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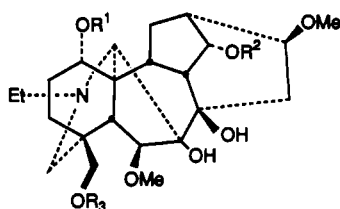
NEW NORDITERPENOID ALKALOIDS FROM THE LEAVES
OF *DELPHINIUM AJACIS*

JING LU, HARIDUTT K. DESAI, SAMIR A. ROSS, HANAA M. SAYED, and S. WILLIAM PELLETIER*

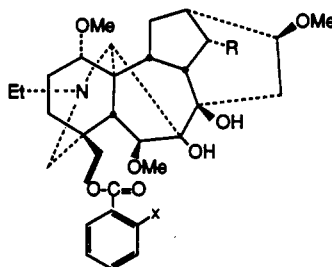
*Institute for Natural Products Research and The Department of Chemistry,
The University of Georgia, Athens, Georgia 30602-2556*

ABSTRACT.—Four new norditerpenoid alkaloids, delajacine [7], delajacirine [8], delajadine [9], and ajanine [10], have been isolated from the leaves of *Delphinium ajacis* along with six known alkaloids. The structures of the new alkaloids 7–10 were determined from the ms and ¹H- and ¹³C-nmr spectra, including DEPT experiments. The structure of 8 was confirmed by its partial synthesis from anthranoyllycoctonine [3].

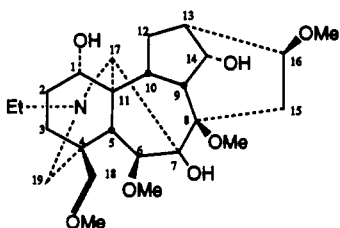
Previous work on *Delphinium ajacis* L. (Ranunculaceae) (1–7) demonstrated the presence of twenty-seven lycoctonine-type norditerpenoid alkaloids. In continuation of our efforts to examine the constituents of various parts of the plant, we recently reported the isolation of six known alkaloids from the leaves (6). Of these delcosine [1], delsoline [2], and anthranoyllycoctonine [3] had been reported previously from this plant, whereas deltraline [4], delpheline [5], and gigactonine [6] were new to this plant. We



- 1 $R^1=R^2=H, R^3=Me$
 2 $R^1=H, R^2=R^3=Me$
 6 $R^1=R^3=H, R^2=Me$
 13 $R^1=R^2=R^3=Me$

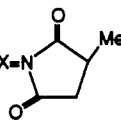


- 3 $R=OMe, X=NH_2$
 7 $R=OMe, X=NHCOCH(Me)Et$
 8 $R=OMe, X=NHCOCH(Me)_2$
 9 $R=OAc, X=NHCOCH(Me)Et$
 10 $R=OCOC(Me)(OH)Et, X=NHCOMe$
 11 $R=OMe, X=NHCOMe$
 12 $R=OAc, X=NHCOMe$

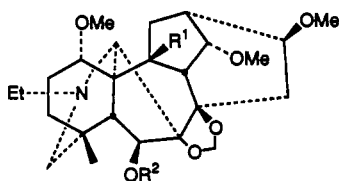


14

- 15 $R=OMe, X=N$



- 16 $R=OAc, X=NH_2$



- 4 $R^1=OH, R^2=Ac$
 5 $R^1=R^2=H$

now report the isolation and identification of four new norditerpenoid alkaloids: delajacine [7], delajacirine [8], delajadine [9], and ajanine [10], besides six known alkaloids: ajacine [11], ajadine [12], delphatine [13], deltatsine [14], methyllycaconitine [15], and 14-acetyldelectine [16]. The known compounds 11–16 have not previously been reported from the leaves. The new compounds 7 and 9 are the first two alkaloids bearing a 2-methylbutyryl group on the -NH side chain of the C-18 anthranilic acid ester. Similarly, compound 8 is the first alkaloid that possesses an isobutyryl group on the -NH side chain. The structure of 8 was confirmed by its partial synthesis from anthranoyllycoctonine [3]. Ajanine [10] is the first norditerpenoid alkaloid bearing a 2-hydroxy-2-methylbutyric acid ester group at C-14. The stereochemistry of the new compounds was determined by comparison of chemical shifts of protons and carbons with known compounds of similar stereochemistry.

