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METHOD FOR THE QUANTITATIVE DETERMINATION OF VINCARINE, HERBADINE, AND HERBAMINE IN THE HERBAGE OF Vinca herbacea

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The alkaloids vincarine, herbadine, and herbamine isolated from the herbage of Vinca herbacea Waldstet. et Kit. [1, 2] are pharmacologically active [3]. We propose a method for the quantitative determination in the plant raw material which consists in obtaining the combined alkaloids, separating them by TLC, and determining the alkaloids in the eluates by spectrophotometry on an SF-26 instrument. To separate the herbadine, herbamine, and vincarine from the accompanying bases, the total material was chromatographed on a fixed layer of type KSK No. 2 silica gel in the ethyl acetate–methanol (95:5) system. Elution with chloroform achieved 98-100% desorption of the material from the plate.

The essence of the method was as follows: 25 g of comminuted air-dry raw material was placed in a conical flask with a ground-in stopper, the alkaloids were exhaustively extracted with a 2% aqueous solution of sulfuric acid, the amorphous combined alkaloids were obtained by the usual methods [4], and these were dissolved in chloroform. The solution was transferred quantitatively to a 25-ml measuring flask and it was made up to the mark with chloroform, after which 3 ml of the resulting solution was deposited on a plate (24 × 18 cm) in the form of a continuous line 14 cm long and, beside it at a distance of 1.5 cm, as marker, was deposited 0.1 ml of the same solution in the form of a continuous line 1.5 cm long.

Chromatography was performed by the ascending method in the above-mentioned system. The finished plate was dried in the air, and only the marker band was stained with a solution of cerium ammonium sulfate in 85% orthophosphoric acid. The corresponding sections of the sorbent at the levels of vincarine (orange-red band with Rf 0.16), herbadine (orange-red band with Rf 0.52), and herbamine (carmine-red band with Rf 0.7), were transferred quantitatively to flasks, covered with chloroform, and extracted on a universal shaking machine for 2 h. After separation, the chloroform extract from each zone was evaporated to dryness and the residue was dissolved in 2 ml of methanol. The optical densities were determined after appropriate dilution. The amounts of vincarine, herbadine, and herbamine in the raw material (X, %), calculated on the absolutely dry weight of the raw material, were calculated from the formula

$$X = \frac{K \cdot 100 \cdot D \cdot V_1 \cdot V_3 \cdot V_5}{a\,(100-b) \cdot E_{1\text{cm}}^{1\%} \cdot V_2 \cdot V_4},$$

where D is the optical density of the solution under investigation; $E_{1\,cm}^{1\%}$ is the specific absorption index of vincarine at a wavelength of 243 nm, which is 165; or of herbadine at 292 nm, 90; or of herbamine at 295 nm, 85.5; V_1 is the volume of the chloroform solution, ml; V_2 is the volume of the chloroform solution deposited on the plate, ml; V_3 is the volume of the methanol solution, ml; V_4 is the volume of the methanol solution taken for dilution, ml; V_5 is the volume of the diluted methanol solution, ml; a is the rate of raw material, g; b is the loss in weight on the drying of the raw material, %; and K is a correction factor for the alkaloid being determined on TLC separation.

Below we give the results of a statistical treatment of the figures obtained in the determination of vincarine, herbadine, and herbamine in the raw material. The amount of vincarine in the raw material, calculated on the absolutely dry weight of the plant, was 0.064%; of herbadine, 0.081%; and of herbamine, 0.077%.

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Alkaloid	f	$\bar{X}$	S^2	S	P	$t_{p,f}$	ΔX	$E, \%$ $E_m, \%$
Vincarine	9	0,625	0,000149	±0,012210	95	2,26	±0,027596	4,41 ±2,55
Herbadine	9	1,128	0,000479	±0,021899	95	2,26	±0,049493	4,39 ±2,53
Herbamine	9	1,881	0,001405	±0,037491	95	2,26	±0,084730	4,50 ±2,60

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FORMATION OF THE RADICAL ANIONS $O_2^{\cdot}$ IN THE OXIDATION OF LIGNIN

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It is known that the oxygen radical anion ($O_2^{\cdot}$) is formed in the autooxidation of phenols in alkaline media [1]. In the opinion of Renard et al. [2], $O_2^{\cdot}$ plays an active part in the oxidation of lignin by a radical chain mechanism. However, there are no experimental proofs of the formation of the oxygen radical anion in the oxidation of lignin and its model compounds in the literature.

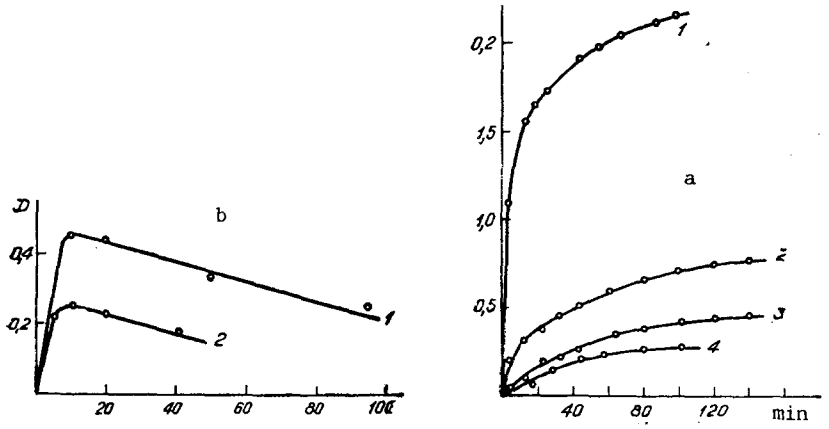

Fig. 1. Change in the optical density in the zone of the maximum absorption of diformazan on the oxidation of lignin (1.67 × 10^{-2} M), in the presence of Tetrazolium Blue ($5 \cdot 10^{-4}$ M): a) air, 30°C: 1) 1 N NaOH; 2-4) aqueous buffer, pH 9.3—DMF (1:2); 2) C_{SOD} = 0; 3) S_{SOD} = $9 \cdot 10^{-6}$ M; 4) C_{SOD} = $3.6 \cdot 10^{-5}$ M. b) pO_2 = 1 atm, 60°C, aqueous buffer, pH 3—DMF (1:2): 1) C_{SOD} = 0; 2) C_{SOD} = 10^{-5} M.

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