3-PHENOXY-1,2,3,4-TETRAHYDROBENZO[h]-QUINOLINE*

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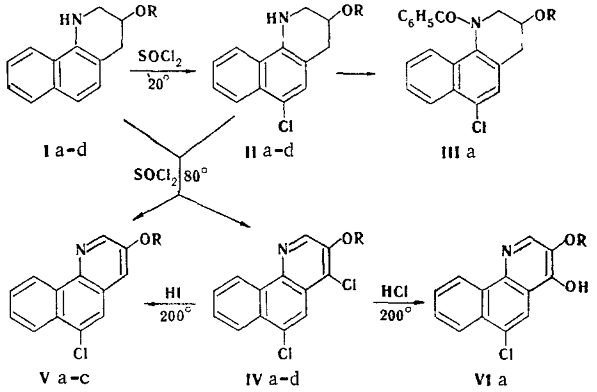
Thionyl chloride chlorinates 3-phenoxy-1,2,3,4-tetrahydrobenzo[h]quinolines in the 6 position at room temperature, but at 75-80°C it also partially chlorinates these compounds in the 4 position, which is accompanied by aromatization of the tetrahydropyridine ring. In the case of 3-(2,4,6-tribromophenoxy)-1,2,3,4-tetrahydrobenzo[h]quinoline, a tribromophenol residue is partially split out to give 4,6-dichlorobenzo[h]quinoline.

It has been shown [2, 3] that thionyl chloride chlorinates 3-hydroxy- or 3-chloro-1,2,3,4-tetrahydrobenzo[h]quinoline in the 6 position at room temperature, while, in addition to chlorination, a substituent is split out when the mixture is refluxed, and the tetrahydropyridine ring is aromatized. In the present study it is shown that chlorination in the 6 position occurs smoothly when 3-phenoxy-1,2,3,4-tetrahydrobenzo[h]quinoline (Ia) [4] is allowed to react with thionyl chloride at room temperature, and 3-phenoxy-6-chloro-1,2,3,4-tetrahydrobenzo[h]quinoline (IIa) is formed. However, when Ia is refluxed with SOCl$_2$, a mixture of 3-phenoxy-4,6-dichlorobenzo[h]quinoline (IVa) and 3-phenoxy-6-chlorobenzo[h]quinoline (Va) is formed, i.e., the tetrahydropyridine ring is aromatized, and the starting compounds undergo partial dichlorination.



Here and subsequently, a R=C$_6$H$_5$, b R=o-C$_6$H$_4$Cl, c R=p-C$_6$H$_4$Cl, d R= sym-C$_6$H$_2$Br$_3$.

It might have been assumed that the second chlorine atom enters the benzene ring of the phenoxy group. We therefore carried out the alternative synthesis of such compounds starting from 7a,8-dihydro-7H-azirino[1,2-a]benz[g]indole (VII), which reacts with o- or p-chlorophenol to give a mixture of 3-chlorophenoxy-1,2,3,4-tetrahydrobenzo[h]quinoline (Ib, c) and 2-chlorophenoxymethyl-2,3-dihydro-1H-benz[g]indole (VIIIb, c). The latter are isolated as the acetyl derivatives.

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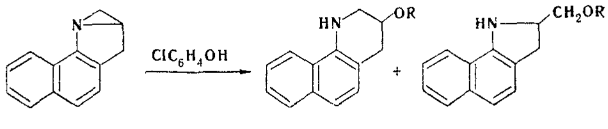
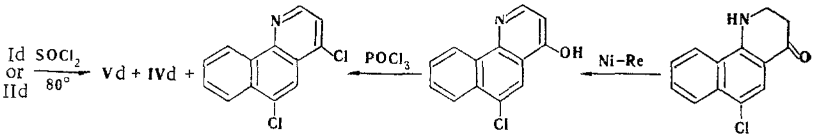


VII I b, c VIII b, c

Compounds Ib, c react with thionyl chloride at room temperature to give 3-chlorophenoxy-6-chloro-1,2,3,4-tetrahydrobenzo[h]quinolines (IIb, c), which are aromatized to 3-chlorophenoxy-6-chlorobenzo[h]quinolines (Vb, c) on refluxing. Neither of these products are identical to IVa, and, in addition, substances with three halogen atoms – 3-chlorophenoxy-4,6-dichlorobenzo[h]quinolines (IVb, c) – are formed along with thionyl chloride.

