

Synthesis and Alkylation of some New Reissert Compounds

Frank D. Popp

Department of Chemistry, University of Missouri-Kansas City, Kansas City, MO 64110

Robert E. Buhts and Dennis K. Chesney

Department of Chemistry, Clarkson College of Technology, Potsdam, N. Y. 13676

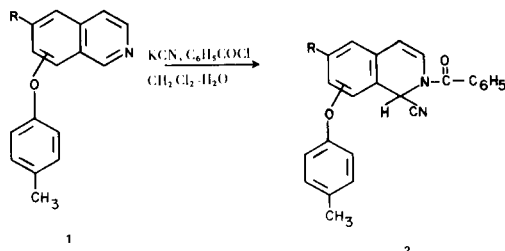
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The synthesis of several new isoquinoline Reissert compounds is described. Alkylation reactions of the anions of these new Reissert compounds and a rearrangement reaction are reported.

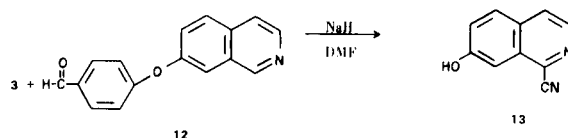
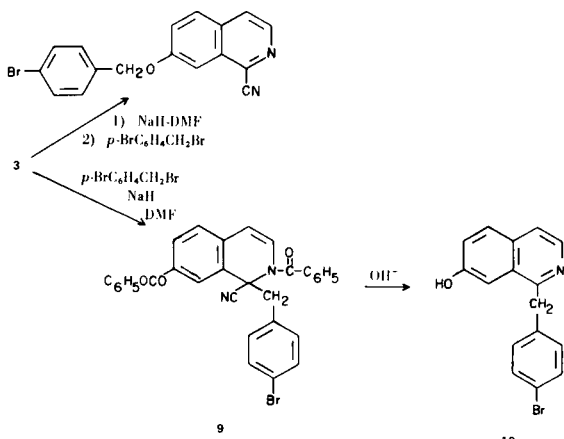
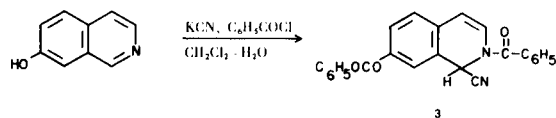
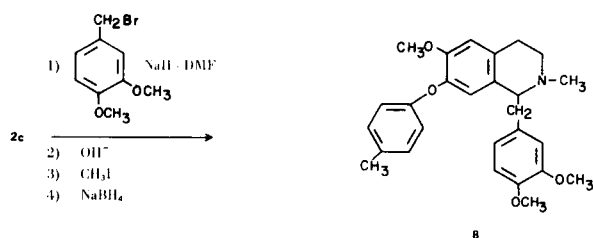
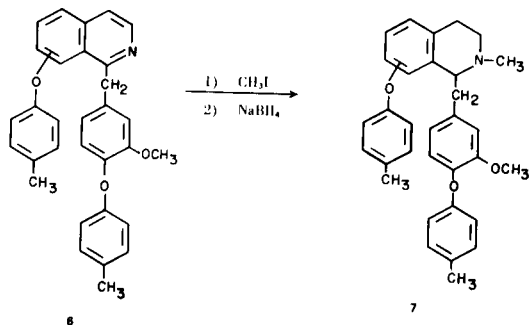
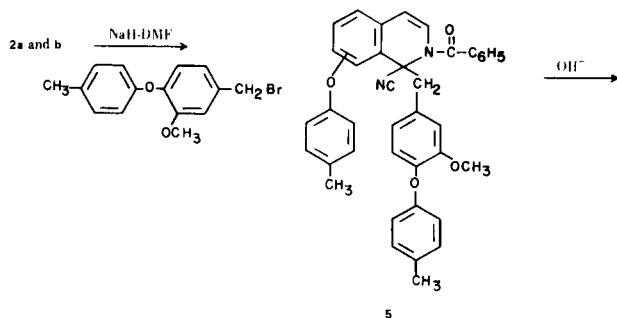
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In connection with other work in progress in these laboratories we had need of a number of isoquinoline derivatives as model compounds. The most convenient route to these compounds is through the appropriate Reissert compound (1) and this paper describes the synthesis of those compounds.

