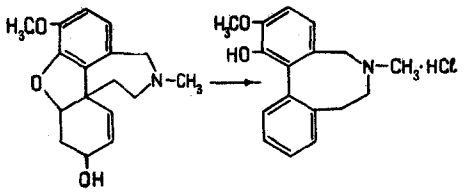

I II

The starting material for this preparation is galanthamine (I) [1–3]. The action of mineral acids on galanthamine forms methylapogalanthamine [4, 5].

Concentration of hydrochloric acid, %	Time of heating						
	1 hr	1 hr 30 min	2 hr	2 hr 30 min	3 hr	3 hr 30 min	4 hr
	Yield of methylapogalanthamine hydrochloride, % of theory						
15	25	35	47	55	65	67	70
20	35	50	68	70	85	84	80
25	40	55	65	71	85	75	71
30	38	52	60	68	80	72	67

To obtain methylapogalanthamine hydrochloride we heated galanthamine hydrobromide with hydrochloric acid (1 : 5) under various conditions (table).

Thus, the maximum yield of the preparation is obtained when galanthamine is heated with 20% hydrochloric acid for 3 hr.

Galanthamine hydrobromide (5.0 g) was heated in the boiling water bath with 25 ml of 20% hydrochloric acid for 3 hr. The crystals that deposited were separated off, the solution was treated with alkali, and the base that separated was converted by means of hydrochloric acid into the hydrochloride. Yield 3.52 g (85%). Recrystallization from ethanol (1 : 2.5) gave 3.3 g of a preparation with mp 163–166° C (with foaming) in the form of white crystals with a slight creamy tinge readily soluble in water and ethanol.

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ALKALOIDS OF DICTAMNUS ANGUSTIFOLIUS

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Dictamnine and skimmianine have previously been found in D. angustifolius (D. albus Linn.) [1]. We have studied the seeds and roots of D. angustifolius collected in the autumn of 1966 in the Angren valley (village of Karabauvsai, Tashkent region). The cleaned seeds (1000 g) were defatted with petroleum ether giving 400 g (40%) of oil. Chloroform extracted 0.025% of total alkaloids from the defatted seeds.

Chromatography of the combined bases on alumina gave three alkaloids which were identified with known specimens: skimmianine, dictamnine, and dubamine [2]. Chloroform extraction of 600 g of the roots of D. angustifolius gave 1.3 g (0.21%) of total alkaloids. Separation of the mixture of bases on alumina yielded dubinidine and haploperine [3] for the first time, in addition to skimmianine, dictamnine, and dubamine.

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ISOLATION OF VINCAMINE

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The hydrochloride of the alkaloid vincamine [1-3] (vinkametrin) is a new preparation approved by the Pharmaceutical Committee of the Ministry of Public Health of the USSR for use in medicine. We have investigated the epigeal part of Vinca erecta with the aim of isolating the alkaloid vincamine by ion-exchange absorption.

The air-dry comminuted raw material (10 kg) was placed in a 40-liter extractor and was covered with 25 *l* of a 1% solution of sulfuric acid. A total of six decantations of 15 *l* each was made. The resulting extract was passed through a column of KU-1 cation exchanger in the hydrogen form (800 g) from the bottom upwards at the rate of 4 *l*/hr. The "breakthrough" of the alkaloids was monitored with Dragendorff's reagent. Then the resin was washed with water and the alkaloids were desorbed with a 1.5% solution of ammonia in 85% ethanol. The alkaloids were eluted at the rate of 0.2-0.3 *l*/hr. The ethanolic eluate (10 *l*) was treated with acetic acid to give pH 2 and the ethanol was distilled off in vacuum (720-730 mm Hg). The solution of the alkaloids (1 *l*) was brought to pH 10 with 25% ammonia and extracted with benzene (6 *l*). The benzene was evaporated in vacuum (720-730 mm) to 200 ml. On standing, the vincamine crystallized out, yield 2 g.

To obtain vincamine from the epigeal part of V. erecta by the adsorption method, more than 100 kg of the plant was treated. It was found that this method is the most suitable for the industrial production of vincametrine.

It was also found that V. erecta collected in the village of Darbaz (Tadzh SSR) is a better raw material for the isolation of vincamine than the plants from the Fergana, Osh, and Tashkent Oblasts. The content of vincamine in the plants from Darbaz amounts to 0.03-0.035% (of the weight of the raw material, i.e., approximately 3-4 times more than in the raw material from the oblasts mentioned.

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