DERIVATIVES OF AZAINDOLES.

65.* SYNTHESIS OF 1-BENZYL-4-METHYL-5-CYANO-6-HYDROXY-7-AZAINDOLINE

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Treatment of the ammonium (I) or benzylammonium salt of 2,6-dihydroxy-3-(β-hydroxyethyl)-4-methyl-5-cyanopyridine (II)† with a mixture of benzylamine and phosphorus pentoxide yielded 2-benzylamino-3-(β-hydroxyethyl)-4-methyl-5-cyano-6-hydroxypyridine (III), which, when heated with phosphorus oxychloride, is converted to 1-benzyl-4-methyl-5-cyano-6-hydroxy-7-azaindoline (IV). The products of thermal fragmentation of II with benzylamine were studied by the method of chromato-mass spectrometry. In addition to compound III, N,N'-dibenzylurea (V) and the dibenzylamide of malonic acid (VI) were preparatively isolated from the reaction products. The cyclization of I and II to 4-methyl-6-hydroxy-2,3-dihydro-7-azabenzofuran (VII) and 4-methyl-5-cyano-6-hydroxy-2,3-dihydro-7-azabenzofuran (VIII) was carried out. Heating VIII with benzylamine at 200-210°C led to compound III.

In recent years data have appeared [3, 4] on the intensification of the β-adrenoblocking activity and the appearance of vasodilative, as well as hypotensive properties when cyano-substituents are introduced into pyridine or indole molecules together with the 3-isopropylamino-2-hydroxypropoxy group. These compounds evoked interest in the synthesis and study of new cyano-containing β-adrenoblockers of the N-heteroaromatic series. According to the data of [1], 1-benzyl-6-(3-isopropyl-amino-2-hydroxypropoxy)-7-cyano-5-azaindoline is 10 times as active as the known drug preparations propranolol and pindalol (visken) in β-adrenoblocking activity in experiments *in vitro*. To obtain the 7-azaindolinyl analog of this compound, 1-benzyl-4-methyl-5-cyano-6-hydroxy-7-azaindoline (I) can be used as the starting material. The production of compound I, undescribed in the literature, is a nontrivial problem. General methods of synthesis of 7-azaindolines developed earlier on the basis of the reactions of amines with 2-chloro-3-(β-chloroethyl)pyridines [5] or 2,3-dihydro-7-azabenzofurans [6] do not give positive results in this case: the first since the corresponding 2-chloro-3-(β-chloroethyl) pyridine cannot be obtained from the ammonium salt of 2,6-dihydroxy-3-(β-hydroxyethyl)-4-methyl-5-cyanopyridine (II) without elimination of the cyano group, and the second since 4-methyl-5-cyano-6-hydroxy-2,3-dihydro-7-azabenzofuran (III) undergoes thermal fragmentation at the temperatures necessary for conversion of the dihydrofuran ring to a pyrroline ring.

We developed the synthesis of 1-benzyl-4-methyl-5-cyano-6-hydroxy-7-azaindoline (I) according to the scheme cited on the following page.

The ammonium salt of II, produced by the reaction of cyanoacetamide with α-acetobutyrolactone according to the method of Stevens et al. [7], modified by M. V. Rubtsov [8], in the case of treatment with sufficiently strong basic and relatively nonvolatile amines, is converted to new ammonium salts and, in particular, forms the benzylammonium salt IV with a yield of 92% when it is heated with benzylamine (4 h at 150-160°C). Interaction of the salt II with stronger bases — aromatic amines — does not permit the production of analogous arylammonium salts: Even after prolonged boiling with aniline, compound II is recovered virtually entirely unchanged.

*For communication 64, see [1].

†Here and henceforth the names of the compounds will be cited without consideration of the state of the lactam–lactim tautomeric equilibrium, which, according to the data of [2], usually is greatly shifted in the direction of the 2-hydroxy-6-oxo-tautomers for 2,6-dihydroxypyridine compounds. The structural formulas of the compounds reflect the predominant tautomeric forms.

