DETERMINATION OF ALKALOIDS IN THE HERB *Vinca minor*

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Vinca minor L. (common periwinkle) is of great practical interest as a source of domestic hypotensive agents. Methods are known for the analysis of the raw material of the common periwinkle which are based on gravimetric, extraction-photometric, and spectrophotometric procedures and the method of nonaqueous titration [1-4].

Having generalized the literature information on the analysis of the periwinkle, we have developed a convenient and fairly accurate method for estimating the total amount of alkaloids it contains by using the method of nonaqueous titration in view of the high accuracy of the methods and the possibility of performing the determination without the vincamine standard that is necessary when optical methods are used.

We have determined the optimum conditions for the extraction of the raw material and the titration of the total alkaloids extracted. The purest total material was obtained with the highest yield when chloroform was used as the extractant. Of a series of solvents and mixtures of them used for the titration of weak bases [5, 6], the most convenient proved to be acetic anhydride. On titration in acetic anhydride, the jumps in potential almost doubled in comparison with glacial acetic acid, which permitted the use of the indicator method of determining the equivalence point with good reproducibility. The correspondence of the change in the color of the indicator from violet to green with the titration jump was established by the potentiometric method.

The investigations performed have been made the basis of a procedure for the quantitative determination of the total alkaloids.

About 5 g (weighed with an accuracy of 0.01 g) of raw material that had been ground and passed through a No. 30 sieve with apertures having a diameter of 3 mm was placed in a 750-ml conical flask with a ground-in stopper and was carefully moistened with 5 ml of 10% ammonia; after 10-15 min, 300 ml of chloroform was added and the mixture was shaken and was left for 4 h with periodic shaking. The resulting extract was filtered through four layers of gauze.

The extract obtained (100 ml) was placed in a separatory funnel and the alkaloids were extracted with a 0.5 N solution of sulfuric acid (3 × 15 ml), with shaking each time for 2 min. The sulfuric acid extracts were then filtered through filter paper into a second separatory funnel. The combined sulfuric acid extracts were made alkaline with 10% ammonia solution to pH 10 according to universal indicator paper and were extracted with chloroform (4 × 15 ml), with shaking for 2 min each time. The chloroform extracts were then filtered into a 100-ml flask through a paper filter containing 3 g of anhydrous sodium sulfate that has previously been moistened with chloroform. The chloroform was distilled off on the water bath to dryness. The residue was dissolved in 10 ml of acetic anhydride, two drops of a 0.1% solution of Crystal Violet was added, and the solution was titrated with a 0.05 N solution of perchloric acid in acetic acid until the color of the indicator changed to green.

The alkaloid content as a percentage was calculated as free vincamine (mol. mass 354.44).

The accuracy of the method is ±1.36% ($n = 5$, $\bar{x} = 0.59$, $\delta\bar{x} = 0.0025$).

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ALKALOIDS OF *Peganum harmala*

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The plant *Peganum harmala* L. (harmel peganum) is widespread on the territory of our country, including Central Asia. More than 20 alkaloids have been isolated from *P. harmala* from various growth sites [1], but the alkaloid composition of this plant from the territory of Tadzhikistan has not yet been investigated.

We have studied the alkaloid composition of *P. harmala* growing in the regions of Lyaura, Leninskii region (900 m above sea level) and in the basin of the R. Yagnob, Anzobskii region (1500 m above sea level) of the Tadshik SSR.

From 18 kg of air-dry epigeal part of *P. harmala* chloroform extraction yielded 139.82 g (0.77%) of technical alkaloids. A solution of the total alkaloids was separated on a column of alumina (Brockman activity grade II). Elution was performed in the following solvent systems: benzene–ethyl acetate (2:1); ethyl acetate; and chloroform–ethanol (7:3). The fractions obtained were separated and analyzed by TLC (alumina fixed with gypsum) in the systems mentioned.

The alkaloids were repurified on a column of alumina and were identified by thin-layer chromatography with markers, and also on the basis of the UV, IR, and NMR spectra, which agreed with those given in the literature [1]. The amounts of the various alkaloids are given in Table 1.

As can be seen from Table 1, the largest amount of alkaloids was present in the ripe fruit and the smallest amount in the stems and leaves. The percentages of the individual alkaloids in various organs of *P. harmala* were determined by gas chromatography on a Chron-5 chromatograph. Flame ionization detector, 1.20 m × 3 mm column, 5% of SE-30 on Chromaton N, temperature of the evaporator 310°C, temperature of the detector 280°C.

The areas of the peaks were determined with the aid of an IT-2 electronic integrator. The combined error of chromatograph and integrator in the determination of the areas was 2%. Analysis was peformed under the regime of the programming of the thermostat temperature. After injection, the temperature was kept at 120°C for 4 min, and then it was raised in the course of 4 min to 260°C.

TABLE 1. Amounts of Alkaloids in *Peganum harmala* in the Flowering–Fruit-Bearing Phases

Plant organ	Sum of the alkaloids, % on the air-dry weight		Amounts of alkaloids in sample 2, % on the total				
	sample 1	sample 2	harmine	harmaline	deoxyvasicinone	peganine	total identified
Leaves	0,3	0,4					
Stems	0,9	0,6	10	5	70	5	10
Flowers	2,6	1,3	—	—	—	—	100
Fruit							
green	4 6	5,7	75	5	5	5	10
ripe	5 4	6,3	15	10	15	40	20
Roots	2,5	2,3	60	5	5	20	10

Note. Sample 1 was collected in the Lyaura region in June, 1981; sample 2 was collected in the R. Yagnob basin in July, 1982.

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