10 br s	2.50 d(1)	2.01 (ov)
9	2.52 t(5.9)	1.79~1.82 m	2.97 m	1.80 m	2.34 m
10	2.00 m	1.61~1.65 m	2.20 m	1.75 m	1.90 m
12	*α* 1.82 dd(14.4, 4.6)	2.29~1.31 m	*α* 2.18 m	*α* 1.45 m	2.26 m
	β 2.10 m	1.61~1.65 m	*β* 2.68 m	*β* 2.50 m	2.52 m
13	2.36 m	2.39~2.42 m		1.30 m	
14	4.20 m	3.57 t(5)	4.88 d(5.2)	4.20 t(5)	3.97 d(5.2)

Table 2-21-14 (continued)

H	2-21-44	2-21-45	2-21-46	2-21-47	2-21-48
15	*α* 3.00 dd(16.2, 8.9)	1.69~1.71 m	*α* 2.46 dd(14.2, 5.6)	2.70 dd(7, 14)	2.25 m
	β 2.38 m	2.52 dd(12, 14)	*β* 2.97 m	3.00 dd(12, 14)	2.49 m
16	3.42 t(8.3, 6.8)	3.60 dd(7, 12)	3.30 m	3.72 dd(7, 12)	3.36 d(8.4)
17	2.81 s	2.87 s	2.80 br s	2.60 br s	3.09 s
18	3.59 d(8.1)	3.32 d(10)	*α* 3.01 d(8.4)	0.88 s	3.36 ABq(8.4)
	3.12 d(8.1)	3.58 d(10)	*β* 3.65 d(8.4)		3.78 ABq(8.4)
19	2.71 d(11.0)	3.85 s	2.53 ABq(11.4)	1.80 m	2.57 m
	2.41 m		2.58 ABq(11.4)	3.15 m	2.78 d(10.8)
21	2.74 m	2.59~2.61 m	2.61 m	2.40 m	2.55 m
	2.65 m	2.43~2.47 m	2.68 m	2.65 m	
22	1.20 t(7.2)	1.07 t(7)	1.16 t(7.2)	1.07 t(7)	1.97 t(7.2)
OMe	3.04 s(6-OMe)	3.38 s(1-OMe)	3.31 s(1-OMe)	3.38 s(1-OMe)	3.23 s(1-OMe)
	3.35 s(16-OMe)	3.36 s(6-OMe)	3.18 s(6-OMe)	3.36 s(6-OMe)	3.40 s(16-OMe)
	3.29 s(18-OMe)	3.34 s(14-OMe)	3.52 s(16-OMe)	3.33 s(16-OMe)	3.31 s(18-OMe)
	3.91 s(3′-OMe)	3.33 s(16-OMe)	3.27 s(18-OMe)		
	3.92 s(4′-OMe)		3.87 s(4′-OMe)		
8-OAc			1.36 s		
1-OH	3.38 s				
2′	7.54 d(1.7)		8.01 d(8.8)		
3′			6.91 d(8.8)		
5′	6.85 d(8.4)		6.91 d(8.8)		
6′	7.66 dd(8.4, 1.7)		8.01 d(8.8)		

Table 2-21-15: ^{1}H NMR spectroscopic data of B1-type diterpenoid alkaloids **2-21-49**~**2-21-53**.

H	2-21-49	2-21-50	2-21-51	2-21-52	2-21-53
1	3.44 br s	3.12 t(6.4)	3.66 br s	3.36 m	3.70 br s
2	*α* 1.37 m	*α* 2.34 dt(13.6, 6.4)	*α* 1.85 m	2.41 m	1.65 m
	β 2.30 br d(11.6)	*β* 1.86 dt(13.6, 4.8)	*β* 1.60 m	1.95 m	1.55 m
3	4.21 br s	3.80 dd(6.4, 4.8)	1.61 m	1.76 dd(14, 3)	1.89 m
				1.55 m	1.68 m
5	2.95 br d(5.6)	2.08 m	2.70 br s	2.25 s	2.31 br s
6	4.21 br d(5.6)	4.03 d(6.4)	3.95 d(6.4)	4.40 d(6)	4.00 d(6)
7	2.25 br s	2.67 m	2.25 m	5.76 d(6)	2.73 br s
9	2.54 t(4.2)	2.61 m	2.75 t(5.2)	3.20 br s	2.82 dd(7, 5)
10	2.22 dd(9.6, 4.2)	2.06 m	2.1 m	2.66 m	2.15 m
12	*α* 1.77 br d(9.6)	*α* 2.10 m	*α* 2.20 m	2.12 t(11)	2.32 m
	β 2.22 t(9.6)	*β* 2.55 dd(14, 4)	*β* 2.25 m	2.01 m	2.21 m
14	4.93 d(4.5)	4.79 d(5.2)	4.85 d(4.9)	5.06 br s	4.91 d(5)
15	4.76 d(5.2)	4.52 d(6.4)	4.46 dd(5.2, 2.6)	3.90 dd(12, 8)	4.51 d(5)
				2.64 m	
16	3.10 br d(5.2)	3.24 m	3.37 d(5.2)	3.35 m	3.43 d(5)

Table 2-21-15 (continued)

H	2-21-49	2-21-50	2-21-51	2-21-52	2-21-53
17	3.33 br s	2.87 br s	3.06 br s	4.37 s	3.07 br s
18	3.55 d(8.4)	3.43 d(8.8)	3.05 d(9.2)	3.15 d(9)	3.57 d(8)
	3.53 d(8.4)	3.55 d(8.8)	3.50 d(9.2)	3.04 d(9)	3.11 d(8)
19	3.52 d(12.4)	2.43 m	2.25 m	2.44 m	3.27 d(11)
	3.26 d(12.4)	2.70 m	3.22 m	2.04 m	2.30 m
NH					3.96 br s
21	3.26 m, 3.29 m	2.43 m, 2.91 m		2.60 m, 2.41 m	
22	1.34 t(6.5)	1.07 t (7.2)		1.01 t(7)	
OAc			1.34 s		1.42 s
OEt		3.29 m, 3.46 m, 0.54 t(6.4)			
1-OMe	3.30 s	3.23 s		3.37 s	
6-OMe	3.35 s	3.23 s	3.12 s		3.19 s
16-OMe	3.70 s	3.71 s	3.70 s	3.47 s	3.78 s
18-OMe	3.24 s	3.28 s	3.26 s	3.28 s	3.33 s
2′, 6′	8.03 d(7.2)	8.01 d(7.6)	7.97 d(7.6)	8.07 d(8)	8.04 d(7)
3′, 5′	7.43 t(7.2)	7.41 t(7.6)	7.40 d(7.6)	7.45 t(8)	7.47 t(7)
4′	7.53 t(7.2)	7.53 t(7.6)	7.55 t(7.6)	7.57 t(8)	7.59 t(7)
3-OH	7.82 br s				

Table 2-21-16: ^{1}H NMR spectroscopic data of B1-type diterpenoid alkaloids **2-21-54**~**2-21-58**.

H	2-21-54	2-21-55	2-21-56	2-21-57	2-21-58
1	3.14 br d(9.3)	3.15 dd(7.6, 6.4)	3.24 dd(10.8, 6.4)	3.19 m (ov)	3.14 m
2	2.18 m, 2.34 m	*α* 1.98 m, *β* 2.33 m	*α* 1.93 m, *β* 2.48 m	*α* 2.34 m, *β* 1.82 m	*α* 1.92 m, *β* 1.28 m
3	3.77 m	3.76 dd(9.6, 4.8)	1.52 ddd(12, 4.4, 2.4)	*α* 2.04 m	*α* 1.68 m (ov)
			1.77 ddd(13.6, 4.8, 2.0)	*β* 1.50 m	*β* 1.33 m
5	2.01 d(6.8)	2.06 br s	2.21 s	1.90 d(4.4)	2.08 d(5.6)
6	4.15 d(6.8)	4.13 d(6.4)	4.39 d(6.0)	4.70 dd(4.4, 2.8)	*α* 1.94 m (ov)
					β 1.42 m
7	3.08 s	2.15 m	5.71 d(5.6)	2.29 br s ($W_{1/2}$ = 2.0)	1.65 m (ov)
9	3.19 t(5.6)	2.13 m	2.83 br s	1.84 m	2.14 dd(4.8, 4.4)
10	2.19 d(5.9)	1.94 m	2.42 m	1.62 m	1.69 m
12	2.18 m	*α* 2.51 m	*α* 2.00 m	*α* 1.84 m	*α* 1.80 m
	2.90 br d(9.3)	*β* 1.92 m	*β* 1.93 m	*β* 1.46 m	*β* 1.66 m
13				2.38 m	2.36 br s ($W_{1/2}$ = 16.8)

Table 2-21-16 (continued)

H	2-21-54	2-21-55	2-21-56	2-21-57	2-21-58
14	4.92 d(5.1)	3.81 d(5.2)	4.04 br s	4.05 dd(4.9, 2.6)	4.10 br s ($W_{1/2}$ = 4.6)
15	4.62 m	4.23 d(6.4)	α 3.09 m β 2.59 m	α 2.08 m β 1.98 m	2.04 m 2.58 dd(17.5, 8.4)①
16	3.39 d(5.1)	3.09 d(6.4)	3.31 m	3.45 m	3.44 m
17	3.16 s	3.06 s	4.32 br s	3.35 s	β 4.08 s
18	3.55 d(9.0) 3.63 d(9.0)	3.35 ABq(8.4) 3.70 ABq(8.4)	3.06 ABq(9.2) 3.16 ABq(9.2)	α 3.20 ABq(8, 8) β 2.95 ABq(8, 8)	α 3.35 (ov) β 3.08 ABq(8.0)
19	2.37 d(11.7) 2.79 d(11.5)	α 2.78 (ov) β 2.44 (ov)	α 2.44 ABq(11.0) β 2.04 ABq(11.0)	4.35 s	7.27 s
21	2.40 br s	2.47 m, 2.81 m	2.42 m, 2.60 m	α 2.82 m, β 2.54 m	
22		1.11 t(7.2)	0.99 t(7.2)	1.02 t(7.2)	
1-OMe	3.31 s	3.26 s	3.31 s	3.25 s	3.34 s
6-OMe	2.87 s	3.37 s			
16-OMe	3.75 s	3.58 s	3.42 s	3.33 s	3.20 s
18-OMe	3.26 s	3.29 s	3.29 s	3.27 s	3.32 s
2′, 6′	7.68 br d(7.1)				
3′, 5′	7.16 br t(8.1)				
4′	7.35 br t(7.5)				
2″, 6″	7.84 br d(7.1)				
3″, 5″	7.12 br t(7.8)				
4″	7.26 br t(7.5)				
8-OH				3.74 s	
14-OH				5.35 d(5.2)	

① Typographic error exists in the literature, with two signals for H-15β being given.

Table 2-21-17: ^{1}H NMR spectroscopic data of B1-type diterpenoid alkaloids **2-21-59**~**2-21-61**.

H	2-21-59	2-21-60	2-21-61
1	3.15 t(3.9)	3.88 t(3.9)	3.78 br s ($W_{1/2}$ = 5.4)
2α	2.10~2.12 m	2.08~2.10 m	2.08 m
2β	1.26~1.28 m	1.76~1.78 m	1.61 m
3α	2.26~2.28 m	2.24~2.26 m	1.84 m
3β	1.96~1.98 m	1.91~1.93 m	1.60 m
5	3.62 d(6.4)	3.60 d(6.4)	1.86 m
6	4.08 d(6.4)	4.04 d(6.4)	α 2.13 m, β 1.68 m
7	2.88 s	2.78 t(8.2)	2.10 m
9	2.80 t(5.9)	3.16 t(5.9)	2.26 m
10	2.10~2.12 m	2.18~2.20 m	1.94 m

Table 2-21-17 (continued)

H	2-21-59	2-21-60	2-21-61
12*α*	1.96~1.98 m	1.81 d(14.3)	2.13 m
12*β*	2.12~2.14 m	2.34~2.38 m	1.73 m
13	2.18 m		2.65 dd(7.4, 4.8)
14	4.84 d(5.0)	4.85 d(4.5)	4.86 t(4.8)
15*α*	2.44 dd(15.3, 8.9)	2.44 dd(15.3, 8.9)	1.88 m
15*β*	2.87~2.91 m	3.20~3.23 m	2.28 m
16	3.45 t(6.8)	3.48 t(6.8)	3.27 m
17	3.72 s	3.77 s	2.78 s
18	*α* 3.81 d(8.3)	*α* 3.80 d(8.3)	3.89 ABq(10.8)
	β 3.64 d(8.3)	*β* 3.63 d(8.3)	4.09 ABq(10.8)
19	8.04 s	7.30 s	2.16 ABq(10.8)
			2.41 ABq(10.8)
21			2.54 m
22			1.14 t(7.2)
1-OMe	3.12 s	3.10 s	
6-OMe	3.55 s	3.53 s	
16-OMe	3.20 s	3.18 s	3.27 s
18-OMe	3.30 s	3.26 s	
2′,6′	7.98 d(8.9)	7.97 d(8.9)	
3′,5′	6.91 d(8.9)	6.89 d(8.9)	
4′-OMe	3.85 s	3.85 s	
OAc	1.29 s	1.36 s	2.06 s
3″			7.27 d(7.6)
4″			7.69 t(7.6)
5″			7.54 t(7.6)
6″			8.06 d(6.4)
2‴,3‴			2.92 ABq(6.8)
			2.95 ABq(6.8)

Table 2-21-18: ^{1}H NMR spectroscopic data of B1-type diterpenoid alkaloids **2-21-62**~**2-21-64**.

H	2-21-62	2-21-63	2-21-64
1	3.13 dd(7.5, 5.8)	*β* 4.02 br s	3.19 br t(6)
2	*β* 1.90 dt(13, 5.1)	–	1.98 m, 2.35 m
3	3.79 dd(9.1, 4.8)	–	3.88 m
5	2.06 d(6.5)	2.42 d(6.1)	2.05~2.20 m (ov)
6	4.07 d(6.6)	*β* 4.14 d(6.4)	4.05 d(6)
7	2.68 s	2.02 br s	2.80 br s
9	–	2.06 d(5.2)	2.59 br d
10	–		2.05~2.20 m (ov)
12	–	2.30 d(15.1)	2.05~2.20 m (ov)
14	4.82 d(5.1)	*β* 4.66 dd(5.2, 5.2)	4.83 d(5.1)
15	4.54 br s	2.35 d(9.1, 15.7)	4.56 d(5.9)
		2.10 dd(6.1, 15.7)	

Table 2-21-18 (continued)

H	2-21-62	2-21-63	2-21-64
16	–	–	3.31 (ov)
17	2.87 s	2.53 br s	2.93 br d
18	3.48 d(8.5)	3.68 d(8.0)	3.54 d(9)
	3.61 d(8.5)	3.26 d(8.0)	3.60 d(9)
19	*α* 2.90 d(11.3)	2.72 d(10.5)	2.57 br m
		2.32 d(10.5)	
21	–	–	2.52 br s
22	1.09 t(7)	1.13 t(7.2)	
OMe	3.25 s, 3.26 s	3.35 s, 3.34 s	3.31 s(1-OMe)
	3.30 s, 3.73 s	3.33 s	3.29 s(6-OMe)
			3.15 s(8-OMe)
			3.75 s(16-OMe)
			3.31 s(18-OMe)
OH		7.51 br d(8.8, 1-OH)	3.05 br
		3.01 br s	3.70 br
15-OH	3.63 s		
Ar-H	7.42~8.05 m		
8-$OCH_2\underline{CH_3}$	0.60 t(7)		
2′, 6′			8.02 d
3′, 5′			7.44 t(8)
4′			7.53 t(8)

Table 2-21-19: ^{1}H NMR spectroscopic data of B1-type diterpenoid alkaloids **2-21-65**~**2-21-69**.

H	2-21-65	2-21-66	2-21-67	2-21-68	2-21-69
1	3.76 t(3)	3.14 d(4.5)	*β* 3.74 t(2.8)	3.69 t(2.5)	–
2	1.60 m, 1.58 m	4.02 m	*α* 1.64 m, *β* 1.57 m	*α* 1.62 m	–
				β 1.55 m	
3	1.85 m	*α* 2.02 dd(14.8, 3.6)	*α* 1.87 m(6.9, 6.9)	*α* 1.46 td(3.0, 13.0)	*β* 3.73 m
	1.64 m	*β* 1.71 dd(14.8, 2.0)	*β* 1.62 m	*β* 1.71 dt(6.0, 13.0)	
			(14.8, 10.8, 5.4, 14.7)		
5	1.75 d(8)	2.25 d(6.0)	1.85 d(8.1)	1.58	2.38 d(6.5)
6	1.79 dd(14, 8)	4.00 d(6.0)	1.51 dd(14.3, 8.1)	*α* 1.55	*β* 4.08 d(6.5)
	1.56 m		1.79 m(14.3, 8.0)	*β* 1.88 q(7.0, 15.0)	
7	2.42 d(8)	2.92 s	2.42 d(8.0)	2.05 m	–
9	2.10 dd(10.4, 4.7)	2.97 m	2.09 dd(10.4, 4.7)	2.20 m	2.90 t

Table 2-21-19 (continued)

H	2-21-65	2-21-66	2-21-67	2-21-68	2-21-69
10	1.94 m	2.25 m	1.92 m	1.80 m	–
12	2.05 m	*α* 2.78 m	2.04 m(13.4, 7.9)	*α* 2.00 m	2.77 d
	1.62 m	*β* 2.30 m	1.62 m (ov)	*β* 1.60 m	
13	2.34 m		2.38 m	2.30 t(5.0)	
14	4.14 t(5)	4.90 d(4.4)	4.10 t(4.7)	4.18 t(4.8)	4.86 d(5.0)
15	2.15 dd(15, 5)	4.46 dd(4.4, 3.2)	2.14 m(9.0)	*α* 2.35 m	*β* 4.46 dd(5.3, 2.6)
	3.30 dd(15, 8.5)		2.16 dd(10.3, 9.0)	*β* 2.05 m	
16	3.25 br d(9)	3.26 d(4.4)	3.35 t(9.0)	3.35 dd(5.0, 9.0)	–
17	2.71 br s	3.06 s	2.71 br s	2.74 s	3.43 br s
18	4.15 d(11)	*α* 3.68 d(8.8)	3.45 AB(10.4)	0.85 s	–
	4.22 d(11)	*β* 2.97 d(8.8)	3.28 AB(10.4)		
19	2.70 d(13)	*α* 2.58 d(11.2)	2.34 AB(11.7)	*α* 2.25 AB(10.5)	–
	2.80 d(13)	*β* 2.51 d(11.2)	2.07 AB(11.7)	*β* 2.05 AB(10.5)	
21	2.54 m	2.88 m	2.54 (octet)	2.40 m	–
	2.48 m	2.24 m	2.48 (octet)	2.49 m	
22	1.32 t(4.5)	1.15 t(7.2)	1.11 t(7.5)	1.10 t(7.5)	1.42 t(7.2)
OH		3.87 s(13-OH)			4.37 d(2.6, 15-OH)
		4.35 d(3.2, 15-OH)			
OMe	3.56 s(16-OMe)	3.33 s(1-OMe)	3.14 s(8-OMe)	3.31 s(16-OMe)	3.24 s(1-OMe)
		3.19 s(6-OMe)	3.38 s(16-OMe)		3.21 s(6-OMe)
		3.72 s(16-OMe)			3.68 s(16-OMe)
		3.28 s(18-OMe)			3.26 s(18-OMe)
Ar-NH	11.4 s				
OAc	3.60 s	1.41 s			1.43 s
2′					8.02 d(7.5)
3′	7.96 dd(8.5, 1.5)				7.43 t(7.5)
4′	7.48 ddd(8.5, 7.5, 1.5)				7.56 t(7.5)
5′	7.04 ddd(8.5, 7.5, 1.5)				7.43 t(7.5)
6′	8.62 dd(7.5, 1.5)				8.02 d(7.5)
3″	7.17 br d(8.3)				
4″	7.45 td(8.3, 1.2)				
5″	6.75 td(8.3, 1.2)				
6″	7.90 dd(8.3, 1.5)				
Ar-H		8.03 d(7.2)			
		7.46 t(7.2)			
		7.58 t(7.2)			

Table 2-21-20: ^{1}H NMR spectroscopic data of B1-type diterpenoid alkaloids **2-21-70**~**2-21-74**.

H	2-21-70	2-21-71	2-21-72	2-21-73	2-21-74
1	–	*β* 3.62 m	3.68 br s	3.15 m	3.65 s
2*α*	–	–	1.55 m	2.38 m	1.56 m
2*β*	–	–	1.55 m	2.00 m	1.56 m
3	1.65 m	1.55 m	*α* 1.62 m	3.78 m	*α* 1.62 m
			β 1.90 m		*β* 1.88 m
5	–	2.31 s	2.20 d(6.4)	2.12 d(6.3)	2.27 d(6.4)
6	–	*β* 4.09 d(6.5)	4.12 d(6.4)	4.03 d(6.3)	4.09 d(6.4)
7	3.28 br s	–	2.00 s	2.85 s	2.91 s
9	2.81 br t	2.52 m	2.25 t(5.4)	2.92 m	2.36 t(5.4)
10	*β* 2.40 br d(4.2)	–	1.90 m	2.13 m	1.92 m
12	–	1.84 m	*α* 1.80 dd	*α* 2.15 m	*α* 1.88 m
		1.90 m	(13.5, 4.9)	*β* 2.74 m	*β* 2.07 m
			β 2.10 m		
13		2.42 m	2.62 dd(7.8, 4.9)		2.30 dd(7.6, 4.9)
14	–	*β* 4.80 t(4.7)	4.86 t(4.9)	4.88 d(4.8)	4.12 t(4.9)
15	2.44 dd(16.1)	2.08 m	*α* 2.32 m	4.48 dd(5.1, 3.0)	4.41 dd(5.2, 2.8)
	2.95 dd(16.1)	2.15 m	*β* 1.92 m		
16	–	–	3.30 m	3.34 d(5.1)	3.22 d(5.2)
17	–	3.18 br s	2.66 s	3.11 s	2.79 s
18	–	–	3.25 d(8.8)	3.49 d(8.9)	3.05 d(9.0)
			3.64 d(8.8)	3.63 d(8.9)	3.63 d(9.0)
19	3.35 d(11)	–	*α* 2.31 d(10.8)	*α* 2.35 d(11.2)	*α* 2.26 d(10.6)
	3.73 d(11)		*β* 2.68 d(10.8)	*β* 2.89 d(11.2)	*β* 2.63 d(10.6)
21	3.31 s	–	2.48 dq(13.2, 7.1)	2.40 m	2.43 m
			2.57 dq(13.2, 7.1)	2.74 m	2.79 m
22		1.44 t(7.1)	1.13 t(7.1)	1.10 t(7.1)	1.13 t(7.1)
15-OH				4.40 d(3.0)	
1-OMe	3.22 s			3.27 s	
6-OMe	3.12 s	3.28 s	3.35 s	3.15 s	3.28 s
16-OMe	3.45 s	3.31 s	3.27 s	3.76 s	3.49 s
18-OMe	3.20 s	3.28 s	3.33 s	3.30 s	3.33 s
8-OAc	1.25 s	1.97 s			2.09 s
14-OAc		2.04 s	2.07 s		
2′, 6′	8.02 d(7.7)			8.04 d(8.4)	
3′, 5′	7.42 t(7.6)			7.45 t(8.4)	
4′	7.52 t(7.5)			7.58 t(8.4)	
2″				2.20 dd(16.5, 5.3)	
				2.48 dd(16.5, 6.1)	
3″				5.04 m	
4″				5.08 m	
5″				1.86 m	
6″				0.89 t(7.1)	

Table 2-21-21: ^{1}H NMR spectroscopic data of B1-type diterpenoid alkaloids **2-21-75**~**2-21-78**.

H	2-21-75	2-21-76	2-21-77	2-21-78
1	*β* 3.73 br s	*β* 4.16 br s	*α* 4.23 br s	*β* 4.07 br s
2	1.80~1.50 m	1.67~1.72 m	1.50~1.65 m	1.72 m
3	1.80~1.50 m	*α* 1.73~1.82 m	1.50~1.65 m	1.72 m, 1.50 m
		β 1.49~1.57 m	1.42 m	
5	*β* 1.80~1.50 m	1.67~1.72 m	1.71 d(7.7)①	1.60 m
6		*α* 1.49~1.57 m	1.88 m	*α* 1.60 m, *β* 1.90 m
		β 1.73~1.82 m	1.50~1.65 m	
7	3.18 d(7.6)	3.19 d(4.7)	1.95~2.05 m	2.08 m
9	*β* 2.43 dd(4.5, 7.0)	2.50~2.64 m	2.42 m	2.35 m
10	*β* 1.91 dd(7.1, 4.7)①	1.73~1.82 m	2.20~2.40 m	1.72 m
12*α*	1.80~1.50 m	4.23 d(2.7)	3.97 d(2.9)	4.24 d(2.9)
12*β*	2.02 m			
13	*β* 2.38 dd(4.5, 7.4)	2.25~2.35 m	2.42 m	2.35 m
14*β*	3.61 t(4.5)	4.05 t(4.5)	4.16 t(4.6)	4.13 t(4.7)
15	2.79 dd(15.0, 8)	*α* 2.25~2.35 m	2.20~2.40 m	2.26 dd(8.4, 14.8)
	2.23 dd(8, 15)	*β* 2.80 dd(8.3, 14.9)	1.95~2.05 m	2.04 dd(8.4, 14.8)
16*α*	3.28 t(8.3)	3.32 t(8.5)	3.19 t(8.5)	3.32 t(8.6)
17	2.72 br s	2.66 br s	2.20~2.40 m	2.65 br s
18		0.88 s	0.79 s	0.89 s
19	2.24 d(12)	2.25~2.35 m	2.20~2.40 m	2.35 m, 2.08 m
	2.04 d(12)	2.06 d(11.1)	1.95~2.05 m	
21	2.54 m, 2.47 m	2.50~2.64 m	2.20~2.40 m	2.50 m
22	1.12 t(7.0)	1.14 t(7.2)	1.02 t(7)	1.13 t(7.2)
14-OMe	3.41 s	3.44 s	3.43 s	3.43 s
16-OMe	3.36 s	3.37 s	3.36 s	3.36 s
OAc	1.98 s	1.98 s		

Table 2-21-22: ^{1}H NMR spectroscopic data of B1-type diterpenoid alkaloids **2-21-79** and **2-21-80**.

H	2-21-79	2-21-80	H	2-21-79	2-21-80
1*β*	3.26 t(6.7)	3.28 dd(9.0, 7.1)	15	*α* 2.13 dd(15.7, 4.5)	1.94 dd(17.1, 7.6)
				β 2.28 dd(15.9, 8.3)	
2	*α* 1.79 m, *β* 1.49 m	–	16*α*	3.42 m	3.46 m
3	*β* 1.33 m	–	17	3.99 br s	3.63 d(3.1)
5	1.65 br s	1.75 dd(3.2, 1.5)	18	1.13 s	1.19 s
6	*α* 5.07 d(6.8)	*α* 5.11 dd(6.9, 1.5)	19	7.20 s	
7	2.79 d(7.0)	2.73 d(6.8)	21		2.83 sext(7.2)
					3.87 sext(7.2)
9	2.83 t(5.6)	2.91 t(5.1)	22		1.15 t(7.2)
10	2.01 m	–	1-OMe	3.21 s	3.24 s
12*β*	1.91 m	–	8-OMe	3.07 s	3.07 s
13	2.38 br t(6.2)	2.44 m	16-OMe	3.37 s	3.38 s
14*β*	4.05 dt(5.1, 5.3)	3.97 dt(8.4, 4.7)	8-OAc	2.04 s	2.06 s

Table 2-21-23: ^{1}H NMR spectroscopic data of B1-type diterpenoid alkaloids **2-21-81**~**2-21-84**.

H	2-21-81	2-21-82	2-21-83	2-21-84
1β	3.11 dd(9.9, 7.2)	3.07 dd(9.8, 7.3)	–	3.19 dd(10.5, 6.7)
3α	–	1.52 dm(16.9)	–	–
5	1.44 br s	1.39 br s	1.39 br s	1.72 s
6α	5.17 d(7.4)	5.16 d(7.3)	5.24 d(7.4)	
7	2.62 d(7.4)	2.69 d(7.2)	2.72 d(7.3)	2.67 s
9				2.09 t(4.7)
14β	4.96 t(4.9)	4.68 t(4.8)	3.56 t(4.6)	3.96 dt(6.9, 5.1)
16α	3.46 dd(12.5, 7.2)	–	–	3.47 m
17	2.97 d(2)	2.89 d(2)	2.92 d(1.7)	3.48 br s
18	0.82 s	0.81 s	0.84 s	0.93 s
19α	–	2.59 d(12.9)	–	
19β				2.58 d(12.1)
21	–	2.40 m	–	2.34 dq(12.1, 7)
22	1.04 t(7)	1.02 t(7)	1.05 t(7.1)	1.08 t(7.1)
OMe	3.11 s, 3.28 s	2.98 s, 3.24 s	3.04 s, 3.27 s	3.30 s, 3.33 s
	3.38 s	3.36 s	3.35 s, 3.37 s	3.37 s
OAc	1.93 s	1.97 s, 1.97 s	2.08 s	
Ar-H	7.43 m(3H)			
	8.04 m(2H)			
14-OH				3.74 d(7.7)

Table 2-21-24: ^{1}H NMR spectroscopic data of B1-type diterpenoid alkaloids **2-21-85** and **2-21-86**.

H	2-21-85	2-21-86	H	2-21-85	2-21-86
1β	–	3.71 d(9.6, 7.4)	14β	3.07 dd(10.5, 6.91)[①] 4.16 t(4.8)[①]	4.55 t(4.8)
3α	1.56 dm(12.9)	–	16α	–	3.31 m
5	1.42 br s	1.69 br s	17	–	3.01 s
6α	4.32 d(7.2)	5.31 d(7.3)	18	0.95 s	0.86 s
7	–	2.72 d(7.4)	19	α 2.56 d(11.6)	2.62 d(11.8)
9	–	2.82 d(5)	22	1.04 t(7.1)	1.06 t(7.1)
12α	–	2.96 d(15.8)	OMe	3.26 s, 3.35 s	3.08 s, 3.26 s
					3.35 s
12β	–	1.74 dd(15.7, 7.4)	8-OAc		2.05 s

[①] Typographic error exists in the literature, with two signals for H-14β being given.

Table 2-21-25: ^{1}H NMR spectroscopic data of B1-type diterpenoid alkaloids **2-21-87**~**2-21-89**.

H	2-21-87	2-21-88	2-21-89
1	2.96~3.08 m	3.08~3.18 m	3.04 br t
2	1.90~2.11 m	1.95~2.11 m	1.90~2.08 m
	2.18~2.34 m	2.25~2.45 m	2.19~2.33 m
3	1.64~1.73 m	3.76 dd(9, 5)	1.55~1.72 m
5	2.08~2.11 m	2.05~2.16 m	2.04~2.12 m
6	3.96 m	4.02 d(6.4)	4.02 d(6.6)

Table 2-21-25 (continued)

H	2-21-87	2-21-88	2-21-89
7	2.40~2.57 m	2.90~2.08 m	2.03~2.17 m
9	2.85~2.98 m	2.87~2.91 m	2.47~2.61 m
10	2.00~2.11 m	2.05~2.15 m	2.04~2.12 m
12	2.00~2.08 m	2.05~2.15 m	2.00~2.09 m
	2.71~2.82 m	2.55~2.69 m	2.49~2.69 m
14	4.87 d(5.2)	4.87 d(5)	5.15 d(4.8)
15	2.40~2.57 m	2.40~2.55 m	2.19~2.33 m
	2.96~3.08 m	3.06 m	2.50~2.69 m
16	3.38 dd(8, 5)	3.36~3.43 m	3.26~3.39 m
17	3.00 br s	3.00 br s	3.00~3.09 m
18	3.17 m	3.51 m	3.26~3.39 m
	3.61 d(8.4)	3.64 d(8.9)	3.66 d(8.4)
19	2.40~2.57 m	2.25~2.45 m	2.19~2.33 m
	2.96~3.08 m	2.89 m	2.41~2.69 m
21	2.48~2.71 m	2.36~2.60 m	2.41~2.69 m
22	1.09 t(7)	1.10 t(7.1)	1.09 t(7.1)
1-OMe	3.26 s	3.26 s	3.27 s
6-OMe	3.15 s	3.16 s	3.26 s
16-OAc	3.52 s	3.53 s	3.39 s
18-OAc	3.28 s	3.30 s	3.30 s
3′-OMe	3.91 s	3.92 s	3.93 s
4′-OMe	3.94 s	3.94 s	3.94 s
8-OAc	1.31 s	1.33 s	
3-OH		2.09 s	
8-OH			2.25 s
13-OH	3.82 s	3.86 s	3.86 s
2′	7.61 d(1.8)	7.62 d(1.2)	7.59 d(1.7)
5′	6.89 d(8.4)	6.90 d(8.0)	6.90 d(8.4)
6′	7.71 dd(1.8, 8.4)	7.70 dd(1.2, 8)	7.69 dd(1.7, 8.4)

Table 2-21-26: ^{1}H NMR spectroscopic data of B1-type diterpenoid alkaloids **2-21-90**~**2-21-93**.

H	2-21-90	2-21-91	2-21-92	2-21-93
1	3.12 dd(8, 6)	3.10~3.13 m	3.13 br t	3.72 dd(11, 7)
2	2.03~2.17 m	1.97~2.16 m	1.81~1.95 m	*α* 2.40 m
	2.25~2.45 m	2.31~2.43 m	2.25~2.45 m	*β* 2.45 m
3	3.64~3.78 m	3.75~3.80 m	3.83 m	*β* 4.94 dd(12.5, 6)
5	2.03~2.13 m	2.06~2.16 m	2.00~2.20 m	*β* 2.62 d(6.5)
6	4.07 d(6.7)	4.04 d(6.4)	4.07 d(6.8)	*β* 4.12 dd(6.5, 0.5)
7	2.45~2.51 m	2.83 m	2.95 s	*β* 2.91 b(0.5)
9	2.51~2.57 m	2.88~2.91 m	2.45~2.60 m	*β* 2.90 m
10	2.03~2.13 m	2.10~2.16 m	2.07~2.20 m	
12	2.03~2.13 m	2.10~2.16 m	2.07~2.20 m	*α* 3.55 d(16)
	2.31~2.57 m	2.71~2.78 m	2.51~2.66 m	*β* 2.05 bd(16)
14	5.12 d(5)	4.87 d(5.2)	5.00 d(5)	*β* 5.40 d(5)
15	2.22~2.42 m	4.47 dd(6, 3)	4.52 m	*β* 4.50 dd(5, 2.5)
	2.51~2.68 m			

Table 2-21-26 (continued)

H	2-21-90	2-21-91	2-21-92	2-21-93
16	3.31~3.42 m	3.34 d(5.4)	3.22~3.29 m	*α* 3.28 d(5)
17	3.00 br s	3.10~3.13 m	3.25 s	*β* 2.96 m
18	3.64~3.78 m	3.50 d(8.9)	3.45~3.53 m	3.75 d(9)
		3.63 d(8.9)	3.61 d(8.8)	2.92 d(9)
19	2.42 d(12)	2.31~2.39 m	2.43 d(11)	2.65 d(11.5)
	2.94 d(12)	2.83~2.91 m	2.86 d(11)	2.32 d(11.5)
21	2.39~2.57 m	2.31~2.43 m	2.28~2.50 m	2.36 s
		2.66~2.84 m	2.51~2.73 m	
22	1.12 t(7)	1.09 t(6.9)	1.09 t(7)	
1-OMe	3.27 s	3.16 s	3.22 s	3.31 s
6-OMe	3.25 s	3.26 s	3.29 s	3.17 s
16-OMe				3.73 s
18-OMe				3.17 s
3-OAc				2.06 s
8-OAc		1.39 s		1.41 s
16-OAc	3.42 s	3.30 s	3.29 s	
18-OAc	3.31 s	3.75 s	3.70 s	
3′-OMe	3.93 s			
4′-OMe	3.94 s			
3-OH	2.06 s	2.73 s	1.91 s	
8-OH	2.27 s		2.48 s	
10-OH				4.30 s
13-OH	3.86 s	3.96 s	3.83 s	4.02 s
15-OH		4.39 d(3)	4.31 s	4.30 d(2.5)
2′	7.59 d(1.7)	8.00~8.04 d	7.98~8.02 m	8.08 dd(8.3, 1.5)
3′		7.41~7.49 m	7.36~7.44 m	7.47 dt(8.3, 1.5)
4′		7.54~7.63 m	7.51~7.59 m	7.59 dt(8.3, 1.5)
5′	6.90 d(8.4)	7.41~7.49 m	7.36~7.44 m	7.47 dt(8.3, 1.5)
6′	7.67 dd(1.7, 8.4)	8.00~8.06 m	7.98~8.02 m	8.08 dd(8.3, 1.5)

Table 2-21-27: ^{1}H NMR spectroscopic data of B1-type diterpenoid alkaloids **2-21-94** and **2-21-95**.

H	2-21-94	2-21-95	H	2-21-94	2-21-95
1	3.73 m	4.95 m	9	*β* 2.82 m	3.38 d(5.0)
2	*α* 2.33 m, *β* 2.03 m	2.04 m, 2.11 m	12	*α* 3.27 m	2.95 ABq(15.5)
				β 2.09 d(15)	4.34 ABq(15.5)
3	*β* 3.25 m	1.92 m	14	*β* 5.38 d(5)	6.29 d(5.0)
5	*β* 2.36 m	3.28 d(7.3)	15	*β* 4.40 dd(5.5, 3)	5.10 dd(5.2, 2.9)
6	*β* 4.03 br d(6.5)	4.35 d(7.7)	16	*α* 3.28 m	3.90 d(5.2)
7	*β* 2.89 m	3.21 s	17	*β* 2.89 m	4.60 s
18	3.63 d(9)	3.28 ABq(8.4)	10-OH	4.07 s	6.62 s or 7.16 s
	3.46 d(9)	3.77 ABq(8.4)	13-OH	4.07 s	6.62 s or 7.16 s

Table 2-21-27 (continued)

H	2-21-94	2-21-95	H	2-21-94	2-21-95
19	2.85 m	3.55 ABq(13.3)	15-OH	4.32 d(3)	5.32 d(2.9)
	2.75 m	4.37 ABq(13.3)	2′, 6′	8.04 dd(8, 1.5)	
21	2.33 br s		3′, 5′	7.47 br t(8)	
1-OMe	3.30 s		4′	7.58 dt(8, 1.5)	
6-OMe	3.16 s	3.27 s	Ar-H		7.39 t(7.3)
16-OMe	3.74 s	3.78 s			7.52 t(7.4)
18-Ome	3.29 s	3.06 s			8.26 d(7.9)
8-OAc	1.40 s	1.37 s	CHO		8.87 s
1-OH		6.55 d(3.9)			

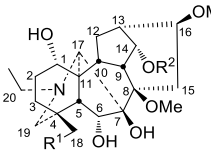
Table 2-21-28: ^{1}H NMR spectroscopic data of B1-type diterpenoid alkaloids **2-21-96**~**2-21-98**.

H	2-21-96	2-21-97	2-21-98
1	4.96 m	3.85 m	4.77 br s
2	2.09 m	1.98 m	1.98 m
3	1.92 m	1.78 m, 1.86 m	1.98 m, 2.10 m
5	3.24 d(6.7)	2.31 d(6.8)	3.23 d(7.0)
6	4.33 d(5.8)	4.19 d(6.9)	4.29 d(6.4)
7	3.18 s	3.05 s	3.22 s
9	3.39 d(5.0)	3.12 m	3.34 d(3.9)
10		2.28 m	
12	2.97 ABq(15.6)	3.77 m	2.94 ABq(14.8)
	4.35 ABq(15.6)	2.63 t(14.0)	3.45 ABq(14.8)
14	6.29 d(5.1)	5.49 d(4.6)	6.24 d(4.8)
15	5.09 dd(5.5, 3.4)	4.96 m	5.00 m
16	3.83 d(5.6)	3.77 m	3.77 d(5.7)
17	4.65 s	4.65 s	3.56 s
18	3.30 ABq(8.3)	3.44 ABq(8.1)	3.12 ABq(8.1)
	3.80 ABq(8.3)	3.69 ABq(8.1)	3.73 ABq(8.1)
19	3.51 ABq(13.3)	3.48 ABq(13.2)	2.42 ABq(10.8)
	4.32 ABq(13.3)	4.62 ABq(13.2)	3.52 ABq(10.8)
6-OMe	3.27 s	3.29 s	3.28 s
16-OMe	3.81 s	3.78 s	3.75 s
18-OMe	3.06 s	3.12 s	3.08 s
N-OAc	2.90 s	2.80 s	
8-OAc	1.38 s	1.44 s	1.41 s
1-OH	6.57 d(3.9)	6.52 d(3.8)	6.52 br s
OH	6.60 s, 7.12 s		
15-OH	5.33 d(3.2)	5.30 d(3.0)	5.18 d(2.4)
Ar-H	7.38 t(7.7)	7.39 t(7.5)	7.35 t(7.8)
	7.51 t(7.3)	7.70 t(7.6)	7.48 t(7.3)
	8.25 d(7.7)	8.25 d(8.3)	8.23 d(7.7)

2.21.4 B2-type diterpenoid alkaloids

Table 2-21-29: Cos, MFs, and TSs of B2-type diterpenoid alkaloids **2-21-99**~**2-21-132**.

