tion for the nonhydrogen atoms. The positions of the H atoms were calculated and not refined (the H atoms of the methyl groups were not taken into account). The final divergence factors were R = 0.056 and R_w = 0.065. The coordinates of the nonhydrogen atoms are given in Table 2.

Compound (II). The cell parameters and the intensities of the reflections were measured at a low temperature on a Syntex $P2_1$ automatic four-circle diffractometer (150 K, λMoK_α, graphite monochromator, $\theta/2\theta$ scanning, $2\theta \leq 50°$). The crystals were triclinic, a = 9.485(5), b = 12.327(4), c = 19.02(1) Å, α = 92.14(4), β = 98.69(4), γ = 111.66(4)°, V = 2033(2) Å^3, d_{calc} = 1.27 g/cm^3, Z = 4 [$C_{10}H_{15}N_2$ + (iso-$P_2O)_2PO^-$], space group P1, four independent formula units (A, B, C, and D).

In the calculation we used 5971 reflections with $I \geq 3\sigma$. The structure was interpreted by the heavy-atom method and was refined by block-diagonal MLS in the anisotropic approximation for the nonhydrogen atoms. The positions of the H atoms were calculated and were not refined (the H atoms of the methyl groups were not taken into account). The final divergence factors were R = 0.063 and R_w = 0.085. The coordinates of the nonhydrogen atoms are given in Table 3.

All the calculations were performed on Eclipse S/200 computer by means of the INEXTL program [5].

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PREPARATION OF DEOXYPEGANINE HYDROCHLORIDE

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UDC 547.856+661.12

Details are given of the technology of the synthesis and stagewise analysis of the alkaloids deoxyvasicinone and deoxypeganine hydrochlorides.

Deoxypeganine hydrochloride (1,2,3,9-tetrahydropyrrolo[2,1-b]quinazoline hydrochloride) is the hydrochloric acid salt of the alkaloid deoxypeganine, which has been isolated from Peganum harmala [1] and is a highly effective relatively nontoxic anticholinesterase drug which has been introduced into medical practice [2]. The process of its isolation from the plant has serious defects owing to the low yield of product (0.35% on the weight of the raw material), difficulties in the collection and drying of the raw materials, the multistage nature of the process and, as a consequence, its high cost [3].

Investigations of synthetic possibilities of obtaining the alkaloids deoxyvasicinone and deoxypeganine [4, 5] have predetermined the development of a convenient technological scheme for their synthesis which avoids the disadvantages of the technology from the plant raw material.

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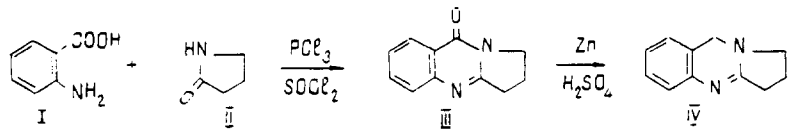

As can be seen from the scheme, the process of obtaining deoxypeganine (IV) is a two-stage batchwise process including the cyclocondensation of anthranilic acid (I) with pyrrolidone (II) in the presence of phosphorus trichloride or thionyl chloride and the reduction of the deoxyvasicinone (III) with zinc in dilute (pH 1-2) sulfuric acid. The deoxypeganine hydrochloride, obtained in 50% yield, is isolated from the reaction mixture by treatment with ammonia followed by its extraction in the form of the base with chloroform, after which the chloroform is distilled off, the residue is treated with hydrochloric acid, and the technical deoxypeganine hydrochloride is isolated by precipitation with acetone.

As our investigation showed, the maximum yield of (III), 70%, is achieved on the use of a twofold excess of (II) and of phosphorus trichloride with respect to the (I) and the performance of the cyclocondensation reaction at 95-105°C for 1.5 h.

The results of mass-spectrometric analysis showed that after the reduction of (III) the technical deoxypeganine contained 4.5-5.2% of the initial deoxyvasicinone and up to 1% of dihydrodeoxyvasicinone [6].

The conditions found for the performance of the cyclocondensation and reduction reactions have been reproduced in an experimental apparatus with a 100-liter reactor.

EXPERIMENTAL

The qualitative and quantitative analysis of the deoxypeganine hydrochloride was performed on a MKh-1310 mass spectrometer by the multipeak monitoring method [7].

