

was 150°C, the ionizing voltage was 70 V, and the emission current was 1.0 mA. The high-resolution mass spectra were recorded with an MS-902 spectrometer. The mass spectra are presented in Table 1.

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SYNTHESIS OF 5-SUBSTITUTED 6-METHOXY-1,2,3,4-TETRAHYDRO- β -CARBOLIN-1-ONES

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The nitration and bromination of 6-methoxy-1,2,3,4-tetrahydro- β -carbolin-1-one were studied. 5-Nitro and 5-bromo derivatives were obtained. 5-Acetyl-1,2,3,4-tetrahydrocarbolin-1-one oxime was obtained, and its Beckmann rearrangement was studied. The use of lithium aluminum hydride leads to reduction of the 5-acetyl group to give an alcohol group, whereas reduction of the acetyl group to an ethyl group occurs in the case of reduction with a palladium catalyst. Saponification of 5-substituted carbolin-1-ones with alcoholic alkali makes it possible to obtain 4-substituted tryptamines with a carbonyl group in the 2 position. The structures of the compounds were established by means of the PMR and mass spectra.

Studies of the electrophilic substitution reactions of 5-methoxyindole and its derivatives by a number of researchers [1-5] have shown that the direction of attack by the electrophilic reagent in this case is determined mainly by the methoxy group of the benzene ring in the para position relative to the indole nitrogen atom. Instead of the electrophilic substitution in the 3 position that is classical for other indoles, the new substituent enters the 6 position in 5-methoxyindole compounds.

In the opinion of Yudin, Kost, and co-workers [5], realization of the process in acidic media, in which indoles that are protonated at the pyrrole nitrogen atom undergo substitution, is decisive for this sort of reaction pathway. According to the data in [4], the introduction in the 2 position of 5-methoxyindoles of an additional alkoxycarbonyl group, which changes the electron density distribution and evidently the site of protonation of the molecule, has a substantial effect on the direction of electrophilic attack. Thus, for example, the bromination of 2-ethoxycarbonyl-5-methoxyindole in acidic media leads to the formation of a 4-bromo derivative rather than a 6-bromo derivative in high yield.

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6-Methoxy-1,2,3,4-tetrahydro- β -carbolin-1-one (I) is a 5-methoxyindole derivative with a carbonyl function included in an additional six-membered ring.

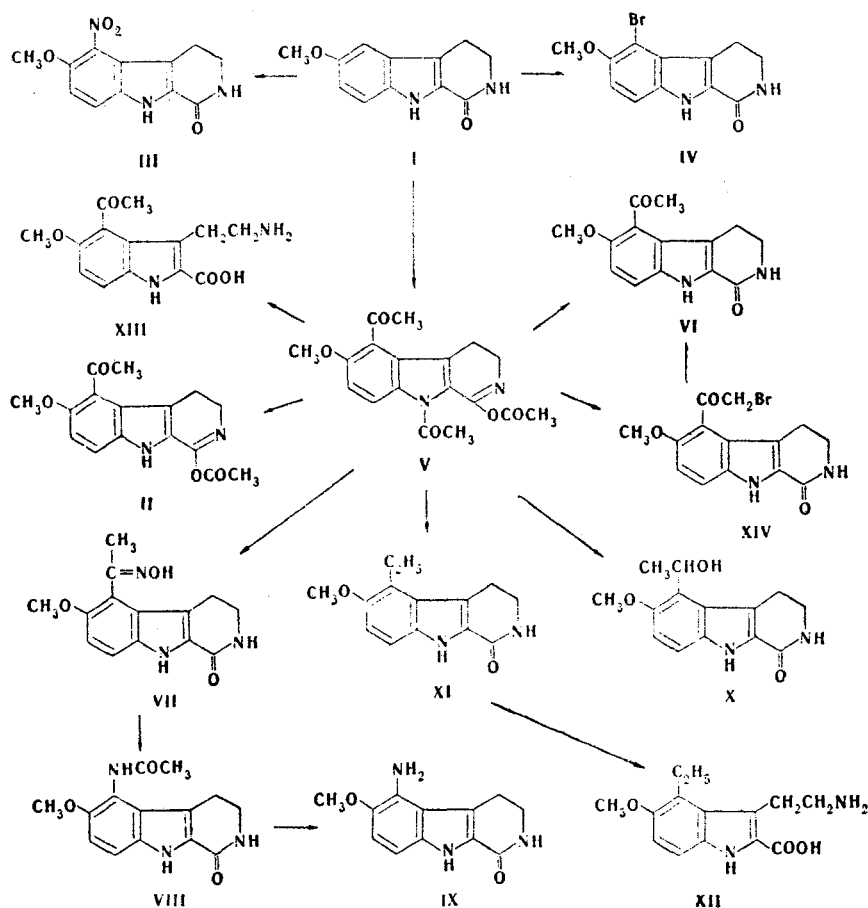
We have previously shown [6] that the dichlorophosphoryl derivative of this compound is readily acetylated by acetic anhydride in the 5 position, which corresponds to the 4 position of 2-ethoxycarbonyl-5-methoxyindole.

