AZAINDOLES.

69.* SOME REACTIONS OF 5-CYANO-6-CHLORO-7-AZAINDOLES AND LACTAM–LACTIM TAUTOMERISM IN 5-CYANO-6-HYDROXY-7-AZAINDOLINES

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Electrophilic substitution in the 3-position of 1-benzyl-4-methyl-5-cyano-6-chloro-7-azaindole requires more severe conditions than in 7-azaindoles without the 5-cyano-substituent. Increased ease of nucleophilic replacement of the chlorine atom by the methoxy group has been observed in 1-benzyl- (and 1-butyl) -4-methyl-5-cyano-6-chloro-7-azaindoles, and the cyano-group in these compounds has been found to be resistant to hydrolysis and alcoholysis. The introduction into 1-benzyl- (and 1-butyl) -4-methyl-6-hydroxy-7-azaindoles of a 5-cyano-substituent results in a shift of the lactam–lactim tautomeric equilibrium towards the lactim forms.

We have previously developed a general method for the synthesis of 4-methyl-5-cyano-7-azaindolines containing an aralkyl or alkyl substituent in the 1-position and hydroxy or halogen in the 6-position [2], and we have examined the conversion of these compounds into the corresponding 7-azaindoles [3]. The development of these methods enabled us to undertake a comparative examination of the reactivity and tautomerism of the compounds obtained and of analogous compounds without a cyano-group in the 5-position.

In contrast to the previously described oxidation of 1-benzyl-4-methyl-6-chloro-7-azaindoline with chloranil in boiling benzene, when in addition to dehydrogenation of the 7-azaindoline system N-debenzylation also occurred with the formation of 4-methyl-6-chloro-7-azaindoline as the main product [4], treatment of 1-benzyl-4-methyl-5-cyano-6-chloro-7-azaindoline (Ia) with activated manganese dioxide in carbon tetrachloride at 20°C gave more than 70% of 1-benzyl-4-methyl-5-cyano-6-chloro-7-azaindole (IIa). Similar treatment of 1-n-butyl-4-methyl-5-cyano-6-chloro-7-azaindoline (Ib) gave more than 65% of the corresponding azaindole (IIb).

From the general features of the effects of substituents on the reactivity of azaindoles [5], it would be expected that the introduction of the electron-acceptor cyano-group in the 5-position would hinder electrophilic substitution in the 3-position which is characteristic of 7-azaindoles. The correctness of this assumption has been shown by experiment. We have examined the electrophilic replacement of hydrogen in the 3-position of 1-benzyl-4-methyl-5-cyano-6-chloro-7-azaindole (IIa) by halogens and the acetyl and dialkylaminomethyl residues.

In these electrophilic substitution reactions, the presence of the 5-cyano substituent in the 7-azaindole resulted in more severe conditions being required than in the reactions of compounds without this group. For example, bromination of (IIa) to (III) by dioxane dibromide in dioxane at 20°C required the use of double the amount of brominating agent and a doubling of the reaction time as compared with the analogous reaction with 4-methyl-6-chloro-7-azaindole [6]. A doubling of the reaction time and an increase in temperature were also required in the acylation of (IIa) to (IV) by acetyl chloride, as compared with the analogous reaction with 6-azaindole [7].

Even greater differences in reactivity between the 5-cyano- and unsubstituted 7-azaindoles was apparent when Mannich reagents were used to obtain (V) and (VI). Normally, the Mannich reaction in the 7-azaindole series gives quite good yields (60%) on boiling in

*For communication 68, see [1].

