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METHOD FOR THE QUANTITATIVE DETERMINATION OF DEOXYPEGANINE FROM Peganum harmala

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The epigeal part of Peganum harmala (family Lygophyllaceae) contains a number of alkaloids [1]. We propose a method for the quantitative determination of deoxypeganine in the plant raw material which consists in obtaining the combined alkaloids from this material separating them by chromatography, and determining the deoxypeganine in the eluate by micro nonaqueous titration [2]. The deoxypeganine was separated from the accompanying alkaloids in a thin nonfixed layer of alumina in the chloroform–benzene–acetone (12 : 6 : 9) system. The R_f value for peganine, deoxypeganine, vasicinone, deoxyvasicinone, peganol, harmine, harmaline, peganidine, deoxypeganine and pegamine are, respectively, 0.02, 0.25, 0.10, 0.76, 0.55, 0.40, 0.06, 0.02, 0.07, and 0.04. On elution with chloroform, 98-100% desorption was achieved.

Below we give the characteristics of the statistical treatment of the results of the determination of deoxypeganine (0.5-2.0 mg) by acid–base titration in anhydrous acetic acid using a solution of perchloric acid as the titrant:

h	$\bar{x}$	S^2	S	$S_{\bar{x}}$	a	$t_{a,k}$	E_a	E_{rel}
8	100,40%	2.760	1.661	0.587	0.95	2.365	1.388	1.38 %

The amounts of deoxypeganine in the raw material was found in the following way. A 20-g portion of the comminuted air-dry raw material was wetted with 20 ml of a 10% solution of ammonia, and the alkaloids were extracted exhaustively with chloroform. The extract was concentrated to 25 ml and from 5 ml of this extract the total alkaloids were obtained in the usual way [3] and were dissolved in 5 ml of ethanol. On a plate (13 × 18 cm) with a layer of alumina (activity grade III, particle size 0.2-0.1 mm; layer thickness 1.5 mm, pH of a 10% aqueous suspension 4.2-4.5) was deposited 0.5 ml of the ethanolic solution of the combined alkaloids and chromatography was carried out in the above-mentioned system. A "marker" – 0.5 ml of a 0.5% ethanolic solution of deoxypeganine hydrochloride – was deposited on the same plate, which was treated in the moist state with Dragendorff's reagent. The alkaloids were eluted with 100 ml of chloroform, the resulting solution was evaporated to dryness, and the residue was dissolved in 5 ml of glacial acetic acid and titrated with 0.01 N perchloric acid until the solution acquired a blue color (indicator Crystal Violet). The amount of deoxypeganine in the raw material (x, %), referred to the absolutely dry weight, was calculated from the formula

$$x = \frac{50 \cdot 17.22 \cdot V_1}{p\,(100 - h)},$$

where V_1 is the volume of 0.01 N perchloric acid solution consumed in the titration, ml; p is the weight of the raw material, g; h is the moisture content of the raw material, %; and 1 ml of 0.01 N perchloric acid solution corresponds to 0.001722 g of deoxypeganine. A control experiment was performed in parallel. The method

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developed was used to find the amount of deoxypeganine in the herb Peganum harmala collected in the Dzhizak oblast of the Uzbek SSR – from 0.3 to 0.08 % of the weight of the dry raw material. The relative error of the method is ±5%.

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ALKALOIDS OF Ammodendron eichwaldi

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The leaves of Ammodendron eichwaldi Ldb. (family Leguminoseae) collected in the environs of Krasnovodsk in the fruit-bearing period (April 8, 1972) contained 1.8% of total alkaloids.

There is information in the literature only on the qualitative composition of the alkaloids in this plant [1, 2]. The investigation showed that alkaloids are present in all the parts of the plant and they were found in the greatest amount (2.7%) in the glumes of the pods. The alkaloids were determined quantitatively in the various organs by the gravimetric method.

By extraction, 5 kg of air-dry leaves of the plant concerned yielded 45 g of combined alkaloids. This material was separated by successive extraction from alkaline solution with the following solvents: petroleum ether, benzene, and chlorofrom. The petroleum ether fraction (16 g) consisted mainly of pachycarpine, which was identified from the melting point of its hydriodide and by a mixed melting point.

The benzene extract (18 g) by chromatography on a column of alumina (activity grade II) and elution with benzene (fractions 1-15) yielded a base with R_f 0.58 (3.7g) giving a perchlorate with mp 213-215°C a mixture of which with *l*-lupanine perchlorate melted without depression. When the column was eluted with chloroform–benzene (1 : 1), fractions 16-35 yielded a base with R_f 0.41 (0.8 g). The base with R_f 0.41 was an oil giving a crystalline picrate with mp 252-253°C. According to its spectra (UV, IR, and mass spectra) and a direct comparison with an authentic sample, this alkaloid was identified as anagyrine. Fractions 36-52 [chloroform–benzene (1 : 1)] yielded a base with R_f 0.27 (7.2 g), mp 135-136°C, which was identified from its physicochemical properties and spectral characteristics as methylcytisine.

When 13 g of the chloroform fraction was chromatographed on a column of alumina (activity grade II), elution with chloroform–benzene (2 : 1) gave cytisine with mp 155-156°C (0.7 g) (fractions 1-10). From fractions 11-27 we isolated yet another base, with R_f 0.81 (0.6 g), mp 58-60°C, which gave a perchlorate with mp 198-200°C. A mixture of this base with an authentic sample of ammodendrine gave no depression of the melting point. This is the first time that all six of these bases have been isolated from Ammodendron eichwaldi.

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