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ALKALOIDS OF *Diptychocarpus strictus*

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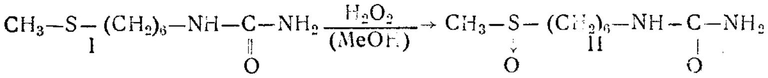
UDC 547.944/945

Continuing a study of the alkaloid composition of the seeds of *Diptychocarpus strictus* collected in the territory of the Chimkent oblast (September, 1976), by the usual chloroform extraction we obtained 0.13% of total alkaloids. On separating these alkaloids by polybuffer distribution, from the pH 2.2 fraction we isolated a new crystalline base with mp 118-119°C (acetone—MeOH) with the composition $C_8H_{18}N_2OS$ (I). The IR spectrum of (I) showed absorption bands at 3220, 3390 cm^{-1} (stretching vibrations of a NH bond) and 1660 cm^{-1} (amide carbonyl).

The NMR spectrum of the base (C_5D_5N) was characterized by the following signals (ppm): 1.10-1.42 (8 H, m, methylene protons); 1.18 (3 H, s, $S{-}CH_3$), and 2.20 (2 H, t, $S{-}CH_3$). The mass spectrum of (I) contained, in addition to the peak of the molecular ion with m/e 190, strong peaks of ions with m/e 175 $(M-15)^+$, 143 $(M-57)^+$, 119, 73, 61, and 44.

In a comparative study of the mass spectra it was found that base (I) and diptocarpaine (I) contain the same set of peaks, with the exception of the peaks of the molecular ions, which differ by 16 m/e. The IR spectrum of one differs from the spectrum of diptocarpaine by the absence of the absorption band of an S—O bond.

On the basis of what has been said above, we assumed that base (I) is a deoxy product of diptocarpaine. In fact, when base (I) was oxidized with hydrogen peroxide we obtained a product the spectral (IR, mass, and NMR) characteristics of which proved to be identical with those of diptocarpaine (II). Thus, base (I) is 6-thiomethyl-N-n-hexylurea:



$$\mathrm{CH_3{-}\underset{I}{S}{-}(CH_2)_6{-}NH{-}\underset{\underset{O}{\|}}{C}{-}NH_2} \xrightarrow[(MeOH)]{H_2O_2} \mathrm{CH_3{-}\underset{\underset{O}{\downarrow}}{S}{-}\underset{II}{(CH_2)_6}{-}NH{-}\underset{\underset{O}{|}}{C}{-}NH_2}$$

The epigeal part of the plant collected in the Dzhizak oblast (April, 1976) contained 0.1% of combined alkaloids from which by separation of a column of alumina we isolated a base with the composition $C_4H_{10}N_2O$ (III), mp 157-158°C.

IR spectrum (cm^{-1}): 3230 and 3360 (NH, NH_2), 1660 (amide carbonyl), and 1605, 1555 (amide II bands). The NMR spectrum showed the signals of the protons of an isopropyl group with δ 1.08 (6 H, d, J = 6 Hz, $-HC\langle^{CH_3}_{CH_3}$, 3.32 ppm (1 H, m, >CH), and the signals of NH and NH_2 groups at 4.41 ppm.

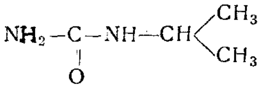
The mass spectrum of the base showed the peak of the molecular ion with m/e 102 and the peaks of ions with m/e 87 $(M-15)^+$, 58, and 44 (100%).

The mass spectrometric fragmentation of (III) was similar to that of N,N'-diisopropylurea [2], isolated previously from this plant. The difference of 42 m/e in the molecular weights shows that base (III) contains one isopropyl group. This was also confirmed by the NMR spectrum, which had a signal in the 1.1 ppm in the form of a doublet (6 H) from one isopropyl group.

Institute of the Chemistry of Plant Substances, Academy of Sciences of the Uzbek SSR, Tashkent. Translated from Khimiya Prirodnyk Soedinenii, No. 4, pp. 535-536, July-August, 1978. Original article submitted March 16, 1978.

0009-3130/78/1404-0463$07.50

All that has been said above, the composition, the melting point, and the spectral characteristics, and also a comparison with the literature [3], permitted the assumption that base (III) was isopropylurea, and this is the first time that we have isolated it from a plant:



$NH_2-C(=O)-NH-CH(CH_3)_2$

III

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THE STRUCTURE OF SEVERZININE

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UDC 547.943

Continuing the separation of the total alkaloids of *Corydalis sewerzowi* [1], from the nonphenolic part of the combined alkaloids we have isolated a new base with mp 90-91°C (with foaming, from ethanol), $[\alpha]_D$ + 109° (c 1.2; chloroform), which we have called severzinine (I). UV spectrum: $\lambda_{max}^{ethanol}$ 290 nm (log ε 4.04). The IR spectrum of (I) showed absorption bands at (cm^{-1}) 3400 (OH), 1510 and 1490 (aromatic ring), and 1050 and 930 (CH_2O_2). The mass spectrum of (I) has the peaks of ions with m/e 369 (M^+, 336, 322 (100%), 292, and 190. The NMR spectrum contains the signals from a N-methyl group at 2.47 ppm (3 H, singlet), and from two methylenedioxy groups at 5.76 ppm (2 H, singlet) and 5.92 ppm (2 H, quadruplet).

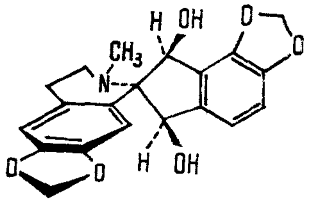
In the aromatic region of the spectrum there are signals in the form of two one-proton singlets at 6.14 and 6.56 ppm (paraaromatic protons) and a two-proton singlet at 6.76 ppm. At 5.10 and 5.33 ppm there are one-proton singlets, at 2.35 ppm a two-proton singlet, and at 2.5-3.5 ppm a multiplet. The features of its UV, IR, NMR, and mass spectra permit the assignment of (I) to the spirobenzylisoquinoline alkaloids with two hydroxy groups in the five-membered ring [2, 3].

The presence in the mass spectrum of an ion with m/e 190 shows that there is one methylenedioxy group in the isoquinoline moiety of the molecule. The presence of one of the singlets of the aromatic protons at 6.14 ppm, which is characteristic for C_1, shows that the methylenedioxy group is located at C_2 and C_3.

The second methylenedioxy group is present in ring D. To determine its position we compared severzinine with dihydrosibiricine, obtained by the reduction of sibiricine with $NaBH_4$ [4].

A direct comparison of the two substances showed their identity (melting point, TLC, IR spectra).

On the basis of what has been said above, it may be concluded that severzinine has the following structure:



Institute of the Chemistry of Plant Substances, Academy of Sciences of the Uzbek SSR, Tashkent. Translated from Khimiya Prirodnykh Soedinenii, No. 4, pp. 536-537, July-August, 1978. Original article submitted April 6, 1978.

0009-3130/78/1404-0464$07.50