



JOURNAL OF THE AMERICAN CHEMICAL SOCIETY

J. Am. Chem. Soc., 1997, 119(43), 10539-10540, DOI: [10.1021/ja971901j](https://doi.org/10.1021/ja971901j)

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Experimental Procedure

General Considerations. All reactions were performed under an inert atmosphere of nitrogen or argon in oven- or flame-dried glassware. Toluene was distilled from molten sodium under argon. Unless otherwise noted, all reagents were purchased from commercial sources and were used without further purification.

Preparative flash chromatography was performed using ICN Flash Silica Gel, 230-400 mesh. Yields refer to the average of two isolated yields of 95% or higher purity as determined by GC, ^1H NMR, and elemental analysis for new compounds. All products were characterized by ^1H NMR, ^{13}C NMR, and infrared (IR) spectroscopy. New compounds were further characterized by C, H elemental analysis from E & R Microanalytical Laboratories, 96-34 Corona Ave, Corona, NY 11368. NMR spectra were recorded on a Varian XL-300 or a Varian Unity 300 MHz spectrometer. Yields indicated in this section refer to a single experiment, while those reported in the tables are an average of two or more runs, so the numbers may differ slightly. All ^1H NMR spectra are reported in δ units ppm downfield from tetramethylsilane internal standard. All ^{13}C spectra are reported in δ units relative to the central line of the triplet for CDCl_3 at 77 ppm. IR spectra were recorded on a Perkin-Elmer 1600 Series Fourier Transform spectrometer. Gas chromatography analyses were performed on a Hewlett-Packard 5890 Gas Chromatograph, with a FID, a 25 meter capillary column with a dimethylpolysiloxane stationary phase, and a 3392A integrator. Melting points were determined using a Haake Buchler Melting Point Apparatus and are uncorrected.

Cu-Catalyzed Coupling of Phenols with Aryl Halides.

General Procedure A. The aryl halide (2.5 mmol), phenol (3.5 or 5.0 mmol, see Table 1), Cs_2CO_3 (3.5 or 5.0 mmol, see Table 1), $(\text{CuOTf})_2\cdot\text{PhH}$ (0.0625

mmol, 5.0 mol % Cu), ethyl acetate (0.125 mmol, 5.0 mol %), and toluene (2.0 mL) were added to an oven-dried test tube which was then sealed with a septum, purged with argon, and heated to 110 °C under argon until the aryl halide was consumed as determined by GC analysis. The reaction mixture was then allowed to cool to room temperature, diluted with Et₂O and washed sequentially with 5% aqueous NaOH, water and brine. The organic layer was dried over Mg₂SO₄ and concentrated under vacuum to give the crude product. Purification by flash column chromatography on silica gel afforded the analytically pure product.

General Procedure B. The aryl halide (2.5 mmol), phenol (3.5 or 5.0 mmol, see Table 1), Cs₂CO₃ (3.5 or 5.0 mmol, see Table 1), (CuOTf)₂•PhH (0.0625 mmol, 5.0 mol % Cu), ethyl acetate (0.125 mmol, 5.0 mol %), 1-naphthoic acid (3.5 mmol), molecular sieves 5 Å (625 mg) and toluene (1.5 mL) were added to an oven-dried test tube which was then sealed with a septum, purged with argon, and heated to 110 °C under argon until the aryl halide was consumed as determined by GC analysis. Upon cooling at room temperature, dichloromethane was added and the solvent was removed by filtration. The remaining molecular sieves were stirred with another portion of dichloromethane for 1 h at room temperature, and the solvent was removed by filtration. The combined organic phases were washed with 5% aqueous NaOH. The aqueous layer was then extracted three times with dichloromethane and the combined organic layers were washed with brine. The organic layer was dried over Mg₂SO₄ and concentrated under vacuum to give the crude product. Purification by flash column chromatography on silica gel afforded the analytically pure product.

