

*Scopolia tangutica* Maxim., which is cultivated at the Scientific-Research Station of the V. L. Komarov Botanical Institute of the Academy of Sciences of the USSR in the Leningrad oblast, is a plant with a high content of alkaloids [1-3].

We have studied the alkaloid composition of the roots of five-year-old plants collected in September, 1969 (fruit-bearing phase). The total amount of alkaloids, found by the weight method [4], was 1.56% (of the absolutely dry weight of the raw material), the tropane fraction (by Reimers' method [5]) being 32.8% of the total. By ascending chromatography on paper (German rapid-filtering paper of types FN-1 and FN-11 in the butan-1-ol-acetic acid-water (4:1:5) system [6], the spots being revealed by the Dragendorff-Munier reagent [7]) and by chromatography in a thin nonfixed layer of "basic" alumina (activity grade IV) [8] [chloroform-methanol (20:1) [9], the spots being revealed with iodine vapor and the Dragendorff-Munier reagent] we showed the presence of hyoscyamine, scopolamine, cuscohygrine, tropine, and two identified bases [10].

To separate the mixture of alkaloids isolated by the extraction method [4], we used a combined process consisting in countercurrent liquid extraction and vacuum distillation. By selective countercurrent extraction at pH 6.7 with a buffer solution [11], we isolated the tropane fraction (hyoscyamine and scopolamine, yield 31.6%) from the liquid fraction (cuscohygrine and two unknown alkaloids, yield 66.8%). At pH 6.4 [11], the scopolamine in the mixture of tropane alkaloids (16.7%) was separated from the hyoscyamine (81.4%) [12]. (The pH of the solutions was checked on an LPU-01 potentiometer; the fractions were extracted with chloroform, and the process was monitored by thin-layer and paper chromatography.)

The liquid fraction was subjected to vacuum distillation. Three separate fractions were obtained: I - at 38-44°C/1.5 mm, yield 3.9%; II - at 97-104°C/1.5 mm, yield 86.7%; and III - at 139-144°C/1.5 mm, yield 4.9%. For purification, the fractions were redistilled. Fraction I deposited crystals in the form of needles with mp 79-80°C which, after washing with ether, gave on paper and thin-layer chromatography spots with low  $R_f$  values (on paper,  $R_f$  0.13, the  $R_f$  value of cuscohygrine being 0.20, that of scopolamine 0.51, and that of hyoscyamine 0.62; on alumina  $R_f$  0.36, the  $R_f$  value of cuscohygrine being 0.79, that of scopolamine 0.69, and that of hyoscyamine 0.58). Fraction II [a light yellow liquid,  $d_4^{20}$  0.9767,  $n_D^{20}$  1.4852, melting point of the nitrate 204-205°C (decomp); a mixture with a cuscohygrine nitrate reference sample gave no depression] formed spots with  $R_f$  values corresponding to those of cuscohygrine, and fraction III (a yellowish-brown viscous oil,  $n_D^{20}$  1.5219) formed spots with high  $R_f$  values (on paper chromatography,  $R_f$  0.65, on thin-layer chromatography 0.82).

## LITERATURE CITED

1. A. A. Ryabinin and M. N. Semenova, *Zh. Obshch. Khim.*, **25**, No. 1, 181 (1955).
2. S. A. Minina, *Med. Prom. SSSR*, **9**, 11 (1958).
3. A. V. Aleksandrova, *Planta Medica*, **9**, No. 1, 94 (1961).
4. F. I. Burmistrov, N. I. Libizov, V. N. Murav'eva, and B. S. Nikolskaya, in: *Himalayan Scopolia* [in Russian], Moscow (1953), p. 69.
5. F. Reimers, *Quart. J. Pharm. Pharmacol.*, **21**, No. 4, 470 (1948).

Leningrad Chemical and Pharmaceutical Institute. Translated from *Khimiya Prirodnikh Soedinenii*, No. 3, pp. 379-380, May-June, 1971. Original article submitted January 4, 1971.

© 1973 Consultants Bureau, a division of Plenum Publishing Corporation, 227 West 17th Street, New York, N. Y. 10011. All rights reserved. This article cannot be reproduced for any purpose whatsoever without permission of the publisher. A copy of this article is available from the publisher for \$15.00.

6. I. M. Hais and K. Macek, Paper Chromatography [Russian translation from Czech], Moscow (1962), p. 121.
7. R. Munier and M. Macheboeuf, Bull. Soc. Chim. Biol., 32, No. 3 192 (1950).
8. A. A. Akhrem and A. I. Kuznetsova, in: Thin-layer Chromatography [in Russian], Moscow (1964).
9. L. N. Slepova, B. L. Moldaver, and A. M. Khaletskii, Tr. Leningrad. Khim.-Farm. Inst., 28, 69 (1969).
10. I. A. Barene and S. A. Minina, Tr. Leningrad. Khim.-Farm. Inst., 24, 88 (1969).
11. State Pharmacopoeia of the USSR [in Russian], 10thed., Moscow (1969), p. 789.
12. V. M. Bashilova and N. I. Figurovskii, Apteknoe Delo, 3, 31 (1963).