- 2. V. M. Rodionov, Selected Works [in Russian], Moscow (1958), p. 39.
- 3. C. Weygand, Organisch-Chemische Experimentierkunst [Russian translation from the German], Moscow, Vol. 2 (1952), p. 622.
- 4. T. Makao and T. Mutsuo, Yakugaku Zasshi, J. Pharm. Soc. Jpn., 85, 77 (1965).

ALKALOIDS OF Rauwolfia littoralis

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It has been reported previously that 3.5-3.8% of total alkaloids and 0.03% of reserpine accumulate in the roots of <u>Rauwolfia littoralis</u> Pierre ex Pitard (<u>R. indochinensis</u> M. Pichon, <u>R. macrocarpa</u> Standl) [1, 2]. Continuing a chemical study of this plant, we have investigated the bark of the roots and have isolated six alkaloids.

The dried and comminuted root bark was wetted with 10% ammonia solution, and the alkaloids were extracted with 96% C_2H_5OH . The extract was 2/3-evaporated in vacuum, acidified with 10% HCl, and filtered. The residue was heated with 20% CH_3COOH , and the mixture was cooled and filtered. The acetic acid solution was washed with petroleum ether to eliminate fats. The ether was driven off from the acetic acid solution by heating in the water bath. After this, the alkaloids were extracted from the acetic acid solution with C_6H_6 . The solvent was distilled off in vacuum to a dry residue, which was dissolved in $CHCl_3$, and the solution was neutralized with 10% ammonia to pH 7. Then it was stirred with a small amount of Al_2O_3 , and the $CHCl_3$ was driven off by blowing with air. The mixture was transferred to a chromatographic column of Al_2O_3 and chromatographed. This led to the isolation of substance (I) — yellowish (30 mg/500 g; 0.006%), $C_{33}H_{40}N_2O_9$, mp 260-262°C; UV spectrum: $\lambda_{max} C_2H_5OH$ (nm): 223, 268, 295; λ_{min} 247. IR spectrum: $\lambda_{max} P^{araffin}$ oil (cm⁻¹): 3435, 1735, 1715, 1500, 1590, 1125. Substance (I) was identified as reserpine [3].

The filtrate was treated with CHCl₃; the chloroform fraction (A) contained a mixture of weak bases. The acid solution was made alkaline to pH 8 and was filtered and extracted with CHCl₃ (fraction B). The mother solution was alkalinized to pH 10-11 and was extracted with ethyl acetate, giving fraction C of strong bases. The chromatography of fraction A on a column of Al_2O_3 with elution of the alkaloids by ethyl acetate and ethyl acetate—ethanol yielded substances (II), (III), (IV), and (V).

Substance (II) with the composition $C_{42}H_{44}N_{4}O_{6}$, mp 260-265°C, $\lambda_{max}^{}C_{2}H_{5}OH$ (nm): 225, 258, 292, 307, 370, was serpentinine [4, 5]. Yield: 200 mg/500 g; 0.04%.

Substance (III) with the composition $C_{21}H_{20}N_2O_3$, mp 203-205°C, $\lambda_{max}C_2H_5OH$ (nm): 252, 307, 370; λ_{min} 282, 327, was identified as alstonine [5]. Yield 25 mg/500 g; 0.005%.

Substance (IV) (yield: 10 mg/500 g; 0.002%), mp 323-325°C; $\lambda_{\rm max}^{\rm C_2H_5OH}$ (nm): 224, 257, 307; was not identified.

Substance (V) consisted of an oily liquid (1 ml/0.5 g; 0.1%). $\lambda_{max}^{C_2H_5OH}$ (nm): 241. It was not identified.

Substance (VI), isolated from fraction C, was yellow. Yield: 100 mg/500 g; 0.02%, mp 170-175°C. $\lambda_{\text{max}}^{\text{C}_2\text{H}_5\text{OH}}$ (nm): 253, 307, 368; λ_{min} 281 was serpentine [5, 6].