It seemed of interest to ascertain how general this pathway of electrophilic substitution reactions is for I and to use the resulting synthetic possibilities for the preparation of previously difficult-to-obtain 5,6-disubstituted β -carbolines and the corresponding 4,5-disubstituted tryptamines.

It should be noted that electrophilic substitution reactions in the 6-methoxy-1,2,3,4-tetrahydro- β -carbolin-1-one series have not been previously studied.

We investigated the nitration and bromination of I.

The nitration of carbolinone I or its diacetyl derivative (II) with fuming nitric acid or a mixture of fuming nitric acid and concentrated sulfuric acid at 0°C was accompanied by pronounced resinification and did not make it possible to isolate the individual nitration products. 5-Nitro-6-methoxy-1,2,3,4-tetrahydro- β -carbolin-1-one (III) was obtained in 66% yield when the reaction was carried out in glacial acetic acid with equimolar ratios of carbolinone I and the nitrating agent. The mass spectrum of nitro compound III contains a molecular-ion peak with m/e 261 and ion peaks with m/e 231 ($[M - NO]^+$), and 204 ($[M - CONHCH_2]^+$), as well as an intense peak with m/e 244 ($[M - OH]^+$), the formation of which is evidently due to the ortho orientation of the methoxy and nitro groups. The subsequent fragmentation of the $[M - OH]^+$ ion entails cleavage of the piperidine ring and elimination of a CO group.



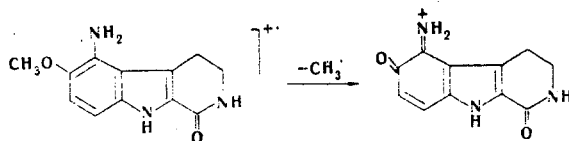
The PMR spectrum of III contains two triplets of the CH_2CH_2 group of the tetrahydro-pyridine ring at 3.91 and 3.15 ppm, a singlet of a 6-methoxy group at 4.09 ppm, two doublets of aromatic protons attached to C_7 and C_8 at 7.74 and 7.38 ppm with spin-spin coupling constant (SSCC) $J = 9$ Hz, and a broad singlet of the proton of the NH group at 10.46 ppm; the spectrum does not contain the signal of the proton attached to C_5 of the aromatic ring that

is characteristic for the PMR spectra of 5-unsubstituted carbolin-1-one. This set of data provides unambiguous evidence for incorporation of the substituent in the 5 position of the carboline system in the nitration of I, as was also observed in the case of acetylation of I.

The bromination of carbolinone I in glacial acetic acid with an equimolar amount of bromine led to 5-bromo-6-methoxy-1,2,3,4-tetrahydrocarbolin-1-one (IV) in 97% yield, the structure of which was also established by means of its PMR and mass spectra. The ratio of the isotope peaks of the molecular ions with m/e 294/296 in the mass spectrum of IV constitutes evidence for the presence in the molecule of one bromine atom, and the absence of fragments formed by elimination of a bromine atom from the molecular or fragment ions indicates that it is located in the aromatic portion of the molecule. The fragmentation of the bromo derivative is realized in the form of stepwise disintegration of the saturated ring to give fragments with m/e 279/281 ($[M - CH_3]^+$), 265/267 ($[M - NHCH_2]^+$) retrodiene decomposition, 251/253 ($[M - CO, -NH]^+$), and 237/239 ($[M - CONHCH_2]^+$).

The PMR spectrum of IV, which contains two triplets of a CH_2CH_2 group at 3.93 and 3.64 ppm, a singlet of a 6-methoxy group at 4.10 ppm, two doublets of aromatic protons at 7.51 and 7.33 ppm ($J \sim 9$ Hz), and a broad signal of the proton of the NH group at 9.94 ppm, is similar to the PMR spectrum of the 5-nitro-6-methoxycarboline derivative (III).