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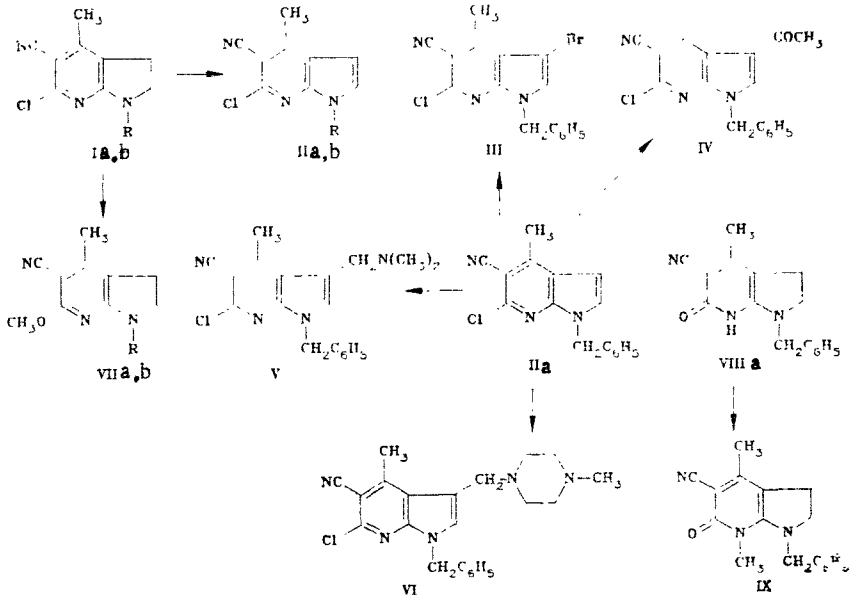
Scheme

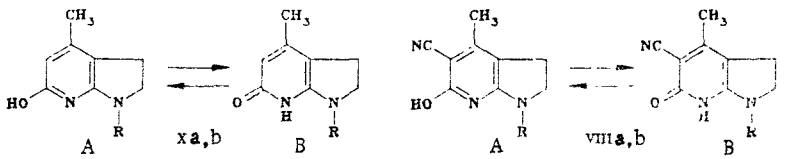


I, II, VII a $R=CH_2C_6H_5$, b $R=n\text{-}C_4H_9$

butanol for 5-30 min [6]. In the present case, however, even on boiling the azaindole (IIa) with a mixture of dimethylamine hydrochloride and paraformaldehyde for 30 h, the yield of (V) was only 13.8%. Only when a large excess of crystalline bis(dimethylamino)methane was used, added portionwise, in place of the mixture of paraformaldehyde and dimethylamine hydrochloride, and on prolonged boiling (up to 5 h) in butanol was the yield of (V) increased to 30%. As would be expected, the presence of the electron-acceptor 5-cyano group facilitates the nucleophilic replacement of the chlorine in the 6-position of the azaindoline. We have previously noted that 1-benzyl-4-methyl-5-cyano-6-chloro-7-azaindoline is readily converted into N-substituted 1-benzyl-4-methyl-5-cyano-6-amino-7-azaindolines on treatment with primary and secondary amines [3]. Reaction of 5-cyano-6-chloro-7-azaindolines (Ia, b) with sodium methoxide in dry dimethylformamide at 80-82°C for 6 h gave high yields (71-73%) of the products of replacement of the chlorine in the 6-position by methoxy, the cyano-group being completely unaffected, to give (VIIa, b). It is noteworthy that, as in other 5-cyano-6-chloro-7-azaindolines [8], the 5-cyano-group in (Ia, b) is chemically quite inert, failing to undergo alcoholysis on treatment of the methanolic solution with gaseous hydrogen chloride for 5 h at 20°C, and being resistant to hydrolysis by prolonged boiling with 10% hydrochloric acid or 7% aqueous sodium hydroxide.

Of considerable interest is a study of lactam–lactim tautomerism in the 5-cyano-6-hydroxy-7-azaindolines (VIIIa, b).

We have shown previously that the –I effect of the nitrogen in the five-membered ring in 6-hydroxy-7-azaindolines of general formula (Xa, b) results in a shift in the lactam–lactim tautomeric equilibrium towards the lactims A, and in solution these compounds exist as mixtures of comparable amounts of tautomeric forms A and B [9-11]. In the case of 7-substituted 4-chloro-6-hydroxy-2,3-dihydro-5-azabenzofurans, it has been found that the –I effects of substituents in the m-position to the nitrogen of the pyridine ring and in the position α to the hydroxy group result in a shift in the tautomeric equilibrium towards the lactam form [12].