An attempt to hydrolytically cleave the ether bond of the phenoxy group in IVa by heating with hydrochloric or hydriodic acids was unsuccessful. Replacement of one chlorine atom by a hydroxyl group occurred on reaction with hydrochloric acid under pressure. It is known [5-7] that the halogen atoms in the 2 and 4 positions of quinoline compounds have increased lability. In this case also, the 4-Cl atom apparently also undergoes hydrolysis to give 3-phenoxy-4-hydroxy-6-chlorobenzo[h]quinoline (VIa). Compound IVa is reduced to Va by the action of hydriodic acid. These transformations indicate that the second chlorine atom is located in the pyridine ring.

The action of thionyl chloride on 3-(2,4,6-tribromophenoxy)-1,2,3,4-tetrahydrobenzo[h]quinoline [4] (Id) at room temperature gives 3-(2,4,6-tribromophenoxy)-6-chloro-1,2,3,4-tetrahydrobenzo[h]quinoline (IId). 4,6-Dichlorobenzo[h]quinoline (IX) was isolated along with IVd and Vd by heating it with SOCl$_2$, and traces of 6-chlorobenzo[h]quinoline were detected by chromatography, i.e., splitting out of a tribromophenol residue is observed in this case. Compound IX is identical to the compound synthesized by the method in [8], and this confirms not only its structure but also the assumption of the presence of halogen in the 4 position rather than in the 2 position in IVa-d.



IX XI X

On passing from the PMR spectrum of Ia to the spectrum of IIa, the multiplet signal of the 7-H proton at 7.86-8.20 ppm can be isolated from the unresolved multiplet of aromatic protons due to the effect of the magnetic anisotropy of the 6-Cl atom. On examination of the PMR spectra of IVa, Va, c and IX it is seen that the differences in the signals of the 7-H, 8-H, 9-H, and 10-H protons are insignificant. However, the singlet of the 5-H proton is found at 7.4 ppm in the case of monochloro derivative Va, while the singlet of the 5-H proton is found at 8.07-8.17 ppm in the case of dichloro derivatives IVa, c and IX. This shift in the signal can be explained by the effect of 4-Cl and is incompatible with the assumption that the second chlorine atom is in the 2 position. The doublet of the 2-H proton (8.65 ppm), with $J_{2,4}=2.7$ Hz for Va, is converted to a singlet in the case of IVa, c while in the case of IX it is expressed as a doublet with $J_{2,3}=4.8$ Hz, which is close to the observed $J_{2,3}$ (and not $J_{3,4}$) value for quinoline derivatives. As a result of hydrolysis of one of the chlorine atoms, a change in the position of the 2-H and 5-H singlets is observed in the PMR spectra of hydroxy derivative VIa and its benzoate, while the position of the 7-H signal remains practically unchanged. This serves as a confirmation of the fact that 4-Cl rather than 6-Cl is hydrolyzed. It is known [9] that 4-hydroxyquinoline derivatives exist in a tautomeric quinolone form, as a result of which a $\nu_{C=O}$ band is observed in the IR spectra at 1610-1638 cm^{-1}. A $\nu_{C=O}$ band also appears in the IR spectrum of VIa at 1624 cm^{-1}. The certain shift to strong field of the 10-H signal in the PMR spectra of VIa and XI, as well as the increase in $J_{2,3}$ in the case of XI, should apparently be explained by the contribution of the quinolone structure. However, the compounds apparently participate primarily in the hydroxy form in the benzoylation reaction, inasmuch as products other than the O-benzoyl derivatives could not be detected.

Thus, in addition to chlorination in the 6 position, chlorination in the 4 position and aromatization of the tetrahydropyridine ring, primarily with retention of the substituent in the 3 position, are also observed in the reaction of thionyl chloride with 3-aryloxy-1,2,3,4-tetrahydrobenzo[h]quinolines. It should be noted that chlorination in the 4 position and splitting out of a tribromophenol residue, as well as the previously investigated chlorination in the 6 position, proceed in the step involving the tetrahydro derivatives, i.e., prior to aromatization, inasmuch as aromatized compounds Va, d do not react with thionyl chloride. Chlorination in the 4 position and aromatization of 3-aryloxy-1,2,3,4-tetrahydrobenzo[h]quinolines also occur when a mixture of thionyl chloride and the starting materials is allowed to stand for a long time at room temperature.

EXPERIMENTAL

The PMR spectra were recorded with a Perkin–Elmer R-12 spectrometer at 60 MHz with tetramethylsilane as the internal standard. The IR spectra were recorded with an IKS-14 spectrometer.