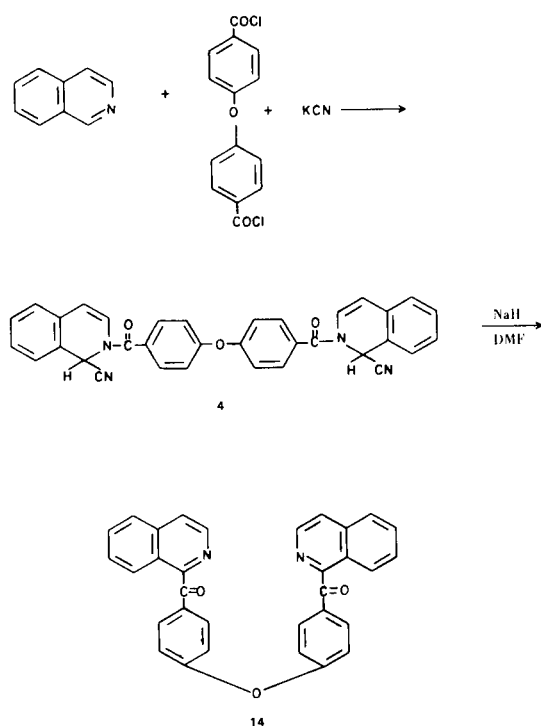
The *p*-tolyl isoquinolyl ethers (1) (2) were reacted with potassium cyanide and benzoyl chloride in methylene chloride-water to give the substituted 2-benzoyl-1,2-dihydro-



- a) ArO in 5-position, R = H
 b) ArO in 7-position, R = H
 c) ArO in 7-position, R = OCH₃



droisoquinaldonitriles (Reissert compounds) (2). In a similar manner 7-hydroxyisoquinoline gave the benzoate of 7-hydroxy-2-benzoyl-1,2-dihydroisoquinaldonitrile (3), and 7-methoxy-8-bromoisoquinoline and 5-bromoisoquinoline gave the expected Reissert compounds. Reaction of isoquinoline, potassium cyanide, and 4,4'-oxydibenzoyl chloride gave the *bis* Reissert compound (4).



The *p*-tolylxy-Reissert compounds (**2a**) and (**2b**) were alkylated with 4-(*p*-tolylxy)benzyl bromide in the presence of sodium hydride-dimethylformamide to give the alkylation products (**5**) which were hydrolyzed to 1-[3-methoxy-4-(*p*-tolylxy)benzyl]-5- and 7-(*p*-tolylxy)isoquinolines (**6**). These benzylisoquinolines (**6**) were reacted with methyl iodide and sodium borohydride to give 2-methyl-1-[3-methoxy-4-(*p*-tolylxy)benzyl]-5- and 7-(*p*-tolylxy)-1,2,3,4-tetrahydroisoquinolines (**7**). In a similar manner the Reissert compound from 4'-methyl-6-methoxy-7-isoquinolyphenyl ether (**2c**) was reacted with 3,4-dimethoxybenzyl bromide to give after hydrolysis, methylation, and reduction 1-(3,4-dimethoxybenzyl)-2-methyl-6-methoxy-7-tolylxy-1,2,3,4-tetrahydroisoquinoline (**8**). Alkylation of the 7-hydroxyisoquinoline Reissert compound (**3**) with 4-bromobenzyl bromide in the presence of sodium hydride-dimethylformamide gave the expected alkylation product (**9**) which was hydrolyzed to 1-(*p*-bromobenzyl)-7-hydroxyisoquinoline (**10**). Addition of the sodium hydride before the 4-bromobenzyl bromide in the alkylation reaction with 4-bromobenzyl bromide gave the isoquinaldonitrile derivative (**11**) instead of **9**. Conversion of Reissert compounds to isoquinaldonitriles have been previously reported (1) under these conditions. An attempt to react the anion of Reissert compound (**3**) with 7-(4-formylbenzoyloxy)isoquinoline (**12**) (**2**) gave a mixture from which 7-hydroxy-1-cyanoisoquinoline (**13**) was the only pure product isolated. This is again another example of base catalyzed conversion of a Reissert compound to an isoquinaldonitrile.

Rearrangement of the *bis* Reissert compound (**4**) with

sodium hydride-dimethylformamide gave the diketo diaryl ether (**14**). Although the yield was low it is possible that in certain cases this may provide an attractive alternative to alkylation (**3**) as a route to bisbenzylisoquinolines.

