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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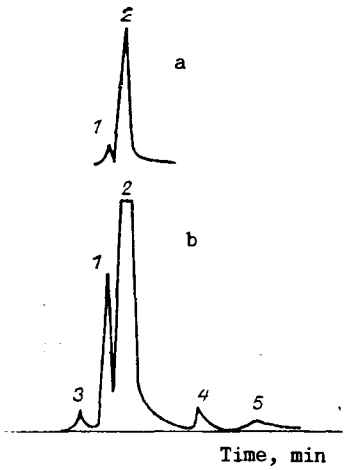

Fig. 1. Chromatogram of allapinin: 1) DAL; 2) lappaconitine; 3-5) unidentified impurities.

TABLE 1. Component Analysis of Industrial Batches of Allapinin, % (Fig. 1b)

Batch of the preparation	Lappaconitine (peak 2)	N-DAL (peak 1)	Unidentified substances		
			peak 3	peak 4	peak 5
211086	88,35	8,97	0 44	1,68	0,56
20287	89,60	8,93	0,19	1,09	0,19
300287	91,88	6,17	0,56	1,21	0,18
80388	90,25	7,89	0,36	1,13	0,46
100488	91,20	7,02	0,43	1,23	0,12

sensitivity of 0.1 (Fig. 1). In Fig. 1a, peak 1 corresponds to N-DAL, and peak 2 (the main one) to lappaconitine; in Fig. 1b are shown three additional peaks corresponding to unidentified impurities. The correspondence of peak 1 to N-DAL was shown by chromatographing the substance with the addition of N-DAL. In these circumstances, an increase in the analytical signal (area of the peak) by a magnitude directly proportional to the concentration of N-DAL was observed.

Table 1 gives comparative results on the component analysis of various batches of allapinin. The metrological characterization of the methods showed that the relative error does not exceed ±2%. The threshold of detection of allapinin is 50 ng.

Thus, the proposed method is two orders more sensitive than the TLC method and three orders more sensitive than the UV-spectroscopic method, and permits not only a qualitative analysis but also a quantitative evaluation of the preparation.

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