Copper(II)-Catalyzed Ether Synthesis from Aliphatic Alcohols and Potassium Organotrifluoroborate Salts

Tan D. Quach and Robert A. Batey.*

*Davenport Research Laboratories, Department of Chemistry, University of Toronto,
80 St. George Street, Toronto, Ontario, M5S 3H6, Canada*

rbatey@chem.utoronto.ca

General Synthetic Methods

CH₂Cl₂ was distilled from CaH₂ under argon. All other commercial solvents and reagents were used as received from the Aldrich Chemical Company, Fischer Scientific Ltd., Strem or BDH. All glassware was oven dried at 210 °C and allowed to cool under a stream of dry nitrogen.

References following compound names indicate literature articles where ¹H and ¹³C NMR data have previously been reported.

Silica gel (60 Å, 230-400 mesh) used in flash column chromatography was obtained from Whatman Company, Toronto Research Chemicals, Inc. or Silicycle, and was used as received. Analytical thin-layer chromatography (TLC) was performed on pre-coated silica gel plates (Ultra Pure Silica Gel Plates purchased from Silicycle), visualized with a Spectroline UV₂₅₄ lamp, and stained with a 20% phosphomolybdic acid in ethanol solution. Solvent systems associated with R_f values and flash column chromatography are reported as v/v ratios.

Melting points were obtained using a Fisher-Johns melting point apparatus, and are uncorrected. Specific optical rotations were determined on a Perkin-Elmer 243B Polarimeter under the conditions indicated using the sodium D line (589 nm). Sample concentrations are reported as grams per 100 mL solvent. Analytical chiral HPLC (of **2t**) was performed on a Chiralcel OD column (4.6 x 250 mm) obtained from Daicel Chemical Industries, Ltd. ¹H and ¹³C NMR were recorded at 400 or 300 MHz and 100 or 75 MHz respectively on a Varian Unity 400, Gemini 300 or Mercury 300 spectrometer. Proton chemical shifts were internally referenced to the residual proton resonance in CDCl₃ (δ 7.26 ppm). Carbon chemical shifts were internally referenced to the dueterated solvent signals in CDCl₃ (δ 77.20 ppm). FT-IR spectra were recorded on a Perkin-Elmer Spectrum 1000 spectrometer with samples loaded as neat films on NaCl plates or as pressed KBr discs. Low and high resolution mass spectra were recorded on a Bell and Howell 21-490 spectrometer and an AEI MS3074 spectrometer respectively.

Synthetic Methods and Characterization Data

Representative Procedure for the Cross-Coupling of $\text{PhBF}_3^-\text{K}^+$ with Aliphatic Alcohols

A suspension of $\text{PhBF}_3^-\text{K}^+$ (0.736 g, 4.00 mmol), $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$ (0.040 g, 0.20 mmol), DMAP (0.050 g, 0.40 mmol) and powdered 4Å molecular sieves (1.50 g) in CH_2Cl_2 (15.0 mL) was stirred for 5 minutes at room temperature. To this stirring suspension was added (*E*)-cinnamyl alcohol (0.268 g, 2.00 mmol). The stirring reaction mixture was then sealed with a rubber septa and stirred under an atmosphere of oxygen (delivered via an oxygen tank connected to a manifold). Following a period of 24 h, the crude reaction mixture was filtered through a plug of celite to remove the molecular sieves and any insoluble byproducts, and then concentrated *in vacuo* to afford the crude product mixture. The product (**2a**) was isolated by silica gel column chromatography (eluting with hexanes:EtOAc 9:1 ~ 3:1 gradient) as a white crystalline solid in 89 % yield (0.374 g, 1.78 mmol).

Products of Experiments Summarized in Table 1

(*E*)-Cinnamylphenyl ether (2a) (Keinan, E.; Sahai, M.; Roth, Z. *J. Org. Chem.* **1985**, *50*, 3558-3566.)

Isolated as a white, crystalline solid in 89 % yield: R_f = 0.6 (9:1 hexanes:EtOAc); ^1H NMR (CDCl_3 , 300 MHz) δ 7.48 (2H, d, J = 7.0 Hz), 7.44 - 7.28 (5H, m), 7.11 - 6.95 (3H, m), 6.80 (1H, d, J = 16.0 Hz), 6.48 (1H, dt, J = 16.0, 6.0 Hz), 4.75 (2H, dd, J = 6.0, 1.5 Hz); ^{13}C NMR (CDCl_3 , 75 MHz) δ 158.66, 136.54, 133.05, 129.58, 128.67, 127.97, 126.67, 124.63, 121.00, 114.91, 68.76.

Phenyl-(3-phenylpropyl) ether (2b)

Isolated as a light yellow oil in 95 % yield: R_f = 0.7 (9:1 hexanes:EtOAc); ^1H NMR (CDCl_3 , 300 MHz) δ 7.40 - 7.24 (7H, m), 7.04 - 6.94 (3H, m), 4.03 (2H, t, J = 6.5 Hz), 2.90 (2H, t, J = 7.5 Hz), 2.22-2.14 (2H, m); ^{13}C NMR (CDCl_3 , 75 MHz) δ 158.87, 141.45, 129.38, 128.48, 128.37, 125.89, 120.57, 114.54, 66.93, 32.49, 31.19; IR (film) ν 3062, 3027, 2946, 2870, 1736, 1600, 1586, 1497, 1470, 1454, 1389, 1301, 1246, 1172, 1080, 1039, 940, 882, 806, 752 cm^{-1} ; LRMS (EI): m/z = 213 (12), 212 (67), 119 (21), 118 (80), 117 (44), 94 (22), 92 (12), 91 (100), 77 (13), 65 (21); HRMS (EI): m/z calcd. for ($\text{C}_{15}\text{H}_{16}\text{O}^+$) = 212.1201, found = 212.1206.

Phenylpropyl ether (2c) (Chen, C. T.; Marder, S. R.; Cheng, L. T. *J. Amer. Chem. Soc.* **1994**, *116*, 3117-3118.)

