LITERATURE CITED

1. I. A. Israilov, M. S. Yunusov, and S. Yu. Yunusov, Khim. Prirodn. Soedin., 428 (1977).
2. G. Nonaka and I. Nishioka, Chem. Pharm. Bull., 23, 294 (1975).
3. A. R. Battersby, I. R. C. Bick, W. Klyne, J. P. Jennings, P. M. Scopes, and M. J. Vernengo, J. Chem. Soc., 2239 (1965).

X-RAY STRUCTURAL INVESTIGATION OF THE ALKALOID SOPHORIDINE

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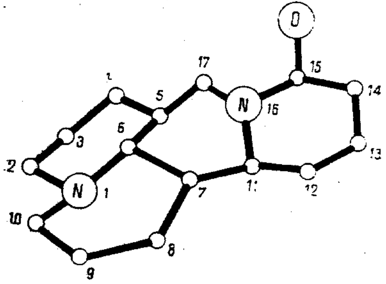
UDC 548.737

Information on the spatial structure of sophoridine up to 1975 was ambiguous and only investigations by the INDOR method and by PMR spectroscopy at 300 MHz [1] made it possible to reduce this ambiguity. It was shown that sophoridine has a conformation with two rings in the boat form (rings B and C). However, the IR spectra of sophoridine in the liquid and solid phases proved to be different [2] and, in view of the lability of the conformation of this alkaloid found in a study of chemical reactions in solutions, it could be assumed that the spatial structure of sophoridine in the crystalline state was different from that in solutions. In order to determine the conformational state of the sophoridine molecule in crystals we have made an x-ray structural investigation of this compound.

Single crystals of sophoridine obtained by crystallization from petroleum ether have an acicular form. The parameters of the monoclinic cell measured on precession x-ray diagrams and refined on a "Sinteks P2$_1$" diffractometer were as follows: $a = 5.458(1)$, $b = 10.640(2)$, $c = 11.989(2)$ Å, $\gamma = 99.7(3)°$, $V = 686.2$ Å^3, mol. wt. = 258, $d_{calc} = 1.19$ g/cm^3, $Z = 2$, space group P2$_1$. The intensities of 920 reflections were measured by $\theta/2\theta$ scanning in CuK$_\alpha$ radiation (graphite monochromator) up to $2\theta \leq 110°$ on the above-mentioned diffractometer; 802 reflections with $I \geq 2\sigma$ were used in the calculations.

A model of the structure was found by using the "Roentgen-75" system of programs [3] and was refined by the method of least squares in the anisotropic approximation to R = 0.090 by a program of the "Kristall" group [4]. The geometry of the molecule is shown in Fig. 1.



Ring A has the chair form with the same departure of the C(3) and C(6) apical atoms from the C(1) C(2) C(4) C(5) plane of ±0.68 Å, i.e., less than for the ideal cyclohexane C$_6$ chair of ±0.73 Å [5]. Rings B and C have the ^{1,8}B and ^{5,11}B unsymmetrical boat conformations with different bends of the apices. The deviations of the angles in these rings from the plane of the "bottom" of the boat are 0.68 Å and 0.60 Å for the N(1) and C(5) and for the C(8) and C(11) atoms, respectively. The conformation of ring D is close to the half-chair conformation; the C(12) and C(13) atoms deviate in opposite directions by 0.60 and 0.13 Å from the plane of the other atoms of the ring. Thus, the conformation of ring D of sophoridine in the crystal differs from the conformation of this ring in solution [1].

The A/B, A/C, and C/D ring linkages are trans and the B/C linkage is cis. The coordination of the N(1) nitrogen is pyramidal. Because of conjugation in the lactam fragment of the

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Institute of Bioorganic Chemistry, Academy of Sciences of the Uzbek SSR, Tashkent. V. I. Lenin Tashkent State University. Translated from Khimiya Prirodnykh Soedinenii, No. 4, pp. 538-539, July-August, 1978. Original article submitted May 4, 1978.

0009-3130/78/1404-0466$07.50

molecule, the coordination of the N(16) molecule is plane-trigonal. The lengths of the C(15)—O and C(15)—N(16) bonds are 1.22 and 1.32 Å, respectively (accuracy of the determination of the interatomic distances not worse than 0.015 Å).

LITERATURE CITED

1. F. T. Kamaev, V. B. Leont'ev, Kh. A. Aslanov, Yu. V. Ustynyuk, and A. S. Sadykov, Khim. Prirodn. Soedin., 744 (1974).
2. T. K. Yunusov, A. P. Matveeva, V. B. Leont'ev, F. G. Kamaev, Kh. A. Aslanov, and A. S. Sadykov, Khim. Prirodn. Soedin., 200 (1974).
3. V. I. Andrianov, Z. Sh. Safina, and B. L. Tarnopol'skii, Zh. Strukt. Khim., 15, 911 (1974).
4. L. A. Muradyan and V. I. Simonov, Kristallografiya, 18, 75 (1974).
5. G. I. Birnbaum, Acta Crystallogr., B26, 755 (1970).

THE GLOBULINS OF COTTON SEEDS

XV. PRIMARY STRUCTURE OF A GLYCOPEPTIDE OF SUBUNIT C11 OF THE S-GLOBULIN

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UDC 547.962.5

Previously, from a tryptic hydrolysate of subunit C we obtained a fraction of peptides insoluble at pH 2.2 [1]. In the present paper we give information on the separation and purification of this fraction and the determination of the primary structure of a glycopeptide. To separate the peptides we used gel filtration on a column (2 × 70 cm) of Sephadex G-50 (fine) equilibrated with 50% acetic acid containing 6 M urea. The eluate was collected in 2.5 ml fractions and the absorption of the solution was measured at 280 nm. The results are given in Fig. 1.

The purity of the combined fractions was checked by TLC and PC under the conditions described previously [1] and by the determination of the N-terminal amino acids. Fraction 1 was homogeneous, and the presence of sugars in it was shown by the reaction with a 0.2% solution of anthrone in concentrated H_2SO_4. Fractions 2-5 of the peptides were separated by PC and electrophoresis as described previously in [1].

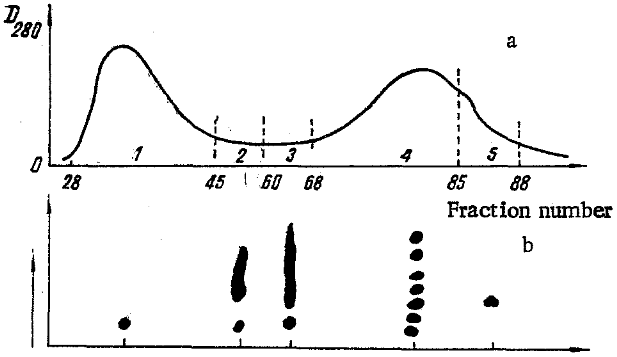

Fig. 1. Chromatography of the acid-insoluble tryptic peptides on a column of Sephadex G-50 (fine) (a) and thin-layer chromatography of the combined fractions (b).

Institute of the Chemistry of Plant Substances, Academy of Sciences of the Uzbek SSR, Tashkent. Translated from Khimiya Prirodnykh Soedinenii, No. 4, pp. 539-540, July-August, 1978. Original article submitted March 27, 1978.

0009-3130/78/1404-0467$07.50