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ISOLATION OF HELIOTRINE BY THE ION-EXCHANGE METHOD

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The alkaloid heliotrine has been isolated from the plants Heliotropium lasiocarpum [1, 2] and H. dasycarpum [3]. The seeds and epigeal part of H. lasiocarpum cause serious poisoning [4]. At the present time, heliotrine is the only agent in experimental medicine for obtaining a true picture of hepatitis and cirrhosis of the liver in experimental animals. We have developed a semiindustrial method for obtaining heliotrine from the plant H. dasycarpum. The process of extracting the alkaloids from the plant material was investigated with various organic solvents, water, and dilute aqueous solutions of acids. The most acceptable results were given by extraction of the plant material with 1% hydrochloric acid. The adsorption of the alkaloids was studied on various cation-exchange resins (KU-1, KU-2, SBS, KB-4P-2, etc.). Good results were obtained with the use of KU-1 resin.

Extraction was carried out with 1% hydrochloric acid in 250-*l* extractors containing 50 kg of comminuted epigeal part of the raw material. Five decantations were carried out, at four-hour intervals. The acid extract of the alkaloids was passed through a battery of adsorbers consisting of three columns (d = 0.15 m, *l* =0.4 m) each containing 2.0–2.2 kg of KU-1 ion-exchanger in the H form with a moisture content of 40–50%. The rate of adsorption varied from 4 to 9 *l*/hr.

The desorbents used were ammoniacal solutions of ethanol and methanol with various pH values, a 1.5% solution of ammonia in 85% ethanol, and an alkaline mixture of chloroform and ethanol (1 : 1). 95% ethanol saturated with gaseous ammonia to pH 5 proved to be a good desorbent. The ethanolic solution obtained from the adsorbers was evaporated in vacuum to dryness. The yield of combined alkaloids was 0.90–0.92% (of the weight of the raw material).

To isolate the heliotrine, the total combined alkaloids were dissolved in 15% hydrochloric acid, and the N-oxide forms of the alkaloids were reduced with zinc dust. Then the mixture was made alkaline with ammonia and extracted with chloroform. The chloroform extract, after drying with potassium carbonate, was distilled in vacuum to dryness. Acetone treatment of the residue gave crystals of heliotrine with mp 125–126° C (from acetone). The yield of heliotrine was 0.40–0.45% (of the weight of the air-dry raw material).

REFERENCES

1. G. P. Men'shikov, Ber., **65**, 974, 1932, **66**, 875, 1933, **68**, 1051, 1555, 1935; Izv. AN SSSR, 969, 1802, 1936.

2. S. Yu. Yunusov and G. P. Sidyakin, DAN UzSSR, no. 1, 3, 1950.

3. S. T. Akaramov, F. Kiyamitdinova, and S. Yu. Yunusov, DAN UzSSR, no. 4, 30, 1961.

4. Proceedings of the Department of Pathological Physiology, Questions of Regional Pathology [in Russian], Tashkent, 1956.

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STUDY OF THE ALKALOIDS OF THE FAMILY BORAGINACEAE

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Heliotropium olgae Bge. The plant was collected at Tashkumyr in the Andizhan region of UzSSR in the flowering and incipient fruit-bearing stage (10 October 1957). The epigeal part yielded 0.85% and the roots 0.32% of combined alkaloids.

The combined alkaloids were separated through their solubility in acetone; heliotrine [1] was isolated. Paper chromatography showed that the mother liquor contained three other alkaloids besides heliotropine, with R_f 0.32, 0.67 and 0.73, which could not be isolated in the crystalline state. The solvent system was butanol–acetic acid–water (20:1:20) and the revealing agent iodine vapor.

Solenanthus coronatus Rgl. We studied four species of plants of the genus Solenanthus [2, 3] and from them we isolated rinderine, turkestanine, and caratégine. From the epigeal part of S. coronatus prepared in Sary-Dasht, Tadzhik SSR in the budding stage (12 June 1964) we obtained 0.82% of a mixture of alkaloids on the total weight of the plant.

The combined alkaloids were separated according to their solubility in acetone. Echinatine [3] was isolated (53% of the total). Paper chromatography showed the presence in the mother liquor of two noncharacterized alkaloids with R_f 0.42 and 0.51.

Lindelofia stylosa (K. et K.) Brand. We have studied various organs (except the seeds) of this plant collected from various sites in the Kirgiz SSR previously [4], isolating three alkaloids. To study this plant we used seed of the 1964 crop (Alai valley, KirgSSR).

The alkaloids were extracted from the defatted seeds with chloroform and then with methanol. This gave 1.34% (from the chloroform extract) and 1.19% (from the methanolic extract) of combined alkaloids referred to the weight of the seeds (a total of 2.53%).

The combined alkaloids isolated by chloroform extraction were separated according to the solubility of the alkaloids in acetone, and viridiflorine [5] and echinatine [3] were obtained. The combined alkaloids obtained by methanolic extraction were chromatographed on alumina. The methanol–chloroform fractions (5:95) and (10:90), respectively. yielded echinatine N-oxide [6] and viridiflorine N-oxide [4].

By paper chromatography, the mother liquor was found to contain a mixture of these four bases.

REFERENCES

1. G. P. Men'shikov, Ber., 65, 974, 1932.
2. S. T. Akramov, F. Kiyamitdinova, and S. Yu. Yunusov, DAN UzSSR, no. 10, 29, 1962.
3. S. T. Akramov, A. S. Samatov, and S. Yu. Yunusov, DAN UzSSR, no. 6, 28, 1964.
4. S. T. Akramov, F. Kiyamitdinova, and S. Yu. Yunus v, DAN UzSSR, no. 6, 35, 1961.
5. G. P. Men'shikov, ZhOKh, 16, 1311, 1946; 17, 343, 1947; 18, 1736, 1948.
6. S. T. Akramov, F. Kiyamitdinova, and S. Yu. Yunusov, DAN UzSSR, no. 4, 35, 1965.

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CONDENSATION PRODUCT OF LUPININIC ACID WITH PIPERIDINE

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The condensation product of piperidine with lupininic acid, obtained from the alkaloid lupinine, a crystalline substance with mp 240–241° C and $[\alpha]_D$ +17.7°, has been described previously [1].

A further study of the reaction products has shown that the product of the condensation of lupininic acid with piperidine is a liquid with bp 228–230° C (5 mm), $[\alpha]_D$ +54.2° (c 2.13; ethanol), R_f 0.78, composition $C_{15}H_{26}N_2O$, melting point of the perchlorate 199–200° C (from water) and of the hydrochloride 114–115° C (from acetone).

The crystalline substance that we had isolated previously [1] proved to be a mixture of the hydrochlorides of the condensation product and piperidine.