Isolated as a clear liquid in 78 % yield: $R_f = 0.8$ (9:1 hexanes:EtOAc); ^1H NMR (CDCl_3 , 300 MHz) δ 7.40 - 7.28 (2H, m), 7.00 - 6.90 (3H, m), 3.95 (2H, t, $J = 7.0$ Hz), 1.85 (2H, sextet, $J = 7.0$ Hz), 1.09 (3H, t, $J = 7.0$ Hz); ^{13}C NMR (CDCl_3 , 75 MHz) δ 159.01, 129.37, 120.47, 114.53, 69.57, 23.00, 10.96.

Benzylphenyl ether (2d) (Forrester, J.; Jones, R. V. H.; Newton, L.; Preston, P. N. *Tetrahedron* **2001**, *57*, 2871-2884.)

Isolated as a white, crystalline solid in 71 % yield: $R_f = 0.7$ (9:1 hexanes:EtOAc); ^1H NMR (CDCl_3 , 300 MHz) δ 7.47 - 7.25 (7H, m), 7.02 - 6.93 (3H, m), 5.09 (2H, s); ^{13}C NMR (CDCl_3 , 75 MHz) δ 158.67, 137.05, 129.45, 128.56, 127.91, 127.46, 120.95, 114.90, 70.12.

(4-Bromobenzyl)phenyl ether (2e)

Isolated as a white, crystalline solid in 94 % yield: $R_f = 0.5$ (3:1 hexanes:toluene); mp = 91 - 92 °C (CH_2Cl_2); ^1H NMR (CDCl_3 , 300 MHz) δ 7.52 (2H, d, $J = 8.0$ Hz), 7.34 - 7.25 (4H, m), 7.02 - 6.94 (3H, m), 5.03 (2H, s); ^{13}C NMR (CDCl_3 , 75 MHz) δ 158.36, 136.07, 131.67, 129.52, 129.04, 121.18, 121.18, 114.87, 69.34; IR (film) ν 3080, 3048, 2922, 2878, 1916, 1600, 1490, 1456, 1406, 1384, 1240, 1079, 1032, 1016, 994, 871, 811, 753, 691 cm^{-1} ; LRMS (EI): $m/z = 264$ (21), 262 (22), 172 (12), 171 (98), 170 (14), 169 (100), 90 (30), 89 (19), 65 (9), 63 (10), 51 (6); HRMS (EI): m/z calcd. for ($\text{C}_{13}\text{H}_{11}\text{BrO}^+$) = 261.9993, found = 261.9994.

(4-Nitrobenzyl)phenyl ether (2f) (Maslak, P.; Guthrie, R. D. *J. Am. Chem. Soc.* **1986**, *108*, 2628-2636.)

Isolated as a white, crystalline solid in 75 % yield: $R_f = 0.3$ (9:1 hexanes:toluene); ^1H NMR (CDCl_3 , 300 MHz) δ 8.23 (2H, d, $J = 8.5$ Hz), 7.61 (2H, d, $J = 8.5$ Hz), 7.32 (2H, t, $J = 7.5$ Hz), 7.05 - 6.96 (3H, m), 5.19 (2H, s); ^{13}C NMR (CDCl_3 , 75 MHz) δ 157.95, 147.41, 144.51, 129.61, 127.53, 123.76, 121.53, 114.80, 68.74; LRMS (EI): $m/z = 230$ (23), 229 (93), 137 (15), 136 (100), 106 (42), 90 (28), 89 (25), 84 (14), 78 (45), 65 (14), 63 (11); HRMS (EI): m/z calcd. for ($\text{C}_{13}\text{H}_{11}\text{NO}_3^+$) = 229.0739, found = 229.0734.

(4-Methoxybenzyl)phenyl ether (2g) (Forrester, J.; Jones, R. V. H.; Newton, L.; Preston, P. N. *Tetrahedron* **2001**, *57*, 2871-2884.)

Isolated as a white, crystalline solid in 80 % yield: R_f = 0.4 (9:1 hexanes:EtOAc); ^1H NMR (CDCl_3 , 300 MHz) δ 7.40 - 7.28 (4H, m), 7.03 - 6.92 (5H, m), 5.01 (2H, s), 3.84 (3H, s); ^{13}C NMR (CDCl_3 , 75 MHz) δ 159.30, 158.71, 129.42, 129.20, 129.07, 120.85, 114.86, 114.03, 69.88, 55.54.

2-Phenoxymethyl furan (2h) (Katritzky, A. R.; Abdel-Megeed, M. F.; Lhommet, G.; Ramsden, C. A. *J. Chem. Soc. Perkin Trans. 1* **1979**, 426-429.)

Isolated as a clear oil in 90 % yield: R_f = 0.6 (9:1 hexanes:EtOAc); ^1H NMR (CDCl_3 , 300 MHz) δ 7.46 (1H, dd, J = 2.0, 1.0 Hz), 7.35 - 7.28 (2H, m), 7.04 - 6.96 (3H, m), 6.45 (1H, dd, J = 3.5, 0.5 Hz), 6.39 (1H, dd, J = 3.5, 2.0 Hz), 5.02 (2H, s); ^{13}C NMR (CDCl_3 , 75 MHz) δ 158.24, 150.24, 142.98, 129.44, 121.23, 114.94, 110.53, 109.91, 62.57.

(2-Butynyl)phenyl ether (2i) (Larock, R. C.; Harrison, L. W. *J. Amer. Chem. Soc.* **1984**, 106, 4218-4227.)

Isolated as a clear oil in 92 % yield: R_f = 0.7 (9:1 hexanes:EtOAc); ^1H NMR (CDCl_3 , 300 MHz) δ 7.31 (2H, t, J = 7.5 Hz), 7.02 - 6.94 (3H, m), 4.66 (2H, q, J = 2.0 Hz), 1.88 (3H, t, J = 2.0 Hz); ^{13}C NMR (CDCl_3 , 75 MHz) δ 157.69, 129.37, 121.19, 114.83, 83.77, 74.26, 56.56, 4.11.

