

THE QUESTION OF THE ISOLATION OF PSORALEN FROM THE SEEDS  
OF Psoralea drupaceae Bge.

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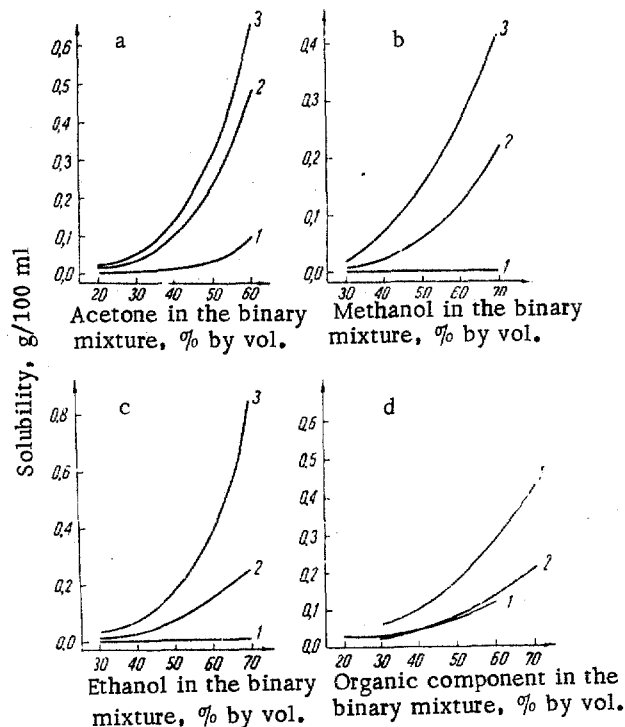
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Psoralen has been used successfully to treat the disease of vitiligo [1]. It is isolated from Psoralea corylifolia L. and Ps. drupaceae Bge. [2, 3]. According to the order drawn up by the Institute of Chemistry and Plant Substances AS UzSSR, the Tashkhimfarmzavod have set up the production of the material from the roots of Ps. drupaceae. However, there is 2-2.5 times as much psoralen in the seeds of this plant as in the roots (about 1%) [3].

To develop an industrial method for obtaining psoralen from the seeds of Ps. drupaceae it was necessary to find a selective solvent which would enable this substance to pass into the extract without extracting the fatty and resinous substances [4]. For this purpose we have determined the solubility of psoralen both in pure solvents (table) and in some binary mixtures in which fats and resins are practically insoluble.

The binary mixtures consisted of the following components:  
1) water-acetone; 2) water-methanol; 3) water-ethanol. The acetone content of the binary mixture was varied from 20 to 60% and that of methanol and ethanol from 30 to 70%. The solubility of the psoralen in these mixtures was determined at 25, 50, and 60°C and the boiling point of the mixture under investigation (figure).

Solvent	Solubility of psoralen, g/100 ml of solvent	
	25°C	at bp
Acetone	1.30	4.00
Methanol	0.40	3.90
Ethanol	0.26	5.00
Benzene	1.10	2.77
Chloroform	6.00	12.50
Gasoline (extraction)	0.0006	0.20
Methanol: gasoline (3:2)	0.33	2.00



Solubility of psoralen in mixtures in relation to the temperature and content of the organic component: a) in mixtures of water and acetone; b) in mixtures of water and methanol; c) in mixtures of water and ethanol: 1, 2, 3) at 25, 50, and 60°C, respectively; d) in binary mixtures of water and organic solvents at the boiling point: 1, 2, 3) aqueous acetone, methanol, and ethanol, respectively.

Consequently, selective extraction from plant raw material (without an appreciable extraction of the accompanying fat-like and resinous substances) can be carried out with aqueous solutions of acetone, ethanol, and methanol.

We have extracted the seeds with aqueous acetone and ethanol on the semi-industrial scale. Up to 80% of the psoralen present in the seeds was isolated.

#### REFERENCES

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#### STRUCTURE OF LEONTALBINE

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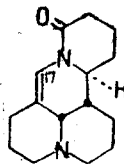
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We have extracted the alkaloids taspine, N-methylcytisine, and leontalbine from the epigeal parts of Leontice alberti Bge. [1]. The reduction of leontalbine ( $C_{15}H_{22}N_2O$ ) with lithium aluminum hydride in ether gave deoxyleontalbine  $C_{15}H_{24}N_2$  with mp 54-56°C (from absolute ether). The IR spectrum of the latter lacks an absorption band of an amide carbonyl group.

The hydrogenation of deoxyleontalbine in alcohol in the presence of a platinum catalyst led to deoxydihydroleontalbine  $C_{15}H_{26}N_2$  with mp 60-61°C,  $[\alpha]_D + 13.8^\circ$  (c 0.1; ethanol), picrate with mp 254-256°C, hydriodide with mp 306-308°C, hydrochloride with mp 328-330°C. The properties of deoxydihydroleontalbine are similar to those of deoxymatrine [2] and differ from it only by the sign of the specific rotation. The melting points of mixtures of the picrates and hydriodides are 4-5° higher than for the individual salts. Thus, deoxydihydroleontalbine is the enantiomorphic form of deoxymatrine.

Leontalbine perchlorate is not hydrogenated by sodium borohydride, while deoxyleontalbine perchlorate is readily hydrogenated, forming deoxydihydroleontalbine. This property shows that the double bond in leontalbine is located between the  $\alpha$  and  $\beta$  carbon atoms relative to an inert nitrogen atom. The NMR spectrum of leontalbine has the signal of one olefinic proton in the  $C_{17}$  position ( $\delta = 6.9$  ppm) in the form of a singlet [3, 4].

Consequently, the double bond in the leontalbine molecule is located between carbon atoms 5 and 17, and the structural formula of this base will therefore have the form



A compound with this structure was obtained from matrine by Bohlmann, who called it 5, 17-dehydromatrine [5]. For comparison, we synthesized 5, 17-dehydromatrine, modifying the reaction conditions. Matrine was dehydrogenated with mercuric acetate. The resulting 5-hydroxy-6, 7-dehydromatrine was hydrogenated with hydrogen in alcohol in the presence of a Raney Ni catalyst. The yield of 5-hydroxymatrine was twice as high as that given in the literature [5]. By the reaction of phosphorus pentoxide, the 5-hydroxymatrine was converted into 5, 17-dehydromatrine, the perchlorate of which melted at 245°C,  $[\alpha]_D + 128.2^\circ$  (c 0.73; ethanol). Leontalbine perchlorate has mp 247°C,  $[\alpha]_D - 131.2^\circ$  (c 0.82; ethanol). A mixture of the perchlorates melted at 245-247°C and their IR spectra coincided completely.

Thus, leontalbine is the optical antipode of 5, 17-dehydromatrine.