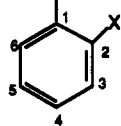
RESULTS AND DISCUSSION

The molecular formula of delajacine [7], $C_{37}H_{54}N_2O_9$, was derived from the ms m/z 670 $[M]^+$ and ^{13}C -nmr spectral data. The ir spectrum exhibited bands at 3470 and 3310 (OH), 1690 and 1682 (C=O), 1600, 1585, and 1552 cm^{-1} (aromatic system); the DEPT study indicated 8 quarternary, 14 methine, 8 methylene, and 7 Me carbons (Table 1). The 1H -nmr spectrum indicated the presence of an *N*-ethyl group 1.08 ppm (3H, t, $J=7$ Hz), a *C*-ethyl group at 0.98 ppm (3H, t, $J=7$ Hz), a secondary Me group at 1.26 ppm (3H, d, $J=6$ Hz), four methoxyls at 3.27, 3.35, 3.38, 3.43 ppm (3H each, s), and resonance typical of a C-18 ester residue at 7.12, 7.58 (1H, each, t, $J=8$ Hz) and 8.00, 8.80 ppm (1H each, d, $J=8$ Hz) such as is found in ajacine [11] (8) except for the replacement of the acetate group attached to the nitrogen by a 2-methylbutyryl group. The presence of the 2-methylbutyryl group in the molecule was supported by comparison of the chemical shifts of 7 with those reported for the *C*-ethyl and the secondary Me shifts for the C-14 ester side chain in glaucedine (9), glaucenine (9), and elanine (10). The ^{13}C -nmr spectrum of 7 showed signals characteristic of the above functional groups and compares well with those of ajacine [11] (8) except for the replacement of the group attached to the -NH side chain of the anthranilic acid ester. The presence of a fragment at m/z 85 in the ms is characteristic of $[COCH(Me)Et]^+$, and the base peak at m/z 57 for $[CH(Me)Et]^+$ is also consistent with the presence of a 2-methylbutyryl group in the structure.

Delajacirine [8] is a homologue of delajacine [7]. Careful chromatography of these two compounds was carried out on a Chromatotron rotor to achieve separation. The molecular formula $C_{36}H_{52}N_2O_9$ was deduced from the eims m/z 656 $[M]^+$ and the ^{13}C -nmr spectral data. The 1H -nmr spectrum revealed the presence of the following groups: *N*-ethyl (1.08, 3H, t), four methoxyls (3.27, 3.36, 3.39, 3.42, 3H each, s), a C-18 ester residue at 7.12, 7.58 (1H, each t, $J=8$ Hz) and 8.00, 8.78 (1H each, d, $J=8$ Hz) as in ajacine [11] (8) except for the absence of an acetate group, and a -CH(Me)₂ group (1.28, 6H, d). The ^{13}C -nmr signals at 37.5 d and 19.5 q ppm in 8 can be assigned to the carbons of a -CH(Me)₂ group by comparison with those reported for the -CH(Me)₂ group in glaucerine (9) and occidentalidine (11). The other ^{13}C -nmr signals are similar to those of ajacine [11] and delajacine [7] except for the -NHCO chain of the C-18 anthranilic acid ester group, which suggested that the -NH chain bears an isobutyryl [COCHMe₂] group. The DEPT spectrum showed 8 quaternary, 14 methine, 7 methylene, and 7 Me carbons (Table 1), in support of the assignment of structure 8. The ms with $[M]^+$ at m/z 656 is 14 mass units less than that of delajacine [7], indicating that the 2-methylbutyryl group in 7 is replaced by an isobutyryl group. The significant fragment at m/z 71 is characteristic of $[-COCHMe_2]^+$, and the base peak at m/z 43 $[-CHMe_2]^+$ is also consistent

TABLE 1. ^{13}C -nmr Chemical Shifts and Assignments for Compounds **3** and **7-12**.