3-Phenoxy-6-chloro-1,2,3,4-tetrahydrobenzo[h]quinoline (IIa). A mixture of 2.8 g (0.01 mole) of 3-phenoxy-1,2,3,4-tetrahydrobenzo[h]quinoline (Ia) and 10 ml of thionyl chloride was allowed to stand at room temperature for 15 min, after which it was decomposed with ice, and the resulting precipitate was removed by filtration and shaken with 10% sodium hydroxide solution in the presence of ether. The ether was removed, and the residual crystals were recrystallized from butyl acetate to give 2.4 g (78%) of a product with mp 166.5-167.5°. Found: Cl 11.3; N 4.5%. $C_{19}H_{16}ClNO$. Calculated: Cl 11.4; N 4.5%.

3-(2-Chlorophenoxy)-6-chloro-1,2,3,4-tetrahydrobenzo[h]quinoline (IIb). This compound was similarly obtained and had mp 120.5-122° (from alcohol). Found: Cl 20.6; N 4.1%. $C_{19}H_{15}Cl_2NO$. Calculated: Cl 20.6; N 4.1%.

3-(4-Chlorophenoxy)-6-chloro-1,2,3,4-tetrahydrobenzo[h]quinoline (IIc). This compound was similarly obtained and had mp 111.2-112.5° (from alcohol). Found: Cl 20.4; N 4.1%. $C_{19}H_{15}Cl_2NO$. Calculated: Cl 20.6; N 4.1%.

3-(2,4,6-Tribromophenoxy)-6-chloro-1,2,3,4-tetrahydrobenzo[h]quinoline (IId). This compound was similarly obtained in 80% yield and had mp 194.5-195.5° (from chloroform). Found: halogens 50.2; N 2.5%. $C_{19}H_{13}BrClNO$. Calculated: halogens 50.4; N 2.6%.

1-Benzoyl-3-phenoxy-6-chloro-1,2,3,4-tetrahydrobenzo[h]quinoline (IIIa). A mixture of 1.2 g (4 mmole) of IIa, 0.6 g (4 mmole) of benzoyl chloride, and 8 ml of pyridine was heated at 95° for 5 h. The mixture was worked up to give 1.1 g (67%) of IIIa with mp 171.5-172.5° (from alcohol). Found: Cl 8.6; N 3.5%. $C_{26}H_{20}ClNO_2$. Calculated: Cl 8.6; N 3.4.

3-Phenoxy-4,6-dichlorobenzo[h]quinoline (IVa) and 3-Phenoxy-6-chlorobenzo[h]quinoline (Va). A mixture of 5.5 g (0.02 mole) of Ia and 22 ml of thionyl chloride was refluxed for 2 h. The mixture was treated with ice, and the resulting precipitate was removed by filtration and dissolved in acetone–alcohol in the presence of sodium acetate. The solvent was partially removed, and the resulting crystals were removed by filtration, washed with water, and recrystallized from acetone to give 2.3 g (34%) of IVa with mp 156-157°. Found: C 66.7; H 3.3; Cl 21.0; N 4.1%. $C_{19}H_{11}Cl_2NO$. Calculated: C 67.1; H 3.3; Cl 20.8; N 4.1%.

The mother liquor remaining after the isolation of IVa was evaporated, and the oily residue was extracted with petroleum ether. The petroleum ether was partially removed, and the resulting crystals were removed by filtration and recrystallized from petroleum ether and alcohol to give 0.8 g (13%) of Va with mp 84-86°. In order to completely separate the IVa impurity, the isolated substance was heated under pressure with hydrochloric acid, and the mixture was made alkaline with sodium hydroxide and extracted with ether to give a product with mp 87.3-88.3° (from alcohol). Found: C 74.9; H 4.1; Cl 11.6; N 4.5%. $C_{19}H_{12}ClNO$. Calculated: C 74.7; H 4.0; Cl 11.6; N 4.6%.

Similar results were obtained under the same conditions when IIa was used.

Reaction of Ia with thionyl chloride at room temperature for 70 h gave Va with mp 86.2-87.2° (from alcohol) in 7% yield.