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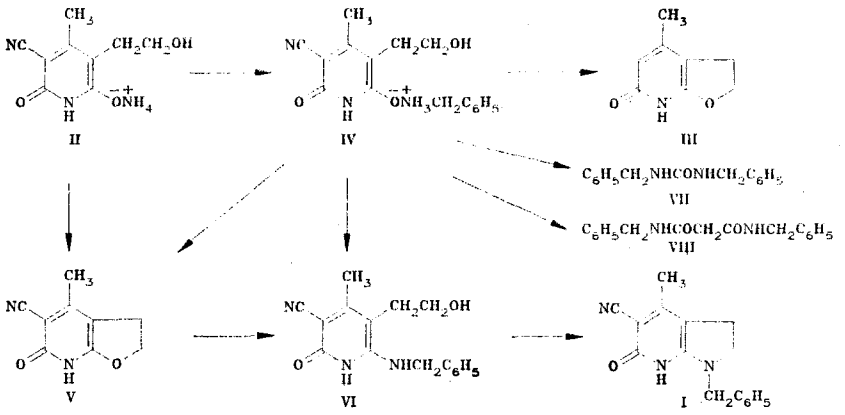

Heating the benzylammonium salt IV with 65% sulfuric acid, analogously to that described for compound II [8], leads to a loss of the cyano group, closing of the dihydrofuran ring, and the formation of 4-methyl-6-hydroxy-2,3-dihydro-7-azabenzofuran (III). The use of phosphorus oxychloride in this reaction instead of sulfuric acid permitted us to carry out the cyclization without elimination of the cyano group and to obtain 4-methyl-5-cyano-6-hydroxy-2,3-dihydro-7-azabenzofuran (V) with a yield of 97%. In contrast to the previously described [6] recyclization of 2,3-dihydro-5-azabenzofurans in the case of heating to 200°C with benzylamines to the corresponding substituted 5-azaindolines, compound V under the same conditions does not form a bicyclic 7-azaindoline derivative I but is converted with a 79% yield to monocyclic 2-benzylamino-3-(β-hydroxyethyl)-4-methyl-5-cyano-6-hydroxypyridine (VI). Conducting the reaction at 245°C leads to the appearance of negligible amounts of the 7-azaindoline compound I, detected in the reaction products by the method of thin-layer chromatography. However, the process of fragmentation of the pyridine molecule chiefly occurs; one of its products — N,N'-dibenzylurea (VII) — could be preparatively isolated with a 30% yield. Fragmentation of the pyridine ring and thermal conversions of benzylamine also occur when benzylammonium (IV) or ammonium (II) salts of 2,6-dihydroxy-3-(β-hydroxyethyl)-4-methyl-5-cyanopyridine are heated with benzylamine to 200-210°C (10 h). The yield of the 2-benzylamino derivative VI in the first case is 44-50%, in the second 38%. Products of fragmentation of the pyridine ring — N,N'-dibenzylurea (VII) and the dibenzylamide of malonic acid (VIII), identified according to mixed melting points and according to the IR spectra with known samples produced by counter-synthesis according to the methods of [9, 10] — were preparatively isolated. Chromato-mass spectrometric analysis of the complex mixture remaining suggested possible structures (yield) for the other fragmentation products: 5-hydroxy-3-pentyn-2-one (6%), 2-pentan-4-on-1-al (22%), 3-(α-methyl-β-formylvinyl)-4,5-dihydrofuran (13%), dibenzylamine (9%), 1-benzyl-3-acetyl-2,3-dihydropyrrolidine (2%), 1,6-dihydroxy-3-vinyl-4-methylpyridine (2.8%), and 2-acetylbenzylamine (2%). More than 10 additional minor unidentified substances were detected in this mixture.

The benzylammonium salt of IV is converted most smoothly to 2-benzylamino-3-(β-hydroxyethyl)-4-methyl-5-cyano-6-hydroxypyridine (VI) in treatment with a mixture of benzylamine and phosphorus pentoxide for 12 h at 150-170°C, i.e., under the conditions recommended earlier [11] for one-step conversion of hydroxy(oxo)-N-heteroaromatic compounds to the corresponding amino derivatives. The yield of compound VI according to the method of [11] is 73%.

Closing of the pyrroline ring in compound VII is accomplished by heating with phosphorus oxychloride in the presence of dimethylaniline. The cyclization process is not accompanied by replacement of the hydroxy group by chlorine in the 6-position, although under analogous conditions [12], 6-hydroxy-7-cyano-5-azaindolines are smoothly converted to the corresponding 6-chloro-7-cyano-5-azaindoline derivatives.