No.	Compounds	MFs	Test solvents	References
2-21-99	18-demethylpubescenine	$C_{25}H_{39}NO_8$	$CDCl_3$	[608]
2-21-100	18-demethoxypubescenine	$C_{25}H_{39}NO_7$	$CDCl_3$	[609]
2-21-101	8-*O*-methylconsolarine	$C_{23}H_{37}NO_6$	$CDCl_3$	[571]
2-21-102	18-*O*-benzoyl-14-*O*-deacetyl-18-*O*-demethyl-pubescenine	$C_{30}H_{41}NO_8$	$CDCl_3$	[571]
2-21-103	isodeleatine	$C_{24}H_{37}NO_6$	$CDCl_3$	[610]
2-21-104	giraldine D	$C_{24}H_{37}NO_6$	$CDCl_3$	[611]
2-21-105	giraldine E	$C_{25}H_{39}NO_7$	$CDCl_3$	[611]
2-21-106	giraldine F	$C_{23}H_{33}NO_6$	$CDCl_3$	[611]
2-21-107	18-*O*-methyldelterine	$C_{26}H_{43}NO_8$	$CDCl_3$	[612]
2-21-108	10-hydroxynudicaulidine	$C_{24}H_{39}NO_7$	$CDCl_3$	[612]
2-21-109	14-*O*-acetyltakaosamine	$C_{25}H_{39}NO_8$	$CDCl_3$	[609]
2-21-110	14-*O*-benzoyltakaosamine	$C_{30}H_{41}NO_8$	$CDCl_3$	[571]
2-21-111	1–*O*-demethyltricornine	$C_{26}H_{41}NO_8$	$CDCl_3$	[571]
2-21-112	jiufengdine	$C_{36}H_{52}N_2O_9$	$CDCl_3$	[613]
2-21-113	jiufengtine	$C_{30}H_{42}N_2O_8$	$CDCl_3$	[613]
2-21-114	trifoliolasine A	$C_{35}H_{50}N_2O_9$	$CDCl_3$	[614]
2-21-115	bearline	$C_{37}H_{48}N_2O_{11}$	$CDCl_3$	[615]
2-21-116	14-acetylbearline	$C_{39}H_{50}N_2O_{12}$	$CDCl_3$	[615]
2-21-117	16-deacetylgeyerline	$C_{36}H_{48}N_2O_{10}$	$CDCl_3$	[615]
2-21-118	10-hydroxymethyllycaconitine	$C_{37}H_{50}N_2O_{11}$	$CDCl_3$	[612]
2-21-119	dehydrodeltatsine	$C_{25}H_{39}NO_7$	$CDCl_3$	[609]
2-21-120	1-*O*,19-didehydrotakaosamine	$C_{23}H_{35}NO_7$	$CDCl_3$	[571]
2-21-121	olivimine	$C_{24}H_{37}NO_7$	$CDCl_3$	[576]
2-21-122	acovulparine	$C_{23}H_{35}NO_7$	$CDCl_3$	[616]
2-21-123	olividine	$C_{26}H_{39}NO_8$	$CDCl_3$	[576]
2-21-124	consolidine	$C_{25}H_{41}NO_7$	$CDCl_3$	[617]
2-21-125	finetiadine	$C_{38}H_{52}N_2O_{12}$	$CDCl_3$	[618]
2-21-126	blacknine	$C_{23}H_{35}NO_6$	$CDCl_3$	[619]
2-21-127	blacknidine	$C_{23}H_{37}NO_5$	$CDCl_3$	[619]
2-21-128	siwanine A	$C_{27}H_{39}NO_8$	$CDCl_3$	[620]
2-21-129	siwanine B	$C_{26}H_{37}NO_8$	$CDCl_3$	[620]
2-21-130	siwanine C	$C_{26}H_{37}NO_7$	$CDCl_3$	[620]
2-21-131	siwanine D	$C_{25}H_{35}NO_8$	$CDCl_3$	[620]
2-21-132	tatsiensine	$C_{27}H_{39}NO_7$	$CDCl_3$	[620]



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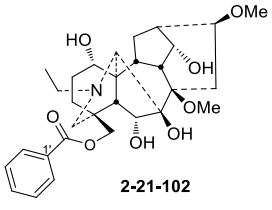
2-21-99	OH	Ac
2-21-100	H	Ac
2-21-101	H	H



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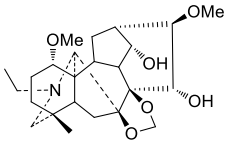

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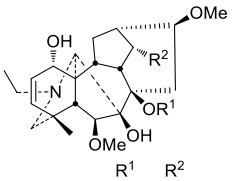
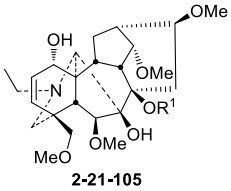


	R^1	R^2
2-21-104	Me	OH
2-21-106	H	=O



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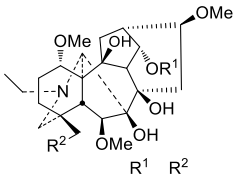
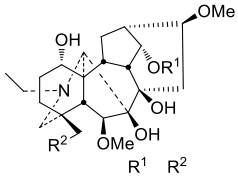
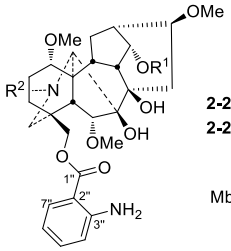


	R^1	R^2
2-21-107	Me	OMe
2-21-108	H	H

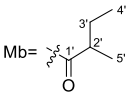


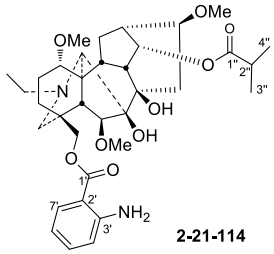
	R^1	R^2
2-21-109	Ac	OH
2-21-110	Bz	OH
2-21-111	Me	OAc



	R^1	R^2
2-21-112	Mb	Et
2-21-113	Me	H

Mb=





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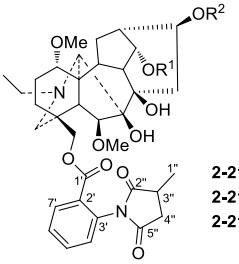
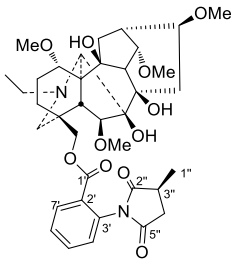


	R^1	R^2
2-21-115	H	Ac
2-21-116	Ac	Ac
2-21-117	Me	H



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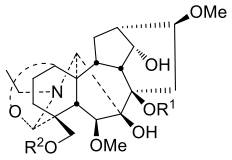
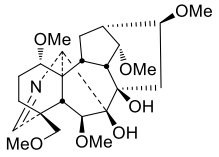


	R^1	R^2
2-21-119	Me	Me
2-21-120	H	H



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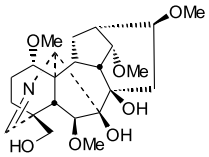

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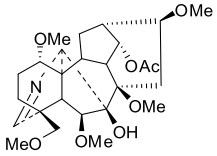

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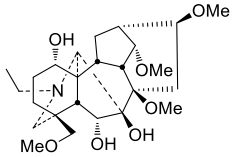

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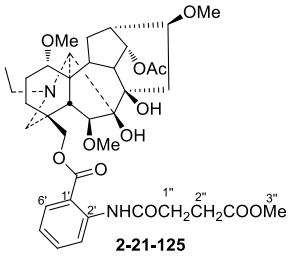

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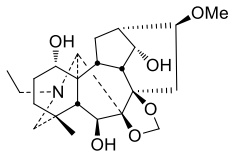

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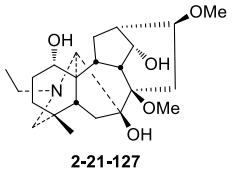
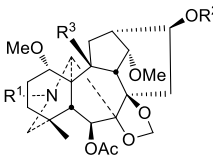


2-21-127



	R^1	R^2	R^3
2-21-128	Et	Me	OH
2-21-129	Et	H	OH
2-21-130	Me	Me	H
2-21-131	Me	H	OH
2-21-132	Et	Me	H

Table 2-21-30: ^{1}H NMR spectroscopic data of B2-type diterpenoid alkaloids **2-21-99**~**2-21-103**.

H	2-21-99	2-21-100	2-21-101	2-21-102	2-21-103
1	3.66 br s	3.63 br s ($W_{1/2}$ = 6.9)	3.66 br s ($W_{1/2}$ = 7.4)	3.71 m	3.04 dd(9.6, 7.6)
2α	1.60 m	1.55 m	1.54 m	1.71 m	2.13 m
2β	1.49 m	1.52 m	1.52 m	1.54 dddd(14.6, 14.6, 6.1, 3.1)	2.02 m
3α	1.62 m	1.72 ddd(12.7, 12.7, 5.0)	1.75 m	1.67 m	1.58 m

Table 2-21-30 (continued)

H	2-21-99	2-21-100	2-21-101	2-21-102	2-21-103
3β	1.66 m	1.44 m	1.46 ddd(13.2, 3.6, 3.6)	1.78 m	1.20 m
5	2.16 d(6.9)	1.92 d(6.4)	1.91 d(6.8)	2.1 d(6.1)	1.26 s
6	4.48 d(6.9)	4.50 dd(6.4, 6.4)	4.53 d(6.8)	4.56 m	1.81 m, 1.77 m
9	2.30 t(6.0)	2.28 dd(7.0, 5.1)	2.16 dd(6.9, 4.5)	2.11 m	3.65 m
10	1.92 m	1.92 m	1.87 ddd(11.9, 6.9, 5.1)	1.89 ddd(11.6, 6.8, 5.1)	1.95 m
12α	1.81 dd(14.3, 4.6)	1.81 dd(14.2, 4.6)	1.75 dd(14.4, 5.2)	1.76 dd(14.4, 5.0)	2.51 dd(9.8, 8.4)
12β	2.07 m	2.07 ddd(14.2, 11.4, 7.3)	2.04 ddd(14.4, 11.0, 7.3)	2.05 m	2.04 m
13	2.48 dd(7.2, 4.6)	2.48 dd(7.3, 4.6)	2.31 dd(7.6, 4.5)	2.34 dd(7.3, 4.7)	2.40 m
14	4.76 t(4.5)	4.77 t(4.6)	4.05 t(4.5)	4.07 t(4.7)	4.10 t(4.5)
15	2.64 dd(14.6, 8.5)	2.62 dd(14.5, 8.6)	2.68 dd(15.3, 8.9)	2.76 dd(15.1, 8.8)	4.19 d(6.0)
	1.97 dd(14.6, 8.1)	1.96 dd(14.5, 8.6)	1.77 dd(15.3, 6.7)	2.05 dd(15.1, 6.9)	
16	3.45 t(8.5)	3.43 t(8.6)	3.48 t(5.5)	3.48 t(6.0)	3.45 dd(10.8, 6.0)
17	2.75 s	2.72 s	2.79 s	2.81 s	3.28 s
18	3.89 d(10.6)	1.27 s	1.29 s	5.10 d(10.5)	0.93 s
	3.54 d(10.6)			4.21 d(10.5)	
19	2.80 d(11.0)	2.71 d(11.0)	2.39 d(11.0)	2.50 d(10.3)	2.69 m
	2.35 d(11.0)	2.34 d(11.0)	2.78 d(11.0)	3.01 d(10.3)	2.28 m
20	3.02 dq(12.7, 7.3)	2.98 dq(14.2, 7.2)	3.04 dq(14.0, 7.0)	3.07 dq(13.9, 7.3)	2.64 m
	2.91 dq(12.7, 7.3)	2.88 dq(14.2, 7.2)	2.91 dq(14.0, 7.0)	2.97 dq(13.9, 7.3)	2.76 m
21	1.13 t(7.2)	1.12 t(7.2)	1.14 t(7.2)	1.18 t(7.2)	1.03 t(7.2)
2′				8.09 d(7.6)	
3′				7.50 t(7.7)	
4′				7.62 t(7.3)	
5′				7.50 t(7.7)	
6′				8.09 d(7.6)	
1-OMe					3.23 s
8-OMe	3.42 s	3.41 s	3.45 s	3.49 s	
16-OMe	3.39 s	3.38 s	3.41 s	3.45 s	3.35 s
7,8-OCH_2O					5.17 s„5.07 s
14-OAc	2.05 s	2.05 s			

Table 2-21-31: ^{1}H NMR spectroscopic data of B2-type diterpenoid alkaloids **2-21-104**~**2-21-108**.

H	2-21-104	2-21-105	2-21-106	2-21-107	2-21-108
1	3.70 d(4.8)	3.75 d(4.4)	3.87 d(4.8)	3.57 dd(7.6, 10.0)	3.68 dd(7.4, 10.2)
2	5.77 dd(4.8, 9.2)	5.82 dd(4.4, 9.6)	5.84 dd(4.8, 9.2)	2.08 m, 2.11 m	2.02 m, 2.15 m
3	5.66 d(9.2)	5.86 d(9.6)	5.69 d(9.2)	1.58 m, 1.60 m	1.22 m, 1.60 m
5	3.49 s	2.06 d(1.6)	3.08 d(2.0)	1.94 br s	1.65 br s
6	3.69 s	3.93 s	3.93 s	3.91 br s	3.89 s
9	3.31 m	2.98 m	2.20 m	2.86 d(4.6)	2.97 d(4.9)
10	2.28 m	2.00 m	2.23 m		
12	*α* 1.92 m *β* 2.08 m	*α* 1.92 m *β* 2.20 dd(4.0, 13.2)	2.50 m	1.69 dd(8.3, 15.8) 3.05 d(15.8)	1.70 dd(8.6, 16.0) 2.52 d(16.0)
13	2.04 m	2.40 m	2.44 m	2.48 dd(4.6, 8.3)	2.53 dd(4.9, 8.6)
14	4.00 t(4.8)	4.80 t(4.8)	–	4.10 t(4.6)	4.61 t(4.9)
15	*α* 2.67 dd(18.8, 7.6) *β* 1.83 m	*α* 2.64 dd(8.8, 14.8) *β* 1.74 dd(8.8, 14.8)	2.72 dd(15.6, 8.0) 1.35 m	1.71 dd(5.8, 15.3) 2.64 dd(9.0, 15.3)	1.75 dd(2.1, 17.4) 2.62 dd(8.7, 17.4)
16	3.40 m	3.30 t(8.8)	3.86 m	3.17 dd(5.8, 10.1)	3.43 dd(3.2, 8.7)
17	2.86 s	2.84 d(2.8)	3.25 s	2.82 br s	3.05 br s
18	1.07 s	3.17 ABq(6.8) 3.47 ABq(6.8)	1.13 s	2.98 d(9.0) 3.40 d(9.0)	0.99 s
19	2.43 ABq(11.6) 2.47 ABq(11.6)	2.33 ABq(11.2) 2.41 ABq(11.2)	2.40 m 2.52 m	2.59 d(11.6) 2.84 d(12.0)	2.46 d(12.0) 2.65 d(12.0)
20	2.88 m 3.00 m	2.80 m 3.02 m	3.02 m 2.88 m	2.77 dq(7.1, 12.8) 2.90 dq(7.1, 12.8)	2.81 dq(6.7, 12.8) 2.90 dq(6.8, 12.8)
21	1.06 t(7.2)	1.06 t(7.2)	1.09 t(7.2)	1.05 t(7.1)	1.05 t(7.1)
OMe	3.48 s(6-OMe) 3.41 s(8-OMe) 3.39 s(16-OMe)	3.38 s(6-OMe) 3.42 s(14-OMe) 3.36 s(16-OMe) 3.36 s(18-OMe)	3.37 s(6-OMe) 3.35 s(16-OMe)	3.25 s(1-OMe) 3.42 s(6-OMe) 3.44 s(14-OMe) 3.33 s(16-OMe) 3.30 s(18-OMe)	3.24 s(1-OMe) 3.42 s(6-OMe) 3.35 s(16-OMe)

Table 2-21-32: ^{1}H NMR spectroscopic data of B2-type diterpenoid alkaloids **2-21-109**~**2-21-113**.

H	2-21-109	2-21-110	2-21-111	2-21-112	2-21-113
1	3.68 br s ($W_{1/2}$ = 10.2)	3.74 br s ($W_{1/2}$ = 8.3)	3.71 br s ($W_{1/2}$ = 6.0)	3.01 dd(9.6, 6.8)	3.25 m
2	*α* 1.66 m *β* 1.48 dddd(13.9, 13.9, 6.6, 3.2)	*α* 1.67 m *β* 1.53 dddd(13.9, 13.9, 6.2, 3.2)	*α* 1.67 m *β* 1.49 dddd(13.8, 13.8, 4.3, 2.5)	*α* 2.08 m *β* 2.16 m	*α* 1.77 m *β* 2.01 m
3	*α* 1.63 m, *β* 1.93 m	*α* 1.70 m, *β* 1.96 m	*α* 1.73 m, *β* 1.83 m	*α* 1.79 m, *β* 1.72 m	1.81 m
5	1.85 d(1.9)	1.91 d(1.8)	1.82 d(1.8)	1.74 m	1.95 br s

Table 2-21-32 (continued)

H	2-21-109	2-21-110	2-21-111	2-21-112	2-21-113
6	3.96 s	4.00 s	3.96 s	3.91 s	4.01 br s
9	3.09 dd(7.1, 4.7)	3.21 dd(7.0, 4.7)	2.96 dd(6.8, 4.5)	3.15 dd(6.8, 4.8)	3.09 dd(6.8, 4.8)
10	2.01 ddd(11.9, 7.1, 4.9)	2.10 ddd(11.8, 7.0, 4.7)	1.98 ddd(11.7, 6.8, 4.5)	2.04 m	2.04 m
12	*α* 1.71 dd(14.2, 4.7)	*α* 1.80 dd(14.2, 4.7)	*α* 1.74 dd(13.1, 4.5)	*α* 2.46 m	1.78 m
	β 2.09 ddd(14.2, 11.9, 7.6)	*β* 2.21 ddd(14.2, 11.8, 7.5)	*β* 2.07 ddd(13.1, 11.7, 7.4)	*β* 1.89 m	
13	2.45 dd(7.6, 4.7)	2.63 dd(7.5, 4.7)	2.41 dd(7.4, 4.5)	2.42 m	2.37 dd(6.8, 4.8)
14	4.77 t(4.7)	5.09 t(4.7)	3.64 t(4.5)	4.79 t(5.0)	3.62 t(4.8)
15	2.67 dd(15.0, 8.8)	*α* 2.78 dd(15.1, 8.9)	*α* 2.63 dd(14.6, 8.3)	1.59 dd(15.2, 6.4)	*α* 2.66 dd(14.8, 8.8)
	1.60 dd(15.0, 8.4)	*β* 1.77 dd(15.1, 7.7)	*β* 1.77 dd(14.6, 8.3)	2.64 dd(15.2, 9.2)	*β* 1.75 m
16	3.31 m	3.41 m	3.30 t(8.3)	3.26	3.22 m
17	2.79 d(1.9)	2.87 d(1.8)	2.84 d(1.8)	2.96 d(1.0)	3.05 d(2)
18	3.64 d(10.5)	3.66 d(10.4)	4.00 d(11.0)	4.10 d(11.2)	4.11 d(11.2)
	3.37 d(10.5)	3.40 d(10.4)	3.97 d(11.0)	4.15 d(11.2)	4.15 d(11.2)
19	2.40 d(11.8)	2.46 d(11.6)	2.53 d(11.5)	2.43 (ov)	2.85 d(13.2)
	2.43 d(11.8)	2.44 d(11.6)	2.50 d(11.5)	2.73 d(11.6)	2.95 d(13.2)
20	2.95 dq(12.8, 7.2)	2.99 dq(12.8, 7.2)	2.99 dq(12.8, 7.2)	2.82 m	
	2.81 dq(12.8, 7.2)	2.85 dq(12.8, 7.2)	2.86 dq(12.8, 7.2)	2.93 m	
21	1.08 t(7.2)	1.12 t(7.2)	1.12 t(7.2)	1.06 t(7.2)	
2′		8.11 d(7.5)		2.36 m	
3′		7.43 t(7.5)		1.48 m	
				1.76 m	
4′		7.53 t(7.5)		0.90 t(7.2)	
5′		7.43 t(7.5)		1.15 d(7.2)	
6′		8.11 d(7.5)			
4″				6.67 br d(8.4)	6.69 br d(8.4)
5″				7.29 td(8.4, 1.2)	7.29 td(8.0, 1.2)
6″				6.67 td(8.2, 1.2)	6.64 td(8.4, 1.2)
7″				7.79 dd(8.2, 1.6)	7.76 dd(8.0, 1.6)
OMe	3.37 s(6-OMe)	3.39 s(6-OMe)	3.32 s(6-OMe)	3.26 s(1-OMe)	3.31 s(1-OMe)
	3.32 s(16-OMe)	3.34 s(16-OMe)	3.42 s(14-OMe)	3.37 s(6-OMe)	3.37 s(6-OMe)
			3.37 s(16-OMe)	3.29 s(16-OMe)	3.42 s(14-OMe)
					3.34 s(16-OMe)
OAc	2.04 s(14-OAc)		2.11 s(18-OAc)		
3″-NH_2				5.73 br s	

Table 2-21-33: ^{1}H NMR spectroscopic data of B2-type diterpenoid alkaloids **2-21-114**~**2-21-118**.

H	2-21-114	2-21-115	2-21-116	2-21-117	2-21-118
1	3.01 dd(7.2, 5.2)	2.95 m	2.98 m	2.97 m	3.59 t(7.8)
2	*α* 2.08 m, *β* 2.16 m	2.02 m, 2.19 m	2.07 m, 2.15 m	2.11 m, 2.19 m	2.12 m, 2.19 m
3	*α* 1.59 dd(14.4, 6.0)	1.57 m	1.57 m	1.55 m	1.54 m
	β 1.74 m	1.70 m	1.73 m	1.75 m	1.75 m
5	1.81 s	1.78 br s	1.76 br s	1.78 br s	1.99 br s
6	3.91 s	3.89 br s	3.87 br s	3.86 br s	3.91 br s
9	3.17 dd(10.0, 7.2)	3.08 m	3.24 m	3.23 m	2.89 d(4.6)
10	2.04 m	1.96 m	1.98 m	1.80 m	
12	*α* 2.46 m	1.92 m	1.91 m	1.78 m	1.70 dd(8.0, 15.6)
	β 1.89 m	2.48 m	1.97 m	1.86 m	3.09 d(15.6)
13	2.42 m	2.30 t(5.2)	2.37 t(5.6)	2.37 br s	2.49 dd(4.8, 7.2)
14	4.80 t(4.8)	4.10 t(4.2)	4.76 t(4.8)	3.67 m	4.12 t(4.6)
15	*α* 2.64 dd(13.6, 9.2)	1.63 dd(5.4, 16.8)	1.56 dd(5.6, 16.8)	1.72 m	1.72 dd(8.0, 15.0)
	β 1.56 dd(13.6, 5.2)	2.74 dd(9.2, 16.8)	2.71 dd(9.2, 16.8)	2.71 m	2.67 dd(8.0, 15.0)
16	3.26 (ov)	4.89 dd(5.4, 9.2)	4.86 dd(5.6, 9.2)	3.67 m	3.18 t(8.0)
17	2.96 d(2.4)	3.02 m	3.02 d(2.0)	3.06 m	2.86 br s
18	4.10 d(11.6)	4.09 d(11.2)	4.08 br s	4.07 d(11.2)	4.00 d(11.2)
	4.15 d(11.6)	4.12 d(11.2)		4.14 d(11.2)	4.20 d(11.2)
19	2.43 ABq(10.4)	2.47 d(11.4)	2.43 d(12.0)	2.43 d(11.6)	2.48 d(12.6)
	2.73 ABq(10.4)	2.74 d(11.4)	2.70 d(12.0)	2.72 d(11.6)	2.70 d(12.6)
20	2.82 m	2.82 m	2.79 m	2.82 m	2.82 m
	2.93 m	2.96 m	2.94 m	2.97 m	2.96 m
21	1.08 t(7.2)	1.07 t(7.1)	1.06 t(7.2)	1.06 t(7.2)	1.07 t(7.5)
4′	6.67 dd(8.0, 1.2)	7.25 dd(1.0, 8.0)	7.27 dd(1.0, 8.0)	7.28 dd(1.0, 8.0)	7.27 dd(1.0, 7.6)
5′	6.67 ddd(8.0, 7.2, 1.2)	7.55 dt(2.0, 9.2)	7.55 dt(2.0, 9.2)	7.53 dt(2.0, 9.2)	7.68 dt(1.0, 7.6)
6′	7.29 ddd(8.0, 7.6, 1.6)	7.69 dt(2.0, 9.2)	7.69 dt(2.0, 9.2)	7.69 dt(2.0, 9.2)	7.54 dt(1.0, 7.6)
7′	7.80 dd(7.8, 1.2)	8.04 dd(1.0, 8.0)	8.04 dd(1.0, 8.0)	8.05 dd(1.0, 8.0)	8.04 dd(1.0, 7.6)
1″		1.46 br s	1.47 br s	1.47 br s	1.45 d(7.0)
2″	2.57 m				
3″	1.18 d(7.2)	3.08 br s	3.04 br s	3.06 br s	3.05 m
4″	1.18 d(7.2)	2.50 m	2.51 m	2.54 m	2.53 m, 3.11 m
1-OMe	3.26 s	3.25 s	3.24 s	3.25 s	3.21 s
6-OMe	3.39 s	3.37 s	3.36 s	3.39 s	3.37 s
14-OMe					3.43 s
16-OMe	3.30 s			3.45 s	3.34 s
16-OAc		2.06 s	2.08 s		
14-OAc			2.02 s		

Table 2-21-34: ^{1}H NMR spectroscopic data of B2-type diterpenoid alkaloids **2-21-119**~**2-21-123**.

H	2-21-119	2-21-120	2-21-121	2-21-122	2-21-123
1	3.71 d(5.2)	3.70 d(5.1)	3.22 t(3.8)	3.24 t(3.8)	3.20 t(4.6)
2α	1.75 m	1.80 ddd(12.6, 9.2, 5.1)	1.77 (ov)	1.71 m	1.68 (ov)
2β	1.47 m	1.51 ddd(12.6, 9.2, 9.2)	1.36 dddd(12.6, 12.6, 10.8, 3.4)	1.42 m	1.48 dddd(12.0, 11.6, 10.5, 3.4)
3α	1.66 m	1.60 dd(12.0, 9.2)	1.55 ddd(11.4, 11.4, 6.5)	1.73 m	1.66 (ov)
3β	1.68 m	1.68 ddd(12.0, 9.2, 9.2)	1.80 ddd(13.4, 8.7, 3.0)	1.64 m	1.66 (ov)
5	1.56 br s ($W_{1/2}$ = 5.3)	1.58 br s ($W_{1/2}$ = 4.1)	1.83 s	1.80 s	1.69 s
6	3.57 d(1.9)	3.97 d(1.4)	3.78 s	3.88 br s	3.66 s
9	2.91 dd(4.8, 7.0)	2.73 dd(4.8, 6.9)	2.38 dd(4.6, 7.3)	2.85 t(5.5)	3.14 dd(6.8, 4.7)
10	2.02 ddd(11.5, 7.0, 7.0)	1.96 ddd(11.2, 6.9, 6.9)	1.94 ddd(11.7, 11.7, 6.6)	1.99 m	2.03 (ov)
12α	1.09 dd(14.0, 7.0)	1.08 dd(14.0, 6.9)	1.47 dd(13.5, 4.8)	1.48 dd(13.0, 4.4)	1.47 br d(7.9)
12β	1.84 ddd(14.0, 11.5, 7.8)	1.85 ddd(14.0, 11.2, 6.9)	2.02 ddd(11.8, 11.8, 7.5)	2.03 m	2.02 (ov)
13	2.43 ddd(7.8, 4.8, 1.7)	2.46 m	2.83 t(6.0)	2.41 dd(6.5, 4.4)	2.55 t(6.3)
14	4.08 dt(4.8, 5.5) 3.73 d(5.5)	4.10 t(4.8)	3.65 t(4.5)	3.66 t(4.4)	4.81 t(3.9)
15	2.66 dd(16.4, 8.7)	2.68 dd(17.1, 8.8)	2.86 dd(14.8, 8.6)	2.80 dd(15.0, 8.5)	2.79 dd(15.7, 8.7)
	2.14 dd(16.4, 4.7)	1.81 dd(17.1, 8.1)	1.74 dd(14.5, 7.7)	1.70 m	2.00 dd(16.0, 6.4)
16	3.34 dd(4.7, 8.7)	3.39 m	3.28 t(10.1)	3.27 t(8.5)	3.39 t(9.0)
17	2.40 d(2.9)	2.60 d(2.7)	3.78 s	3.76 br s	3.87 d(2.4)
18	3.37 d(9.1)	3.69 d(10.6)	3.63 d(9.3)	3.81 d(10.8)	3.50 d(9.3)
	3.24 d(9.1)	3.62 d(10.6)	3.28 d(9.3)	3.77 d(10.8)	3.44 d(9.3)
19	3.98 s	4.07 s	7.29 s	7.46 br s	7.52 s
20	3.00 dq(12.3, 7.2)	2.96 dq(12.3, 7.0)			
	2.56 dq(12.3, 7.2)	2.71 dq(12.3, 7.0)			
21	1.10 t(7.2)	1.10 t(7.0)			
OMe	3.45 s(6-OMe)	3.41 s(6-OMe)	3.15 s(1-OMe)	3.16 s(1-OMe)	3.15 s(1-OMe)
	3.59 s(8-OMe)	3.37 s(16-OMe)	3.38 s(6-OMe)	3.44 s(6-OMe)	3.40 s(6-OMe)
	3.40 s(16-OMe)		3.43 s(14-OMe)	3.42 s(14-OMe)	3.50 s(8-OMe)
	3.36 s(18-OMe)		3.35 s(16-OMe)	3.36 s(16-OMe)	3.33 s(16-OMe)
			3.34 s(18-OMe)		3.39 s(18-OMe)
14-OAc					2.05 s
8-OH			4.15 s		

Table 2-21-35: ^{1}H NMR spectroscopic data of B2-type diterpenoid alkaloids **2-21-124**~**2-21-127**.

H	2-21-124	2-21-125	2-21-126	2-21-127
1	3.58 br s	3.05	3.75 br s	3.65 br m
2	1.48 m	2.20	1.29 m, 1.65 m	1.59 m, 1.76 m
3	*α* 1.70 m, *β* 1.56 m	1.55, 1.73	1.52 m, 1.79 m	1.52 m, 1.67 m
5	2.05 m	1.75	1.47 br s	1.54 br s
6	4.43 br s	3.90	4.27 br s	1.65 m
9	2.15 d(6.5)	3.23	3.61 br s	2.35 m
10	1.78 m	2.05	2.17 m	1.85 m
12	*α* 1.98 m, *β* 1.75 m	1.90, 2.50		1.90 m, 2.03 m
13	2.38 m	2.40	1.21 m, 2.18 m	2.33 m
14	3.46 t(4.2)	4.75 t(4.8), 2.06 (OAc)	4.20 br t	4.03 br t
15	*α* 2.50 m, *β* 2.08 m	1.55, 2.65	1.82 m, 1.90 m	1.90 m, 2.87 m
16	3.35 m	3.25	3.40 m	3.45 m
17	2.69 s	2.95	3.07 br s	2.76 br s
18	3.61 AB(7.5) 3.38 AB(7.5)	4.15	1.05 s	0.92 s
19	*α* 2.88 AB(11) *β* 2.36 AB(11)	2.45, 2.75	2.32 AB(11.6) 2.55 AB(11.6)	2.39 AB(11.5) 2.64 AB(11.5)
20	2.83 m, 2.92 m	2.80	2.75 m	2.94 m
21	1.06 t	1.07 t(7.2)	1.12 t(7.2)	1.12 t(7.1)
2′		8.70 d(8.0)		
3′		7.56 t(8.0)		
4′		7.13 t(8.0)		
5′		7.96 d(8.0)		
1″		2.74		
2″		2.74		
3″		3.70		
OMe	3.38 s(8-OMe) 3.20 s(14-OMe) 3.35 s(16-OMe) 3.31 s(18-OMe)	3.26 (1-OMe) 3.37 (6-OMe) 3.33 (16-OMe)	3.45 s(16-OMe)	3.45 s(8-OMe) 3.40 s(16-OMe)
OCH_2O			5.09 s, 5.15 s	
OH			2.85 s(6-OH) 1.65 br s(14-OH)	5.98 s(1-OH) 1.25 br s(7-OH) 1.25 br s(14-OH)

Table 2-21-36: ^{1}H NMR spectroscopic data of B2-type diterpenoid alkaloids **2-21-128**~**2-21-132**.

H	2-21-128	2-21-129	2-21-130	2-21-131	2-21-132
1	3.80 d(4.0)	3.84 d(4.0)	3.86 d(4.0)	3.84 d(4.0)	3.45 d(4.0)
2	5.99 dd(4.0, 10.0)	5.99 dd(4.0, 10.0)	6.01 dd(4.0, 10.0)	5.98 dd(4.0, 10.0)	5.97 dd(4.0, 10.0)
3	5.47 d(10.0)	5.68 d(10.0)	5.69 d(10.0)	5.65 d(10.0)	5.68 d(10.0)
5	1.73 s	1.86 s	1.58 s	1.62 s	1.54 s
6	5.47 br s	5.47 br s	5.50 br s	5.47 br s	5.42 br s
9	3.20 m	3.49 m	3.50 m	3.46 m	3.56 m

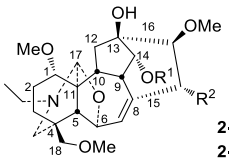
Table 2-21-36 (continued)

H	2-21-128	2-21-129	2-21-130	2-21-131	2-21-132
10			2.10 m		2.06 m
12	2.60 m, 1.80 m	2.70 m, 1.73 m	1.86 m, 2.52 m	1.73 m, 2.59 m	1.93 m, 2.25 m
13	2.50 m	2.49 m	2.60 m	2.49 m	2.40 m
14	4.13 t(4.8)	4.24 t(4.8)	4.26 t(4.8)	4.25 t(4.8)	3.71 t(4.8)
15	1.90 m, 2.53 m	2.66 m, 1.81 m	1.76 m, 2.62 m	1.82 m, 2.70 m	1.87 m, 2.47 m
16	2.97 m	3.65 m	3.64 m	3.64 m	3.26 m
17	3.19 s	3.15 s	2.99 s	3.15 s	3.10 s
18	1.00 s	0.99 s	1.00 s	0.99 s	1.00 s
19	2.47 m, 2.85 m	2.47 m, 2.65 m	2.50 m, 2.66 m	2.48 m, 2.67 m	2.52 m, 2.75 m
20	2.55	2.56	2.53 s	2.55 s	2.55
21	1.07 t(7.2)	1.07 t(7.2)			1.07 t(7.2)
1-OMe	3.33 s	3.32 s	3.35 s	3.31 s	3.31 s
14-OMe	3.44 s	3.47 s	3.48 s	3.45 s	3.41 s
16-OMe	3.34 s		3.36 s		3.34 s
6-OAc	2.08 s	2.07 s	2.10 s	2.05 s	2.05 s
OCH_2O	4.94, 4.98	4.91, 4.97	4.91, 4.99	4.93, 4.98	4.91, 4.98

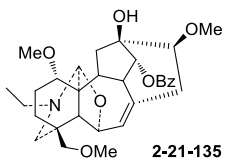
2.21.5 B5-type diterpenoid alkaloids

Table 2-21-37: Cos, MFs, and TSs of B5-type diterpenoid alkaloids **2-21-133**~**2-21-135**.

No.	Compounds	MFs	Test solvents	References
2-21-133	beiwudine	$C_{31}H_{41}NO_8$	$CDCl_3$	[621]
2-21-134	francheline	$C_{24}H_{37}NO_6$	$CDCl_3$	[591]
2-21-135	13-hydroxyfranchetine	$C_{31}H_{41}NO_7$	$CDCl_3$	[589]



	R^1	R^2
2-21-133	Bz	OH
2-21-134	H	H



2-21-135

Table 2-21-38: ^{1}H NMR spectroscopic data of B5-type diterpenoid alkaloids **2-21-133**~**2-21-135**.

H	2-21-133	2-21-134	2-21-135
1	3.30 m	3.24 dd(10.8, 6.4)	3.36 m
2	1.80 m, 2.40 m	α 1.93 m, β 2.48 m	2.41 m, 1.95 m
3	1.50 m	1.52 ddd(12, 4.4, 2.4)	1.76 dd(14,3)
	1.70 m	1.77 ddd(13.6, 4.8, 2.0)	1.55 m

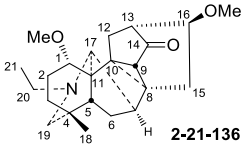
Table 2-21-38 (continued)

H	2-21-133	2-21-134	2-21-135
5	2.29 s	2.21 s	2.25 s
6	4.55 d(6.4)	4.39 d(6.0)	4.40 d(6)
7	6.08 d(6.4)	5.71 d(5.6)	5.76 d(6)
9	3.10 m	2.83 br s	3.20 br s
10	2.65 m	2.42 m	2.66 m
12	2.00 m	α 2.00 m, β 1.93 m	2.12 t(11), 2.01 m
14	5.04 br s($W_{1/2}$ = 4.6)	4.04 br s	5.06 br s
15	5.04 d(6)	3.09 m, 2.59 m	3.90 dd(12, 8), 2.64 m
16	2.85 d	3.31 m	3.35 m
17	4.37 s	4.32 br s	4.37 s
18	3.02 ABq(9.2), 3.25 ABq(9.2)	3.06 ABq(9.2), 3.16 ABq(9.2)	3.15 d(9), 3.04 d(9)
19	2.03 (ov), 2.35 (ov)	α 2.44 (11.0), β 2.04 (11.0)	2.44 m, 2.04 m
20	2.35 m, 2.55 m	2.42 m, 2.60 m	2.60 m, 2.41 m
21	1.00 t(7.1)	0.99 t(7.2)	1.01 t(7)
2′/6′	8.07 d(7)		8.07 d(8)
3′/5′	7.45 d(7.2)		7.45 t(8)
4′	7.57 d(7.2)		7.57 t(8)
1-OMe	3.30 s	3.31 s	3.37 s
16-OMe	3.67 s	3.42 s	3.47 s
18-OMe	3.26 s	3.29 s	3.28 s

2.21.6 B6-type diterpenoid alkaloids

Table 2-21-39: Co, MF, and TS of B6-type diterpenoid alkaloid **2-21-136**.

No.	Compound	MF	Test solvent	Reference
2-21-136	vilmoraconitine	$C_{23}H_{33}NO_3$	$CDCl_3$	[622]



2-21-136

Table 2-21-40: ^{1}H NMR spectroscopic data of B6-type diterpenoid alkaloid **2-21-136**.

H	2-21-136	H	2-21-136
1	3.43 dd(10.8, 6.3)	15	2.25 dd(14.7, 6.8), 2.01 dd(14.7, 2.9)
2	2.18~2.20 m, 1.97~2.00 m	16	3.60~3.63 m

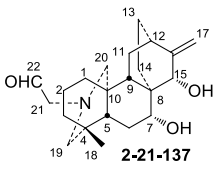
Table 2-21-40 (continued)

H	2-21-136	H	2-21-136
3	1.58~1.62 m, 1.15~1.19 m	17	3.50 br s
5	1.21 d(6.7)	18	0.73 s
6	1.37~1.42 m, 1.07~1.11 m	19	2.49 d(11.5), 2.00~2.23 (ov)
7	2.22~2.23	20	2.58~2.64 m, 2.34~2.38 m
9	2.12 s	21	1.02 t(7.2)
12	2.15~2.16 (ov)	1-OMe	3.34 s
13	2.46 t(4.8)	16-OMe	3.25 s

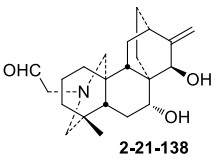
2.21.7 C1-type diterpenoid alkaloids

Table 2-21-41: Cos, MFs, and TSs of C1-type diterpenoid alkaloids **2-21-137**~**2-21-157**.

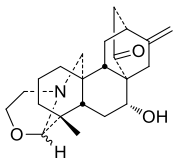
No.	Compounds	MFs	Test solvents	References
2-21-137	spiratine A	$C_{22}H_{33}NO_3$	CD_3OD	[623]
2-21-138	consorientaline	$C_{22}H_{33}NO_3$	$CDCl_3$	[624]
2-21-139	spiramine Z-2	$C_{22}H_{31}NO_3$	$CDCl_3$	[625]
2-21-140	spiramine Z-3	$C_{22}H_{31}NO_3$	$CDCl_3$	[625]
2-21-141	spiramide	$C_{26}H_{35}NO_6$	CD_3OD	[628]
2-21-142	deacetylspiramine S	$C_{22}H_{31}NO_3$	$CDCl_3$	[629]
2-21-143	ouvrardiandine A	$C_{28}H_{37}NO_7$	$CDCl_3$	[584]
2-21-144	ouvrardiandine B	$C_{30}H_{33}NO_7$	$CDCl_3$	[584]
2-21-145	spiraeaine A	$C_{22}H_{33}NO_3$	$CDCl_3$	[627]
2-21-146	spiramine X	$C_{26}H_{35}NO_6$	$CDCl_3$	[626]
2-21-147	spiramine Y	$C_{24}H_{33}NO_5$	$CDCl_3$	[626]
2-21-148	spiramine P	$C_{22}H_{33}NO_4$	C_5D_5N	[630]
2-21-149	spiramine U	$C_{24}H_{35}NO_5$	$CDCl_3$	[630]
2-21-150	spiratine B	$C_{24}H_{33}NO_5$	$CDCl_3$	[623]
2-21-151	spiramine Z	$C_{26}H_{37}NO_5$	$CDCl_3$	[626]
2-21-152	19-*O*-deethylspiramine N	$C_{20}H_{29}NO_3$	C_5D_5N	[629]
2-21-153	isoazitine	$C_{20}H_{29}NO$	$CDCl_3$	[631]
2-21-154	13-(2-methylbutyryl)azitine	$C_{25}H_{37}NO_3$	–	[632]
2-21-155	19-oxodihydroatisine	$C_{22}H_{33}NO_3$	$CDCl_3$	[631]
2-21-156	22-*O*-acetyl-19-oxodihydroatisine	$C_{24}H_{35}NO_4$	$CDCl_3$	[631]
2-21-157	uncinatine	$C_{22}H_{33}NO_3$	$CDCl_3$	[633]



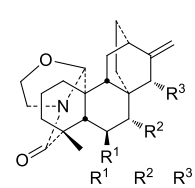
2-21-137



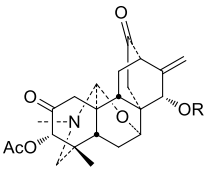
2-21-138



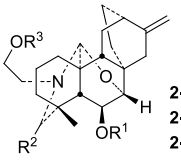
2-21-139 19α H
2-21-140 19β H



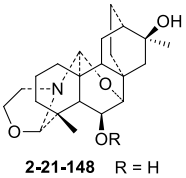
	R^1	R^2	R^3
2-21-141	OAc	OAc	H
2-21-142	H	H	OH



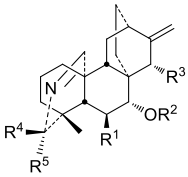
2-21-143 R = MeBu
2-21-144 R = Bz



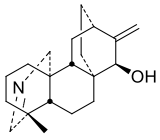
	R^1	R^2	R^3
2-21-145	H	H	H
2-21-146	Ac	=O	Ac
2-21-147	Ac	=O	H



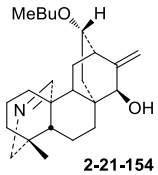
2-21-148 R = H
2-21-149 R = Ac



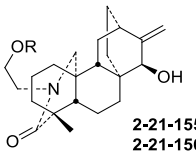
	R^1	R^2	R^3	R^4	R^5
2-21-150	OAc	Ac	H	OH	H
2-21-151	OAc	Ac	H	H	OEt
2-21-152	H	H	OH	OH	H



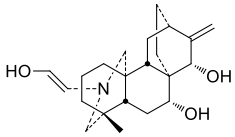
2-21-153



2-21-154



2-21-155 R = H
2-21-156 R = Ac



2-21-157

Table 2-21-42: ^{1}H NMR spectroscopic data of C1-type diterpenoid alkaloids **2-21-137**~**2-21-141**.

H	2-21-137	2-21-138	2-21-139	2-21-140	2-21-141
1	1.58 m, 2.02 m	*α* 2.88 m, *β* 1.90 m	1.22 m, 1.43 m	1.22 m, 1.43 m	0.95 ddd(4.6, 4.8, 13.3) 2.41 br dd(3.2, 13.3)
2	1.65 m, 1.36 m	*α* 1.86 br d, *β* 1.03 m	2.28 m, 1.45 m	2.28 m, 1.45 m	1.39 m, 1.46 m
3	1.74 m, 1.48 m	*α* 1.63 m, *β* 1.40 br d	1.97 m, 1.91 m	1.97 m, 1.91 m	1.43 m, 1.78 m
5	1.59 m	1.64 br s ($W_{1/2}$ = 5.2)	0.89 m	0.71 m	1.84 d(11.6)
6	1.05 m, 1.79 m	*α* 1.60 m *β* 1.21 dd(2.1, 12.3)	1.43 m 1.58 m	1.43 m 1.58 m	5.33 dd (9.9, 11.6)
7	3.76 m	3.84 br d	3.03 d(2.5)	3.03 d(2.5)	4.76 d(9.9)
9	1.67 m	2.14 d(3.7)	1.50 m	1.50 m	1.48 m
11	1.79 m, 1.82 m	*α* 1.91 dd(3.1, 12.3) *β* 1.50 m	1.58 m, 1.98 m	1.58 m, 1.98 m	1.73 m 2.09 ddd(2.4, 7.2, 13.5)

Table 2-21-42 (continued)

H	2-21-137	2-21-138	2-21-139	2-21-140	2-21-141
12	2.42 d(2.6)	2.37 br s	2.86 m	2.86 m	2.25 br s
13	1.73 m	*α* 1.80 m, *β* 1.91 m	2.16 m, 2.23 m	2.16 m, 2.23 m	1.52 dd(6.5, 12.8) 1.65 br d(12.1)
14	1.17 m, 1.99 m	*α* 1.90 m, *β* 1.82 m			1.60 dd(6.5, 12.8) 1.89 br d(12.8)
15	4.02 s	4.26 d(3.8)	2.99 s 2.24 dd(2.5, 8)	2.99 s 2.24 dd(2.5, 8)	1.98 br d(16.0) 2.20 br d(16.4)
17	5.05 br s, 4.04 br s	5.02 br s, 5.10 br s	4.87 br s, 4.69 br s	4.83 br s, 4.62 br s	4.60 d(1.7), 4.77 d(1.7)
18	1.08 s	0.84 s	1.04 s	1.01 s	1.15 s
19	3.75 m, 3.81 m	3.40 d(10.9), 3.75 d(10.9)	3.89 br s	3.70 br s	
20	4.04 m, 3.96 m	4.10 br s	2.65 m, 3.04 m	2.65 m, 3.04 m	5.06 s
21	4.16 m, 4.19 m	3.60 m, 3.85 m	3.41 m, 3.46 m	3.41 m, 3.46 m	3.29 ddd(3.7, 8.2, 11.2) 3.97 ddd(8.2, 8.2, 11.2)
22	8.73 s	8.74 s	3.75 m	3.75 m	3.84 dt(8.2, 8.2) 4.15 ddd(3.7, 8.2, 8.2)
6-OAc					1.98 s
7-OAc					1.92 s

Table 2-21-43: ^{1}H NMR spectroscopic data of C1-type diterpenoid alkaloids **2-21-142**~**2-21-146**.

