Isolation and Structure Elucidation of a Novel Alkaloid, Incartine, a Supposed Biosynthetic Intermediate, from Flowers of *Lycoris incarnata*

Masaru KIHARA,*[,a] Lai XU,[a] Keiji KONISHI,[a] Kiyoshi KIDA,[a] Yoshimitsu NAGAO,[a] Shigeru KOBAYASHI,[b] and Tetsuro SHINGU[c]

Faculty of Pharmaceutical Sciences, The University of Tokushima,[a] Sho-machi, Tokushima 770, Japan, Faculty of Home Economics, Shikoku University,[b] Ohjin-cho, Tokushima 771–11, Japan, and School of Pharmacy, Kobe Gakuin University,[c] Ikawadani-cho, Nishi-ku, Kobe 651–21, Japan. Received July 26, 1993; accepted September 8, 1993

A novel alkaloid incartine (1), a supposed biosynthetic intermediate from galanthine (2) to narcissidine (3), was isolated from flowers of *Lycoris incarnata* (Amaryllidaceae) together with the known alkaloids galanthine (2), ungiminorine (4), ungiminorine *N*-oxide (5), galanthamide (6), galanthamine *N*-oxide (7), lycoramine (8), sanguinine (9), lycorine (10), and *O*-demethyllycoramine (11). 1-Palmitoyl-2-linoleoylphosphatidylethanolamine (12) and 1-palmitoyl-2-linoleoylphosphatidylmethanol sodium salt (13) were also identified in the flower.

Keywords incartine; Amaryllidaceae alkaloid; biosynthetic intermediate; *Lycoris incarnata*; flower; phospholipid

The Amaryllidaceae alkaloids have been studied extensively because of the variety of their structures and biological activities and also from the biosynthetic viewpoint.[1)] However, flowers of Amaryllidaceae plants have not attracted much attention from phytochemists.[2)] In a previous paper,[3)] we reported the isolation of a new alkaloid, hippeastrine *N*-oxide, together with known alkaloids from flowers of *Lycoris radiata* HERB. Recently, we have isolated[4)] a novel alkaloid, incartine (**1**), which was supposed to be a biosynthetic intermediate[5)] from galanthine (**2**) to narcissidine (**3**), from flowers of *Lycoris incarnata*. This paper describes in detail the isolation and the structural elucidation of incartine (**1**), together with nine known alkaloids, galanthine (**2**), ungiminorine (**4**), ungiminorine *N*-oxide (**5**), galanthamine (**6**), galanthamine *N*-oxide (**7**), lycoramine (**8**), sanguinine (**9**), lycorine (**10**), and *O*-demethyllycoramine (**11**), from fresh flowers of this plant. 1-Palmitoyl-2-linoleoylphosphatidylethanolamine (**12**) and 1-palmitoyl-2-linoleoylphosphatidylmethanol sodium salt (**13**) were also isolated.

Crude extract of fresh flowers of *Lycoris incarnata* obtained by the modified method of Ghosal *et al.*[2)] was subjected to column and preparative thin layer chromatographies (PTLC), as described in Experimental to give compounds **1**, **2**, **4**—**12** and **13**.

The new compound, incartine, $C_{18}H_{23}NO_5$, was isolated as colorless prisms, mp 183—185 °C. The infrared (IR) spectrum showed hydroxy group absorption at 3433 cm^{-1}, but no absorption due to a carbonyl group. The proton nuclear magnetic resonance (^{1}H-NMR)

