Table 2

Method of extraction of the alkaloids	Percentage of the weight	
	combined alkaloids	lycorine
With 1% hydrochloric acid	0.72	0.38
With 1% hydrochloric acid using the cation-exchanger KU-1 in the H 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 Rgl. 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. trasphaera 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.

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ISOLATION OF PERFORINE AND FOLIOSIDINE

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

We have developed the technical isolation of perforine from the defatted seeds of H. perforatum 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 perforine (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.

The defatted seeds were extracted with 1% sulfuric acid by steeping. The extraction was carried out for 6 hr 7–8 times. The acid extract was filtered and passed through a column of KU-1 cation-exchanger.

Ammoniacal solutions of alcohols (methanol and ethanol) and also mixtures of organic solvents were used to elute the alkaloids. It was found that a 1.0–1.5% of ammonia in 80–86% ethanol or methanol is a good desorbing solvent. On the basis of the investigation performed, an industrial scheme for the production of perforine from the seeds of H. perforatum has been developed. The yield of combined alkaloids by this scheme is 0.15% and the yield of the perforine 0.07–0.08% of the weight of the dry seeds (from 300 kg of seeds).

We have studied the method of isolating foliosidine from Haplophyllum foliosum [4, 5]. The preparation has proved to be pharmacologically active [6]. At the present time it is at the stage of introduction into medical practice.

The total alkaloids obtained by an adsorption method from the epigeal parts of H. foliosum [5] were separated according to their basicities in a continuous apparatus for polybuffer distribution, but no satisfactory results were obtained. By using the different solubilities of the alkaloids in organic solvents (acetone and ether) we succeeded in isolating the foliosidine (yield 0.015–0.02% of the weight of raw material) and developing an industrial scheme for its production.

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THE STRUCTURE OF VINCANICINE

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By separating the combined ethereal alkaloids of Vinca erecta on a column of Al_2O_3, we have obtained vincanine, vinervidine, and a new base, which we have called vincanicine.

Vinervidine, $C_{19}H_{20}ON_2$, mp 190–191° C (acetone), R_f 0.23 [TLC, SiO_2; benzene–methanol (9 : 1)], $[\alpha]_D^{30}$ ±0° (c 0.6; methanol), has previously been isolated from the roots of this plant [1]. Its UV spectrum $\lambda_{max}^{C_2H_5OH}$ 244, 300, 362 mμ (lg ε 4.08; 3.67; 4.33)] and IR spectrum (743, 1575, 1665, 3220 cm^{-1}) are similar to those of vincanine [2].

The mass spectra of vinervidine and vincanine (table) proved to be identical. Consequently, vinervidine is dl-vincanine.

Substance	m/e (relative intensity, %)			
	M^+	M–15	M–29	C [5]
Vincanine	292 (83)	277 (9)	263 (20)	121 (100)
Vinervidine	292 (62)	277 (9)	263 (17)	121 (100)
Vincanicine	322 (60)	307 (7)	293 (15)	121 (100)

Vincanicine is an amorphous base with R_f 0.3 [TLC on SiO_2; benzene–methanol (9 : 1)], $[\alpha]_D^{24}$ −438.0° (c 0.6; chloroform). Its UV spectrum [$\lambda_{max}^{C_2H_5OH}$ 248, 293, 376 mμ lg ε 3.90; 3.29; 4.04] is similar to that of vincanidine [3].