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DAURINE — A NEW ALKALOID FROM *Haplophyllum dauricum*

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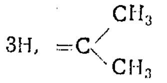
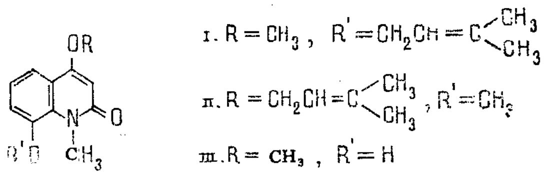
UDC 547.944/945

From the total alkaloids of the roots of *Haplophyllum dauricum* (L.) G. Con., collected in Mongolia, by column chromatography we have isolated an optically inactive base with mp 117-118°C, which has been called daurine. The base is soluble in ethanol and chloroform and crystallizes from acetone.

The IR spectrum of daurine has intense absorption in the 1650 cm^{-1} region (2-quinoline system); there is no absorption band corresponding to active hydrogen. The UV spectrum has absorption maxima at (nm) 215, 235, 254, 268 shoulder, 276 shoulder, 286 shoulder, 328, which are characteristic for alkaloids of the 4,8-dialkoxy-N-methyl-2-quinoline series [1].

The PMR spectrum of daurine ($CDCl_3$, 0 — TMS, taken on a Varian XL-200 instrument) contains signals characterized by the following values (δ, ppm): 1.73 and 1.80 (singlets, 3 H each,  $3H, =C(CH_3)_2$) : 3.91 and 3.93 (singlets, 3 H each, NCH_3 and OCH_3); 4.57 and 5.52 (doublet, 2 H, ^3J = 6.8 Hz; triplet, 1 H, ^3J = 6.8 Hz; protons of a $-O-CH_2-CH=$ fragment forming a three-spin system of the A_2X type); 6.05 (singlet, 1 H, H-3); 7.09 (quartet, 1 H, J_{ortho} = 7.9 Hz and J_{meta} = 2.4 Hz, H-7); 7.13 (triplet, 1 H, J_{ortho} = 7.8 Hz and 7.2 Hz, H-6); 7.60 (quartet, 1 H, J_{ortho} = 7.2 Hz and J_{meta} = 2.4 Hz, H-5) — aromatic protons forming a system of the ABC type; a similar pattern is observed in the spectrum of edulitine [2].

What has been stated above and also the presence in the mass spectrum of the alkaloid of intense peaks of ions with m/z 273 (M^+, 33%), 205 (M — 69, 100%), and 69 (M — 205, 18%) gives grounds for the assumption that daurine may have a 4-methoxy-8-(γ,γ-dimethylallyloxy)-N-methyl-2-quinolone (I) or an 8-methoxy-4-(γ,γ-dimethylallyloxy)-N-methyl-2-quinolone (II) structure.



I. $R = CH_3$, $R' = CH_2CH=C(CH_3)_2$

II. $R = CH_2CH=C(CH_3)_2$, $R' = CH_3$

III. $R = CH_3$, $R' = H$

The choice between these structures was made on the basis of the following facts. In an alkaline medium, the UV spectrum of daurine did not change, while on the addition to an ethanolic solution of the base of a drop of concentrated hydrochloric acid it became identical with the spectrum of folifidine (III) taken under similar conditions. An examination of the sample obtained from the acidified ethanolic solution showed its identity with (III) (GLC, IR spectra).

Institute of the Chemistry of Plant Substances, Academy of Sciences of the Uzbek SSR, Tashkent. Translated from Khimiya Prirodnykh Soedinenii, No. 1, p. 116, January-February, 1983. Original article submitted July 2, 1982.

0009-3130/83/1901-0117$07.50

Thus, we have established that daurine has the structure (I).

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ALKALOIDS OF *Veratrum lobelianum*.

VIII. THE STRUCTURE OF GERMINALINE

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UDC 547.944/945

The isolation of a base with mp 220-222°C has been recorded previously. By a study of the products of alkaline hydrolysis, it was characterized as germanitrine [1, 2]. A determination of its elementary composition (mol. wt. 793, $C_{41}H_{63}NO_{14}$) on a MKh-1310 high-resonance mass spectrometer and a renewed study of its PMR spectrum (JNM-4H 100/100 MHz, internal standard HMDS) of the alkaloid showed that the compound was germitetrine (I) (melting point, IR spectrum) [3, 4].

Continuing the separation of the total alkaloids of the epigeal part of *V. lobelianum* collected in the Caucasus [5, 6] on a column of silica gel (eluent benzene—acetone), we have isolated an alkaloid with the composition $C_{39}H_{61}NO_{13}$, mol. wt. 751 (mass spectrometry), mp 138-140°C (II). The IR spectrum of (II) had absorption bands at (cm^{-1}) 3450 (OH), 1745, and 1250 (ester C=O). The main chemical shifts of the protons in the PMR spectrum of alkaloids (I) and (II) taken in $CDCl_3$ are given below (δ, ppm: s — singlet; d — doublet; t — triplet; m — multiplet):

	Gemitetrine (I)	*Alkaloid (II)*
19-CH_3 (s)	0,94	0,89
21-CH_3 (s)	1,10	1,11
27-CH_3 (d)	1,02	1,00
$OCOCH_3$ (s)	1,88; 2,00	1,89
$\underline{C}H—O$-acyl (m)	5,71; 5,04 (3H)	4,98; 5,23; 5,91
Side chain:		
$—\underset{OH}{\underset{\vert}{C}}—CH_3$ (s)	1,28	1,26
$—CH—CH_3$ (d)	1,06; 1,22	1,08; 1,21
$—CH_2—CH_3$ (t)	0,82	0,82

On acetylation, the alkaloid (II) formed a diacetyl derivative (III) with mp 210-212°C, M^+ 835, and a triacetyl derivative (IV) with mp 200-202°C M^+ 877. This alkaloid proved to be identical with germinaline (melting point and IR spectrum) [7]. However, a comparison of the elementary composition of (II) (M^+ 751) with that of germinaline, which we isolated previously from the epigeal part of *V. lobelianum* [7] showed that they differed by one oxygen atom. We have compared the acetyl derivatives of (III) and (IV) with the corresponding acetyl derivatives of germerine (V) [8-10] — diacetylgermerine (VI), with mp 220-222°C, M^+ 777, and triacetylgermerine (VII) with mp 200-202°C, M^+ 819.

In spite of the identical R_f values of (III) and (VI), and of (IV) and (VII), these substances differ from one another by 58 mass units, respectively. Consequently, the elementary composition of germaline was previously determined incorrectly, and the acetyl derivatives of germinaline are not identical with those of germerine.

Institute of the Chemistry of Plant Substances of the Academy of Sciences of the Uzbek SSR, Tashkent. Translated from Khimiya Prirodnykh Soedinenii, No. 1, pp. 116-118, January-February, 1983. Original article submitted July 2, 1982.

0009-3130/83/1901-0118$07.50