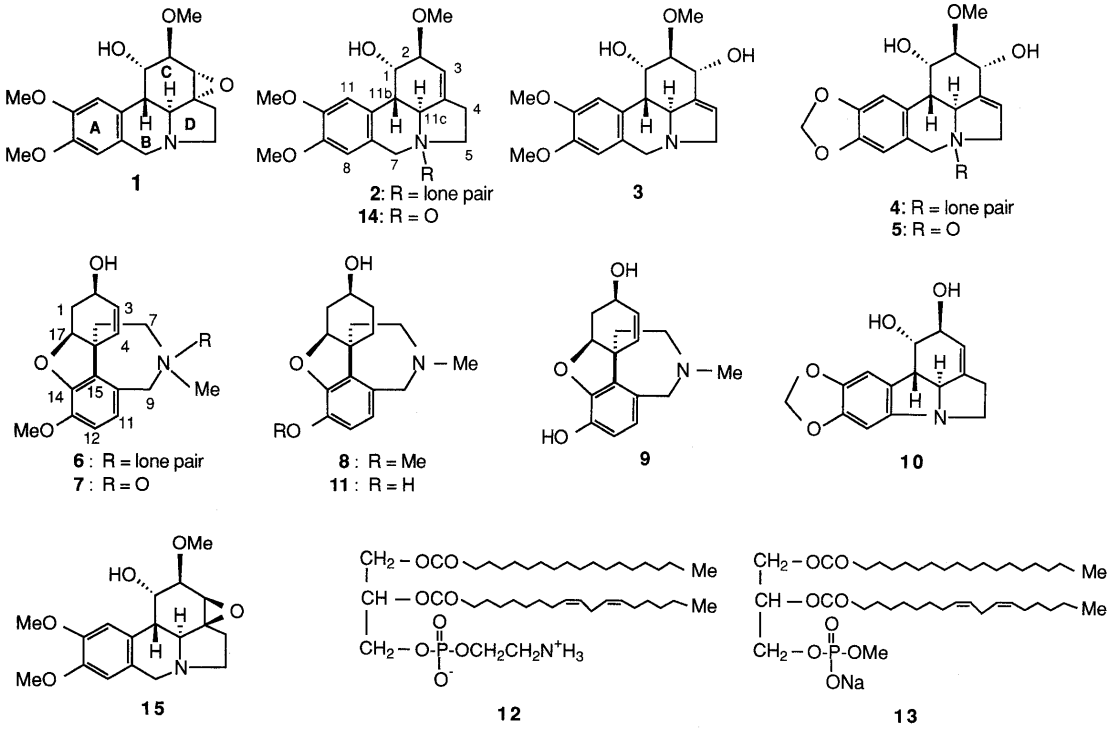


Chart 1

spectrum revealed the presence of two *para*-oriented aromatic protons (δ 6.97 and 6.95), two aromatic methoxy groups (δ 3.92 and 3.90), one aliphatic methoxy group (δ 3.57), and benzyl protons (δ 4.71 and 4.31, each doublet, $J=13.0$ Hz), but no *N*-methyl signal. These data suggested that the compound has a lycorine-type skeleton. This suggestion was supported by a two-dimensional ^1H–^1H shift correlation spectroscopy (2D-COSY) experiment, which indicated the presence of the sequence H_{11c}-H_{11b}-H_1-H_2-H_3 (see also below). These findings and the molecular formula ($C_{18}H_{23}NO_5$) indicate that the structure of incartine should be very similar to that of narcissidine (**3**),[6] galanthine *N*-oxide (**14**),[7] or the 3,3a-epoxy derivative of galanthine (**2**). Since the ^1H-NMR spectrum of incartine did not show an olefinic proton signal, the compound was considered to be an α- or β-3,3a-epoxy derivative (**1** or **15**) of galanthine (**2**).

In order to elucidate the structure and the stereochemistry of incartine, a decoupling experiment and the nuclear magnetic double resonance (NMDR) analysis were carried out. The completely assigned chemical shifts, the coupling patterns (see Experimental), and the nuclear Overhauser effect (NOE) enhancements (Fig. 1) suggested the relative configuration of the 3,3a-epoxy ring of incartine to be α. The conformation of α-3,3a-epoxygalanthine (**1**) inspected from a Dreiding model was compared with that of the 3α,3aα-glycol derivative (**16**) reported by Toda *et al.*[8] (see Fig. 2). It was reported[8] that the C-ring of **16** took a distorted boat conformation with *trans* diaxial orientation of H-2 and H-3 ($J=8.3$ Hz). On the contrary, a small coupling ($J=1.2$ Hz) of the corresponding hydrogens in incartine suggested the dihedral angle between them to be *ca.* 90°. A long-range coupling ($J=0.8$ Hz) between H-1 and H-3 was observed in this case. These observations show that the C-ring takes a distorted chair conformation in the α-3,3a-epoxy compound. The β-orientation of the epoxy ring, such as in **15**, would give a different coupling pattern. From these findings, incartine was concluded to be galanthine α-3,3a-epoxide (**1**).