4-Chloro-3',4'-dimethyldiphenylether (entry 1). Procedure A was used to convert 1-chloro-4-iodobenzene and 3,4-dimethylphenol to the title product. Purification by flash column chromatography (1% Et₂O/pentane) gave the analytically pure product as a clear oil (530 mg, 91% yield). ¹H NMR (300 MHz, CDCl₃) δ 7.22 (d, *J* = 8.5 Hz, 2 H), 7.07 (d, *J* = 8.3 Hz, 1 H), 6.88 (d, *J* = 8.5 Hz, 2 H), 6.79 (br s, 1 H), 6.73 (br d, *J* = 8.3 Hz, 1 H), 2.22 (s, 6 H); ¹³C NMR (75 MHz, CDCl₃) δ 156.7, 154.5, 138.3, 131.9, 130.9, 129.5, 127.5, 120.5, 119.4, 116.5, 19.8, 18.9; IR (neat): 3025, 2931, 2851, 1878, 1584, 1490, 1249, 1155, 1091, 1002 cm⁻¹. Anal. Calcd for C₁₄H₁₃OCl: C, 72.26; H, 5.63. Found: C, 72.42; H, 5.54. Using 0.25 mol % (CuOTf)₂•PhH the yield was 89%.

4-(3',4'-Dimethylphenoxy)-ethylbenzoate (entry 2). Procedure A was used with 625 mg of molecular sieves 5 Å to convert ethyl-4-bromobenzoate and 3,4-dimethylphenol to the title product. Purification by flash column chromatography (1% Et₂O/pentane) gave the analytically pure product as a clear oil (425 mg, 63% yield). ¹H NMR (300 MHz, CDCl₃) δ 7.98 (d, *J* = 8.6 Hz, 2 H), 7.12 (d, *J* = 8.0 Hz, 1 H), 6.95 (d, *J* = 8.6 Hz, 2 H), 6.85 (d, *J* = 2.4 Hz, 1 H), 6.79 (dd, *J* = 8.0 Hz, 2.3 Hz, 1 H), 4.35 (q, *J* = 7.0 Hz, 2 H), 2.25 (s, 6 H), 1.37 (t, *J* = 7.0 Hz, 3 H); ¹³C NMR (75 MHz, CDCl₃) δ 166.1, 162.2, 153.3, 138.5, 132.8, 131.5, 130.8, 124.4, 121.3, 117.4, 116.8, 60.7, 19.9, 19.1, 14.3; IR (neat): 2978, 2931, 1713, 1595, 1495, 1367, 1261, 1226, 1161, 1102 cm⁻¹. Anal. Calcd for C₁₇H₁₈O₃: C, 75.53; H, 6.71. Found: C, 75.77; H, 6.67.

3,4,4'-Trimethyldiphenylether (entry 3). Procedure A was used to convert 4-iodotoluene and 3,4-dimethylphenol to the title product. Purification by flash column chromatography (1% Et₂O/pentane) gave the analytically pure product as a

clear oil (458 mg, 86% yield). ^1H NMR (300 MHz, CDCl_3) δ 7.11 (d, J = 8.3 Hz, 2 H), 7.06 (d, J = 8.1 Hz, 1 H), 6.88 (d, J = 8.2 Hz, 2 H), 6.80 (br s, 1 H), 6.73 (br d, J = 8.3 Hz, 1 H), 2.32 (s, 3 H), 2.22 (s, 6 H); ^{13}C NMR (75 MHz, CDCl_3) δ 155.4, 155.3, 138.0, 132.2, 131.0, 130.5, 130.1, 119.9, 118.5, 115.9, 20.6, 19.8, 18.9; IR (neat): 2919, 2849, 1608, 1496, 1449, 1249, 1214, 1161 cm^{-1} . Anal. Calcd for $\text{C}_{15}\text{H}_{16}\text{O}$: C, 84.87; H, 7.60. Found: C, 84.69; H, 7.68. Using 0.25 mol % $(\text{CuOTf})_2\cdot\text{PhH}$ the yield was 86%. Using 0.05 mol % $(\text{CuOTf})_2\cdot\text{PhH}$ the yield was 72%.