H	2-21-142	2-21-143	2-21-144	2-21-145	2-21-146
1	*α* 2.37 m, *β* 0.89 m	*α* 2.28 br s, *β* 2.29 br s	*α* 2.37 br s, *β* 2.37 br s	1.66 m, 1.38~1.59 m	1.38 m, 1.68 m
2	*α* 1.45 m, *β* 1.30 m			1.38~1.59 m, 1.25~1.38 m	1.46 m, 1.71 m
3	*α* 1.42 m, *β* 1.84 m	4.54 s	4.57 s	1.38~1.59 m, 1.25~1.38 m	1.52 m, 1.58 m
5	1.52 d(9.6)	2.02 br s	2.07 br s	1.25 m	1.49 br s
6	*α* 1.95 m *β* 1.75 m	*α* 1.62 d(12.0) *β* 2.05 dd(12.0, 6.0)	*α* 1.67 d(12.0) *β* 2.10 dd(12.0, 6.0)	4.40 br s	5.12 t(3.8)
7	3.73 dd(5.3, 8.8)	4.03 d(6.0)	4.05 dd(6.0)	3.75 d(4.8)	3.65 d(3.8)
9	1.12 dd(5.9, 8.6)	2.41 m	2.50 m	1.64 d(8.4)	1.40 m

Table 2-21-43 (continued)

H	2-21-142	2-21-143	2-21-144	2-21-145	2-21-146
11	*α* 1.69 m *β* 1.42 m	*α* 1.83 m *β* 2.20 m	*α* 1.88 m *β* 2.27 m	1.59~1.38 m 1.07 ddd(13.6, 13.6, 7.2)	2.07 m 2.36 m
12	2.36 m	3.10 t(2.9)	3.19 t(2.8)	2.27 m	2.33 m
13	*α* 1.30 m, *β* 1.41 m			2.61 m, 1.25~1.58 m	1.37 m, 1.87 m
14	*α* 1.42 m *β* 1.68 m	*α* 2.40 d(ov) *β* 2.92 d(19.0)	*α* 2.59 d(19.6) *β* 3.04 d(19.6)	2.15 m 1.59~1.38 m	1.40 m 1.90 m
15	3.93 d(7.8)	5.56 s	5.85 s	3.46 d(15.6), 2.15 m	1.29 m, 1.79 m
17	5.09 s, 5.06 s	5.10 s, 5.24 s	5.24 s, 5.32 s	4.88 s, 4.74 s	4.66 br s, 4.82 br s
18	1.21 s	1.29 s	1.31 s		1.13 s
19		2.77 ABq(10.2) 2.84 ABq(10.2)	2.79 ABq(10.2) 2.85 ABq(10.2)	2.69 d(11.6) 2.35 d(11.6)	
20	5.11 s	4.33 s	4.35 s	4.71 s	4.78 d(1.8)
21	*α* 3.28 m *β* 3.90 m	2.36 s	2.39 s	3.16 dt(14.0, 5.6) 2.91 dt(14.0, 5.6)	3.28 m 4.01 m
22	*α* 3.87 m, *β* 4.18 m			3.90 m, 3.85 m	4.18 m
2′		2.41 m	8.03 d(8.0)		
3′		1.48 m, 1.71 m	7.47 t(8.0)		
4′		0.92 t(7.3)	7.62 t(8.0)		
5′		1.16 d(7.1)	7.47 t(8.0)		
6′			8.03 t(8.0)		
3-OAc		2.14 s	2.14 s		
6-OAc					2.04 s
22-OAc					2.02 s
OH				6.47 br s(6-OH) 5.47 br s(22-OH)	

Table 2-21-44: ^{1}H NMR spectroscopic data of C1-type diterpenoid alkaloids **2-21-147**~**2-21-151**.

H	2-21-147	2-21-148	2-21-149	2-21-150	2-21-151
1	1.37 m, 1.70 m	1.32 m, 1.21 m	1.68 m, 1.18 m	1.76 d(11.5), 1.17 m	1.09 m, 1.66 m
2	1.48 m, 1.75 m	2.26 m, 1.39 m	2.03 m, 1.40 m	1.45 m	0.98 m, 1.94 m
3	1.55 m, 1.61 m	1.40 m, 1.52 m	1.46 m, 1.20 m	2.05 m, 1.06 m	1.34 m
5	1.50 br s	1.38 br s	1.08 d(2.0)	1.65 d(11.0)	1.52 d(10.2)
6	5.13 t(3.8)	5.09 dd(2.1, 4.9)	5.61 dd(2.0, 4.9)	5.20 dd(9.5, 11.0)	5.08 t(10.2)
7	3.73 d(4.8)	3.70 d(4.9)	3.53 d(4.9)	4.74 d(9.5)	4.65 d(10.2)
9	1.44 m	2.03 dd(2.9, 10.5)	1.53 m	1.62 m	1.50 m
11	2.08 m, 2.38 m	1.60 m, 1.23 m	1.50 m, 1.31 m	1.85 m, 1.83 m	1.78 m

Table 2-21-44 (continued)

H	2-21-147	2-21-148	2-21-149	2-21-150	2-21-151
12	2.35 m	1.83 m	1.51 m	2.35 br s	2.27 m
13	1.36 m, 1.90 m	2.65 m, 1.48 m	1.91 m, 1.38 m	1.66 m, 1.09 m	1.52 m, 1.58 m
14	1.35 m, 1.85 m	2.12 m, 1.50 m	1.80 m, 1.21 m	1.67 m, 1.64 m	1.48 m, 1.56 m
15	1.33 m	3.06 dd(3.2, 12.4)	1.90 dd(3.4, 12.4)	2.27 br d(17.1)	1.92 d(17.2)
	1.77 m	1.89 d(12.4)	1.28 d(12.4)	2.00 br d(17.1)	2.19 d(17.2)
17	4.66 br s	1.71 s	1.36 s	4.79 br s	4.54 s
	4.82 br s			4.62 br s	4.71 s
18	1.13 s	1.40 s	1.07 s	0.98 s	0.84 s
19		3.91 s	3.83 s	5.11 s	4.56 s
20	4.78 d(1.6)	4.64 s	4.54 s	7.76 br s	7.74 s
21	3.20 m, 3.63 m	3.38 m, 3.18 m	3.26 m, 3.17 m		3.59 m, 4.03 m
22	3.79 m	3.75 m, 3.40 m	3.63 m, 3.43 m		1.13 t(7.0)
6-OAc	2.04 s		2.00 s	2.03 s	1.95 s
7-OAc				1.96 s	1.89 s

Table 2-21-45: ^{1}H NMR spectroscopic data of C1-type diterpenoid alkaloids **2-21-152**~**2-21-154**.

H	2-21-152	2-21-153	2-21-154
1	α 2.46 m	1.70 m	α 1.72 br d(14.5)
	β 1.04 m	1.00 m	β 1.06 br d(12.7)
2	α 1.41 m, β 1.76 m	1.50 m, 1.27 m	α 1.48 dd(6.6, 13.8), β (ov)
3	α 1.61 m,	1.49 m	α 1.42 dd(5.1, 18.0)
	β 0.99 m	1.28 m	β 1.24 m
5	1.21 d(14.0)	0.98 m	(ov)
6	α 1.65 m, β 2.34 m	1.56 m, 0.99 m	α 1.60 m, β 1.19 m
7	3.90 dd(4.1, 7.4)	1.68 m	α 2.05 m
		1.12 br dt(13.5, 3)	β 1.33 m
9	1.27 d(5.4)	1.58 m	1.94 br d(8.4)
11	α 2.02 d(13.0), β 1.56 m	1.72 m, 1.36 ddd(12.5, 7.7, 2)	α 1.78 m, β 1.62 m
12	2.31 d(16.0)	2.34 br s	2.38 m
13	1.15 m, 1.62 m	1.57 m, 1.57 m	5.04 dd(4.7, 11.9)
14	1.18 m	α 2.14 ddd(15, 11, 4.5)	α 1.65 m
	1.76 m	β 0.92 dddd(15, 11, 7.2)	β 1.30 m
15	4.26 d(4.1)	3.61 br t(2)	3.74 br t
17	5.11 s, 5.33 s	5.10 t(1.5), 5.04 t(1.5)	5.08 br s, 5.17 br s
18	1.10 s	1.07 s	0.86 s
19	5.30 s	7.43 br s	3.45 d(10.7)
20	7.99 s	3.92 dt(19, 2), 3.42 dd(19, 3)	7.93 br d
2′			2.57 m
3′			α 1.70 m, β 1.50 m
4′			0.92 t(7.3)
5′			1.16 d(7.0)

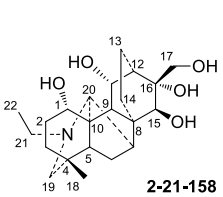
Table 2-21-46: ^{1}H NMR spectroscopic data of C1-type diterpenoid alkaloids **2-21-155**~**2-21-157**.

H	2-21-155	2-21-156	2-21-157
1	1.81 m, 1.19 m	1.79 m, 1.12 m	*α* 1.7 m, *β* 2.8 br d(14)
2	1.57 m, 1.51 m	1.56 m, 1.41 tt(13.5, 4.5)	*α* 1.8 ddd(3, 10, 12) *β* 1.2 dd(3, 12)
3	1.80 m, 1.36 td(13.5,4.5)	1.78 m, 1.33 td(13.5, 4.5)	*α* 1.87 dd(5, 14) *β* 2.1 ddd(5, 12, 14)
5	1.69 m	1.15 m	1.72 dd(2, 14)
6	1.68 m, 1.14 m	1.68 m, 1.14 m	*α* 1.24 dd(2, 12), *β* 1.6 m
7	1.71 m, 1.47 m	1.72 m, 1.16 m	3.9 d(5)
9	1.74 m	1.73 m	2.2 d(3)
11	1.73 m, 1.12 m	1.74 m, 1.19 m	*α* 2.0 dd(3, 12), *β* 1.5 m
12	2.32 br s	2.32 m	2.4 d(3)
13	1.62 m, 1.46 m	1.62 br t(13), 1.48 m	*α* 1.8 m, *β* 2.05 m
14	*α* 2.13 ddd(15, 11.5, 3) *β* 0.97 ddd(15, 12, 7)	*α* 2.15 ddd(15, 11.5, 3) *β* 0.97 ddd(15, 12, 7)	1.9 m
15	3.63 br t(2)	3.63 br t(2)	4.2 d(3)
16	5.10 t(1.5)	5.10 t(1.5)	
17	5.04 t(1.5)	5.04 t(1.5)	5.02 br s, 5.12 br s
18	1.15 s	1.11 s	1.07 s
19			*α* 3.60 d(10), *β* 3.45 d(10)
20	3.72 dd(13, 1.5), 3.12 br d(13)	3.76 dd(13, 1.5), 3.09 d(13)	4.15 d(12)
21	3.58 ddd(14.5, 6, 4.5, 3.6) 3.51 ddd(14.5, 7, 4.5, 3.7)	3.76 ddd(14.5, 6, 4.5) 3.44 ddd(14.5, 7, 4.5)	7.08 d(8.5)
22	3.83 ddd(11.5, 6, 4.5, 3.6) 3.79 ddd(11.5, 7, 4.5)	4.31 ddd(11.5, 6, 4.5) 4.20 ddd(11.5, 7, 4.5)	6.75 d(8.5)
22-OAc		2.02 s	

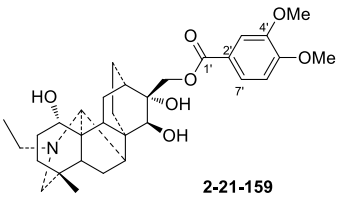
2.21.8 C2-type diterpenoid alkaloids

Table 2-21-47: Cos, MFs, and TSs of C2-type diterpenoid alkaloids **2-21-158**~**2-21-166**.

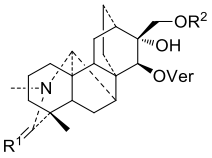
No.	Compounds	MFs	Test solvents	References
2-21-158	11-*epi*-16*α*,17-dihydroxylepenine	$C_{22}H_{35}NO_5$	C_5D_5N	[634]
2-21-159	*N*-ethyl-1*α*-hydroxy-17-veratroyldictizine	$C_{31}H_{43}NO_7$	$CDCl_3$	[575]
2-21-160	15-veratroyldictizine	$C_{30}H_{41}NO_6$	$CDCl_3$	[575]
2-21-161	15-veratroyl-17-acetyldictizine	$C_{32}H_{43}NO_7$	$CDCl_3$	[575]
2-21-162	15-veratroyl-17-acety-19-oxodictizine	$C_{32}H_{41}NO_8$	$CDCl_3$	[575]
2-21-163	kirinine B	$C_{22}H_{31}NO_3$	$CDCl_3$	[635]
2-21-164	kirinine C	$C_{22}H_{29}NO_4$	$CDCl_3$	[635]
2-21-165	lepenine	$C_{22}H_{33}NO_3$	$CDCl_3$	[635]
2-21-166	ajaconine	$C_{22}H_{33}NO_3$	$CDCl_3$	[617]



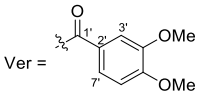
2-21-158

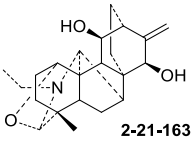


2-21-159

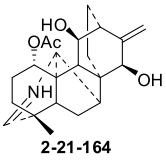


	R^1	R^2
2-21-160	H_2	H
2-21-161	H_2	Ac
2-21-162	O	Ac

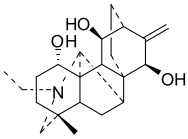




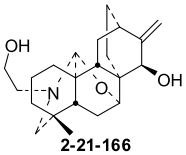
2-21-163



2-21-164



2-21-165



2-21-166

Table 2-21-48: ^{1}H NMR spectroscopic data of C2-type diterpenoid alkaloids **2-21-158**~**2-21-162**.

H	2-21-158	2-21-159	2-21-160	2-21-161	2-21-162
1	4.30 dd(11, 6)	3.86 dd(10.5.7)	1.89 dd(13, 4.7)	1.89 dd(13.4, 4.5)	2.01 m
			1.32 m	1.40 m	1.42 m
2	*α* 2.86 br d(11)	2.50 m	2.23 m	2.22 m	2.21 m
	β 2.06 m	2.18 m	1.44 br dd(13.5, 4.7)	1.45 m	1.42 m
3	*α* 1.60 m	1.62 m	1.56 br d(13)	1.56 br d(13.1)	1.82 br d(13.1)
	β 1.35 m	1.35 m	1.18 dddd(12, 12, 4, 2)	1.17 m	1.18 m
5	1.42 d(8)	1.53 m	1.15 br d(7.3)	1.16 br d(7)	1.35 br d(8.6)
6	*α* 1.33 m	3.23 dd(13.7, 7.9)	2.37 dd(13.8, 7.9)	2.29 dd(13.4, 7.5)	2.58 dd(13.9, 7.9)
	β 3.64 dd(13, 8)	1.30 m	1.30 m	1.26 m	1.32 m
7	2.23 d(5)	2.34 m	2.12 br d(5.5)	2.12 br d(4.8)	2.10 br d(5)
9	2.35 d(7)	2.04 t(10)	1.80 t(10, 10)	1.85 dd(11.5, 9.5)	1.93 t(10.5)
11	4.85 dd(7, 5)	2.17 t(12), 1.84 m	1.71 m, 1.31 t(13.1)	1.64 m, 1.30 m	1.67 m, 1.40 m
12	2.63 br s	1.82 br s	1.90 br s	1.81 m	1.86 br s
13	*α* 2.50 m, *β* 2.43 m	2.06 m, 1.5 m	2.07 m, 1.38 m	2.13 m, 1.35 m	1.56 m, 0.81 m
14	*α* 2.48 m, *β* 1.32 m	2.05 m, 1.31 m	2.12 m, 1.38 m	2.02 m, 1.36 m	2.01 m, 1.50 m
15	4.69 br s	4.11 s	5.08 s	5.17 s	5.19 s

Table 2-21-48 (continued)

H	2-21-158	2-21-159	2-21-160	2-21-161	2-21-162
17	*α* 4.33 d(11)	4.68 d(12.3)	3.69 d(11.5)	4.30 d(11.6)	4.29 d(11.6)
	β 4.74 d(11)	4.56 d(12.3)	3.62 d(11.5)	4.23 d(11.6)	4.22 d(11.6)
18	0.73 s	0.86 s	0.66 s	0.65 s	1.02 s
19	*α* 2.54 m	3.18 m	2.40 br d(11.2)	2.45 br d(11.6)	
	β 2.25 d(10)	2.49 m	2.23 dd(11.2, 2)	2.20 dd(11.6, 1.8)	
20	4.11 br s	4.37 br s	3.38 br s	3.40 br s	3.72 br s
21	2.52 m, 2.42 m	3.23 m, 2.92 m	2.24 br s	2.24 br s	2.89 br s
22	1.01 t(7)	1.55 t(7)			
3′		7.51 d(2)	7.52 d(2)	7.50 d(2)	7.49 d(2)
6′		6.89 d(8.5)	6.91 d(8.5)	6.9 d(8.5)	6.91 d(8.5)
7′		7.64 dd(8.5, 2)	7.65 dd(8.5, 2)	7.64 dd(8.5, 2)	7.62 dd(8.5, 2)
4′-OMe		3.93 s	3.90 s	3.89 s	3.90 s
5′-OMe		3.95 s	3.93 s	3.92 s	3.94 s
17-OAc				1.77 s	1.76 s

Table 2-21-49: ^{1}H NMR spectroscopic data of C2-type diterpenoid alkaloids **2-21-163**~**2-21-166**.

H	2-21-163	2-21-164	2-21-165	2-21-166
1	4.19 d(5.3)	5.30 dd(10.8, 7.2)	4.17 dt(10.8, 6.9)	*α* 1.55 m, *β* 1.17 m
2	1.24 m, 1.83 m	1.30 m, 1.98 m	1.82 m, 2.35 m	*α* 2.27 m, *β* 1.40 m
3	1.56 m, 1.63 m	1.23 m, 1.51 m	1.32 m, 1.64 m	*α* 1.58 m, *β* 1.23 m
5	1.61 m	1.43 m	1.37 d(7.6)	1.22 s
6	1.67 m	1.20 m	1.25 m	*α* 2.42 m
	2.35 dd(12.6, 8.5, 2.0)	2.91 dd(14.0, 7.8, 1.3)	2.74 dd(13.0, 7.6)	*β* 1.80 t(3.4)
7	1.84 m	2.17 m	2.21 m	3.67 t(9.2)
9	1.28 d(9.6, 6.8)	1.35 d(9.2)	1.37 d(9.5)	1.50 m
11	3.74 dd(9.6, 6.8)	3.84 dd(9.2, 1.4)	4.46 dd(9.5, 6.7)	*α* 1.65 m, *β* 1.40 m
12	2.21 dd(5.3, 5.2)	2.15 m	2.21 m	2.36 t(3.7)
13	1.47 m, 1.71 m	1.48 m, 1.69 m	1.47 m, 1.72 m	1.85 m
14	1.97 ddd(14.0, 11.7, 7.0)	1.96 m	1.94 m	*α* 2.15 m
	1.21 m	1.25 m	1.14 m	*β* 1.35 m
15	4.28 dt(6.8, 2.0, 2.0)	4.28 br s	4.28 dt(7.7, 2.1, 2.1)	4.15 br s
17	5.04 t(2.0, 2.0)	5.22 t(2.0, 2.0)	5.08 d(2.1, 2.1)	5.11 s
	5.23 t(2.0, 2.0)	5.22 t(2.0, 2.0)	5.28 t(2.1, 2.1)	4.99 s
18	0.78 s	0.98 s	0.70 s	0.73 s
19	3.68 s	7.25 s	2.23 m	*α* 2.81 AB(11.3)
			2.50 m	*β* 2.27 AB(11.3)
20	3.04 dd(4.1, 2.1)	4.67 br s	3.68 br s	4.57 br s
21	2.63~2.69 m		2.30~2.50 m	*α* 2.94 m, *β* 2.83 m
22	0.99 t(7.3, 7.3)		1.05 t(7.0, 7.0)	*α* 3.67 m, *β* 2.82 m
1-OAc		2.05 s	2.32 br s	
OH	1.76 d(6.8)	1.86 br s	2.50 br s	2.46 br d(15-OH)
	1.40 d(6.8)	1.81 br s	2.08 d(7.7)	4.08 br d(22-OH)

2.21.9 C3-type diterpenoid alkaloids

Table 2-21-50: Cos, MFs, and TSs of C3-type diterpenoid alkaloids **2-21-167**~**2-21-179**.

No.	Compounds	MFs	Test solvents	References
2-21-167	delcarduchol	$C_{21}H_{27}NO_3$	$CDCl_3$	[636]
2-21-168	2-dehydrodeacetylheterophylloidine	$C_{21}H_{25}NO_3$	$CDCl_3$	[637]
2-21-169	variegatine	$C_{21}H_{27}NO_2$	$CDCl_3$	[575]
2-21-170	carduchoron	$C_{21}H_{25}NO_3$	$CDCl_3$	[636]
2-21-171	racemulodine	$C_{21}H_{27}NO_4$	$CDCl_3$	[638]
2-21-172	spirafine II	$C_{22}H_{31}NO_2$	C_5D_5N	[639]
2-21-173	spirafine III	$C_{22}H_{31}NO_2$	C_5D_5N	[639]
2-21-174	trabzonine	$C_{22}H_{33}NO_3$	$CDCl_3$	[640]
2-21-175	naviculine B	$C_{20}H_{27}NO_2$	CD_3OD	[641]
2-21-176	naviculine A	$C_{20}H_{27}NO_2$	CD_3OD	[641]
2-21-177	deacetylheterophylloidine	$C_{21}H_{27}NO_3$	$CDCl_3$	[642]
2-21-178	hetidine	$C_{21}H_{27}NO_4$	$CDCl_3$-C_5D_5N	[642]
2-21-179	hetisinone	$C_{20}H_{27}NO_3$	$CDCl_3$	[603]

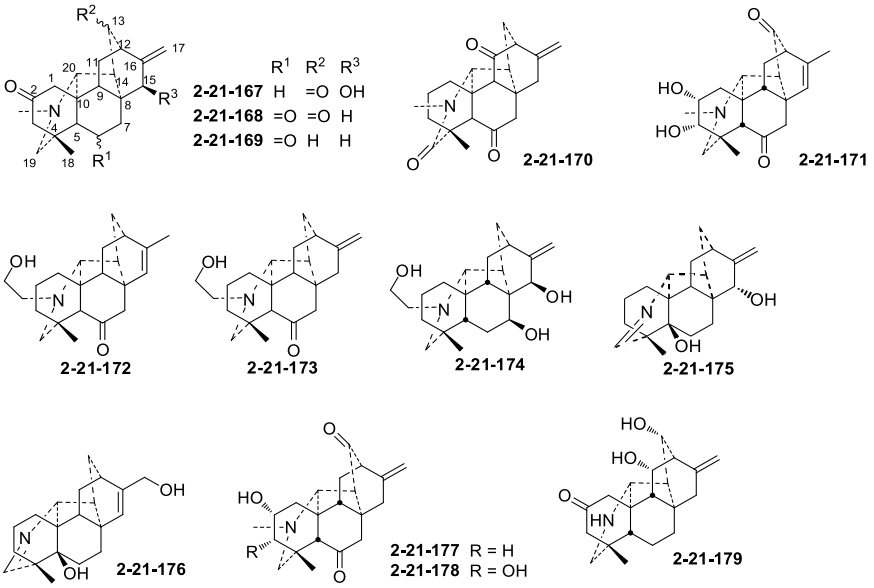


Table 2-21-51: ^{1}H NMR spectroscopic data of C3-type diterpenoid alkaloids **2-21-167**~**2-21-171**.

H	2-21-167	2-21-168	2-21-169	2-21-170	2-21-171
1	*α* 3.26 d(13)	2.45 dd(15, 1.5)	2.61 dd(15.1, 2.0)	*α* 2.01 dd(5,12)	*α* 2.14 dd(14.2, 2.0)
	*β*1.30 d(13)	2.23 d(15)	2.28 d(15.0)	*β* 1.60 br d(12)	*β* 1.82 dd(14.2, 4.4)

Table 2-21-51 (continued)

H	2-21-167	2-21-168	2-21-169	2-21-170	2-21-171
2				*α* 1.75 m, *β* 1.40 m	3.92 sept($W_{1/2}$ = 2.0)
3	*α* 2.90 d(12)	2.21 d(15.3)	2.24 m	*α* 1.85 m	3.35 d(5.6)
	β 1.90 d(12)	2.17 dd(15.3, 1.6)	2.13 dd(14.5, 2.1)	*β* 1.40 m	
5	1.86 br s	2.14 br s	2.00 s	2.50 s	1.85 s
6	*α* 2.85 m, *β* 1.60 m				
7	*α* 2.75 m	2.64 d(19.2)	2.57 d(19.0)	*α* 2.75 d(18)	2.79 br s
	β 1.65 m	2.36 dd(19.2, 1.3)	2.17 d(19.0)	*β* 2.25 d(18)	
9	2.04 dd(4, 7)	2.10 m	1.85 dd(10.7)	1.66 s	1.76 dt(10.4, 2.0)
11	*α* 1.80 m	1.90 m	1.82 dd(14.0, 2.5)		*α* 1.99 ddd(14.0, 3.0, 1.6)
	β 2.35 m	1.88 m	1.67 ddt(14, 11, 2)		*β* 1.55 ddd(14.0, 10.4, 2.0)
12	2.60 br d(3)	2.93 t(2.6)	2.20 m	2.30 br s	2.98 m ($W_{1/2}$ = 5.7)
13			1.97 tt(12.3, 2.2)	*α* 1.90 m	
			1.39 dd(12.3, 3.7)	*β* 1.40 m	
14	1.65 m	2.50 br d(2.5)	1.97 br s	1.80 m	2.30 d(2.8)
15	3.95 br s	2.47 dt(18, 2.5)	2.34 dt(18.0, 2.3)	*α* 2.26 d(14)	5.50 s
		2.37 dt(18, 2)	2.25 m	*β* 2.38 d(14)	
17	5.01 t(1.5)	4.96 br t(2.5)	4.70 dd(4.0, 2.3)	4.97 br s	1.86 d(2.0)
	4.97 t(1.5)	4.80 t(2)	4.53 dd(3.7, 2.0)	4.78 br s	
18	1.00 s	1.38 s	1.45 s	1.50 s	1.16 s
19	*α* 2.16 d(13)	2.43 d(12.5)	2.46 d(12.3)		1.88 ABq(12.4)
	β 1.98 d(13)	1.92 d(12.5)	1.78 d(12.3)		2.64 ABq(12.4)
20	2.97 br s	2.30 br s	1.93 s	2.02 d(3)	3.06 d(3.2)
NMe	2.37 s	2.10 s	2.20 s	2.50 s	2.45 s

Table 2-21-52: ^{1}H NMR spectroscopic data of C3-type diterpenoid alkaloids **2-21-172**~**2-21-176**.

H	2-21-172	2-21-173	2-21-174	2-21-175	2-21-176
1	1.63 m	1.72 m	1.84	1.65~1.67 m	1.70~1.72 m
				1.60~1.62 m	1.51~1.53 m
2	1.52 m	1.57 m	1.52	1.53~1.55 m	1.52~1.54 m
				1.22~1.26 m	1.17~1.19 m
3	1.26 m	1.25 m	*α* 1.33, *β* 1.30	1.8 dd(14, 4.5)	1.76~1.79 m
				1.24~1.28 m	1.22~1.25 m
5	1.52 s	1.57 s	1.18 d		
6			*α* 2.16, *β* 1.93	1.67~1.69 m	1.68~1.71 m
				1.35~1.39 m	1.46~1.48 m
7	2.84 dd(1.8, 8.2)	2.66 dd(1.2, 7.2)	4.24	2.00 dd(12.0, 4.5)	2.00~2.04 m
				1.47 dd(12.0, 4.5)	1.71~1.73 m

Table 2-21-52 (continued)

H	2-21-172	2-21-173	2-21-174	2-21-175	2-21-176
9	1.53 s	1.57 s	1.55	1.74~1.76 m	1.67~1.69 m
11	1.86 m	2.02 m	α 1.92, β 1.56	1.88~1.91 m	1.63~1.65 m
				1.50~1.52 m	1.37 t(12.4)
12	2.19 m	2.13 m	2.26	2.31 s	2.45 s
13	1.85 m	1.62 m	α 1.62, β 1.49	2.16 dd(13.5, 8.5)	1.83 dd(12.0, 4.0)
				1.86~1.88 m	1.53~1.55 m
14	1.85 m	1.62 m	2.11	1.90~1.92 m	1.55~1.57 m
15	5.25 s	2.20 s	4.52	3.89 s	5.50 d(1.4)
17	1.76 s	4.66 br s, 4.49 br s	4.98, 4.87	4.86 br s, 4.85 br s	4.09 s
18	1.508 s	1.492 s	0.97	1.04 s	1.05 s
19	2.68 dd(11.9, 14.6)	2.66 dd(11.9)	α 2.73, β 2.13	7.37 d(2.4)	7.39 d(2.6)
20	2.33 s	2.20 s	2.35	3.53 s	3.51 s
21	3.05 m	3.05 m	2.86, 2.51		
22	3.73 m	3.73 m	3.71, 3.54		

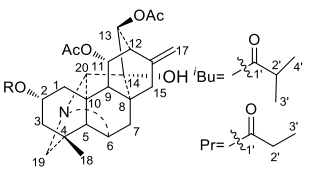
Table 2-21-53: ^{1}H NMR spectroscopic data of C3-type diterpenoid alkaloids **2-21-177**~**2-21-179**.

H	2-21-177	2-21-178	2-21-179
1α	2.02 dd(13.8, 4.4)	2.15 dd(14.5, 3.5)	3.33 d(14)
1β	1.61 dd(13.8, 5.5)	1.73 dd(14.5, 3.5)	2.75 d(14.6)
2β	3.92 br s($W_{1/2}$ = 5.0)	3.95 br t(3.5, 3.5, 5.3)	
3α	1.80 m		–
3β	1.72 m	3.36 d(5.3)	–
5	1.85 s	1.83 s	–
6			3.31 br s
7α	2.41 m	2.48 m	1.76 dd(13.6, 3.2)
7β	2.41 m	2.48 m	1.64 dd(13.6, 2.5)
9	1.91 m	1.92 m	–
11α	2.07 m	2.07 m	
11β	1.85 m	1.85 m	4.23 d(10.3)
12	2.92 br d($W_{1/2}$ = 7.5)	2.95 br d($W_{1/2}$ = 7.0)	2.42 br s
13			4.23 d(10.3)
14	2.60 br t($W_{1/2}$ = 6.0)	2.61 br d($W_{1/2}$ = 3.0)	–
15α	2.35 AB(18.1)	2.38 AB(18.0)	–
15β	2.49 AB(18.1)	2.49 AB(18.0)	–
17	4.76 br s($W_{1/2}$ = 5.0),	4.79 br s($W_{1/2}$ = 7.0),	4.88 s
	4.94 br s($W_{1/2}$ = 5.0)	4.96 br s($W_{1/2}$ = 7.0)	
18	1.08 s	1.18 s	1.15 s
19α	2.10 AB(8.0)	1.94 AB(12.4)	2.72 d(12.8)
19β	2.40 AB(8.0)	2.73 AB(12.4)	
20	3.21 s	3.17 br d($W_{1/2}$ = 3.0)	2.95 s
21	2.45 s	2.47 s	
2-OH	6.62 br s		

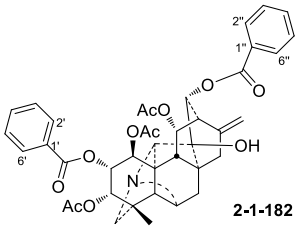
2.21.10 C4-type diterpenoid alkaloids

Table 2-21-54: Cos, MFs, and TSs of C4-type diterpenoid alkaloids **2-21-180**~**2-21-209**.

No.	Compounds	MFs	Test solvents	References
2-21-180	guanfu base P	$C_{28}H_{37}NO_7$	–	[643]
2-21-181	guanfu base R	$C_{27}H_{35}NO_7$	$CDCl_3$	[644]
2-21-182	delphigraciline	$C_{40}H_{41}NO_{11}$	$CDCl_3$	[645]
2-21-183	guanfu base Q	$C_{22}H_{27}NO_5$	$CDCl_3$	[646]
2-21-184	guanfu base S	$C_{24}H_{29}NO_5$	$CDCl_3$	[647]
2-21-185	jaluenine	$C_{29}H_{33}NO_6$	$CDCl_3$	[648]
2-21-186	orochrine	$C_{21}H_{28}NO_3$	CD_3OD	[649]
2-21-187	2-*O*-acetylorochrine	$C_{23}H_{30}NO_4$	CD_3OD	[649]
2-21-188	2-*O*-acetyl-7*α*-hydroxyorochrine	$C_{23}H_{30}NO_5$	CD_3OD	[649]
2-21-189	7*α*-hydroxycossonidine	$C_{20}H_{27}NO_3$	$CDCl_3$	[576]
2-21-190	14-hydroxyhetisinone *N*-oxide	$C_{20}H_{25}NO_5$	$CDCl_3$	[645]
2-21-191	glandulosine	$C_{30}H_{39}NO_9$	$CDCl_3$	[650]
2-21-192	9-deoxyglanduline	$C_{27}H_{37}NO_7$	$CDCl_3$	[650]
2-21-193	11,13-*O*-diacetylglanduline	$C_{31}H_{41}NO_{10}$	$CDCl_3$	[650]
2-21-194	9-*O*-acetylglanduline	$C_{29}H_{39}NO_9$	$CDCl_3$	[650]
2-21-195	orientinine	$C_{20}H_{23}NO_5$	$CDCl_3$	[651]
2-21-196	acorientine	$C_{20}H_{27}NO_3$	$CDCl_3$	[651]
2-21-197	11,13-*O*-diacetyl-9-deoxyglanduline	$C_{31}H_{41}NO_9$	$CDCl_3$	[652]
2-21-198	13-*O*-acetyl-9-deoxyglanduline	$C_{29}H_{39}NO_8$	$CDCl_3$	[652]
2-21-199	14-*O*-acetyl-9-dexoglanduline	$C_{29}H_{39}NO_8$	$CDCl_3$	[652]
2-21-200	13-*O*-acetylganduline	$C_{29}H_{39}NO_9$	$CDCl_3$	[652]
2-21-201	ganduline	$C_{27}H_{37}NO_8$	$CDCl_3$	[652]
2-21-202	13-*O*-acetylvakhmutine	$C_{22}H_{29}NO_5$	$CDCl_3$	[653]
2-21-203	cossonidine	$C_{20}H_{27}NO_2$	$CDCl_3$	[654]
2-21-204	1,15-diacetylcossonidine	$C_{24}H_{31}NO_4$	$CDCl_3$	[654]
2-21-205	15-dehydrocossonidine	$C_{20}H_{25}NO_2$	$CDCl_3$	[654]
2-21-206	1,15-didehydrocossonidine	$C_{20}H_{23}NO_2$	$CDCl_3$	[654]
2-21-207	delnuttaline	$C_{22}H_{27}NO_5$	C_5D_5N	[602]
2-21-208	delnuttidine	$C_{20}H_{25}NO_3$	C_5D_5N	[602]
2-21-209	delnuttine	$C_{22}H_{29}NO_4$	CD_3OD-$CDCl_3$	[602]



2-21-180 R = ^{i}Bu
2-21-181 R = Pr



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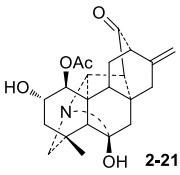

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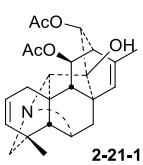

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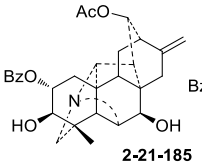

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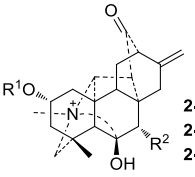
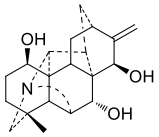


	R^1	R^2
2-21-186	H	H
2-21-187	Ac	H
2-21-188	Ac	OH



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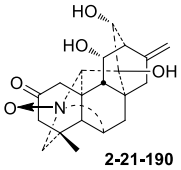

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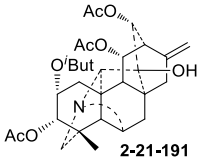

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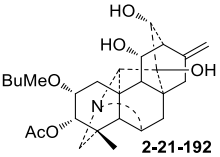

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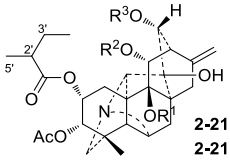
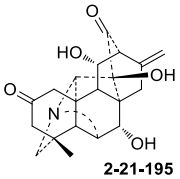


	R^1	R^2	R^3
2-21-193	H	Ac	Ac
2-21-194	Ac	H	H



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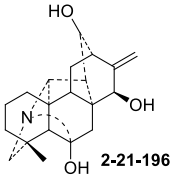

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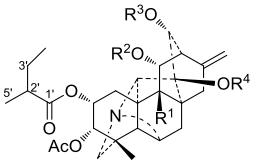
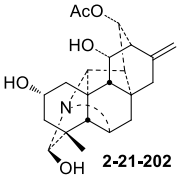
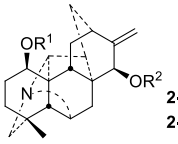


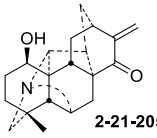
	R^1	R^2	R^3	R^4
2-21-197	H	Ac	Ac	H
2-21-198	H	H	Ac	H
2-21-199	H	H	H	Ac
2-21-200	OH	H	Ac	H
2-21-201	OH	H	H	H



2-21-202



	R^1	R^2
2-21-203	H	H
2-21-204	Ac	Ac



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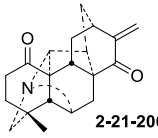

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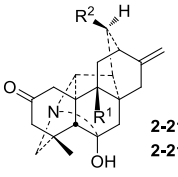
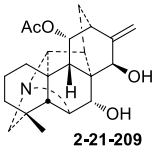


	R^1	R^2
2-21-207	OH	OAc
2-21-208	H	OH



2-21-209

Table 2-21-55: ^{1}H NMR spectroscopic data of C4-type diterpenoid alkaloids **2-21-180**~**2-21-183**.

H	2-21-180	2-21-181	2-21-182	2-21-183
1	2.74, 1.58	1.62 d, 2.93 d(15.2)	6.18 d(3.7)	4.73 d(2.2)
2	5.11 m	5.25 m	5.96 dd(3.0, 5.1)	3.98 m
3	1.52, 1.75	1.58 m, 2.00 m	5.21 d(5.1)	1.67 m, 1.85 m
5	1.49	1.91 m	2.29 s	2.01
6	3.08 br s	4.10 m	3.26 br s ($W_{1/2}$ = 5.5)	
7	1.36	1.56 m	α 2.03 dd(3.5, 14.0)	2.15 d(14.2)
	1.80	2.60 dd(14.6, 2.6)	β 1.55 dd(3.5, 14.0)	2.37 d(14.2)
9	2.17 d	2.39 d(9.0)	2.45 d(9.3)	2.08 m
11	5.02 d(9)	5.11 d(9.0)	5.43 d(9.5)	1.60 m, 1.78 m
12	2.62 d	2.72 m	2.53 d(2.7)	2.92 d(3.4)
13	4.94 m	5.09 br s	5.51 d(1.9)	
14				2.72 d
15	2.08	2.18 d(17.9)	α 2.35 dt(2.5, 18.0)	2.48 m
	1.96	2.34 d(17.9)	β 2.22 dt(2.5, 18.0)	2.58 m
17	4.75 br s, 4.94 br s	4.86 br s, 5.04 br s	5.0 br s, 4.90 br s	4.87 m, 4.98 m
18	0.94 s	1.19 s	1.02 s	1.52 s
19	2.82 d, 2.50 d	3.41 d(12.1), 3.59 d(12.1)	α 3.27 d(12.7), β 2.46 d(12.7)	3.38 d(11.5), 3.49 d(11.5)
20	3.37 s	4.75 s	3.28 s	4.11 s
2′	2.45 m	2.51 q(7.5)	7.55 dd(7.10, 1.0)	
3′	1.13 d	1.16 t(7.5)	6.99 t(7.7)	
4′	1.13 d		7.16 t(7.5)	
5′			6.99 t(7.7)	
6′			7.55 dd(7.10, 1.0)	
2″,6″			7.75 dd(7.1, 1.0)	
3″,5″			7.29 t(7.7)	
4″			7.42 t(7.5)	
1-OAc			2.14 s	2.05 s
3-OAc			1.83 s	
11-OAc	1.96 s	2.03 s	1.97 s	
13-OAc	1.97 s	2.04 s		

Table 2-21-56: ^{1}H NMR spectroscopic data of C4-type diterpenoid alkaloids **2-21-184**~**2-21-187**.

H	2-21-184	2-21-185	2-21-186	2-21-187
1	α 2.61~2.69 m	2.44 dd(4.8, 15.3)	1.59 d(15)	1.65 d(17.5)
	β 2.02~2.10 m	2.18 (ov)	1.75 d(15)	
2	5.48~5.54 m	5.41 m($W_{1/2}$ = 10.2)	4.14 br s	5.11 d(3)
3	5.64 d(8.5)	3.76 br d(1.8)	1.60 d(15), 1.93 t(15)	1.66 d(15.5), 1.92 d(15.5)
5	1.61 br s	2.04 br s	2.15 s	2.17 s
6	2.74 br s	3.35 br s		

Table 2-21-56 (continued)

H	2-21-184	2-21-185	2-21-186	2-21-187
7	*α* 2.24 d(13.8)	3.62 d(3.0)	2.23 d(12.5)	2.23 d(14.5)
	β 1.77 dd(13.8, 2.1)		2.31 d(15)	2.27 d(14.5)
9	1.89 d(9.3)	2.20 (ov)	2.20 s	2.17 s
11	4.84 dd(9.3, 1.4)	1.03 dt(3.0, 14.4)	1.86 d(14)	1.84 m
		1.90 ddd(3.0, 11.1, 14.4)	2.03 d(14.5)	
12	2.68~2.70 m	2.51 (ov)	2.97 br s	2.89 d(3.5)
13	4.88 t(2.0)	5.08 d(4.8)		
14		1.50 br d(1.8)	2.98 br s	2.97 d(2)
15	5.27 t(1.4)	2.20 (ov), 2.77 dt(1.5, 17.7)	2.52 d(17.5), 2.69 d(17)	2.47 d(17),
				2.62 d(17.5)
17	1.86 s	4.74 br s	5.04 br s, 4.94 br s	4.86 br s,
				4.95 br s
18	1.08 s	1.15 s	1.48 s	1.43 s
19	*α* 2.72 d(10.5)	2.52 d(12.6)	3.35 d(11.5)	3.37 d(12)
	β 2.23 d(10.5)	3.05 d(12.6)	4.30 d(11.5)	3.79 d(12)
20	3.10 s	2.95 br s	4.27 s	3.76 s
2′, 6′		7.95 dt(1.5, 7.2)		
3′, 5′		7.45 tt(1.2, 7.2)		
4′		7.57 dt(1.2, 7.2)		
NMe			2.90 s	2.90 s
OAc	2.04 s(11-OAc)	1.99 s(13-OAc)		
	2.08 s(13-OAc)			2.06 s(2-OAc)

Table 2-21-57: ^{1}H NMR spectroscopic data of C4-type diterpenoid alkaloids **2-21-188**~**2-21-191**.

H	2-21-188	2-21-189	2-21-190	2-21-191
1	1.65 d(16.5)	4.20 br s($W_{1/2}$ = 6.0)	*α* 3.70 d(18.3)	*α* 2.85 dd(15.4, 2.5)
	1.74 d(15.5)		*β* 2.14 d(18.4)	*β* 1.81 dd(15.4, 4.6)
2	5.21 s	*α* 1.78 m, *β* 1.75 m		5.47 ddd(4.6, 4.6, 2.5)
3	1.65 d(17)	*α* 1.31 m	*α* 3.19 br d(14.0)	4.92 d(4.8)
	1.93 d(16.5)	*β* 1.80 m	*β* 2.36 d(14.0)	
5	2.12 s	1.93 s	1.41 s	1.80 s
6		3.39 br s($W_{1/2}$ = 7.0)	3.38 br s	3.13 br s($W_{1/2}$ = 7)
7	4.35 s	3.97 d(2.7)	*α* 2.62 dd(14.1, 4.0)	*α* 1.90 dd(14.0, 3.4)
			β 1.61 br d(14.0)	*β* 1.41 dd(14.1, 2.3)
9	2.20 d(7.5)	1.97 m	2.16 d(9.6)	2.23 d(9.0)
11	1.86 d(6.5)	*α* 1.95 m, *β* 1.78 m	4.44 d(9.4)	5.11 d(8.9)
12	2.95 s	2.25 m	2.68 t(2.5)	2.68 d(2.8)
13		*α* 1.08 dt(13.5, 2.8)	4.54 br s	5.02 dd(2.9, 1.7)
		β 1.76 m		
14	3.00 s	2.18 br d(11.1)		
15	2.48 d(17.5)	4.51 s	*α* 2.57 dt(18.0, 2.0)	*α* 2.21 d(16.3)
	2.96 d(17.5)		*β* 1.89 dt(18.0, 2.4)	*β* 2.11 dt(17.9, 2.6)
17	4.92 br s	*Z* 5.00 s	5.04 br s	*Z* 5.01 t(2.1)
	5.00 br s	*E* 4.97 s	4.90 br s	*E* 4.83 br s

Table 2-21-57 (continued)

H	2-21-188	2-21-189	2-21-190	2-21-191
18	1.52 s	1.04 s	1.19 s	1.03 s
19	3.42 d(11.5)	2.49 d(12.6)	3.11 d(12.4)	*α* 3.34 d(12.5)
	3.61 d(10.0)	2.43 d(12.5)	3.14 d(12.4)	*β* 2.50 d(12.5)
20	3.63 s	2.45 s	4.94 br s	3.56 s
2′				2.59 sept(7.0)
3′				1.18 d(6.8)
4′				1.25 d(6.4)
NMe	3.12 s			
OAc	2.01 s(2-OAc)			1.99 s(3-OAc)
				2.01 s(11-OAc)
				1.98 s(13-OAc)

Table 2-21-58: ^{1}H NMR spectroscopic data of C4-type diterpenoid alkaloids **2-21-192**~**2-21-196**.

