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QUANTITATIVE DETERMINATION OF SPHAEROPHYSINE BENZOATE

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Sphaerophysine — an alkaloid obtained from the plant salt globe-pea (*Sphaerophysa salsula*) is produced by the domestic pharmaceutical chemistry in the form of sphaerophysine benzoate.

The determination of sphaerophysine benzoate by acid–base titration in a nonaqueous medium [1] is characterized by low sensitivity and inconvenient execution. A method is known for the quantitative analysis of sphaerophysine by the colorimetric examination of a sample obtained in the chromatographic separation of the alkaloids of the globe-pea and its treatment with ninhydrin [2]. A defect of this method is its low selectivity, since ninhydrin is used in the analysis of many drugs belonging to various classes of compounds [3].

The object of the present investigation was to increase the sensitivity of the determination of sphaerophysine benzoate. For this purpose we have studied the possibility of using alloxane hydrate for the quantitative analysis of this drug by a spectrophotometric method in the visible region. It has been established that alloxane hydrate reacts with sphaerophysine benzoate in dimethyl formamide with the formation of a crimson solution. The optimum conditions for the reaction are heating in the boiling water bath, the use of alloxane hydrate in the form of a 10% solution in dimethylformamide, and the use of kh. ch. ["chemically pure"] dimethylformamide as solvent.

The figures given below show the high sensitivity of the reaction of alloxane hydrate with sphaerophysine benzoate

Analytical index	Numerical values
Absorption maximum, nm	479
Molar absorption coefficient	12,050
Specific absorption	0.0272
Sandell coefficient	0.0367
Koch and Koch–Dedic coefficient	1.835
Minimum detectable concentration, μg/ml	1.836

The absorption spectrum of the product of the interaction of the drug with the reagent has λ_{max} 479 nm. We have calculated the specific absorption index ($E^{1\%}_{1cm}$) for this wavelength. The basic law of light absorption is obeyed within the concentration of 1.2-3.6 mg of sphaerophysine per 100 ml of solution.

The quantitative determination of sphaerophysine benzoate in the substance was carried out in the following way. An accurately weighed sample of between 0.0124 and 0.0229 g was dissolved in dimethylformamide in a 50-ml measuring flask and was made up to the mark with dimethylformamide. To 2 ml of this dilution was added 2 ml of a solution of alloxane hydrate in dimethylformamide. The reaction mixture was heated on the boiling water bath for 5 min and was then artificially cooled, and the solution was transferred quantitatively to a 25-ml measuring flask and was made up to the mark with dimethylformamide. The optical density of the colored solution was measured in a SF-4A spectrophotometer in quartz cells with a layer thickness of 1 cm at an analytical wavelength of 479 nm.

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The percentage content of sphaerophysine benzoate was calculated from the formula

$$C = \frac{D \cdot 50 \cdot 25}{E^{1\%}_{1\,cm} \cdot p \cdot 2},$$

where D is the optical density. $E^{1\%}_{1\,cm}$ is the specific absorption index, which is 229.33±8.65, and p is the weight of the sample, g.

The results of the determination are given below:

Weight of sample, g	Found, %	Metrological characteristics
0,0124	98,90	$\overline{X} = 99,60$
0,0156	99,58	$S = 0,451$
0,0174	100,24	$Sr = 0,0045$
0,0183	99,78	$St = \pm 1,11$
0,0229	99,73	$\overline{X} \pm St = 99,60 \pm 1,11$
0,0200	99,34	

The method developed is characterized by high sensitivity and by accuracy of the results. The relative standard deviation Sr does not exceed 0.0045.

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MACRANTALINE — A MINOR ALKALOID OF *Papaver lisae*

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Protopine, isocorydine, mecambridine, oridine, and N-methyloridine have been isolated previously from the epigeal part of *Papaver lisae* N. Busch [1].

In an investigation of the epigeal part of *P. lisae* collected in North Ossetia in the mass flowering phase, we have obtained the total alkaloids with a yield of 0.35% on the weight of the dry raw material. By using the method of separation into phenolic and nonphenolic alkaloids the solubilities of the hydrochlorides in chloroform, and column chromatography, from the total alkaloids, in addition to protopine, isocorydine, mecambridine, and oridine, we have isolated a nonphenolic base new for this plant, with mp 140-141°C (from acetone–diethyl ether–petroleum ether), $[\alpha]_D^{19}$ +30.2±0.3° (c 3.4; chloroform).

The UV spectrum of the base (in methanol) has absorption maxima at 285, 238 nm (log ε 3.64 and 4.04) and a minimum at 256 nm (log ε 2.95, corresponding to the isoquinoline bases). According to the IR spectra, the base contains no carbonyl group, but it does contain a hydroxy group bound by an intramolecular hydrogen bond (3150 cm^{-1}).

The mass spectrum of the base contains peaks at m/e 220 (100%) and 205 (20%) and according to the PMR spectrum the alkaloid contains a N-CH_3 group (3 H, singlet at 2.19 ppm), three OCH_3 group (3 H, singlet at 4.16 ppm, 3 H, singlet at 3.82 ppm, and 3 H, singlet at 3.86 ppm), a methylenedioxy group (2 H, singlet at 5.84 ppm), and one isolated and two ortho aromatic protons (1 H, singlet at 6.29 ppm, 1 H, doublet at 6.81 ppm, and 1 H, doublet at 7.04 ppm, J = 7 Hz). The PMR spectrum also contained two one-proton doublets with J = 10 Hz at 4.39

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