

Thus, we have isolated the previously unknown xeniolide (IV) with a 14(15)-double bond in the side chain. Cases of differences in the chemical compositions of terpenoids from gorgonians of one species but different habitats were known previously for cembranolides [3].

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### CHROMATOSPECTROPHOTOMETRIC DETERMINATION OF CYTISINE IN ÉKSTRAKT TERMOPSIS ZHIDKII (LIQUID EXTRACT OF THERMOPSIS)

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The standardization of medicinal plant material and preparations from it must be carried out in relation to identical biologically active substances. In view of this, the aim of the present investigation was to develop a method for the quantitative evaluation of ékstrakt termopsis zhidkii [liquid extract of thermopsis] (1:2) with respect to cytisine.

As the standard sample we used cytisine corresponding to the demands of the State Pharmacopeia of the USSR, 10th edn. Ékstrakt termopsis zhidkii (1:2) marketed by the Geguzhes Pirmoi pharmaceutical factory was investigated.

The total *Thermopsis* alkaloids were separated by thin-layer chromatography. For this purpose, about 10 g (accurately weighed) of the liquid extract was placed in a 100-ml flask and evaporated on the water bath to a volume of 2-3 ml. After cooling, 2 ml of a solution of ammonia and 60 ml of chloroform, accurately measured, were added and the mixture was shaken on a vibration apparatus for 5-10 min. Then 5 g of anhydrous sodium sulfate was added and shaking was continued for 5 min. The chloroform extract was strained through absorbent cotton, with protection of the liquid from evaporation. Then 40-50 ml, accurately measured, of the chloroform extract was filtered into a flask through a paper filter containing 3 g of anhydrous sodium sulfate and 1.5 g of alumina [activity grade (II)] that had previously been wetted with chloroform. This was washed with chloroform (3 × 5 ml), the washings being added to the main filtrate. The chloroform was distilled off on the water bath to dryness. The dry residue in the flask was dissolved in 2 ml of 95% ethanol.

On a Silufol plate (15 × 15 cm) divided into five equal sections 0.075-ml portions of the alcoholic extract of liquid ékstrakt termopsis zhidkii were deposited on the starting line (bands 2, 3, and 4), and in the fifth position 0.075 ml (150 µg) of a 0.2% ethanolic solution of cytisine, one band being left as control. The plate with the deposited substances was placed in a previously saturated chamber containing the mixed solvent chloroform-acetone-diethylamine (5:4:1) [1]. When the solvent front had traveled 12 cm, the plate was removed from the chamber and dried, and the substances were detected in UV light (254 nm): cytisine ( $R_f$  0.29), pachycarpine (0.48), methylcytisine (0.60), and an unidentified alkaloid (0.70).

As the comparison solution we used the eluate from the control band corresponding to the zone of the standard cytisine, which was prepared in the following way. Cytisine (0.15 g, accurately weighed) was placed in a 100-ml measuring flask and dissolved in 95% ethanol, the solution was made up to the mark with ethanol and was carefully mixed, and 5 ml of the resulting solution was placed in a 50-ml measuring flask, made up to the mark with 95% ethanol, and carefully mixed. This solution contained 0.00015 g of cytisine in 1 ml.

The sections of the sorbent with the standard and experimental samples and the control bands at the level of the zone of the standard sample of cytisine were transferred quantitatively to flasks with ground-in stoppers and were each covered with 10 ml of 95% ethanol and shaken in the vibration apparatus for 10 min, after which the extracts were filtered through folded paper filters.

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The optical densities of the eluates obtained were measured on a spectrophotometer in a cell with a layer thickness of 10 mm at a wavelength of 311 nm, using as comparison solution the eluate from the control band.

The percentages of cytisine ( $X$ ) were calculated from the formula

$$X = \frac{D_{\text{exp}} \cdot 60 \cdot 2 \cdot 10 \cdot 1,05 \cdot 0,00015}{D_{\text{st}} \cdot a \cdot 50} \cdot 100\%$$

where  $D_{\text{st}}$  and  $D_{\text{exp}}$  are the optical densities of the eluates from the standard cytisine and the sample being tested, respectively.

The metrological characteristics of the chromatographic determination of cytisine in ékstrakt termopsis zhidkii (1:2) are given below:

Ser.	$n$	$x$ , %	$S$	$S_x$	$P$	$t(p, f)$	$\Delta, \bar{x}$	$E$ , %
010186	5	0,504	0,018	0,008	95	2,78	0,022	4,37
010187	5	0,502	0,016	0,073	95	2,78	0,020	3,98
010188	5	0,498	0,015	0,007	95	2,78	0,018	3,61

The error of a single determination at a confidence level of 95% does not exceed  $\pm 4.5\%$ .

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### ALKALOIDS OF *Berberis vulgaris*. XII

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Continuing investigations of alkaloids of the genus *Berberis*, we have studied various organs of *Berberis vulgaris* gathered in the foothill region of Stavropol' krai, close to Kislovodsk in the flowering and fruit-ripening phases. Oxyacanthine, berbamine, and berberine have been isolated from this species of barberry growing in the Soviet Union [1]. Abroad, a number of other known alkaloids have been isolated from the roots of this plant [2-4]. From the roots, bark of the stems, leaves, and fruit collected in the fruit-ripening phase 2.19, 1.48, 0.1, and 0.05% of total alkaloids have been isolated by ethanolic extraction. From the leaves collected in the flowering phase, 0.39% of total bases have been isolated by chloroform extraction.

By separating the total quaternary bases from the roots on a column of KSK silica gel we have isolated bases in the form of chlorides which, from their spectral characteristics, have been assigned to the diisoquinoline type and by direct comparison with authentic samples have been identified as berberine, columbamine, and palmatine. The total ether-extracted material from the roots was separated into phenolic and nonphenolic fractions. From the phenolic fraction by separation on a column of silica gel with elution by chloroform and chloroform-methanol have been isolated oxyacanthine, berbamine, berbamine, and a phenolic base (I) with mp 158-159°C (methanol),  $[\alpha]_D^{22} +19^\circ$  (CHCl<sub>3</sub>).

When (I) was methylated with methyl iodide, oblongine iodide [5] was obtained and shown to be identical with an authentic sample. From this, and from spectral characteristics, (I) was identified as juziphine [6]. The total chloroform-extracted material from the roots was separated similarly. Oxyacanthine and bases (II) and (III) were isolated. Base (II) with mp 163-164°C had, in its UV spectrum, absorption in the regions ( $\lambda_{\text{max}}^{\text{ethanol}}$ , nm) 280 (log  $\epsilon$  4.21) and 368 (log  $\epsilon$  4.30), which is characteristic for dihydroprotoberberines [7].

Base (III), mp 200-201°C had absorption in the IR spectrum at 1650 cm<sup>-1</sup>. When (II) and (III) were reduced with NaBH<sub>4</sub> in methanol, a crystalline base with mp 163-169°C (ethanol) was isolated, which was identified by TLC and from the absence of the depression of the melting point of a mixture as ( $\pm$ )-tetrahydroberberine. On the basis of their physicochemical

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