Fuganti *et al.*[5] suggested that galanthine (**2**) is probably transformed to narcissidine (**3**) *via* the α-,3,3a-epoxide (**1**), since they found that galanthine (**2**) was converted to narcissidine (**3**) in *Sempre avanti* daffodil with loss of *pro*-S hydrogen from C-4 of the lycorane skeleton. Toda *et al.*[8] gave chemical support to this elimination step. However, the proposed intermediate, the α-epoxide (**1**), has never been isolated or synthesized. Therefore, this paper is the first to report isolation of the proposed epoxy intermediate.

Compound **5**, $[\alpha]_D$ −58.9° (EtOH), was isolated as a pale yellow oil. The mass (MS) spectrum showed the molecular formula $C_{17}H_{19}NO_6$, suggesting the presence of one more oxygen atom than in ungiminorine (**4**). The ^1H-NMR spectrum of **5** showed a similarity to that of **4** except for deshielding of the protons at C-5, C-7 and C-11c. This alkaloid was identical with ungiminorine *N*-oxide (**5**) isolated from *Pancratium maritimum* (Amaryllidaceae).[9]

Compounds **2**, **4**, **6**—**10** and **11** were identified as galanthine, ungiminorine, galanthamine, galanthamine *N*-oxide, lycoramine, sanguinine, lycorine, and *O*-demethyllycoramine, respectively, by direct comparisons of their physical and spectral data and thin layer chromatographic (TLC) behavior with those of the authentic samples.

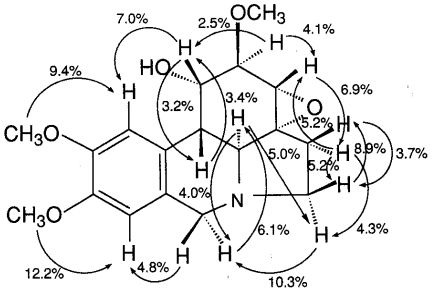
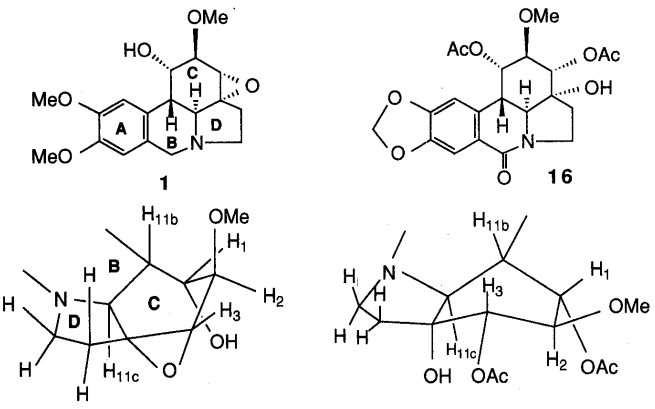


Fig. 1. NOE Enhancements for Incartine (**1**) in $CDCl_3$



chemical shifts (ppm)

	H_1	H_2	H_3	H_{11b}	H_{11c}
1:	4.55(br s)	3.91(br s)	3.44(br s)	2.60(dd)	3.52(d)
16:	5.72(dd)	3.61(dd)	5.22(d)	3.24(dd)	3.97(d)

coupling constants (Hz)

	$J_{1\text{-}2}$	$J_{2\text{-}3}$	$J_{1\text{-}3}$	$J_{1\text{-}11b}$	$J_{11b\text{-}11c}$
1:	2.9	1.2	0.8	3.0	13.4
16:	2.0	8.3	—	3.0	14.0