3-(2-Chlorophenoxy)-4,6-dichlorobenzo[h]quinoline (IVb) and 3-(2-Chlorophenoxy)-6-chlorobenzo[h]quinoline (Vb). These compounds were obtained by the method described above for IVa and Va. When IIb was used, the yield of IVb with mp 164.5-165.5° (from acetone) was 38%. Found: C 60.6; H 3.0; Cl 28.6; N 3.8%. $C_{19}H_{10}Cl_3NO$. Calculated: C 61.0; H 2.7; Cl 28.4; N 3.7%. The yield of Vb with mp 116-117° (from alcohol) was 15%. Found: C 66.8; H 3.4; Cl 20.9; N 4.2%. $C_{19}H_{11}Cl_2NO$. Calculated: C 67.1; H 3.3; Cl 20.8; N 4.1%.

3-(4-Chlorophenoxy)-4,6-dichlorobenzo[h]quinoline (IVc) and 3-(4-Chlorophenoxy)-6-chlorobenzo[h]quinoline (Vc). These compounds were obtained by the procedure used to obtain IVa and Va. When IIc was used, the yield of IVc with mp 155.5-156.5° (from acetone) was 43%. Found: Cl 28.3; N 3.9%. $C_{19}H_{10}Cl_3NO$. Calculated: Cl 28.4; N 3.7%. The yield of Vc with mp 89-90° (from alcohol) was 14%. Found: Cl 20.8; N 4.3%. $C_{19}H_{11}Cl_2NO$. Calculated: Cl 20.8; N 4.1%.

Reaction of IIc with thionyl chloride at room temperature for 240 h gave IVc in 8% yield and Vc in 9% yield.

3-Phenoxy-6-chlorobenzo[h]quinoline (Va). A mixture of 1 g (3 mmole) of IVa and 16 ml of 57% hydriodic acid was heated in a sealed ampul at 200° for 4 h. The oily layer was separated, washed with water, and extracted with petroleum ether. The petroleum ether was removed, and the residue was recrystallized from alcohol to give 0.3 g (33%) of a product with mp 87-88°. No melting-point depression was observed for a mixture of this product with a sample of the product obtained by the method described above.

3-Phenoxy-4-hydroxy-6-chlorobenzo[h]quinoline (VIa). A mixture of 0.5 g (1.5 mmole) of IVa and 10 ml of concentrated HCl was heated in a sealed ampul at 200° for 4 h. The solid material was removed by filtration, washed with water, and recrystallized from alcohol in the presence of sodium acetate to give 0.4 g (85%) of a product with mp 284-285°. Found: Cl 11.0; N 4.5%. $C_{19}H_{12}ClNO_2$. Calculated: Cl 11.0; N 4.4%.

3-Phenoxy-4-benzoxy-6-chlorobenzo[h]quinoline. A mixture of 0.3 g (1 mmole) of VIa, 0.14 g (1 mmole) of benzoyl chloride, and 10 ml of pyridine was refluxed for 4 h. The mixture was treated with 25% sulfuric acid and diluted with water, and the resulting crystals were removed by filtration and recrystallized from alcohol to give 0.3 g (75%) of a product with mp 165-166°. PMR spectrum (in DMSO), ppm: 6.92-7.35 (protons of the phenyl ring of the phenoxy group), 7.45-7.70 and 7.96-8.17 (protons of the phenyl ring of the benzoxy group), 7.70-7.90 (8-H and 9-H), 7.88 s* (5-H), 8.07-8.34 (7-H), 8.73 s (2-H), 9.0-9.29 (10-H). IR spectrum (of a KBr pellet): 1736, 1237, and 1022 cm^{-1} (OCO). Found: Cl 8.2; N 3.5%. $C_{26}H_{16}ClNO_3$. Calculated: Cl 8.3; N 3.3%.

3-(2,4,6-Tribromophenoxy)-6-chlorobenzo[h]quinoline (Vc), 3-(2,4,6-Tribromophenoxy)-4,6-dichlorobenzo[h]quinoline (IVd), and 4,6-Dichlorobenzo[h]quinoline (IX). A mixture of 5.5 g (0.01 mole) of Id or IId and 18 ml of thionyl chloride was refluxed for 2 h. It was then cooled, and the resulting crystals of the hydrochloride of Vd were removed by filtration and recrystallized from acetone–alcohol in the presence of sodium acetate to give 0.8 g (15%) of a product with mp 194-195° (from acetone). PMR spectrum (in DMSO), ppm: 7.60 d, $J_{4,2}=2.9$ Hz (4-H); 7.69-7.93 (8-H and 9-H); 7.99 s (5-H); 8.03 s (3'-H and 5'-H); 8.13-8.41 (7-H); 8.83 d, $J_{2,4}=2.9$ Hz (2-H); 9.06-9.35 (10-H). Found: C 42.3; H 1.9; halogens 50.8; N 2.5%. $C_{19}H_9Br_3ClNO$. Calculated: C 42.1; H 1.7; halogens 50.7; N 2.6%.