VIII a $R=CH_2C_6H_5$, b $R=n\text{-}C_4H_9$; X a $R=n\text{-}C_4H_9$, b $R=C_6H_5$

The lactam–lactim tautomerism has been studied by UV spectroscopy.* The "fixed" forms used for the lactims A were the 6-methoxy derivatives (VIIa, b), the synthesis of which has

*The UV spectra were obtained by T. Yu. Kurbatova, whom the authors thank.

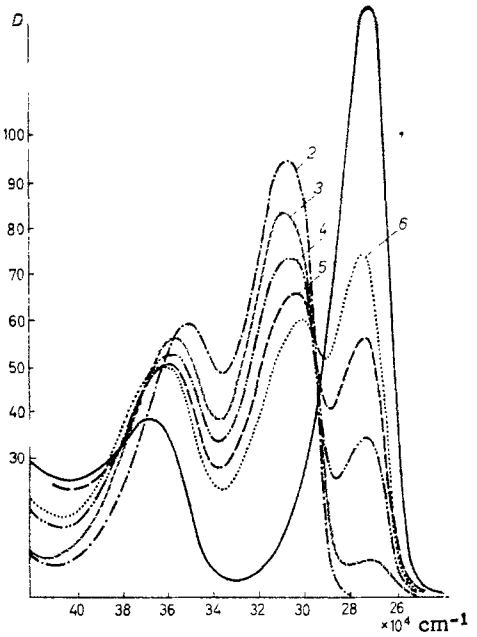


Fig. 1

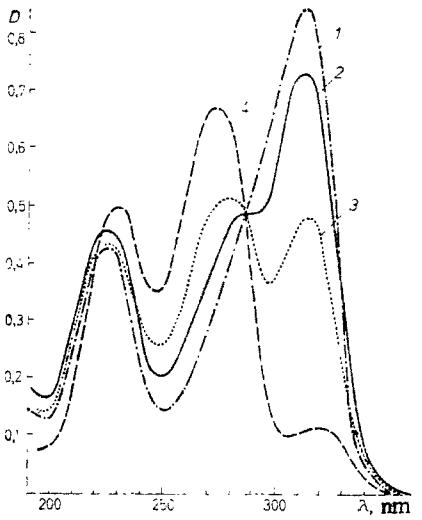


Fig. 2

Fig. 1. UV spectra of (IXa) in alcohol (1), (VIIa) in dioxane (2), (VIIIa) in alcohol (3) and in mixtures of alcohol and dioxane: 90% dioxane (4), 50% dioxane (5), and 20% dioxane (6).

Fig. 2. UV spectra of (VIIIb) in alcohol (1) and in mixtures of alcohol and dioxane: 20% dioxane (2), 50% dioxane (3), and 90% dioxane (4).

been described above, and for the lactams B, 1-benzyl-4,7-dimethyl-5-cyano-6-oxo-7-azaindoline (IXa), obtained by methylating (VIIIa) with dimethyl sulfate in the presence of sodium methoxide. The positions and intensities of the UV absorption maxima for (VII) and (IXa) remained virtually unchanged in solvents of differing polarity (dioxane and alcohol). In contrast, in the spectra of 5-cyano-6-hydroxy-7-azaindolines (VIII) the positions and intensities of the absorption maxima were highly dependent on the nature of the solvent (Figs. 1 and 2). For example, in dioxane the UV spectra of (VIIIa, b) approximate to those of the "fixed" lactim forms (VIIa, b) for which the λ_{max} (log ε) values are 285 (3.40 ± 0.04), 325 nm (3.6 ± 0.3) for (VIIb) and 365 nm (3.63 ± 0.03) for (VIIa). As the amount of alcohol in the solvent mixture was increased, intensity of the maximum at 325 nm gradually decreased, with a simultaneous increase in the UV absorption at longer wavelengths (365 nm), reaching a maximum in 100% alcohol, when the appearance of the curves approximated to the UV spectrum of the "fixed" lactam form (IXa), for which the λ_{max} (log ε) values are 369 (3.7 ± 0.3) and 323 nm (3.41 ± 0.3). An isobestic point is clearly apparent on the plots, indicating the presence of two tautomers. The changes in the spectra with changes in the solvent are reversible, and are clearly due to shifts in the lactam–lactim equilibrium. The proportions of the lactam and lactim forms were determined as percentages from the UV spectral data, and the tautomeric equilibrium constants K_T calculated. The results obtained are shown in Table 1. Comparison of the K_T values of (X) [10] and (VIIIb) leads to the conclusion that the presence of the CN group in the 5-position in (VIIIb) slightly increases the acidity of the NH group, and the probability of formation of the lactim form is thereby increased.