1, 8-diphenoxy-octa-3, 5-diyne (2j)

Isolated as a beige, needle-like, crystalline solid in 61% yield: R_f = 0.7 (9:1 hexanes:EtOAc); mp = 133 - 135 °C (hexanes); ^1H NMR (CDCl_3 , 300 MHz) δ 7.28 (4H, t, J = 8.0 Hz), 6.96 (2H, t, J = 8.0 Hz), 6.89 (4H, d, J = 8.0 Hz), 4.09 (4H, t, J = 7.0 Hz), 2.76 (4H, t, J = 7.0 Hz); ^{13}C NMR (CDCl_3 , 75 MHz) δ 158.17, 129.50, 121.18, 114.70, 74.06, 66.81, 65.77, 20.73; IR (film) ν 2936, 1597, 1499, 1470, 1390, 1304, 1246, 1176, 1082, 1041, 883, 812, 752, 689 cm^{-1} ; LRMS (EI): m/z = 291 (18), 290 (68), 289 (15), 262 (19), 261 (16), 183 (41), 182 (17), 181 (23), 157 (15), 153 (16), 133 (52), 107 (34), 79 (31), 78 (15), 77 (100), 65 (19), 63 (17), 51 (29); HRMS (EI): m/z calcd. for ($\text{C}_{20}\text{H}_{18}\text{O}_2$) $^+$ = 290.1307, found = 290.1307.

Phenyl-2,2,2-trifluoroethyl ether (2k) (Keegstra, M. A.; Peters, T. H. A.; Brandsma, L. *Tetrahedron* **1992**, 48, 3633-3652.)

Isolated as a clear oil in 82 % yield: R_f = 0.7 (9:1 hexanes:EtOAc); ^1H NMR (CDCl_3 , 300 MHz) δ 7.38 - 7.30 (2H, m), 7.10 - 7.02 (1H, m), 6.99 - 6.93 (2H, m), 4.36 (2H, q, J = 8.0 Hz); ^{13}C NMR (CDCl_3 , 75 MHz) δ 157.30, 129.73, 122.54, 114.99, 66.08 (q, J = 35.5 Hz); ^{19}F NMR (CDCl_3 , 282 MHz) δ -73.98 (3F, t, J = 8.0 Hz).

(3-Chloropropyl)phenyl ether (2l) (Foubelo, F.; Saleh, S. A.; Yus, M. *J. Org. Chem.* **2000**, *65*, 3478-3483.)

Isolated as a clear oil in 95 % yield: R_f = 0.7 (9:1 hexanes:EtOAc); ^1H NMR (CDCl_3 , 300 MHz) δ 7.34 - 7.26 (2H, m), 7.00 - 6.90 (3H, m), 4.13 (2H, t, J = 6.0 Hz), 3.77 (2H, t, J = 6.0 Hz), 2.26 (2H, quintet, J = 6.0 Hz); ^{13}C NMR (CDCl_3 , 75 MHz) δ 158.57, 129.47, 120.91, 114.56, 64.38, 41.85, 32.66.

(3-Bromopropyl)phenyl ether (2m) (Reinholz, E.; Becker, A.; Hagenbruch, B.; Schaefer, S.; Schmitt, A. *Synthesis* **1990**, *11*, 1069-1071.)

Isolated as a clear oil in 93 % yield: R_f = 0.7 (9:1 hexanes:EtOAc); ^1H NMR (CDCl_3 , 300 MHz) δ 7.36 - 7.31 (2H, m), 7.03 - 6.94 (3H, m), 4.14 (2H, t, J = 6.0 Hz), 3.65 (2H, t, J = 6.0 Hz), 2.36 (2H, quintet, J = 6.0 Hz); ^{13}C NMR (CDCl_3 , 75 MHz) δ 158.79, 129.66, 121.06, 114.63, 65.27, 32.54, 30.29.

Phenyl-*p*-tolyl ether (2n) (Haga, N.; Takayanagi, H. *J. Org. Chem.* **1996**, *61*, 735-745.)

Isolated as a clear oil in 82 % yield: R_f = 0.60 (5:1 hexanes:toluene); ^1H NMR (300 MHz, CDCl_3) δ 7.50 - 7.41 (2H, m), 7.30 - 7.05 (7H, m), 2.48 (3H, s); ^{13}C NMR (100 MHz, CDCl_3) δ 158.00, 154.90, 133.03, 130.40, 129.81, 123.37, 119.30, 118.51, 20.86.

4-Cyanodiphenyl ether (2o) (Gujadhur, R.; Venkataraman, D. *Synth. Commun.* **2001**, *31*, 2865-2880.)

Isolated as a thick, yellow oil in 77 % yield: R_f = 0.70 (3:1 hexanes:EtOAc); ^1H NMR (300 MHz, CDCl_3) δ 7.60 (2H, ddd, J = 9.5, 5.0, 2.5 Hz), 7.42 (2H, t, J = 7.5 Hz), 7.23 (1H, tt, J = 7.0, 1.0 Hz), 7.06 (2H, ddd, J = 7.5, 3.5, 1.0 Hz), 7.00 (2H, ddd, J = 9.5, 5.0, 2.5 Hz); ^{13}C NMR (75 MHz, CDCl_3) δ 161.84, 154.99, 134.30, 130.41, 125.32, 120.58, 119.02, 118.09, 106.00.

Allylphenyl ether (2p) (Annunziata, A.; Galli, C.; Marinelli, M.; Pau, T. *Eur. J. Org. Chem.* **2001**, *7*, 1323-1330.)

Isolated as a clear oil in 93 % yield: R_f = 0.8 (9:1 hexanes:EtOAc); ^1H NMR (CDCl_3 , 300 MHz) δ 7.38 - 7.28 (2H, m), 7.00 - 6.91 (3H, m), 6.09 (1H, ddt, J = 17.5, 10.5, 5.5 Hz), 5.44 (1H, dd, J = 17.5, 1.5 Hz), 5.31 (1H, dd, J = 10.5, 1.5 Hz), 4.55 (2H, dt, J = 5.5, 1.5 Hz); ^{13}C NMR (CDCl_3 , 75 MHz) δ 158.71, 133.49, 129.58, 120.97, 117.76, 114.85, 68.82.