EXPERIMENTAL

Preparation of 2-Benzoyl-1,2-dihydro-7-(*p*-tolylxy)isoquinaldonitrile (**2b**).

To a stirred mixture of 3.76 g. of 4'-methyl-7-isoquinolyphenyl ether (**1b**) (0.016 mole) in 20 ml. of dichloromethane and 3.12 g. potassium cyanide (0.048 mole) in 8 ml. of water was added 4.5 g. of benzoyl chloride (0.032 mole). The mixture was stirred at room temperature for a total of 7 hours and the layers were separated and the aqueous phase extracted with dichloromethane. The dichloromethane layers were washed successively with 5% hydrochloric acid, water, 5% sodium hydroxide and water. Evaporation of the solvent gave a gum which was chromatographed over alumina with carbon tetrachloride (first fraction) and then chloroform (product fraction). Evaporation of the chloroform gave 2.5 g. of product, m.p. 162-165° (from carbon tetrachloride-hexane), ir: 1675, 1632, 1225 cm^{-1} .

Anal. Calcd. for $\text{C}_{24}\text{H}_{18}\text{N}_2\text{O}_2$: C, 78.67; H, 4.95; N, 7.65. Found: C, 78.33; H, 5.27; N, 7.39.

Preparation of 1-[3-methoxy-4-(*p*-tolylxybenzyl)-7-(*p*-tolylxy)isoquinoline (**6b**).

A mixture of 4.79 g. of the Reissert compound (**2b**) and 4.0 g. of 3-methoxy-4-(*p*-tolylxy)benzyl bromide in 50 ml. of dimethylformamide under a nitrogen atmosphere was cooled to 5° and 0.8 g. of sodium hydride-oil was added. The mixture was stirred for 1 hour at 5° and 2 hours at room temperature and was poured on crushed ice and filtered to give 5.5 g. of gummy material.

A mixture of 5.5 g. of **5b** in 100 ml. of 95% ethanol and 34.4 g. of potassium hydroxide in 100 ml. water was refluxed for 3 hours and then the ethanol was removed by distillation. After 100 ml. of water was added the solution extracted with chloroform. Evaporation of the chloroform gave a gum. Chromatography over alumina with chloroform again gave a gum after evaporation of the solvent. Attempts to crystallize this material failed. Compound **6b** was converted to the methiodide and recrystallized from ethyl acetate, m.p. 170-173°, ir: 1605 and 1230 cm^{-1} .

Anal. Calcd. for $\text{C}_{32}\text{H}_{30}\text{INO}_3$: C, 63.68; H, 5.01; N, 2.32. Found: C, 63.67; H, 5.07; N, 2.22.

Preparation of *N*-Methyl-1-[3-methoxy-4-(*p*-tolylxy)benzyl]-7-(*p*-tolylxy)-1,2,3,4-tetrahydroisoquinoline Methiodide (**7b**-Methiodide).

To 160 mg. of **6b** methiodide in 80 ml. methanol and 15 ml. water was added to 2.0 g. of sodium borohydride and the solution was refluxed for 2.5 hours. The methanol was evaporated and the resulting milky solution poured on ice. The mixture was extracted with chloroform and dried over magnesium sulfate. Evaporation of the solvent gave a gum. This material was converted to the methiodide of **7b** and recrystallized from ethyl acetate-methanol-diethyl ether, m.p. 193-196°.

Anal. Calcd. for $\text{C}_{33}\text{H}_{36}\text{INO}_3$: N, 2.25; I, 20.41. Found: N, 2.40; I, 20.80.

Preparation of 2-Benzoyl-1,2-dihydro-5-(*p*-tolylxy)isoquinaldonitrile (**2a**).

The procedure was the same as in **2b** except that chromatography was over Florisil with chloroform. The isoquinolyphenyl ether (**1a**) (1.88 g., 0.008 mole) gave 0.45 g. of the Reissert com-

pound after recrystallization from 95% ethanol, m.p. 192-195°, ir: 1675, 1625, 1250 cm^{-1} .

Anal. Calcd. for $\text{C}_{24}\text{H}_{18}\text{N}_2\text{O}_2$: C, 78.66; H, 4.95; N, 7.64. Found: C, 78.66; H, 4.97; N, 7.64.

Preparation of 2-Benzoyl-1,2-dihydro-1-[3-methoxy-4-(*p*-tolylxy)-benzyl]-5-(*p*-tolylxy)isoquinaldonitrile (**5a**).