EXPERIMENTAL

Mass spectra were obtained on a Varian MAT-112 with direct introduction of the sample into the source, ionizing electron energy 70 eV, ionization chamber temperature 180°C; IR spectra on a Perkin-Elmer 457 spectrometer in Vaseline oil; and UV spectra on Perkin-Elmer 575 and Specord M-40 ultraviolet spectrophotometers. The method of determination of K_T and the amounts of the tautomeric forms have been described [9]. The mean error in the determination of the compositions of the tautomeric mixtures was not greater than 4%. The cells used were of optically transparent quartz, thickness 1 cm.

TABLE 1. Amounts of Tautomeric Forms and Tautomeric Equilibrium Constants for 1-Substituted 4-Methyl-5-cyano-6-hydroxy-7-azaindolines (VIIIa, b) in Solvents of Differing Polarity

Compound	Amount of lactam form (%) (K_T) in the solvent*						
	1	2	3	4	5	6	7
VIIIa	61 (1,56)	58 (1,36)	46 (0,84)	28 (0,4)	— —	6 (0,06)	—
VIIIb	68 (2,13)	69 (2,2)	58 (1,37)	39 (0,64)	20 (0,26)	9 (0,09)	5 (0,05)

*Mixture of dioxane and alcohol. Percentage amounts of dioxane in alcohol: 1) 0; 2) 10; 3) 30; 4) 50; 5) 70; 6) 90; 7) 100.

1-Benzyl-4-methyl-5-cyano-6-chloro-7-azaindole (IIa). A solution of 1 g (3.53 mmole) of the azaindoline (Ia) in 75 ml of hot CCl_4 was cooled to 20°C, and 5 g of activated γ-manganese dioxide added at 20°C with stirring in one-gram portions over 15 h. The solid was filtered off, washed with 50 ml of CCl_4, the filtrate evaporated, and the residue crystallized from hexane–benzene (1:1) to give 0.7 g (71%) of yellowish crystals, mp 134-135°C, soluble in benzene, chloroform, and acetone, sparingly soluble in hexane and alcohols, insoluble in water. IR spectrum: 2215 (CN) cm^{-1}. Mass spectrum: 281* (98) $[M]^+$, 204 (21) $[M-C_6H_5]^+$, 191 (17) $[M-C_7H_6]^+$, 91 (100) $[CH_2Ph]^+$. UV spectrum, λ_{max}(log ε): 370 (3.10), 290 (3.62). Found, %: C 68.3, H 4.3, Cl 12.6, N 14.7. $C_{16}H_{12}ClN_3$. Calculated, %: C 68.3, H 4.3, Cl 12.5, N 15.0.

1-Butyl-4-methyl-5-cyano-6-chloro-7-azaindole (IIb) was obtained similarly, from 4 g of the azaindoline (Ib) and 20 g of γ-manganese dioxide. Yield 2.6 g (66%), yellowish crystals, mp 95-96°C (from hexane), readily soluble in benzene and chloroform, sparingly in hexane and alcohols, and insoluble in water. IR spectrum: 2210 cm^{-1} (CN). Mass spectrum: 247 $[M]^+$, 232 (7) $[M-CH_3]^+$, 204 (100) $[M-C_3H_7]^+$, 191 (71) $[M-C_4H_8]^+$. Found %: C 62.9. , H 5.5, Cl 14.1, N 17.2. $C_{13}H_{13}ClN_3$. Calculated, %: C 63.3, H 5.3, Cl 14.4, N 17.0.

