QUANTITATIVE DETERMINATION OF VINCANIDINE IN THE ROOTS OF Vinca erecta

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Apart from vincanine [1], the roots of Vinca erecta contain a number of other alkaloids [2], one of which – vincanidine [3] – is of practical interest, since it is a physiologically active substance. In view of this, we have developed a method for the analysis of vincanidine in the raw material.

To determine the amount of vincanidine we have proposed an extraction-photometric method based on the capacity of organic bases for forming with certain dyes addition products which are soluble in organic solvents [4]. As the dye we used Tropaeolin 000.

The results of a study of the dependence of the optical density of solutions of vincanidine tropaeolinate on the pH of the medium showed that the maximum light-absorption of the solution is found in the acid region (pH 1.5-2.5). The composition of the complex was determined by the saturation method and by the method of isomolar series. Both methods showed that the stoichiometric ratio of the reactants in vincanidine tropaeolinate is 1:1. The values of the molar absorption coefficient and of the equilibrium constant of the complex-forming reaction were determined by Tolmachev's method [5]; they are 10,000 and 13.1, respectively. The absorption of the complex obeys the Lambert–Beer law over a wide range of concentrations.

To separate the vincanidine from the residual alkaloids, we used chromatography on alumina. In the chromatographic separation, vincanidine is retained on the sorbent and the other alkaloids are eluted by benzene or chloroform. To elute the vincanidine from the alumina the most suitable agent proved to be a mixture of tartaric and hydrocholoric acids.

The results of the determination of the degree of desorption of the vincanidine from alumina are given in Table 1. The eluent – a mixture of tartaric and hydrochloric acids – is a suitable solvent both for the elution of the vincanidine from the sorbent and also for the direct determination of the amount of base in it. The results of the analysis of a model mixture of alkaloids are given in Table 2.

Using the method developed, we determined the amounts of vincanine and vincanidine in the roots of Vinca erecta. The objectivity of the procedure for the quantitative determination of these compounds in one sample of raw material was checked by the method of additives (Table 3).

EXPERIMENTAL

Extraction of the Alkaloids from the Raw Material. The comminuted roots of Vinca erecta (10 g) were moistened with 8 ml of a 5% aqueous solution of ammonia and were left for 1 h, and they were then charged into a Soxhlet apparatus and extracted exhaustively with chloroform. The chloroform extract was concentrated in vacuum and was transferred to a 50-ml measuring flask.

TABLE 1

Vincanidine added, mg	Found	
	mg	%
0,25	0,24	96,0
0,50	0,49	98,0
0,75	0,73	97,3
1,00	0,97	97,0

Chromatographic Separation. A 2- to 5-ml portion of the extract was deposited quantitatively on a column of alumina (activity grade II according to Brockmann; column 10 cm long and 1 cm in diameter). The vincanine was eluted with 50 ml of chloroform or benzene and was determined quantitatively by a known method [1].

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TABLE 2

Taken, mg		Found				Statistical characteristics for	
vincanine	vincanidine	vincanine		vincanidine		vincanine	vincanidine
		mg	%	mg	%		
1,45	0,30	1,42	97,9	0,29	96,6	$\bar{x}$=97,5%	$\bar{x}$=96,9
1,37	0,42	1,34	97,8	0,41	97,6	S^2= 0,51	S^2= 0,47
0,85	0,50	0,84	98,8	0,49	98,0	Sx= 0,71	Sx= 0,69
0,52	0,27	0,51	98,0	0,26	96,2	$S\bar{x}$= 0,31	$S\bar{x}$= 0,28
0,32	0,32	0,31	96,8	0,31	96,8	α= 0,95	α= 0,95
						$t\alpha$, κ= 2,306	$t\alpha$, κ= 2,306
						$\Sigma\alpha$= 0,76	$\Sigma\alpha$= 0,69
						E_{rel} =±0,78%	E_{rel} =±0,70%

TABLE 3

Found in extract, mg		Added, mg		Found, mg	
vincanine	vincanidine	vincanine	vincanidine	vincanine	vincanidine
3,2	1,62	0,50	0,25	98,8	97,8
3,2	1,62	1,00	0,50	99,0	97,6
3,2	1,62	1,50	0,75	98,6	98,0
3,2	1,62	2,00	1,00	98,6	97,4

Method of Determining Vincanidine. The vincanidine was eluted with 25 ml of a mixture of 20% tartaric acid and 0.1 M HCl (9 : 1). To eluate was added 5 ml of a 0.1% solution of Tropaeolin 000, it was transferred to a separating funnel, and the vincanidine tropaeolinate formed was extracted with chloroform. The chloroform extracts were combined in a 25-ml measuring flask and were made up to the mark with chloroform, and the optical density was determined on an FEK-M photocolorimeter with a blue filter in a cell having a layer thickness of 1 cm.

As the standard solution was used the eluate formed in the chromatography of 0.3-0.4 ml of a solution of vincanidine (c 1 mg/ml) under the conditions as the extract of the raw material.

CONCLUSIONS

1. An extraction-photometric method for the quantitative determination of vincanidine is proposed.

2. A method for the separate determination of the amounts of vincanine and vincanidine in a single sample of raw material has been developed.

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