Fig. 2. Analysis of ^1H-NMR Spectra of **1** (at 400 MHz) and **16** (at 100 MHz, Ref. 8) in $CDCl_3$

Compounds **12** and **13** gave a blue-violet color with molybdenum blue reagent.[10] Compound **12** showed a pale orange color with Dragendorff's reagent but compound **13** did not. The IR spectra of **12** and **13** revealed ester absorptions at 1741 and 1742 cm^{-1}, and phosphate absorptions at 1045 and 1050 cm^{-1}, respectively. ^1H-NMR spectra of **12** and **13** indicated the presence of palmitate and linoleate groups[3] in both compounds (see Experimental). The EI-MS spectra of **12** and **13** showed similar fragmentation patterns. The intensities (80 and 82%) of the fragments of *m/z* 313 generated by cleavage of a linoleate group from the fragments (*m/z* 575)[3] of **12** and **13** were higher than those (65 and 31%) of *m/z* 337 generated by cleavage of a palmitate group, respectively. These facts suggest that **12** and **13** are 1-palmitoyl-2-linoleoylphosphatidyl derivatives.[3,11] In addition, the ^1H-NMR spectrum of **12** showed the presence of an aminoethyl group and the FAB-MS revealed the molecular ion peak of *m/z* 716 (M+1) for $C_{39}H_{74}NO_8P$. From these data, compound **12** was concluded to be 1-palmitoyl-2-

linoleoylphosphatidylethanolamine.[12] The FAB-MS of **13** showed the molecular ion peak of *m/z* 709 (M+1) for $C_{38}H_{70}NaO_8P$ and the ^{1}H-NMR spectrum indicated the presence of a methoxy group. These findings suggested that compound **13** is 1-palmitoyl-2-linoleoylphosphatidylmethanol sodium salt. Compound **13** may be an artifact derived from a corresponding phosphatidylcholine or a phosphatidylethanolamine during the extraction of the flower with MeOH–$CHCl_3$.

Experimental

All melting points are given as uncorrected values. The spectrophotometers used were a Perkin–Elmer 1720 infrared Fourier-transform spectrophotometer for IR spectra, a JEOL JMS-D 300 for MS, a Union PM-201 for optical rotations, and JEOL JNM-FX 200, JEOL JNM-GSX 400 and Bruker AM-400 spectrometers for ^{1}H-NMR spectra with tetramethylsilane as an internal standard. The plates used for PTLC were coated with silica gel (Kieselgel PF_{254}, Merck) and aluminum oxide (PF_{254}, Merck). The following solvent systems were used: 1) $CHCl_3$–MeOH (5:1); 2) $CHCl_3$–MeOH–H_2O (60:35:10); 3) $CHCl_3$–AcOEt–MeOH–H_2O (70:30:10:2); 4) $CHCl_3$–MeOH–NH_4OH–H_2O (70:26:2:2); 5) $CHCl_3$–MeOH (10:1). UV light, I_2 vapor, Dragendorff's reagent and molybdenum blue reagent[10] were used for location of compounds.

Extraction Following the modified method of Ghosal *et al.*,[2] fresh flowers (5.3 kg) of *Lycoris incarnata* collected in our Faculty plot were ground in 8.74 l of $CHCl_3$–MeOH (2:1) in a mixer. The extract was warmed at 60 °C for 1 h, then 0.1 M EDTA (87 ml) was added to retard the phospholipase activity and the mixture was kept at room temperature overnight, then filtered to give two layers. The $CHCl_3$ and MeOH–H_2O layers were concentrated *in vacuo* to afford sticky extracts, 18.5 and 98 g, respectively.