Carbon	7	8	9	10	11	12	3
C-1	83.9 d	83.9 d	83.7	83.9 d	83.8 d	83.7	83.9
C-2	26.1 t	26.1 t	26.0	25.9 t	26.0 t	26.0	26.1
C-3	32.2 t	32.2 t	32.2	32.2 t	32.1 t	32.1	32.2
C-4	37.6 s	37.6 s	37.6	37.5 s	37.5 s	37.5	37.5
C-5	43.2 d	43.2 d	42.5	43.2 d	43.2 d	42.4	43.2
C-6	90.9 d	90.9 d	90.6	90.6 d	90.9 d	90.6	90.8
C-7	88.5 s	88.5 s	88.3	88.4 s	88.5 s	88.3	88.5
C-8	77.5 s	77.5 s	77.4	75.2 s	77.4 s	77.4	77.5
C-9	50.4 d	50.4 d	50.0	50.1 d	50.3 d	50.0	50.2
C-10	46.1 d	46.1 d	45.7	45.5 d	46.0 d	45.7	46.1
C-11	49.0 s	49.1 s	49.0	48.8 s	49.0 s	49.0	49.0
C-12	28.7 t	28.7 t	28.1	28.0 t	28.6 t	28.1	28.7
C-13	38.1 d	38.2 d	38.0	37.6 d	38.0 d	38.2	38.2
C-14	83.9 d	83.9 d	75.9	76.2 d	83.8 d	75.6	84.0
C-15	33.7 t	33.7 t	33.6	32.9 t	33.6 t	33.7	33.5
C-16	82.5 d	82.5 d	82.3	81.8 d	82.5 d	82.2	82.5
C-17	64.5 d	64.5 d	64.5	64.5 d	64.5 d	64.5	64.5
C-18	69.7 t	69.5 t	69.5	69.5 t	69.7 t	69.5	68.6
C-19	52.4 t	52.3 t	52.2	52.2 t	52.3 t	52.2	52.2
N-CH ₂	51.0 t	51.0 t	51.0	51.0 t	51.0 t	51.0	51.0
CH ₃	14.0 q	14.1 q	14.1	14.1 q	14.0 q	14.1	14.1
1'	55.8 q	55.9 q	55.8	55.9 q	55.8 q	55.8	55.8
6'	57.8 q	57.9 q	58.0	58.1 q	57.8 q	58.0	57.8
14'	58.1 q	58.0 q	—	—	58.0 q	—	57.8
16'	56.3 q	56.3 q	56.3	56.0 q	56.3 q	56.3	56.3
C=O	168.0 s	168.1 s	168.1	168.0 s	168.0 s	168.0	167.8
1	114.5 s	114.5 s	114.4	114.3 s	114.3 s	114.4	110.2
2	142.1 s	142.1 s	142.1	141.8 s	141.8 s	141.8	150.7
3	120.7 d	120.6 d	120.7	120.6 d	120.5 d	120.5	116.8
4	135.0 d	135.0 d	135.1	135.1 d	134.9 d	135.0	134.4
5	122.3 d	122.3 d	122.5	122.6 d	122.5 d	122.6	116.8
6	130.3 d	130.3 d	130.3	130.2 d	130.2 d	130.2	130.8
X=NHCO	175.8 s		175.8				
-CH	45.0 d		45.0				
(CH ₃)	17.3 q		17.3				
-CH ₂	27.3		27.3				
CH ₃	11.8		11.8				
X=NHCO		176.2 s					
CH		37.5 d					
(CH ₃) ₂		19.5 q					
X=NHCO				169.1 s	169.1 s	169.1	
CH ₃				25.5 q	25.5 q	25.5	
OC=O			172.0			171.9	
CH ₃			21.5			21.5	
OC=O				176.2 s			
-C-OH				77.2 s			
(CH ₃)				25.0 q			
-CH ₂				33.9 t			
CH ₃				8.0 q			



with the presence of an isobutyryl group in the molecule. Structure **8** for delajacirine was confirmed by its partial synthesis from anthranoyllycoctonine [**3**] and isobutyryl chloride.

The molecular formula of delajadine [**9**], $\text{C}_{38}\text{H}_{54}\text{N}_2\text{O}_{10}$, was deduced from its eims m/z 698 $[\text{M}]^+$ and ^{13}C -nmr spectral data. The ir spectrum exhibited bands at 3470 and 3310 (OH), 1740, 1690, and 1682 (C=O), 1600, 1585, 1525 cm^{-1} (aromatic system).

Its ^1H -nmr spectrum indicated the presence of an *N*-ethyl at 1.08 ppm (3H, t, $J=7$ Hz), three methoxyls at 3.28, 3.34, 3.80 ppm (3H, each, s), resonance typical of a C-18 ester residue at 7.13, 7.60 (1H each, t, $J=8$ Hz) and 7.98, 8.80 (1H each, d, $J=8$ Hz) such as is found in delajacine [7], and an acetate group at 2.07 ppm (3H, s). The location of the acetate group at C-14 was discerned from the upfield ^{13}C -nmr signal at 75.9 ppm compared with that of 83.9 ppm in delajacine [7], and was confirmed by comparing ^1H - and ^{13}C -nmr spectra with those of ajadine [12] (8), which bears a C-14 acetate group. The ^{13}C -nmr shifts, which compare well with those of delajacine [7] except for the C-14 acetate group (172.0, 21.5 ppm), suggested the structure for delajadine to be *N*-deacetyl-*N*-2-methylbutyrylajadine [9]. Besides the presence of the significant fragments at m/z 85 [$\text{COCH}(\text{Me})\text{Et}$] $^+$ and m/z 57 [$\text{CH}(\text{Me})\text{Et}$] $^+$, which showed the presence of the 2-methylbutyryl group, the ms exhibited $[\text{M}]^+$ at m/z 698, 28 mass units more than that of delajacine [7], supporting the presence of monoacetate group. The base peak at m/z 43 is also indicative of the acetate group.