After separation of Vd, the filtrate was decomposed with ice, and the resulting precipitate was removed by filtration, washed with water, treated with sodium acetate, and extracted with alcohol. The alcohol was partially removed, and the residual solution was cooled. The resulting crystals were removed by filtration and recrystallized from alcohol–acetone to give 0.9 g (36%) of IX with mp 146-147° (from acetone). PMR spectrum (in CCl_4), ppm: 7.51 d, $J_{3,2}=5.0$ Hz (3-H); 7.64-7.91 (8-H and 9-H); 8.16 s (5-H); 8.16-8.44 (7-H); 8.75 d, $J_{2,3}=4.8$ Hz (2-H); 9.15-9.45 (10-H). Found: C 63.0; H 3.0; Cl 28.3; N 5.7%. $C_{13}H_7Cl_2N$. Calculated: C 62.9; H 2.8; Cl 28.6; N 5.6%.

6-Chlorobenzo[h]quinoline [R_f 0.61, ether–hexane (1:4)] was detected in the filtrate after separation of IX by thin-layer chromatography (TLC) on aluminum oxide.

The alcohol-insoluble residue was extracted with petroleum ether. The solvent was removed, and the residue was recrystallized from acetone to give 0.1 g of IVd with mp 187.5-189.5°. PMR spectrum (in DMSO), ppm: 7.70-7.91 (8-H and 9-H); 8.02 s (3'-H and 5'-H); 8.0-8.37 (7-H); 8.19 s and 8.27 s (2-H and 5-H); 8.93 and 9.20 (10-H). Found: N 2.6%. $C_{19}H_8Br_3Cl_2NO$. Calculated: N 2.4%.

Reaction of 2 g (4 mmole) of Id and 16 ml of thionyl chloride at room temperature for 360 h gave 0.2 g (20%) of IX and 0.06 g (2.6%) of IVd.

4,6-Dichlorobenzo[h]quinoline (IX). A mixture of 0.12 g (0.5 mmole) of 4-hydroxy-6-chlorobenzo[h]quinoline and 2.5 ml of phosphorus oxychloride was refluxed for 2 h. The mixture was then decomposed with water, made alkaline with sodium hydroxide, and extracted with ether. The ether was removed to give 0.1 g (81%) of a product with mp 146-146.5° (from acetone). No melting-point depression was observed for a mixture of this product with the sample obtained by the method described above.

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*Here and subsequently, s is singlet and d is doublet.

4-Hydroxy-6-chlorobenzo[h]quinoline (XI). A mixture of 2.3 g (0.01 mole) of 4-oxo-6-chloro-1,2,3,4-tetrahydrobenzo[h]quinoline, 9.4 g (0.1 mole) of phenol, and 0.4 g of Raney nickel was heated at 195° for 60 h. The mixture was then diluted with ether, and the resulting precipitate was removed by filtration and dissolved in aqueous sodium hydroxide. The alkaline solution was acidified with hydrochloric acid, andthe resulting crystals were removed by filtration and recrystallized from alcohol in the presence of sodium acetate to give 0.3 g (13%) of a product with mp 327-328°. PMR spectrum (in DMSO), ppm: 6.38 d, $J_{3,2}$ = 6.7 Hz (3-H); 7.71-7.86 (8-H and 9-H); 7.97 d, $J_{2,3}$ = 6.7 Hz (2-H); 8.15 s (5-H); 8.0-8.34 (7-H); 8.56-8.86 (10-H). Found: N 6.1%. $C_{13}H_8ClNO$. Calculated: N 6.1%.

4-Benzoxy-6-chlorobenzo]h]quinoline. A solution of 0.12 g (0.5 mmole) of XI, 10 ml of pyridine, and 0.1 g (0.7 mmole) of benzoyl chloride was refluxed for 4 h. The mixture was then treated with 25% sulfuric acid and diluted with water. The resulting crystals were removed by filtration to give 0.13 g (78%) of a product with mp 183.7-184.5° (from alcohol). Found: Cl 10.6; N 4.2. $C_{20}H_{12}ClNO_2$ Calculated: Cl 10.6; N 4.2 .