3',4'-Dimethyl-4-*tert*-butyldiphenylether (entries 4 and 5). Procedure A was used to convert 4-*tert*-butylbromobenzene or 4-*tert*-butyliodobenzene and 3,4-dimethylphenol to the title product. Purification by flash column chromatography (pentane) gave the analytically pure product as a clear oil (575 mg, 90% yield for the iodo compound; 550 mg, 86% yield for the bromo compound). ^1H NMR (300 MHz, CDCl_3) δ 7.30 (d, J = 8.5 Hz, 2 H), 7.06 (d, J = 8.4 Hz, 1 H), 6.90 (d, J = 8.4 Hz, 2 H), 6.82 (br s, 1 H), 6.74 (br d, J = 8.3 Hz, 1 H), 2.22 (s, 6 H), 1.31 (s, 9 H); ^{13}C NMR (75 MHz, CDCl_3) δ 155.4, 155.2, 145.4, 138.0, 131.1, 130.5, 126.3, 120.3, 117.9, 116.2, 34.2, 31.4, 19.9, 19.1; IR (neat): 2956, 2867, 1600, 1496, 1452, 1253, 1216, 1179, 1105, 1002 cm^{-1} . Anal. Calcd for $\text{C}_{18}\text{H}_{22}\text{O}$: C, 84.99; H, 8.72. Found: C, 85.23; H, 8.85.

3',4'-Dimethyl-4-methoxydiphenylether (entry 6). Procedure A was used to convert 4-iodoanisole and 3,4-dimethylphenol to the title product. Purification by flash column chromatography (1% Et_2O /pentane) gave the analytically pure product as a clear oil (445 mg, 78% yield). ^1H NMR (300 MHz, CDCl_3) δ 7.03 (d, J = 8.1 Hz, 1 H), 6.94 (d, J = 8.2 Hz, 2 H), 6.85 (d, J = 8.2 Hz, 2 H), 6.75 (br s, 1 H), 6.68 (br d, J = 8.3 Hz, 1 H), 3.77 (s, 3 H), 2.20 (s, 6 H); ^{13}C NMR (75 MHz, CDCl_3) δ 156.1, 155.5, 150.6, 137.9, 130.5, 130.4, 120.1, 119.1, 115.0, 114.8, 55.3, 19.8,

18.8; IR (neat): 2931, 2837, 1508, 1496, 1455, 1284, 1226, 1179, 1149, 1102, 1032 cm^{-1} . Anal. Calcd for $\text{C}_{15}\text{H}_{16}\text{O}_2$: C, 78.92; H, 7.06. Found: C, 79.12; H, 7.16.

3',4'-Dimethyl-4-(N,N-dimethylamino)diphenylether (entry 7).

Procedure A was used to convert 4-bromo-N,N-dimethylaniline and 3,4-dimethylphenol to the title product. Purification by flash column chromatography (5% EtOAc/hexane) gave the analytically pure product as a clear oil (561 mg, 83% yield). ^1H NMR (300 MHz, CDCl_3) δ 7.02 (d, J = 8.1 Hz, 1 H), 6.91-6.96 (m, 2 H), 6.65-6.75 (m, 4 H), 2.92 (s, 6 H), 2.20 (s, 6 H); ^{13}C NMR (75 MHz, CDCl_3) δ 157.0, 148.6, 147.6, 137.7, 130.4, 130.1, 120.4, 119.1, 115.0, 114.2, 41.2, 19.7, 18.7; IR (neat): 2973, 2913, 1677, 1606, 1515, 1496, 1445, 1355, 1249, 1224 cm^{-1} . Anal. Calcd for $\text{C}_{16}\text{H}_{19}\text{ON}$: C, 79.63; H, 7.94. Found: C, 79.48; H, 7.72.

3,3',4,5'-Tetramethyldiphenylether (entry 8). Procedure A was used to convert 5-iodo-*m*-xylene and 3,4-dimethylphenol to the title product. Purification by flash column chromatography (0.7% Et_2O /pentane) gave the analytically pure product as a clear oil (513 mg, 91% yield). ^1H NMR (300 MHz, CDCl_3) δ 7.06 (d, J = 8.2 Hz, 1 H), 6.81 (br s, 1 H), 6.74 (d, J = 8.2 Hz, 1 H), 6.69 (br s, 1 H), 6.60 (br s, 2 H), 2.26 (s, 6 H), 2.22 (s, 6 H); ^{13}C NMR (75 MHz, CDCl_3) δ 157.8, 155.0, 139.4, 138.0, 131.3, 130.5, 124.5, 120.5, 116.4, 116.0, 21.3, 19.9, 19.0; IR (neat): 3025, 2919, 2849, 1596, 1496, 1461, 1302, 1244, 1196, 1155, 1026 cm^{-1} . Anal. Calcd for $\text{C}_{16}\text{H}_{18}\text{O}$: C, 84.91; H, 8.02. Found: C, 85.07; H, 7.97.