Treatment of the $CHCl_3$ Extract The $CHCl_3$ extract was subjected to column chromatography using HCl-washed Florisil (25 × 5.5 cm). Elution was carried out successively with benzene (1.85 l), $CHCl_3$ (6 l), $CHCl_3$–MeOH (95:5, 2.85 l, fraction (fr.) I, 1.627 g), $CHCl_3$–MeOH (9:1, 1.75 l, fr. II, 488 mg), $CHCl_3$–MeOH (3:1, 3.45 l, fr. III, 305 mg), and $CHCl_3$–MeOH (1:1, 3.49 l, fr. IV, 360 mg).

Fraction I (1.627 g) was subjected to column chromatography on SiO_2. Elution was carried out successively with $CHCl_3$–MeOH (10:1), $CHCl_3$–MeOH (5:1), and MeOH. The MeOH fraction gave an oil, which was subjected to PTLC (SiO_2, solvent 2) to afford incartine (**1**) (*Rf* 0.53—0.62, 14 mg).

Fraction II (488 mg) was subjected to PTLC (SiO_2, solvent 1) to afford an oil (*Rf* 0.28—0.36, 45.4 mg). This crude material was further purified by PTLC (SiO_2, solvent 4) to give 1-palmitoyl-2-linoleoylphosphatidylethanolamine (**12**) (*Rf* 0.68—0.74, 7.7 mg).

Fraction III (305 mg) was subjected to PTLC (SiO_2, solvent 1) to give two fractions (*Rf* 0.38—0.41, fr. III-A, 17.2 mg; *Rf* 0.13—0.23, fr. III-B, 52.9 mg). Purification of fr. III-A and fr. III-B by PTLC (Al_2O_3, solvent 3 and 1) gave ungiminorine (**4**) (*Rf* 0.65—0.75, 7.2 mg) and ungiminorine *N*-oxide (**5**) (*Rf* 0.61—0.65, 2.9 mg), respectively.

Fraction IV (360 mg) was subjected to PTLC (SiO_2, solvent 4) to afford two fractions (*Rf* 0.80—0.92, fr. IV-A, 33 mg, *Rf* 0.45—0.55, fr. IV-B, 139.4 mg). Fraction IV-A was subjected to PTLC (SiO_2, solvent 1) to give galanthamine (**6**) (*Rf* 0.19—0.27, 5 mg) and lycoramine (**8**), (*Rf* 0.37—0.44, 8.5 mg). Fraction IV-B was subjected to PTLC (SiO_2, solvent 4) to afford an oil (*Rf* 0.59—0.69, 75.1 mg), which was further separated by PTLC (Al_2O_3, solvent I) to give galanthamine *N*-oxide (**7**) (*Rf* 0.76—0.81, 1.6 mg) and 1-palmitoyl-2-linoleoylphosphatidylmethanol sodium salt (**13**) (*Rf* 0.02—0.08, 47 mg).

Treatment of the MeOH–H_2O Extract The MeOH–H_2O extract (98 g) was successively triturated with hot hexane, benzene, $CHCl_3$, and $CHCl_3$–MeOH (1:1) (fr. V, 6.454 g). The insoluble material was an oil (fr. VI, 55 g), which was soluble in MeOH.

Fraction V (6.454 g) was subjected to flash chromatography on SiO_2 with solvent 4 to give two fractions (550 ml, fr. V-A, 165.8 mg; 500 ml, fr. V-B, 119.8 mg). Fraction V-A was subjected to PTLC (SiO_2, solvent 2) to give three fractions (*Rf* 0.55—0.63, fr. V-A-1, 34.7 mg; *Rf* 0.66—0.74, fr. V-A-2, 76 mg; *Rf* 0.86—0.89, fr. V-A-3, 2.1 mg of galanthine (**2**)). Fraction V-A-1 was purified by PTLC (Al_2O_3, solvent 3) to give sanguinine (**9**) (*Rf* 0.50—0.59, 3.9 mg). Fraction V-A-2 was triturated with MeOH to afford lycorine (**10**) (53.3 mg). Fraction V-B was subjected to PTLC (SiO_2, solvent 2) to give an oil (21.3 mg), which was further purified by PTLC (Al_2O_3, solvent 1) to afford *O*-demethyllycoramine (**11**) (*Rf* 0.13—0.44, 1.9 mg).