Triacetyl derivative V, which was described in a previous communication [6], on treatment with hydroxylamine readily splits out labile N- and O-acetyl groups and undergoes conversion, through diacetate II, to 5-acetyl-6-methoxy-1,2,3,4-tetrahydro- β -carbolin-1-one (VI), which readily reacts with excess hydroxylamine to give the corresponding oxime (VII). Beckmann rearrangement of oxime VII in a mixture of acetic and sulfuric acids led to 5-acetamido-6-methoxy-1,2,3,4-tetrahydro- β -carbolin-1-one (VIII), which was saponified to give the corresponding 5-amino-6-methoxycarboline derivative (IX). Two intense peaks with m/e 231 (M^+) and 216 ($[M - CH_3]^+$) are observed in the mass spectrum of IX. The high stability of the $[M - CH_3]^+$ ion is evidently explained by the ortho orientation of the methoxy and amino substituents and the advantageousness of detachment of a methyl group, as a result of which the charge is localized on the nitrogen atom of the primary aromatic amine.



Only detachment of the N- and O-acetyl groups to give VI occurs when triacetyl derivative V is treated with sodium borohydride.

The use of lithium aluminum hydride leads to reduction of the 5-acetyl group to give an alcohol group and conversion of V to carbinol X. More profound reduction occurs in the case of hydrogenation of triacetate V with a palladium catalyst: the acetyl group in the 5 position is reduced to an ethyl group.

The spectrum of carbinol X contains an intense molecular-ion peak with m/e 260. The formation of the $[M - CH_3]^+$ fragment, which gives an intense peak with m/e 245, is explained by the advantageousness of elimination of a methyl group from the α position with respect to the hydroxy group. The intense $[M - H_2O]^+$ ion peak provides evidence for the presence of a hydroxy group in the molecule. Subsequent stepwise fragmentation of the saturated ring in the $[M - H_2O]^+$ and $[M - CH_3]^+$ ions leads to the formation of fragments with m/e 227, 217, 203, 202, 184, 174, etc.

The most intense peaks in the mass spectrum of XI are due to M^+ (m/e 244) and $[M - CH_3]^+$ (m/e 229) ions. The formation of the $[M - CH_3]^+$ ion attests to the presence of an ethyl group in the aromatic ring; β cleavage of the exocyclic C-C bond leads to a rearranged ion with a tropylium structure. The subsequent fragmentation of the M^+ and $[M - CH_3]^+$ ions entails cleavage of the saturated ring.

The saponification of 5-ethyl-6-methoxy-1,2,3,4-tetrahydro- β -carbolin-1-one (XI) with alcoholic alkali makes it possible to obtain a 4,5-disubstituted tryptamine (XII) with a carbonyl group in the 2 position, which readily undergoes recyclization to carbolinone XI when it is heated without a solvent or in concentrated hydrochloric acid. A similar picture is also observed in the case of 5-acetylcarbolinones V and VI. In alkaline media these compounds are converted in virtually quantitative yield to 4-acetyl-5-methoxytryptamine-2-

carboxylic acid (XIII), which undergoes cyclization to carbolinone VI when it is heated without a solvent or with hydrochloric acid. Only removal of the N-acetyl group to give 1-acetoxy-5-acetyl-6-methoxy-3,4-dihydro- β -carboline (II) occurs when triacetate V is hydrogenated in methanol in the presence of a nickel catalyst at 40°C and a hydrogen pressure of 50 atm. Triacetyl derivative V is readily brominated in the side chain with an equimolar amount of bromine in chloroform to give 5-bromoacetyl-6-methoxy-1,2,3,4-tetrahydro- β -carbolin-1-one (XIV). The bromine in XIV displays the properties of a positive halogen. Replacement of the bromine by hydrogen to give 5-acetyl-6-methoxy-1,2,3,4-tetrahydro- β -carbolin-1-one (VI) occurs in all cases in the reaction of bromoacetyl derivative XIV with aniline, piperidine, potassium phthalimide, and sodium methoxide.

The PMR spectrum of XIV is characterized by the presence of two triplets of a CH_2CH_2 group at 3.95 and 3.18 ppm, a singlet of a 6-methoxy group at 4.10 ppm, a doublet of aromatic protons attached to C(7) and C(8) at 7.80 and 7.34 ppm ($J \sim 9$ Hz), a broad singlet of the proton of an NH group at 10.13 ppm, and a singlet of a CH_2 group of the 5-monobromoacetyl substituent at 4.74 ppm.