1-Benzyl-3-bromo-4-methyl-5-cyano-6-chloro-7-azaindole (III). To a solution of 0.57 g (2 mmole) of the azaindole (IIa) in 10 ml of dioxane was added dropwise at 20°C 1 g (4 mmole) of dioxane dibromide in 14 ml of dioxane. The mixture was stirred for 2 h at 20°C, and evaporated under reduced pressure. The residue was crystallized from hexane–benzene (1:1) to give 0.5 g (69%) of colorless crystals, mp 186-187°C, readily soluble in benzene, acetone, and chloroform, sparingly in hexane, insoluble in water. Mass spectrum: 359 (42) $[M]^+$, 280 (15) $[M-Br]^+$, 91 (100) $[CH_2C_6H_5]^+$. Found, %: C 53.3, H 2.8, Br 22.4, Cl 9.7, N 11.5. $C_{16}H_{11}BrClN_3$. Calculated, %: C 53.3, H 3.1, Br 22.2, Cl 9.7, N 11.7.

1-Benzyl-3-acetyl-4-methyl-5-cyano-6-chloro-7-azaindole (IV). To a suspension of 2.4 g (18 mmole) of anhydrous aluminum chloride in 20 ml of dry dichloroethane was added at 0°C 1.7 ml (24 mmole) of acetyl chloride, and the mixture was stirred for 15 min. To the resulting complex was added with stirring over 30 min at 20°C 1.18 g (4.2 mmole) of the azaindole (IIa) in 20 ml of dry dichloroethane. The mixture was kept for 2 h at 20°C, boiled for 30 min, cooled to 20°C, and poured into a solution of 20.3 g (72 mmole) of potassium sodium tartrate and 3.2 g (80 mmole) of sodium hydroxide in 100 ml of water, extracted with chloroform (3 × 50 ml), the chloroform extract dried over $MgSO_4$, and evaporated. The residue was recrystallized from benzene to give 1.01 g (74%) of yellowish crystals, mp 186-187°C, soluble in acetone and chloroform, sparingly soluble in benzene and alcohols, and insoluble in water. Mass spectrum: 323 (32) $[M]^+$, 308 (24) $[M-CH_3]^+$, 280 (5) $[M-COCH_3]^+$ 91 (100) $[CH_2C_6H_5]^+$. Found, %: C 67.0, H 4.1, Cl 11.0, N 13.0. $C_{18}H_{14}ClN_3O$. Calculated, %: C 66.9, H 4.3, Cl 10.8, N 13.0.

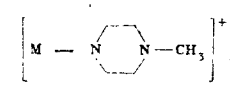
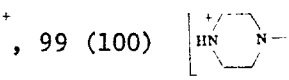
1-Benzyl-3-dimethylaminomethyl-4-methyl-5-cyano-6-chloro-7-azaindole (V). A. To a solution of 1.26 g (4.5 mmole) of the chloroazaindole (IIa) in 40 ml of butanol was added

*Here and subsequently, ions containing ^{35}Cl are given.

4.42 g (54.5 mmole) of dimethylamine hydrochloride and 0.6 g of paraformaldehyde. The mixture was stirred at the boil for 20 h, and evaporated under reduced pressure. The residue was cooled to 20°C and treated with 50 ml of 25% potassium carbonate solution, extracted with chloroform (3 × 50 ml), and the chloroform extracts dried over magnesium sulfate and evaporated under reduced pressure. The residue was chromatographed on a column (10 × 3 cm) containing 53 g of silica gel L 40/100. Elution with a mixture of benzene and methanol (99:1) gave 0.98 g (78%) of the starting chloroazaindole (IIa), and with 9:1 benzene–methanol, 0.2 g (14%) of the azaindole (V), as colorless crystals, mp 116-117°C (from hexane), soluble in benzene, chloroform, acetone, and alcohols, sparingly soluble in hexane, insoluble in water. Mass spectrum: 338 (4) M^+, 294 (16) $[M-(CH_2{=}N{-}CH_3)]^+$, 293 (12) $[M-N(CH_3)_2]^+$, , 91 (100) $[CH_2C_6H_5]^+$. Found, %: C 67.7, H 5.6, Cl 10.5, N 16.6. $C_{19}H_{19}ClN_4$. Calculated, %: C 67.5, H 5.6, Cl 10.4, N 16.6.