Fraction VI (55 g) was subjected to flash chromatography on SiO_2 with solvent 4. The solid obtained from the first fraction (1000 ml) was washed with MeOH to afford lycorine (**10**) (114 mg). The MeOH solution gave an oil (208 mg), which was subjected to PTLC (SiO_2, solvent 2) to give an amorphous solid (68.6 mg). This was further purified by PTLC (Al_2O_3, solvent 5) to afford ungiminorine (**4**) (*Rf* 0.38—0.44, 2.6 mg) and lycoramine (**8**) (*Rf* 0.78—0.82, 5.6 mg).

Incartine (1) Colorless prisms (from MeOH), mp 183—185 °C. IR (KBr): 3433 (OH), 1615, 1516, 1456, 1326, 1290, 1085 cm^{-1}. High MS *m/z* [M^+]: Calcd for $C_{18}H_{23}NO_5$: 333.1573. Found: 333.1538. EI-MS *m/z* (%): 333 (M^+, 50), 332 (100), 296 (25), 295 (22), 294 (20), 266 (17), 259 (74), 258 (65), 244 (30), 242 (23). ^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 6.97 (1H, s, H-8), 6.95 (1H, s, H-11), 4.71 (1H, d, $J_{7\beta\text{-}7\alpha}$=13.0 Hz, H-7$\beta$), 4.55 (1H, br s, $J_{1\text{-}2}$=2.9 Hz, $J_{1\text{-}11b}$=3.0 Hz, $J_{1\text{-}3}$=0.8 Hz, H-1), 4.31 (1H, d, $J_{7\alpha\text{-}7\beta}$=13.0 Hz, H-7$\alpha$), 4.08 (1H, dd, $J_{5\beta\text{-}5\alpha}$=10.8 Hz, $J_{5\beta\text{-}4\beta}$=7.2 Hz, H-5β), 3.92 (3H, s, OCH_3-10), 3.90 (3H, s, OCH_3-9), 3.91 (1H, br s, $J_{2\text{-}1}$=2.9 Hz, $J_{2\text{-}3}$=1.2 Hz, H-2), 3.76 (1H, ddd, $J_{5\alpha\text{-}5\beta}$=10.8 Hz, $J_{5\alpha\text{-}4\beta}$=14.2 Hz, $J_{5\alpha\text{-}4\alpha}$=5.3 Hz, H-5$\alpha$), 3.57 (3H, s, OCH_3-2), 3.52 (1H, d, $J_{11c\text{-}11b}$=13.4 Hz, H-11c), 3.44 (1H, br s, $J_{3\text{-}2}$=1.2 Hz, $J_{3\text{-}1}$=0.8 Hz, H-3), 3.38 (1H, ddd, $J_{4\beta\text{-}4\alpha}$=13.1 Hz, $J_{4\beta\text{-}5\alpha}$=14.2 Hz, $J_{4\beta\text{-}5\beta}$=7.2 Hz, H-4β), 2.60 (1H, dd, $J_{11b\text{-}11c}$=13.4 Hz, $J_{11b\text{-}1}$=3.0 Hz, H-11b), 2.04 (1H, dd, $J_{4\alpha\text{-}4\beta}$=13.1 Hz, $J_{4\alpha\text{-}5\alpha}$=5.3 Hz, H-4$\alpha$).

Galanthine (2) Pale yellow oil, $[\alpha]_D^{29}$ −65.9° (*c*=0.046, $CHCl_3$) (lit.[13] $[\alpha]_D^{22}$ −94.7° (*c*=0.686, $CHCl_3$)). High MS *m/z* [M^+]: Calcd for $C_{18}H_{23}NO_4$: 317.1628. Found: 317.1603. ^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 6.68 (1H, s, H-11), 6.63 (1H, s, H-8), 5.61 (1H, br s, H-3), 4.67 (1H, s, H-1), 4.17 and 3.50 (each 1H, d, *J*=14 Hz, CH_2-7), 3.90 (3H, s, OCH_3-9), 3.87 (3H, s, OCH_3-10), 3.53 (3H, s, OCH_3-2).