A mixture of 3.0 g. of Reisert compound (**2a**) and 2.46 g. of 3-methoxy-4-(*p*-tolylxy)benzyl bromide was reacted as in the preparation of **5b**. Chromatography over alumina with chloroform gave a gum which crystallized with scratching. Recrystallization from 95% ethanol gave 5.0 g. of **5a**, m.p. 72-75°.

Anal. Calcd. for $\text{C}_{39}\text{H}_{32}\text{N}_2\text{O}_4$: C, 79.03; H, 5.44; N, 4.72. Found: C, 79.18; H, 5.53; N, 4.62.

Preparation of 1-[3-methoxy-4-(*p*-tolylxy)benzyl]-5-(*p*-tolylxy)-isoquinoline (**6a**).

Four g. of **5a** was subjected to the usual hydrolysis conditions. The resulting gum (**6a**) was chromatographed over alumina with chloroform but could not be crystallized. A pierate was formed and recrystallized from 95% ethanol, m.p. 85-88°.

Anal. Calcd. for $\text{C}_{37}\text{H}_{30}\text{N}_4\text{O}_{10}$: C, 64.34; H, 4.37; N, 8.11. Found: C, 64.45; H, 4.43; N, 8.02.

Preparation of *N*-Methyl-1-[3-methoxy-4-(*p*-tolylxy)-5-(*p*-tolylxy)-1,2,3,4-tetrahydroisoquinoline (**7a**).

The gummy methiodide (2.27 g.) of **6a** was reduced in the usual manner and the resulting gum chromatographed over alumina with chloroform. The reduced free base (**7a**) was converted to its methiodide and recrystallized from ethyl acetate-methyl iodide (trace)-diethyl ether, m.p. 118-122°.

Anal. Calcd. for $\text{C}_{33}\text{H}_{36}\text{INO}_3$: C, 63.76; H, 5.83; N, 2.25; I, 20.41. Found: C, 63.67; H, 5.85; N, 2.31; I, 20.47.

Preparation of Reisert Compound (**2c**) of 4'-Methyl-6-methoxy-7-isoquinolyphenyl Ether.

Benzoyl chloride (2.25 g., 0.016 mole) was added dropwise to a stirred mixture of 2.12 g. (0.008 mole) of 4'-methyl-6-methoxy-7-isoquinolyphenyl ether (**1c**), 1.56 g., (0.024 mole) of potassium cyanide, 20 ml. of dichloromethane and 8 ml. of water. The solution was stirred for 9 hours and worked up in the usual manner. A gum was isolated which was crystallized from hexane-carbon tetrachloride and then hexane, m.p. 94-97°, ir: 1640 cm^{-1} .

Anal. Calcd. for $\text{C}_{25}\text{H}_{20}\text{N}_2\text{O}_3$: C, 75.74; H, 5.08. Found: C, 76.30; N, 5.25.

Reaction of the Reisert Compound of 4'-Methyl-6-methoxy-7-isoquinolyphenyl Ether (**2c**) and 3,4-Dimethoxybenzylbromide.

A mixture of 4.0 g. of the above Reisert compound (0.01 mole) and 2.31 g. of 3,4-dimethoxybenzylbromide was dissolved in 50 ml. of dimethylformamide and the solution cooled to zero degrees. Sodium hydride (0.24 g.) was added and the solution stirred at zero degrees (under nitrogen) for one hour and at room temperature for two hours. The solution was poured on ice and allowed to stir overnight. The resulting suspension was filtered to give 4 g. of a white solid. Recrystallization from ethyl acetate-hexane gave the alkylation product, m.p. 105-108°, ir: 3000, 2900, 1635, 1500, 1450 and 1400 cm^{-1} .

Anal. Calcd. for $\text{C}_{34}\text{H}_{31}\text{N}_2\text{O}_5$: C, 74.57; H, 5.71; N, 5.12. Found: C, 74.28; H, 6.00; N, 4.70.

Hydrolysis of the Above Reisert Adduct.

A mixture of 3.0 g. of the above Reisert adduct in 100 ml. of 95% ethanol and 15 g. of potassium hydroxide in 100 ml. of water was refluxed with stirring for 3 hours, cooled and extracted three times with chloroform. The chloroform extract was washed

with water, dried over magnesium sulfate and evaporated to give a dark gum. The gum was refluxed with methyl iodide for two hours, cooled and poured into diethyl ether and stirred overnight. The resulting powder was filtered and washed with ether several times, m.p. 139-143°.