(E)-Pent-2-enylphenyl ether (2q)

Isolated as a clear oil in 74 % yield: R_f = 0.4 (3:1 hexanes:toluene); ^1H NMR (CDCl_3 , 300 MHz) δ 7.28 (2H, d, J = 8.0 Hz), 6.98 - 6.90 (3H, m), 5.91 (1H, dt, J = 15.5, 6.0 Hz), 5.72 (1H, ddd, J = 15.5, 6.0, 1.0 Hz), 4.49 (2H, dd, J = 6.0, 1.0 Hz), 2.14 (2H, ddq, J = 7.5, 7.5, 1.0 Hz), 1.06 (3H, t, J = 7.5 Hz); ^{13}C NMR (CDCl_3 , 75 MHz) δ 158.61, 137.10, 129.37, 123.84, 120.66, 114.74, 68.94, 25.72, 13.60; IR (film) ν 2964, 1600, 1496, 1461, 1381, 1301, 1241, 1172, 1078, 1030, 1011, 968, 881, 753, 691 cm^{-1} ; LRMS (EI): m/z = 162 (8), 95 (12), 94 (100), 77 (8), 69 (16), 68 (19), 67 (8), 66 (6), 65 (9); HRMS (EI): m/z calcd. for ($\text{C}_{11}\text{H}_{14}\text{O}^+$) = 162.1045, found = 162.1037.

(Z)-Pent-2-enylphenyl ether (2r)

^1H NMR analysis of the starting material reveals a 1:20 ratio of E:Z geometric isomers which is carried through to the cross-coupled products. Isolated as a clear oil in 83 % yield: R_f = 0.6 (3:1 hexanes:toluene); ^1H NMR (CDCl_3 , 300 MHz) δ 7.32 - 7.25 (2H, m), 6.98 - 6.90 (3H, m), 5.70 - 5.65 (2H, m), 4.60 (2H, d, J = 5.5 Hz), 2.23 - 2.15 (2H, m), 1.05 (3H, t, J = 7.5 Hz); ^{13}C NMR (CDCl_3 , 75 MHz) δ 158.60, 135.96, 129.39, 124.17, 120.71, 114.74, 63.99, 21.52, 14.49; IR (film) ν 3028, 2965, 2874, 1600, 1586, 1496, 1300, 1242, 1172, 1078, 1031, 1011, 991, 881, 753, 691 cm^{-1} ; LRMS (EI): m/z = 162 (30), 95 (12), 94 (100), 77 (13), 69 (20), 68 (24), 67 (11), 66 (10), 65 (20), 53 (7), 51 (10).

Geranylphenyl ether (2s) (Nakamura, S.; Ishihara, K.; Yamamoto, H. *J. Amer. Chem. Soc.* **2000**, *122*, 8131-8140.)

Isolated as a clear oil in 87 % yield: R_f = 0.6 (4:1 hexanes:toluene); ^1H NMR (CDCl_3 , 300 MHz) δ 7.34 - 7.26 (2H, m), 7.00 - 6.91 (3H, m), 5.53 (1H, tq, J = 3.5, 1.5 Hz), 5.18 - 5.00 (1H, m), 4.57 (2H, d, J = 6.0 Hz), 2.23 - 2.08 (4H, m), 1.78 (3H, s), 1.73 (3H, s), 1.66 (3H, s); ^{13}C NMR (CDCl_3 , 75 MHz) δ 158.74, 140.91, 131.70, 129.34, 123.83, 120.55, 119.64, 114.71, 64.95, 39.86, 26.67, 26.03, 18.07, 17.02.

(R)-2-Methyl-3-phenoxypropionic acid methyl ester (2t) (Toda, S.; Miyamoto, M.; Kinoshita, H.; Inomata, K. *Bull. Chem. Soc. Jpn.* **1991**, *64*, 3600-3606.)

Isolated as a clear oil in 90 % yield: R_f = 0.5 (9:1 hexanes:EtOAc); $[\alpha]_D^{22}$ = -13.2 ° (c = 3.1, CH_2Cl_2) for >99% ee (Chiralcel OD, 1% isopropanol/hexanes, 1.0 mL/min, retention times for R and S enantiomers are 8.26 and 9.98 min respectively) with determination of the absolute stereochemistry by comparison of the specific rotation with the literature value; ^1H NMR (CDCl_3 , 300 MHz) δ 7.32 - 7.25 (2H, m), 6.99 - 6.87 (3H, m), 4.20 (1H,

dd, $J = 9.0, 6.5$ Hz), 4.00 (1H, dd, $J = 9.0, 6.5$ Hz), 3.73 (3H, s), 2.97 (1H, sextet, $J = 7.0$ Hz), 1.32 (3H, d, $J = 7.0$ Hz); ^{13}C NMR (CDCl_3 , 75 MHz) δ 174.44, 158.49, 129.39, 120.96, 114.68, 69.65, 52.11, 40.07, 14.38.

Methyl-(2-phenoxyethyl)carbamic acid *tert*-butyl ester (2u)

Isolated as a clear oil in 93 % yield: $R_f = 0.6$ (9:1 hexanes: EtOAc); ^1H NMR (CDCl_3 , 300 MHz) δ 7.28 (2H, t, $J = 7.5$ Hz), 6.94 (1H, t, $J = 7.5$ Hz), 6.89 (2H, d, $J = 7.5$ Hz), 4.09 (2H, t, $J = 5.5$ Hz), 3.60 (2H, t, $J = 5.5$ Hz), 2.99 (3H, s), 1.47 (9H, s); ^{13}C NMR (CDCl_3 , 75 MHz) δ 158.47, 155.53, 129.41, 120.81, 114.41, 79.69, 66.49 (broad), 48.57, 36.10 (broad), 28.75; IR (film) ν 2976, 1697, 1600, 1497, 1391, 1366, 1303, 1246, 1157, 1046, 882, 754 cm^{-1} ; LRMS (EI): $m/z = 251$ (5), 196 (11), 179 (17), 178 (90), 158 (31), 120 (19), 108 (14), 107 (11), 103 (10), 102 (100), 94 (15), 77 (29), 58 (11), 57 (59); HRMS (EI): m/z calcd. for ($\text{C}_{14}\text{H}_{21}\text{NO}_3 + \text{H}^+$) = 252.1600, found = 252.1609.