B. To a solution of 2.36 g (8.4 mmole) of the chloroazaindole (VIa) in 100 ml of butanol was added 8.32 g (127 mmole) of dimethylamine hydrochloride and 1.2 g (40 mmole) of paraformaldehyde. The mixture was boiled for 30 h, evaporated under reduced pressure, the residue treated with 150 ml of 1 N HCl, and extracted with benzene (3 × 50 ml). The benzene extract was dried over magnesium sulfate, evaporated, and the residue crystallized from hexane–benzene (1:1) to give 1.51 g (64%) of the starting chloroazaindole (IIa). The solution was basified with concentrated sodium hydroxide solution to pH 9, extracted with chloroform (3 × 50 ml), the chloroform layer dried over magnesium sulfate, evaporated, and the residue crystallized from hexane–benzene (5:1) to give 0.5 g (18%) of (V). The compound gave no depression of melting point on admixture with the material obtained by method A.

C. To a solution of 0.56 g (2 mmole) of the azaindole (IIa) in 20 ml of butanol was added at 115-118°C four portions of 0.54 g (5 mmole) of crystalline bis(dimethylamino)-methane at intervals of 3 h. Following addition of the last portion, the mixture was boiled for 3 h, evaporated under reduced pressure, cooled to 20°C, 50 ml of 1 N HCl added, stirred, and the mixture extracted with benzene (3 × 50 ml). The benzene layer was dried over magnesium sulfate and evaporated, giving 0.37 g (66%) of the starting azaindole (IIa). The acid solution following extraction with benzene was basified with concentrated sodium hydroxide solution to pH 9, and extracted with benzene (3 × 50 ml). The benzene extracts were dried over sodium sulfate and evaporated under reduced pressure to give 0.2 g (30%) of the azaindole (V). The compound gave no depression of melting point on admixture with a sample of material obtained by method A.

1-Benzyl-3-(4'-methylpiperazinyl-1'-methyl)-4-methyl-5-cyano-6-chloro-7-azaindole (VI). To a solution of 0.6 g (2.14 mmole) of the chloroazaindole (IIa) in 40 ml of butanol was added 3.3 g (19 mmole) of N-methylpiperazine hydrochloride and 0.36 g (12 mmole) of paraformaldehyde. The mixture was boiled with stirring for 20 h, evaporated under reduced pressure, and the residue treated with 50 ml of 25% potassium carbonate solution. The mixture was extracted with chloroform (3 × 50 ml), the chloroform extracts dried over magnesium sulfate, evaporated under reduced pressure, and the residue applied to a column (12 × 3 cm) of silica gel L 40/100 (29 g). Elution with benzene–methanol (99:1) gave 0.3 g (50%) of the starting azaindole (IIa), followed by benzene–methanol (9:1) to give 0.2 g (24%) of (VI) as colorless crystals, mp 166-167°C (from hexane–benzene, 1:1), soluble in benzene, acetone, chloroform, and alcohols, sparingly soluble in hexane and ether, insoluble in water. Mass spectrum: 393 (28) $[M]^+$, 349 (5) $[M-CH_2{-}NH(CH_3)]^+$, 334 (7) $[M-CH_2{-}NH(CH_3)-CH_3]^+$, 322 (21) $[M-CH_2{=}CH{-}N(CH_3)_2]^+$, 294 (17)  , 99 (100)  , 91 (72) $[CH_2C_6H_5]^+$, 56 (50) $[99{-}CH_2NCH_3]$. Found, %: C 67.0, H 5.9, Cl 8.9, N 17.6. $C_{22}H_{23}ClN_5$. Calculated, %: C 67.2, H 5.9, Cl 9.2, N 17.8.