1-Acetyl-2-(2-chlorophenoxymethyl)-2,3-dihydro-1H-benz[g]indole and 3-(2-Chlorophenoxy)-1,2,3,4-tetrahydrobenzo[h]quinoline (Ib). A solution of 9.1 g (0.05 mole) of 7a,8-dihydro-7H-azirino[1,2-a]benz[g]-indole (VII) and 6.4 g of o-chlorophenol in 20 ml of chlorobenzene was refluxed for 2 h. The mixture was then treated with aqueous sodium hydroxide solution and extracted with ether. The solvent was removed, 10 ml of acetic anhydride was added, and the mixture was held at room temperature for 4 h. It was then shaken with aqueous sodium carbonate solution in the presence of ether. The ether was removed, and the resulting crystals were removed by filtration and washed with ether to give 2.8 g (16%) of a product with mp 163-164° (from acetone). Found: Cl 10.1; N 4.1%. $C_{21}H_{18}ClNO_2$. Calculated: Cl 10.1; N 4.0%.

After isolation of 1-acetyl-2-(2-chlorophenoxymethyl)-2,3-dihydro-1H-benz[g]indole, the oily residue was chromatographed on aluminum oxide with elution with ether–petroleum ether (1 : 1). The fraction with R_f 0.64 was collected to give 3.1 g (20%) of Ib with mp 106.5-108° (from alcohol). Found: Cl 11.4; N 4.6%. $C_{19}H_{16}ClNO$. Calculated: Cl 11.4; N 4.5%.

1-Acetyl-2-(4-chlorophenoxymethyl)-2,3-dihydro-1H-benz[g]indole and 3-(4-Chlorophenoxy)-1,2,3,4-tetrahydrobenzo[h]quinoline (Ic). These compounds were similarly synthesized by acylation of the mixture obtained in the reaction of VII with p-chlorophenol. The yield of 1-acetyl-2-(4-chlorophenoxymethyl)-2,3-dihydro-1H-benz[g]indole with mp 147.5-148.5° (from alcohol) was 3.0 g (17%). Found: Cl 10.3; N 4.0%. $C_{21}H_{18}ClNO_2$. Calculated: Cl 10.1; N 4.0%. The yield of Ic with mp 94-95.5° (from hexane) was 16%. Found: Cl 11.3; N 4.5%. $C_{19}H_{16}ClNO$. Calculated: Cl 11.4; N 4.5%.

2-(2-Chlorophenoxymethyl)-2,3-dihydro-1H-benz[g]indole (VIIIb). A solution of 1.8 g (0.005 mole) of 1-acetyl-2-(2-chlorophenoxymethyl)-2,3-dihydro-1H-benz[g]indole, 60 ml of ethanol, and 10 ml of concentrated HCl was refluxed for 2 h. It was then shaken with aqueous sodium carbonate solution in the presence of ether, after which the ether was removed, and the oily residue was treated with alcohol to give 1.2 g (78%) of a product with mp 53-54.5° (from alcohol). Found: Cl 11.4; N 4.6%. $C_{19}H_{16}ClNO$. Calculated: Cl 11.4; N 4.5%.

2-(4-Chlorophenoxymethyl)-2,3-dihydro-1H-benz[g]indole (VIIIc). This compound, with mp 78-79.5° (from hexane), was similarly obtained in 84% yield from 1-acetyl-2-(4-chlorophenoxymethyl)-2,3-dihydro-1H-benz[g]indole. Found: Cl 11.4; N 4.6%. $C_{19}H_{16}ClNO$. Calculated: Cl 11.4; N 4.5%.

1-Acetyl-3-(2-chlorophenoxy)-1,2,3,4-tetrahydrobenzo[h]quinoline. A 0.3-g (0.1 mmole) sample of Ib was heated in 6 ml of acetic anhydride at 80° for 2 h, after which the mixture was shaken with aqueous sodium carbonate solution in the presence of ether. The ether was removed, and the residue was recrystallized from alcohol to give 0.25 g (71%) of a product with mp 110-111°. Found: Cl 10.1; N 4.1%. $C_{21}H_{18}ClNO_2$. Calculated: Cl 10.1; N 4.0%.

1-Acetyl-3-(4-chlorophenoxy)-1,2,3,4-tetrahydrobenzo[h]quinoline. This compound was similarly obtained from Ic. The yield of product with mp 128.5-129.5° (from alcohol) was 0.3 g (86%). Found: Cl 10.1; N 4.0%. $C_{21}H_{18}ClNO_2$. Calculated: Cl 10.1; N 4.0%.

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