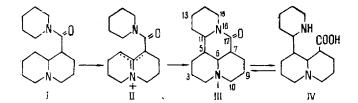
ponents with  $R_f$  0.26, 0.78, and 0.35 (in a thin layer of alumina with acetone as the solvent and iodine vapor as the revealing agent). The mixture of dehydro products was hydrogenated with sodium borohydride in methanol.

By means of adsorption chromatography on alumina and elution with acetone, two individual substances were isolated from the hydrogenation products. One of them (yield 3.4%;  $R_f 0.24$ ;  $[\alpha]_D +18.1^\circ$ ; c 2.1; ethanol) had the composition  $C_{15}H_{24}ON_2$  (III). The IR spectrum of the base III exhibits bands at 1630 cm<sup>-1</sup> (>N-CO group) and 2763 cm<sup>-1</sup> (trans-quinolizidine).

By heating the base III with 18% hydrochloric acid in a sealed tube at  $160-180^{\circ}$  C for 20 hr we obtained an amino acid (IV) giving an ethyl ester (picrate mp 70-72° C) and a N-benzoyl derivative (hydrochloride, mp 220-222° C).

The cyclization of IV by heating at 240-250° C regenerated the base III.

The reduction of the base III with lithium aluminum hydride gave allomatridine. Consequently, the base that we obtained is a new structural isomer of allomatrine and the lactam carbonyl in it is present at  $C_{(17)}$  (III).



The second base (yield 85%, Rf 0.78) is optically inactive. When it was boiled with 20% sulfuric acid for 18 hr, piperidine and the racemate of epilupinic acid was obtained.

The second base is a racemate of epilupinoylpiperidine (I) which is probably formed by the hydrogenation of an intermediate product, 6,7-dehydroepilupinoylpiperidine (II).

## REFERENCES

1. Kh. A. Aslanov, T. K. Kasymov, A. S. Sadykov, and A. I. Ishbaev, KhPS [Chemistry of Natural Compounds], 3, 412, 1967.

2. N. I. Leonard, F. P. Hauck, J. Am. Chem. Soc., 79, 5279, 1957.

3. C. Schöpf, K. Keller, Naturw., 43, 325, 1956; RZhKh, 8, 26989, 1957.

4. E. Ochiai. Sh. Okuda, H. Minato. J. Pharm. Soc. Jap., 72, 781, 1952, C. A. 2724, 1954.

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## LYCORINE FROM UNGERNIA TRISPHAERA

A. Abdusamatov, Kh. Allayarov, Kh. A. Abduazimov, and S. Yu. Yunusov

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Lycorine is used in the treatment of acute and chronic bronchitis, bronchiectatic diseases, and bronchial asthma.

	Table	T		
Time of gathering	Height of the leaves, cm	Percentage of the weight of the dry leaves		
		com- bined alka- loids	lycor- ine	
25 March 8 April 15 April 21 April 5 May 15 May 5 June	$\begin{array}{r} 3-6\\ 6-10\\ 10-14\\ 20-25\\ 25-30\\ 30-32\\ 30-32 \end{array}$	$1.3 \\ 1.25 \\ 1.09 \\ 1 \\ 0.77 \\ 0.30 \\ 0,15$	$\begin{array}{c} 0.61 \\ 0.55 \\ 0.52 \\ 0.5 \\ 0.45 \\ 0.1 \\ 0.02 \end{array}$	

Table	2
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	Percentage of of the weight	
Method of extraction of the alkaloids	com- bined alka- loids	lycor- ine
With 1% hydrochloric acid With 1% hydrochloric acid using the cation-exchanger KU-1 in the H	0.72	0,38
form	0.64	0.34
With 1% hydrochloric acid using bentonite KB-1	0.59	0.31
With methylene chloride	0.76	0.40
With chloroform	0.81	0.41
With chloroform at 35-40° C	0.91	0.46

The raw material for the preparation of this substance consists of plants of the genus Ungernia [1,2]. U. trisphaera Rg1. has a particularly high content of this substance (up to 0.6%) [3].

We have studied the dynamics of the accumulation of lycorine according to the vegetation periods in <u>U. trasphaera</u> collected in the Ashkhabad region of the Turkmenian SSR (foothills of the Kopet-Dagh) in 1964 (Table 1). It can be seen from the table that it is desirable to collect the leaves in April and at the beginning of May (length of the leaves 20-25 cm).

In order to develop a method for the isolation of lycorine from the plant we performed extraction by various methods (Table 2).

Consequently, the maximum yield of the product is obtained by extraction with chloroform at 35-40° C.

The comminuted leaves of U. trisphaera (400 g) were wetted with 0.4 l of 5% aqueous ammonia, charged into the extractor, and covered with 1.6 l of chloroform. Extraction was carried out at 35-40° C for 2 hr and then the extract was poured off and more solvent was added. In this way four portions of extract were obtained which were then evaporated to 0.5 l and treated with 10% sulfuric acid until the alkaloids had been extracted completely, after which the acid solution was filtered and made alkaline with 25% ammonia, with cooling. The crystalline precipitate that deposited was separated off, boiled with acetone twice, and filtered off with suction. This gave 1.65 g of a preparation with mp 252-254° C. The alkaline solution was extracted with chloroform and the extract was evaporated. This yielded a further 0.2 g of lycorine.

## REFERENCES

1. S. Yu. Yunusov and Kh. A. Abduazimov, DAN UzSSR, no. 6, 44, 1953.

2. S. Yu. Yunusov and Kh. A. Abduazimov, ZhOKh. 27, 3357, 1957.

3. Kh. Allayarov, Kh. A. Abduazimov, and S. Yu. Yunusov, DAN UZSSR, no. 7, 50, 1962.

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## ISOLATION OF PERFORINE AND FOLIOSIDINE

T. T. Shakirov and L. T. Avazmukhamedov

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The alkaloid perforine has been obtained from the seeds of <u>Haplophyllum perforatum</u> [1]. It possesses valuable pharmacological properties.

We have developed the technical isolation of performe from the defatted seeds of <u>H. perforatum</u> after the extraction of the haplophyllidine [3]. Various organic solvents and weak aqueous solutions of acids were tested for extraction. Of the organic solvents, chloroform gave a good yield of performe (0.1-0.11%).

Satisfactory results were given by extraction with 1% hydrochloric acid or 1% sulfuric acid using a number of cation-exchangers (KU-1, SDV-3T, SBS), of which the best was KU-1.