Another new alkaloid isolated from this plant is ajanine [10]. Its molecular formula, $\text{C}_{38}\text{H}_{54}\text{N}_2\text{O}_{11}$, was deduced on the basis of the ms and ^{13}C -nmr spectral data. The ^1H -nmr spectrum exhibited the presence of an *N*-ethyl group (1.08, 3H, t), three MeO groups (3.28, 3.30, 3.39, each 3H, s), resonance typical of a C-18 ester residue at 7.13, 7.60 (1H, each, t, $J=8$ Hz) and 7.97, 8.73 (1H each, d, $J=8$ Hz) such as is found in ajacine [11] (8) or ajadine [12] (8), a *C*-ethyl group (0.90, 3H, t), and a tertiary Me group (1.42, 3H, s). The ^{13}C -nmr spectrum, which shows signals characteristic of the above functional groups, compares well with those of ajacine [11] and ajadine [12] except for the C-14 ester group (176.2 ppm signal). The DEPT experiment revealed 10 quarternary, 13 methine, 8 methylene, and 7 Me carbons (Table 1). The presence of 10 quaternary carbons in 10 suggests an extra tertiary carbon on the C-14 chain as compared with the other 9 quaternary carbons in the molecule of ajadine [12] (8) and, therefore, a replacement of the ester group on the C-14 chain in 10. The presence of a significant fragment at m/z 73 in the ms of 10 is characteristic of [$-\text{C}(\text{Me})(\text{OH})-\text{Et}$] $^+$, and indicates the cleavage of a 2-hydroxyl-2-methylbutyryl ester side chain at C-14.

EXPERIMENTAL

GENERAL EXPERIMENTAL PROCEDURES.—Mp's were taken on a Thomas-Koffler hot stage equipped with a microscope and a polarizer. Optical rotations were measured on a Perkin-Elmer model 141 polarimeter in CHCl_3 . ^1H and ^{13}C nmr were recorded on a Bruker AC-300 spectrometer in CDCl_3 . Ms were determined on a Finnigan Quadrupole 4023 instrument. For chromatographic separations on a Chromatotron (12,13), rotors were coated with a 1-mm-thick layer of Al_2O_3 60 PF 254+365 (type E, EM. 1104), or Si gel GF 254 (EM, 7749). For separation by vlc, (14), Al_2O_3 60 H (type E, EM. 1085) was employed. All the known compounds isolated were identified by comparing their spectral data and tlc behavior with those of authentic samples.

PLANT MATERIAL.—The plants of *D. ajacis* were cultivated in September 1989 in the Experimental Station of the Faculty of Pharmacy, Assiut University, Assiut, Egypt, and collected during the flowering stage in April 1990. The plants were identified by Professor Naeem El-Keltawy, Faculty of Agriculture, Assiut University. A voucher specimen (No. 105) has been deposited in the Herbarium of the Department of Pharmacognosy.

EXTRACTION OF THE ALKALOIDS.—The air-dried and powdered leaves of *D. ajacis* (1.5 kg) were first defatted with hexane (1×5 liters). The defatted leaves were extracted with 80% EtOH (2×6 liters) at room temperature. The EtOH extract was concentrated under reduced pressure to give 200 g of dry extract. The extract was fractionated by a gradient pH extraction technique (15) to yield 10 g of a neutral fraction and 4.85 g (pH 8) and 0.88 g (pH 12) of crude alkaloids.

ISOLATION OF DELAJACINE [7], DELAJACIRINE [8], DELAJADINE [9], AND AJACINE [11].—The neutral fraction (10 g) was chromatographed (vlc) (14) on Al_2O_3 . Elution was made with hexane, Et_2O , and EtOH in order of increasing polarity. Two alkaloid-containing fractions were collected. Fraction 1 (1.7 g) eluted

with 40%, 60%, and 80% Et₂O/hexane, and fraction 2 (0.86 g) eluted with Et₂O and 2%, 5%, and 8% EtOH/Et₂O.