The unambiguous conversion of the salts II and IV through 2-benzylamino derivative VI to the 7-azaindoline I convincingly shows that in 2,6-dihydroxy-3-(β-hydroxyethyl)-4-methyl-5-cyanopyridine, the most acid is the hydroxyl in the 2-position, and salt formation of the indicated compound with ammonia and amines proceeds, if not entirely than largely at this hydroxyl. All the aforementioned permits us to give up the previously accepted image of such salts as dihydroxypyridine derivatives bonded to amines through a point [7, 8] and change to the writing of structural formulas of the type of II and IV.

EXPERIMENTAL

The IR spectra were recorded on a Perkin-Elmer 457 spectrometer in liquid petrolatum, the PMR spectra on a JNH-4H-100 instrument with internal standard TMS, the mass spectra on a Varian MAT-112 instrument at 70 eV; the chromato-mass spectrometric investigations were conducted on a chromato-mass spectrometer from Varian, chromatographic column 1 m long with cross-section 0.2 cm, stationary phase OV-101 on chromosorb WHP, column temperature 100°C, temperature of inlet 240°C, temperature of detection 230°C, carrier gas helium. For a quantitative analysis, benzylamine was used as the standard; an exact weighed sample of it with C ~ 1/2 of the weight of the test sample was introduced into the instrument as an internal standard; analysis was performed by comparing the areas of the test and standard samples, and the yield was calculated on the basis of the amounts of the starting materials taken for the reaction. The retention time of the peaks is cited in min (for benzylamine under these conditions, the retention time is 0.4 min). Thin-layer chromatography was conducted on Silufol UV-254. Chromatography was conducted in a mixture of CH_3COOH—C_4H_9OH—H_2O; 1:5:4.*

Benzylammonium Salt of 2,6-Dihydroxy-3-(β-hydroxyethyl)-4-methyl-5-cyanopyridine (IV). A mixture of 4 g (18 mmoles) of the ammonium salt II and 5.67 g (54 mmoles) benzylamine was mixed for 4 h at 150-160°C. The cooled reaction mass was triturated with 200 ml of ether, the precipitate filtered off and washed with 200 ml of acetone. Yield 5.28 g (93%) of the benzylammonium salt IV. Colorless crystals, mp 222-223°C (from methanol). The substance is soluble in water, DMFA, and hot alcohols and insolute in ether, benzene, and acetone; R_f 0.46. IR spectrum: 3450, 3250, 3060 (NH, NH_2), 2170 (C≡N), 1600 cm^{-1} (CO). Mass spectrum†: 194 $[M_1]^{+\bullet}$ (20); 176 $[M - H_2O]^{+\bullet}$ (82); 163 $[M - CH_2OH]^+$ (100); 107 $[M_2]^{+\bullet}$‡. PMR spectrum (in CD_3OD): 2.24 (3H, s, CH_3), 2.67 (2H, t, CH_2CH_2OH, J = 5.2 Hz), 3.59 (2H, t, CH_2CH_2OH, J = 5.2 Hz), 4.10 (2H, s, $CH_2C_6H_5$); 7.46 ppm (5H, s, $CH_2C_6H_5$). Found: C 63.7; H 6.4; N 13.7%. $C_{16}H_{19}N_3O_3$. Calculated: C 63.8; H 6.3; N 13.9%.

4-Methyl-6-hydroxy-2,3-dihydro-7-azabenzofuran (III). To 1 g (3.3 mmoles) of the benzylammonium salt IV, a hot freshly prepared mixture of 4 h of sulfuric acid (d 1.83) and 1.7 ml of water was added, and the mixture was boiled for 6 h. The homogeneous solution formed was diluted with an equal volume of water and treated upon cooling with a concentrated aqueous solution of ammonia to pH 5-6. The precipitate formed was filtered off, washed with water until sulfate ions disappeared in the filtrate (test with $BaCl_2$), and dried first in air and then in a drying oven for 5 h at 110-120°C. Yield 0.43 g (86%) of the furan III, identical according to a mixed melting point test and according to the IR spectrum with a known sample of III prepared from the salt II according to the method of [8].

