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SYNTHESIS OF DEOXYPEGANINE AND ITS ANALOGS BY THE AMIDOMETHYLATION OF ACETANILIDE WITH N-(HYDROXYMETHYL)LACTAMS

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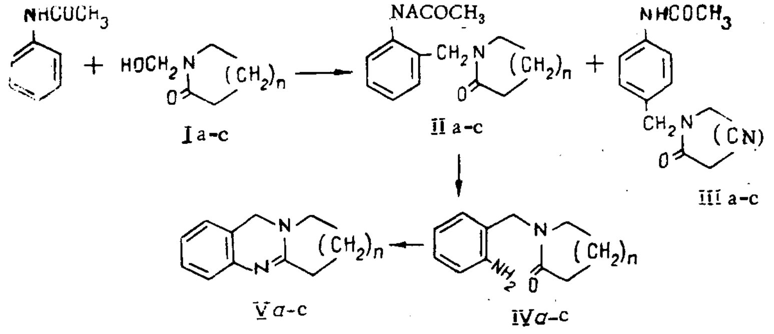
Continuing investigations on the synthesis of deoxypeganine derivatives [1, 2], we have performed the reactions of acetanilide and of p-methoxyacetanilide with N-(hydroxymethyl)lactams. By the reaction of acetanilide with N-(hydroxymethyl)pyrrolidone (Ia), N-(hydroxymethyl)piperidone (Ib), and N-(hydroxymethyl)caprolactam (Ic) in concentrated sulfuric acid at 70°C, we obtained a mixture of the o- and p-acetylaminobenzyl derivatives of pyrrolidone (IIa) and (IIIa), of piperidone (IIb) and (IIIb), and of caprolactam (IIc) and (III)c, respectively.

Performance of the reaction at room temperature led to the formation only of substance (III). The hydrolysis of (II) in an acid medium gave the corresponding o-aminobenzyl derivatives of pyrrolidone (IVa), of piperidone (IVb), and of caprolactam (IVc). The latter, on being treated with water-abstracting agents, cyclized to form 2,3-trimethylene-, 2,3-tetramethylene-, and 2,3-pentamethylene-3,4-dihydroquinazolines (Va, b, and c, respectively). In its physicochemical properties, (Va) was completely identical with deoxypeganine [1, 3]. Substance (Vb) was identical with an alkaloid isolated from the plant Mackilaya [4] and proved to belong to a new class of quinazoline alkaloids. Compound (Vc) has been obtained previously by a multistage method from 1-(o-aminobenzyl)hexamethyleneimine [5]. The reaction of p-methoxyacetanilide with (Ia) at 20°C formed (5-acetylamino-2-methoxybenzyl)pyrrolidone (VI). Its structure was shown by its PMR spectrum, which contained the signals of protons in the following regions: (ppm): 2.0 (the protons of two methyl groups); 2.3, 3.2, and 3.7, (the protons of three methylene groups) 4.4 (aminomethylene group); and 7.1 and 7.3 ppm (protons of an aromatic ring).



Throughout a: $n = 1$; b: $n = 2$; c: $n = 3$

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TABLE 1

Reaction product	Reaction temperature, °C	Yield, %	Solvent for recrystallization	Chloroform–ether (1:1), Al_2O_3	Mol. wt. (mass spectrometrically)
IIa	70	5	124–125 (hexane)	0 50*	—
IIIa	"	41	162–163 (benzene)	0,24*	232
IIIa	20	44,5	162–163 (benzene)	0,24*	232
IIb	70	4,3	92–94 (hexane)	0,26	246
IIIb	"	22,4	154 (benzene)	0,09	—
IIc	70	2,6	132–134 (hexane)	0,32	—
IIIc	"	21	166–167 (benzene)	0.1	—
IVa†	—	32	72–73 (benzene–petroleum ether)	—	—
IVb†	—	30	68–69 (hexane)	0,09	—
Va‡	—	35	86–87 (hexane)	0,16	172
Vb	—	33,3	79–81 (hexane)	0,15	—
Vc	—	24	94–96 (ether)	0,12	—
VI	20	54	163–164 (benzene)	0,04	—

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*The R_f values were determined on silica gel in the chloroform–methanol (25 :1) system.

†Compound (IVa, b) were obtained by the hydrolysis (IIa, b)

‡Compounds (Va-c) were isolated from (IVa-c).

The yields and some physicochemical properties of the compounds obtained are given in Table 1.

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ALKALOIDS OF Corydalis severtzovii

STRUCTURE OF SEVERTZINE

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Continuing a study of the alkaloid composition of various organs of the plant Corydalis severtzovii from different growth sites [1-3], we have investigated the epigeal part of the plant collected on April 21, 1973 in the Chimkent oblast of the Kazakh SSR in the flowering period. Chloroform extraction gave 1.06% of the total alkaloids. From the nonphenolic ether-soluble alkaloids we isolated protopine, allocryptopine, cryptopine, corlumine, and a base with mp 195-196°C; from the phenolic ether-soluble alkaloids we isolated four bases which were identified by comparing their spectral characteristics, by the preparation of salts, and also by mixed melting points with authentic samples as *l*-scoulerine [4], coramine [5], coclaurine [6], and isoboldine [7], and a new base with mp 94-95°C (with foaming; from methanol), $[\alpha]_D - 52°$ (c 0.91; chloroform) which we have called severtzine.

The UV spectrum of the base has three maxima at 221, 291, and 326 nm (log 4.45, 3.64, and 3.76, and the IR spectrum has absorption bands at (cm^{-1}), 920, 1050 (methylenedioxy group), 1600 (aromatic

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