1-Benzyl-3-(4'-methylpiperazinyl-1'-methyl)-4-methyl-5-cyano-6-chloro-7-azaindole Dihydrochloride. Mp 266-267°C (from DMF). The compound was insoluble in benzene, ether, and acetone, and sparingly soluble in alcohols, DMF, and water. Found, %: C 56.7, H 5.6, Cl 22.9, N 14.9. $C_{22}H_{24}ClN_5 \cdot HCl$. Calculated, %: C 56.7, H 5.4, Cl 22.9, N 15.0

1-Benzyl-4-methyl-5-cyano-6-methoxy-7-azaindoline (VIIa). To sodium methoxide, obtained from 30 ml of methanol and 1 g of sodium, was added slowly 1 g of the azaindoline (Ia) in 20 ml of dry DMF, and the mixture stirred for 6 h at 80-82°C. It was then evaporated under reduced pressure, and residual DMF removed by adding xylene (3 × 20 ml) followed by distilling

off under reduced pressure. After cooling to 20°C, the mixture was treated with 20 ml of water and extracted with chloroform (5 × 30 ml), the chloroform extract dried over magnesium sulfate and evaporated under reduced pressure. The residue was recrystallized from cyclohexane to give 0.72 g (73%) of bright yellow crystals, mp 125-126°C, readily soluble in benzene, chloroform, acetone, and alcohols, sparingly soluble in hexane, and insoluble in water. IR spectrum: 2200 cm^{-1} (CN). Mass spectrum: 279 (100) $[M]^+$, 264 (24) $[M - CH_3]^+$, 202 (35) $[M - C_6H_5]^+$, 188 (29) $[M - CH_2C_6H_5]^+$. Found, %: C 73.4, H 6.2, N 14.9. $C_{17}H_{17}N_3O$. Calculated, %: C 73.1, H 6.1, N 15.1.

1-Butyl-4-methyl-5-cyano-6-methoxy-7-azaindoline (VIIb) was obtained as for (VIIa), from 0.57 g (2.3 mmole) of the azaindoline (Ib) and 1.92 g (30 mmole) of sodium methoxide. Yield 0.4 g (71%), yellowish crystals, mp 67-68°C (from hexane), readily soluble in acetone, chloroform, and alcohols, sparingly soluble in hexane, and insoluble in water. IR spectrum: 2200 cm^{-1} (CN). Mass spectrum: 245 (50) $[M]^+$, 202 (100) $[M - C_3H_7]^+$, 189 (11) $[M - C_4H_8]^+$, 188 (7) $[M - C_4H_9]^+$. Found, %: C 68.4, H 7.9, N 17.3. $C_{14}H_{19}N_3O$. Calculated, %: C 68.6, H 7.8, N 17.1.

1-Benzyl-4,7-dimethyl-5-cyano-6-oxo-7-azaindoline (IX). To a freshly prepared "solution" of 0.16 g (7 mmole) of metallic sodium in 6 ml of methanol was added 1.86 g (7 mmole) of the azaindoline (VIIIa), the solution cooled to 20°C, and 1.4 g (8 mmole) of dimethyl sulfate added over 5 min with stirring. The mixture was kept at 65°C for 2 h, cooled, 20 ml of water added, and the solid filtered off and washed with 50 ml of chloroform to give 0.4 g (21.5%) of the starting azaindoline (VIII). The aqueous solution was extracted with chloroform (3 × 30 ml), the chloroform extracts and washings combined, dried over magnesium sulfate, and evaporated. The residue was dissolved at the boil in 500 ml of hexane–benzene (1:1), cooled, and the crystals which separated filtered off to give 0.2 g (11%) (IX) as yellowish crystals, mp 197-198°C, soluble in chloroform, acetone, and alcohols, sparingly soluble in hexane and benzene, insoluble in water. IR spectrum: 2200 (CN), 1610 cm^{-1} (CO). Mass spectrum: 279 (100) $[M]^+$, 264 (49) $[M - CH_3]^+$, 202 (17) $[M - C_6H_5]^+$, 188 (49) $[M - CH_2C_6H_5]^+$, 91 (79) $[CH_2C_6H_5]^+$. Found, %: C 73.2, H 6.2, N 15.1. $C_{17}H_{17}N_3O$. Calculated, %: C 73.1, H 6.1, N 15.1. The filtrate was evaporated and the residue crystallized from hexane–benzene (1:1) to give 1 g (54.6%) of the azaindoline (VIIa).

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