H	2-21-192	2-21-193	2-21-194	2-21-195	2-21-196
1	*α* 2.91 dd(15.8, 4.3)	*α* 2.65 dd(14.9, 2.0)	*α* 2.95 dd(16.1, 2.4)	1.53	1.35
	β 2.05 dd(16.1, 4.3)	*β* 1.83 dd(14.8, 4.6)	*β* 2.00 dd(16.0, 4.3)	2.03	1.92
2	5.42 ddd(4.4, 4.4, 2.5)	5.45 ddd(4.5, 4.5, 2.2)	5.35 ddd(4.3, 4.3, 2.8)		1.10, 1.55
3	4.95 d(4.9)	4.88 d(4.8)	4.78 d(4.8)	1.75, 1.60	1.58
5	1.80 s	2.62 s	2.76 s	2.03	1.83
6	3.15 br s($W_{1/2}$ = 7)	3.05 br s($W_{1/2}$ = 7)	3.49 br s($W_{1/2}$ = 7)	3.47	
7	*α* 1.88 dd(13.9, 3.3)	*α* 1.65 dd(14.0, 3.0)	*α* 1.83 dd(13.2, 2.9)	4.50	2.12
	β 1.41 dd(13.9, 2.2)	*β* 1.82 dd(14.5, 2.3)	*β* 1.81 dd(14.2, 2.1)		2.35
9	2.00 d(9.3)			2.00	2.10
11	4.25 d(9.0)	4.86 s	3.97 s	4.24	1.95, 2.35
12	2.50 d(2.2)	2.73 d(2.9)	2.38 br s($W_{1/2}$ = 6)	2.90	2.65
13	4.09 t(2.4)	4.98 t(2.7)	3.91 t(2.0)		3.98
14					2.45
15	*α* 2.01 m	*α* 2.22 dt(17.8, 3.1)	*α* 1.87 br d(17.7)	2.30	4.02
	β 2.09 br d(17.8)	*β* 2.03 br d(17.6)	*β* 2.04 dt(17.8, 2.6)		
17	*E* 4.71 br s	*E* 4.85 br s	*E* 4.64 br s	4.86	5.16
	Z 4.90 br s	*Z* 5.02 br s	*Z* 4.82 br s	4.98	5.27
18	1.04 s	1.03 s	1.05 s	1.02	1.42
19	*α* 3.38 d(12.5)	*α* 3.34 d(12.6)	*α* 3.59 d(12.5)	2.31	2.37
	β 2.51 d(12.4)	*β* 2.50 d(12.6)	*β* 2.72 d(12.5)	2.62	2.67
20	3.74 s	3.55 s	4.15 s	3.64	3.86
2′	2.40 sext(7.0)	2.36 sext(7.0)	2.36 sext(7.0)		
3′	1.69 ddq(14.9, 7.5, 7.5)	1.71 ddq(14.0, 7.0, 7.0)	1.59 ddq(14.0, 7.0, 7.0)		
	1.49 ddq(13.8, 7.3, 7.3)	1.49 ddq(14.4, 7.2, 7.2)	1.40 ddq(14.0, 7.0, 7.0)		
4′	0.92 t(7.4)	0.92 t(7.4)	0.83 t(7.4)		
5′	1.20 d(7.0)	1.25 d(7.0)	1.10 d(7.0)		
OAc	1.99 s(3-OAc)	1.99 s(3-OAc)	1.90 s(3-OAc)		
		2.10 s(11-OAc)	1.88 s(9-OAc)		
		2.00 s(13-OAc)			

Table 2-21-59: ^{1}H NMR spectroscopic data of C4-type diterpenoid alkaloids **2-21-197**~**2-21-201**.

H	2-21-197	2-21-198	2-21-199	2-21-200	2-21-201
1α	2.85 dd(15.3, 1.8)	3.07 dd(16.2, 2.2)	3.03 br d(15.5)	3.13 dd(16.6, 2.0)	3.04 br d(16.0)
1β	1.83 dd(15.3, 4.5)	2.07 dd(16.2, 4.4)	2.11 dd(14.5, 5.5)	2.09 dd(16.6, 4.7)	2.15 dd(16.0, 3.7)
2β	5.47 m ($W_{1/2}$ = 14.0)	5.50 m ($W_{1/2}$ = 14.0)	5.46 m ($W_{1/2}$ = 14.0)	5.50 m ($W_{1/2}$ = 14.0)	5.45 m ($W_{1/2}$ = 14.0)
3β	4.92 d(4.7)	4.98 d(4.4)	4.95 d(4.6)	4.90 d(4.7)	4.90 d(4.7)
5	1.80 s	1.79 s	1.98 s	2.59 s	2.72 br s
6	3.14 br s ($W_{1/2}$ = 6.2)	3.13 br s ($W_{1/2}$ = 6.4)	3.51 br s ($W_{1/2}$ = 6.3)	3.10 br s ($W_{1/2}$ = 6.1)	3.34 br s ($W_{1/2}$ = 6.4)
7α	1.91 dd(14.0, 3.3)	1.89 dd(14.0, 3.4)	2.16 dd(14.0, 3.5)	1.70 dd(13.4, 3.0)	1.80 d(18.0)
7β	1.44 dd(14.0, 2.0)	1.41 dd(14.0, 2.5)	1.50 br d(14.0)	1.75 dd(13.8, 2.2)	1.85 d(18.0)
9	2.23 d(9)	2.04 d(8.9)	2.08 d(8.7)		
11β	5.11 d(9)	4.28 d(8.9)	4.24 d(8.8)	4.10 s	4.12 s
12	2.68 d(2.4)	2.64 d(2.5)	2.56 s	2.65 d(2.2)	2.51 d(1.8)
13β	5.02 br s	5.06 t(2.2)	4.14 s	4.96 d(2.2)	4.09 br s
15α	2.20 d(14.0)	2.17 d(17.9)	2.15 d(17.7)	2.04 d(18.0)	2.10 d(16.0)
15β	2.12 d(14.0)	2.02 m	2.04 d(17.7)	1.99 d(18.0)	2.00 d(16.0)
17*E*	4.83 s	4.77 s	4.73 s	4.78 s	4.74 s
17*Z*	5.02 s	4.97 s	4.93 s	4.97 s	4.91 s
18	1.02 s	1.02 s	1.12 s	1.03 s	1.10 s
19α	3.34 d(12.5)	3.35 d(12.5)	3.65 d(12.5)	3.38 d(12.5)	3.59 d(12.5)
19β	2.50 d(12.5)	2.50 d(12.5)	2.73 d(12.5)	2.54 d(12.5)	2.70 d(12.5)
20	3.57 s	3.54 s	4.21 s	3.62 s	4.06 s
2′	2.38 sext(7.4)	2.35 sext(7.0)	2.46 sext(7.0)	2.36 sext(7.0)	2.45 sext(7.0)
3′	1.70 ddq(4.8, 7.4, 7.4)	1.69 ddq(14.0, 7.0, 7.0)	1.70 ddq(14.0, 7.0, 7.0)	1.68 ddq(14.6, 7.3, 7.3)	1.70 ddq(14.0, 7.0, 7.0)
	1.50 ddq(14.8, 7.4, 7.4)	1.48 ddq(14.0, 7.0, 7.0)	1.49 m(14.0, 7.0, 7.0)	1.48 ddq(14.6, 7.3, 7.3)	1.49 ddq(14.0, 7.0, 7.0)
4′	0.92 t(7.4)	0.89 t(7.4)	0.94 t(7.4)	0.89 t(7.4)	0.92 t(7.4)
5′	1.24 d(7.0)	1.25 d(7.0)	1.21 d(7.0)	1.23 d(7.0)	1.18 d(17.0)
OAc	2.02 s(3-OAc)	2.01 s(3-OAc)	2.00 s(3-OAc)	2.02 s(3-OAc)	2.01 s(3-OAc)
	2.00 s(111-OAc)	1.99 s(13-OAc)	1.99 s(14-OAc)	1.99 s(13-OAc)	
	1.99 s(13-OAc)				

Table 2-21-60: ^{1}H NMR spectroscopic data of C4-type diterpenoid alkaloids **2-21-202**~**2-21-206**.

H	2-21-202	2-21-203	2-21-204	2-21-205	2-21-206
1	α 2.66 br d(15.0)	4.19 br s	5.23 br s	4.29 br s ($W_{1/2}$ = 6.4)	
	β 1.82 dd(15.0, 4.0)				
2	4.18 br s	α 1.79 m, β 1.77 m	–	–	–

Table 2-21-60 (continued)

H	2-21-202	2-21-203	2-21-204	2-21-205	2-21-206
3	*α* 1.98 br d(7.8) *β* 1.55 dd(7.8, 2.1)	*α* 1.25 m *β* 1.74 m	*α* 1.29 br d(13.2) *β* –	–	–
5	1.45 s	1.89 s	1.86 s	1.89 s	2.12 s
6	3.55 br s ($W_{1/2}$ = 4.0)	3.40 br s ($W_{1/2}$ = 6)	3.37 br s ($W_{1/2}$ = 7)	3.39 br s ($W_{1/2}$ = 7)	3.48 br s ($W_{1/2}$ = 7)
7*α*	1.71 dd(14.0, 2.7)	1.68 dd(13.2, 3.1)	–	–	–
7*β*	1.56 m	2.02 dd(13.2, 2.4)	–	–	–
9	1.91 d(9.0)	2.01 d(11.5)	–	–	–
11	4.23 d(9.0)	*α* 1.92 dd(14.2, 4.2) *β* 1.76 m	*β* 1.53 dd(13.8, 9.2)	*α* 2.14 dd(14.2, 4.0)	*α* 2.47 dd(14.2, 5.2)
12	2.42 d(2.5)	2.21 m	2.17 t(3.2)	2.64 m	2.54 br s ($W_{1/2}$ = 10)
13	5.00 br d(9.0)	*α* 1.07 dt(13.2, 2.7) *β* 1.80 m	*α* 1.10 dt(13.5, 2.8)	–	*α* 1.34 dt(13.6, 2.8)
14	2.38 d(9.0)	1.90 m	–	2.19 dt(11.9, 1.6)	–
15	*α* 2.03 AB(18.0) *β* 2.18 AB(18.0)	4.00 s	5.43 s	–	–
16				2.64 m	
17	*E* 4.86 s	*E* 4.94 s	*E* 4.92 d(1.4)	*E* 5.08 d(1.5)	*E* 5.08 d(1.5)
17	*Z* 4.70 s	*Z* 4.97 s	*Z* 4.95 t(1.3)	*Z* 5.89 d(1.5)	*Z* 5.89 d(1.5)
18	1.00 s	1.02 s	1.02 s	1.03 s	1.05 s
19*α*	4.71 s	2.39 d(12.5)	2.38 d(12.6)	2.40 d(12.5)	2.70 d(12.7)
19*β*		2.56 d(12.5)	2.57 d(12.6)	2.54 d(12.5)	2.77 d(12.7)
20	3.28 s	2.49 s	2.56 s	2.61 s	3.36 s
OAc	2.22 s(13-OAc)		2.09 s, 2.11 s		

Table 2-21-61: ^{1}H NMR spectroscopic data of C4-type diterpenoid alkaloids **2-21-207**~**2-21-209**.

H	2-21-207	2-21-208	2-21-209
1	*α* 2.87 d(13) *β* 2.68 d(13.0)	3.29 d(13.7) 2.35 d(13.7)	1.20 m 1.33~1.40 m
2			1.50~1.55 m, 1.33~1.40 m
3	*α* 2.34 d(13.9) *β* 2.49 d(13.9)	2.52 d(14.5) 2.40 d(14.5)	2.17~2.26 m 1.33~1.40 m
5	3.08 s	2.25 s	1.50 s
6			3.20 br s
7	2.77 d(12.7), 1.90 d(12.7)	2.74 d(13.7), 2.27 d(13.7)	3.87 d(2.8)
9		1.91 d(8.7)	2.17~2.26 m
11	*α* 2.60 d(14.2) *β* 1.79 d(14.5)	*α* 2.12 d(14.5) *β* 1.49 dd(8.7, 14.5)	5.18 d(8.3)

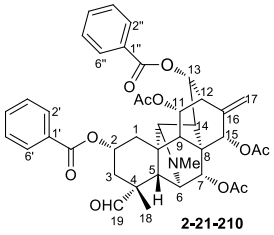
Table 2-21-61 (continued)

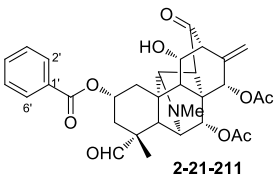
H	2-21-207	2-21-208	2-21-209
12	2.41 br s		2.17~2.26 m
13	5.09 br d(9.6)	4.24 d(9.3)	1.33~1.40 m
14	2.62 d(9.6)	3.37 d(9.3)	2.07 br d(10.5)
15	2.58 d(17.8), 1.92 d(17.8)	2.17 d(17.4), 1.95 d(17.4)	4.37 s
17	4.91 br s, 4.70 br s	4.80 br s, 4.61 br s	5.02 br s, 4.97 br s
18	1.68 s	1.65 s	0.93 s
19	3.55 d(12.2), 2.38 d(12.2)	3.85 d(12.1), 2.83 d(12.1)	2.39 s
20	2.55 s	3.90 s	2.58 s
OAc	2.27 s(13-OAc)		1.99 s(11-OAc)

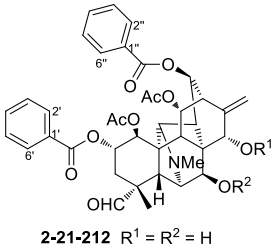
2.21.11 C5-type diterpenoid alkaloids

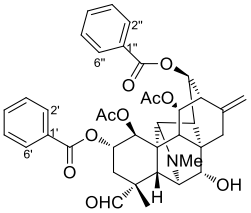
Table 2-21-62: Cos, MFs, and TSs of C5-type diterpenoid alkaloids **2-21-210**~**2-21-216**.

No.	Compounds	MFs	Test solvents	References
2-21-210	tangutisine A	$C_{41}H_{43}NO_{11}$	$CDCl_3$	[655]
2-21-211	tangutisine B	$C_{32}H_{35}NO_9$	$CDCl_3$	[656]
2-21-212	trifoliolasine E	$C_{39}H_{41}NO_{11}$	CD_3OD-$CDCl_3$	[657]
2-21-213	trifoliolasine D	$C_{43}H_{45}NO_{13}$	$CDCl_3$	[657]
2-21-214	trifoliolasine F	$C_{39}H_{41}NO_{10}$	$CDCl_3$	[657]
2-21-215	barbaline	$C_{34}H_{37}NO_{11}$	$CDCl_3$	[658]
2-21-216	7-acetylbarbaline	$C_{36}H_{39}NO_{12}$	$CDCl_3$	[658]









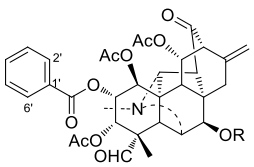


Table 2-21-63: ^{1}H NMR spectroscopic data of C5-type diterpenoid alkaloids **2-21-210**~**2-21-213**.

H	2-21-210	2-21-211	2-21-212	2-21-213
1	*α* 2.98 dd(3.6, 15.1)	*α* 2.23 br d(14.5)	5.93 d(3.2)	5.86 d(4.0)
	β 1.86 dd(3.6, 15.1)	*β* 1.95 dd(3.6, 15.0)		
2	5.51 m	5.55 m	5.76 m	5.80 d(3.2)
3*α*	2.29 br d(15.5)	2.47 br s	2.01 s	2.05 t(15.6)
3*β*	1.49 dd(3.2, 15.5)	1.53 dd(3.2, 15.5)	1.81 dd(16.0, 4.0)	1.72 dd(15.6, 3.6)
5	2.04 s	2.08 s	2.54 s	2.23 s
6	3.05 d(3.3)	3.18 d(3.3)	3.18 d(4.0)	3.15 d(3.6)
7	5.28 d(3.6)	5.30 d(3.5)	4.37 d(4.0)	5.28 d(3.6)
9	3.01 br s	2.76 d(2.2)	2.97 dd(9.2, 2.0)	2.90 dd(9.6, 2.4)
11	5.57 dd(9.0, 2.6)	4.14 d(4.5)	5.35 dd(10.0, 2.4)	5.45 d(9.6)
12	2.81 d(2.6)	3.34 d(4.5)	2.67 d(2.4)	2.69 d(2.0)
13	5.14 m		5.37 d(9.6)	5.35 dt(10.0, 2.0)
14	2.91 dd(1.9, 9.7)	2.51 br d(2.3)	3.26 dt(10.0, 2.0)	3.25 dd(10.0, 2.0)
15	5.68 s	5.94 s	4.58 t(2.0)	5.76 d(2.0)
17	5.36 s	5.53 s	5.26 d(2.4)	5.10 d(2.0)
	5.23 s	5.38 s	5.41 d(1.2)	5.26 d(2.0)
18	1.00 s	1.05 s	1.07 s	1.03 s
19	9.37 s	9.50 s	8.93 br s	9.05 br s
20	3.67 s	3.38 br s	3.90 s	3.93 s
NMe	2.43 s	2.40 s	2.47 s	2.48 s
2′/6′	7.62 dd(1.1, 7.3)		7.75 dd(8.4, 1.2)	7.76 dd(8.0, 1.2)
3′/5′	7.33 (ov)		7.06 t(7.6)	7.10 t(8.0)
4′	7.49 m		7.30 m	7.30 m
2″/6″	7.75 dd(1.2, 7.5)	7.89 dd(1.1, 7.2)	7.53 m	7.55 dd(8.0, 1.2)
3″/5″	7.08 (ov)	7.44 (ov)	7.33 m	7.29 t(8.0)
4″	7.33 (ov)	7.57 m	7.51 m	7.47 m
1-OAc			2.02 s	2.10 s
7-OAc	2.17 s	2.13 s		2.01 s
11-OAc	2.09 s		2.11 s	2.13 s
15-OAc	2.07 s	2.07 s		2.10 s

Table 2-21-64: ^{1}H NMR spectroscopic data of C5-type diterpenoid alkaloids **2-21-214**~**2-21-216**.

H	2-21-214	2-21-215	2-21-216
1	5.91 d(3.6)	5.55 d(4.2)	5.57 d(4.2)
2	5.78 dd(6.4, 3.6)	6.09 t(4.2)	6.09 t(4.2, 3.9)
3	*α* 2.01 m, *β* 1.71 dd(16.0, 3.6)	5.22 d(3.9)	5.23 d(3.9)
5	1.94 s	2.52 s	2.35 s
6	3.27 br s	3.03 br d(4.0)	3.06 br d(4.0)
7	3.76 d(1.5)	3.94 d(4.0)	5.12 d(4.0)
9	2.41 dd(9.6, 2.0)	2.96 dd(4.0, 9.5)	2.89 dd(4.0, 9.5)
11	5.49 d(9.6)	5.43 dd(2.0, 9.5)	5.46 br dd(9.5, 2.0)

Table 2-21-64 (continued)

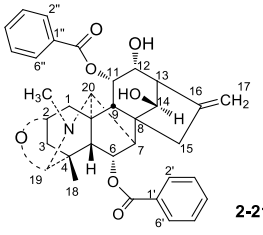
H	2-21-214	2-21-215	2-21-216
12	2.56 d(2.4)	2.84 d(2.0)	2.83 d(2.0)
13	5.32 dt(10.0, 2.0)		
14	3.32 dd(9.6, 2.0)	2.80 br d(4.0)	2.88 br d(4.0)
15	*α* 2.83 dt(18.0, 1.0)	2.93 dt(18.0, 1.5)	2.64 dt(18.0, 1.5)
	β 2.18 m	2.77 d(18.0)	2.29 br d(18.0)
17	4.89 br s, 5.04 br s	5.06 br s, 4.96 br t(1.5)	5.05 br t(1.5), 4.95 br s
18	1.06 s		
19	8.87 s	9.69 br s	9.81 br s
20	3.92 s	3.81 s	3.81 s
NMe	2.61 s	2.43 s	2.47 s
2′, 6′	7.79 d(8.4)	7.98 m, 7.48~7.66 m	7.88 m①
3′, 5′	7.06 t(8.0)	7.98 m, 7.48~7.66 m	7.55 m①
4′	7.26 m	7.98 m, 7.48~7.66 m	7.62 m①
2″, 6″	7.55 d(8.4)		
3″, 5″	7.28 t(8.0)		
4″	7.46 m		
1-OAc	2.05 s	2.03 s	2.05 s
3-OAc		1.96 s	1.96 s
7-OAc			2.18 s
11-OAc	1.99 s	2.10 s	2.12 s
18-Me		1.16 s	1.12 s

① Data not specifically assigned in the literature.

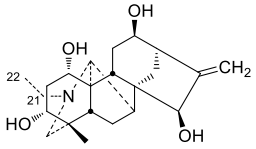
2.21.12 C6-type diterpenoid alkaloids

Table 2-21-65: Cos, MFs, and TSs of C6-type diterpenoid alkaloids **2-21-217**~**2-21-220**.

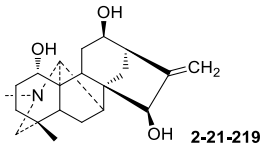
No.	Compounds	MFs	Test solvents	References
2-21-217	racemulotine	$C_{35}H_{37}NO_7$	$CDCl_3$	[659]
2-21-218	3*α*-hydroxy-12-*epi*-napelline	$C_{22}H_{33}NO_4$	$CDCl_3$	[660]
2-21-219	*N*-deethyl-*N*-methyl-12-*epi*-napelline	$C_{21}H_{31}NO_3$	$CDCl_3$	[634]
2-21-220	16,17-dihydro-12*β*,16*β*-epoxynapelline	$C_{22}H_{33}NO_3$	$CDCl_3$	[634]



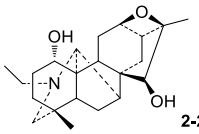
2-21-217



2-21-218



2-21-219



2-21-220

Table 2-21-66: ^{1}H NMR spectroscopic data of C6-type diterpenoid alkaloids **2-21-217**~**2-21-220**.

H	2-21-217	2-21-218	2-21-219	2-21-220
1	*α* 3.48 d(16.8) *β* 2.18 dd(17.2, 5.2)	3.76 br	3.91 dd(8, 6)	3.88 dd(8, 7)
2	5.56 br s($W_{1/2}$ = 6.0)	*α* 1.86 m, *β* 1.52 m	*α* 1.99 m, *β* 1.87 m	*α* 2.22 m, *β* 1.91 m
3	*α* 1.99 d(16.2) *β* 1.76 dd(16.2, 4.4)	3.78 br	*α* 1.66 m *β* 1.33 m	*α* 1.61 dt(13, 4) *β* 1.33 m
5	2.39 br s	1.32 m	1.35 br s	1.35 d(8)
6	4.00 m($W_{1/2}$ = 8.0)	*α* 2.40 d(8.0), *β* 1.29 m	*α* 1.36 m *β* 2.32 dd(13, 8)	*α* 1.29 dd(13, 5) *β* 2.56 dd(13, 8)
7	2.39 s	2.11 m	2.11 d(5)	2.07 d(5)
9	2.57 d(2.8)	2.07 m	1.84 m	1.94 d(7)
11	5.31 dt(10.2, 2.0)	*α* 2.40 m *β* 1.67 ddd, (14.4, 6.8, 2.0)	*α* 2.16 dd(14, 5) *β* 1.62 m	*α* 2.11 m *β* 1.86 m
12	4.36 d(9.2)	4.18 dt(8.8, 2.0)	4.19 dd(9, 6)	4.82 dd(8, 4)
13	3.13 dd(9.8, 1.8)	2.81 dd(8.8, 3.2)	2.80 dd(9, 4)	2.72 dd(8, 4)
14	3.71 s	*α* 1.13 dd(12.0, 3.2) *β* 1.81 d(12.0)	*α* 1.75 d(12) *β* 1.09 dd(12, 4)	*α* 1.77 d(13) *β* 1.06 m
15	*α* 2.19 br d(17.2) *β* 2.74 br d(18.0)	4.25 br	4.23 br s	3.51 s
17	4.85 br s, 4.97 br s	5.10 br, 5.29 br	5.34 br s, 5.12 br s	1.38 s
18	1.07 s	0.76 s	0.76 s	0.74 s
19	5.45 s	*α* 2.42 m *β* 2.15 m	*α* 2.38 d(11) *β* 2.26 d(11)	*α* 2.47 d(11) *β* 2.18 m
20	3.71 br s	3.24 br	3.19 br s	3.42 br s
21	2.97 s	2.48 m, 2.58 m	2.29 s	2.51 m, 2.38 m
22		1.17 t(7.2)		1.04 t(7)
2′, 6′	7.36 dd(8.4, 1.2)			
3′, 5′	7.25 t(7.6)			
4′	7.44 ddt(7.6, 7.6, 1.2)			
2″, 6″	7.66 dd(8.4, 1.2)			
3″, 5″	6.94 t(7.6)			
4″	7.19 ddt(7.6,7.6,1.2)			

2.21.13 C8-type diterpenoid alkaloids

Table 2-21-67: Co, MF, and TS of C8-type diterpenoid alkaloid **2-21-221**.

No.	Compound	MF	Test solvent	Reference
2-21-221	racemulosine	$C_{22}H_{32}N_2O_3$	$CDCl_3$-CD_3OD	[661]

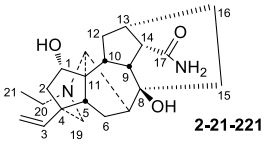


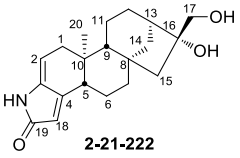
Table 2-21-68: ^{1}H NMR spectroscopic data of C8-type diterpenoid alkaloid **2-21-221**.

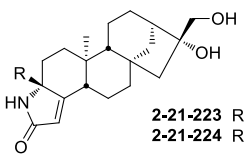
H	2-21-221	H	2-21-221
1	3.86 d(8.8)	12eq	2.15 m
2ax	1.63 m	13	2.54 m
2eq	2.24 dd(14.4, 9.6)	14	2.66 t(4.0)
3	5.63 dd(17.6, 11.2)	15	1.72 m
5	1.93 t(7.8)	16ax	1.70 m
6ax	1.50 m	16eq	2.10 m
6eq	2.00 m	18	4.90 dd(17.2, 1.2), 4.95 dd(11.0, 1.2)
7	2.10 m	19	2.37 ABq(10.8), 2.61 ABq(10.8)
9	2.38 m	20	3.00 br s
10	2.08 m	21	2.58 q(7.0)
12ax	1.30 m	22	1.11 t(7.2)

2.21.14 C10-type diterpenoid alkaloids

Table 2-21-69: Cos, MFs, and TSs of C10-type diterpenoid alkaloids **2-21-222**~**2-21-225**.

No.	Compounds	MFs	Test solvents	References
2-21-222	tricalysiamide A	$C_{20}H_{27}NO_3$	C_5D_5N	[662]
2-21-223	tricalysiamide B	$C_{20}H_{29}NO_3$	C_5D_5N	[662]
2-21-224	tricalysiamide C	$C_{21}H_{31}NO_4$	C_5D_5N	[662]
2-21-225	tricalysiamide D	$C_{20}H_{31}NO_4$	C_5D_5N	[662]





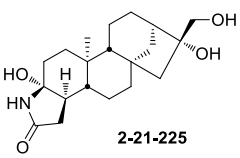


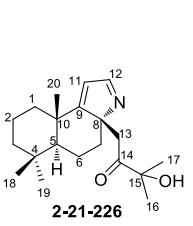
Table 2-21-70: ^{1}H NMR spectroscopic data of C10-type diterpenoid alkaloids **2-21-222**~**2-21-225**.

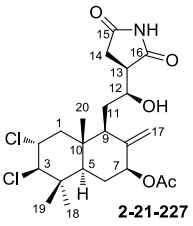
H	2-21-222	2-21-223	2-21-224	2-21-225
1	2.35 dd(17.5, 6.7)	1.68 m	1.62 m	1.76 m
	1.84 m	0.97 td(13.7, 3.7)	1.38 td(13.6, 4.0)	1.08 m
2	5.48 d(6.7)	2.14 m	2.33 dt(13.6, 2.4)	2.36 m
		1.47 m	1.82 m	2.33 m
3		3.90 dd(10.9, 7.2)		
4				2.24 dd(11.7, 6.6)
5	2.21 dt(11.8, 2.3)	1.88 m	2.08 m	0.97 t(12.1)
6	1.71 m	1.51 m	1.44 m	1.42 m
	1.44 m	1.46 m	1.56 m	1.22 td(11.9, 3.1)
7	1.66 dt(13.0, 3.0)	1.64 m	1.66 m	1.61 m
	1.56 td(13.0, 3.6)	1.53 m	1.57 d(9.1)	1.36 m
9	1.26 d(8.7)	1.15 d(8.8)	1.25 d(8.7)	0.93 d(7.2)
11	1.71 m, 1.48 m	1.71 m, 1.49 m	1.72 m, 1.49 m	1.70 m, 1.56 m
12	1.89 m, 1.49 m	1.87 m, 1.47 m	1.89 m, 1.48 m	1.90 m, 1.49 m
13	2.46 d-like(3.0)	2.45 d-like(2.9)	2.47 d-like(2.9)	2.47 s-like
14	2.05 dd(11.6, 4.5)	2.05 dd(11.4, 4.1)	2.07 m	2.05 dd(10.9, 3.3)
	1.94 d(11.6)	1.96 d(11.4)	1.98 d(11.4)	2.00 d(10.9)
15	1.85 d(13.6)	1.85 d(14.3)	1.87 d(14.2)	1.84 d(14.0)
	1.72 d(13.6)	1.73 dd(14.3, 1.3)	1.76 d(14.2)	1.72 d(14.0)
17	4.15 dd(10.9, 5.3)	4.15 dd(10.9, 5.2)	4.15 dd(10.9, 5.3)	4.15 dd(10.7, 4.5)
	4.07 dd(10.9, 5.3)	4.06 dd(10.9, 5.2)	4.06 dd(10.9, 5.3)	4.06 dd(10.7, 4.5)
18	5.98 s	5.83 s	5.93 s	3.21 dd(16.4, 6.5)
				2.35 m
20	0.93 s	0.71 s	0.77 s	1.04 s
NH	10.83 s	8.87 s	9.29 s	9.16 s
3-OMe			3.09 s	
3-OH				7.35 s
16-OH	5.22 s	5.22 s	5.19 s	5.17 s
17-OH	6.13 t(5.3)	6.12 t(5.2)	6.14 t(5.3)	6.15 t(4.5)

2.21.15 M-type diterpenoid alkaloids

Table 2-21-71: Cos, MFs, and TSs of M-type diterpenoid alkaloids **2-21-226**~**2-21-235**.

No.	Compounds	MFs	Test solvents	References
2-21-226	chamobtusin A	$C_{20}H_{31}NO_2$	CD_3OD	[663]
2-21-227	haterumaimide A	$C_{22}H_{31}Cl_2NO_5$	DMSO-d_6	[664]
2-21-228	haterumaimide B	$C_{20}H_{27}Cl_2NO_4$	DMSO-d_6	[664]
2-21-229	haterumaimide C	$C_{20}H_{27}Cl_2NO_4$	CD_3OD	[664]
2-21-230	haterumaimide D	$C_{20}H_{27}Cl_2NO_4$	CD_3OD	[664]
2-21-231	haterumaimide E	$C_{20}H_{29}Cl_2NO_4$	DMSO-d_6	[664]
2-21-232	haterumaimide F	$C_{20}H_{30}ClNO_4$	DMSO-d_6	[665]
2-21-233	haterumaimide G	$C_{20}H_{28}ClNO_4$	DMSO-d_6	[665]
2-21-234	haterumaimide H	$C_{20}H_{28}ClNO_4$	DMSO-d_6	[665]
2-21-235	haterumaimide I	$C_{20}H_{28}ClNO_4$	DMSO-d_6	[665]


2-21-226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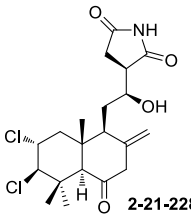
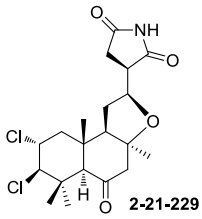
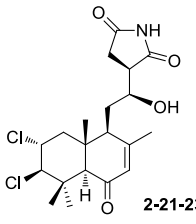
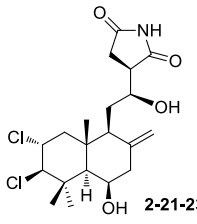
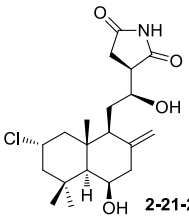
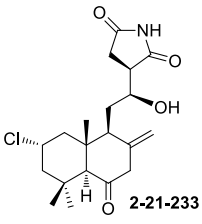

2-21-228

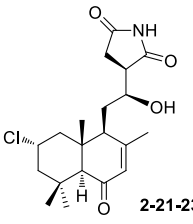

2-21-229


2-21-230


2-21-231


2-21-232


2-21-233


2-21-234

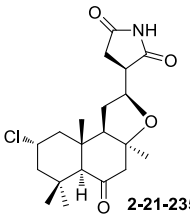

2-21-235

Table 2-21-72: ^{1}H NMR spectroscopic data of M-type diterpenoid alkaloids **2-21-226**~**2-21-230**.

H	2-21-226	2-21-227	2-21-228	2-21-229	2-21-230
1	1.82 m	*α* 1.62 dd(13.0, 2.0)	*α* 1.84 dd(13.0, 12.5)	*α* 1.83 dd(13.3, 12.3)	*α* 1.90 dd(12.8, 12.1)
	1.67 m	*β* 2.24 dd(13.0, 4.2)	*β* 2.34 dd(13.0, 4.5)	*β* 2.29 dd(13.3, 4.2)	*β* 2.47 dd(12.8, 4.2)
2	1.58 m	4.45 td(12.0, 4.2)	4.48 td(12.5, 4.5)	4.22 ddd(12.3, 11.0, 4.2)	4.28 ddd(12.1, 11.0, 4.2)
3	1.45 m 1.22 m	3.90 d(11.0)	3.86 d(11.0)	3.79 d(11.0)	3.78 d(11.0)
5	0.75 m	1.63 dd(12.0, 2.0)	2.84 s	2.34 s	2.55 s
6	1.74 m	*α* 1.65 m *β* 1.40 q(12.0)			
7	0.65 m	*α* 5.09 m	*α* 2.89 d(13.5)	*α* 2.56 d(18.7)	*α* 5.81 dd(2.8, 1.3)
	2.61 m		*β* 3.32 d(13.5)	*β* 2.72 d(18.7)	
9		1.70 dd(10.9, 4.0)	2.27 br t(9.5)	2.26 dd(9.0, 7.7)	2.50 dddd(7.0, 2.8, 2.0, 1.3)

Table 2-21-72 (continued)

H	2-21-226	2-21-227	2-21-228	2-21-229	2-21-230
11	6.09 br s	*α* 1.98 ddd(13.0, 7.0, 4.0) *β* 1.40 ddd(13.0, 10.9, 7.0)	*α* 1.64 ddd(14.0, 9.5, 4.0) *β* 1.49 dt(14.0, 9.5)	*α* 2.20 ddd(12.9, 9.0, 5.7) *β* 1.73 ddd(12.9, 10.1, 7.7)	*α* 1.72 dt(15.2, 7.0) *β* 1.61 ddd(15.2, 7.5, 2.0)
12	7.96 br s	3.95 td(7.0, 2.1)	4.01 m	4.33 ddd(10.1, 5.7, 4.3)	4.25 ddd(7.5, 7.0, 2.4)
13	3.38 d(17.8) 3.52 d(17.8)	2.85 ddd (10.8, 4.5, 2.1)	2.96 ddd (9.0, 5.0, 2.0)	3.12 ddd(9.1, 5.1, 4.3)	3.13 ddd(9.2, 4.7, 2.4)
14*α*		2.49 dd(17.6, 10.8)	2.53 dd(18.0, 9.0)	2.75 dd(18.0, 9.1)	2.66 dd(17.8, 9.2)
14*β*		2.55 dd(17.6, 4.5)	2.62 dd(18.0, 5.0)	2.64 dd(18.0, 5.1)	2.79 dd(17.8, 4.7)
16	1.20 s				
17	1.19 s	5.09 br s, 4.86 br s	4.96 br s, 4.88 br s	1.29 s	2.04 t(1.3)
18	0.88 s	1.05 s	1.14 s	1.41 s	1.39 s
19	0.97 s	0.85 s	1.21 s	1.23 s	1.25 s
20	1.15 s	0.70 s	0.63 s	1.08 s	0.95 s
22		2.10 s			
NH		11.03 s	11.10 s		
OH		3.34 br s	5.05 d(5.0)		

Table 2-21-73: ^{1}H NMR spectroscopic data of M-type diterpenoid alkaloids **2-21-231**~**2-21-235**.

H	2-21-231	2-21-232	2-21-233	2-21-234	2-21-235
1*α*	1.59 t(12.5)	1.31 t(12.0)	1.50 t(12.0)	1.59 t(12.0)	1.52 t(12.0)
1*β*	2.20 dd(12.5, 4.5)	2.06 ddd(12.0, 4.0, 1.5)	2.20 ddd(12.0, 4.0, 1.5)	2.21 ddd(12.0, 4.0, 1.5)	2.01 ddd(12.0, 3.5, 1.5)
2	4.40 td(12.5, 4.5)	4.33 tt(12.0, 4.0)	4.34 tt(12.0, 4.0)	4.33 tt(12.0, 4.0)	4.27 tt(12.0, 3.5)
3	3.84 d(11.0)	*α* 1.45 t(12.0) *β* 1.81 ddd(12.0, 4.0, 1.5)	*α* 1.43 t(12.0) *β* 1.78 ddd(12.0, 4.0, 1.5)	*α* 1.48 dd(12.5, 12.0) *β* 1.80 ddd(12.0, 4.0, 1.5)	*α* 1.52 t(12.0) *β* 1.79 ddd(12.0, 3.5, 1.5)
5	1.34 br d(3.0)	1.11 br d(4.0)	2.51 s	2.26 s	2.10 s
6	*α* 4.23 m	4.23 m			
7*α*	2.18 dd(13.5, 4.5)	2.15 br d(13.0)	3.35 d(13.5)		2.61 d(18.5)
7*β*	2.27 dd(13.5, 2.5)	2.26 dd(13.0, 2.5)	2.84 d(13.5)	5.73 q(1.5)	2.43 d(18.5)
9	1.63 dd(8.5, 5.5)	1.63 m	2.29 br dd(8.5, 7.5)	2.47 ddd(7.5, 5.4, 1.5)	2.17 dd(9.0, 7.5)

Table 2-21-73 (continued)

H	2-21-231	2-21-232	2-21-233	2-21-234	2-21-235
11	α 1.64 m	1.63 m	1.62 ddd(12.5, 8.5, 2.5)	1.56 ddd(12.0, 9.5, 5.4)	2.10 dt(12.0, 9.0)
	β 1.37 m	1.36 ddd(11.5, 10.5, 6.0)	1.48 m	1.46 m	1.54 ddd(12.0, 9.0, 5.5)
12	4.00 dddd(10.5, 5.5, 4.5, 1.5)	4.00 dddd(10.5, 5.5, 5.0, 2.5)	4.05 m	4.03 dddd(9.5, 5.5, 4.5, 2.5)	4.24 ddd(9.0, 5.5, 3.0)
13	2.84 ddd(9.5, 5.0, 1.5)	2.86 ddd(9.5, 4.5, 2.5)	2.98 ddd(9.0, 4.8, 2.5)	3.08 ddd(9.5, 4.5, 2.5)	3.05 ddd(9.0, 5.0, 3.0)
14	α 2.44 dd(17.5, 9.5)	2.54 dd(17.5, 5.0)	2.59 dd(17.0, 5.0)	2.63 dd(17.5, 4.5)	2.66 dd(17.5, 9.0)
	β 2.55 dd(17.5, 5.0)	2.45 dd(17.5, 9.5)	2.54 dd(17.0, 9.0)	2.51 dd(17.5, 9.5)	2.50 dd(17.5, 5.0)
17	4.88 br s, 4.84 br s	4.86 br s, 4.82 br s	4.92 br s, 4.85 br s	1.94 br d(1.5)	1.20 s
18	1.14 s	0.97 s	0.95 s	1.11 s	1.15 s
19	1.24 s	1.17 s	1.15 s	1.12 s	1.11 s
20	0.95 s	0.94 s	0.58 s	0.80 s	0.94 s
NH	11.03 s	11.01 s	11.05 s	11.11 s	11.10 s
OH	4.94 d(4.5) 4.36 d(4.0)				
12-OH		4.91 d(5.0)	5.04 d(4.5)	5.29 d(4.5)	
6-OH		4.15 d(3.5)			

Bibliography

[555] Tan JJ, Tan CH, Ruan BQ, et al. J Asian Nat Prod Res, 2006, 8: 535.
[556] Csupor D, Forgo P, Wenzig EM, et al. J Nat Prod, 2008, 71: 1779.
[557] Feng F, Ye WC, Liu JH, et al. J Chin Pharm Sci, 2000, 9: 167.
[558] Ulubelen A, Meriçli AH, Meriçli F, et al. Phytochemistry, 1999, 50: 513.
[559] Wang FP, Peng CS, Jian XX, et al. J Asian Nat Prod Res, 2001, 3: 15.
[560] Shamma M, Chinnasamy P, Miana GA, et al. J Nat Prod, 1979, 42: 615.
[561] Ross SA, Pelletier SW, Aasen AJ. Tetrahedron, 1992, 48: 1183.
[562] Ding LS, Chen WX. Acta Pharm Sin, 1990, 25: 441.
[563] Cai L, Chen DL, Wang FP. Nat Prod Commun, 2006, 1: 191.
[564] Yu DQ, Das BC. Planta Med, 1983, 49: 85.
[565] Joshi BS, Pelletier SW, Zhang XL, et al. Tetrahedron, 1991, 47: 4299.
[566] Peng CS, Wang FP, Wang JZ, et al. Acta Pharm Sin, 2000, 35: 201.
[567] Song L, Liang XX, Chen DL, et al. Chem Pharm Bull, 2007, 55: 918.
[568] Kolak U, Öztürk M, Özgökçe F, et al. Phytochemistry, 2006, 67: 2170.
[569] Fuente G, Orribo T, Gavin J, et al. Heterocycles, 1993, 36: 1455.
[570] Lao AN, Wang HC, Uzawa J, et al. Heterocycles, 1990, 31: 27.
[571] Alva A, Grandez M, Madinaveitia A, et al. Helv Chim Acta, 2004, 87: 2110.
[572] Zinurova E, Khakimova T, Spirikhin L, et al. Chem Nat Compd, 2000, 36: 387.
[573] Almanza G, Bastida J, Codina C, et al. Phytochemistry, 1997, 45: 1079.

[574] Li J, Chen DL, Jian XX, et al. Molecules, 2007, 12: 353.
[575] Díaz JG, Ruiza JG, Herz W. Phytochemistry, 2005, 66: 837.
[576] Grandez M, Madinaveitia A, Gavín JA, et al. J Nat Prod, 2002, 65: 513.
[577] Cai L, Chen DL, Liu SY, et al. Chem Pharm Bull, 2006, 54: 779.
[578] Ulubelen A, Arfan M, Sönmez U, et al. Phytochemistry, 1998, 48: 385.
[579] Xie GB, Chen QH, Chen DL, et al. Heterocycles, 2003, 60: 631.
[580] Xie GB, Wang FP. Chem J Chin Univ, 2004, 25: 482.
[581] Yan H, Chen DL, Jian XX, et al. Helv Chim Acta, 2007, 90: 1133.
[582] Csupor D, Forgo P, Csedö K, et al. Helv Chim Acta, 2006, 89: 2981.
[583] Meriçli AH, Pirildar S, Süzgeç S, et al. Helv Chim Acta, 2006, 89: 210.
[584] Hou LH, Chen DL, Jian XX, et al. Chem Pharm Bull, 2007, 55: 1090.
[585] Ulubelen A, Meriçli A, Meriçli F, et al. Helv Chim Acta, 2000, 53: 2279.
[586] Wei XY, Xie HH, Liu MF, et al. Heterocycles, 2000, 53: 2027.
[587] Ji H, Wang FP. J Asian Nat Prod Res, 2006, 8: 619.
[588] Gao LM, Wei XM, Yang L. Chin Chem Lett, 2005, 16: 475.
[589] Zhang F, Peng SL, Luo F, et al. Chin Chem Lett, 2005, 16: 1043.
[590] Ishimi K, Makino M, Asada Y, et al. J Nat Med, 2006, 60: 255.
[591] Ji H, Chen DL, Wang FP. Heterocycles, 2004, 63: 2363.
[592] Peng CS, Wang FP, Jian XX. Chin Chem Lett, 2002, 13: 233.
[593] Yang XD, Yang S, Yang J, et al. Helv Chim Acta, 2008, 91: 569.
[594] Fuente G, Reina M, Valencia E. Heterocycles, 1989, 29: 1577.
[595] Takayama H, Yokota M, Aimi N, et al. J Nat Prod, 1990, 53: 936.
[596] Hang GY, Cai P, Wang JZ, et al. J Nat Prod, 1988, 51: 364.
[597] Lao AN, Wang Y, Wang HC. Heterocycles, 1996, 43: 1267.
[598] Meriçli AH, Meriçli F, Seyhan V, et al. Heterocycles, 1997, 45: 1955.
[599] Ulubelen A, Desai HK, Srivastava SK, et al. J Nat Prod, 1996, 59: 360.
[600] Bai Y, Desai HK, Pelletier SW. J Nat Prod, 1995, 58: 929.
[601] Liu HM, Katz A. J Nat Prod, 1996, 59: 135.
[602] Bai YL, Sun F, Benn M, et al. Phytochemistry, 1994, 37: 1717.
[603] Fuente GDL, Ruiz-Mesía L. Phytochemistry, 1995, 39: 1459.
[604] Fuente GDL, Meriçli AH, Ruiz-Mesía L, et al. Phytochemistry, 1995, 39: 1467.
[605] Hanuman JB, Katz A. J Nat Prod, 1993, 56: 801.
[606] Zhu DY, Lin LZ, Cordell GA. Phytochemistry, 1993, 32: 767.
[607] Chen ZG, Lao AN, Wang HC, et al. Heterocycles, 1989, 29: 997.
[608] Hohmann J, Forgo P, Haidú Z, et al. J Nat Prod, 2002, 65: 1069.
[609] Alva A, Grandez M, Madinaveitia A, et al. Chem Pharm Bull, 2004, 52: 530.
[610] He YQ, Ma ZY, Yang Q, et al. Acta Pharm Sin, 2008, 43: 934.
[611] Zhou XL, Chen QH, Wang FP. Heterocycles, 2004, 63: 123.
[612] Batbayar N, Enkhzaya S, Tunsag J, et al. Phytochemistry, 2003, 62: 543.
[613] Shen XL, Zhou XL, Chen QH, et al. Chem Pharm Bull, 2002, 50: 1265.
[614] Zhou XL, Chen QH, Wang FP. Chem Pharm Bull, 2004, 52: 381.
[615] Gardner DR, Manners GD, Panter KE, et al. J Nat Prod, 2000, 63: 1127.
[616] Csupor D, Forgo P, Máthé I, et al. Helv Chem Acta, 2004, 87: 2125.
[617] Ulubelen A, Desai HK, Hart BP, et al. J Nat Prod, 1996, 59: 907.
[618] Wu G, Jiang SH, Zhu DY. Phytochemistry, 1996, 42: 1253.
[619] Park JC, Desai HK, Pelletier SW. J Nat Prod, 1995, 58: 291.
[620] Zhang SM, Ou QY. Phytochemistry, 1998, 48: 191.
[621] ang FP, Li ZB, Che CT. J Nat Prod, 1998, 61: 1555.
[622] Xiong J, Tan NH, Ji CJ, et al. Tetrahedron Lett, 2008, 49: 4851.
[623] He HP, Shen YM, Zhang JX, et al. J Nat Prod, 2001, 64: 379.