Ungiminorine (4) Colorless prisms (from acetone), mp 207—208 °C (lit.[14] mp 208—211 °C), $[\alpha]_D^{24}$ −42° (*c*=0.46, EtOH) (lit.[14] $[\alpha]_D^{24}$ −49° (*c*=0.67, EtOH)). High MS *m/z* [M^+]: Calcd for $C_{17}H_{19}NO_5$: 317.1264. Found: 317.1236. ^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 6.68 (1H, s, H-11), 6.63 (1H, s, H-8), 5.88 (2H, s, OCH_2O), 5.56 (1H, br s, H-4), 4.65 (2H, m, H-1 and 3), 4.01 and 3.50 (each 1H, d, *J*=13 Hz, CH_2-7), 3.84 (1H, br s, H-11c), 3.43 (3H, s, OCH_3), 2.67 (1H, d, *J*=11.2 Hz, H-11b).

Ungiminorine *N*-Oxide (5) Pale yellow oil, $[\alpha]_D^{28}$ −58.9° (*c*=0.11, EtOH). IR (KBr): 3425, 2900, 1485 cm^{-1}. High MS *m/z* [M^+]: Calcd for $C_{17}H_{19}NO_6$: 333.1213. Found: 333.1219. ^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 6.98 (1H, s, H-11), 6.84 (1H, s, H-8), 5.97 and 5.95 (each 1H, d, *J*=1.7 Hz, OCH_2O), 5.62 (1H, br s, H-4), 4.65 and 4.53 (each 1H, br s, CH_2-5), 4.50 and 4.16 (each 1H, d, *J*=13 Hz, CH_2-7), 4.20 (1H, br s, H-11c), 3.34 (3H, s, OCH_3), 2.65 (1H, d, *J*=12.5 Hz, H-11b).

Galanthamine (6) Colorless prisms (from acetone), mp 126—127 °C (lit.[15] mp 127—128 °C), $[\alpha]_D^{25}$ −91.6° (*c*=0.19, EtOH) (lit.[15] $[\alpha]_D^{23}$ −109.2° (*c*=0.85, EtOH)). High MS *m/z* [M^+]: Calcd for $C_{17}H_{21}NO_3$: 287.1522. Found: 287.1495. ^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 6.67 and 6.62 (each 1H, d, *J*=8 Hz, H-11 and 12), 6.01—6.05 (2H, m, H-3 and 4), 4.62 (1H, br s, H-17), 4.16 (1H, m, H-2), 4.08 and 3.68 (each 1H, d, *J*=15 Hz, H-9), 3.84 (3H, s, OCH_3), 2.40 (3H, s, NCH_3).

Galanthamine *N*-Oxide (7) Colorless oil, $[\alpha]_D^{24}$ −133.8° (*c*=0.05, MeOH) (lit.[15] $[\alpha]_D^{26}$ −122.9° (*c*=0.38, MeOH)). IR (KBr): 3437, 2922, 1632, 1512 cm^{-1}. High MS *m/z* [M^+]: Calcd for $C_{17}H_{21}NO_4$: 303.1469. Found: 303.1450. ^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 6.77 (2H, s, H-11 and 12), 6.08—6.03 (2H, m, H-3 and 4), 4.65 (1H, br s, H-16), 4.70 and 4.40 (each 1H, d, *J*=15 Hz, CH_2-9), 4.20 (1H, br s, H-2), 3.88 (3H, s, OCH_3), 3.10 (3H, s, NCH_3).