(3-Phenoxypropyl)carbamic acid benzyl ester (2v)

Isolated as a clear oil in 85 % yield: $R_f = 0.4$ (3:1 hexanes: EtOAc); ^1H NMR (CDCl_3 , 300 MHz) δ 7.38 - 7.25 (7H, m), 6.98 - 6.86 (3H, m), 5.12 (2H, s), 5.08 (1H, s, broad), 4.03 (2H, t, $J = 6.0$ Hz), 3.42 (2H, q, $J = 6.0$ Hz), 2.02 (2H, quintet, $J = 6.0$ Hz); ^{13}C NMR (CDCl_3 , 75 MHz) δ 158.51, 156.32, 136.53, 129.44, 128.47, 128.04, 120.87, 114.48, 66.83, 65.85, 38.96, 29.72 (one signal absent); IR (film) ν 3336, 3064, 3033, 2949, 2878, 1702, 1600, 1587, 1530, 1498, 1471, 1455, 1243, 1173, 1142, 1080, 1041, 754, 693 cm^{-1} ; LRMS (EI): $m/z = 193$ (12), 192 (84), 177 (7), 121 (5), 108 (9), 107 (9), 94 (20), 92 (10), 91 (100), 84 (6), 79 (10), 77 (15), 65 (17), 56 (10), 51 (8); HRMS (EI): m/z calcd. for ($\text{C}_{17}\text{H}_{19}\text{NO}_3^+$) = 285.1365, found = 285.1374.

Methyl-(2-phenoxyethyl)phenyl amine (2w)

Isolated as a clear oil in 30 % yield: $R_f = 0.5$ (9:1 hexanes: EtOAc); ^1H NMR (CDCl_3 , 300 MHz) δ 7.34 - 7.25 (4H, m), 7.01 - 6.90 (3H, m), 6.83 - 6.73 (3H, m), 4.17 (2H, t, $J = 6.0$ Hz), 3.80 (2H, t, $J = 6.0$ Hz), 3.09 (3H, s); ^{13}C NMR (CDCl_3 , 75 MHz) δ 158.83, 149.07, 129.64, 129.45, 121.02, 116.63, 114.60, 112.25, 65.21, 52.05, 39.30; IR (film) ν 3027, 2923, 1599, 1497, 1368, 1301, 1245, 1172, 1077, 1034, 992, 884, 749, 691 cm^{-1} ; LRMS (EI): $m/z = 228$ (9), 227 (48), 212 (11), 121 (14), 120 (100), 105 (8), 104 (8), 91 (5), 84 (6), 77 (19), 51 (7); HRMS (EI): m/z calcd. for ($\text{C}_{15}\text{H}_{17}\text{NO}^+$) = 227.1310, found = 227.1307.

Isopropylphenyl ether (2x) (Keegstra, M. A.; Peters, T. H. A.; Brandsma, L. *Tetrahedron* **1992**, 48, 3633-3652.)

Isolated as a clear oil in 71 % yield: $R_f = 0.8$ (9:1 hexanes:EtOAc); ^1H NMR (CDCl_3 , 300 MHz) δ 7.30 - 7.24 (2H, m), 6.98 - 6.88 (3H, m), 4.57 (1H, septet, $J = 6.0$ Hz), 1.37 (6H, d, $J = 6.0$ Hz); ^{13}C NMR (CDCl_3 , 75 MHz) δ 157.77, 129.43, 120.49, 115.94, 69.94, 22.46.

(1-Methylallyl)phenyl ether (2y)

Isolated as a clear oil in 69 % yield: $R_f = 0.8$ (9:1 hexanes:EtOAc); ^1H NMR (CDCl_3 , 300 MHz) δ 7.38 - 7.30 (2H, m), 6.96 - 6.89 (3H, m), 5.93 (1H, ddd, $J = 17.5, 10.5, 6.0$ Hz), 5.28 (1H, dd, $J = 17.5, 1.0$ Hz), 5.18 (1H, dd, $J = 10.5, 1.0$ Hz), 4.82 (1H, ddq, $J = 6.0, 6.0, 1.0$ Hz), 1.46 (3H, d, $J = 6.0$ Hz); ^{13}C NMR (CDCl_3 , 75 MHz) δ 157.88, 139.19, 129.31, 120.73, 116.07, 115.57, 74.72, 21.67; IR (film) ν 3040, 2981, 1585, 1488, 1287, 1239, 1164, 1072, 1024, 990, 928, 866, 797, 750 cm^{-1} .

Benzhydrylphenyl ether (2z)

Isolated as a clear liquid in 75 % yield: $R_f = 0.6$ (9:1 hexanes: EtOAc); ^1H NMR (CDCl_3 , 300 MHz) δ 7.54 - 7.26 (12H, m), 7.07 - 6.95 (3H, m), 6.30 (1H, s); ^{13}C NMR (CDCl_3 , 75 MHz) δ 158.25, 141.43, 129.53, 128.77, 127.89, 127.06, 121.13, 116.24, 81.80; IR (film) ν 3062, 3029, 1597, 1586, 1493, 1454, 1306, 1235, 1172, 1078, 1029, 916, 880, 817, 753, 699, 655 cm^{-1} ; LRMS (EI): $m/z = 262$ (43), 260 (10), 183 (25), 168 (24), 167 (100), 166 (17), 165 (39), 152 (20), 65 (12); HRMS (EI): m/z calcd. for $(\text{C}_{19}\text{H}_{16}\text{O}^+)$ = 260.1201, found = 260.1196.

Cyclohexylphenyl ether (2aa) (Tateiwa, J.; Nishimura, T.; Horiuchi, H.; Uemura, S. *J. Chem. Soc. Perkin Trans. I* **1994**, 23, 3367-3372.)