EXPERIMENTAL

The PMR spectra of solutions of the compounds in trifluoroacetic acid were recorded with a JNM-4H-100 spectrometer with tetramethylsilane as the internal standard. The mass spectra were recorded with an MKh-1303 spectrometer with direct introduction of the samples into the ion source at 50 eV.

5-Nitro-6-methoxy-1,2,3,4-tetrahydro- β -carbolin-1-one (III). A solution of 0.19 g (3 mmole) of fuming nitric acid (sp. gr. 1.5) in 4 ml of glacial acetic acid was added gradually with stirring to a solution of 0.5 g (2.3 mmole) of carbolinone I in 100 ml of glacial acetic acid, and the mixture was stirred for 3 h and allowed to stand overnight. It was then vacuum evaporated at room temperature to give 0.4 g (66,5%) of nitrocarboline III with mp 282-283°C (from methanol). The product was soluble in chloroform but only slightly soluble in ether, benzene, acetone, and water. Found: C 54.8; H 4.2; N 16.4%. $\text{C}_{12}\text{H}_{11}\text{N}_3\text{O}_4$. Calculated: C 55.1; H 4.2; N 16.2%.

5-Bromo-6-methoxy-1,2,3,4-tetrahydro- β -carbolin-1-one (IV). A solution of 0.23 g (2.3 mmole) of bromine in 50 ml of glacial acetic acid was added gradually with stirring to a solution of 0.5 g (2.3 mmole) of carbolinone I in 200 ml of glacial acetic acid, and the mixture was allowed to stand overnight. The resulting precipitate was removed by filtration to give 0.61 g (97%) of bromo derivative IV with mp 277-278°C (from methanol). The product was soluble in chloroform but only slightly soluble in benzene, ether, and water. Found: C 49.2; H 3.9; Br 27.2; N 9.4%. $\text{C}_{12}\text{H}_{11}\text{BrN}_2\text{O}_2$. Calculated: C 48.8; H 3.7; Br 27.2; N 9.5%.

1-Acetoxy-5-acetyl-6-methoxy-3,4-dihydro- β -carboline (II). A 0.07-g (1 mmole) sample of hydroxylamine hydrochloride and 0.08 g (1 mmole) of ammonium acetate were added to 0.34 g (1 mmole) of triacetate V in a mixture of 5 ml of alcohol and 1 ml of water, and the mixture was refluxed for 3 h. The resulting precipitate was removed by filtration to give 0.22 g (85%) of diacetate II with mp 214-215°C (from methanol). No melting-point depression was observed for a mixture of the product with a sample of II obtained by the method described in [6], and the two samples had identical mass spectra.

5-Acetyl-6-methoxy-1,2,3,4-tetrahydro- β -carbolin-1-one Oxime (VII). A 0.21-g (3 mmole) sample of hydroxylamine hydrochloride and 0.24 g (3 mmole) of ammonium acetate were added to a solution of 0.34 g (1 mmole) of triacetate V in a mixture of 7 ml of alcohol and 2 ml of water, and the mixture was refluxed for 30 h; the end of the reaction was monitored by thin-layer chromatography (TLC) on Silufol [methanol-chloroform (1:4)]. The precipitate that formed when the mixture was cooled was removed by filtration to give 0.16 g (62%) of oxime VII with mp 233-234°C (from ethanol). The product was only slightly soluble in ether, benzene, chloroform, and water. Found: C 61.8; H 5.5; N 15.4%. $\text{C}_{14}\text{H}_{15}\text{N}_3\text{O}_3$. Calculated: C 61.5; H 5.5; N 15.4%.

4-Acetamido-5-methoxy-1,2,3,4-tetrahydro- β -carbolin-1-one (VIII). A mixture of 2.5 g of oxime VII, 15 ml of glacial acetic acid, and 10 ml of concentrated sulfuric acid was refluxed for 1 h, after which it was cooled to 20°C and poured into 30 ml of water. The aqueous mixture was allowed to stand in a refrigerator overnight and was then filtered to give 1.1 g (44%) of acetamidocarbolinone VIII with mp 271-272°C (from methanol). The product was only slightly soluble in water and organic solvents. Found: C 61.5; H 5.5; N 15.4%. $\text{C}_{14}\text{H}_{15}\text{N}_3\text{O}_3$. Calculated: C 61.5; H 5.5; N 15.4%.

