juziphine, domesticine, isoboldine, corytuberine, stylopine, sendaverine, and the new alkaloids sendaverine N-oxide and juziphine N-oxide have been isolated from the epigeal part of *Corydalis gortschakovii* growing in the upper regions of the R. Pskem.

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ALKALOIDS OF *Delphinium iliense*

M. G. Zhamierashvili, V. A. Tel'nov, M. S. Yunusov, and S. Yu. Yunusov

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We have investigated the epigeal part of *Delphinium iliense* Huth collected in the budding and incipient flowering stage in the Trans-Ili Ala-Tau on the R. Purgen'. An unidentified alkaloid with mp 192.5-193°C has been isolated from this plant previously [1].

Chloroform extraction of the epigeal part of *D. iliense* yielded 0.24% of combined alkaloids, from which we isolated delcorine, lycoctonine, eldeline, a base (I), and a base with mp 141-143°C (II).

The mass spectrum of (I) was characteristic for diterpene alkaloids with a lycoctonine skeleton and had the maximum peak M − 31 [2]. The IR spectrum of (I) contained an absorption band at 1750 cm^{-1} due to a carbonyl group in a five-membered ring, and the NMR spectrum contained the signals from an N-ethyl group (1.03 ppm, 3 H, triplet), from four methoxy groups (3.23, 3.26, 3.30, 3.35; 3 H each, singlets), and from a methylenedioxy group (5.03 and 5.46 ppm; one-proton doublets, J = 1.5 Hz). Such a difference in the chemical shifts of the protons of a methylenedioxy group is observed in lycoctonine alkaloids for a C_7—C_8 methylenedioxy group when a keto group is present at C_6 [3].

A comparison of (I) with dehydrodelcorine [3] showed that these compounds were identical. Because of the small amount of (I) it was impossible to obtain it in the crystalline form. This is the first time that dehydrodelcorine has been isolated from a plant.

The base (II), having the composition $C_{25}H_{37}NO_7$, proved to be new and we have called it ilidine.

Its spectral characteristics enable (II) to be assigned to the lycoctonine group of alkaloids. The presence in the mass spectrum of the maximum ion M − 31 shows that there is a methoxy group at C-1 [2]. The IR spectrum of (II) shows the absorption bands of a hydroxy group (3445 cm^{-1}) and of a carbonyl group (1745 cm^{-1}). According to its NMR spectrum, (II) contains an N-ethyl group (1.02 ppm, triplet, J = 7 Hz), three methoxy groups (3.25, 3.27, 3.31 ppm, 3 H each, singlets), and a methylenedioxy group (5.07 and 5.53 ppm, poorly resolved one-proton doublets, J = 1.5 Hz). Consequently, the developed formula of ilidine can be given in the following form: $C_{18}H_{20}$ (N—C_2H_5) (OH) (CO) $(OCH_3)_3$ (CH_2O_2).

In its spectral characteristics (II) is similar to dehydrodelcorine, and its composition differs from that of dehydrodelcorine by a methylene group. The methylation of the base with methyl iodide in the presence of sodium hydride gave O-methylilidine, which was identical with dehydrodelcorine.

In order to determine the position of the hydroxy group, ilidine was acetylated with acetic anhydride in the presence of pyridine, which gave a monoacetyl derivative (III), the NMR spectrum of which contained the signal of an acetyl group (1.99 ppm, 3 H, singlet), and

Institute of the Chemistry of Plant Substances, Academy of Sciences of the Uzbek SSR, Tashkent.

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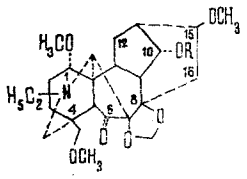
a poorly resolved triplet with an intensity of one proton unit at 4.74 ppm (J = 5 Hz).



I R = CH_3; II R = H; III R = $OCOCH_3$

The chemical shift and the nature of the splitting of the signal correspond to a proton at C_{10} geminal to an acetyoxy group [4, 5]. Consequently, the hydroxy group in ilidine is present at C_{10} and it has the structure (II).

EXPERIMENTAL

The homogeneity of the substances was checked by chromatography in a thin layer of type ShSK silica gel in the benaene–methanol (4:1) system. The IR spectra were taken on a UR-20 instrument in KBr tablets and in chloroform, the NMR spectra (deuterochloroform) on a JNM-4H-100/100 MHz instrument with HMDS as internal standard (the values are given in the δ scale), and the mass spectra on an MKh-1303 instrument fitted with a system for direct introduction into the ion source.