Isolated as a clear oil in 67 % yield: $R_f = 0.8$ (9:1 hexanes:EtOAc); ^1H NMR (CDCl_3 , 300 MHz) δ 7.40 - 7.25 (2H, m), 6.98 - 6.90 (3H, m), 4.27 (1H, m), 2.10 - 1.98 (2H, m), 1.91 - 1.80 (2H, m), 1.68 - 1.50 (3H, m), 1.48 - 1.28 (3H, m); ^{13}C NMR (CDCl_3 , 75 MHz) δ 157.72, 129.38, 120.47, 116.09, 75.50, 32.20, 26.02, 24.18.

O-Phenyl stigmasterol (2bb)

Isolated as a clear crystal in 48 % yield: $R_f = 0.9$ (9:1 hexanes: EtOAc); $[\alpha]_D^{22} = -40.7^\circ$ ($c = 1.4$, CH_2Cl_2); ^1H NMR (CDCl_3 , 300 MHz) δ 7.29 - 7.23 (2H, m), 6.94 - 6.88 (3H, m), 5.40 (1H, d, $J = 5.0$ Hz), 5.17 (1H, dd, $J = 15.0, 8.5$ Hz), 5.02 (1H, dd, $J = 15.0, 8.5$ Hz), 4.18 - 4.07 (1H, m), 2.54 - 2.36 (2H, m), 2.11 - 1.90 (4H, m), 1.76 - 1.66 (2H, m),

1.65 - 1.34 (8H, m), 1.33 - 1.12 (6H, m), 1.10 - 1.02 (9H, m), 0.90 - 0.80 (9H, m), 0.73 (3H, s); ^{13}C NMR (CDCl_3 , 75 MHz) δ 157.62, 140.33, 138.28, 129.45, 129.30, 122.29, 120.62, 115.98, 77.20, 57.15, 56.25, 51.54, 50.54, 42.56, 40.83, 40.02, 39.06, 37.56, 37.21, 32.31, 32.27, 29.30, 28.65, 25.81, 24.76, 21.64, 21.50, 21.48, 19.86, 19.41, 12.70, 12.50; IR (film) ν 3037, 2950, 2851, 2361, 1583, 1486, 1379, 1366, 1290, 1230, 1152, 1088, 1037, 1021, 972, 866, 839, 806, 758, 693 cm^{-1} ; LRMS (EI): m/z = 488 (1), 397 (18), 396 (27), 395 (64), 311 (10), 255 (28), 161 (14), 159 (15), 149 (13), 147 (17), 145 (13), 139 (41), 135 (12), 133 (14), 121 (19), 119 (11), 109 (15), 107 (16), 105 (20), 97 (23), 95 (24), 94 (17), 93 (22), 91 (18), 84 (14), 83 (100), 81 (31), 79 (18), 77 (10), 71 (19), 69 (47), 67 (16), 57 (28), 55 (42); HRMS (EI): m/z calcd. for $(\text{C}_{35}\text{H}_{52}\text{O}^+)$ = 488.4018, found = 488.4027.

Products of Experiments Summarized in Table 2**2-(4-Methoxyphenoxyethyl) furan (3a)**

Isolated as a clear, crystalline solid in quantitative yield: $R_f = 0.4$ (9:1 hexanes: EtOAc); ^1H NMR (CDCl_3 , 300 MHz) δ 7.44 (1H, dd, $J = 2.0, 1.0$ Hz), 6.93 (2H, d, $J = 9.0$ Hz), 6.84 (2H, d, $J = 9.0$ Hz), 6.41 (1H, d, $J = 3.0$ Hz), 6.38 (1H, dd, $J = 3.0, 2.0$ Hz), 4.95 (2H, s), 3.78 (3H, s); ^{13}C NMR (CDCl_3 , 75 MHz) δ 154.11, 152.37, 150.47, 142.92, 116.21, 114.62, 110.48, 109.80, 63.53, 55.89; IR (film) ν 2933, 2834, 1592, 1508, 1464, 1371, 1232, 1150, 1108, 1037, 1010, 923, 825, 780, 744 cm^{-1} ; LRMS (EI): $m/z = 204$ (32), 123 (18), 95 (10), 82 (8), 81 (100), 53 (18), 52 (6); HRMS (EI): m/z calcd. for ($\text{C}_{12}\text{H}_{12}\text{O}_3^+$) = 204.0786, found = 204.0779.

2-(3-Chlorophenoxyethyl) furan (3b)

Isolated as a clear oil in 71 % yield: $R_f = 0.5$ (3:1 hexanes: toluene); ^1H NMR (CDCl_3 , 300 MHz) δ 7.46 (1H, dd, $J = 2.0, 1.0$ Hz), 7.21 (1H, t, $J = 8.0$ Hz), 7.02 - 6.96 (2H, m), 6.88 (1H, ddd, $J = 8.0, 2.5, 1.0$ Hz), 6.44 (1H, d, $J = 3.0$ Hz), 6.40 (1H, dd, $J = 3.0, 2.0$ Hz), 4.99 (2H, s); ^{13}C NMR (CDCl_3 , 75 MHz) δ 158.89, 149.59, 143.16, 134.78, 130.19, 121.39, 115.36, 113.40, 110.59, 110.27, 62.73; IR (film) ν 3123, 3070, 2933, 2874, 1594, 1477, 1371, 1284, 1247, 1151, 1099, 1073, 1011, 924, 898, 768, 744, 680 cm^{-1} ; LRMS (EI): $m/z = 210$ (6), 208 (8), 143 (5), 141 (16), 128 (11), 113 (7), 111 (13), 101 (6), 99 (16), 83 (7), 82 (11), 81 (100), 75 (10), 73 (8), 64 (6), 63 (12), 53 (30), 52 (6), 51 (9); HRMS (EI): m/z calcd. for ($\text{C}_{11}\text{H}_9\text{ClO}_2^+$) = 208.0291, found = 208.0293.