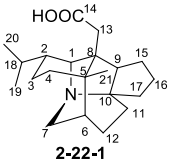
[624] Meriçli F, Meriçli AH, Ulubelen A, et al. J Nat Prod, 2001, 64: 787.
[625] Zuo GY, He HP, Hong X, et al. Heterocycles, 2000, 55: 487.
[626] Wang BG, Li L, Yang XS, et al. Heterocycles, 2000, 53: 1343.
[627] Wu TS, Hwang CC, Kuo PC, et al. Heterocycles, 2002, 57: 1495.
[628] Shen YM, He HP, Zhang YS, et al. Chin Chem Lett, 2000, 11: 789.
[629] Zuo GY, He HP, Hong X, et al. Chin Chem Lett, 2001, 12: 147.
[630] Wang BG, Hong X, Zuo GY, et al. J Asian Nat Prod Res, 2000, 2: 271.
[631] Díaz JG, Ruíz JG, Fuente GDL. J Nat Prod, 2000, 63: 1136.
[632] Shrestha PM, Katz A. J Nat Prod, 2004, 67: 1574.
[633] Ulubelen A, Arfan M, Sönmez U, et al. Phytochemistry, 1998, 47: 1141.
[634] Zhang F, Peng SL, Liao X, et al. Planta Med, 2005, 71: 1073.
[635] Feng F, Liu JH, Zhao SX. Phytochemistry, 1998, 49: 2557.
[636] Meriçli AH, Meriçli F, Doğru E, et al. Phytochemistry, 1999, 51: 337.
[637] Díaz JG, Ruiz JG, Herz W. Phytochemistry, 2004, 65: 2123.
[638] Peng CS, Jian XX, Wang FP, et al. Chin Chem Lett, 2000, 11: 411.
[639] Li M, Du XB, Shen YB, et al. Chin Chem Lett, 1999, 10: 827.
[640] Meriçli AH, Meriçli F, Desai HK, et al. Heterocycles, 2000, 53: 1987.
[641] Cao JX, Li LB, Ren J, et al. Helv Chim Acta, 2008, 91: 1954.
[642] Ulubelen A, Desai HK, Joshi BS, et al. J Nat Prod, 1995, 58: 1555.
[643] Yang CH, Zhang HJ, Liu JH. Chin Tradit Herb Drugs, 2004, 35: 1328.
[644] Jiang K, Yang CH, Liu JH, et al. Acta Pharm Sin, 2006, 41: 128.
[645] Reina M, Mancha R, Gonzalez-Coloma A, et al. Nat Prod Res, 2007, 21: 1048.
[646] Tang FQ, Yang CH, Liu JH, et al. Acta Pharm Sin, 2005, 40: 640.
[647] Yang CH, Wang XC, Tang QF, et al. Helv Chim Acta, 2008, 91: 759.
[648] Shim SH, Kim JS, Kang SS, et al. J Asian Nat Prod Res, 2006, 8: 451.
[649] Wangchuk P, Bremner JB, Samosorn S. J Nat Prod, 2007, 70: 1808.
[650] Ruiz-Mesía L, Madinaveitia A, Reina M, et al. J Nat Prod, 2002, 65: 496.
[651] Ulubelen A, Meriçli AH, Meriçli F, et al. Phytochemistry, 1996, 41: 957.
[652] Almanza G, Bastida J, Codina C, et al. Phytochemistry, 1997, 44: 739.
[653] Venkateswarlu V, Srivastava SK, Joshi BS, et al. J Nat Prod, 1995, 58: 1527.
[654] Reina M, Gavín JA, Madinaveitia A, et al. J Nat Prod, 1996, 59: 145.
[655] Li L, Zhao JF, Wang YB, et al. Helv Chim Acta, 2004, 87: 866.
[656] Wang YB, Huang R, Zhang HB, et al. Helv Chim Acta, 2005, 88: 1081.
[657] Zhou XL, Chen DL, Chen QH, et al. J Nat Prod, 2005, 68: 1076.
[658] Manners GD, Wong RY, Benson M, et al. Phytochemistry, 1996, 42: 875.
[659] Peng CS, Jian XX, Wang FP. J Asian Nat Prod Res, 2001, 3: 49.
[660] Gao LM, Wei XM, Jin XL, et al. Heterocycles, 2004, 63: 1181.
[661] Wang FP, Peng CS, Yu KB. Tetrahedron, 2000, 56: 7443.
[662] Nishimura K, Hitotsuyanagi Y, Sugeta N, et al. J Nat Prod, 2007, 70: 758.
[663] Zhang YM, Tan NH, Lu Y, et al. Org Lett, 2007, 9: 4579.
[664] Uddin MJ, Kokubo S, Suenaga K, et al. Heterocycles, 2000, 54: 1039.
[665] Uddin MJ, Kokubo S, Ueda K, et al. J Nat Prod, 2001, 64: 1169.

2.22 Triterpenoid alkaloids

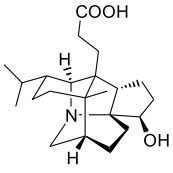
2.22.1 Daphniphylline-type triterpenoid alkaloids

Table 2-22-1: Cos, MFs, and TSs of daphniphylline-type triterpenoid alkaloids **2-22-1**~**2-22-13**.

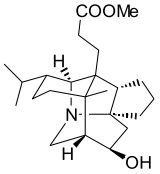
No.	Compounds	MFs	Test solvents	References
2-22-1	daphnezomine O	$C_{21}H_{33}NO_2$	CD_3OD	[666]
2-22-2	17-hydroxyhomodaphniphyllic acid	$C_{22}H_{35}NO_3$	C_5D_5N	[667]
2-22-3	caldaphnidine Q	$C_{23}H_{37}NO_3$	CD_3OD	[668]
2-22-4	calyciphylline L	$C_{23}H_{35}NO_2$	CD_3OD	[669]
2-22-5	yunnandaphninine H	$C_{30}H_{49}NO_3$	$CDCl_3$	[670]
2-22-6	daphnilongeranin D	$C_{30}H_{47}NO_4$	CD_3OD	[671]
2-22-7	daphnioldhanine H	$C_{32}H_{49}NO_6$	$CDCl_3$	[672]
2-22-8	calyciphylline P	$C_{30}H_{49}NO_4$	$CDCl_3$	[673]
2-22-9	daphnezomine E	$C_{32}H_{49}NO_6$	$CDCl_3$	[674]
2-22-10	daphmacropodine	$C_{32}H_{51}NO_4$	$CDCl_3$	[675]
2-22-11	calyciphylline M	$C_{32}H_{51}NO_5$	$CDCl_3$	[669]
2-22-12	yunnandaphninine F	$C_{30}H_{47}NO_4$	$CDCl_3$	[670]
2-22-13	yunnandaphninine G	$C_{30}H_{47}NO_3$	$CDCl_3$	[670]



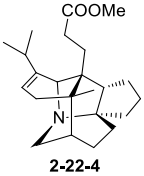
2-22-1



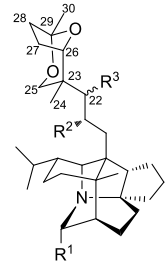
2-22-2



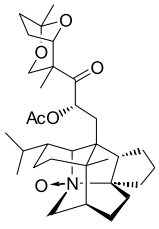
2-22-3



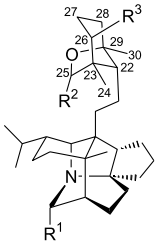
2-22-4



	R^1	R^2	R^3
2-22-5	H	H	OH
2-22-6	H	OH	=O
2-22-7	OH	OAc	=O
2-22-8	OH	H	OH



2-22-9



	R^1	R^2	R^3
2-22-10	H	OH	OAc
2-22-11	OH	OH	OAc
2-22-12	OH	=O	OH
2-22-13	H	=O	OH

Table 2-22-2: ^{1}H NMR spectroscopic data of daphniphylline-type triterpenoid alkaloids **2-22-1**~**2-22-4**.

H	2-22-1	2-22-2	2-22-3	2-22-4
1	4.08 br s	3.50 d(3.5)	2.94 d(4.4)	3.91 br s
2	2.05 m	1.46 m	1.47 m	
3	1.76 m, 2.06 m	1.68 m, 1.41 m	1.89 m, 1.61 m	6.31 m
4	1.60 m, 2.05 m	1.93 m, 1.34 m	1.98 m, 1.48 m	2.48 m, 2.18 m
6	1.81 m	1.50 m	1.55 m	1.76 m
7	3.14 d(13.5)	3.52 br d(13.6)	3.27 m	3.58 br d(12.6)
	3.71 d(13.5)	3.40 br d(13.6)	2.75 br d(14.5)	3.14 br d(12.6)
9	2.82 dd(7.4, 10.6)	2.48 dd(10.4, 7.7)	2.65 m	2.70 m
11	1.50 m, 1.86 m	2.28 m, 1.60 m	4.01 t-like(8.0)	2.07 m, 1.83 m
12	1.78 m, 2.02 m	1.70 m, 1.59 m	2.06 m, 1.60 m	2.07 m, 1.83 m
13	2.26 m	2.32 ddd(15.0, 10.0, 6.5)	2.08 m	1.82 m
	2.43 m	1.68 m	1.36 m	1.54 m
14		2.75 ddd(16.8, 10.5, 6.5)	2.56 m	2.63 m
		2.59 ddd(16.8, 10.0, 4.8)	2.51 m	2.52 m
15	1.82 m, 2.16 m	1.68 m, 1.52 m	1.82 m, 1.47 m	2.01 m, 1.75 m
16	1.69 m, 1.99 m	1.97 m, 1.63 m	1.69 m	2.03 m, 1.62 m
17	1.53 m, 2.10 m	4.60 dd(11.4, 6.0)	2.16 m, 1.38 m	2.12 m, 1.92 m
18	1.57 m	1.84 m	1.60 m	2.32 m
19	1.02 d(6.5)	1.20 d(6.4)	1.01 d(6.3)	1.16 d(6.4)
20	1.08 d(6.5)	0.75 d(6.5)	0.94 d(6.3)	1.14 d(6.4)
21	1.03 s	0.95 s	0.95 s	1.17 s
OMe			3.66 s	3.69 s

Table 2-22-3: ^{1}H NMR spectroscopic data of daphniphylline-type triterpenoid alkaloids **2-22-5**~**2-22-9**.

H	2-22-5	2-22-6	2-22-7	2-22-8	2-22-9
1	2.74~2.79 m	2.87 d(4.8)	3.62 d(4.4)	3.44 m	3.69 br s
2	1.37~1.40 m	1.54 m	1.75 m	1.53 m	1.76 m
3	1.52~1.57 m	1.90 m	1.54 m	1.93 m	1.88 m
	1.77~1.82 m	1.54 m	2.01 m	1.54 m	2.02 m
4	1.38~1.42 m	1.72 m	2.24 m	1.96 m	1.64 m
	1.86~1.90 m	1.35 m	2.31 m	1.58 m	1.99 m
6	1.84~1.86 m	1.33 m	1.93 m	1.83 m	1.92 m
7	2.74~2.82 m	3.27 br d(14.2)	5.72 br s	5.69 s	4.15 br m
	3.22~3.30 m	2.68 dd(15.7, 3.7)			
9	2.12~2.18 m	2.39 dd(10.3, 7.3)	2.54 t(9.2)	2.38 m	2.49 m
11	1.56~1.59 m	1.80 m	1.60 m	2.73 m	1.79 m
	1.82~1.86 m	1.50 m	2.10 m	1.53 m	1.96 m
12	1.55~1.59 m	1.92 m	2.32 m	2.03 m	1.61 m
	1.83~1.86 m	1.59 m		1.80 m	

Table 2-22-3 (continued)

H	2-22-5	2-22-6	2-22-7	2-22-8	2-22-9
13	1.42~1.46 m	2.71 br d(14.0)	1.54 m	1.93 m	1.55 m
	1.84~1.88 m	1.07 dd(15.8, 7.2)	2.66 dd(9.2, 2.8)	1.54 m	2.64 dd(3.2, 15.7)
14	1.24~1.27 m	4.85 dd(7.2, 3.7)	5.66 dd(10.4, 2.8)	1.68 m	5.61 dd(3.2, 12.5)
	1.81~1.85 m			1.22 m	
15	1.68~1.71 m	2.04 m	1.60 m	2.03 m	1.53 m
	1.83~1.86 m	1.40 m	2.60 m	1.42 m	2.36 m
16	1.21~1.26 m	1.70 m	1.46 m	1.93 m	1.38 m
	1.68~1.72 m	1.39 m	1.93 m	1.54 m	1.89 m
17	1.30~1.33 m	2.01 m	1.61 m	2.12 m	1.82 m
	1.79~1.83 m	1.39 dd(14.4, 8.4)	2.03 m	1.84 m	2.48 m
18	1.72~1.77 m	1.65 m	1.46 m	1.68 m	2.27 m
19	0.91 d(6.5)	0.98 d(6.4)	0.96 d(6.4)	0.92 d(6.6)	0.90 d(6.4)
20	1.03 d(6.5)	0.99 d(6.3)	1.13 d(6.0)	1.04 d(6.6)	1.09 d(6.2)
21	0.88 s	0.93 s	1.07 s	1.01 s	1.04 s
22	3.33 dd(2.0, 9.0)			3.37 d(10.8)	
24	1.11 s	0.90 s	0.91 s	1.08 s	0.89 s
25	3.40 dd(16.0, 11.0)	4.53 dd(12.7, 1.8)	4.49 br d(13.6)	3.39 m	3.71 d(13.3)
		3.70 d(12.7)	3.75 br d(13.6)		4.46 dd(1.7, 13.3)
26	4.16 d(7.0)	4.78 br d(5.9)	4.57 d(6.4)	4.10 d(6.6)	4.53 d(6.8)
27	1.94~1.98 m	2.00 m	1.98 m	1.93 m	1.94 m
	2.03~2.07 m		2.00 m	1.87 m	2.02 m
28	1.73~1.77 m	2.10 m	1.93 m	2.06 m	1.89 m
	1.95~1.98 m	1.83 m	2.08 m	1.78 m	2.08 m
30	1.46 s	1.35 s	1.47 s	1.47 s	1.45 s
OAc			2.12 s		2.09 s

Table 2-22-4: ^{1}H NMR spectroscopic data of daphniphylline-type triterpenoid alkaloids **2-22-10**~**2-22-13**.

H	2-22-10	2-22-11	2-22-12	2-22-13
1	3.33 br s	3.44 m	2.77 d(3.5)	3.35 d(4.0)
2	1.44~1.48 m	1.44 m	1.15~1.21 m	1.44~1.49 m
3	1.90~1.92 m	1.87 m	1.31~1.35 m	1.56~1.60 m
	1.27~1.29 m	1.44 m	1.74~1.78 m	1.98~2.03 m
4	1.96~2.00 m	1.98 m	1.30~1.35 m	1.53~1.60 m
	1.61~1.62 m	1.65 m	1.76~1.83 m	2.00~2.03 m
6	1.71~1.76 m	1.88 m	1.44~1.47 m	1.72~1.75 m
7	3.54 br d(13.7)	5.74 br d(3.4)	4.97 br s	3.40 br d(14.5)
	3.34 br d(13.5)			3.59 dd(5.0, 14.5)
9	1.61~1.66 m	2.48 m	2.07~2.13 m	2.48 t(7.0)
11	1.86~1.87 m	2.76 m	1.28~1.33 m	1.60~1.65 m
	1.74~1.79 m	1.64 m	2.16~2.24 m	2.19~2.26 m

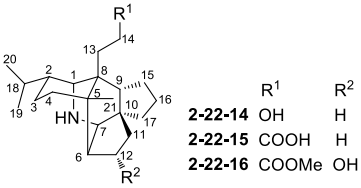
Table 2-22-4 (continued)

H	2-22-10	2-22-11	2-22-12	2-22-13
12	2.18 dd(15.7, 9.4)	2.04 m	1.59~1.67 m	1.77~1.80 m
	1.63~1.69 m	1.80 m		1.90~1.94 m
13	2.08 dd(14.0, 9.1)	2.10 m	0.67~0.75 m	0.90~0.94 m
	1.35~1.39 m	1.39 m	1.78~1.82 m	2.14~2.17 m
14	1.97~1.98 m	1.94 m	1.35~1.40 m	1.47~1.51 m
	1.00~1.02 m	1.00 m	2.25~2.34 m	2.42~2.46 m
15	1.90~1.95 m	1.96 m	1.24~1.28 m	1.46~1.49 m
	1.42~1.44 m	1.52 m	1.58~1.63 m	1.81~1.86 m
16	1.90~1.94 m	1.90 m	1.25~1.28 m	1.05~1.10 m
	1.42~1.45 m	1.52 m	1.61~1.65 m	1.61~1.65 m
17	2.27 dd(14.0, 6.3)	2.16 m	1.25~1.29 m	1.84~1.90 m
	1.93~1.96 m	1.90 m	1.65~1.70 m	2.21~2.25 m
18	1.91~1.96 m	1.70 m	1.44~1.49 m	1.23~1.28 m
19	0.93 d(6.4)	0.94 d(6.3)	0.90 d(6.0)	1.06 d(6.5)
20	1.10 d(6.4)	1.00 d(6.3)	0.79 d(6.0)	0.95 d(6.5)
21	1.08 s	1.17 s	0.89 s	1.08 s
22	2.44 dd(10.1, 7.2)	1.59 m	1.51~1.55 m	1.63~1.66 m
24	1.02 s	1.05 s	1.18 s	1.34 s
25	4.83 s	4.88 br s		
26	4.75 d(5.2)	4.79 br d(5.2)	3.56 d(4.0)	3.80 d(5.0)
27	1.90~1.95 m	1.95 m	1.46~1.51 m	1.47~1.52 m
	1.60~1.65 m	1.62 m	1.74~1.78 m	1.82~1.86 m
28	1.44~1.48 m	1.53 m	1.71~1.67 m	1.81~1.86 m
	1.30~1.36 m	1.35 m	1.78~1.74 m	1.92~1.97 m
30	1.29 s	1.33 s	1.32 s	1.43 s
OAc	2.05 s	2.09 s		

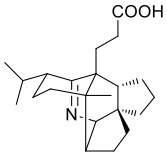
2.22.2 Secodaphniphylline-type triterpenoid alkaloids

Table 2-22-5: Cos, MFs, and TSs of secodaphniphylline-type triterpenoid alkaloids **2-22-14**~**2-22-21**.

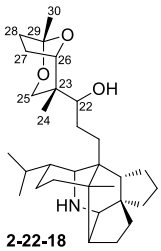
No.	Compounds	MFs	Test solvents	References
2-22-14	caldaphnidine D	$C_{21}H_{35}NO$	CD_3OD	[676]
2-22-15	daphnezomine M	$C_{22}H_{35}NO_2$	CD_3OD	[666]
2-22-16	calyciphylline O	$C_{23}H_{37}NO_3$	$CDCl_3$	[673]
2-22-17	daphnezomine N	$C_{22}H_{33}NO_2$	CD_3OD	[666]
2-22-18	caldaphnidine E	$C_{30}H_{49}NO_3$	CD_3OD	[676]
2-22-19	daphnezomine C	$C_{30}H_{45}NO_4$	$CDCl_3$	[674]
2-22-20	daphnilongeridine	$C_{32}H_{51}NO_4$	$CDCl_3$	[675]
2-22-21	daphnezomine D	$C_{32}H_{49}NO_5$	$CDCl_3$	[674]



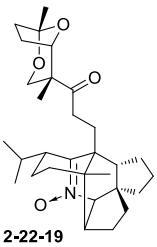
	R^1	R^2
2-22-14	OH	H
2-22-15	COOH	H
2-22-16	COOMe	OH



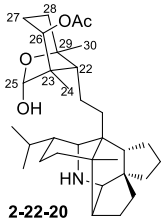
2-22-17



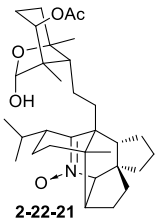
2-22-18



2-22-19



2-22-20



2-22-21

Table 2-22-6: ^{1}H NMR spectroscopic data of secodaphniphylline-type triterpenoid alkaloids **2-22-14**~**2-22-17**.

H	2-22-14	2-22-15	2-22-16	2-22-17
1	2.98 br s	3.46 s	2.97 br s	
2	1.06 dd(17.1, 8.3)	1.42 m	0.91 m	3.26 dt(5.2, 10.6)
3	1.54 m	1.41 m, 1.83 m	1.49 m	1.49 m, 2.57 m
4	1.65 m	1.37 m	1.58 m	1.25 br d(5.5)
	1.18 m	1.77 m	1.29 m	1.97 m
6	1.92 t(5.0)	2.18 t(4.5)	1.81 d(4.6)	2.15 m
7	2.55 d(4.3)	3.15 d(4.5)	3.03 d(4.6)	4.33 d(4.5)
9	1.73 m	2.03 t(16.0)	1.67 m	1.57 m
11	1.68 m	1.75 m	2.04 dd(14.6, 7.2)	1.83 m
	1.52 m	1.86 m	1.58 m	2.05 m
12	1.46 m, 1.67 m	1.63 m, 1.84 m	4.20 d(6.9)	2.12 m
13	1.54 m	1.67 m, 1.78 m	1.65 m, 1.58 m	2.38 m, 2.48 m
14	3.68 m, 3.53 m	2.23 m, 2.43 m	2.37 m, 2.20 m	2.49 m, 2.68 m
15	1.78 m, 1.60 m	1.29 m, 1.58 m	1.70 m, 1.26 m	0.99 m, 2.11 m
16	1.79 m, 1.48 m	1.57 m, 1.82 m	1.75 m, 1.40 m	1.52 m, 1.59 m
17	1.84 m	1.74 m	1.75 m	1.74 ddd(6.6, 10.1, 13.1)
		1.81 m	1.58 m	1.91 m
18	1.50 m	1.47 m	1.52 m	2.20 m
19	0.92 d(6.4)	1.05 d(6.0)	0.91 d(6.3)	1.06 d(6.5)
20	0.90 d(6.4)	1.01 d(6.0)	0.91 d(6.9)	1.01 d(6.5)
21	0.80 s	0.94 s	0.82 s	1.21 s
OMe			3.64 s	

Table 2-22-7: ^{1}H NMR spectroscopic data of secodaphniphylline-type triterpenoid alkaloids **2-22-18**~**2-22-21**.

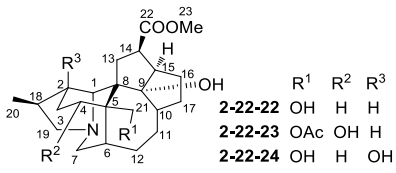
H	2-22-18	2-22-19	2-22-20	2-22-21
1	3.09 br s		3.40 br s	
2	1.13 m	2.26 dt(3.4, 11.5)	0.98~1.01 m	1.76 m
3	1.51 m	1.65 m	1.85~1.93 m	1.69 m
		2.01 m	1.61~1.66 m	2.05 m
4	1.68 m	1.09 dd(4.7, 14.9)	1.59~1.63 m	1.12 dd(4.4, 14.5)
	1.16 m	1.87 m	1.24~1.29 m	1.90 m
6	1.90 m	2.19 br q(3.0)	2.07~2.09 m	2.19 br s
7	2.52 d(4.5)	4.02 d(4.8)	2.88 br s	4.03 d(4.7)
9	1.71 m	1.75 t(8.0)	1.74~1.79 m	2.26 td(11.7)
11	1.69 m	1.60 m	2.08~2.13 m	1.59 m
	1.52 m	1.85 m	1.55~1.63 m	1.76 m
12	1.54 m	1.67 m	1.70~1.74 m	1.68 m
			1.31~1.34 m	1.74 m
13	1.68 m	1.88 m	1.63~1.70 m	
		2.34 ddd(4.1, 11.4, 15.1)	1.49~1.65 m	
14	1.26 m	2.86 m	1.44~1.53 m	
	1.04 m	3.14 ddd(5.1, 11.2, 18.5)	1.31~1.44 m	
15	1.76 m	1.33 m	1.80~1.87 m	1.39 m
	1.62 m	1.91 m	1.73~1.82 m	2.09 m
16	1.80 m	1.38 m	1.39~1.47 m	
	1.44 m	1.64 m	1.27~1.29 m	
17	1.82 m	1.78 m	1.72~1.78 m	1.85 m
	1.51 m	1.83 m	1.53~1.59 m	1.90 m
18	1.49 m	2.84 m	1.73~1.78 m	2.87 m
19	0.89 d(6.8)	0.83 d(6.8)	0.94 d(6.6)	0.86 d(6.6)
20	0.90 d(6.8)	1.05 d(6.6)	1.03 d(6.4)	1.05 d(6.3)
21	0.69 s	0.95 s	0.81 s	0.94 s
22	3.41 d(9.8)		1.56~1.63 m	1.73 m
24	1.03 s	0.82 s	1.01 s	1.06 s
25	3.33 d(11.3)	3.55 d(12.2)	4.85 s	4.88 s
	3.27 d(11.3)	4.29 dd(1.7, 12.2)		
26	4.10 d(6.3)	4.69 d(6.1)	4.76 d(5.3)	4.80 d(5.1)
27	1.95 m	1.95 m	1.89~1.94 m	1.74 m
	2.13 m	2.10 m	1.59~1.64 m	1.95 m
28	1.98 m	1.93 m	1.51~1.60 m	1.70 m
	1.88 m	2.08 m	1.31~1.38 m	2.05 m
30	1.40 s	1.43 s	1.32 s	1.40 s
OAc			2.08 s	2.04 s

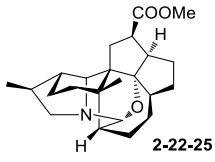
2.22.3 Yuzurimine-type triterpenoid alkaloids

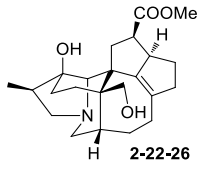
Table 2-22-8: Cos, MFs, and TSs of Yuzurimine-type triterpenoid alkaloids **2-22-22**~**2-22-46**.

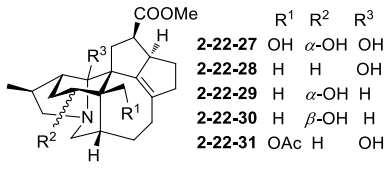
No.	Compounds	MFs	Test solvents	References
2-22-22	daphnioldhanin A	$C_{23}H_{35}NO_4$	$CDCl_3$-CD_3OD(10:1)	[677]
2-22-23	daphnioldhanin B	$C_{25}H_{37}NO_6$	$CDCl_3$	[677]
2-22-24	daphlongamine D	$C_{23}H_{35}NO_5$	$CDCl_3$-CD_3OD(4:1)	[678]
2-22-25	daphcalycine	$C_{23}H_{33}NO_3$	C_5D_5N	[679]
			$CDCl_3$	[679]
2-22-26	daphlongamine C	$C_{23}H_{33}NO_4$	$CDCl_3$-CD_3OD(4:1)	[678]
2-22-27	daphnezomine K	$C_{23}H_{33}NO_5$	$CDCl_3$-CD_3OD(9:1)	[680]
2-22-28	yunnandaphnine A	$C_{23}H_{33}NO_3$	$CDCl_3$	[681]
2-22-29	yunnandaphnine B	$C_{23}H_{33}NO_3$	$CDCl_3$	[681]
2-22-30	yunnandaphnine C	$C_{23}H_{33}NO_3$	$CDCl_3$	[681]
2-22-31	daphniglaucin J	$C_{25}H_{35}NO_5$	CD_3OD	[682]
2-22-32	daphniglaucin K	$C_{25}H_{35}NO_5$	CD_3OD	[682]
2-22-33	calyciphylline E	$C_{23}H_{31}NO_4$	$CDCl_3$	[683]
2-22-34	macropodumine F	$C_{23}H_{31}NO_5$	$CDCl_3$	[684]
2-22-35	caldaphnidine I	$C_{23}H_{31}NO_4$	CD_3OD	[668]
2-22-36	macropodumine H	$C_{26}H_{35}NO_6$	$CDCl_3$	[684]
2-22-37	17-oxoyuzurimine	$C_{27}H_{35}NO_8$	$CDCl_3$	[684]
2-22-38	pordamacrine A	$C_{23}H_{31}NO_5$	CD_3OD	[685]
2-22-39	pordamacrine B	$C_{23}H_{31}NO_4$	CD_3OD	[685]
2-22-40	caldaphnidine G	$C_{23}H_{31}NO_4$	$CDCl_3$	[668]
2-22-41	caldaphnidine H	$C_{23}H_{31}NO_4$	CD_3OD	[668]
2-22-42	caldaphnidine J①	$C_{23}H_{31}NO_2$	CD_3OD	[668]
2-22-43	caldaphnidine A	$C_{23}H_{29}NO_3$	CD_3OD	[676]
2-22-44	daphtenidine D	$C_{27}H_{35}NO_7$	CD_3OD	[686]
2-22-45	macropodumine G	$C_{23}H_{29}NO_5$	$CDCl_3$	[684]
2-22-46	daphnezomine J	$C_{25}H_{34}NO_5$	$CDCl_3$	[680]

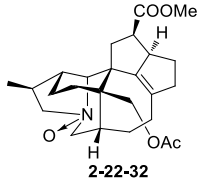
① The structure in the literature was uncorrect. There is not hydroxyl on C-1 judged by the NMR and MS spectra.

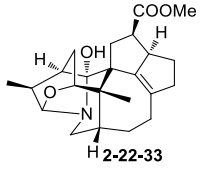


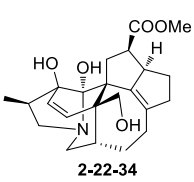




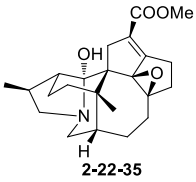




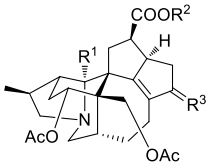




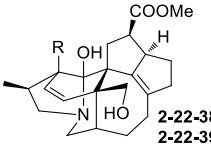
2-22-34



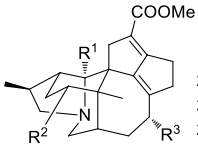
2-22-35



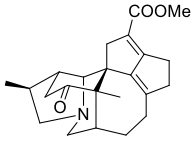
	R^1	R^2	R^3
2-22-36	H	H	H_2
2-22-37	OH	Me	O



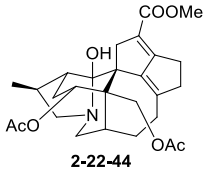
2-22-38 R = OH
2-22-39 R = H



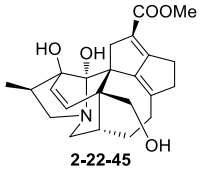
	R^1	R^2	R^3
2-22-40	OH	OH	H
2-22-41	OH	H	OH
2-22-42	H	H	H



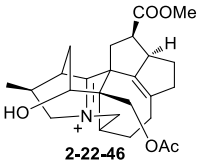
2-22-43



2-22-44



2-22-45



2-22-46

Table 2-22-9: ^{1}H NMR spectroscopic data of yuzurimine-type triterpenoid alkaloids **2-22-22**~**2-22-24**.

H	2-22-22	2-22-23	2-22-24
1	4.07 m	3.96 m	3.07 m
2	2.26 m	2.35 dd(9.6, 14.4)	
3	1.71 m, 1.39 m	1.73 m	1.50 m
4	2.04 m, 1.62 m	4.22 br s	α 1.94 m, β 1.69 m
6	2.36 m	2.45 m	2.26 t(5.0)
7	3.85 m, 3.12 m	3.11 m, 4.08 m	α 3.25 m β 2.89 t(11.5)
10	2.23 m	2.21 m	2.12 m
11	1.86 m, 1.37 m	1.27 m, 1.47 m	α 1.36 m, β 1.90 m
12	1.98 m, 1.44 m	1.47 m	α 1.27 m, β 1.80 m
13	2.35 m, 1.92 m	2.05 m, 2.32 m	α 2.55 dd(15.0, 7.5) β 2.11 m

Table 2-22-9 (continued)

H	2-22-22	2-22-23	2-22-24
14	3.08 m	3.10 m	3.09 m
15	2.77 dd(19.5, 9.2)	2.83 dd(9.2, 18.8)	2.61 dd(19.0, 9.0)
16	1.44 m, 1.76 m	1.31 m, 1.81 m	α 1.60 m, β 1.13 m
17	1.60 m, 1.48 m	1.65 m	α 1.36 m, β 1.50 m
18	2.52 m	1.67 m	1.98 m
19	4.10 m, 2.63 m	2.68 br s, 4.22 br s	α 3.47 t(10.0) β 2.14 d(10.0)
20	1.01 d(6.7)	1.05 d(6.0)	0.93 d(7)
21	4.02 m, 3.84 m	4.28 d(11.2), 4.66 d(11.2)	4.13 d(10.9), 3.67 d(10.9)
23	3.60 s	3.68 s	3.75 s
OAc		2.09 s	

Table 2-22-10: ^{1}H NMR spectroscopic data of yuzurimine-type triterpenoid alkaloids **2-22-25**~**2-22-27**.

H	2-22-25 (C_5D_5N)	2-22-25 ($CDCl_3$)	2-22-26	2-22-27
1	4.15 d(7.4)	3.68 d	2.45 m	
2	2.23 m	2.11 m		2.37 m
3	1.53 m, 1.45 m	1.57 m, 1.52 m	α 1.72 m, β 1.42 m	1.60 m, 2.05 m
4	1.39 m, 1.35 m	1.45 m, 1.42 m	α 1.54 m, β 1.67 m	4.38 dd(7.3, 11.8)
6	1.98 dd(3.3, 7.1)	1.92 ddd	2.01 m	2.69 t(7.1)
7	4.93 d(3.3)	4.55 d	α 2.95 d(16.0) β 2.85 m	3.50 dd(8.9, 15.0) 3.61 d(15.0)
10	2.13 m	2.22 m		
11	1.56 m	1.65 m	α 1.88 m β 2.10 m	2.10 dd(4.3, 17.7) 2.50 m
12	1.56 m, 1.83 m	1.63 m, 1.92 m	α 1.24 m, β 1.94 m	1.46 m, 1.91 m
13	1.84 dd(7.1, 13.3)	1.75 dd	α 2.80 m	2.34 dd(9.8, 15.1)
	2.23 t(13.3)	2.11 t	β 2.24 dd(17.0, 3.5)	2.94 dd(2.1, 15.1)
14	3.49 ddd(7.1, 13.3, 17.6)	3.25 ddd	2.75 s	3.04 m
15	3.09 ddd(2.4, 8.9, 17.6)	2.83 ddd	3.32 m	3.77 m
16	1.82 m, 0.97 m	1.88 m, 0.86 m	α 1.01 m, β 1.69 m	1.07 m, 1.93 m
17	1.30 m, 1.64 m	1.30 m, 1.70 m	α 2.11 m, β 2.45 m	2.27 dd(8.2, 15.1), 2.53 m
18	2.71 m	2.52 m	1.87 m	3.15 m
19	3.40 t(6.7)	3.16 t	α 3.18 m	2.58 dd(6.4, 11.6)
	2.76 dd(6.7, 15.3)	2.66 dd	β 1.94 m	4.13 t(11.6)
20	0.90 d(6.6)	1.03 d	0.88 d(7.0)	1.11 d(7.3)
21	0.99 s	1.12 s	3.67 d(11.5) 3.62 d(11.5)	3.68 d(11.9)4.19 d(11.9)
23	3.68 s	3.62 s	3.45 s	3.64 s

Table 2-22-11: ^{1}H NMR spectroscopic data of yuzurimine-type triterpenoid alkaloids **2-22-28**~**2-22-31**.

H	2-22-28	2-22-29	2-22-30	2-22-31
1		2.97 m	3.11 m	
2	2.13 m	2.31 m	2.43 m	2.48 m
3	1.50 m	*α* 1.43 m	*α* 1.68 m	1.88 m
		β 1.92 m	*β* 1.82 dd(14.0, 4.0)	1.80 m
4	*α* 1.42 m	3.73 dd(11.5, 6.0)	3.60 m	2.02 m
	β 1.65 dt(13.5, 7.0)			1.77 m
6	1.90 m	2.52 m	1.95 m	2.44 m
7	*α* 3.19 dd(12.5, 9.0)	*α* 3.05 dd(12.0, 10.0)	*α* 2.95 m	3.66 m
	β 3.31 d(12.5)	*β* 3.47 br d(12.0)	*β* 3.57 m	3.80 m
11	*α* 2.01 m	*α* 2.12 dd(18.0, 5.5)	*α* 2.20 m	2.22 dd(4.9, 17.5)
	β 2.41 m	*β* 2.52 m	*β* 2.47 m	2.46 m
12	*α* 1.37 m, *β* 2.08 m	*α* 1.54 m, *β* 1.99 m	*α* 1.50 m, *β* 2.05 m	1.58 m, 1.97 m
13	*α* 2.42 dd(14.5, 10.0)	*α* 1.88 dd(14.0, 9.0)	*α* 2.05 m	2.36 dd(9.6, 15.3)
	β 2.62 dd(14.5, 4.5)	*β* 2.69 dd(14.0, 4.5)	*β* 2.88 dd(15.0, 6.5)	2.77 dd(3.2, 15.3)
14	3.04 td(10.5, 4.0)	2.92 m	2.94 m	3.01 dt(3.1, 10.2)
15	3.53 m	3.58 m	3.70 m	3.85 m
16	*α* 1.84 m, *β* 1.35 m	*α* 1.92 m, *β* 1.21 m	*α* 1.92 m, *β* 1.18 m	1.83 m, 1.29 m
17	*α* 2.67 m	*α* 2.83 m	*α* 2.78 m	2.42 m
	β 2.31 dd(15.0, 8.5)	*β* 2.27 m	*β* 2.23 dd(14.5, 8.0)	2.73 m
18	2.77 m	2.43 m	2.40 m	3.11 m
19	*α* 3.60 m	*α* 3.86 m	*α* 3.94 m	4.09 t(11.7)
	β 2.26 dd(11.5, 7.5)	*β* 2.37 m	*β* 2.43 m	2.88 dd(6.5, 12.2)
20	1.03 d(7.5)	1.08 d(6.5)	1.05 d(5.5)	1.20 d(7.3)
21	1.12 s	1.25 s	1.26 s	4.41 d(11.9)
				4.44 d(12.0)
OMe	3.63 s	3.64 s	3.64 s	3.68 s
OAc				2.06 s

Table 2-22-12: ^{1}H NMR spectroscopic data of yuzurimine-type triterpenoid alkaloids **2-22-32**~**2-22-36**.

H	2-22-32	2-22-33	2-22-34	2-22-35	2-22-36
1	3.93 d(4.3)				3.26 s
2	2.79 m	2.16 m		2.32 m	2.37~2.41 m
3	1.74 m	2.03 m	5.80 d(10.3)	1.88 m	*α* 1.38~1.43 m
		1.75 dd(13.7, 6.4)		1.74 m	*β* 2.02~2.05 m
4	1.75 m, 2.03 m	3.21 m	5.87 d(10.3)	1.98 m, 1.55 m	5.27 dd(11.5, 6.4)
6	2.52 m	2.49 m	1.82~1.85 m	2.13 m	2.68 t(7.9)
7	4.42 s	3.44 dd(14.3, 9.8)	3.11~3.15 m	3.73 m	3.62 d(13.8)
	4.44 m	3.02 dd(14.3, 8.9)	2.84~2.86 m	3.45 br d(14.7)	3.00 dd(13.8, 4.2)
11	2.23 m	1.97 m	2.43~2.47 m	2.30 m	1.94~1.97 m
	2.33 m	1.60 m	2.05~2.08 m	2.19 m	2.40~2.43 m

Table 2-22-12 (continued)

H	2-22-32	2-22-33	2-22-34	2-22-35	2-22-36
12	1.66 m	2.16 m	1.98~2.02 m	2.21 m	1.53~1.57 m
	2.01 m		1.49~1.53 m	1.44 m	1.83~1.87 m
13	2.19 m	2.49 m	3.04~3.06 m	3.26 m	1.87~1.91 m
	2.75 dd(3.4, 15.3)	2.28 dd(13.6, 8.6)	2.51 dd(14.0, 2.3)	3.14 m	2.98~3.03 m
14	3.00 dt(3.4, 6.2)	3.14 dt(11.4, 8.4)	3.00~3.04 m		2.75~2.79 m
15	3.60 m	3.71 m	3.66~3.69 m		3.40~3.45 m
16	1.34 m	1.84 m	*α* 1.86~1.90 m	2.30 m	*α* 1.80~1.85 m
	1.93 m	1.20 m	*β* 1.25~1.28 m	2.21 m	*β* 1.45~1.48 m
17	2.43 m	2.49 m	*α* 2.67~2.72 m	2.71 m	*α* 2.51~2.57 m
	2.50 m	2.20 m	*β* 2.30~2.33 m	2.08 m	*β* 2.16 dd(14.7, 8.1)
18	2.92 m	2.41 m	2.27~2.32 m	3.05 m	2.46~2.51 m
19	4.04 m	4.30 br s	3.44~3.46 m	4.05 dd(11.9, 10.7)	3.85 t(11.3)
	4.02 t(11.9)		2.08~2.12 m	2.80 dd(11.9, 6.5)	2.30 dd(12.4, 8.2)
20	1.09 d(6.9)	1.14 d(6.9)	1.16 d(7.5)	1.15 d(7.4)	1.00 d(6.6)
21	4.02 m	0.97 s	4.10 d(11.9)	1.02 s	4.35 d(11.5)
	4.46 m		3.76 d(11.9)		4.47 d(11.5)
23	3.70 s	3.63 s	3.65 s	3.75 s	
OAc	2.07 s				

Table 2-22-13: ^{1}H NMR spectroscopic data of yuzurimine-type triterpenoid alkaloids **2-22-37**~**2-22-40** and **2-22-46**.