Fraction 1 was repeatedly fractionated on Al₂O₃ rotors to give 16 mg of delajacine [7] eluted with 25% Et₂O/hexane; 5 mg of delajacirine [8] eluted with 25% Et₂O/hexane; and 31 mg of ajacine [11] (8) eluted with 50% Et₂O/hexane, mp 135–137° (from Et₂O/hexane).

Delajacine [7].—Mp 73–76°, [α]_D +58.9° (*c*=0.1); ir (Nujol) 3470 and 3310 cm⁻¹ (OH), 1690 and 1682 cm⁻¹ (C=O), 1600, 1585, 1525 cm⁻¹ (C=C); eims *m/z* (%) [M]⁺ 670 (0.8), [M-Me]⁺ 655 (2.2), [M-OMe]⁺ 639 (41), [-COCH(Me)Et]⁺ 85 (13), [CH(Me)Et]⁺ 57 (100); ¹H nmr δ 0.98 (3H, t, *J*=7 Hz, -CH₂CH₃), 1.08 (3H, t, *J*=7 Hz, -NCH₂CH₃), 1.26 (3H, d, *J*=6.8 Hz, -CH(CH₃)Et), 3.27, 3.35, 3.38, 3.42 (3H each, s, 4×OMe), 3.61 (1H, t, *J*=4.5 Hz, H-14β), 3.92 and 3.96 (1H each, s), 4.18 (2H, s, H-18), 7.12 and 7.58 (1H each, t, *J*=8 Hz, Ar-H-4 and H-5), 8.00, and 8.80 (1H, d, *J*=8 Hz, Ar-H-3 and H-6), 11.0 (1H, s, -NH); ¹³C nmr see Table 1.

Delajacirine [8].—Amorphous: [α]_D +39.62° (*c*=0.2); ir (Nujol) 3470 and 3310 cm⁻¹ (OH), 1690 and 1682 cm⁻¹ (C=O), 1600, 1585, and 1525 cm⁻¹ (C=C); eims *m/z* (%) [M]⁺ 656 (0.5), [M-Me]⁺ 641 (1.6), [M-OMe]⁺ 625 (24.9), [COCHMe₂]⁺ 71 (26.4), [-CHMe₂]⁺ 43 (100); ¹H nmr 1.08 (3H, t, *J*=7 Hz, -NEt), 1.28 (6H, d, *J*=9 Hz, -CH Me₂), 3.27, 3.36, 3.39, 3.42 (3H each, s, 4×OMe), 3.61 (1H, t, *J*=4.5 Hz, H-14β), 3.92 and 3.96 (1H each, s), 4.19 (2H, s, H-18), 7.12 and 7.58 (1H, each, t, *J*=8 Hz, Ar-H-4 and H-5), 8.00 and 8.78 (1H each, d, *J*=8 Hz, Ar-H-3 and H-6), and 11.0 (1H, s, -NH); ¹³C nmr see Table 1.

Fraction 2 was subsequently chromatographed on an Al₂O₃ rotor to give 11 mg of delajadine [9], eluted with 15% Et₂O in hexane.

Delajadine [9].—Amorphous: [α]_D +54.0° (*c*=0.1); ir (Nujol) 3470 and 3310 cm⁻¹ (OH), 1740, 1690, and 1682 cm⁻¹ (C=O), 1600, 1585, and 1525 cm⁻¹ (C=C); eims *m/z* (%) [M]⁺ 698 (0.01), [M-Me]⁺ 683 (0.28), [M-OMe]⁺ 667 (6.98), [-COCH(Me)Et]⁺ 85 (6.12), [-CH(Me)Et]⁺ 57, (46.41), [-OCOMe]⁺ 43 (100); ¹H nmr δ 0.98 (3H, t, *J*=7 Hz, -CH₂CH₃), 1.08 (3H, t, *J*=7 Hz, -NCH₂CH₃), 1.26 (3H, d, *J*=7 Hz, CH(CH₃)Et), 2.07 (3H, s, -OAc), 3.28, 3.34, 3.80 (3H each, s, 3×OMe), 3.83 and 3.93 (1H each, s), 4.78 (1H, t, H-14β), 7.13 and 7.60 (1H each, t, *J*=8 Hz, Ar-H-4 and H-5), 7.98 and 8.80 (1H each, d, *J*=8 Hz, Ar-H-3 and H-6), 11.0 (1H, s, -NH); ¹³C nmr see Table 1.