Conditions for recording the spectra: SVP-5 direct-introduction system; ionizing voltage 50 V; collector current 40 μA; voltage of the multiplier 5 kV; temperature of the ionizing chamber 100°C; resolving capacity 2000; rate of scanning 12.3 sec per mass decade; recorder chart speed 10 mm/sec.

Production of Deoxyvasicinone (III). A 100-liter reactor was charged with 5.5 liters of pyrrolidone and 15 liters of toluene, and then, with stirring and cooling, 6.5 liters of phosphorus trichloride in 10 liters of toluene was added. The mass so obtained was treated with 5.0 kg of anthranilic acid and 25 liters of toluene, and it was heated with the temperature being maintained at 100°C for one and a half hours. After the end of the reaction, the toluene was separated off and was extracted with 10 liters of water. The aqueous extract and another 40 liters of water were again charged into the reactor and were stirred until the solid matter had dissolved. Then the reaction mixture was neutralized with aqueous ammonia (20 liters), brought to pH 8-9, and extracted four times with 15-liter portions of chloroform. The chloroform extracts were combined, and the (III) was extracted with 10% sulfuric acid solution (six times with 5 liters each time).

To eliminate impurities of nonbasic nature, the acid solution of (III) was washed with 20 liters of chloroform. To determine the yield of (III), 50 ml of the acid solution was taken and was brought to pH 8-9 with aqueous ammonia and was then extracted with chloroform. The chloroform extract was evaporated, and concentrated hydrochloric acid and then acetone were added to the residue. The hydrochloride of (III) so obtained was recalculated to the total volume of the reaction mixture. The yield of (III) was 4.5 kg (70%).

Production of Technical Deoxypeganine Hydrochloride (IV). The acid solution of (III) obtained was charged into the reactor and, with stirring, 6.0 kg of zinc dust was added in such a way as to give a uniform evolution of hydrogen. Then the reaction mixture was heated to the boil and was boiled for 5 h. After the end of the reaction, the cooled solution was filtered from unchanged zinc and was brought to pH 8-9 by treatment with aqueous ammonia. The product was extracted four times with chloroform, the extracts were combined and dried with Na_2SO_4, and the chloroform was distilled off. The evaporated chloroform solution was treated with 35% hydrochloric acid to pH 2-3 and with a tenfold volume of acetone. After filtration, 2 kg of technical product containing 94-95% of deoxypeganine hydrochloride was obtained.

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MASS-SPECTROMETRIC INVESTIGATION OF SOME NEW DERIVATIVES OF THE ALKALOID LUPININE

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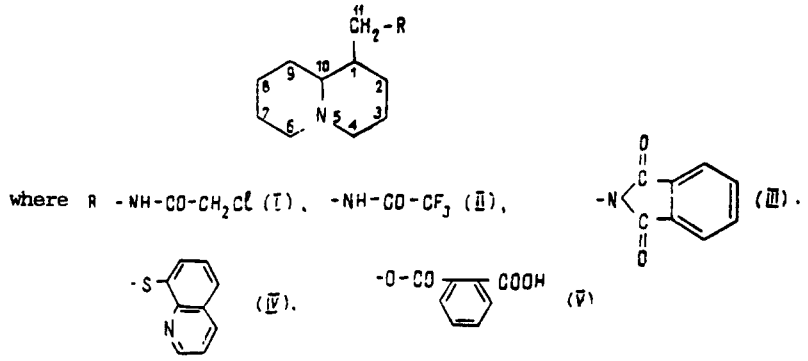
The mass spectra of some new derivatives of lupinine have been studied with the use of the spectra of metastable ions. On the basis of the results of an investigation of DADI spectra it has been shown that in the formation of low-mass quinolizidine ions the ions with m/z 152-150 are of considerable importance.

In the present work we consider the mass spectra of derivatives of 1-methylquinolizidine synthesized with the aim of finding new physiologically active compounds.



Features of the fragmentation of bases of the quinolizidine series have been described fairly fully in the literature [1-6], but at the same time some characteristics of such fragmentation remained unconsidered. We have examined these characteristics for the case of lupinine derivatives, using the spectra of metastable ions. The peaks of the molecular ions of amides (I) and (II) in the corresponding ordinary spectra are fairly strong and the pathways of their fragmentation are reproduced along the line of the ejection of the hydrocarbon units of the quinolizidine ring and of the formation of key nitrogen-containing fragments. The process of fragmentation in the spectrum of monochloroacetylaminolupinane (I) is accompanied by the appearance of the far more intense fragment $(M-HCl)^+$ with m/z 206 and of ions

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