Isolation of the Alkaloids. The comminuted epigeal part of *D. iliense* (48 kg) was moistened with a 5% solution of sodium carbonate and extracted with chloroform six times. The combined chloroform extracts were treated with 5% sulfuric acid. The acid solution was washed with ether and was made alkaline with sodium carbonate, with cooling. Extraction with ether yielded 96.82 g and with chloroform 17.58 g of mixed alkaloids, this amounting to 0.24% of the weight of the air-dry plant.

When the ethereal extract of the mixture of bases was treated with acetone, 14.5 g of a base with mp 200-202°C (acetone) separated out, and this was identified by its mass, IR, and NMR spectra and a mixed milting point as delcorine.

The material from the residual mother liquor was dissolved in ethanol and the solution was acidified with ethanolic perchloric acid. On cooling, the perchlorate of a base with mp 179-180°C (29.94 g) separated out. The alkaloid obtained from the perchlorate was identified as lycoctonine.

The perchlorates from the mother liquor were dissolved in water and the solution was made alkaline with sodium carbonate and was extracted successively with hexane, ether, and chloroform. The hexane and ether extracts, on treatment with acetone, yielded a base with mp 189-190°C (10.54 g) which was identified spectrally and also by comparison with an authentic sample as eldeline.

The residue was chromatographed on a column of alumina, and by elution with ether a base (I) (0.04 g), mol. wt. 477, was isolated. Then the mother liquor from base (I) was separated on a column of silica gel with elution by benzene–methanol (100:1); ilidine was isolated with mp 141-143°C (0.085 g) mol. wt. 463.

Ilidine Acetate (III). A solution of 70 mg of the base in 4 ml of acetic anhydride and 0.5 ml of pyridine was left at room temperature for 72 h. The excess of acetic anhydride was eliminated on a rotary evaporator, the residue was dissolved in ice water, the solution was made alkaline with sodium carbonate, and the reaction product was extracted with ether. This gave amorphous ilidine monoacetate, mol. wt. 505.

O-Methylilidine (I). Ilidine (60 mg) was boiled in dioxane in the presence of sodium hydride and 2 ml of methyl iodide for 7 h. After cooling, the solution was separated from the deposit and the solvent was evaporated off, giving a residue which was dissolved in 5% sulfuric acid. The acid solution was washed with ether, made alkaline with sodium carbonate with cooling, and the (I) was extracted with ether. Dehydrodelcorine with mp 133-134°C was isolated.

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ISOLATION AND PURIFICATION OF gamma-GLOBULIN FROM BLOOD PLASMA AND SERUM BY ABSORPTION ON AMINOSILOCHROME

M. A. Tel'nova, T. S. Kotova,
S. V. Belyaev, and V. M. Stepanov

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Aminosilochrome [1, 2] — a derivative of macroporous silica containing covalently bound alipatic amino groups

$$-O-\overset{|}{\underset{|}{Si}}-CH_2-CH_2-CH_2-NH_2,$$

— may be used in the chromatography of proteins as an anion-exchange resin which is favorably distinguished from the ion-exchange resins based on cellulose that are usually used by simplicity of preparation, stability to a number of chemical and microbiological effects, and, which is particularly important, by a low hydrodynamic resistance. Another feature of aminosilochrome that is of practical importance is the very high rate of settling of aqueous suspensions of this sorbent which make it particularly convenient for the sorption of proteins under static conditions — a process which is distinguished by rapidity and is applicable to mixtures of extremely complex composition.

In the present work we consider the use of selective sorption on aminosilochrome under static conditions to isolate and purify the gamma-globulins of blood.

Chromatography on columns of DEAE-cellulose [3] or other ion-exchange resins or sorption from solutions on the same ion-exchange resins under static conditions [4] is frequently used to obtain pure gamma-globulins.

The conditions for the sorption of the gamma-globulins on aminosilochrome under static conditions are basically similar to those developed for the column chromatography of these proteins on aminosilochrome [2]. The sorption of the gamma-globulins on aminosilochrome is performed at pH 7.5, and the sorbent binds not only the gamma-globulins but the albumins of the serum and some other proteins. At the same time, a considerable part of the proteins — fraction I, amounting to 46% of their total amount — is not bound at this pH and is readily

TABLE 1. Chromatography of Human Blood Serum on Aminosilochrome C-80

Fraction	Desorption conditions	Volume, ml	Protein content	
			opt. units	%
Initial serum	—	0.7	24,5	100
I	0.01 M phosphate buffer, pH 7.5	69	11,3	46
II	0.01 M acetate buffer, pH 4.5	40	6,0	24,6
III	1 M HCl	65	7,3	29,6
Total				100,2

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