2,3',4'-Trimethyldiphenylether (entry 9). Procedure A was used to convert 5-iodo-*m*-xylene and *o*-cresol to the title product. Purification by flash column chromatography (1% Et_2O /pentane) gave the analytically pure product as a clear oil (463 mg, 87% yield). ^1H NMR (300 MHz, CDCl_3) δ 7.24 (d, J = 6.5 Hz, 1 H),

7.16 (t, $J = 7.5$ Hz, 1 H), 7.05 (t, $J = 7.3$ Hz, 1 H), 6.89 (d, $J = 8.0$ Hz, 1 H), 6.69 (s, 1 H), 6.53 (s, 2 H), 2.26 (s, 6 H), 2.24 (s, 3 H); ^{13}C NMR (75 MHz, CDCl_3) δ 157.9, 154.6, 139.5, 131.3, 129.9, 127.1, 124.1, 123.7, 119.7, 115.0, 21.3, 16.2; IR (neat): 2919, 1584, 1490, 1378, 1302, 1226, 1185, 1138, 1108, 1026 cm^{-1} . Anal. Calcd for $\text{C}_{15}\text{H}_{16}\text{O}$: C, 84.87; H, 7.60. Found: C, 84.99; H, 7.66.

3,4',5-Trimethyldiphenylether (entry 10). Procedure A was used to convert 5-iodo-*m*-xylene and *p*-cresol to the title product. Purification by flash column chromatography (pentane) gave the analytically pure product as a clear oil (155 mg, 29% yield). Using procedure B, the yield was 80%. ^1H NMR (300 MHz, CDCl_3) δ 7.13 (d, $J = 8.3$ Hz, 2 H), 6.91 (d, $J = 8.2$ Hz, 2 H), 6.71 (s, 1 H), 6.61 (s, 2 H), 2.33 (s, 3 H), 2.27 (s, 6 H); ^{13}C NMR (75 MHz, CDCl_3) δ 157.8, 154.9, 139.5, 132.7, 130.2, 124.6, 119.1, 116.1, 21.3, 20.7; IR (neat): 3025, 2919, 2861, 1590, 1502, 1467, 1302, 1220, 1161, 1132, 1102, 1020 cm^{-1} . Anal. Calcd for $\text{C}_{15}\text{H}_{16}\text{O}$: C, 84.87; H, 7.60. Found: C, 85.00; H, 7.80.

2,2',5-Trimethyldiphenylether (entry 11). Procedure A was used to convert 2-iodo-*p*-xylene and *o*-cresol to the title product. Purification by flash column chromatography (hexane) gave the analytically pure product as a clear oil (448 mg, 84% yield). ^1H NMR (300 MHz, CDCl_3) δ 7.21 (d, $J = 7.3$ Hz, 1 H), 7.08 (t, $J = 7.3$ Hz, 2 H), 6.97 (t, $J = 7.3$ Hz, 1 H), 6.80 (d, $J = 7.8$ Hz, 1 H), 6.69 (d, $J = 8.0$ Hz, 1 H), 6.55 (s, 1 H), 2.28 (s, 3 H), 2.22 (s, 6 H); ^{13}C NMR (75 MHz, CDCl_3) δ 155.4, 154.9, 136.9, 131.2, 131.0, 128.6, 126.9, 125.7, 123.8, 122.8, 118.5, 117.4, 21.0, 16.1, 15.7; IR (neat): 3024, 2922, 2860, 1578, 1506, 1490, 1255, 1227, 1186, 1121 cm^{-1} . Anal. Calcd for $\text{C}_{15}\text{H}_{16}\text{O}$: C, 84.87; H, 7.60. Found: C, 85.11; H, 7.75.