4-Methyl-5-cyano-6-hydroxy-2,3-dihydro-7-azabenzofuran (V). A mixture of 4.22 g (20 mmoles) of the ammonium salt II, 60 ml $POCl_3$, and 2 ml dimethylaniline was boiled for 5 h and evaporated under vacuum; the residue was treated with 20 g of crushed ice. The mass obtained was boiled for 30 min, cooled, and neutralized with a 10% potassium hydroxide solution. The precipitate was filtered off and washed with 150 ml of water and 30 ml of acetone. Yield 3.4 g (97%) of compound V. The crystals were white with a pink tinge, mp 269-270°C (from methanol). The substance was soluble in DMFA and hot alcohols, insoluble in water, ether, ethyl acetate, chloroform, and acetone. IR spectrum: 2200 (C≡N), 1600 cm^{-1} (CO). Mass spectrum: 176 $[M]^{+\bullet}$ (100), 161 $[M-CH_3]^+$ (5), 158 $[M-H_2O]^{+\bullet}$ (2), 148 $[M-C_2H_4]^{+\bullet}$ (9). Found: C 61.6; H 4.7; N 16%. $C_9H_8N_2O_2$. Calculated: C 61.4; H 4.6; N 15.9%.

*The PMR spectra were recorded by K. F. Turchin and T. Ya. Filipenko, the IR spectra by Yu. I. Pomerantsev, the mass spectra with the participation of O. S. Anisimova at the Laboratory of Physicochemical methods of investigation of the All-Union Pharmaceutical Chemistry Scientific-Research Institute (Supervisor: Professor Yu. N. Sheinker), microanalysis in R. A. Dubinskii's group, gas—liquid chromatographic analysis by V. A. Kuzovskin and L. M. Budanova in the analytical laboratory of the All-Union Pharmaceutical Chemistry Scientific-Research Institute (Supervisor: Candidate of Chemical Sciences V. E. Degtyarev).

†The values of m/z of the molecular peaks and some characteristic fragments are cited; the intensities are given in % relative to the intensity of the maximum peak.

‡Under the conditions of a mass spectrometric experiment, salts of the type of IV appear in the form of a summary spectrum of the corresponding base and acid. At the temperature of fractionation of the sample in an ion source, the spectrum of the benzylamide is initially observed ($[M_2]^{+\bullet}$ 107), and upon further heating, the spectrum of the corresponding dihydroxypyridine derivative ($[M_1]^{+\bullet}$ 194).

2-Benzylamino-3-(β-hydroxyethyl)-4-methyl-5-cyano-6-hydroxypyridine (VI). A. A mixture of 1.2 g (4 mmoles) of the benzylammonium salt IV, 1.4 g (12 mmoles) benzylamine and 0.57 g (4 mmoles) phosphorus pentoxide was exposed for 12 h at 150-170°C, treated with 100 ml of water, the precipitate filtered off and washed with 70 ml of hot acetone. Yield 0.82 g (73%) of substance VI. Colorless crystals, mp 284-285°C (from glacial acetic acid). The substance was soluble in conc. H_2SO_4, hot acetic acid, and DMFA, and insoluble in the other usual organic solvents and in water. IR spectrum: 3080, 3020 (NH), 2200 (C≡N), 1600 cm^{-1} (CO). PMR spectrum (in DMFA-D_6): 2.14 (3H, s, CH_3); 3.06 (2H, t, CH_2CH_2OH, J = 5.2 Hz); 3.14 (2H, t, CH_2CH_2OH, J = 5.2 Hz); 4.42 (2H, s, $CH_2C_6H_5$); 7.50 ppm (5H, m, $CH_2C_6H_5$). Mass-spectrum: 283 $[M]^{+\bullet}$ (29); 265 $[M-H_2O]^{+\bullet}$ (18); 176 $[M-NH_2CH_2C_6H_5]^+$ (100). Found: C 67.6; H 5.8; N 14.8%. $C_{16}H_{17}N_3O_3$. Calculated: C 67.8; H 6.0; N 14.8%.

B. To 1 g (5.6 mmoles) of the azabenzofuran V we added 5 g (46.7 mmoles) of benzylamine, heated for 8 h, mixing, at 200-210°C and evaporated under vacuum. The precipitate was washed by boiling with 10 ml of acetone and filtered off. Yield 1.27 g (79%) of substance VI, mp 284-285°C.

