LUPININE ALKALOIDS FROM *Sophora alopecuroides*

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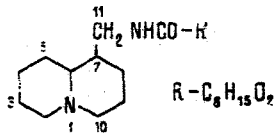
The plant *Sophora alopecuroides* L. (family Legumineosae), which is rich in alkaloids, has been widely studied [1-7]. From 4 kg of the air-dry plant (collected in the village of Toktagul, Kirghiz SSR, in August, 1976) by extraction with methanol we have isolated 60 g of total bases (yield 1.5%). From a benzene extract of the combined material (22 g), by chromatography on a column (alumina, activity grade (II), 50:1, eluents chloroform and chloroform–methanol (100:1, 100:5, 10:1, 2:1, and 1:1)) we have isolated sophoridine, (0.51%), dehydrosophoridine (0.0075%), matrine (0.12%), sophocarpine (0.15%), neosophoramine (0.06%), sophoramine (0.081%), aloperine (0.045%), cytisine (0.0075%), and methylcytisine (0.005%). From a chloroform extract (6 g) by column chromatography (cellulose (1:1) with butan-1-ol–acetic acid–water (100:15:27) (system 1) as eluent) we have isolated sophoridine N-oxide (0.23%), matrine N-oxide (0.09%), and sophocarpine N-oxide (0.16%).

The mother liquor after the extraction of the combined alkaloids with benzene and chloroform was evaporated to dryness in a rotary evaporator. The dry residue was treated with methanol. When the methanolic fraction of the mother liquor was separated on a column of cellulose (system 1), new bases were isolated: (8) with mp 59-60°C, $[\alpha]_D^{23}$ −18.9° (c 0.98; ethanol), M^+ 338, and (9) with M^+ 296.

The mass spectrum of base (8) had the peak of the molecular ion M^+ 338 and the peaks of ions with m/e M−15(4%), 296(7%), 265(7%), 168(21%), 167(3%), 152(30%) and ions with m/e 151(40%), 150(24%), 149(6%), 138(100%), 136(20%), 124(10%), 111(38%), 110(42%), 109(15%), 98(19%), 96(23%), 84(19%), 83(50%), which are characteristic for the lupinine alkaloids [8, 9]. The maximum (100%) intensity of the ion with m/e 138 shows the presence of a trans-quinolizidine system, which was confirmed by the presence of a Bohlmann band in the IR spectrum. The IR spectrum also contained strong absorption bands at 1150 and 1170 cm^{-1} corresponding to a —C—O—C— fragment, at 1524 cm^{-1} corresponding to free NH bonds, and at 1605 and 1683 cm^{-1} corresponding to the stretching vibrations of a secondary amide group (—NH—CO—). In the 3080 and 3300 cm^{-1} regions there are bands characteristic for the stretching vibrations of an associated NH bond of a secondary amide group.

In the PMR spectrum of base (8) there is a broadened one-proton triplet at 6.08 ppm. A two-proton triplet at 3.95 ppm has the splitting constant J = 6 Hz. The chemical shift and the nature of the splitting of this signal indicated the presence of a $-OCH_2-CH_2-$ fragment in the molecule. Another two two-proton multiplets are located at 2.9-3.4 and 3.62 ppm. The latter signal is characteristic for the H_{2e} and H_{10e} protons of lupinine derivatives. In addition, a three-proton triplet at 0.89 ppm shows the presence of a $-CH_2CH_2CH_3$ chain.

A consideration of the spectral characteristics permits the assumption of a lupinine skeleton in the molecule of base (8) and the suggestion of the partial structure (I) for it.



R-$C_8H_{15}O_2$

I

As spectral analysis showed, base (9) also has a lupinine skeleton and differs from base (8) only by the substituent R = $C_5H_9O_2$.

Thus, new alkaloids having a lupinine skeleton have been isolated from the plant *S. alopecuroides* L.

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MONITORING OF THE PURITY OF ANABASINE HYDROCHLORIDE BY THIN-LAYER CHROMATOGRAPHY

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Anabasine hydrochloride has been recommended by the Pharmacological Committee of the Ministry of Public Health of the USSR for manufacture and wide medicinal use. The starting material for the production of anabasine hydrochloride is N-nitrosoanabasine, isolated from technical anabasine sulfate by nitrosation [1]. The possibility of the presence of N-nitrosoanabasine in a pharmacopoeial preparation of anabasine hydrochloride must be avoided. The chromatographic analysis of N-nitroanabasine in a pharmacopoeial preparation of anabasine hydrochloride in a nonfixed layer of alumina has been described only in a provisional pharmacopoeial paper [2]. The main disadvantage of this method is the inconvenience of its performance, which is connected with the free-flowing nature of the alumina and its low sensitivity for the determination of N-nitrosoanabasine (500 μg).

There have been no publications devoted to the thin-layer chromatography of anabasine hydrochloride in a fixed layer of silica gel. Consequently, we have made a choice of the optimum system of solvents and of the amount of deposited substance.

For thin-layer chromatography we used type KSK silica gel prepared by the method of the State Pharmacopoeia [3]. Silica gel (2 g) and gypsum (0.1 g) were carefully mixed with distilled water (8 ml), and a uniform layer was deposited on a glass plate with dimensions of 50×200 mm. The prepared plate was activated by drying at room temperature for 24 h. On the starting line, 125 μg of the preparation in the form of a solution in methanol was deposited as a spot. Chromatography was carried out by the ascending method in a cylindrical chamber (80×200 mm). The plate was treated with the Dragendorff reagent. The following solvent systems were used: 1) chloroform–methanol (2:1), 2) chloroform–methanol–acetone (25:7:3), 3) ether–methanol (50:1), and 4) benzene–methanol–acetone (10:5:2). The best separation was achieved in system 2. The selected solvent system permits the separation of anabasine hydrochloride (R_f 0.35) from N-nitrosoanabasine (R_f 0.92). The minimum detectable amount of anabasine hydrochloride is 1 μg and of N-nitroanabasine 1.2 μg.

The chromatographic method in a thin layer of sorbent that we have developed is recommended for introduction into the draft Pharmacopoeia paper on anabasine hydrochloride and the Provisional Pharmacopoeial paper on anabasine hydrochloride – a standard as a test for the purity of the latter.

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