2-(3',4'-Dimethylphenoxy)benzoic acid (entry 12). Procedure A was used to convert 2-bromobenzoic acid and 3,4-dimethylphenol to the title product. Purification by flash column chromatography (20% Et₂O/pentane) gave the analytically pure product as a white solid (466 mg, 77% yield), mp: 98 °C. ¹H NMR (300 MHz, CDCl₃) δ 8.18 (d, *J* = 8.0 Hz, 1 H), 7.45 (t, *J* = 7.9 Hz, 1 H), 7.20-7.15 (m, 2 H), 6.90-6.83 (m, 3 H), 2.25 (s, 6 H); ¹³C NMR (75 MHz, CDCl₃) δ 167.2, 157.7, 152.5, 138.8, 134.9, 133.8, 133.1, 130.9, 123.1, 121.2, 119.4, 117.6, 117.3, 19.9, 19.0; IR (KBr): 2931, 2590, 1685, 1596, 1572, 1482, 1402, 1302, 1243, 1214, 1149 cm⁻¹. Anal. Calcd for C₁₅H₁₄O₃: C, 74.36; H, 5.82. Found: C, 74.20; H, 5.85.

2-isopropyl-4'-methyldiphenylether (entry 14). Procedure A was used to convert 4-iodotoluene and 2-isopropylphenol to the title product. Purification by flash column chromatography (hexane) gave the analytically pure product as a clear oil (428 mg, 76% yield). ¹H NMR (300 MHz, CDCl₃) δ 7.28-7.32 (m, 1 H), 7.03-7.12 (m, 4 H), 6.79-6.85 (m, 3 H), 3.32 (septet, *J* = 7.0 Hz, 1 H), 2.28 (s, 3 H), 1.22 (d, *J* = 7.1 Hz, 1 H); ¹³C NMR (75 MHz, CDCl₃) δ 155.5, 151.3, 145.7, 131.9, 129.9, 124.3, 121.0, 120.4, 117.4, 112.9, 55.9, 20.5; IR (neat): 3026, 2941, 2868, 1506, 1490, 1265, 1188, 1120 cm⁻¹. Anal. Calcd for C₁₆H₁₈O: C, 84.91; H, 8.02. Found: C, 84.77; H, 8.16.

2',5'-dimethyl-2-isopropyldiphenylether (entry 15). Procedure A was used to convert 2-iodo-*p*-xylene and 2-isopropylphenol to the title product. Purification by flash column chromatography (hexane) gave the analytically pure product as a clear oil (500 mg, 83% yield). ¹H NMR (300 MHz, CDCl₃) δ 7.32 (dd, *J* = 2.2, 7.1 Hz, 3 H), 7.02-7.13 (m, 3 H), 6.82 (d, *J* = 7.2 Hz, 1 H), 6.67 (dd, *J* = 2.1, 7.2 Hz, 1 H), 6.58 (s, 1 H), 3.35 (septet, *J* = 6.8 Hz, 1 H), 2.23 (s, 6 H), 1.27 (d, *J* = 6.6 Hz, 6 H); ¹³C NMR (75 MHz, CDCl₃) δ 155.2, 154.5, 138.8, 137.0, 131.0,

126.72, 126.66, 125.9, 123.8, 122.9, 118.7, 117.3, 27.2, 22.9, 21.0, 15.8; IR (neat): 3029, 2961, 2868, 1620, 1576, 1506, 1490, 1449, 1282, 1188, 1120 cm^{-1} . Anal. Calcd for $\text{C}_{17}\text{H}_{20}\text{O}$: C, 84.96; H, 8.39. Found: C, 84.72; H, 8.24.

2-methoxy-4'-methyldiphenylether (entry 16). Procedure B was used to convert 2-bromoanisole and *p*-cresol to the title product. Purification by flash column chromatography (2% EtOAc/hexane) gave the analytically pure product as a clear oil (429 mg, 81% yield). ^1H NMR (300 MHz, CDCl_3) δ 7.11-7.15 (m, 3 H), 6.88-7.03 (m, 4 H), 3.87 (s, 3 H), 2.34 (s, 3 H); ^{13}C NMR (75 MHz, CDCl_3) δ 156.0, 154.1, 139.9, 131.8, 130.1, 126.81, 126.76, 123.8, 119.3, 117.7, 27.1, 23.0, 20.6; IR (neat): 3021, 2955, 1610, 1590, 1508, 1219, 1110, 1020 cm^{-1} . Anal. Calcd for $\text{C}_{14}\text{H}_{14}\text{O}_2$: C, 78.48; H, 6.59. Found: C, 78.27; H, 6.52.