ISOLATION OF AJANINE [10], AJACINE [11], AJADINE [12], ANTHRANOYLLYCOCTONINE [3], 14-ACETYLDELECTINE [16], METHYLLYCAONITINE [15], DELSOLINE [2], DELPHATINE [13], DELTATSINE [14], DELCOSINE [1], AND GIGACTONINE [6].—The pH 8 alkaloid fraction (4.85 g) was chromatographed (vlc) (14) on Al₂O₃. Elution was performed with hexane, Et₂O, EtOH, and MeOH in order of increasing polarity. In all, six fractions were collected.

Fraction 2 (0.38 g, eluted with 50% and 60% Et₂O/hexane) was fractionated four times on Al₂O₃ rotors to provide 14 mg of ajadine [12] (8), eluted with 60% Et₂O/hexane, and 10 mg of deltatsine [14] (16), eluted with 80% Et₂O/hexane.

Fraction 3 (0.97 g, eluted with 70 and 100% Et₂O/hexane) was fractionated repeatedly on Al₂O₃ rotors to obtain 200 mg of delsoline [2] (8), mp 212–214° (from Me₂CO/hexane), eluted with Et₂O; 10 mg of delphatine [13] (8), eluted with Et₂O; 12 mg of ajanine [10], eluted with 80% Et₂O/hexane; 15 mg of 14-acetyldelectine [16] (7), eluted with 60% Et₂O/hexane; 45 mg of ajacine [11] (8), mp 136–138° (from Me₂CO/hexane), eluted with Et₂O; and 40 mg of anthranoyllycoctonine [3] (8), eluted with 2% EtOH/Et₂O.

Ajanine [10].—Amorphous: [α]_D +47.37° (*c*=0.1); ir (Nujol) 3470 and 3310 cm⁻¹ (OH), 1690 and 1680 cm⁻¹ (C=O), 1600, 1585, and 1525 cm⁻¹ (C=C); eims *m/z* (%) [M]⁺ 714 (0.43), [M-Me]⁺ 699 (1.69), [M-OMe]⁺ 683 (43.78), [-C(Me)(OH)CH₂Me]⁺ 73 (11.61), [Ac]⁺ 43 (100); ¹H nmr δ 0.90 (3H, t, *J*=7 Hz, -CH₂CH₃), 1.08 (3H, t, *J*=7 Hz, -NCH₂CH₃), 1.42 (3H, s, -CMe), 3.28, 3.30, 3.39 (3H each, s, 3×OMe), 4.92 (1H, t, H-14β), 3.79 and 3.92 (1H each, s), 7.13 and 7.60 (1H each, t, *J*=8 Hz, Ar-H-4, and H-5), 7.97 and 8.73 (1H each, d, *J*=8 Hz, Ar-H-3 and H-6), 11.0 (1H, s, -NH); ¹³C nmr see Table 1.

Fraction 4 (0.53 g, eluted with Et₂O and 2% EtOH in Et₂O) on fractionation (Si gel rotor) gave 43 mg of ajacine [11] (8), eluted with 40% CHCl₃/hexane.

Fraction 5 (0.48 g, eluted with 5% EtOH in Et₂O) was chromatographed on Al₂O₃ rotors. Elution was performed with hexane, Et₂O and Me₂CO to get 15 mg of methyllycaconitine [15] (8), eluted with 3% Me₂CO/Et₂O, and 20 mg of gigactonine [6] (8), eluted with 10% Me₂CO/Et₂O.

Fraction 6 (0.87 g, eluted with MeOH) was purified on a small column of Si gel and fractionated on an Al₂O₃ rotor. Elution was carried out with hexane, Et₂O, and Me₂CO in a manner of increasing polarity to provide 97 mg delcosine [1] (8), mp 206–208° (from Me₂CO/Et₂O), eluted with 4% Me₂CO/Et₂O; and 30 mg of gigactonine [6] (8), eluted with 10% Me₂CO/Et₂O.

PARTIAL SYNTHESIS OF DELAJACIRINE [8].—To an ice-cooled solution of anthranoyllycoctonine [3] (40 mg) in C₆H₆ (5 ml) containing pyridine (0.25 ml), isobutyryl chloride (0.25 mg) was added, and the solution was left in the refrigerator for 20 h. Usual workup and fractionation of the crude product on an Al₂O₃ rotor gave synthetic delajacirine [8] (23.1 mg). The synthetic product had tlc behavior, ir, and ¹H- and ¹³C-nmr spectra identical with those of the natural compound.

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