Anal. Calcd. for $\text{C}_{27}\text{H}_{28}\text{INO}_4$: I, 22.77; N, 2.51. Found: I, 22.70; N, 2.58.

Reduction of the Above Methiodide to give **8**.

To 1.0 g. of the above methiodide in 80 ml. of methanol and 10 ml. of water was added 5.0 g. of sodium borohydride in small portions with stirring. The mixture was then refluxed for 3 hours and poured on ice. The solution was extracted with chloroform, and the chloroform extracts washed with water, dried (magnesium sulfate), and evaporated to give a gum. This was chromatographed over alumina with chloroform. The resulting gum was dissolved in diethyl ether and hydrogen chloride bubbled through to give a yellow powder which was recrystallized from ethyl acetate/diethyl ether, m.p. 119-122°.

Anal. Calcd. for $\text{C}_{27}\text{H}_{32}\text{ClNO}_4$: C, 68.99; H, 6.86; N, 2.98; Cl, 7.54. Found: C, 69.23; H, 6.53; N, 3.15; Cl, 7.49.

Preparation of 7-Hydroxyisoquinoline Reisert Compound (**3**).

A mixture of 1.45 g. (0.01 mole) 7-hydroxyisoquinoline in 25 ml. dichloromethane and 1.95 g. (0.03 mole) potassium cyanide in 10 ml. water was stirred while 4.2 g. (0.03 mole) benzoyl chloride was added over a 20 minute period. The mixture was stirred at room temperature for 4 hours and the layers were separated. The aqueous layer was washed with 10 ml. of dichloromethane and the combined dichloromethane was extracted with 2 x (25 ml. of 10% hydrochloric acid, 25 ml. of 10% sodium hydroxide) and 25 ml. of water. The dichloromethane solution was dried over magnesium sulfate and evaporated *in vacuo* to yield 1.92 g. (50%) of **3**, m.p. 210-211° from ethanol; ir: 1750, 1660 cm^{-1} .

Anal. Calcd. for $\text{C}_{24}\text{H}_{16}\text{N}_2\text{O}_3$: C, 75.78; H, 4.24; N, 7.36. Found: C, 75.42; N, 7.60.

Alkylation of **3** with 4-Bromobenzyl Bromide.

A suspension of 4.19 g. (0.011 mole) of Reisert compound (**3**) and 2.8 g. (0.012 mole) of 4-bromobenzyl bromide was stirred in 50 ml. of dry dimethylformamide while 0.52 g. (0.012 mole) 52% sodium hydride was added. The mixture was stirred for 4 hours at room temperature and was poured onto 400 ml. of crushed ice and filtered to yield a wet white powder. The material was dried, washed, with 20 ml. of ethanol twice and filtered to yield 4.08 g. (68%), m.p. 180.5-181.5° from ethanol; ir: 1740, 1670 and 1210 cm^{-1} .

Anal. Calcd. for $\text{C}_{31}\text{H}_{21}\text{BrN}_2\text{O}_3$: C, 67.77; H, 3.85; N, 5.10. Found: C, 67.53; H, 3.87; N, 5.28.

Hydrolysis of **9** to 1-(4-Bromobenzyl)-7-hydroxyisoquinoline (**10**).

A mixture of 1.00 g. (0.00182 mole) of Reisert adduct (**9**) and 0.50 g. (0.089 mole) of potassium hydroxide in 10 ml. of 50% aqueous ethanol was refluxed for 45 minutes. The reaction mixture was diluted with 20 ml. of water and washed with 2 x 10 ml. of dichloromethane. The aqueous layer was made slightly acidic with 10% hydrochloric acid and buffered with sodium bicarbonate. The solution was extracted with 2 x 20 ml. of dichloromethane which was dried over magnesium sulfate and evaporated *in vacuo* to yield 0.45 g. (79%), m.p. 223-225° from ethanol; ir: 3410, 1615, 1240 cm^{-1} .

Anal. Calcd. for $\text{C}_{16}\text{H}_{12}\text{BrNO}$: C, 61.16; H, 3.85; N, 4.46. Found: C, 61.14; H, 3.87; N, 4.57.

