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

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

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

The metrological characteristics of the chromatospectrophotometric 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 ±4.5%.

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ALKALOIDS OF *Berberis vulgaris*. XII

M. M. Yusupov, A. Karimov, and K. L. Lutfullin

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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, berbamunine, and a phenolic base (I) with mp 158-159°C (methanol), $[\alpha]_D^{22}$ +19° ($CHCl_3$).

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 ($\lambda_{max}^{ethanol}$, nm) 280 (log ε 4.21) and 368 (log ε 4.30), which is characteristic for dihydroprotoberberines [7].

Base (III), mp 200-201°C had absorption in the IR spectrum at 1650 cm^{-1}. When (II) and (III) were reduced with $NaBH_4$ 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 (±)-tetrahydroberberine. On the basis of their physicochemical

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characteristics, chemical transformation, and direct comparison with synthetic samples, (II) and (III) were identified as lambertine and berlambine, respectively. The latter had been isolated previously from *B. lamberti* [8].

Thus, the roots, bark of the stems, the leaves, and the fruit of *B. vulgaris* have been studied for their alkaloid contents. By separating the total alkaloids of the roots we have isolated nine bases. Of them, berlambine, lambertine, and berbamunine have been isolated from this species for the first time, while juziphine has not previously been isolated from the genus *Berberis*.

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MONITORING THE QUALITY OF THE PREPARATION ALLAPININ BY HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY

A. D. Sakhibov, Sh. A. Sadikova, and G. L. Genkina

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Allapinin is an effective antiarrhythmic agent. The active principle of the preparation is the hydrobromide of the alkaloid lappaconitine with an admixture of accompanying alkaloids obtained from the herb aconite (monkshood) [1].

It has been shown by TLC on Silufol UV-254 plates in the chloroform–benzene–diethylamine (10:40:3) system that allapinin contains, in addition to lappaconitine (main, violet, spot), N-deacetyllappaconitine (N-DAL), two compounds of unestablished structure, and a compound of an unknown class.

Earlier [1], for the quantitative determination of allapinin as such a titrimetric method was proposed (using an 0.2-g sample), the prescribed total amount of alkaloids being not less than 96%.

The alkaloids accompanying lappaconitine in the preparation have similar spectral properties. The molar extinction of lappaconitine hydrobromide is low ($\lambda_{max}^{H_2O}$ 298 nm, log ε 3.6) and, therefore, to monitor the purity of allapinin we propose the HPLC method, which is distinguished by high sensitivity and selectivity and permits the rapid determination in the native form of all the components of the mixture from a single sample [2-4].

For HPLC we used a TRACOR-955 liquid chromatograph (USA) with an UV detector at a wavelength of 220 nm.

Conditions of chromatography: Perkin–Elmer II-X column (2.6 × 250 mm) filled with the reversed phase ODS-C18. Mobile phase – a mixture of two solutions, A and B, in a ratio of 35:65. Solution A was a mixture consisting of 95% ethanol, water, and phosphoric acid in a ratio of 20:79:1. Solution B contained the same ingredients in a ratio of 2:97:1. The rate of elution was 1 ml/min and the temperature of column 40°C. The time of analysis was 40 min.

Allapinin corresponding to the official documentation [1], in an amount of 5 mg, was dissolved in 5 ml of 20% methanol, and 20 μl (20 μg) of the resulting solution was transferred to the column with the aid of a loop injector.

To determine the area of the peak of the main substance, the preparation was chromatographed at a sensitivity of the detector of 1.0. The areas of the peaks of the impurities were determined by rechromatographing the same sample at a detector

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

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