4-phenoxybenzonitrile (entry 17).¹ Procedure B was used to convert 4-bromobenzonitrile and phenol to the title product. Purification by flash column chromatography (3% EtOAc/hexane) gave the analytically pure product as a clear oil (405 mg, 86% yield). ^1H NMR (300 MHz, CDCl_3) δ 7.57 (d, J = 8.7 Hz, 2 H), 7.40 (t, J = 7.9 Hz, 2 H), 7.22 (t, J = 7.6 Hz, 1 H), 7.05 (d, J = 7.9 Hz, 2 H), 6.98 (d, J = 8.8 Hz, 2 H); ^{13}C NMR (75 MHz, CDCl_3) δ 161.4, 154.6, 133.9, 130.0, 124.9, 120.2, 118.6, 117.7, 105.6; IR (neat): 3068, 2226, 1586, 1483, 1245, 1165 cm^{-1} .

4-phenoxy-*tert*-butylbenzene (entry 18).² Procedure B was used to convert 4-*t*-butylbromobenzene and phenol to the title product. Purification by flash column chromatography (2% EtOAc/hexane) gave the analytically pure product as a clear oil (457 mg, 81% yield). ^1H NMR (300 MHz, CDCl_3) δ 7.22-7.34 (m, 4 H), 6.90-7.05 (m, 5 H), 1.30 (s, 9 H); ^{13}C NMR (75 MHz, CDCl_3) δ 157.6, 154.7, 146.0, 129.6,

126.5, 122.9, 118.6, 118.5, 34.3, 31.5; IR (neat): 3038, 3962, 8867, 1590, 1508, 1489, 1240 cm^{-1} .

4-(4'-chlorophenoxy)acetophenone (entry 19). Procedure B was used to convert 4-iodoacetophenone and *p*-chlorophenol to the title product. Purification by flash column chromatography (4% EtOAc/hexane) gave the analytically pure product as a light yellow solid (580 mg, 94% yield), mp: 51-53 °C. ^1H NMR (300 MHz, CDCl_3) δ 7.91-7.96 (m, 2 H), 7.31-7.36 (m, 2 H), 6.96-7.02 (m, 4 H), 2.56 (s, 3 H); ^{13}C NMR (75 MHz, CDCl_3) δ 196.5, 161.5, 154.3, 132.4, 130.7, 130.7, 129.8, 121.4, 117.5, 26.5; IR (KBr): 2955, 1676, 1600, 1504, 1358, 1254, 1086 cm^{-1} . Anal. Calcd for $\text{C}_{14}\text{H}_{11}\text{O}_2\text{Cl}$: C, 68.16; H, 4.49. Found: C, 67.95; H, 4.64.

4-methyl-4'-chlorodiphenylether (entry 20). Procedure B was used to convert 4-iodotoluene and *p*-chlorophenol to the title product. Purification by flash column chromatography (2% EtOAc/hexane) gave the analytically pure product as a white solid (433 mg, 79% yield), mp: 47.5-49 °C. ^1H NMR (300 MHz, CDCl_3) δ 7.23 (d, J = 8.6 Hz, 2 H), 7.12 (d, J = 8.4 Hz, 2 H), 6.87-6.92 (m, 4 H), 2.32 (s, 3 H); ^{13}C NMR (75 MHz, CDCl_3) δ 156.4, 154.2, 133.2, 130.2, 129.5, 127.6, 119.4, 119.0, 20.8; IR (KBr): 3096, 3037, 2978, 2872, 1890, 1590, 1480, 1237, 1096 cm^{-1} . Anal. Calcd for $\text{C}_{13}\text{H}_{11}\text{OCl}$: C, 71.40; H, 5.07. Found: C, 71.67; H, 5.41.

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