2-(Thiophen-3-yloxyethyl) furan (3c)

Isolated as a yellow oil in 76 % yield: $R_f = 0.5$ (3:1 hexanes: toluene); ^1H NMR (CDCl_3 , 300 MHz) δ 7.46 (1H, dd, $J = 2.0, 1.0$ Hz), 7.18 (1H, dd, $J = 5.0, 3.0$ Hz), 6.80 (1H, ddd, $J = 5.0, 1.5, 1.0$ Hz), 6.45 (1H, d, $J = 3.5$ Hz), 6.41 - 6.37 (2H, m), 4.97 (2H, s); ^{13}C NMR (CDCl_3 , 75 MHz) δ 156.89, 150.02, 143.11, 124.74, 119.69, 110.57, 110.24, 98.33, 64.49; IR (film) ν 3117, 2927, 1542, 1503, 1422, 1392, 1359, 1235, 1174, 1150, 1072, 1008, 953, 923, 874, 817, 752, 626 cm^{-1} ; LRMS (EI): $m/z = 180$ (23), 82 (8), 81 (100), 53 (18); HRMS (EI): m/z calcd. for ($\text{C}_{12}\text{H}_{12}\text{O}_3^+$) = 180.0245, found = 180.0245.

2-Styryloxyethyl furan (3d)

Isolated as a clear oil in 61 % yield: $R_f = 0.6$ (9:1 hexanes: EtOAc); ^1H NMR (CDCl_3 , 300 MHz) δ 7.46 (1H, dd, $J = 2.0, 1.0$ Hz), 7.29 - 7.23 (4H, m), 7.18 - 7.06 (1H, m), 7.05 (1H, d, $J = 13.0$ Hz), 6.44 (1H, d, $J = 3.0$ Hz), 6.39 (1H, dd, $J = 3.0, 2.0$ Hz), 5.99 (1H, d, $J = 13.0$ Hz), 4.85 (2H, s); ^{13}C NMR (CDCl_3 , 75 MHz) δ 150.02, 146.99, 143.22, 136.00,

128.57, 125.86, 125.22, 110.55, 110.15, 107.39, 64.28; IR (film) ν 3024, 2924, 1733, 1654, 1637, 1598, 1501, 1448, 1371, 1330, 1276, 1227, 1153, 1072, 1016, 993, 921, 884, 816, 748, 694 cm^{-1} ; LRMS (EI): m/z = 200 (3), 170 (5), 135 (17), 120 (8), 107 (46), 105 (39), 91 (17), 90 (9), 89 (11), 82 (18), 81 (100), 79 (42), 77 (37), 63 (8), 53 (33), 52 (9), 51 (20).

2-Hex-1-enyloxymethyl furan (3e)

Isolated as a clear oil in 55 % yield: R_f = 0.7 (9:1 hexanes: EtOAc); ^1H NMR (CDCl_3 , 300 MHz) δ 7.41 (1H, t, J = 1.5 Hz), 6.35 (2H, d, J = 1.5 Hz), 6.28 (1H, dt, J = 12.5, 1.0 Hz), 4.91 (1H, dt, J = 12.5, 7.5 Hz), 4.64 (2H, s), 1.98 - 1.88 (2H, m), 1.38 - 1.26 (4H, m), 0.94 - 0.86 (3H, m); ^{13}C NMR (CDCl_3 , 75 MHz) δ 150.64, 145.13, 142.89, 110.41, 109.58, 105.76, 63.51, 33.04, 27.68, 22.38, 14.30; IR (film) ν 2958, 2926, 2858, 1672, 1653, 1503, 1456, 1369, 1290, 1226, 1157, 1126, 1080, 1016, 933, 885, 813, 738 cm^{-1} ; LRMS (EI): m/z = 180 (7), 149 (9), 105 (11), 97 (7), 85 (10), 83 (6), 82 (8), 81 (100), 57 (13), 53 (9); HRMS (EI): m/z calcd. for ($\text{C}_{11}\text{H}_{16}\text{O}_2^+$) = 180.1150, found = 180.1154.

phenyl-(3-phenylpropyl) ether (2b)

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

Mercury-300 "mercury300"

Pulse 65.6 degrees

Acq. time 2.276 sec

Width 4492.4 Hz

16 repetitions

OBSERVE H1, 300.0510813 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 0 min, 42 sec

13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

Mercury-300 "mercury300"

Pulse 45.0 degrees

Acq. time 1.000 sec

Width 18797.0 Hz

368 repetitions

OBSERVE C13, 75.4479051 MHz

DECOUPLE H1, 300.0525807 MHz

Power 39 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 54 min, 57 sec

(4-bromobenzyl)phenyl ether (2e)

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
Mercury-300 "mercury300"

Pulse 69.1 degrees
Acq. time 2.276 sec
Width 4492.4 Hz
16 repetitions
OBSERVE H1, 300.0510813 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 0 sec

13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
Mercury-300 "mercury300"

Pulse 48.6 degrees
Acq. time 1.000 sec
Width 18797.0 Hz
684 repetitions
OBSERVE C13, 75.4477451 MHz
DECOUPLE H1, 300.0525807 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 16 min, 22 sec

1,8-diphenoxyocta-3,5-diyne (2j)

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
Mercury-300 "mercury300"

Pulse 65.6 degrees
Acq. time 2.276 sec
Width 4492.4 Hz
16 repetitions
OBSERVE H1, 300.0510813 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 42 sec

13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
Mercury-300 "mercury300"

Pulse 45.0 degrees
Acq. time 1.000 sec
Width 18797.0 Hz
960 repetitions
OBSERVE C13, 75.4478959 MHz
DECOUPLE H1, 300.0525807 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 54 min, 57 sec

(E)-pent-2-enylphenyl ether (2q)**STANDARD 1H OBSERVE**

Pulse Sequence: s2pul
Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
Mercury-300 "mercury300"

Pulse 69.1 degrees
Acq. time 2.276 sec
Width 4492.4 Hz
16 repetitions
OBSERVE H1, 300.0510813 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 42 sec

13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
Mercury-300 "mercury300"

Relax. delay 0.500 sec
Pulse 48.6 degrees
Acq. time 1.000 sec
Width 18797.0 Hz
208 repetitions
OBSERVE C13, 75.4478982 MHz
DECOUPLE H1, 300.0525807 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 25 min, 17 sec

(Z)-pent-2-enylphenyl ether (2r)

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

Mercury-300 "mercury300"