Lycoramine (8) Colorless prisms (from acetone), mp 122—124 °C (lit.[16] mp 122—124 °C, $[\alpha]_D^{22}$ −100.0° (*c*=0.05, EtOH) (lit.[16] $[\alpha]_D^{22}$ −96.0° (*c*=0.71, EtOH)). High MS *m/z* [M^+]: Calcd for $C_{17}H_{23}NO_3$: 289.1679. Found: 289.1663. ^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 6.66 and 6.60 (each 1H, d, *J*=8 Hz, H-11 and 12), 4.37 (1H, m, H-17), 4.05 (1H, m, H-2), 4.01 and 3.62 (each 1H, d, *J*=15 Hz, CH_2-9), 3.86 (3H, s, OCH_3), 2.37 (3H, s, NCH_3).

Sanguinine (9) Pale yellow oil, $[\alpha]_D^{27}$ −100.0° (*c*=0.08, EtOH) (lit.[17] $[\alpha]_D^{27}$ −133.0° (*c*=0.23, EtOH)). High MS *m/z* [M^+]: Calcd for $C_{16}H_{19}NO_3$: 273.1366. Found: 273.1350. ^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 6.67 and 6.43 (each 1H, d, *J*=8 Hz, H-11 and 12), 4.43 (1H, m, H-17), 4.09 (1H, m, H-2), 4.05 and 3.63 (each 1H, d, *J*=15 Hz, CH_2-9), 2.38 (3H, s, NCH_3).

Lycorine (10) Colorless pillars (from MeOH), mp 258—259 °C (lit.[3]

mp 257—258 °C).

***O*-Demethyllycoramine (11)** Pale yellow oil, $[\alpha]_D^{24}$ −103.1° (c=0.21, EtOH) (lit.[16] $[\alpha]_D^{23}$ −111.9° (c=0.59, EtOH)). High MS m/z [M$^+$]: Calcd for $C_{16}H_{21}NO_3$: 275.1522. Found: 275.1513. ^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 6.64 and 6.52 (each 1H, d, J=8 Hz, H-11 and 12), 4.36 (1H, br s, H-17), 4.11 (1H, m, H-2), 3.98 and 3.60 (each 1H, d, J=15 Hz, CH_2-9), 2.37 (3H, s, NCH_3).

1-Palmitoyl-2-linoleoylphosphatidylethanolamine (12) Pale yellow oil. EI-MS m/z (%): 575 (12), 337 (65), 313 (80), 263 (48), 239 (25). FAB-MS m/z: 716 [M+1] (thioglycerol). IR (KBr): 2925, 2854, 1741, 1652, 1446, 1385, 1074, 1045 cm^{-1}. ^{1}H-NMR ($CDCl_3$, 200 MHz), δ: 5.35 (4H, m, –CH=CH–×2), 5.22 (1H, m, –CH–), 4.48—3.78 (4H, m, OCH_2CH–×2), 3.88—3.04 (4H, m, OCH_2CH_2N), 3.48 (3H, s, NH_3), 2.76 (2H, t, J=6 Hz, =C–CH_2–C=), 2.28 (4H, m, $COCH_2CH_2$–×2), 2.03 (4H, m, =C–CH_2–×2), 1.58 (4H, br s, $COCH_2CH_2$–×2), 0.88 (6H, m, CH_3).

1-Palmitoyl-2-linoleoylphosphatidylmethanol Sodium Salt (13) Pale yellow oil. EI-MS m/z (%): 575 (58), 337 (31), 313 (82), 263 (39), 239 (32). FAB-MS m/z: 709 [M+1] (thioglycerol); 731 [M+Na] (thioglycerol+NaI). IR (KBr): 2926, 2855, 1742, 1466, 1234, 1073, 1050 cm^{-1}. ^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 5.34 (4H, m, –CH=CH–×2), 5.25 (1H, m, –CH–), 4.37—3.88 (4H, m, OCH_2CH–×2), 3.57 and 3.51 (3H, each s, OCH_3), 2.77 (2H, t, J=6 Hz, =C–CH_2–C=), 2.30 (4H, m, $COCH_2CH_2$–×2), 2.05 (4H, m, =C–CH_2–×2), 1.59 (4H, br s, $COCH_2CH_2$–×2), 0.89 (6H, m, CH_3).

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