5-Amino-6-methoxy-1,2,3,4-tetrahydro- β -carbolin-1-one (IX). A solution of 0.2 g of acetamidocarbolinone VIII in 4 ml of 10% hydrochloric acid was refluxed for 3 h, after which it was cooled and allowed to stand overnight in a refrigerator. The resulting precipitate was removed by filtration to give 0.15 g (64%) of 5-aminocarbolinone IX with mp 290–291°C (dec., from methanol). The product was soluble in hot water but only slightly soluble in organic solvents. Found: C 44.8; H 5.5; Cl 22.5; N 12.7%. $C_{12}H_{13}N_3O_2 \cdot 2HCl \cdot 2H_2O$. Calculated: C 44.7; H 5.2; Cl 22.1; N 13.1%.

5-Acetyl-6-methoxy-1,2,3,4-tetrahydro- β -carbolin-1-one (VI). A 1-g (26 mmole) sample of sodium borohydride was added with stirring in the course of an hour at 45–50° to a solution of 0.7 g (2 mmole) of triacetyl derivative V in 10 ml of methanol, and the mixture was allowed to stand overnight. The methanol was then removed by vacuum distillation, and 5 ml of water was added to the residue. The aqueous mixture was extracted with four 10-ml portions of chloroform, and the extracts were combined and dried with potassium carbonate. The solvent was removed by vacuum evaporation to give 0.5 g (95%) of monoacetyl derivative VI with mp 207–208°C (from ethanol). Found: C 65.4; H 5.5; N 11.3%. $C_{14}H_{14}N_2O_3$. Calculated: C 65.1; H 5.4; N 10.8%.

5-(α -Hydroxyethyl)-6-methoxy-1,2,3,4-tetrahydro- β -carbolin-1-one (X). A solution of 1.5 g (5 mmole) of triacetate V in 70 ml of tetrahydrofuran (THF) was added gradually with stirring to a suspension of 1 g (26 mmole) of lithium aluminum hydride in 50 ml of refluxing THF, and the mixture was refluxed for 7 h. It was then treated with 2 ml of water, and the aqueous mixture was subjected to vacuum evaporation. Compound X was extracted with boiling absolute alcohol. The alcohol was evaporated, and the residue was recrystallized from absolute alcohol to give 0.2 g (18%) of carbinol X with mp 275–276°C. The product was soluble in chloroform but only slightly soluble in ether, benzene, and water. Found: C 64.5; H 6.4; N 10.7%. $C_{14}H_{17}N_2O_3$. Calculated: C 64.5; H 6.2; N 10.8%.

Any additional amounts of carbinol X that were deposited on the mixture of lithium and aluminum hydroxides were not extracted.

5-Ethyl-6-methoxy-1,2,3,4-tetrahydro- β -carbolin-1-one (XI). A solution of 0.5 g of palladium chloride in 5 ml of 18% hydrochloric acid solution was added to a solution of 1 g (3 mmole) of triacetate V in 100 ml of ethanol, and the mixture was hydrogenated at room temperature (18–20°C) and an excess pressure of 20–30 mm (water column) until hydrogen absorption ceased. The palladium was then removed by filtration, and the solution was vacuum evaporated to give 0.6 g (85%) of 5-ethyl-6-methoxy-1,2,3,4-tetrahydro- β -carbolin-1-one (XI) with mp 204–205°C (from ethanol). The product was soluble in organic solvents but only slightly soluble in water. Found: C 68.6; H 6.2; N 11.1%. $C_{14}H_{16}N_2O_2$. Calculated: C 68.7; H 6.5; N 11.5%.

1-Acetoxy-5-acetyl-6-methoxy-3,4-dihydro- β -carboline (II). A solution of 3 g (9 mmole) of triacetate V in 50 ml of methanol was heated in a rotary autoclave at 40°C with 1 g of a nickel catalyst at an excess hydrogen pressure of 50 atm. After 3 h, the catalyst was removed by filtration, and the solution was vacuum evaporated to give 2.59 g (quantitative yield) of diacetate II. No melting-point depression was observed for a mixture of this product with a sample of II obtained by the method in [6], and the two samples had identical mass spectra.