Addition of 4-Bromobenzyl Bromide **3** After Addition of Sodium Hydride.

A mixture of 12.83 g. (0.033 mole) of Reissert compound (**3**) and 1.56 g. (0.036 mole) of 52% sodium hydride in 50 ml. of dry dimethylformamide was stirred at room temperature for 5-10 minutes, at which time 8.4 g. (0.036 mole) 4-bromobenzyl bromide was added. The mixture was stirred for 4 hours and poured onto 1000 ml. of crushed ice to yield a white precipitate which was filtered and used in crude form for the hydrolysis reaction.

The crude material was dissolved in 120 ml. of 50% aqueous ethanol containing 7.1 g. of potassium hydroxide and was refluxed for 2 hours. This was allowed to stand at room temperature for 16 hours, diluted with 60 ml. of water and evaporated *in vacuo* to remove the ethanol. This mixture was filtered to yield 6.11 g. (54%), of **11**, m.p. 177-178° from ethanol; ir: 2215, 1650, 1225 cm^{-1} ; nmr (deuteriochloroform): δ 8.56 (1, d, J = 3), 7.32-8.04 (10, m), 5.26 (2, s); ms: 341 (6.2), 3.40 (29.5), 339 (6.0), 338 (29.8), 172 (22.2), 171 (100.0), 170 (22.9), 169 (100.0), 141 (4.6), 91 (5.7), 90 (63.5), 89 (32.6), 64 (5.7), 63 (13.4), 51 (4.0), 39 (9.6), 32 (42.4), 28 (99.0).

Anal. Calcd. for $\text{C}_{17}\text{H}_{11}\text{BrN}_2\text{O}$: C, 60.20; H, 3.27; N, 8.26. Found: C, 60.00; H, 3.26; N, 7.98.

Attempt to React **3** with **12**.

A mixture of 1.39 g. (0.0056 mole) of ether (**12**) and 2.12 g. (0.0056 mole) of Reissert compound (**3**) was stirred in 35 ml. dimethylformamide while 0.28 g. (0.0060 mole) of 52% sodium hydride was added. The mixture was stirred at room temperature for 4 hours and poured onto 300 ml. of crushed ice. This was filtered to yield a wet yellow solid that became gummy on standing. This was dissolved in 5 ml. of chloroform and eluted from a column of alumina. The first 50 ml. of eluent was discarded. The next 100 ml. of eluent gave 1.91 g. of brown oil. The next 100 ml. of eluent showed only one spot at $R_f = 0.1$ and evaporation *in vacuo* gave 0.88 g. of **13**, m.p. 225.5-257° from aqueous ethanol; ir: 3400, 2225, 1660, 1560 cm^{-1} ; ms: 171 (16.5), 170 (100.0), 143 (9.7), 142 (12.9), 141 (4.8), 115 (23.0), 114 (10.5), 89 (6.6), 88 (7.7), 63 (10.0), 62 (6.4).

Anal. Calcd. for $\text{C}_{10}\text{H}_6\text{N}_2\text{O}$: C, 70.58; H, 3.55. Found: C, 70.78; H, 3.64.

The 1.91 g. of brown oil was dissolved in 50 ml. of chloroform and extracted with 3 x 25 ml. of 10% hydrochloric acid. The hydrochloric acid extracts were made basic and extracted with chloroform to yield 0.08 g. of pasty solid whose ir was identical to the above product. The chloroform layer was evaporated *in vacuo* to yield 0.96 g. of brown oil, that was purified on a column of alumina to give 0.31 g. of pasty solid whose ir was identical to the starting Reissert compound.

Preparation of Reissert Compound **4**.

To a mixture of 11.28 g. (0.048 mole) of isoquinoline and 0.38 g. (0.144 mole) of potassium cyanide in 16 ml. of water and 16 ml. of dichloromethane was added 9.44 g. (0.032 mole) of 4,4'-oxydibenzoyl chloride in 25 ml. of dichloromethane dropwise over 2 hours. The mixture was stirred for 4 hours after the addition and worked up in the usual manner. The product was chromatographed over alumina with chloroform to give 1.3 g. (8%) of **4**, m.p. 140-163° from ethyl acetate-hexane. Preparation of **4** in chloroform with silver cyanide resulted in yields of 6% after stirring at room temperature for 20 hours and 22% after stirring for 37 hours.