Pulse 69.1 degrees

Acq. time 2.276 sec

Width 4492.4 Hz

16 repetitions

OBSERVE H1, 300.0510813 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 0 min, 42 sec

13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

Mercury-300 "mercury300"

Pulse 48.6 degrees

Acq. time 1.000 sec

Width 18797.0 Hz

488 repetitions

OBSERVE C13, 75.4478973 MHz

DECOUPLE H1, 300.0525807 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 1 hr, 16 min, 22 sec

methyl-(2-phenoxyethyl)carbamic acid *tert*-butyl ester (2u)

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
Mercury-300 "mercury300"

Pulse 65.6 degrees
Acq. time 2.276 sec
Width 4492.4 Hz
16 repetitions
OBSERVE H1, 300.0510813 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 0 sec

13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
Mercury-300 "mercury300"

Pulse 45.0 degrees
Acq. time 1.000 sec
Width 18797.0 Hz
800 repetitions
OBSERVE C13, 75.4479008 MHz
DECOUPLE H1, 300.0525807 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 2 min, 5 sec

(3-phenoxypropyl)carbamic acid benzyl ester (2v)

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
Mercury-300 "mercury300"

Pulse 69.1 degrees
Acq. time 2.276 sec
Width 4492.4 Hz
16 repetitions
OBSERVE H1, 300.0510813 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 42 sec

13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
Mercury-300 "mercury300"

Pulse 48.6 degrees
Acq. time 1.000 sec
Width 18797.0 Hz
800 repetitions
OBSERVE C13, 75.4479002 MHz
DECOUPLE H1, 300.0525807 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 2 min, 5 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

GEMINI-300BB "gemini300"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.998 sec

Width 4500.5 Hz

16 repetitions

OBSERVE H1. 30

OBSERVE HI, 300.073809
DATA PROCESSING

Line broadening 0.2 Hz

Line broaden:
FT size 32768

FT size 32768
Metal time 0 min 53 sec

13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

GEMINI-300BB "gemini300"

Pulse 51.1 degrees

Pulse 51.1 degrees
Acc. time 1.815 sec

Acq. time 1.815
width 18761 7 5

Width 18761.7 E
408 repetitions

408 repetitions
ORIGINAL C13 75 4526003 MY-

OBSERVE C13, 75.4536093 MHz

DECOUPLE H1,

Power 33 dB

continuously on

WALTZ-16 modula

DATA PROCESSING

Line broadening

FT size 131072

(1-methylallyl)phenyl ether (2y)

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
Mercury-300 "mercury300"

Pulse 65.6 degrees
Acq. time 2.276 sec
Width 4492.4 Hz
16 repetitions
OBSERVE H1, 300.0510813 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 0 sec

13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
Mercury-300 "mercury300"

Pulse 45.0 degrees
Acq. time 1.000 sec
Width 18797.0 Hz
688 repetitions
OBSERVE C13, 75.4478968 MHz
DECOUPLE H1, 300.0525807 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 54 min, 57 sec

benzhydrylphenyl ether (2z)

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl₃
 Temp. 25.0 C / 298.1 K
 Mercury-300 "mercury300"

Pulse 65.6 degrees
 Acq. time 2.276 sec
 Width 4492.4 Hz
 16 repetitions
 OBSERVE H1, 300.0510813 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536
 Total time 0 min, 42 sec

O-phenyl stigmasterol (2bb)

13C OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl₃
 Temp. 25.0 C / 298.1 K
 Mercury-300 "mercury300"

Pulse 46.1 degrees
 Acq. time 1.000 sec
 Width 18797.0 Hz
 848 repetitions
 OBSERVE C13, 75.4478956 MHz
 DECOUPLE H1, 300.0525807 MHz
 Power 39 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total time 1 hr, 2 min, 5 sec

2-(4-methoxyphenoxy)methyl furan (3a)

2-(3-chlorophenoxymethyl) furan (3b)

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Mercury-300 "mercury300"

Pulse 65.6 degrees
Acq. time 2.276 sec
Width 4492.4 Hz
16 repetitions
OBSERVE H1, 300.0510813 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 42 sec

13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Mercury-300 "mercury300"

Pulse 46.1 degrees
Acq. time 1.000 sec
Width 18797.0 Hz
352 repetitions
OBSERVE C13, 75.4479016 MHz
DECOUPLE H1, 300.0525807 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 2 min, 5 sec

2-(thiophen-3-yloxymethyl) furan (3c)

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

Mercury-300 "mercury300"

Pulse 65.6 degrees

Acq. time 2.276 sec

Width 4492.4 Hz

16 repetitions

OBSERVE H1, 300.0510813 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 0 min, 0 sec

13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

Mercury-300 "mercury300"

Pulse 45.0 degrees

Acq. time 1.000 sec

Width 18797.0 Hz

624 repetitions

OBSERVE C13, 75.4478994 MHz

DECOUPLE H1, 300.0525807 MHz

Power 39 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 1 hr, 2 min, 5 sec

2-styryloxymethyl furan (3d)

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
Mercury-300 "mercury300"

Relax. delay 5.000 sec
Pulse 83.9 degrees
Acq. time 2.276 sec
Width 4492.4 Hz
16 repetitions
OBSERVE H1, 300.0510813 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min, 2 sec

13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
Mercury-300 "mercury300"

Pulse 46.1 degrees
Acq. time 1.000 sec
Width 18797.0 Hz
272 repetitions
OBSERVE C13, 75.4478994 MHz
DECOUPLE H1, 300.0525807 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 16 min, 22 sec

2-hex-1-enyloxymethyl) furan (3e)

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

Mercury-300 "mercury300"

Pulse 65.6 degrees

Acq. time 2.276 sec

Width 4492.4 Hz

16 repetitions

OBSERVE H1, 300.0510813 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 0 min, 42 sec

13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

Mercury-300 "mercury300"

Pulse 46.1 degrees

Acq. time 1.000 sec

Width 18797.0 Hz

416 repetitions

OBSERVE C13, 75.4478973 MHz

DECOUPLE H1, 300.0525807 MHz

Power 39 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 1 hr, 2 min, 5 sec