3-(β -Aminoethyl)-4-acetyl-5-methoxyindole-2-carboxylic Acid (XIII). Alcohol (35 ml) and a solution of 6 g of potassium hydroxide in 20 ml of water were added to 2 g (7 mmole) of triacetate V, and the mixture was refluxed for 5 h. The alcohol was then removed by vacuum distillation, and the residue was neutralized to pH 7 with glacial acetic acid and allowed to stand in a refrigerator at +5°C. The precipitated acid XIII was removed by filtration to give 1.5 g (93%) of a product with mp 241–242°C (dec.). Found: C 60.5; H 5.8; N 9.9%. $C_{14}H_{16}N_2O_4$. Calculated: C 60.8; H 5.8; N 10.2%. Carbolinone VI was obtained in quantitative yield when 0.5 g of acid XIII was refluxed for 1 h in 5 ml of 5% aqueous hydrochloric acid after cooling and alkalization with potassium hydroxide. Complete resinification was observed when 0.5 g of acid XIII was heated at 260–270°C for 15 min.

5-Bromoacetyl-6-methoxy-1,2,3,4-tetrahydro- β -carbolin-1-one (XIV). A solution of 0.5 g (3.5 mmole) of bromine in 10 ml of chloroform was added with stirring at room temperature to a solution of 1 g (3 mmole) of triacetate V in 40 ml of chloroform, and the mixture was stirred for another hour. It was then subjected to vacuum evaporation to give 0.9 g (90%) of bromoacetate XIV with mp 181–182°C (from methanol). The product was quite soluble in

chloroform but only slightly soluble in ether, benzene, and water. Found: C 50.0; H 3.9; Br 23.5; N 8.3%. $C_{14}H_{13}BrN_2O_3$. Calculated: C 50.0; H 3.8; Br 23.7; N 8.3%.

Reaction of 5-Bromoacetyl-6-methoxy-1,2,3,4-tetrahydro- β -carbolin-1-one (XIV) with Nucleophilic Agents. A) A 0.15-g (2 mmole) sample of piperidine was added to a solution of 0.3 g (1 mmole) of bromo derivative XIV in 5 ml of anhydrous benzene, and the mixture was maintained at room temperature in an argon atmosphere for 12 h. It was then treated with 10 ml of water, and the benzene layer was separated, dried with magnesium sulfate, and evaporated to give 0.23 g (quantitative yield) of carbolinone VI, which, according to its melting point, mass spectrum, and the results of a mixed-melting-point determination, was identical to a genuine sample of VI.

B) In a similar experiment 0.23 g of carbolinone VI was obtained from 0.3 g (1 mmole) of bromo derivative XIV and 0.23 g (2 mmole) of aniline in 5 ml of benzene.

C) In a similar experiment 0.23 g of carbolinone VI was obtained from 0.3 g (1 mmole) of bromo derivative XIV and sodium methoxide [from 0.05 g (2 mg-atom) of sodium and 5 ml of methanol].

D) A 0.33-g (2 mmole) sample of potassium phthalimide was added to a solution of 0.3 g (1 mmole) of bromo derivative XIV in 5 ml of dimethylformamide (DMF), and the mixture was heated at 100°C for 2 h. It was then vacuum evaporated, and the residue was extracted with boiling chloroform. Removal of the chloroform by distillation gave 0.23 g (quantitative yield) of carbolinone VI.

3-(β -Aminoethyl)-4-ethyl-5-methoxyindole-2-carboxylic Acid (XII). A solution of 1.5 g (30 mmole) of potassium hydroxide in 5 ml of water was added to a solution of 0.5 g (2 mmole) of 5-ethylcarbolinone XI in 9 ml of ethanol, and the mixture was refluxed for 5 h. It was then acidified to pH 4 with concentrated hydrochloric acid and allowed to stand in a refrigerator overnight. The resulting precipitate was removed by filtration to give 0.54 g (quantitative yield) of acid XII with mp 204–205°C. Found: C 54.8; H 6.6; Cl 11.8; N 9.0%. $C_{14}H_{18}N_2O_3 \cdot HCl \cdot \frac{1}{2}H_2O$. Calculated: C 54.6; H 6.9; Cl 11.6; N 8.2%. Carbolinone XI was obtained in quantitative yield (0.27 g) when 0.3 g (1 mmole) of acid XII was heated at 250–260°C for 20 min. The product was identical to an authentic sample of XI according to the results of a mixed-melting-point determination and the IR spectra.

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