H	2-22-37	2-22-38	2-22-39	2-22-40	2-22-46
2	2.28~2.32 m		2.49 m	2.38 m	2.46 br s
3	*α* 1.52~1.56 m	5.50 d(10.3)	5.61 dd(10.2, 2.7)	1.88 m	2.29 dd(2.7, 15.0)
	β 2.00~2.05 m			1.60 m	2.39 dd(4.0, 15.0)
4	5.35 dd(12.0, 7.5)	5.99 d(10.3)	5.18 dd(10.2, 1.8)	3.86 dd(11.4, 7.4)	4.29 m
6	2.68~2.72 m	2.13 m	2.09 m	2.34 m	2.84 m
7	3.40~3.44 m	2.86 dd(14.4, 8.9)	2.93 dd(13.0, 8.3)	3.21 m	2.73 m
	3.32~3.36 m	3.46 dd(14.4, 9.3)	3.28 dd(13.0, 5.5)		3.61 m
11	1.75~1.79 m	2.31 m	2.15 m	2.82 m	2.05 m
	1.63~1.66 m		2.45 m	2.07 m	2.11 m
12	2.60~2.65 m	1.47 m	1.51 m	1.83 m	1.74 m
	2.41~2.45 m	2.31 m	2.16 m	1.53 m	1.92 m
13	2.91~2.96 m	2.02 m	2.35 m	3.03 br s	2.25 dd(8.4, 14.5)
	2.72~2.76 m	2.96 dd(13.7, 8.3)	2.50 m		2.95 dd(5.0, 14.5)

Table 2-22-13 (continued)

H	2-22-37	2-22-38	2-22-39	2-22-40	2-22-46
14	3.18 t(9.0)	3.23 m	3.01 ddd(4.8, 10.4, 10.4)		2.81 m
15	3.46~3.49 m	3.52 m	3.47 m		3.43 m
16	α 2.49 d(5.1)	1.27 m	1.42 m	2.71 m	1.34 m
	β 2.49 d(5.1)	1.83 m	1.82 m	2.61 m	1.98 m
17		2.18 m, 2.49 m	2.32 m, 2.49 m	2.93 m, 2.88 m	2.42 m, 2.71 m
18	2.88~2.93 m	2.01 m	2.40 m	2.78 m	2.73 m
19	3.72~3.77 m	2.25 dd(10.6, 7.9)	2.40 m	3.59 dd(11.9, 10.6)	3.06 m
	2.31~2.37 m	3.03 t(7.2)	3.17 m	2.21 dd(11.9, 6.0)	3.09 m
20	1.08 d(7.2)	0.96 d(6.9)	1.10 d(6.8)	1.08 d(7.4)	1.19 d(6.9)
21	4.33 d(11.4)	3.68 d(10.7)	3.78 d(11.2)	0.99 s	4.30 d(12.0)
	3.98 d(11.4)	3.90 d(10.7)	3.92 d(11.2)		4.64 d(12.0)
OMe	3.60 s	3.61 s	3.64 s	3.68 s	3.67 s
OAc					2.13 s

Table 2-22-14: ^{1}H NMR spectroscopic data of yuzurimine-type triterpenoid alkaloids **2-22-41**~**2-22-45**.

H	2-22-41	2-22-42	2-22-43	2-22-44	2-22-45
1		2.96 m	3.09 d(6.2)		
2	2.36 m	2.44 m	2.82 m	2.39 m	
3	2.02 m, 1.64 m	1.79 m, 1.65 m	2.54 m	2.00 m, 1.74 m	5.95 d(10.3)
4	1.78 m, 1.51 m	1.69 m, 1.63 m		5.42 dd(11.2, 8.1)	5.88 d(10.3)
6	1.91 m	2.07 m	1.84 t(5.6)	2.60 m	2.00~2.03 m
7	3.45 dd(13.2, 6.2)	3.41 m	3.05 d(13.0)	3.40 d(13.1)	2.97~3.03 m
	3.10 m	3.34 m	2.86 m	3.26 m	2.96~2.99 m
11	4.02 t-like(3.2)	2.69 m	2.15 m	2.92 m	2.96~2.99 m
		2.16 m	2.98 m	2.09 d(16.8)	2.04~2.07 m
12	2.17 m	2.05 m	1.67 m	1.75 m	1.90~1.93 m
	1.83 m	1.54 m	2.08 m	1.61 m	1.68~1.71 m
13	3.08 br s	3.09 br d(15.6)	2.92 d(12.0)	3.37 m	3.70~3.74 m
		2.72 m	2.97 d(12.0)	3.08 d(16.8)	2.68~2.73 m
16	2.68 m	2.69 m	2.90 m	2.94 m	α 2.65~2.69 m
		2.60 m			β 2.71~2.75 m
17	3.16 m	2.89 m	2.62 m	2.72 m	α 2.90~2.95 m
	2.89 m	2.83 m	2.75 m	2.63 m	β 3.00~3.06 m
18	2.81 m	2.44 m	2.52 m	2.75 m	2.34~2.38 m
19	3.56 dd(11.6, 10.1)	3.49 m	3.21 br t(9.8)	3.58 dd(11.8, 10.0)	3.63~3.68 m
	2.29 dd(11.6, 5.1)	2.52 m	2.49 dd(9.8, 3.8)	2.30 dd(6.2, 11.8)	2.13~2.17 m
20	1.16 d(7.6)	1.15 d(7.4)	1.18 s	1.12 d(7.5)	1.25 d(6.0)

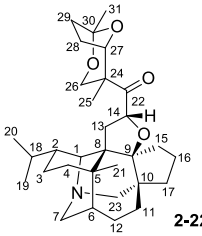
Table 2-22-14 (continued)

H	2-22-41	2-22-42	2-22-43	2-22-44	2-22-45
21	0.86 s	1.02 s	1.04 s	4.22 d(11.2) 4.17 d(11.2)	3.76 d(10.8) 3.71 d(10.8)
23	3.70 s	3.75 s	3.69 s	3.70 s	3.67 s
OAc				1.97 s, 2.02 s	

2.22.4 Daphnilactone-type triterpenoid alkaloids

Table 2-22-15: Cos, MFs, and TSs of Daphnilactone-type triterpenoid alkaloids **2-22-47**~**2-22-56**.

No.	Compounds	MFs	Test solvents	References
2-22-47	daphtenidine A	$C_{31}H_{47}NO_4$	CD_3OD	[686]
2-22-48	daphtenidine B	$C_{24}H_{37}NO_2$	CD_3OD	[686]
2-22-49	caldaphnidine C	$C_{22}H_{33}NO_2$	CD_3OD	[676]
2-22-50	macropodumine I	$C_{22}H_{34}N_2O_2$	CD_3OD	[684]
2-22-51	daphnimacropodine D	$C_{23}H_{35}NO_3$	CD_3OD	[687]
2-22-52	daphnezomine S	$C_{22}H_{33}NO_4$	CD_3OD	[688]
2-22-53	daphnioldhanin C	$C_{22}H_{33}NO_4$	$CDCl_3$	[677]
2-22-54	daphnezomine H	$C_{22}H_{31}NO_3$	$CDCl_3$	[680]
2-22-55	daphnezomine I	$C_{22}H_{31}NO_3$	$CDCl_3$	[680]
2-22-56	calycilactone A	$C_{22}H_{29}NO_3$	$CDCl_3$	[689]



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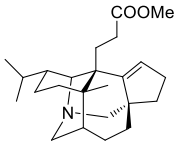

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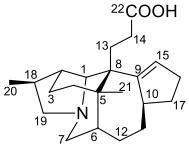

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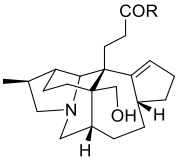
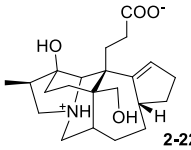


2-22-50 R=NH_2
2-22-51 R=OMe



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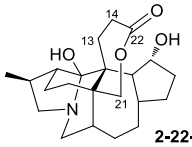

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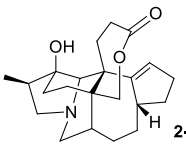

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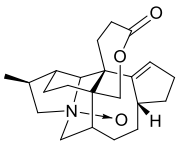

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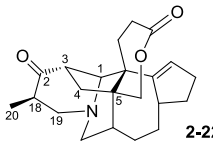


2-22-56

Table 2-22-16: ^{1}H NMR spectroscopic data of daphnilactone-type triterpenoid alkaloids **2-22-47**~**2-22-51**.

H	2-22-47	2-22-48	2-22-49	2-22-50	2-22-51
1	3.29 m	2.58 d(4.4)	3.88 d(3.9)	3.38 br s	3.62 d
2	1.32 m	1.29 m	2.46 m	2.33~2.37 m	2.40 m
3	1.75 m	1.96 m	1.70 m	*α* 1.49~1.52 m	*α* 1.80 m
	1.87 m	1.70 m		*β* 1.67~1.72 m	*β* 1.39 m
4	1.43 m	1.96 m	2.10 m	*α* 1.97~2.00 m	*α* 2.03 m
	1.99 m	1.51 m	1.54 m	*β* 2.05~2.09 m	*β* 2.20 m
6	1.67 m	1.64 m	2.16 m	2.41~2.45 m	2.49 m
7	3.83 d(14.9)	3.73 dd(15.0, 6.2)	3.58 dd(14.8, 11.3)	3.29~3.32 m	*α* 3.79 m
	2.48 d(14.9)	2.65 d(15.0)	3.45 d(14.8)	3.21~3.25 m	*β* 3.15 t(11.5)
10			3.04 t(9.2)	2.96~2.99 m	2.87 m
11	2.17 m	1.90 m	1.76 m	1.69~1.72 m	*α* 2.05 m
	1.60 m	1.57 m	1.51 m	1.57~1.62 m	*β* 1.64 m
12	2.12 m	1.79 m	1.48 m	2.01~2.05 m	*α* 1.45 m
	1.57 m	1.57 m	2.00 m	1.41~1.47 m	*β* 2.00 m
13	2.19 m	2.08 m	2.15 m	2.12~2.16 m	2.07 m
	2.02 m	2.04 m	1.84 m	1.79~1.83 m	1.74 m
14	4.96 t(8.2)	2.40 m, 2.04 m	2.34 m, 2.28 m	2.13~2.17 m	2.24 m, 2.19 m
15	2.27 m, 1.29 m	5.44 s	5.85 br s	5.72 br s	5.80 s
16	1.71 m	2.48 m	2.44 m	*α* 2.25~2.27 m	*α* 2.18 m
	1.69 m	2.24 ddd(16.1, 8.5, 3.1)	2.23 m	*β* 2.46~2.49 m	*β* 2.87 m
17	1.73 m	1.69 m	1.64 m	*α* 1.58~1.62 m	*α* 1.68 m
	1.47 m	1.57 m	2.14 m	*β* 2.13~2.17 m	*β* 2.01 m
18	1.70 m	1.62 m	2.67 m	2.47~2.53 m	2.53 m
19	1.00 d(6.1)	0.93 d(6.5)	3.96 t(11.3)	3.67~3.71 m	*α* 4.46 br s
			2.84 dd(11.3, 8.8)	2.51~2.55 m	*β* 2.52 m
20	0.95 d(6.9)	2.17 d(6.2)	1.09 d(6.8)	1.09 d(6.8)	1.05 d(6.5)
21	1.20 s	0.80 s	1.23 s	4.21 d(10.7)	4.28 d(10.0)
				3.58 d(10.7)	3.65 d(10.0)
23	2.82 d(14.5)	2.96 d(13.3)			
	2.59 d(14.5)	2.77 d(13.3)			
25	0.91 s				
26	4.69 d(12.6)				
	3.69 d(12.6)				
27	4.71 m				
28	2.03 m				
29	1.89 m, 2.10 m				
31	1.37 s				
COOMe		3.68 s			3.63 s

Table 2-22-17: ^{1}H NMR spectroscopic data of daphnilactone-type triterpenoid alkaloids **2-22-52**~**2-22-56**.

H	2-22-52	2-22-53	2-22-54	2-22-55	2-22-56
1	3.52 s		3.86 s	4.78 d(2.7)	3.39 d(5.0)
2		2.57 m		2.73 m	
3	1.68 m	1.72 m	1.88 dt(6.5, 15.8)	1.61 m	2.59~2.53 m
	1.85 m	1.75 m	1.94 dt(6.1, 15.8)	1.92 m	
4	1.47 m	1.97 m	1.45 m	1.52 dt(15.5, 8.1)	1.62~1.56 m
	2.32 m		2.54 m	2.27 m	2.51~2.43 m
6	2.41 m	2.26 m	2.29 m	2.34 m	1.66~1.63 m
7	2.58 m	3.21 m	3.67 d(14.4)	4.11 dd(8.8, 15.5)	2.27~2.20 m
	3.27 m	3.01 d(13.6)	3.30 dd(10.3, 14.4)	4.18 d(15.5)	
9		3.26 m			
10	2.94 br t(9.0)	2.59 m	3.07 m	3.11 m	2.81~2.74 m
11	1.70 m	1.74 m	1.74 m	1.61 m	2.09~2.05 m
	1.93 m		2.22 m	2.28 m	1.52~1.43 m
12	1.37 m, 2.24 m	1.45 m	1.45 m, 1.68 m	1.58 m, 1.68 m	1.43~1.36 m
13	2.05 m	2.25 m	2.52 m	2.02 dd(7.7, 14.1)	1.75~1.68 m
	2.28 m	2.11 m		2.19 m	
14	2.06 m	2.59 m	1.76 m	2.49 dd(7.6, 15.0)	2.59~2.47 m
	2.18 m	2.43 m	3.06 t(13.5)	2.63 t(15.0)	2.42~2.36 m
15	5.73 s	4.25 br	6.03 s	5.90 s	5.46 br s
16	1.29 m	2.33 m	2.24 m	2.31 m	2.37~2.31 m
	2.48 m		2.45 m	2.42 m	2.13~2.07 m
17	1.59 m, 2.12 m	1.63 m	1.70 m, 1.76 m	1.70 m, 2.15 m	2.19~2.11 m
18	2.24 m	2.42 m	2.41 m	3.07 m	2.39~2.29 m
19	2.48 m	2.31 m	2.42 m	3.72 dd(9.4, 12.0)	3.54~3.50 m
	3.77 m	3.26 m	4.29 m	4.61 t(12.0)	2.21~2.15 m
20	1.05 d(6.9)	1.14 d(7.1)	1.11 d(6.5)	1.05 d(6.5)	0.94 d(7.0)
21	3.64 d(10.7)	4.27 d(11.2)	3.74 d(13.1)	3.79 d(13.2)	3.79 d(13.5)
	4.28 d(10.7)	3.81 d(11.2)	4.94 d(13.1)	4.84 d(13.2)	4.75 d(13.5)

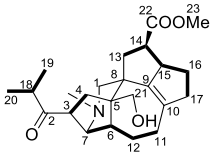
2.22.5 Yuzurine-type triterpenoid alkaloids

Table 2-22-18: Cos, MFs, and TSs of yuzurine-type triterpenoid alkaloids **2-22-57**~**2-22-69**.

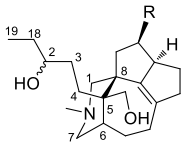
No.	Compounds	MFs	Test solvents	References
2-22-57	daphnilongerine	$C_{24}H_{35}NO_4$	CD_3OD	[690]
2-22-58	daphnoldine A	$C_{22}H_{37}NO_3$	$CDCl_3$	[691]
2-22-59	daphnoldine B	$C_{23}H_{37}NO_4$	$CDCl_3$	[691]
2-22-60	yuzuric acid	$C_{23}H_{35}NO_4$	CD_3OD	[692]
2-22-61	daphnezomic acid	$C_{24}H_{37}NO_4$	CD_3OD	[692]
2-22-62	11-hydroxydaphnigracine	$C_{24}H_{37}NO_5$	CD_3OD	[693]

Table 2-22-18 (continued)

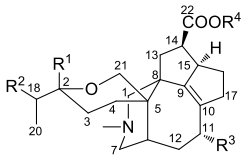
No.	Compounds	MFs	Test solvents	References
2-22-63	daphnezomine R	$C_{25}H_{39}NO_4$	CD_3OD	[688]
2-22-64	dehydrodaphnigraciline	$C_{23}H_{33}NO_3$	$CDCl_3$	[672]
2-22-65	longistylumphylline B	$C_{23}H_{33}NO_3$	CD_3OD	[694]
2-22-66	daphnezomine P	$C_{40}H_{57}NO_{13}$	CD_3OD	[688]
2-22-67	daphnezomine Q	$C_{39}H_{55}NO_{13}$	CD_3OD	[688]
2-22-68	daphmacropodosidine A	$C_{40}H_{57}NO_{13}$	CD_3OD	[695]
2-22-69	daphmacropodosidine B	$C_{39}H_{53}NO_{12}$	C_5D_5N	[695]



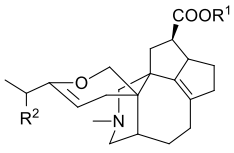
2-22-57



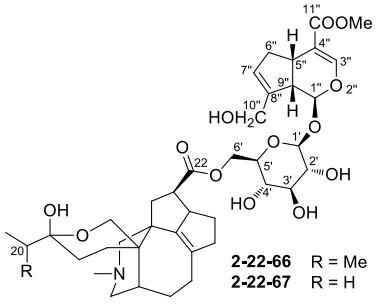
2-22-58 R = CH_2OH
2-22-59 R = COOMe



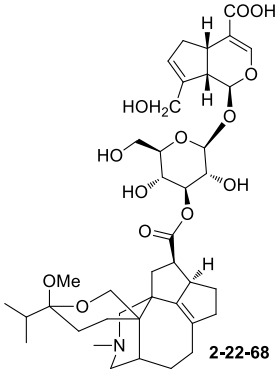
	R^1	R^2	R^3	R^4
2-22-60	OMe	H	H	H
2-22-61	OMe	Me	H	H
2-22-62	OH	Me	OH	Me
2-22-63	OMe	Me	H	Me



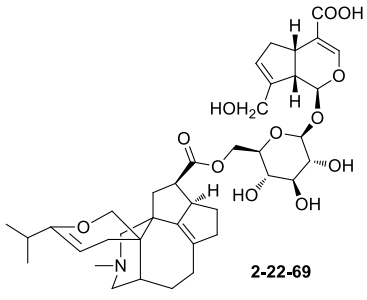
	R^1	R^2
2-22-64	Me	H
2-22-65	H	Me



2-22-66 R = Me
2-22-67 R = H



2-22-68



2-22-69

Table 2-22-19: ^{1}H NMR spectroscopic data of yuzurine-type triterpenoid alkaloids **2-22-57**~**2-22-61**.

H	2-22-57	2-22-58	2-22-59	2-22-60	2-22-61
1	*α* 2.62 br d(11.7)	*α* 2.62~2.68 m	*α* 2.62~2.69 m	2.95 br s	2.73 d(12.2)
	β 2.08 br d(11.7)	*β* 2.55~2.59 m	*β* 2.51~2.60 m		2.67 d(12.2)
2		3.64~3.69 m	3.41~3.48 m		
3	3.25 dd(9.2, 5.4)	1.48~1.54 m	1.86~1.94 m	1.64~1.67 m	1.72~1.76 m
		1.24~1.27 m	1.14 dt(10.0, 2.4)		1.36 br d(10.3)
4	*α* 1.51 dd(9.2, 13.4)	1.94~1.97 m	1.85~1.94 m	*α* 1.92~1.98 m	*α* 1.94~1.96 m
	β 2.21~2.15 m	1.64~1.67 m	1.72~1.81 m	*β* 1.69~1.71 m	*β* 1.66~1.69 m
6	2.04~2.00 m	1.82~1.86 m	1.77~1.86 m	2.55 br s	2.33~2.37 m
7	2.87~2.86 m	*α* 2.32~2.36 m	*α* 2.29~2.36 m	3.30~3.32 m	3.09 br d(12.8)
		β 2.37~2.41 m	*β* 2.37~2.43 m	3.24 dd(13.1, 5.4)	2.97~2.99 m
11	*α* 2.24~2.21 m	*α* 2.54~2.58 m	*α* 2.45~2.58 m	2.35~2.42 m	2.39~2.44 m
	β 1.93~1.89 m	*β* 1.88~1.94 m	*β* 1.88~1.94 m	2.42~2.29 m	2.20 dd(16.2, 5.4)
12	*α* 2.29~2.27 m	*α* 1.70~1.74 m	*α* 1.33~1.41 m	1.74~1.76 m	1.68~1.71 m
	β 1.74~1.71 m	*β* 1.52~1.57 m	*β* 1.22~1.26 m		
13	*α* 1.65 dd(14.0, 8.7)	*α* 1.47~1.56 m	*α* 2.94~3.03 m	*α* 1.61~1.64 m	*α* 1.55~1.58 m
	β 2.45 dd(14.0, 6.9)	*β* 1.38~1.43 m	*β* 1.47~1.56 m	*β* 2.82~2.87 m	*β* 2.80~2.85 m
14	2.97~2.91 m	2.03~2.09 m	2.90~2.96 m	3.48 br s	3.44 br s
15	3.53~3.50 m	3.21~3.27 m	3.43~3.52 m	2.76~2.79 m	2.74~2.77 m
16	*α* 1.95~1.92 m	*α* 2.67~2.70 m	*α* 1.71~1.81 m	1.88~1.93 m	1.86~1.90 m
	β 1.32~1.28 m	*β* 2.63~2.66 m	*β* 1.46~1.54 m	1.53~1.61 m	1.57~1.62 m
17	*α* 2.59~2.54 m	*α* 2.62~2.68 m	*α* 2.60~2.68 m	2.60~2.67 m	2.55~2.60 m
	β 2.35~2.31 m	*β* 2.30~2.37 m	*β* 2.27~2.35 m	2.32~2.38 m	2.29~2.35 m
18	2.86~2.80 m	1.70~1.75 m	1.42~1.52 m	1.71~1.74 m	2.01~2.08 m
		1.50~1.54 m		1.39~1.46 m	
19	1.09 d(7.0)	0.94 t(7.4)	0.93 t(7.4)		0.92 d(6.4)
20	1.06 d(7.0)			0.85 t(7.4)	0.84 d(7.0)
21	*α* 3.85 br d(11.5)	4.18 d(12.0)	4.28~4.33 m	*α* 3.82 dd(12.8, 2.2)	*α* 3.86 dd(12.5, 2.9)
	β 3.59 br d(11.5)	3.54~3.58 m	3.34~3.43 m	*β* 4.25 d(12.8)	*β* 4.21 d(12.9)
22		3.56~3.62 m			
		3.38~3.44 m			
OMe	3.62 s		3.64 s	3.19 s	3.18 s
NMe	2.27 s	2.25 s	2.14 s	2.73 s	2.52 s

Table 2-22-20: ^{1}H NMR spectroscopic data of yuzurine-type triterpenoid alkaloids **2-22-62**~**2-22-65**.

H	2-22-62	2-22-63	2-22-64	2-22-65
1	*α* 2.32 d(11.7)	3.14 d(12.5)	2.36 br d(10.5)	3.11 d(12.2)
	β 2.30 d(11.7)	3.20 d(12.5)	2.10 br d(10.5)	3.04 d(12.2)
3	*α* 1.69 m, *β* 1.41 m	1.95 m, 1.71 m	4.32 br d(5.0)	4.46 dd(5.0, 1.5)

Table 2-22-20 (continued)

H	2-22-62	2-22-63	2-22-64	2-22-65
4	*α* 1.97 m	1.93 m	2.22 m	2.29 d(16.9)
	β 1.69 m	1.68 m	1.80 m	2.02 d(16.9)
6	2.39 m	2.58 br s	1.97 m	2.26 m
7	*α* 2.57 dd(12.9, 3.5)	3.48 dd(5.2, 13.8)	2.67 br d(12.0)	3.35 dd(14.2, 5.9)
	β 2.66 d(12.9)	3.53 br d(13.0)	2.49 dd(12.0, 4.5)	3.45 d(13.6)
11	3.98 t(3.1)	1.75 m, 2.42 m	2.22 m, 1.60 m	2.35 m, 2.41 m
12	*α* 1.86 m, *β* 2.39 m	2.34 m, 2.45 m	2.49 m, 1.83 m	2.32 m, 1.67 m
13	*α* 1.76 m	1.79 m	1.60 dd(14.5, 9.0)	1.78 dd(14.9, 9.1)
	β 2.82 dd(15.0, 2.5)	2.76 m	2.40 dd(14.5, 2.5)	2.56 dd(14.9, 2.6)
14	2.95 ddd(9.7, 7.4, 2.5)	2.99 m	2.86 m	2.84 m
15	3.48 m	3.62 m	3.44 br s	3.57 m
16	*α* 1.39 m	1.33 t(10.2)	1.86 m	1.93 m
	β 1.86 m	1.90 m	1.26 m	1.60 m
17	*α* 2.45 dd(15.0, 8.2)	2.39 m	2.33 m	2.70 m
	β 2.88 m	2.69 br s	2.62 m	2.42 m
18	1.72 m	2.08 m	1.99 m	2.22 m
19	0.92 d(6.9)	0.86 d(7.0)	1.02 t(7.5)	1.02 d(6.9)
20	0.92 d(6.9)	0.94 d(6.9)		1.02 d(6.9)
21	*α* 3.57 d(12.6)	3.78 dd(2.7, 12.6)	4.46 dd(11.5, 2.5)	4.42 dd(11.9, 2.6)
	β 4.32 d(12.6)	4.11 d(12.5)	3.96 br d(11.5)	4.06 br d(11.9)
23	3.61 s	3.65 s	3.62 s	
NMe	2.25 s	2.90 s	2.17 s	2.84 s

Table 2-22-21: ^{1}H NMR spectroscopic data of yuzurine-type triterpenoid alkaloids **2-22-66**~**2-22-69**.

H	2-22-66	2-22-67	2-22-68	2-22-69
1	3.13 d(12.5)	3.17 m, 3.19 m	2.55 d(10.8)	2.32 d(10.4)
	3.19 m		2.43 d(10.8)	2.01 d(10.4)
3	1.96 dd(2.7, 13.7)	1.70 m, 1.97 m	1.35 m, 1.73 m	4.45 br d(4.2)
	1.75 m			
4	1.75 m	1.69 m, 1.73 m	1.58 m, 1.96 m	2.26 dd(16.8, 4.2)
	1.70 dd(9.4, 12.3)			1.88 d(16.8)
6	2.57 m	2.63 br s	2.31 m	2.09 m
7	3.48 m, 3.53 d(13.7)	3.47 m, 3.51 m	2.77 (ov), 2.90 d(12.4)	2.46 m, 2.63 d(11.6)
11	1.77 m, 2.35 m	1.77 m, 2.34 m	2.11 m, 1.65 m	2.64 m, 2.40 m
12	2.36 m, 2.40 m	2.29 m, 2.43 m	2.45 m, 2.27 m	1.97 m, 1.62 m
13	1.79 m, 2.77 m	1.77 m, 2.76 m	2.76 m, 1.68 m	2.47 m, 1.54 m
14	2.99 dt(2.7, 10.1)	3.00 m	3.00 m	2.95 m
15	3.61 m	3.65 m	3.55 m	3.53 m
16	1.41 m, 1.90 m	1.60 m, 2.06 m	1.83 m, 1.73 m	1.96 m, 1.56 m
17	2.42 m, 2.67 m	2.40 m, 2.67 m	2.57 m, 2.33 m	2.48 m, 2.36 m
18	2.07 m	1.45 m, 1.90 m	2.04 m	2.33 m

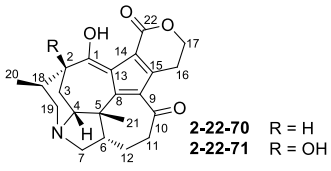
Table 2-22-21 (continued)

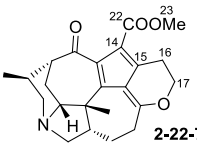
H	2-22-66	2-22-67	2-22-68	2-22-69
19	0.85 d(7.0)	0.86 t(7.4)	0.84 d(6.9)	1.08 d(6.8)
20	0.93 d(6.8)		0.93 d(6.7)	1.08 d(6.8)
21	3.76 dd(2.8, 12.5)	3.86 br d(11.1)	4.06 d(12.4)	4.72 d(11.6)
	4.08 d(12.5)	4.11 m	3.88 d(12.4)	4.38 d(11.6)
NMe	2.90 s	2.90 s	2.34 s	2.11 s
OMe			3.17 s	
1′	4.71 d(7.9)	4.71 d(7.8)	4.77 d(7.6)	5.39 d(7.8)
2′	3.23 dd(8.0, 9.1)	3.23 dd(8.5, 8.5)	3.35 m	4.06 m
3′	3.40 dd(9.1, 9.1)	3.39 dd(9.1, 9.1)	4.95 t(7.0)	4.25 t(8.9)
4′	3.30 dd(9.1, 9.1)	3.31 m	3.46 m	3.99 t(8.9)
5′	3.48 m	3.47 m	3.32 m	4.06 m
6′	4.14 dd(6.3, 12.0)	4.13 m	3.84 d(12.0)	5.01 d(11.3)
	4.48 dd(1.7, 12.0)	4.48 br d(10.8)	3.66 dd(12.0, 5.1)	4.55 dd(11.3, 6.7)
1″	5.01 d(7.9)	5.01 d(7.9)	5.05 d(7.5)	5.68 d(7.3)
3″	7.52 s	7.51 s	7.22 s	7.94 s
5″	3.17 m	3.18 m	3.21 m	3.57 m
6″	2.07 m	2.08 m	2.83 m	3.15 m
	2.86 dd(8.7, 16.6)	2.86 m	2.06 m	2.44 m
7″	5.82 s	5.81 s	5.76 s	6.02 s
9″	2.75 m	2.74 m	2.63 m	3.13 m
10″	4.21 d(14.0)	4.21 d(13.2)	4.27 d(13.2)	4.77 d(14.2)
	4.26 d(14.0)	4.23 d(13.2)	4.16 d(13.2)	4.65 d(14.2)
COOMe	3.35 s	3.35 s		

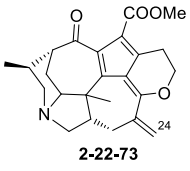
2.22.6 Daphnicyclidine-type triterpenoid alkaloids

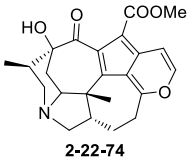
Table 2-22-22: Cos, MFs, and TSs of daphnicyclidine-type triterpenoid alkaloids **2-22-70**~**2-22-80**.

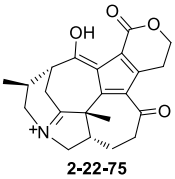
No.	Compounds	MFs	Test solvents	References
2-22-70	daphnicyclidin A	$C_{22}H_{25}NO_4$	$CDCl_3$-CD_3OD(9:1)	[696]
2-22-71	daphnicyclidin C	$C_{22}H_{25}NO_5$	CD_3OD	[696]
2-22-72	daphnicyclidin D	$C_{23}H_{27}NO_4$	$CDCl_3$-CD_3OD(9:1)	[696]
2-22-73	paxiphylline A	$C_{24}H_{27}NO_4$	$CDCl_3$	[697]
2-22-74	paxiphylline B	$C_{23}H_{25}NO_5$	$CDCl_3$	[697]
2-22-75	daphnicyclidin B	$C_{22}H_{24}NO_4$	CD_3OD	[696]
2-22-76	daphnipaxinin	$C_{21}H_{24}N_2O_3$	CD_3OD	[698]
2-22-77	oldhamine B	$C_{21}H_{23}NO_4$	CD_3OD	[699]
2-22-78	oldhamine A	$C_{21}H_{23}NO_4$	CD_3OD	[699]
2-22-79	daphnicyclidin J	$C_{23}H_{25}NO_5$	CD_3OD	[700]
2-22-80	daphnicyclidin K	$C_{23}H_{27}NO_6$	$CDCl_3$-CD_3OD(1:1)	[700]

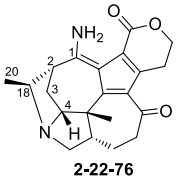


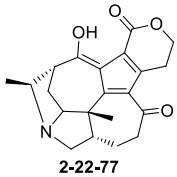


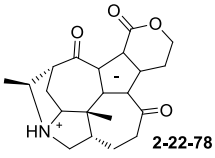


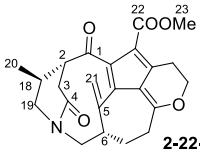












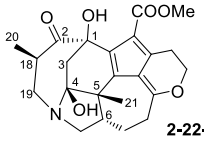


Table 2-22-23: ^{1}H NMR spectroscopic data of daphnicyclidine-type triterpenoid alkaloids **2-22-70**~**2-22-73**.

H	2-22-70	2-22-71	2-22-72	2-22-73
2	2.87 br s		2.58 br s	2.55 m
3	2.26 d(15.8)	2.45 m	2.12 d(15.4)	2.30 m
	2.45 dt(15.8, 6.0)		2.40 m	2.14 m
4	3.74 d(6.7)	3.95 d(7.2)	3.77 m	3.04 m
6	2.64 m	2.81 m	2.58 m	2.37 m
7	2.52 dd(8.7, 12.2)	2.81 m	2.53 m	*α* 2.36 m
	4.07 br t(11.3)	4.11 m	3.96 m	*β* 3.49 m
11	2.61 m	2.66 dd(3.3, 19.7)	2.60 dd(5.5, 18.6)	
		2.74 dd(13.0, 19.7)	2.73 dt(13.0, 18.6)	
12	1.67 m, 2.11 m	1.79 m, 2.19 m	1.46 m, 2.24 m	*α* 2.19 m, *β* 2.82 m
16	2.69 dt(18.8, 5.1)	2.81 m	2.67 dd(4.9, 17.0)	3.01 m
	3.27 m	3.28 ddd(5.7, 9.2, 18.1)	2.96 dd(3.0, 17.0)	2.83 m
17	4.38 dt(4.7, 9.8)	4.46 m	4.04 ddd(3.0, 11.0, 14.5)	4.67 dd(4.8, 10.4)
	4.51 m	4.59 m	4.57 dd(5.4, 11.0)	4.14 ddd(3.6, 10.4, 18.4)
18	2.24 m	2.24 m	2.24 m	2.27 m
19	3.05 dd(3.4, 12.8)	3.36 dd(2.9, 13.2)	3.13 m	*α* 2.98 m
	3.24 br d(13.3)	3.47 br d(13.2)		*β* 2.53 br
20	1.32 d(7.3)	1.32 d(7.3)	1.17 d(6.7)	1.24 d(4.5)
21	1.42 s	1.50 s	1.34 s	1.22 s
23			3.66 s	3.81 s
24				5.95 s, 5.63 s

Table 2-22-24: ^{1}H NMR spectroscopic data of daphnicyclidine-type triterpenoid alkaloids **2-22-74**~**2-22-76**.

H	2-22-74	2-22-75	2-22-76
2		3.13 br s	2.89 dd(8.2, 1.4)
3	2.28 d(6.4)	3.69 dd(2.4, 13.8)	*α* 2.46 ddd(13.9, 8.2, 7.1)
		2.77 m	*β* 2.27 d(13.9)
4	3.57 m		3.40 d(7.1)
6	2.58 m	2.97 m	2.48 m
7	*α* 3.12 m	3.61 br t(11.4)	*α* 2.33 dd(10.8, 10.6)
	β 2.34 m	4.43 dd(8.7, 13.6)	*β* 3.54 dd(10.8, 7.7)
11	3.59 m	2.57 dd(6.6, 18.0)	*α* 2.49 ddd(19.8, 5.7, 1.7)
	2.94 m	2.79 m	*β* 2.74 ddd(19.6, 13.8, 1.6)
12	*α* 2.56 m	1.55 m	*α* 1.43 dddd(13.8, 13.7, 11.2, 1.7)
	β 1.67 m	2.18 m	*β* 2.07 dddd(13.7, 5.7, 5.7, 1.6)
16	7.69 br s	2.81 m	*α* 2.72 ddd(17.9, 5.3, 4.4)
		2.89 ddd(5.0, 13.8, 18.3)	*β* 3.17 ddd(17.9, 10.4, 5.3)
17	7.69 br s	4.20 ddd(3.7, 10.5, 13.8)	*α* 4.26 ddd(10.5, 10.4, 4.4)
		4.39 dd(5.0, 10.5)	*β* 4.41 ddd(10.5, 5.3, 5.3)
18	2.71 m	2.56 m	3.12 qd(6.7, 1.4)
19	*α* 3.11 m	4.10 dd(7.7, 14.2)	
	β 2.62 m	3.32 dd(9.1, 14.2)	
20	0.87 d(6.8)	1.33 d(6.9)	1.26 d(6.7)
21	1.45 s	1.82 s	1.34 s
23	3.84 s		

Table 2-22-25: ^{1}H NMR spectroscopic data of daphnicyclidine-type triterpenoid alkaloids **2-22-77**~**2-22-80**.

H	2-22-77	2-22-78	2-22-79	2-22-80
2	3.26 br d(8.2)	3.00 d(7.9)	2.70 ddd(1.1, 3.0, 5.0)	
3	2.75 m	*α* 2.69 m	2.49 dd(5.0, 15.9)	2.06 d(14.8)
		β 2.60 m	2.85 ddd(1.9, 3.0, 15.9)	2.20 d(14.8)
4	4.25 br d(6.5)	4.19 br d(7.9)		
6	2.65 m	2.62 m	2.99 m	2.68 m
7	*α* 2.89 m	*α* 2.83 m	2.44 d(13.9)	2.48 dd(8.3, 10.9)
	β 4.14 m	*β* 4.10 m	4.69 dd(8.8, 13.9)	3.19 t(7.6)
11	*α* 2.56 dd(16.8, 5.6)	*α* 2.49 dd(17.6, 5.2)	2.56 ddd(1.4, 7.0, 17.6)	2.51 ddd(2.4, 6.7, 10.6)
	β 2.85 m	*β* 2.88 m	2.62 dd(10.2, 17.6)	3.00 ddd(2.5, 12.2, 17.5)
12	*α* 2.12 m	*α* 2.12 m	1.76 m	1.58 m
	β 1.42 m	*β* 1.35 br d(11.9)	2.19 m	2.37 m
16	*α* 2.75 m	*α* 2.74 m	3.05 ddd(5.7, 15.5, 19.0)	2.96 ddd(3.2, 5.7, 18.7)
	β 3.32 m	*β* 3.10 m	3.30 ddd(1.4, 4.4, 19.0)	3.27 ddd(1.4, 4.1, 18.7)
17	4.55 m	*α* 4.29 m	4.22 ddd(4.4, 11.1, 15.5)	4.16 ddd(4.2, 11.0, 15.4)
		β 4.41 m	4.74 ddd(1.4, 5.7, 11.1)	4.62 ddd(1.4, 4.2, 7.1)
18	4.20 m	4.06 m	2.48 m	2.85 m
19			3.32 dd(9.4, 13.3)	3.10 dd(10.2, 15.5)
			2.94 dd(6.0, 13.3)	2.91 dd(2.7, 15.5)

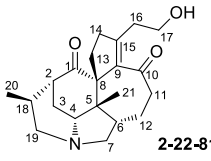
Table 2-22-25 (continued)

H	2-22-77	2-22-78	2-22-79	2-22-80
20	1.59 d(7.0)	1.55 d(6.8)	0.98 d(7.1)	0.88 d(6.8)
21	1.55 s	1.51 s	4.99 d(2.2) 5.07 dd(0.9, 2.2)	1.33 s
23			3.75 s	3.69 s

2.22.7 Calyciphylline-type triterpenoid alkaloids

Table 2-22-26: Cos, MFs, and TSs of calyciphylline-type triterpenoid alkaloids **2-22-81**~**2-22-94**.

No.	Compounds	MFs	Test solvents	References
2-22-81	daphniyunnine B	$C_{21}H_{29}NO_3$	$CDCl_3$-CD_3OD(9:1)	[701]
	longeracinphyllin B	$C_{21}H_{29}NO_3$	$CDCl_3$-CD_3OD(1:9)	[702]
2-22-82	daphnipaxianine C	$C_{21}H_{29}NO_2$	CD_3OD	[703]
2-22-83	daphnilongeranin C	$C_{22}H_{29}NO_3$	$CDCl_3$	[671]
2-22-84	daphniyunnine A	$C_{23}H_{31}NO_3$	CD_3OD	[701]
2-22-85	calyciphylline A	$C_{23}H_{31}NO_4$	CD_3OD	[704]
2-22-86	daphniyunnine D	$C_{21}H_{27}NO_3$	$CDCl_3$	[701]
2-22-87	daphniyunnine E	$C_{21}H_{27}NO_3$	$CDCl_3$	[701]
2-22-88	daphnilongeranin B	$C_{21}H_{27}NO_2$	CD_3OD	[671]
2-22-89	daphnipaxianine A	$C_{21}H_{27}NO_3$	$CDCl_3$	[703]
2-22-90	daphnipaxianine B	$C_{21}H_{27}NO_3$	$CDCl_3$	[703]
2-22-91	daphniyunnine C	$C_{21}H_{27}NO_2$	$CDCl_3$	[701]
2-22-92	longistylumphylline A	$C_{23}H_{29}NO_3$	$CDCl_3$	[694]
2-22-93	macropodumine D	$C_{23}H_{31}NO_5$	$CDCl_3$	[705]
2-22-94	daphnilongeranin A	$C_{23}H_{29}NO_4$	CD_3OD	[671]



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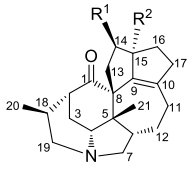
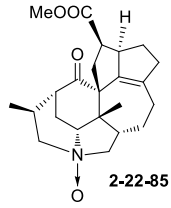


	R^1	R^2
2-22-82	H	OH
2-22-83	COOH	H
2-22-84	COOMe	H



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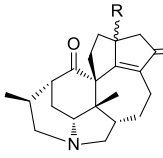
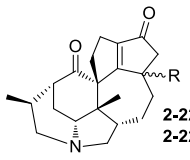
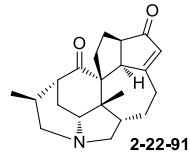


2-22-86 R = α-OH
2-22-87 R = β-OH
2-22-88 R = α-H



2-22-89 R = α-OH
2-22-90 R = β-OH



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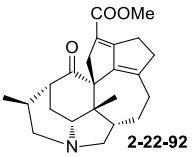

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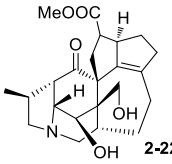
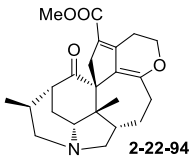


2-22-93



2-22-94

Table 2-22-27: ^{1}H NMR spectroscopic data of calyciphylline-type triterpenoid alkaloids **2-22-81**~**2-22-84**.

H	2-22-81 ($CDCl_3$-CD_3OD,9:1)	2-22-81 ($CDCl_3$-CD_3OD,1:9)	2-22-82	2-22-83	2-22-84
2	2.06 m	2.10 br d(3.3)	2.21 m	2.24 m	2.16 m
3	*α* 2.05 m	2.08 br dd(15.4, 3.8)	2.36 m	*α* 2.06 m	*α* 1.98 m
	β 2.41 m	2.47 m	2.08 m	*β* 2.39 m	*β* 2.15 m
4	3.50 br d(3.9)	3.51 br d(2.2)	3.43 d(4.5)	3.73 br s	3.21 br d(5.0)
6	2.32 m	2.28 m	2.09 m	2.35 m	2.25 m
7	*α* 2.83 m	2.77 m	2.85 m	*α* 2.52 br t(11.4)	*α* 2.66 dd(11.2, 9.9)
	β 2.78 m		2.78 m	*β* 3.23 dd(10.6, 6.8)	*β* 2.88 dd(9.9, 7.1)
11	*α* 2.53 m, *β* 2.35 m	2.28 m, 2.41 m	1.82 m, 1.59 m	*α* 1.94 m, *β* 2.11 m	*α* 2.09 m, *β* 2.03 m
12	*α* 1.79 m	1.78 m	1.66 m	*α* 1.55 m	*α* 1.65 m
	β 1.96 m	2.02 br dd(14.3, 3.3)	1.54 m	*β* 1.94 m	*β* 1.94 m
13	*α* 1.81 m	1.79 m	2.92 m	*α* 2.25 m	*α* 2.32 m
	β 2.66 m	2.67 br dd(11.5, 8.3)	2.16 m	*β* 2.73 dd(13.2, 9.1)	*β* 2.76 m
14	2.48 m	2.36 m, 2.53 m	2.35 m, 2.22 m	2.66 m	2.79 m
15				3.43 m	3.38 m
16	2.66 m, 2.39 m	2.48 m, 2.60 m	2.49 m, 2.13 m	*α* 1.98 m, *β* 1.47 m	*α* 1.87 m, *β* 1.27 m
17	3.78 m	3.67 m	2.23 m	*α* 2.31 m	*α* 2.68 m
	3.76 td(9.7, 4.2)			*β* 2.68 m	*β* 2.35 m
18	2.87 m	2.80 m	2.76 m	2.71 m	2.78 m
19	*α* 2.80 m	2.54 m	2.88 m	*α* 2.87 m	*α* 2.78 m
	β 2.52 m	2.79 m	2.57 dd(11.2, 8.4)	*β* 2.68 m	*β* 2.48 m
20	0.97 d(6.6)	0.99 d(6.0)	1.03 d(6.8)	1.08 d(6.0)	1.01 d(6.5)
21	1.31 s	1.34 s	1.20 s	1.42 s	1.33 s
COOMe					3.62 s

Table 2-22-28: ^{1}H NMR spectroscopic data of calyciphylline-type triterpenoid alkaloids **2-22-85**~**2-22-89**.

