RAPID METHOD FOR THE QUANTITIVE DETERMINATION OF ALLAPININ IN THE HERB Aconitum leucostomum

A. Z. Sadikov, É. V. Safonova, and T. T. Shakirov

UDC 547.94:543.422

The epigeal part of the herb Aconitum leucostomum Worosch (family Ranunculaceae) is the industrial source of the antiarrhythmic drug allapinin [1]. Methods are known for the quantitative determination of allapinin in the herb Aconitum leucostomum [2, 3]. These methods, consisting in the wetting of the comminuted raw material with a soda solution, extraction with chloroform, purification, chromatographic separation of the total alkaloids [2], or without chromatographic separation [3], and the determination of allapinin by spectrophotometry, are very laborious and very time-consuming.

The quality of the A. leucostomum raw material varies according to the vegetation period, the collection site, the conditions of drying and storage, and so on [4]. In view of this, mass industrial processing requires numerous analyses of the herb, which has led to a demand for a rapid method.

To develop a rapid method we studied the process of extracting the alkaloids with 80% ethyl (or isopropyl) alcohol and determined the time of diffusional equilibrium on first contact of the phases with heating to the boiling point of the solution. We found a conversion factor experimentally that enabled us to determine the amount of allapinin in the raw material without extracting it completely.

Identical results were obtained on determining the content of lappaconitine with accompanying alkaloids (allapinin) in more than ten samples of A. leucostomum by the method of VFS [Provisional Pharmacopeal Standard] 4201666-86 and modification No. 1 in comparison with the proposed rapid method.

The method developed permits an analysis of A. leucostomum raw material to be carried out in 3-3.5 h.

Below, we give the metrological characteristics of the method applied to the analysis of A. leucostomum gathered in 1990-1991 in Issyk-Kul' province:

n	f	$\bar{x}$	S	t	P	X	Σ *
10	9	0,121	$1{,}9\cdot10^{-3}$	2,57	95	$4{,}9\cdot10^{-3}$	4,0

The results of the analysis (in % on the air-dry mass of the raw material): 1) 0.120; 2) 0.121; 3) 0.120; 4) 0.118; 5) 0.118; 6) 0.122; 7) 0.124; 8) 0.123; 9) 0.120; 10) 0.121.

In this method, an analytical sample of the raw material is ground to particles passing through a sieve according to TU [Technical Specification] 23.2.2068—89 with apertures having a diameter of 5 mm. About 20 g (accurately weighed) of the ground raw material is extracted with 100 ml of 80% ethanol in a 500-ml round-bottomed flask with a reflux condenser, and the mixture is then cooled and filtered. A 20-ml sample of the filtrate is evaporated to an aqueous residue, the pH is brought 9 (according to universal indicator paper TU 6-09181-71) with 20% sodium carboante solution, and the alkaloids are extracted with chloroform until the reaction with tungstosilicic acid is negative.

The chloroform extract is treated with 5 g of anhydrous sodium sulfate, shaken, and filtered through a paper filter into a distillation flask. The chloroform is distilled off to dryness. The residue is dissolved in 50 ml of 95% alcohol and the solution is filtered through a paper filter into a 50-ml measuring flask. The volume of the solution is made up to the

---

Institute of Chemistry of Plant Substances, Uzbekistan Academy of Sciences, Tashkent.
Translated from Khimiya Prirodnykh Soedinenii, No. 2, pp. 306-307, March-April, 1993. Original article submitted April 22, 1992.

0009-3130/93/2902-0257$12.50 © 1993 Plenum Publishing Corporation

mark with the same solvent, and 2.5 ml of this solution is transferred with a pipet into a 25-ml measuring flask and made up to the mark with the same solvent.

The optical density of the solution obtained is measured at 308 nm on a spectrophotometer in a cell with a layer thickness of 10 mm, using 95% alcohol as the comparison solution. The optical density of a solution of a working standard sample (WSS) of allapinin is measured in parallel.

The percentage of lappaconitine with accompanying alkaloids as recalculated to lappaconitine in the absolutely dry raw material (X) is found from the formula:

$$X = \frac{D_1 \cdot m_0 \cdot C_0 \cdot 89 \cdot 20 \cdot 2}{D_0 \cdot m\,(100 - W) \cdot 100},$$

where D_0 and D_1 are the optical densities of the solution of the WSS of allapinin and of the solution under investigation, respectively; m_0 and m are the weights of the allapinin and of the raw material, respectively; C_0 is the amount of the main substance in the standard sample of allapinin, %; W is the loss in weight on the drying of the raw material, %; and 2 is the conversion factor of the diffusional equilibrium (when isopropyl alcohol is used, the factor is 1.8).

LITERATURE CITED

1. A. Z. Sadikov and T. T. Shakirov, Khim. Prir. Soedin., 91 (1988).
2. Provision Pharmacopeal Standard 42-1666-86 [in Russian].
3. A. U. Makhkamova, É. V. Safonova, A. Z. Sadikov, E. K. Dobronravova, and T. T. Shakirov, Khim. Prir. Soedin., 436 (1989).
4. M. G. Zhamierashvili, V. A. Tel'nov, M. S. Yunusov, S. Yu. Yunusov, A. Nigmatullaev, and K. Taizhanov, Khim. Prir. Sodium., 805 (1980).