A METHOD FOR THE QUANTITATIVE DETERMINATION OF VINCAMINE IN THE EPIGEAL PART OF Vinca erecta

R. A. Mirkina and T. T. Shakirov

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In a preceding communication [1] the technology of the isolation of the alkaloid vincamine from the epigeal part of Vinca erecta was described. We have developed a method for the quantitative determination of vincamine in plant raw material, which consists in the extraction of the alkaloids from the raw material with chloroform, the chromatographic separation of the chloroform extract, and the extraction-photometric determination of the vincamine.

The epigeal part of Vinca erecta contains more than 30 alkaloids [3] and repeated chromatography does not give a complete separation of the vincamine from the accompanying bases, so that double chromatography in a thin layer of silica gel was used [4].

To determine the completeness of the desorption of the vincamine from the silica gel, we carried out double chromatography with a subsequent quantitative determination of the vincamine in the eluate (mean of two determinations).

Added, mg	Found %
0.145	96.0
0.193	95.5
0.242	95.4

We checked the objectivity of the proposed method by the analysis of extracts with the addition of pure vincamine to them (Table 1).

The method developed has been used for the analysis of plant raw material from the 1968 crop (0.026-0.027% of vincamine) and the 1969 crop (0.034-0.035% of vincamine).

EXPERIMENTAL

Extraction of the Alkaloids from the Raw Material. The comminuted air-dry raw material (20 g) was wetted with 15 ml of 10% ammonia solution, left for 1 h, and then exhaustively extracted with chloroform in a Soxhlet apparatus. The extract was concentrated to a volume of 10-15 ml, transferred quantitatively to a 25-ml measuring flask, and made up to the mark with chloroform.

TABLE 1. Results of a Quantitative Determination of Vincamine in Extracts with the Addition of Pure Vincamine.

Amt. added	Nominal amount	Found	Relative error, %
	mg		
0,072	0,180	0,183	+1 8
0,077	0,256	0,244	—4,8
0,053	0,130	0,124	—4,3
0,053	0,160	0,154	—4,0

Chromatographic Separation. On a plate coated with a layer of alkaline silica gel (14 g of KSK silica gel and 30 ml of 0.1 N NaOH solution on a 15 × 24 cm plate), 0.5 ml of the extract was chromatographed in the chloroform–methanol (9.5 : 0.5) system. A "marker" (vincamine) was deposited on the plate as well. The position of the marker was revealed with a 1% phosphoric acid solution of cerium ammonium sulfate. When the chromatograms were sprayed with a solution of cerium ammonium sulfate, the spots of the alkaloids of Vinca erecta gave colors characteristic for each base [5]. The spot of vincamine (R_f 0.8± 0.05) had a characteristic lemon-yellow color.

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The part of the silica gel with the vincamine spot was cut out and eluted with a mixture of chloroform and methanol (8 : 2). In addition to the vincamine, a certain amount of another alkaloid was extracted.

The eluate was distilled until the residue amounted to a few drops and this was transferred quantitatively to a plate with a fixed layer of silica gel and chromatographed in the acetone–butan-1-ol (1 : 1) system. The vincamine (R_f 0.3 ± 0.05) was separated completely from the accompanying alkaloid (pink spot with R_f 0.9 ± 0.05).

After the complete elimination of the butan-1-ol, the part of the silica gel with the vincamine spot was cut out and eluted with a mixture of chloroform and 1% methanolic tartaric acid (9 : 1). The eluate was distilled to dryness, the residue was dissolved in 10 ml of citrate-phosphate buffer solution (pH 3.7-4.0), and the amount of vincamine was determined as described previously [2].

As the standard solution we used an eluate formed in the chromatography of 0.2 ml of a solution of vincamine (c 1 mg/ml) under the same conditions as the extract of the raw material. The content of vincamine (x, %) calculated to the dry raw material was obtained from the formula

$$x = \frac{C_{st} \cdot D_x \cdot 500}{D_{st} \cdot a\,(100 - h)},$$

where C_{st} is the concentration of the solution of the standard sample, mg/ml; D_{st} is the optical density of the standard sample; D_x is the optical density of the sample under investigation; a is the weight of raw material, g; and h is the moisture content, %.

SUMMARY

An extraction-photometric method for the determination of vincamine in its raw material – the epigeal part of Vinca erecta – has been developed.

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