H	2-22-85	2-22-86	2-22-87	2-22-88	2-22-89
2	2.45 br s	2.34 br d(4.0)	2.16 m	2.27 br d(1.3)	2.16 d(4.5)
3	2.44 m	*α* 2.11 m, *β* 2.28 m	*α* 2.05 m, *β* 2.19 m	*α* 2.08 m, *β* 2.28 m	2.31 m, 2.09 m
4	3.80 br s	3.59 br d(3.0)	3.43 br d(4.6)	3.26 br d(4.9)	3.53 d(4.4)
6	2.88 m	2.43 m	2.36 m	2.24 m	2.26 m
7	3.21 dd(11.1, 12.3)	*α* 2.92 m	*α* 2.89 m	*α* 2.89 dd(11.7, 9.9)	2.90 m
	3.35 dd(7.6, 12.3)	*β* 3.11 m	*β* 2.82 m	*β* 2.86 dd(9.9, 8.8)	
11	2.05 br dd(6.2, 17.3)	*α* 2.07 m	*α* 1.89 ddd(16.8, 13.3, 2.1)	*α* 2.02 m	2.03 m
	2.18 br dd(12.1, 16.4)	*β* 2.46 ddd(15.9, 5.7, 2.9)	*β* 2.29 ddd(16.8, 4.7, 2.5)	*β* 2.36 ddd(15.7, 5.6, 3.0)	1.75 m
12	1.59 m	*α* 1.87 m	*α* 1.81 m	*α* 1.75 m	1.78 m
	1.91 m	*β* 1.72 m	*β* 1.64 m	*β* 1.85 m	1.37 m
13	2.80 m	*α* 2.23 m	*α* 2.11 dd(13.0, 6.3)	*α* 1.00 m	2.92 m
	2.41 dd(10.2, 16.2)	*β* 2.63 dd(12.5, 8.0)	*β* 2.90 m	*β* 2.77 dd(13.9, 7.8)	2.25 m
14	2.82 m	*α* 2.23 m	*α* 1.41 td(12.9, 6.3)	*α* 2.13 m	2.41 m
		β 1.43 td(14.7, 8.0)	*β* 2.01 dd(12.9, 5.4)	*β* 1.18 m	
15	3.40 m			2.74 m	
16	1.34 m	*α* 2.72 d(17.7)	2.52 s	*α* 2.58 dd(18.0, 6.2)	
	1.93 m	*β* 2.44 d(17.7)		*β* 2.08 m	
17	2.41 dd(10.2, 16.2)				2.93 m
	2.65 m				2.68 m
18	2.51 m	2.83 m	2.80 m	2.72 m	2.92 m
19	3.56 dd(6.8, 13.5)	*α* 2.96 dd(14.0, 7.4)	*α* 2.81 m	*α* 2.54 dd(14.2, 9.7)	2.88 m
	3.03 dd(8.8, 13.5)	*β* 2.68 m	*β* 2.51 dd(16.9, 13.1)	*β* 2.85 dd(14.2, 7.0)	2.56 dd(11.2, 8.0)
20	1.22 d(6.9)	1.08 d(6.8)	1.00 d(6.4)	1.02 d(6.8)	1.04 d(6.5)
21	1.57 s	1.25 s	1.29 s	1.24 s	1.16 s
COOMe	3.63 s				

Table 2-22-29: ^{1}H NMR spectroscopic data of calyciphylline-type triterpenoid alkaloids **2-22-90**~**2-22-94**.

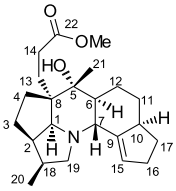
H	2-22-90	2-22-91	2-22-92	2-22-93	2-22-94
2	2.06 m	2.17 br d(3.9)	2.19 d(1.9)	2.60 d(5.9)	2.31 br d(3.9)
3	2.21 m	*α* 2.08 m	2.02 m	3.33 dd(5.6, 3.9)	*α* 2.12 dd(15.4, 3.9)
	2.04 m	*β* 2.35 br dd(15.4, 4.8)			*β* 2.33 m
4	3.45 br s	3.62 br d(5.0)	3.33 d(4.0)	4.42 d(4.4)	3.51 m
6	2.37 m	2.45 m	2.28 m	1.94 m	2.29 m
7	2.79 m	*α* 2.54 dd(12.6, 9.8)	2.86 m	2.51 dd(12.6, 2.7)	2.89 m
	2.85 m	*β* 3.31 dd(9.8, 6.2)		2.18 m	
9		4.12 br d(5.2)			
11	1.79 m	2.89 m	2.01 m	1.80 m	*α* 2.39 m
			2.13 m	1.92 m	*β* 1.96 ddd(16.3, 6.4, 2.0)
12	2.46 m	*α* 1.74 m	1.81 m	1.46 m	*α* 1.79 m
	1.68 m	*β* 2.04 m		1.92 m	*β* 1.87 m
13	2.50 m	*α* 1.90 m	2.87 m	*α* 2.18 m	*α* 2.75 d(17.5)
	2.61 m	*β* 1.77 m	3.49 br d(16.8)	*β* 3.69 m	*β* 3.35 d(17.5)
14	2.22 m	*α* 1.25 m		2.96 m	
	1.99 m	*β* 1.88 m			
15		2.73 dd(9.7, 5.6)		3.85 m	
16			2.71 m	*α* 1.96 m	*α* 3.07 m
				β 1.13 dd(11.0, 8.3)	*β* 2.72 m
17	2.93 d(16.0)	6.08 d(1.5)	3.00 br d(18.2)	*α* 2.72 m	*α* 4.15 ddd(10.8, 5.0, 5.0)
	2.67 d(16.0)		2.90 br d(18.2)	*β* 2.32 dd(14.8, 7.9)	*β* 3.94 ddd(10.8, 10.8, 3.7)
18	2.87 m	2.85 m	2.78 m	2.98 m	2.73 m
19	2.78 m	*α* 2.95 dd(13.9, 7.7)	2.83 m	2.95 m	*α* 2.65 dd(13.8, 10.9)
	2.50 m	*β* 2.64 dd(13.9, 10.5)	2.54 dd(13.7, 9.3)	2.43 m	*β* 2.91 dd(13.8, 6.9)
20	0.98 d(6.8)	1.08 d(6.6)	0.98 d(6.1)	1.09 d(7.1)	1.04 d(6.7)
21	1.31 s	1.23 s	1.18 s	4.54 d(12.1)	1.26 s
				3.71 d(12.1)	
COOMe			3.68 s	3.66 q	3.69 s

Table 2-22-30: Cos, MFs, and TSs of calyciphylline-type triterpenoid alkaloids **2-22-95**~**2-22-105**.

No.	Compounds	MFs	Test solvents	References
2-22-95	longistylumphylline C	$C_{23}H_{35}NO_3$	$CDCl_3$	[694]
2-22-96	caldaphnidine R	$C_{23}H_{35}NO_3$	CD_3OD	[668]

Table 2-22-30 (continued)

No.	Compounds	MFs	Test solvents	References
2-22-97	deoxycalyciphylline B	$C_{22}H_{31}NO_2$	$CDCl_3$	[706]
2-22-98	deoxyisocalyciphylline B	$C_{22}H_{31}NO_2$	$CDCl_3$	[706]
2-22-99	calyciphylline B	$C_{22}H_{31}NO_3$	CD_3OD	[706]
2-22-100	daphnioldhanine J	$C_{22}H_{31}NO_3$	$CDCl_3$	[672]
2-22-101	calydaphninone	$C_{23}H_{31}NO_4$	$CDCl_3$-C_5D_5N(3:1)	[707]
2-22-102	calyciphylline C	$C_{23}H_{31}NO_3$	CD_3OD	[708]
2-22-103	calyciphylline J	$C_{22}H_{29}NO_3$	$CDCl_3$	[669]
2-22-104	calyciphylline D	$C_{29}H_{45}NO_3$	CD_3OD	[709]
2-22-105	calyciphylline F	$C_{22}H_{35}NO_2$	CD_3OD	[683]



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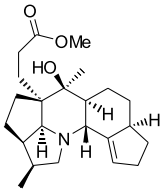

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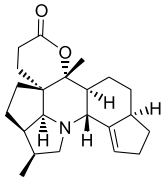

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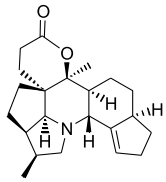

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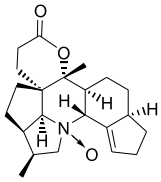

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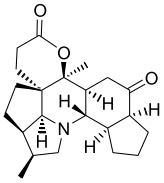

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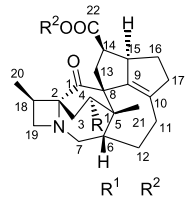
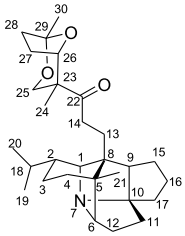


2-22-101 OH Me
2-22-102 H Me
2-22-103 H H



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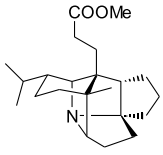


2-22-105

Table 2-22-31: ^{1}H NMR spectroscopic data of calyciphylline-type triterpenoid alkaloids **2-22-95**~**2-22-98**.

H	2-22-95	2-22-96	2-22-97	2-22-98
1	4.49 t(7.2)	3.30 m	3.64 d(4.9)	3.17 d(4.6)
2	2.64 m	2.49 m	2.61 m	2.59 m

Table 2-22-31 (continued)

H	2-22-95	2-22-96	2-22-97	2-22-98
3	1.67 m, 1.58 m	1.46 m	1.62 m	*α* 1.87 m, *β* 1.63 m
4	1.43 m, 2.13 m	1.85 m, 1.43 m	*α* 1.77 m, *β* 1.35 m	*α* 1.60 m, *β* 2.01 m
6	1.66 m	1.46 m	2.05 m	2.47 dd(15.0, 7.4)
7	4.34 br s	3.72 d(2.5)	3.03 d(11.4)	3.78 d(7.2)
10	2.95 br s	2.72 m	2.98 m	2.59 m
11	2.11 m	2.03 m	*α* 1.97 m	*α* 1.90 m
	0.87 dd(22.9, 11.5)	0.91 m	*β* 1.32 m	*β* 1.10 ddd(23.7, 12.2, 3.4)
12	1.65 m	1.73 m	*α* 1.81 dd(12.7, 8.0)	1.75 m
	1.80 m	1.52 m	*β* 1.04 m	
13	1.92 m, 1.65 m	1.66 m, 1.60 m	1.62 m	*α* 1.92 m, *β* 1.63 m
14	2.62 m, 2.23 m	2.39 m	2.46 m	2.75 m
15	5.81 s	5.41 br d(1.7)	5.49 br d(2.0)	5.59 br s
16	2.38 m, 2.32 m	2.28 m	2.32 m	2.26 m
17	2.17 m, 1.34 m	2.12 m, 1.34 m	*α* 2.21 m, *β* 1.41 m	*α* 2.12 m, *β* 1.45 m
18	2.53 m	2.25 m	2.41 dd(12.3, 6.3)	2.39 m
19	3.81 m	2.95 m	*α* 3.12 dd(8.5, 6.1)	*α* 3.08 dd(9.8, 7.5)
	2.77 dd(20.9, 10.3)	2.22 m	*β* 2.02 dd(11.7, 8.5)	*β* 2.51 br d(10.0)
20	0.98 d(6.7)	0.97 d(6.7)	1.02 d(6.8)	1.03 d(6.7)
21	1.39 s	1.25 s	1.29 s	1.49 s
COOMe	3.67 s	3.64 s		

Table 2-22-32: ^{1}H NMR spectroscopic data of calyciphylline-type triterpenoid alkaloids **2-22-99**~**2-22-103**.

H	2-22-99	2-22-100	2-22-101	2-22-102	2-22-103
1	4.04 d(6.5)	3.18 m			
2	3.03 m	2.68 m			
3	1.82 m	1.56 m	*α* 1.80 m, *β* 2.20 m	2.29 m, 1.15 m	2.32 m, 1.38 m
4	*α* 1.86 m, *β* 1.92 m	2.11 m	3.81 br d(9.0)	1.98 m, 1.88 m	1.92 m, 1.82 m
6	2.59 t(13.1)	2.42 m	2.58 m	1.98 m	2.06 m
7	4.03 d(10.6)	3.77 m	2.47 m, 2.78 m	2.61 m, 2.87 m	2.58 m, 2.96 dd(12.0, 4.6)
9		2.18 m			
10	3.15 m	2.68 m			
11	*α* 2.11 m, *β* 1.42 m		1.98 m	2.15 m	2.06 m
12	*α* 1.81 m, *β* 1.15 m	2.64 m	*α* 1.55 m, *β* 1.87 m	2.15 m, 1.71 m	2.06 m, 1.59 m
13	*α* 1.63 ddd(13.9, 9.1, 4.4)	1.66 m	*α* 1.70 dd(9.0, 14.0)	2.78 m	2.74 dd(13.7, 6.3)
		1.92 m	*β* 2.61 dd(6.5, 14.0)	1.98 m	1.92 m
	β 1.81 m				
14	*α* 2.50 ddd(25.9, 12.4, 4.1)	2.74 m	2.98 m	3.11 m	3.11 m
	β 2.63 m				
15	5.99 d(2.3)	1.88 m	3.86 m	3.99 m	4.01 m

Table 2-22-32 (continued)

H	2-22-99	2-22-100	2-22-101	2-22-102	2-22-103
16	2.35 m	2.35 m	α 1.83 m, β 1.18 m	1.98 m, 1.32 m	2.02 m, 1.47 m
17	α 2.32 m, β 1.42 m	1.71 m	α 2.48 m, β 2.14 m	2.61 m, 2.33 m	2.61 m, 2.27 m
18	3.03 m	2.36 m	2.86 m	3.04 m	3.05 m
19	α 3.47 m, β 3.07 m	2.50 m	2.81 m, 2.92 m	3.04 m, 2.96 m	3.10 m
20	1.11 d(6.3)	1.03 d(6.5)	0.96 d(7.0)	1.13 br s	1.12 br d(6.3)
21	1.35 s	1.54 s	1.23 s	1.24 s	1.19 s
COOMe			3.51 s	3.67 s	

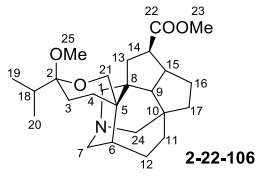
Table 2-22-33: ^{1}H NMR spectroscopic data of calyciphylline-type triterpenoid alkaloids **2-22-104** and **2-22-105**.

H	2-22-104	2-22-105	H	2-22-104	2-22-105
1	3.64 m	3.65 d(1.8)	17	2.17 m, 1.89 m	2.17 m, 1.89 m
2	1.55 m	1.53 m	18	1.53 m	1.52 m
3	1.99 m, 1.63 m	1.99 m, 1.50 m	19	1.07 d(6.0)	1.06 d(6.0)
4	2.06 m, 1.55 m	1.97 m, 1.58 m	20	1.05 d(6.0)	1.04 d(6.0)
6	4.15 m	4.18 t(5.2)	21	1.13 s	1.10 s
9	2.42 m	2.42 m	24	0.85 s	
11	2.08 m, 2.31 m	2.08 m, 2.31 m	25	4.30 dd(12.3, 1.9)	
12	2.19 m	2.18 m		3.65 d(12.3)	
13	1.84 m, 2.04 m	2.12 m	26	4.73 br d(6.3)	
		1.82 ddd(16.4, 11.8, 4.4)	27	2.09 m	
14	3.04 ddd(18.6, 11.2, 5.6)	2.56 ddd(16.4, 11.8, 5.8)	28	2.13 m	
				1.92 m	
	2.81 ddd(18.6, 10.4, 4.5)	2.38 m	30	1.40 s	
			COOMe		3.72 s
15	2.05 m, 1.63 m	2.05 m, 1.66 m			
16	1.99 m, 1.63 m	2.02 m, 1.66 m			

2.22.8 Paxdaphnine A-type triterpenoid alkaloids

Table 2-22-34: Cos, MFs, and TSs of paxdaphnine A-type triterpenoid alkaloids **2-22-106**~**2-22-110**.

No.	Compounds	MFs	Test solvents	References
2-22-106	daphnipaxianine D	$C_{25}H_{39}NO_4$	$CDCl_3$	[703]
2-22-107	daphlongamine A	$C_{24}H_{35}NO_4$	$CDCl_3$	[678]
2-22-108	daphlongamine B	$C_{21}H_{29}NO_3$	$CDCl_3$	[678]
2-22-109	paxdaphnine A	$C_{22}H_{31}NO_3$	CD_3OD	[710]
			$CDCl_3$	[711]
2-22-110	daphlongeramine A	$C_{27}H_{35}NO_5$	C_5D_5N	[711]



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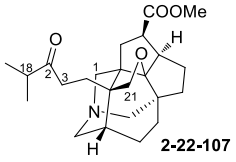

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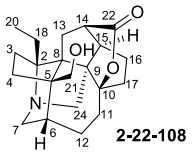
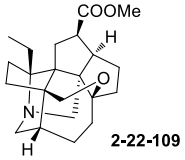


2-22-108



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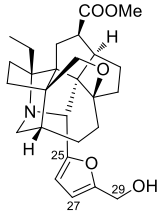


2-22-110

Table 2-22-35: ^{1}H NMR spectroscopic data of paxdaphnine A-type triterpenoid alkaloids **2-22-106**~**2-22-108** and **2-22-110**.

H	2-22-106	2-22-107	2-22-108	2-22-110
1	3.81 d(12.5)	*α* 3.27 d(11.0)		
	3.85 d(12.5)	*β* 2.89 d(11.0)		
3	1.56 m, 1.27 m	2.36 m	1.50 m	*α* 1.65 m, *β* 2.45 m
4	1.83 m, 1.52 m	*α* 1.67 m, *β* 1.86 m	1.92 m	*α* 1.38 m, *β* 1.49 m
6	2.20 m	1.88 m	2.11 m	1.54 m
7	3.40 m	*α* 2.97 d(11.4)	*α* 2.76 d(6.5)	*α* 3.37 d(15.3)
	3.57 m	*β* 3.56 dd(11.4, 4.4)	*β* 3.68 t(6.5)	*β* 3.68 m
9	2.79 m			
11	1.59 m, 2.20 m	*α* 1.58 m, *β* 2.08 t(10.0)	*α* 2.17 m, *β* 2.02 m	*α* 1.46 m, *β* 1.69 m
12	2.20 m, 1.99 m	*α* 1.69 m, *β* 2.19 t(10.4)	*α* 1.47 m, *β* 2.06 m	1.97 m
13	1.52 m, 2.60 m	*α* 1.62 m, *β* 2.80 m	*α* 1.91 m, *β* 1.95 m	*α* 1.62 m, *β* 2.51 t(13.0)
14	2.80 m	2.77 m	3.04 t(4.5)	3.28 m
15	3.38 m	2.63 m	2.60 dd(9.0, 5.0)	3.13 dd(18.4, 9.2)
16	1.78 m, 1.19 m	*α* 1.84 m, *β* 1.94 m	*α* 1.81 m, *β* 1.63 m	*α* 1.67 m, *β* 1.85 m
17	2.60 m, 2.22 m	*α* 1.74 m, *β* 1.81 m	*α* 1.72 m, *β* 2.15 m	*α* 2.42 m, *β* 1.75 m
18	1.96 m	2.60 m	*α* 1.42 m, *β* 1.81 m	1.77 m, 1.25 m
19	0.77 d(7.0)	1.10 d(1.5)		
20	0.86 d(7.0)	1.07 d(1.5)	0.96 t(7.3)	1.06 t(7.0)
21	3.86 dd(12.0, 3.0)	3.88 d(6.8)	4.29 d(10.3)	3.99 d(9.5)
		3.62 d(6.8)	3.64 d(10.3)	3.88 d(9.5)
23	3.56 s	3.66 s		3.72 s
24	3.82 d(14.0)	3.20 d(11.0)	3.16 d(13.6)	4.61 s
	3.86 d(14.0)	3.13 d(11.0)	2.73 d(13.6)	
25	3.09 s			
26				6.44 d(3.1)
27				6.38 br s
29				4.89 s

Table 2-22-36: ^{1}H NMR spectroscopic data of paxdaphnine A-type triterpenoid alkaloid **2-22-109**.

H	2-22-109 (CD_3OD)	2-22-109 ($CDCl_3$)	H	2-22-109 (CD_3OD)	2-22-109 ($CDCl_3$)
3	2.40 dt(8.4, 15.1)	*α* 1.67 m	15	2.61 dd(8.6, 17.4)	2.54 dd(18.0, 9.0)
	1.81 td(1.2, 11.0)	*β* 2.36 m			
4	1.59 m(ov)	*α* 1.50 m	16	*α* 1.58 m(ov)	*α* 1.57 m
	1.68 m(ov)	*β* 1.62 m		*β* 1.48 m	*β* 1.46 m
6	1.79 d(9.9)	1.68 m	17	*α* 1.52 dd(7.2, 12.6)	*α* 1.43 m
				β 1.66 m(ov)	*β* 1.69 m
7	*α* 2.52 d(14.9)	*α* 2.47 d(15.0)	18	1.36 dd(7.3, 13.2)	1.57 m
	β 3.75 dd(9.9, 14.9)	*β* 3.73 dd(15.0, 10.0)		1.65 m(ov)	1.23 m
11	*α* 2.10 m	1.91~2.02 m	20	0.97 t(7.2)	0.93 t(7.2)
	β 1.92 ddd(0.9, 7.5, 13.6)				
12	*α* 1.32 ddd(2.5, 7.1, 11.8)	*α* 1.24 m	21	3.97 d(9.6)	4.08 d(9.6)
		β 2.14 m		4.14 d(9.6)	3.96 d(9.6)
	β 2.20 m				
13	*α* 1.57 m(ov)	*α* 1.52 m	23	3.64 s	3.63 s
	β 2.32 t(12.9)	*β* 2.32 m			
14	3.24 ddd(7.1, 8.9, 12.6)	3.18 m	24	3.22 d(14.1)	3.26 d(14.1)
				3.36 d(14.1)	3.16 d(14.1)

Bibliography

[666] Morita H, Kobayashi J. Tetrahedron, 2002, 58: 6637.
[667] Bitar HE, Nguyen VH, Gramain A, et al. J Nat Prod, 2004, 67: 1094.
[668] Zhang CR, Yang SP, Yue JM. J Nat Prod, 2008, 71: 1663.
[669] Saito S, Yahata H, Kubota T, et al. Tetrahedron, 2008, 64: 1901.
[670] Di YT, Liu LL, Li CS, et al. Helv Chim Acta, 2008, 91: 838.
[671] Yang SP, Zhang H, Zhang CR, et al. J Nat Prod, 2006, 69: 79.
[672] Mu SZ, Wang JS, Yang XS, et al. J Nat Prod, 2008, 71: 564.
[673] Yahata H, Kubota T, Kobayashi J. J Nat Prod, 2009, 72: 148.
[674] Morita H, Yoshida N, Kobayashi J. Tetrahedron, 1999, 55: 12549.
[675] Yang SP, Yue JM. Helv Chim Acta, 2006, 89: 2783.
[676] Zhan ZJ, Zhang CR, Yue JM. Tetrahedron, 2005, 61: 11038.
[677] Mu SZ, Wang Y, He HP, et al. J Nat Prod, 2006, 69: 1065.
[678] Li CS, Di YT, Mu SZ, et al. J Nat Prod, 2008, 71: 1202.
[679] Jossang A, Bitar HE, Pham VC, et al. J Org Chem, 2003, 68: 300.
[680] Morita H, Yoshida N, Kobayashi J. Tetrahedron, 2000, 56: 2641.
[681] Di YT, He HP, Li CS, et al. J Nat Prod, 2006, 69: 1745.
[682] Takatsu H, Morita H, Shen YC, et al. Tetrahedron, 2004, 60: 6279.
[683] Saito S, Kubota T, Kobayashi J. Tetrahedron Lett, 2007, 48: 3809.
[684] Li ZY, Chen P, Xu HG, et al. Helv Chim Acta, 2007, 90: 1353.
[685] Matsuno Y, Okamoto M, Hirasawa Y, et al. J Nat Prod, 2007, 70: 1516.
[686] Kubota T, Matsuno Y, Morita H, et al. Tetrahedron, 2006, 62: 4743.
[687] Kong NC, He HP, Wang YH, et al. J Nat Prod, 2007, 70: 1348.
[688] Morita H, Takatsu H, Kobayashi J. Tetrahedron, 2003, 59: 3575.
[689] Di YT, He HP, Liu HY, et al. Tetrahedron Lett, 2006, 47: 5329.

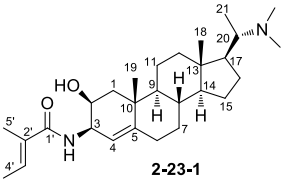
[690] Li L, He HP, Di YT, et al. Tetrahedron Lett, 2006, 47: 6259.
[691] Zhang Q, Di YT, Liu HY, et al. Helv Chim Acta, 2008, 91: 914.
[692] Gan LS, Wang XN, Fan CQ, et al. Helv Chim Acta, 2007, 90: 2395.
[693] Li L, He HP, Di YT, et al. Helv Chim Acta, 2006, 89: 1457.
[694] Chen X, Zhan ZJ, Yue JM. Helv Chim Acta, 2005, 88: 854.
[695] Kong NC, He HP, Wang YH, et al. Helv Chim Acta, 2007, 90: 972.
[696] Kobayashi J, Inaba Y, Shiro M, et al. J Am Chem Soc, 2001, 123: 11402.
[697] Zhang Y, He HP, Di YT, et al. Tetrahedron Lett, 2007, 48: 9104.
[698] Yang SP, Yue JM. Org Lett, 2004, 6: 1401.
[699] Tan CJ, Di YT, Wang YH, et al. Tetrahedron Lett, 2008, 49: 3376.
[700] Morita H, Yoshida N, Kobayashi J. J Org Chem, 2002, 67: 2278.
[701] Zhang H, Yang SP, Fan CQ, et al. J Nat Prod, 2006, 69: 553.
[702] Di YT, He HP, Lu Y, et al. J Nat Prod, 2006, 69: 1074.
[703] Mu SZ, Li CS, He HP, et al. J Nat Prod, 2007, 70: 1628.
[704] Morita H, Kobayashi J. Org Lett, 2003, 5: 2895.
[705] Li ZY, Chen P, Xu HG, et al. Org Lett, 2007, 9: 477.
[706] Yang SP, Yue JM. J Org Chem, 2003, 68: 7961.
[707] Di YT, He HP, Wang YS, et al. Org Lett, 2007, 9: 1355.
[708] Saito S, Kubota T, Fukushi E, et al. Tetrahedron Lett, 2007, 48: 1587.
[709] Saito S, Kubota T, Fukushi E, et al. Org Lett, 2007, 9: 1207.
[710] Fan CQ, Yin S, Xue JJ, et al. Tetrahedron, 2007, 63: 115.
[711] Li CS, He HP, Di YT, et al. Tetrahedron Lett, 2007, 48: 2737.

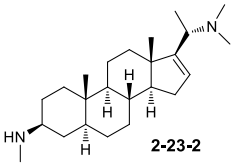
2.23 C_{21}-pregnane alkaloids

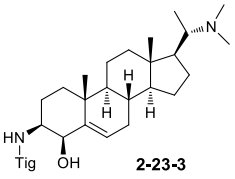
2.23.1 Saracodine-type C_{21}-pregnane alkaloids

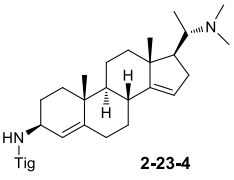
Table 2-23-1: Cos, MFs, and TSs of saracodine-type C_{21}-pregnane alkaloids **2-23-1**~**2-23-18**.

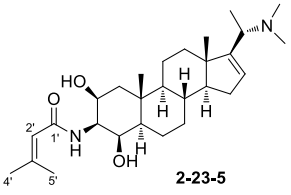
No.	Compounds	MFs	Test solvents	References
2-23-1	2-hydroxysalignarine E	$C_{28}H_{46}N_2O_2$	$CDCl_3$	[712]
2-23-2	5,6-dihydrosarconidine	$C_{24}H_{42}N_2$	$CDCl_3$	[712]
2-23-3	salignarine-F	$C_{28}H_{46}N_2O_2$	$CDCl_3$	[712]
2-23-4	salonine-C	$C_{28}H_{44}N_2O$	$CDCl_3$	[712]
2-23-5	(–)-hookerianamide A	$C_{28}H_{46}N_2O_3$	C_5D_5N	[713]
2-23-6	(+)-hookerianamide B	$C_{30}H_{50}N_2O_4$	$CDCl_3$	[713]
2-23-7	(–)-hookerianamide C	$C_{30}H_{50}N_2O_3$	$CDCl_3$	[713]
2-23-8	(–)-hookerianamine A	$C_{24}H_{42}N_2$	$CDCl_3$	[713]
2-23-9	(+)-phulchowkiamide A	$C_{27}H_{42}N_2O_2$	$CDCl_3$	[713]
2-23-10	salignarine A	$C_{30}H_{48}N_2O_5$	$CDCl_3$	[714]
2-23-11	salignarine B	$C_{28}H_{46}N_2O_2$	$CDCl_3$	[714]
2-23-12	salignarine C	$C_{28}H_{46}N_2O_2$	$CDCl_3$	[714]
2-23-13	salignarine D	$C_{28}H_{46}N_2O$	$CDCl_3$	[714]
2-23-14	salignarine E	$C_{28}H_{46}N_2O$	$CDCl_3$	[714]
2-23-15	hookerianamide J	$C_{28}H_{46}N_2O_2$	$CDCl_3$	[715]
2-23-16	hookerianamide K	$C_{25}H_{42}N_2$	$CDCl_3$	[715]
2-23-17	(20*S*,2′*Z*)-20-(*N*,*N*-dimethylamino)-3β-(2-methyl-2*Z*-butenamido)-pregn-5-en-4-one	$C_{28}H_{44}N_2O_2$	$CDCl_3$	[716]
2-23-18	(20*S*,2′*Z*)-20-(*N*,*N*-dimethylamino)-3β-(2-methyl-2*Z*-butenamido)-pregna-5,14-dien-4-one	$C_{28}H_{42}N_2O_2$	$CDCl_3$	[716]

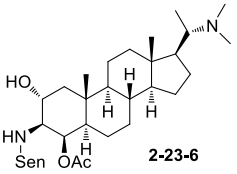


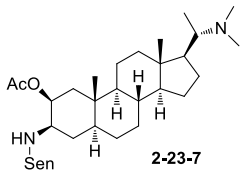


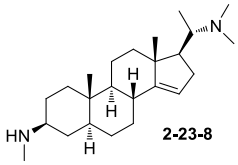


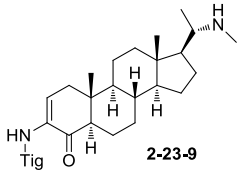


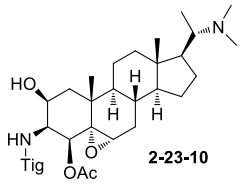


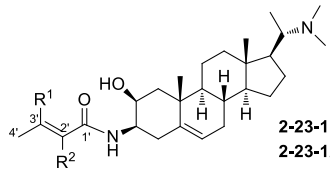


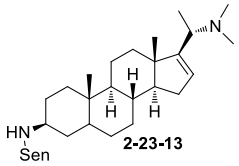


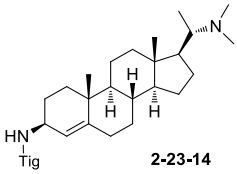


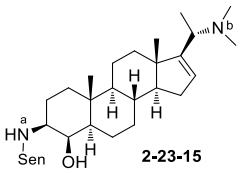


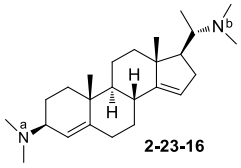


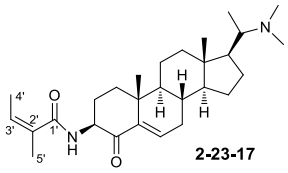












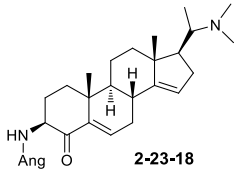


Table 2-23-2: ^{1}H NMR spectroscopic data of saracodine-type C_{21}-pregnane alkaloids **2-23-1**~**2-23-4**.

H	2-23-1	2-23-2	2-23-3	2-23-4
1	1.15 m, 1.85 m	1.90 m, 1.35 m	1.85 m, 1.23 m	1.69 m, 1.15 m
2	3.91 m ($W_{1/2}$=5.6)	1.80 m, 1.75 m	1.6 m, 1.21 m	2.6 m, 1.94 m
3	4.19 m	2.68 m	3.95 br s	4.15 m
4	5.51 br s	2.30 m, 2.60 m	3.99 br s	5.78 br s
5		1.65		
6	1.65 m, 2.11 m	1.32 m, 1.75 m	5.78 br s	1.49 m, 2.15 m
7	1.32 m, 1.84 m	2.00 m, 2.61 m	2.05 m, 2.5 m	1.88 m, 1.34 m
8	1.82	2.40	1.55	1.55
9	0.75	1.38	1.23	0.88
11	1.61 m, 1.42 m	1.79 m, 1.99 m	2.13 m, 1.41 m	2.12 m, 1.39 m
12	1.36 m, 1.84 m	1.58 m, 1.50 m	1.52 m, 1.20 m	1.35 m, 1.23 m
14	1.36	2.45	1.32	
15	2.01 m, 1.88 m	1.85 m, 2.06 m	1.7 m, 1.92 m	5.41 br s
16	1.59 m, 1.61 m	5.55 br s	1.63 m, 1.73 m	2.06 m, 1.65 m
17	1.25		1.2	1.1

Table 2-23-2 (continued)

H	2-23-1	2-23-2	2-23-3	2-23-4
18	0.84 s	0.84 s	0.82 s	0.86 s
19	1.27 s	0.87 s	1.04 s	1.06 s
20	2.80 m	2.68 q(6.6)	2.80 s	2.82 m
21	1.07 d(6.6)	1.13 d(6.5)	1.38 d(6.5)	1.73 d(6.5)
3-NMe		2.14 s		
20-NMe	2.20 s	2.23 s	2.23 s	2.91 s
3′	6.51 q(6.9)		6.34 q(6.9)	6.34 q(6.9)
4′	1.77 d(6.9)		1.73 d(6.9)	1.78 d(6.9)
5′	1.88 s		1.83 s	1.82 s

Table 2-23-3: ^{1}H NMR spectroscopic data of saracodine-type C_{21}-pregnane alkaloids **2-23-5**~**2-23-9**.

H	2-23-5	2-23-6	2-23-7	2-23-8	2-23-9
1	1.62, 1.55	1.21, 1.92	1.13, 1.84	1.38, 1.55	1.21, 1.95
2	4.73 br s($W_{1/2}$=7.2)	3.88 br s($W_{1/2}$=18.1)	5.11 br s ($W_{1/2}$=7.5)	1.40, 1.75	7.62 dd(6.7, 2.4)
3	5.11 m	4.03 m	4.01 m	2.66 m	
4	4.66 dd(4.3, 4.3)	4.09 dd(4.0, 3.9)	1.62, 1.70	1.35, 1.48	
5	1.79	1.49	1.39	1.58	2.29
6	2.42, 1.59	1.41, 1.66	1.15, 1.53	1.69, 1.87	1.38, 1.55
7	1.85, 1.21	1.65, 1.78	1.75, 1.86	1.21, 1.77	1.12, 1.75
8	1.50	1.38	1.38	1.78	1.35
9	0.88	1.42	1.08	1.67	1.11
11	2.39, 1.46	1.23, 1.81	1.01, 1.62	1.41, 1.44	1.32, 1.61
12	1.79, 1.32	1.82, 1.26	1.53, 1.78	1.30, 1.82	1.34, 1.92
14	1.25	1.44	1.35		1.02
15	1.60, 1.85	1.05, 1.66	1.61, 1.72	5.54 br s	1.71, 1.92
16	5.54 br s	1.18, 1.41	1.31, 1.49	1.67, 1.81	1.25, 1.83
17		1.13	1.20	2.00	1.18
18	0.88 s	0.63 s	0.61 s	0.77 s	0.65 s
19	1.42 s	1.20 s	0.94 s	0.79 s	0.85 s
20	2.90 q(6.2)	2.67 m	2.42 m	2.76 m	2.48 m
21	1.03 d(6.5)	1.72 d(6.7)	0.95 d(6.3)	1.02 d(6.5)	1.80 d(6.4)
NMe				2.33 s	2.63 s
NMe_2	2.12 s	2.26 s	2.21 s	2.15 s	
2′	6.13 s	5.31 s	5.45 s		
3′					6.47 q(6.2)
4′	2.27 s	1.85 s	2.08 s		1.75 d(6.9)
5′	1.58 s	1.75 s	1.78 s		1.86 s
2-OAc			2.07 s		
4-OAc		2.06 s			

Table 2-23-4: ^{1}H NMR spectroscopic data of saracodine-type C_{21}-pregnane alkaloids **2-23-10**~**2-23-14**.

H	2-23-10	2-23-11	2-23-12	2-23-13	2-23-14
1	1.30, 2.20	1.15, 1.85	1.20, 1.80	1.30, 1.70	1.40, 1.90
2	4.09	3.92	3.95	1.55, 2.01	1.35, 1.60
3	4.01	4.02	4.00 dd(6.0, 3.3)	4.07	4.12
4	5.32 br s	1.20, 2.01	1.85, 2.15	1.40, 1.80	5.93 d(6.5)
5				2.09	
6	3.76 t(5.5)	5.50 t(3.6)	5.56 t(3.0)	1.60, 1.75	1.45, 1.52
7	1.40, 1.55	1.75, 2.02	1.65, 1.70	1.30, 1.65	1.70, 1.83
8	2.15	1.65	1.75	2.20	1.66
9	2.10	0.72	0.75	1.79	0.80
11	1.55, 1.60	1.59, 1.65	1.35, 1.60	1.35, 1.55	1.45, 1.50
12	1.40, 1.70	1.70, 1.75	1.65, 1.70	1.10, 1.50	1.40, 1.43
14	1.90	1.35	1.30	1.30	1.35
15	1.45, 1.50	1.25, 1.35	1.20, 1.30	1.85, 2.00	1.59, 1.75
16	1.80, 1.95	1.81, 1.90	1.95, 2.05	5.54 br s	1.90, 1.25
17	1.45	1.25	1.75		1.25
18	0.90 s	0.80 s	0.81	0.80	0.80 s
19	1.29 s	1.04 s	1.04	0.80	0.81 s
20	2.83	2.80	2.85	2.79	3.09
21	1.20 d(6.5)	1.07 d(6.5)	1.10 d(6.5)	1.40 d(6.6)	1.13 d(6.5)
NMe_2	2.36 s	2.19 s	2.24 s	2.17 s	2.29 s
2′			5.58 s	5.48 br s	
3′	6.38 q(7.0)	6.33 q(6.7)			6.38 q(6.7)
4′	1.75 d(7.0)	1.70 d(6.7)	2.10 s	2.10 s	1.73 d(6.7)
5′	1.83 s	1.80 s	1.82 s	1.75 s	1.81 s
4-OAc	2.07 s				

Table 2-23-5: ^{1}H NMR spectroscopic data of saracodine-type C_{21}-pregnane alkaloids **2-23-15**~**2-23-18**.

H	2-23-15	2-23-16	2-23-17	2-23-18
1	1.75 m, 1.81 m	1.67 m, 1.55 m	0.95 m, 1.81 m	1.49 m, 1.69 m
2	1.43 m, 1.52 m	1.38 m, 1.75 m	1.28 m, 1.32 m	1.32 m, 1.34 m
3	4.27 m	2.82 ddd(12.2, 4.9, 4.2)	2.24 m	2.21 m
4	3.61 dd(3.9,3.9)	5.26 d(4.9)		
5	1.21 m			
6	1.31 m, 1.46 m	1.22 m, 1.49 m	7.45 dd(6.7, 2.7)	7.45 dd(6.6, 2.7)
7	1.59 m, 1.66 m	1.19 m, 1.24 m	2.35 m, 2.56 m	2.39 m, 2.54 m
8	1.65 m	1.36 m	1.38 m	1.43 m
9	1.35 m	1.35 m	1.12 m	1.18 m
11	1.76 m, 1.68 m	1.63 m, 1.07 m	1.37 m, 2.01 m	1.41 m, 2.01 m
12	1.65 m, 1.72 m	1.55 m, 1.08 m	1.82 m, 1.92 m	1.28 m, 1.31 m

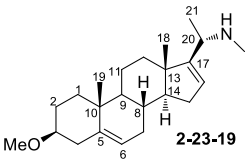
Table 2-23-5 (continued)

H	2-23-15	2-23-16	2-23-17	2-23-18
14	1.26 m		1.20 m	
15	1.28 m, 1.54 m	5.48 br s	1.49 m, 1.55 m	5.85 t(1.3)
16	5.50 br s	1.48 m, 1.87 m	1.59 m, 1.61 m	2.13 m, 2.28 m
17		1.72 m	1.72 m	1.42 m
18	0.81 s	0.60 s	0.77 s	0.89 s
19	0.85 s	0.78 s	0.90 s	0.92 s
20	2.80 d(6.8)	2.39 m	3.31 m	3.42 m
21	1.07 d(6.9)	0.83 d(6.4)	1.34 d(6.6)	1.38 d(6.5)
NMe_2	2.19 s(N_b Me_2)	2.19 s N_aMe_2, 2.14 s N_bMe_2	2.69 s	2.71 s
2′	5.66 s			
3′			6.50 q(5.5)	6.58 q(5.5)
4′	2.13 s		1.84 d(5.5)	1.85 q(5.5)
5′	1.83 s		1.88 s	1.87 s
NH	5.62 d(7.4, N_a-H)			

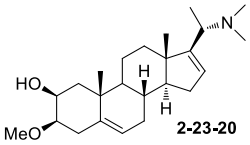
2.23.2 Pachysine-type C_{21}-pregnane alkaloids

Table 2-23-6: Cos, MFs, and TSs of pachysine-type C_{21}-pregnane alkaloids **2-23-19**~**2-23-21**.

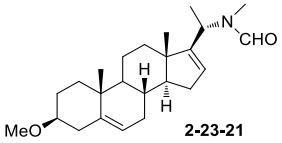
No.	Compounds	MFs	Test solvents	References
2-23-19	salignamine	$C_{23}H_{37}NO$	$CDCl_3$	[712]
2-23-20	2-hydroxysalignamine	$C_{24}H_{39}NO_2$	$CDCl_3$	[712]
2-23-21	*N*-[formyl(methyl)amino]salonine B	$C_{24}H_{37}NO_2$	$CDCl_3$	[712]



2-23-19



2-23-20



2-23-21

Table 2-23-7: ^{1}H NMR spectroscopic data of pachysine-type C_{21}-pregnane alkaloids **2-23-19**~**2-23-21**.

H	2-23-19	2-23-20	2-23-21
1	1.35 m, 2.20 m	1.15 m, 2.20 m	1.35 m, 1.90 m
2	1.80 m, 1.75 m	4.14 br s	1.75 m, 1.80 m
3	3.04 m	3.11 br s	3.03 m($W_{1/2}$ = 17.7)

Table 2-23-7 (continued)

H	2-23-19	2-23-20	2-23-21
4	2.11 m, 2.55 m	2.15 m, 2.55 m	2.14 m, 2.39 m
6	5.36 br s	5.39 br s	5.34 br s
7	2.00 m, 2.60 m	2.00 m, 2.60 m	2.30 m, 1.5 m
8	2.38	1.65	1.45
9	1.27	1.00	1.35
11	1.20 m, 1.60 m	1.20 m, 1.60 m	1.7 m, 1.99 m
12	1.40 m, 1.70 m	1.40 m, 1.70 m	1.53 m, 1.22 m
14	2.14	1.35	1.75
15	1.65 m, 2.10 m	1.70 m, 2.10 m	1.6 m, 1.20 m
16	5.55 br s	5.64 br s	5.78 br s
18	0.84 s	0.86 s	0.75 s
19	1.03 s	1.21 s	1.01 s
20	3.08 d(6.6)	2.90 q(6.6)	4.19 m
21	1.17 d(6.5)	1.14 d(6.5)	1.3 d(6.5)
20-NMe	2.33 s	2.28 s	2.64, 2.72
HCO			8.02, 8.09
3-OMe	3.34 s	3.38 s	3.33 s

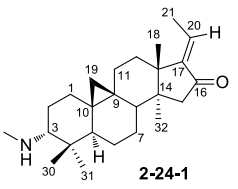
Bibliography

[712] Atta-ur-Rahman Z, Feroz F. Helv Chim Acta, 2004, 87: 439.
[713] Choudhary MI, Devkota KP, Nawaz SA, et al. Helv Chim Acta, 2004, 87: 1099.
[714] Atta-ur-Rahman, Choudhary MI, Khan MR, et al. J Nat Prod, 2000, 63: 1364.
[715] Devkota KP, Lenta BN, Wansi JD, et al. J Nat Prod, 2008, 71: 1481.
[716] Atta-ur-Rahman AS, Farooq A, et al. Phytochemistry, 1997, 46: 771.

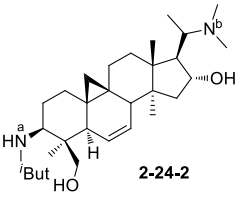
2.24 Cycloregnane alkaloids

Table 2-24-1: Cos, MFs, and TSs of cycloregnane alkaloids **2-24-1**~**2-24-16**.