The structures of all the compounds isolated were confirmed by the results of UV and IR spectroscopies. Ajmaline was detected chromatographically in fraction B. This is the first time that any of these alkaloids have been isolated from R. littoralis.

LITERATURE CITED

 B. P. Korzun, A. F. St. Andre, and P. R. Ulfshafer, J. Am. Pharm. Assoc. Sci. Ed., 46, No. 12, 720 (1957).

Leningrad Institute of Pharmaceutical Chemistry. Translated from Khimiya Prirodnykh Soedinenii, No. 2, pp. 282-283, March-April, 1990. Original article submitted May 31, 1989; revision submitted September 25, 1989.

- 2. D. L. Nhieu, N. Tap, and N. K. Can, Duoc Hoc, No. 6, 12 (1984).
- 3. N. Norbert, H. E. Boar, and J. W. Forbes, J. Am. Chem. Soc., 76, 2463 (1954).
- 4. C. Djerassi and J. Fishman, Chem. Ind. (London), No. 22, 627 (1955).
- 5. E. Schlittler, H. U. Huber, F. E. Bader, and H. Zahnd, Helv. Chim. Acta, 37, 1912 (1954).
- 6. M. S. Habib and W. E. Court, Planta Med., <u>25</u>, No. 4, 331 (1974).

COMPONENTS OF THE LEAVES OF Haplophyllum ramosissimum

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Continuing a systematic study of plants of the genus $\underline{\text{Haplophyllum}}$ [1], we have subjected to chemical investigation the leaves of $\underline{\text{H. ramosissimum}}$ Vved., gathered by one of us in the budding and incipient flowering phase in the Ustyurt desert near Kosbulak.

The dry comminuted raw material (680 g) was extracted with methanol. The total alkaloids (0.35 g; 0.05% of the weight of the dry leaves) were obtained from the evaporated extract in the usual way [2], and their chromatography on silica gel gave the alkaloids evoxine (210 mg), mp $154-155^{\circ}$ C (methanol), methylevoxine (28 mg), mp $122-123^{\circ}$ C (ether), and acetylevoxine (32 mg), mp $161-162^{\circ}$ C (acetone), and also the coumarins scoparone (30 mg), mp $141-142^{\circ}$ C (acetone) and obtusinin (20 mg), mp $140-141^{\circ}$ C (acetone) [3].

This is the first time that obtusinin has been isolated from this plant.

By chromatography on silica gel, the neutral fraction of the methanolic extract, yielded the coumarins scoparone (52 mg), obtusinin (30 mg), and scopoletin (17 mg), mp 199-200°C (acetone), and also cinnamide (10 mg), mp 144-155°C, shown to be identical in its spectral characteristics with a sample isolated from Reseda luteola [4].

All the substances with the exception of the cinnamide were identified by direct comparison with authentic samples.

This is the first time that cinnamide has been isolated from a plant of the family $\underline{\text{Rutaceae}}$.

LITERATURE CITED

- 1. I. A. Bessonova and S. Yu. Yunusov, Khim. Prir. Soedin., 47 (1989).
- 2. I. A. Bessonova, D. Kurbanov, and S. Yu. Yunusov, Khim. Prir. Soedin., 46 (1989).
- 3. A. D. Matkarimov, É. Kh. Batyrov, V. M. Malikov, and E. Seitmuratov, Khim. Prir. Soedin., 328 (1980).
- 4. K. L. Lutfullin, M. M. Tadzhibaev, V. M. Malikov, U. A. Abdullaev, and U. Rakhmankulov, Khim. Prir. Soedin., 826 (1977); M. M. Tadzhibaev, Author's Abstract of Dissertation for Candidate of Chemical Sciences [in Russian], Tashkent (1978).

Institute of the Chemistry of Plant Substances, Uzbek SSR Academy of Sciences, Tashkent. Translated from Khimiya Prirodnykh Soedinenii, No. 2, pp. 284, March-April, 1990. Original article submitted June 8, 1989.