In an analogous experiment, which was carried out in 8 h at 245-250°C, after evaporation of the reaction mass under vacuum and washing of the greatly resinified residue with 10 ml of cold acetone, 0.4 g (30%) of N,N'-dibenzylurea (VII) was obtained, mp 169-170°C. The substance gives no depression in a mixed melting point test with a known sample [9] and has an IR spectrum identical with it. A negligible amount of the 7-azaindoline derivative with R_f 0.85 was detected by the method of thin-layer chromatography in the evaporated acetone mother liquor after the removal of substance VII. The identity of the substance formed with 7-azaindoline (see below) was confirmed by a comparison of the chromatographic mobility in various systems: benzene–methanol, 20:1 (R_f 0.08); benzene–ethyl acetate, 1:1 (R_f 0.40); chloroform–methanol, 9:1 (R_f 0.40). However, on account of the low content of compound I in the reaction mass and the substantial resinification, it could not be isolated in pure form in this experiment.

C. A mixture of 3 g (10 mmoles) of the benzylammonium salt IV and 3.2 g (30 mmoles) of benzylamine was heated for 12 h at 200-210°C. The reaction mass was evaporated under vacuum, the residue triturated with 25 ml of hot acetone. Yield 0.87 g (31%) of compound VI, mp 284-285°C.

D. A mixture of 6 g (17 mmoles) of the ammonium salt II and 8.76 g (81 mmoles) benzylamine was mixed for 12 h at 200-210°C and evaporated under vacuum. The residue was washed with 50 ml of hot acetone. Yield 3.01 g (38%) of substance VI. In recrystallization of the entire reaction mass from 300 ml of glacial acetic acid, 1.77 g of substance VI with mp 284-285°C precipitated. Another 1 g of the less pure substance VI was obtained from the mother liquor after evaporation.

The acetone mother liquor was evaporated and the residue (8.22 g) triturated with 70 ml of ethyl acetate. The ethyl acetate solution was filtered, evaporated, and the residue (6.6 g) mixed with 150 ml of benzene. The substance that did not dissolve in benzene was filtered off and washed with benzene. Yield 0.4 g of N,N'-dibenzylurea (VII), mp 169-170°C. The substance gave no depression in a mixed melting point test with a known sample [9] and had an IR spectrum identical with it.

The benzene mother liquor after removal of urea VII was applied on a column with 250 g of silica gel 100/160 μm (column diameter 30 mm, height 80 cm). It was eluted first with 1 liter of benzene, then with 800 ml of ethyl acetate and 2.6 liters of acetone. When the ethyl acetate eluate was evaporated to 1/4 of the original volume, 0.09 g of crystallized dibenzylamide of malonic acid (VIII) was obtained, mp 138-139°C, identical according to a mixed melting point test and the IR, PMR, and mass spectra with a known sample prepared according to the method of [10]. Benzene, ethyl acetate, and acetone eluates were evaporated to dryness separately, and the residues (0.37, 1.41, and 1.92 g, respectively), were subjected to chromato-mass spectrometric analysis. In this case, the following main products were detected and identified according to their mass spectra (cited: yield in percent of the theoretical on the basis of the starting materials used in the reaction, retention time in min, values of m/z of the peaks in the mass spectra and their relative intensities in percent of the intensity of the maximum peak): in the ethyl acetate eluate – N-acetylbenzylamine {0.6; 1.5; 149 $[M]^{+\bullet}$ (58); 106 $[M-COCH_3]^+$ (100); 91 $[M-NHCOCH_3]^+$ (33)}; dibenzylamine {8.5; 4; 197 $[M]^+$ (18); 120 $[M-C_6H_5]^+$ (14); 106 $[M-C_6H_5CH_2]^+$ (100); 91 $[C_6H_5CH_2]^+$ (79)}; 1-benzyl-3-acetyl-2,3-dehydropyrrolidine {2; 5; 201 $[M]^{+\bullet}$ (100); 158 $[M-COCH_3]^+$ (4); 124 $[M-C_6H_5]^+$ (14); 110 $[M-C_6H_5CH_2]^+$ (57); 91 $[C_6H_5CH_2]^+$ (24)} and dibenzylurea {0.2; 8; 240 $[M]^{+\bullet}$ (10); 149 $[M-C_6H_5CH_2]^+$ (16); 133 [M–