Anal. Calcd. for $\text{C}_{34}\text{H}_{22}\text{N}_4\text{O}_3$: C, 76.39; H, 4.14; N, 10.48. Found: C, 76.34; H, 4.29; N, 10.47.

Rearrangement of **4** to **14**.

To 2.68 g. of **4** (0.005 mole) in 25 ml. of dry dimethylformamide saturated with dry nitrogen was added 0.48 g. of 50% sodium hydride (0.01 mole). The solution turned black immedi-

ately and remained dark for the 2.5 hours it was stirred. The reaction mixture was poured onto crushed ice and allowed to separate into a dark oil layer (bottom) and yellow water layer. (This process required about seven days). The water layer was then decanted, the oil dissolved in chloroform washed with water several times and dried over magnesium sulfate for 24 hours. Evaporation of the solvent gave an oil (**14**) showing carbonyl absorption (1700 cm^{-1}). A picrate was formed, m.p. 100-120°.

Anal. Calcd. for $\text{C}_{38}\text{H}_{23}\text{N}_5\text{O}_{10}$: C, 64.31; H, 3.27; N, 9.87. Found: C, 64.02; H, 3.01; N, 10.17.

5-Bromoisoquinoline Reissert Compound.

A mixture of 1.50 g. (0.00721 mole) of 5-bromoisoquinoline in 20 ml. of dichloromethane and 1.41 g. (0.0216 mole) of potassium cyanide in 5 ml. of water was stirred at room temperature while 2.02 g. (0.0144 mole) of benzoyl chloride was added over a 7 minute period. The mixture was stirred for 4 hours and the layers were separated. The dichloromethane layer was extracted with 20 ml. each of 2 x (10% hydrochloric acid, water, 10% sodium hydroxide, water). The dichloromethane layer was dried over magnesium sulfate and evaporated *in vacuo* to a light brown oil that yielded 0.35 g. of white crystals on stirring with ethanol, m.p. 183-184°, ir: 1670, 1625 cm^{-1} .

Anal. Calcd. for $\text{C}_{17}\text{H}_{11}\text{BrN}_2\text{O}$: C, 60.20; H, 3.27. Found: C, 60.31; H, 3.26.

The acid extract was made basic to yield 0.36 g. of starting material.

7-Methoxy-8-bromoisoquinoline.

A solution of 0.88 g. (0.00505 mole) of 7-methoxy-8-aminoisoquinoline (**4**) in 3 ml. of concentrated hydrobromic acid and 15 ml. of water was cooled in an ice salt bath. After the orange-red mixture was at -1°, a solution of 0.37 g. (0.00536 mole) of sodium nitrite in 8 ml. of water was added dropwise with stirring at a rate such that the temperature did not exceed 0°. This was added dropwise to a heated solution of 4.25 g. (0.0296 mole) of cuprous bromide in 29 ml. of hydrobromic acid. The brown black solution was allowed to stand at room temperature for 14 hours and then made basic with sodium hydroxide. This was steam distilled to yield 0.34 g., m.p. 108-109° from methanol.

Anal. Calcd. for $\text{C}_{10}\text{H}_8\text{BrON}$: C, 50.45; H, 3.39. Found: C, 50.29; H, 3.37.

7-Methoxy-8-bromoisoquinoline Reissert Compound.

A solution of 2.08 g. (0.00874 mole) 7-methoxy-8-bromoisoquinoline in 15 ml. of dichloromethane and 1.71 g. (0.0262 mole) of benzoyl chloride was added dropwise over a period of 45 minutes. The layers were separated and the aqueous layer was washed with 15 ml. of dichloromethane. The combined dichloromethane layer was extracted twice with the following order of reagents: 20 ml. each of 10% hydrochloric acid, water, 10% sodium hydroxide, and water. The dichloromethane layer was dried over magnesium sulfate and evaporated *in vacuo* to yield a pasty tan material. To this was added 5 ml. of ethanol and filtered to yield 1.82 g. (73%), m.p. 161-162.5° from ethanol; ir: 1650, 1623 cm^{-1} .

Anal. Calcd. for $\text{C}_{18}\text{H}_{13}\text{BrN}_2\text{O}_2$: C, 58.55; H, 3.55; N, 7.59. Found: C, 58.38; H, 3.51; N, 7.57.

The combined acid extractions were made basic and the precipitate was collected and dried to yield 0.48 g. whose ir was identical to starting material.

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