

ISOLATION OF HYOSCYAMINE AND SCOPOLAMINE
FROM THE EPIGEAL PART OF *Scopolia*
tangutica

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The highest content of tropane alkaloids has been found in the epigeal part of *Scopolia tangutica* Maxim. (*Tangut scopolia*) [1, 2], in which hyoscyamine and scopolamine have been identified [3, 4].

We have used the epigeal part of the *scopolia* collected in the flowering stage (nursery of the Komarov Botanical Institute, Academy of Sciences of the USSR). The amount of combined alkaloids in the raw material was 0.55% (absolutely dry raw material). Their qualitative and quantitative composition was determined by chromatographic methods [5-8]. It was found that the combined alkaloids consist of 54% of hyoscyamine, 31% of scopolamine, and 14% of an unidentified base.

Scopolia ground to 3-5 mm was extracted by percolation after previous steeping, by the vortex method, and by continuous countercurrent extraction in a spring-blade extractor [9] with distilled water. The continuous countercurrent method of extraction is efficient. The yield with a rate of feed of the raw material of 0.085 kg/h was 92%. When 0.5% solutions of hydrochloric and sulfuric acids were used as the extractant, the yield of alkaloids rose slightly, but the amount of extractive substances of nonalkaloid nature increased simultaneously.

In order to select the sorbent for the subsequent isolation and purification of the alkaloids, the sorption properties of various brands (domestic and foreign) of strongly acid cation-exchange resins in the H form were studied. The most highly selective sorption of the alkaloids from the extracts was found on KU-5 cation-exchange resin with a swelling coefficient of 2.4. Since KU-5 cation-exchange resins with $K_{sw} = 2.4$ possess a high selectivity and good kinetics, we used them for the isolation of the alkaloids from the extracts obtained by the continuous countercurrent method with a concentration of alkaloids of 0.03-0.45%. The optimum pH was 5.2 (initial extract).

At a rate of passage of the extract of 0.250 liter/cm² · h, the capacity of KU-5 cation-exchange resin was 0.72 meq/g, that of SBS-3 0.44 meq/g, and that of KU-1 0.21 meq/g.

Desorption was performed with 1.5-3% solutions of ammonia in methanol, ethanol, and isopropanol of various concentrations. A good eluent was 80% ethanol containing 1.5% of ammonia at a rate of flow of 0.090 liter/cm² · h, giving a yield of 95%.

The eluate was acidified with 10% hydrochloric acid to pH 5.5 and decolorized with type "A" activated carbon with stirring for 30 min at 45°C. The ethanol was distilled off from the filtered eluate under vacuum (650 mm Hg).

The aqueous extract was brought to pH 6 with ammonia, phosphate buffer with pH 6.4 was added, and it was extracted with chloroform [10, 11]. After the separation of the scopolamine, the hyoscyamine remained in the aqueous layer, and this was extracted with chloroform after the pH had been brought to 9-10.

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The completeness of the separation was monitored by paper and thin-layer chromatography.

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