$C_6H_5CH_2NH_2]^+$ (18); 106 $[C_6H_5CH_2NH]^+$ (100); 91 $[C_6H_5CH_2]^+$ (88)}; in the acetone eluate — 3-(α- methyl-β-formylvinyl)-4,5-dihydrofuran {1.3; 1.3; 138 $[M]^{+\bullet}$ (100); 123 $[M-CH_3]^+$ (53); 109 $[M-CHO]^+$ (19)}; 5-hydroxy-3-pentyn-2-one {6; 0.6; 98 $[M]^{+\bullet}$ (31); 83 $[M-CH_3]^+$ (79); 55 $[M-CH_3-CO]^+$ (100)}; 2-penten-4-on-1-al {22; 1.1; 98 $[M]^{+\bullet}$ (33); 83 $[M-CH_3]^+$ (8); 80 $[M-H_2O]^{+\bullet}$ (5); 55 $[M-CH_3CO]^+$ (100); 70 $[M-CO]^{+\bullet}$ (22); 69 $[M-HCO]^+$ (27)}; N-acetylbenzylamine (1.3) and 2,6-dihydroxy-3-vinyl-4-methylpyridine {2.8; 2; 151 $[M]^{+\bullet}$ (88); 123 $[M-CO]^{+\bullet}$ (18); 122 $[M-HCO]^+$ (25); 108 $[M-CONH]^{+\bullet}$ (50); 67 $[M-C_6H_7]^+$ (100)}.

1-Benzyl-4-methyl-5-cyano-6-hydroxy-7-azaindoline (I). A mixture of 1.13 g (4 mmoles) of substance VI, 15 ml of phosphorus oxychloride, and 0.5 ml of dimethylaniline was boiled for 5 h, after which the reaction mass was evaporated under vacuum. To the residue we added 5 g of crushed ice, heated the mixture to boiling, and exposed it for 30 min. Then the mass was cooled, neutralized with a 10% aqueous solution of potassium hydroxide, the precipitate filtered off and washed with 50 ml of water, 20 ml of ether, and 5 ml of acetone. Yield 1.02 g of a mixture (according to the data of thin-layer chromatography) of substances VI and I. To remove the azaindoline I the mixture was boiled with 120 ml of acetone, and the undissolved 0.82 g of compound VI was filtered off. The acetone solution was evaporated, yielding 0.24 g of the azaindoline I. Then 10 ml of phosphorus oxychloride and 0.4 ml of dimethylaniline were added to the 0.82 g of compound VI recovered from the reaction, boiled for 5 h, and the treatment of the reaction mass described above was repeated. We obtained 0.59 g (52%) of unreacted substance VI, which could be reused for the production of the azaindoline I, and 0.19 g of substance I, isolated after evaporation of the acetone solution. The total yield of the azaindoline I was 0.43 g (41%). The crystals were white with a greenish tinge, mp 284-285°C (from acetone). The substance was soluble in DMFA, hot acetone and alcohols, and insoluble in water, ethyl acetate, and chloroform. IR spectrum: 2190 (C≡N), 1600 cm^{-1} (CO). PMR spectrum (in DMSO)-D_6); 2.09 (3H, s, CH_3); 2.85 (2H, t $\mathbf{CH_2}CH_2N$, J = 8.5 Hz); 3.37 (2H, t, CH_2CH_2N, J = 8.5 Hz); 5.01 (2H, s, $\mathbf{CH_2}C_6H_5$); 7.50 ppm (5H, s, $CH_2C_6H_5$). Mass spectrum: 265 $[M]^{+\bullet}$ (100); 250 $[M-CH_3]^+$ (3); 188 $[M-C_6H_5]^+$ (27); 174 $[M-C_6H_5-CH_2]^+$ (18). Found: C 72.8; H 5.7; N 15.9%. $C_6H_{15}N_3O$. Calculated: C 72.5; H 5.7; N 15.8%.

The addition of phosphorus pentachloride to the reaction mass or a 10-fold increase in the amount of dimethylaniline did not permit any increase in the yield of the azaindoline I.

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