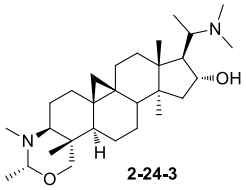
No.	Compounds	MFs	Test solvents	References
2-24-1	buxmicrophylline B	$C_{25}H_{39}NO$	$CDCl_3$	[717]
2-24-2	buxmicrophylline C	$C_{30}H_{50}N_2O_3$	$CDCl_3$	[717]
2-24-3	buxmicrophylline D	$C_{29}H_{50}N_2O_2$	$CDCl_3$	[717]
2-24-4	buxbodine A	$C_{26}H_{45}N$	$CDCl_3$	[718]
2-24-5	buxbodine B	$C_{26}H_{41}NO_2$	$CDCl_3$	[718]
2-24-6	(+)-papillotrienine	$C_{27}H_{44}N_2$	$CDCl_3$	[719]
2-24-7	(+)-N_b-demethylpapillotrienine	$C_{26}H_{42}N_2$	$CDCl_3$	[719]
2-24-8	(+)-N_b-demethylharappamine	$C_{26}H_{42}N_2O$	$CDCl_3$	[719]
2-24-9	papillamidine	$C_{27}H_{42}N_2O_2$	$CDCl_3$	[720]
2-24-10	(+)-papillozine C	$C_{27}H_{44}N_2O$	$CDCl_3$	[721]
2-24-11	(+)-benzoylbuxidienine	$C_{33}H_{48}N_2O_2$	$CDCl_3$	[722]
2-24-12	(+)-buxapapillinine	$C_{37}H_{52}N_2O_6$	$CDCl_3$	[722]
2-24-13	(+)-buxaquamarine	$C_{26}H_{39}NO_2$	$CDCl_3$	[722]
2-24-14	(+)-O^6-buxafurandiene	$C_{35}H_{48}N_2O_6$	$CDCl_3$	[722]
2-24-15	(+)-7-deoxy-O^6-buxafurandiene	$C_{35}H_{48}N_2O_5$	$CDCl_3$	[722]
2-24-16	(–)-2α,16α-diacetoxy-9β,11β-epoxybuxamidine	$C_{37}H_{52}N_2O_6$	$CDCl_3$	[721]



2-24-1



2-24-2



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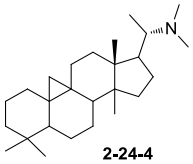
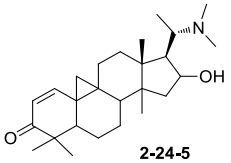
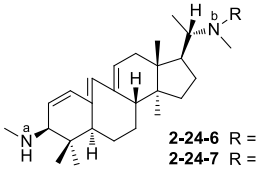


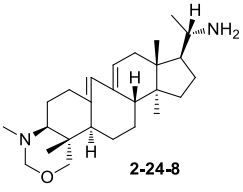
2-24-4



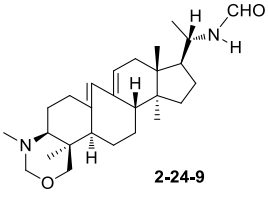
2-24-5



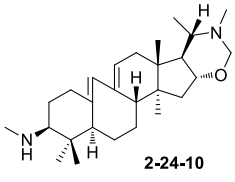
2-24-6 R = Me
2-24-7 R = H



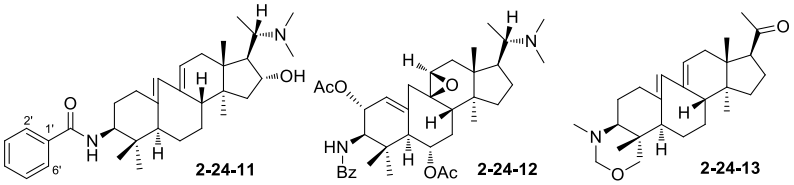
2-24-8



2-24-9



2-24-10



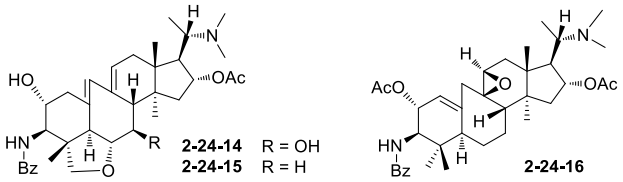


Table 2-24-2: ^{1}H NMR spectroscopic data of cycloregnane alkaloids **2-24-1**~**2-24-5**.

H	2-24-1	2-24-2	2-24-3	2-24-4	2-24-5
1	–	–	–	–	6.77 d(10)
2	–	–	–	–	5.95 d(10)
3	–	4.08 m	2.62 m	–	
6	–	5.35 dd(10.5, 3.2)	–	–	–
7	–	5.65 d(10.5)	–	–	–
16		4.03 sept(2.8, 7.2, 10.0)	4.02 sept(2.8, 7.2, 10.0)	–	4.41 m
17		2.61 m	–	–	–
18	1.38 s	0.99 s	–	0.91 s	0.89 s
19	*α* 0.32 d(4.2)	*α* 0.21 d(4.1)	*α* 0.31 d(4.0)	0.56	0.77
	β 0.57 d(4.2)	*β* 0.70 d(4.0)	*β* 0.58 d(4.0)	0.78 ABq(4.2)	1.33 ABq(4.4)
20	6.54 q(7.5)				2.98 m
21	1.81 s	0.85 d(6.5)	1.25 d(6.6)	0.82 d(6.4)	0.91 d(6.5)
28					
29					
30	1.23 s	3.47 dd(13.4)		1.08 s	1.15 s
		2.95 dd(13.4)			
31	0.91 s	0.57 s	3.74 d(10.8)	1.03 s	1.11 s
			3.30 d(10.8)		
32	0.92 s	0.89 s		0.97 s	0.97 s
N_aMe	2.21 s				
N_bMe_2				2.18 s, 2.18 s	
NCH			4.27 q(5.4)		
$N_aCH\underline{Me}$			1.28 d(5.6)		
$CH(Me)_2$		1.16 d(7.0)			

Table 2-24-3: ^{1}H NMR spectroscopic data of cycloregnane alkaloids **2-24-6**~**2-24-11**.

H	2-24-6	2-24-7	2-24-8	2-24-9	2-24-10	2-24-11
1	5.58 d(9.9)	5.58 d(10)	–	–	–	
2	6.25 dd(1.9, 10.2)	6.33 dd(2.2, 10)	–	–	–	
3	3.42 m	3.56 m	–	–	–	4.36 m
11	5.71 m	5.84 m	5.54 s	5.53 br s	5.52 br s	5.50 br s
16	–	–	–	–	4.45 m	4.10 m
19	6.11 s	6.23 s	5.97 s	5.96 s	5.92 s	5.95 s
20	–	–	–	2.98 m	–	
21	1.32 d(7.3)	1.33 d(6.5)	1.20 d(8.0)	0.96 d(6.4)	1.19 d(6.5)	1.09 d(6.7)
31			*α* 3.25 d(10.6)	*α* 3.24 d(10.6)		
			β 3.82 d(10.6)	*β* 3.81 d(10.6)		
Me	0.71 s, 0.80 s	0.77 s, 0.79 s	0.70 s, 0.74 s	0.67 s, 0.78 s	0.66 s, 0.61 s	0.76 s, 0.78 s
	0.89 s, 0.93 s	0.84 s, 1.17 s	1.03 s	1.03 s	1.01 s, 1.17 s	0.93 s, 1.02 s
N_aH						5.80 d(9.8)
N_aMe	2.13 s	2.21 s	2.12 s	2.11 s	2.23 br s	
N_aCH_2			*α* 3.59 d(7.6)	*α* 3.58 d(7.5)		
			β 4.44 d(7.6)	*β* 4.43 d(7.5)		
N_bMe	2.80 s	2.21 s			2.37 br s	2.49 br s
N_bCH_2					3.87 d(10.8)	
					4.10 d(10.8)	
Ar-H						7.37~7.65 m

Table 2-24-4: ^{1}H NMR spectroscopic data of cycloregnane alkaloids **2-24-12**~**2-24-16**.

H	2-24-12	2-24-13	2-24-14	2-24-15	2-24-16
1	5.55 d(5.4)	–	2.60 m, 1.38 m	2.59, 1.37	5.40
2	5.54 dd(9.9, 5.4)	–	4.08 m	4.09 m	5.37 d(9.8)
3	4.34 ddd	–	4.01 dd(12.0, 9.5)	4.00 dd(12.0, 9.5)	4.05 dd(9.8, 9.6)
	(9.9, 9.6, 0.76)				
5	–	–	2.00 m	1.99 d(12.5)	2.50
6	–	–	3.96 dd(12.8, 11.0)	3.67 m	1.72, 1.45
7	–	–	3.54 dd(11.0, 9.9)	1.89 m, 1.25 m	1.54, 1.29
8	–	–	2.13 m	2.02 m	1.80
11	2.92 dd(10.2, 2.9)	5.60 br s	5.33 br s	5.36 br s	3.02 dd(10.6, 3.9)
12	–	–	2.08 m, 1.94 m	2.10 m, 1.96 m	1.68, 1.34
15	–	–	1.55 m, 1.35 m	1.68 m, 1.37 m	1.77, 1.49
16	4.79 m	–	5.00 m	4.99 m	4.68
17	–	–	1.88 m	1.87 m	2.49

Table 2-24-4 (continued)

H	2-24-12	2-24-13	2-24-14	2-24-15	2-24-16
18			0.92 s	0.91 s	0.74
19		6.00 s	5.85 br s	5.82 s	2.78
20			2.00 m	1.99 m	2.39
21	1.05 d(6.5)		1.03 d(6.5)	1.02 d(6.6)	0.98 d(6.7)
30			0.89 s	0.89 s	0.78
31		α 3.27 d(10.4)	3.86 d(11.0)	3.87 d(10.9)	0.85
		β 3.85 d(10.4)	4.06 d(11.0)	4.04 d(10.9)	
32			0.91 s	0.90 s	1.05
Me	0.84 s, 0.86 s	0.68 s, 0.75 s			
	0.87 s, 0.95 s	1.06 s			
N_aMe		2.13 s			
NH	6.13 d(9.6)				
N_bMe	2.19 br s		2.27 br s	2.27 br s	2.27
N_bCH$_2$		α 3.31 d(9.6)			
		β 4.45 d(9.6)			
2′/6′	7.44~7.77 m		7.26 m	7.24 m	7.63(7.4, 1.3)
3′/5′	7.44~7.77 m		7.49 m	7.48 m	7.30(7.4)
4′	7.44~7.77 m		7.69 m	7.70 m	7.45(7.8, 1.3)
OAc	1.93 s, 2.04 s	2.00 s	2.10 s	2.07 s	1.82, 1.93

Bibliography

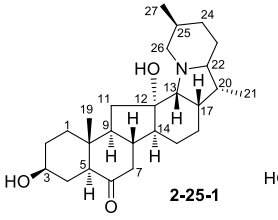
[717] Du J, Chiu MH, Nie RL. J Asian Nat Prod Res, 1999, 1: 239.
[718] Qiu MH, Yang WS, Nie RL. Acta Bot Yunnanica, 2001, 23: 357.
[719] Atta-ur-Rahman, Asif E, Ali SS, et al. J Nat Prod, 1992, 55: 1063.
[720] Rahman AU, Alam M, Ahmed G, et al. Heterocycles, 1988, 27: 89.
[721] Rahman AU, Naz S, Ata A, et al. Heterocycles, 1998, 48: 519.
[722] Babar ZU, Ata A, Meshkatalsadat MH. Steroids, 2006, 71: 1045.

2.25 Cholestane and stigmastane alkaloids

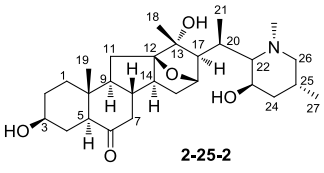
2.25.1 Cholestane-type alkaloids

Table 2-25-1: Cos, MFs, and TSs of cholestane alkaloids **2-25-1**~**2-25-10**.

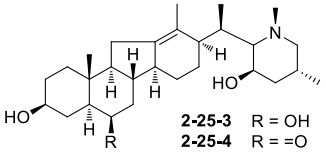
No.	Compounds	MFs	Test solvents	References
2-25-1	pingbeinone	$C_{26}H_{41}NO_3$	$CDCl_3$	[723]
2-25-2	puqienine F	$C_{28}H_{45}NO_5$	C_5D_5N	[724]
2-25-3	puqienine A	$C_{28}H_{47}NO_3$	DMSO-d_6	[725]
2-25-4	puqienine B	$C_{28}H_{45}NO_3$	DMSO-d_6	[725]
2-25-5	*N*-demethylpuqietinone	$C_{27}H_{45}NO_2$	$CDCl_3$	[725]
2-25-6	20-isoveratramine	$C_{27}H_{39}NO_2$	C_5D_5N	[726]
2-25-7	veratramine	$C_{27}H_{39}NO_2$	C_5D_5N	[726]
2-25-8	verapatuline	$C_{29}H_{41}NO_5$	$CDCl_3$	[726]
2-25-9	jervine	$C_{27}H_{39}NO_3$	$CDCl_3$	[726]
2-25-10	puqietinonoside	$C_{34}H_{57}NO_7$	C_5D_5N	[725]



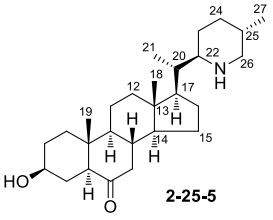
2-25-1



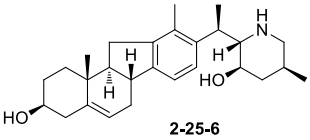
2-25-2



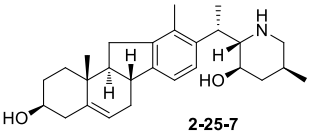
2-25-3 R = OH
2-25-4 R = =O



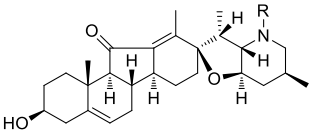
2-25-5



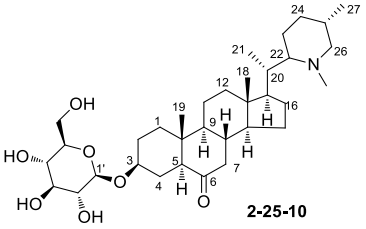
2-25-6



2-25-7



2-25-8 R = COOMe
2-25-9 R = H



2-25-10

Table 2-25-2: ^{1}H NMR spectroscopic data of cholestane alkaloids **2-25-1**~**2-25-5**.

H	2-25-1	2-25-2	2-25-3	2-25-4	2-25-5
1	1.42 t(9.3), 1.60	*α* 1.08 m, *β* 1.39 m	1.04 m, 1.37 m	1.32 m, 1.51 m	1.24 m, 1.78 m
2	1.60, 1.65	*α* 1.90 m, *β* 1.57 m	1.32 m, 1.65 m	1.21 m, 1.65 m	1.40 m, 1.84 m
3	3.60	3.70 br m	3.41 br m	3.37 br m	3.56 br m
4	1.45, 1.92	*α* 2.22 m, *β* 1.74 m	1.49 m, 1.60 m	1.31 m, 2.18 m	1.48 m, 1.90 m
5	2.25 dd(2.5, 12.7)	2.08 dd(12.1, 2.6)	1.03 m	2.29 dd(11.5, 2.6)	2.19 dd(12.5, 2.8)
6			3.67 br m		
7	2.19 t(13.0)	*α* 2.13 m	1.89 m	2.24 m	1.94 t(12.7)
	2.51 dd(4.7, 13.0)	*β* 2.49 m	1.18 m	2.30 m	2.32 dd(12.7, 4.5)
8	1.45	1.92 m(ov)	1.35 m	1.29 m	1.79 m
9	1.96	1.94 m(ov)	1.10 m	1.82 m	1.22 m
11	1.34 t(12.3), 1.9	*α* 2.10 m, *β* 1.69 m	1.93 m, 2.21 m	1.94 m, 2.33 m	1.37 m, 1.62 m
12					1.21 m, 2.03 m
13	2.89 d(9.8)				
14	1.71	2.89 ddd(16.4, 10.8, 5.6)	1.62 m	1.85 m	1.22 m
15	0.80 ddd(2, 10, 24)	*α* 1.80 m	0.92 m	0.89 m	1.36 m
	1.82	*β* 1.52 m	1.67 m	1.66 m	1.78 m
16	1.20 ddd(2, 12.9, 24.1)	4.77 ddd(20.1, 12.3, 7.5)	1.29 m	1.29 m	1.09 m
	1.43		1.71 m	1.69 m	1.54 m
17	2.40	2.32 dd(7.5, 2.9)	2.30 m	2.30 m	1.26 m
18		1.59 s	1.59 s	1.58 s	0.68 s
19	0.70 s	0.71 s	0.90 s	0.60 s	0.75 s
20	2.06 tq(8.5, 9.8)	2.51 m	2.29 m	2.27 m	1.51 m
21	0.86 d(7.0)	1.30 d(6.9)	0.70 d(6.8)	0.66 d(6.8)	0.90 d(6.8)
22	2.67 dt(5.0, 8.5)	1.87 dd(11.4, 3.2)	2.78 dd(10.4, 2.6)	2.76 dd(13.0, 2.8)	2.45 m
23	1.63, 1.69	4.09 br m	3.80 br m	3.77 br m	1.12 m, 1.48 m
24	1.13, 1.56	*α* 1.05 m, *β* 1.93 m	1.17 m, 1.86 m	1.14 m, 1.84 m	0.97 m, 1.80 m
25	1.67	2.24 br m	2.14 br m	2.12 br m	1.43 m
26	2.40 dd(9.1, 11.5)	*α* 1.82 m	2.21 m	2.18 m	2.27 m
	2.79 dd(2.9, 11.5)	*β* 2.76 ddd(11.8, 6.7, 3.5)	2.29 m	2.26 m	
27	0.92 d(6.8)	0.67 d(6.6)	0.75 d(6.6)	0.73 d(6.6)	0.81 d(6.6)
NMe		2.32 s	2.47 s	2.45 s	

Table 2-25-3: ^{1}H NMR spectroscopic data of cholestane alkaloids **2-25-6**~**2-25-10**.

H	2-25-6	2-25-7	2-25-8	2-25-9	2-25-10
1	1.25 td(13.5, 3.5)	1.31 td(13.5, 4)	1.19 td(13.5, 3.5)	1.19 td(14, 4)	0.85 m
	1.76 dt(13.5, 3)	1.80 br d(13.5)	2.57 dt(13.5, 3.5)	2.58 dt(14, 3.5)	1.37 m
2	1.88 m	1.88 dddd(13.5, 12, 11, 3.5)	1.56 tdd(13.5, 11, 3.5)	1.55 m	1.36 m
	2.11 m			1.86 br d(13)	1.85 m
		2.12 br d(12)	1.85 m		
3	3.82 tt(11, 4.5)	3.84 tt(11, 4)	3.52(11, 4.5)	3.52 tt(11.0, 4.5)	3.77 br m
4	2.59 dd(13, 11)	2.60 br dd(13, 11)	2.18 br dd(12.5, 11)	2.17 m	1.52 m
	2.69 ddd(13, 4.5, 1.5)	2.71 br dd(13, 4)	2.35 ddd(12.5, 4.5, 2.0)	2.36 m	2.18 m
5					1.85 dd(12.3, 2.8)
6	5.44 br d(4)	5.40 br d(4)	5.38 br d(5)	5.38 br d(5)	
7	1.91 m	2.02 br dd(15, 12)	1.92 m	1.89 m	1.82 t(12.7)
	2.41 ddd(14, 11, 4)		2.35 m	2.36 m	2.16 dd(12.7, 4.6)
		2.56 m			
8	2.45 ddd(12, 11.0, 5)	2.95 td(12, 5)	1.60 m	1.60 m	1.50 m
9	1.64 td(12, 7.5)	1.80 td(12, 7)	1.67 d(13)	1.66 d(12)	0.95 m
11	2.11 dd(14, 12)	2.50 dd(14.5, 12)			0.98 m,
	2.60 dd(14, 7.5)	2.78 dd(14.5, 7)			1.27 m
12					0.94 m, 1.75 m
14			1.97 m	1.95 m	1.90 m
15	6.87 d(7.5)	7.10 d(7.5)	1.35 br dt(13, 10)	1.37 br t(12.5)	1.26 m
			1.95 m	1.94 m	
16	7.68 d(7.5)	7.66 d(7.5)	1.60 m, 1.92 m	1.52 m, 1.92 m	1.26 m
17					1.02 m
18	2.66 s	2.57 s	2.24 d(1.5)	2.17 s	0.41 s
19	1.07 s	1.11 s	1.02 s	1.01 s	0.43 s
20	4.34 q(7)	4.06 qd(7, 4)	2.95 q(7)	2.52 dq(9, 7)	1.71 m
21	1.86 d(7)	1.64 d(7)	0.95 d(7)	0.96 d(7)	0.79 d(6.6)
22	3.47 dd(10, 7)	2.84 dd(9, 4)	3.21 dd(11, 7)	2.72 t(9)	2.55 m
23	4.51 ddd(11, 10, 4)	3.56 ddd(11, 9, 4.5)	3.61 td(11, 4.5)	3.30 ddd(11.5, 9, 4)	1.02 m
					1.45 m
24	1.55 td(12, 11)	1.35 q(11)	1.11 ddd(12, 11, 9)	1.21 q(11.5)	0.62 m, 1.53 m
	2.39 br dd(12, 4)	2.25 br d(11)	2.21 ddd(12, 6, 4.5)	2.19 dt(11.5, 4)	
25	2.53 m	1.48 m	1.87 m	1.61 m	1.44 m
26	2.59 t(11)	2.20 t(11.5)	2.84 dd(13, 8.5)	2.33 t(12)	1.43 m
	3.42 br d(11)	2.99 br d(11.5)	3.68 dd(13, 4.5)	3.08 dd(12, 4)	

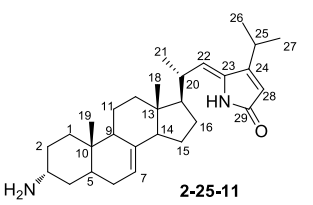
Table 2-25-3 (continued)

H	2-25-6	2-25-7	2-25-8	2-25-9	2-25-10
27	0.75 d(6)	0.74 d(6.5)	1.03 d(6.5)	0.95 d(6.5)	0.58 d(5.2)
1′					4.84 d(7.7)
2′					3.82 dd(8.7, 7.7)
3′					4.05 t(8.7)
4′					4.00 t(8.7)
5′					3.78 m
6′					4.19 dd(11.6, 5.5)
					4.40 dd(11.6, 1.8)
NMe					1.92 s
COOMe			3.72 s		

2.25.2 Stigmastane-type alkaloids

Table 2-25-4: Cos, MFs, and TSs of stigmastane-type alkaloids **2-25-11**~**2-25-19**.

No.	Compounds	MFs	Test solvents	References
2-25-11	plakinamine G	$C_{29}H_{44}N_2O$	CD_3OD	[727]
2-25-12	plakinamine H	$C_{31}H_{48}N_2O$	CD_3OD	[727]
2-25-13	4α-hydroxydemethylplakinamine B	$C_{30}H_{48}N_2O$	CD_3OD	[727]
2-25-14	tetrahydroplakinamine A	$C_{29}H_{50}N_2$	CD_3OD	[727]
2-25-15	plakinamine E	$C_{31}H_{50}N_2O_2$	CD_3OD	[728]
2-25-16	plakinamine F	$C_{31}H_{48}N_2O$	CD_3OD	[728]
2-25-17	plakinamine I	$C_{31}H_{50}N_2$	CD_3OD	[729]
2-25-18	plakinamine K	$C_{32}H_{52}N_2O_2$	CD_3OD	[729]
2-25-19	dihydroplakinamine K	$C_{32}H_{54}N_2O_2$	CD_3OD	[729]



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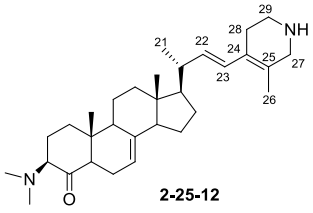

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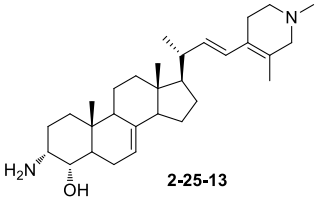

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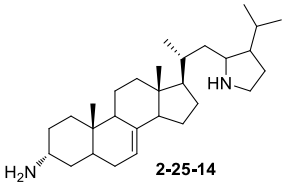

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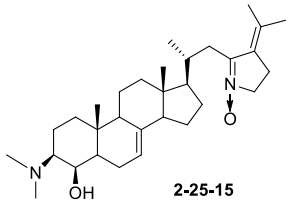

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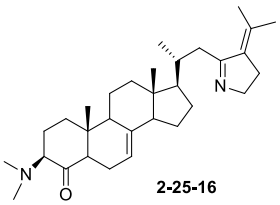

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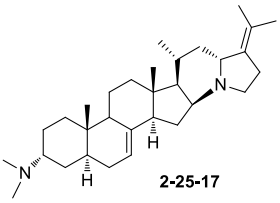

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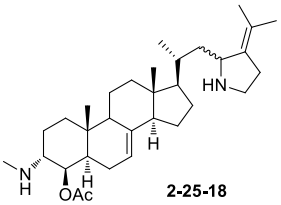

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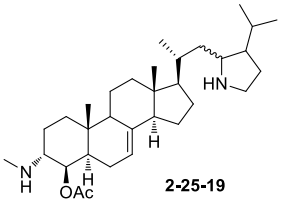


2-25-19

Table 2-25-5: ^{1}H NMR spectroscopic data of stigmastane-type alkaloids **2-25-11**~**2-25-15**.

H	2-25-11	2-25-12	2-25-13	2-25-14	2-25-15
1	1.63	2.09	1.59	1.63	1.98 m
	1.38	1.71	1.34	1.39	1.20 ddd(13.2, 13.2, 2.9)
2	1.73	1.87	1.85	1.73	1.96 m
	1.30		1.76	1.30	1.86 br d(13.7)
3	3.25 br s	–	3.20 br s	3.25 br s	3.09 ddd(12.2, 3.4, 3.4)
4	1.66, 1.45		3.60 dd(11.0, 4.0)	1.66, 1.45	4.09 br s
5	1.65	2.38 dd(11.2, 4.3)	1.48	1.65	1.36 m
6	1.73	2.17	2.27	1.69	2.33 br dd(14.8, 14.2)
		1.89		1.73	1.79 br d(14.8)
7	5.22	5.22 br s	5.25 br d(3.7)	5.22 br s	5.30 br d(3.9)
9	1.83	2.06	1.85	1.83	1.71 m
11	1.65, 1.51	1.71, 1.56	1.66, 1.52	1.65, 1.51	1.56 m, 1.52 m
12	2.08 br d(11.4)	1.75	2.05 br d(12.8)	2.09 br d(12.1)	2.07 br ddd(14.3, 3.0, 3.0)
	1.35	2.07	1.30	1.29	1.32 m
14	1.90	1.89	1.88	1.88	1.91 m
15	1.57, 1.45	1.54, 1.46	1.47	1.57, 1.48	1.62 m, 1.54 m
16	1.87, 1.58	1.72	1.70	1.76, 1.58	2.01 m, 1.43 m
17	1.50	1.36	1.34	1.31	1.40 m
18	0.66 s	0.59 s	0.60 s	0.61 s	0.63 s
19	0.83 s	0.72 s	0.87 s	0.84 s	1.07 s
20	2.68 m	2.18 m	2.17 m	1.52	2.01 m
21	1.13 d(6.6)	1.07 d(6.6)	1.07 d(6.6)	1.06 d(6.6)	0.98 d(6.8)
22	5.36 d(10.7)	5.49 dd(15.5, 9.0)	5.48 dd(15.8, 8.8)	1.81	3.14 br d(14.7)
				1.29	2.61 dd(14.7, 11.2)

Table 2-25-5 (continued)

H	2-25-11	2-25-12	2-25-13	2-25-14	2-25-15
23		6.40 d(15.5)	6.39 d(15.8)	3.09 m	
24				1.78	
25	2.85 m			1.73	
26	1.20 d	3.31	2.91 s	0.99 d(5.9)	2.27 s
27	1.22 d	1.73 s	1.73 s	0.93 d(5.9)	2.04 s
28	5.82 s	2.22	2.28 br t	1.96	2.94 br s
29		2.99 t(5.9)	2.58 t(5.9)	2.97 m	3.95 t(6.8)
$N(Me)_2$		2.35 s	2.33 s		2.90 s

Table 2-25-6: ^{1}H NMR spectroscopic data of stigmastane-type alkaloids **2-25-16**~**2-25-19**.

H	2-25-16	2-25-17	2-25-18	2-25-19
1	2.05 m, 1.70 m	1.74 m, 1.30 td(14.5, 3)	1.58 m, 1.39 m	1.81 m, 1.32 m
2	2.16 m, 1.75 br dd(13.7, 3.4)	2.12 dt(16, 3), 1.88 m	1.88 m	2.11 m, 1.87 m
3	3.25 dd(11.2, 5.9)	3.31 m	2.60 br d(3)	3.31 m
4		1.94 m	4.96 br s	5.09 br s
5	2.37 dd(11.7, 4.4)	1.60 m	1.82 m	1.76 m
6	2.16 m, 1.88 m	1.88 m, 1.80 m	2.01 m, 1.64 m	2.07 m, 1.81 m
7	5.23 br d(3.9)	5.32 br d(3.5)	5.22 br d(4)	5.27 br d(4)
9	2.02 m	1.98 m	1.82 m	1.83 m
11	1.68 m, 1.48 m	1.62 m	1.58 m	1.58 m
12	2.09 ddd(13.7, 3.4, 3.4), 1.32 m	2.02 m, 1.40 m	2.20 m, 1.28 m	2.13 m, 1.35 m
14	1.91 m	1.98 m	1.85 m	1.86 m
15	1.57 m, 1.48 m	2.38 ddd(8, 6, 3), 1.61 m	1.54 m	1.53 m
16	2.00 m, 1.42 m	3.77 dt(10, 8)	1.90 m	1.96 m
17	1.32 m	1.62 m	1.22 m	1.30 m
18	0.59 s	0.78 s	0.62 s	0.63 s
19	0.71 s	0.93 s	1.05 s	1.09 s
20	1.94 m	1.68 m	1.56 m	1.49 m
21	0.89 d(6.5)	1.12 d(6)	1.07 d(6.5)	1.07 d(6)
22	2.82 br d(14.7)	2.23 ddd(12.5, 7.5, 2)	1.54 m	1.78 m
	2.29 dd(14.7, 11.2)	1.40 m	1.04 m	1.46 m
23		4.49 br t(9)	3.84 br d(11)	3.31 m
24				1.83 m
25				1.81 m
26	2.04 s	1.73 br s	1.66 br s	0.93 d(6.5)
27	1.83 s	1.75 br s	1.66 br s	1.01 d(6.5)
28	2.59 m	2.73 m	2.36 m	2.07 m
29	3.70 t(6.6)	3.56 dt(12, 4)	3.01 dt(11, 7.5)	3.27 m
		3.35 m	2.91 m	
$N(Me)_2$	2.34 s	2.92 s, 2.92 s	2.39 s	2.80 s
4-OAc			2.06 s	2.12 s

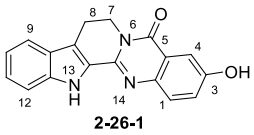
Bibliography

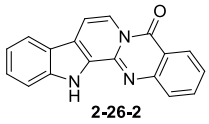
[723] Kitamura Y, Nishizawa M, Kaneko K. Tetrahedron Lett, 1989, 30: 4981.
[724] Li HJ, Jiang Y, Li P, et al. Chem Pharm Bull, 2006, 54: 722.
[725] Jiang Y, Li HJ, Li P, et al. J Nat Prod, 2005, 68: 264.
[726] Tezuka Y, Kikuchi T, Zhao WJ, et al. J Nat Prod, 1998, 61: 1078.
[727] Borbone N, Marino SD, Iorizzi M, et al. J Nat Prod, 2002, 65: 1206.
[728] Lee H-S, Seo YW, Rho JR, et al. J Nat Prod, 2001, 64: 1474.
[729] Ridley CP, Faulkner DJ. J Nat Prod, 2003, 66: 1536.

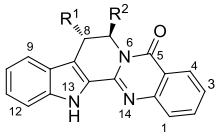
2.26 Quinazoline alkaloids

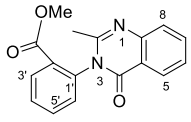
Table 2-26-1: Cos, MFs, and TSs of quinazoline alkaloids **2-26-1**~**2-26-20**.

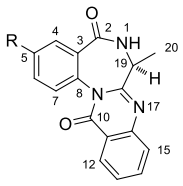
No.	Compounds	MFs	Test solvents	References
2-26-1	3-hydoxyrutaecarpine	$C_{18}H_{13}N_3O_2$	$CDCl_3$	[730]
2-26-2	7,8-dehydrorutaecarpine	$C_{18}H_{11}N_3O$	$CDCl_3$	[731]
2-26-3	7-hydroxyrutaecarpine	$C_{18}H_{13}N_3O_2$	$CDCl_3$	[732]
2-26-4	7,8-dihydroxyrutaecarpine	$C_{18}H_{13}N_3O_3$	CD_3OD	[732]
2-26-5	2-(2-methyl-4-oxo-4*H*-quinazoline-3-yl)-benzoic acid methyl ester	$C_{17}H_{14}N_2O_3$	CD_3OD	[733]
2-26-6	circumdatin C	$C_{17}H_{13}N_3O_3$	CD_3OD	[734]
2-26-7	circumdatin F	$C_{17}H_{13}N_3O_2$	CD_3OD	[734]
2-26-8	circumdatin D	$C_{21}H_{19}N_3O_5$	DMSO-d_6	[734]
2-26-9	circumdatin E	$C_{20}H_{17}N_3O_4$	DMSO-d_6	[734]
2-26-10	sclerotigenin	$C_{16}H_{11}N_3O_2$	$CDCl_3$	[735]
2-26-11	circumdatin G	$C_{17}H_{13}N_3O_3$	CD_3OD	[736]
2-26-12	glycozolone A	$C_{16}H_{16}N_2O$	$CDCl_3$	[737]
2-26-13	glycozolone B	$C_{17}H_{18}N_2O_2$	$CDCl_3$	[737]
2-26-14	samoquasine A	$C_{12}H_8N_2O$	$CDCl_3$	[738]
2-26-15	luotonin F	$C_{18}H_{11}N_3O_2$	$CDCl_3$	[739]
2-26-16	luotonin E	$C_{19}H_{13}N_3O_2$	$CDCl_3$	[739]
2-26-17	luotonin A	$C_{18}H_{11}N_3O$	$CDCl_3$	[740]
2-26-18	luotonin B	$C_{18}H_{11}N_3O_2$	$CDCl_3$	[740]
2-26-19	isaindigotone	$C_{20}H_{18}N_2O_4$	DMSO-d_6	[741]
2-26-20	hydrachine A	$C_{17}H_{19}N_3O_3$	$CDCl_3$	[742]

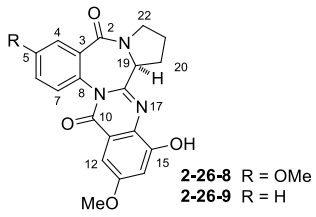


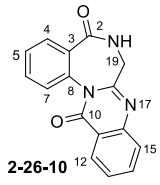


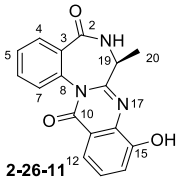


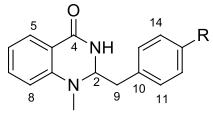


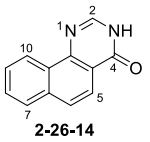


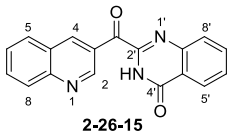


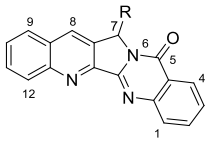












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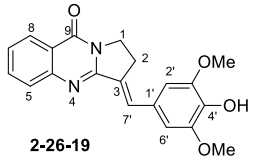
2-26-17 R = H
2-26-18 R = OH



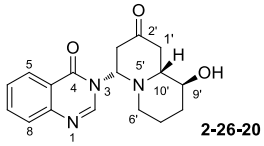


Table 2-26-2: ^{1}H NMR spectroscopic data of quinazoline alkaloids **2-26-1**~**2-26-5**.

H	2-26-1	2-26-2	2-26-3	2-26-4	2-26-5
1	7.56 d(8.8)	7.81 dd(7, 1)	7.58 d(8)	7.74 dd(8, 1)	
2	7.27 dd(8.8, 2.7)	7.82 ddd(8, 7, 1)	7.69 ddd(8, 7.5, 1.5)	7.80 ddd(8.5, 7.5, 1.5)	2.19 s(Me)
3	10.1 br s(OH)	7.46 ddd(8, 8, 1)	7.35 dd(8, 7.5)	7.48 ddd(8, 7.5, 1)	
4	7.48 d(2.7)	8.50 dd(8, 1)	8.21 dd(8, 1.5)	8.24 dd(8, 1.5)	
5					8.16 ddd(0.6, 1.5, 8.1)
6					7.53 ddd(0.9, 7.3, 8.4)
7	4.41 t(6.8)	8.75 d(8)	6.88 dd(5.5, 1.5)	6.70 d(2)	7.86 ddd1.5, 7.2, 8.4)
8	3.14 t(6.8)	7.55 d(8)	ax 3.42 dd(17, 5.5) eq 3.59 dd(17, 1.5)	5.24 d(2)	7.70 br d(7.5)
9	7.59 d(7.4)	8.02 dd(8, 1)	7.60 d(7.5)	7.76 dd(8, 1)	
10	7.05 t(7.4)	7.33 ddd(8, 7, 1)	7.15 ddd(8, 7, 1)	7.16 ddd(8.5, 7.5, 1)	
11	7.22 t(7.4)	7.48 ddd(8, 7, 1)	7.31 ddd(8.5, 7, 1)	7.29 ddd(8.5, 7.5, 1)	
12	7.46 d(7.4)	7.52 dd(8, 1)	7.42 d(8.5)	7.51 dd(8, 1)	
2′					3.69 s(COOMe)
3′					8.22 dd(1.5, 7.8)
4′					7.70 ddd(1.2, 7.8, 7.8)
5′					7.83 ddd(1.5, 7.8, 7.8)
6′					7.49 dd(1.2, 7.8)
NH	11.7 s	10.15 br s			

Table 2-26-3: ^{1}H NMR spectroscopic data of quinazoline alkaloids **2-26-6**~**2-26-10**.

H	2-26-6	2-26-7	2-26-8	2-26-9	2-26-10
1					7.19 br t(6)
4	7.22 d(3.0)	7.92 m(ov)	7.27 d(2.9)	7.81 dd(7.4, 1.9)	7.95 br d(7.8)
5		7.65 m①		7.56(ov)	7.53 m
6	7.05 dd(9, 3)	7.65 m①	7.20 dd(8.9, 2.9)	7.63(ov)	7.62 m(ov)
7	7.43 d(9.0)	7.65 m①	7.49 d(8.9)	7.58(ov)	7.61 m(ov)
12	8.24 ddd(8.0, 1.0, 0.5)	8.31 dd(8.0, 1.6)	6.98 d(2.4)	7.07 d(2.5)	8.29 dd(8.0, 1.4)
13	7.56 ddd(8.0, 7.0, 1.0)	7.73 ddd(8.7, 7.0, 1.7)			7.52 m
14	7.84 ddd(8, 8, 2)	7.92 m(ov)	6.87 d(2.6)	6.87 br s	7.78 ddd(8.1, 8.1, 1.4)
15	7.76 ddd(8.0, 1.0, 0.5)	7.82 d(7.7)			7.66 br d(8.1)
19	4.45 q(7.0)	4.48 q(6.6)	4.60 d(6.4)	4.60 d(6.9)	4.29 dd(15, 5.7) 4.21 dd(15, 6.9)
20	1.66 d(7.0)	1.71 d(6.6)	3.20 m, 2.06 m	3.21 m, 2.04 m	
21			2.11 m, 1.93 m	2.11 m, 1.93 m	
22			3.57 m, 3.43 m	3.58 m, 3.44 m	
23			3.80 s	3.81 s	
24					

① Average value of unresolved signals from three protons.

Table 2-26-4: ^{1}H NMR spectroscopic data of quinazoline alkaloids **2-26-11**~**2-26-15**.

H	2-26-11	2-26-12	2-26-13	2-26-14	2-26-15
2		4.69 m	4.63 m	9.60 s	9.92 d(2.1)
4	7.80 dd(1.5, 8.0)				9.52 d(2.1)
5	7.60 (ov)	7.96 dd(1.5, 7.7)	7.96 br d(7.7)	7.55 d(7.3)	8.05 br d(8.0)
6	7.68 ddd(1.5, 7.0, 8.0)	6.86 br t(7.7)	6.85 br t(7.7)	7.30 d(7.3)	7.72 t(8.0)
7	7.62 (ov)	7.43 br t(7.7)	7.42 br t(7.7)	8.40 dd(1.0, 8.3)	7.93 t(8.0)
8		6.64 br d(7.7)	6.64 br d(7.7)	7.71 dt(1.0, 8.3)	8.23 br d(8.0)
9		3.00 dd(4.4, 13.2) 2.92 dd(8.8, 13.2)	2.95 dd(4.4, 13.2) 2.85 dd(8.8, 13.2)	7.87 dt(1.0, 8.3)	
10				8.12 dd(1.0, 8.3)	
11		7.15 d(7.7)	7.06 d(8.1)		
12	7.59 (ov)	7.32 t(7.7)	6.85 d(8.1)		
13	7.37 dd(8.0, 8.0)	7.25 t(7.7)	3.79 s(OMe)		
14	7.24 dd(1.5, 8.0)	7.32 t(7.7)	6.85 d(8.1)		
15		7.15 d(7.7)	7.06 d(8.1)		
19	4.41 q(6.5)				

Table 2-26-4 (continued)

H	2-26-11	2-26-12	2-26-13	2-26-14	2-26-15
20	1.72 d(6.5)				
5′					8.43 dd(1.5, 8.0)
6′					7.70 dt(1.5, 8.0)
7′					7.90 ddd(1.5, 8.0, 8.6)
8′					7.98 dd(1.5, 8.6)
NMe		2.95 s	2.96 s		
NH		6.07 br	5.92 br		10.21 br s

Table 2-26-5: ^{1}H NMR spectroscopic data of quinazoline alkaloids **2-26-16**~**2-26-20**.

H	2-26-16	2-26-17	2-26-18	2-26-19	2-26-20
1	8.09 dd(1.5, 8.0)	8.11 dd(1.5, 8.0)	8.11 dd(1.5, 8.0)	4.20 t(7.1)	
2	7.85 dt(1.5, 8.0)	7.86 dt(1.5, 8.0)	7.87 dt(1.5, 8.0)	3.30 m	7.88 s
3	7.59 dt(1.5, 8.0)	7.56 dt(1.5, 8.0)	7.60 dt(1.5, 8.0)		
4	8.43 dd(1.5, 8.0)	8.42 dd(1.5, 8.0)	8.44 dd(1.5, 8.0)		
5				7.70 d(7.8)	8.28 dd(8.0, 1.6)
6				7.81 t(7.8)	7.50 ddd(8.0, 7.2, 1.6)
7	6.95 s 3.60 s(OMe)	5.40 br s	7.14 s	7.47 t(7.8)	7.80 ddd(8.4, 7.2, 1.6)
8	8.52 s	8.45 s	8.58 s	8.13 d(7.8)	7.71 dd(8.4, 1.6)
9	8.00 dd(1.5, 8.0)	7.93 dd(1.5, 8.5)	8.00 dd(1.5, 8.0)		
10	7.72 dt(1.5, 8.0)	7.67 dt(1.5, 8.5)	7.73 dt(1.5, 8.0)		
11	7.88 dt(1.5, 8.0)	7.84 dt(1.5, 8.5)	7.89 dt(1.5, 8.0)		
12	8.48 dd(1.5, 8.0)	8.46 dd(1.5, 8.5)	8.48 dd(1.5, 8.0)		
1′					ax 2.50 dd(15.2, 11.2) eq 3.18 dd(15.2, 3.2)
2′				6.95 s	
3′				3.85 s(OMe)	ax 2.84 dd(11.2, 10.4) eq 3.34 dd(10.4, 6.4)
4′					5.70 dd(11.2, 6.4)
5′				3.85 s(OMe)	
6′				6.95 s	ax 2.93 dd(13.0, 4.0) eq 2.18 dt(13.0, 4.0)
7′				7.74 br s	ax 1.80 m, eq 1.74 m
8′					ax 1.29 m, eq 2.07 m
9′					3.46 ddd(11.0, 9.0, 5.0)
10′					2.28 ddd(11.2, 9.0, 3.2)

Bibliography

[730] Li XC, Dunbar DC, ElSohly HN, et al. Phytochemistry, 2001, 58: 627.
[731] Ikuta A, Nakamura T, Urabe H. Phytochemistry, 1998, 48: 285.
[732] Ikuta A, Urabe H, Nakamura T. J Nat Prod, 1998, 61: 1012.
[733] Shim SH, Kim JS, Son KH, et al. J Nat Prod, 2006, 69: 400.
[734] Rahbæk L, Breinholt J. J Nat Prod, 1999, 62: 904.
[735] Joshi BK, Gloer JB, Wicklow DT, et al. J Nat Prod, 1999, 62: 650.
[736] Dai JR, Carté BK, Sidebottom PJ, et al. J Nat Prod, 2001, 64: 125.
[737] Ito C, Kondo Y, Ruangrungsi N, et al. Chem Pharm Bull, 1999, 47: 1491.
[738] Morita H, Sato Y, Chan KL, et al. J Nat Prod, 2000, 63: 1707.
[739] Ma ZZ, Hano Y, Nomura T, et al. Heterocycles, 1999, 51: 1883.
[740] Ma ZZ, Hano Y, Nomura T, et al. Heterocycles, 1997, 46: 541.
[741] Wu XY, Qin GW, Cheung KK, et al. Tetrahedron, 1997, 53: 13323.
[742] Patnam R, Chang FR, Chen CY, et al. J Nat Prod, 2001, 64: 948.

Index of compound names

Index of molecular formula of compounds