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Organosoluble Copper Clusters as Precatalysts for Carbon−Heteroelement Bond-Forming Reactions: Microwave and Conventional Heating

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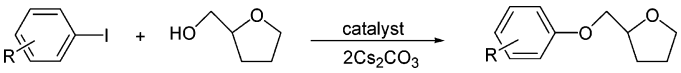

The coupling of aryl iodides with alcohols under mild conditions has been explored using self-assembled octanuclear copper clusters as catalysts. Reactions involving tetrahydrofurfuryl alcohol were typically complete in 4−8 h at 110 °C using an oil bath or 1−3 h with microwave heating. High yields of alkyl aryl ethers were obtained with catalyst loadings as low as 0.4 mol % cluster.

Introduction

The transition-metal-catalyzed formation of carbon−oxygen bonds is an important reaction due to the large number of applications the resulting compounds have in organic, medicinal, and polymer chemistry.[1] While palladium-based catalysts have been successfully used for this transformation,[2] copper catalysts are attractive alternatives due to increased functional group tolerance.[3] One drawback of using copper catalysts is that stoichiometric amounts of the metal are typically required. However, recent reports have shown that catalytic amounts of copper salts, along with several equivalents of a supporting ligand, generate an effective catalyst.[4] The excess ligand has been proposed to coordinate to the copper and prevent the formation of less reactive aggregates.[5]

Microwave-accelerated carbon−carbon and carbon−heteroelement bond-forming reactions have been the subject of an intense amount of research over the past decade.[6] In some cases, reaction times can be dramatically decreased by using microwave irradiation. Several reports have appeared concerning the use of oxygen nucleophiles (phenols and alkoxides) in microwave-accelerated nucleophilic aromatic substitution reactions.[7] To investigate the effectiveness of single component organosoluble copper clusters on the coupling of alcohols with aryl halides, etherification reactions have been carried out using conventional and microwave heating with octanuclear compounds as precatalysts.

$$7\mathrm{CuCl} + 6\mathrm{K[S_2P(OR)_2]} \xrightarrow[[\mathrm{Cu(CH_3CN)_4}]\mathrm{PF_6}]{\mathrm{NMP}} [\mathrm{Cu_8\{S_2P(OR)_2\}_6(\mu_8\text{-}Cl)}]\mathrm{PF_6} \quad (1)$$

$$4[\mathrm{Cu(CH_3CN)_4}]\mathrm{PF_6} + 3\mathrm{K[S_2P(OR)_2]} \xrightarrow[\mathrm{CH_3Cl_2}]{\mathrm{NBu_4Cl}} [\mathrm{Cu_8\{S_2P(OR)_2\}_6(\mu_8\text{-}Cl)}]\mathrm{PF_6} \quad (2)$$

Two different protocols were followed in order to synthesize the Cu_8 clusters (eqs 1 and 2).[8] Although both methods proceeded in moderate yields, the latter (eq 2)

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(1) Nicolaou K. C.; Boddy C. N. C. *J. Am. Chem. Soc.* **2002**, *124*, 10451. (b) Evans, D. A.; Wood, M. R.; Trotter, B. W.; Richardson, T. I.; Barrow, J. C.; Katz, J. L. *Angew. Chem., Int. Ed.* **1998**, *37*, 2700. (c) Evans, D. A.; Dinsmore, C. J.; Watson, P. S.; Wood, M. R.; Richardson, T. I.; Trotter, B. W.; Katz, J. L. *Angew. Chem., Int. Ed.* **1998**, *37*, 2704. (d) Wipf, P.; Lynch, S. M. *Org. Lett.* **2003**, *5*, 1155.

(2) Katoka, N.; Shelby, Q.; Stambuli, J. P.; Hartwig, J. F. *J. Org. Chem.* **2002**, *67*, 5553. (b) Mann, G.; Shelby, Q.; Roy, A. H.; Hartwig, J. F. *Organometallics* **2003**, *20*, 2775. (c) Torraca, K. E.; Huang, X.; Parrish, C. A.; Buchwald, S. L. *J. Am. Chem. Soc.* **2001**, *123*, 10770. (d) Aranyos, A.; Old, D. W.; Kiyomori, A.; Wolfe, J. P.; Sadighi, J. P.; Buchwald, S. L. *J. Am. Chem. Soc.* **1999**, *121*, 4369.

(3) an der Heiden, M. R.; Frey, G. D.; Plenio, H. *Organometallics* **2004**, *23*, 3548.

(4) (a) Wolter, M.; Nordmann, G.; Job, G. E.; Buchwald, S. L. *Org. Lett.* **2002**, *4*, 973. (b) Cristau, H. J.; Celliier, P. P.; Hamada, S.; Sindler, J. F.; Taillefer, M. *Org. Lett.* **2004**, *6*, 913. (c) Gujadhur, R. K.; Bates, C. M.; Venkataraman, D. *Org. Lett.* **2001**, *3*, 4315. (d) Gujadhur, R. K.; Venkataraman, D. *Org. Lett.* **2001**, *31*, 4315. (e) Quach, T. D.; Batey, R. A. *Org. Lett.* **2003**, *5*, 1381.

(5) Kiyomori, A.; Marcoux, J. F.; Buchwald, S. L. *Tetrahedron Lett.* **1999**, *40*, 2657.

(6) For recent reviews see: (a) Mavandadi, F.; Lidstrom, P. *Curr. Top. Med. Chem.* **2004**, *4*, 773. (b) Larhed, M.; Moberg, C.; Hallberg, A. *Acc. Chem. Res.* **2002**, *35*, 717. (c) Lidström, P.; Tierney, J.; Wathey, B.; Westman, J. *Tetrahedron* **2001**, *47*, 9225.

(7) (a) Chaouchi, M.; Loupy, A.; Marque, S.; Petet, A. *Eur. J. Org. Chem.* **2002**, 1278. (b) Feng, L.; Wang, Q.; Ding, Z.; Tao, F. *Org. Lett.* **2003**, *5*, 2169.

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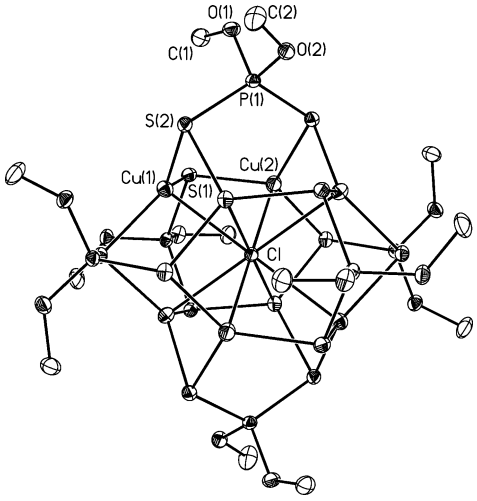
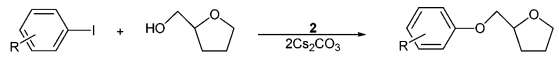


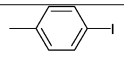
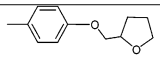
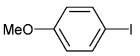
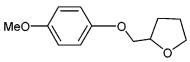
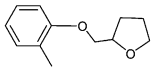
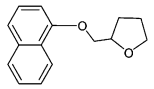
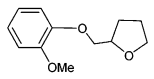
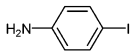
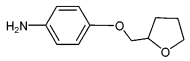
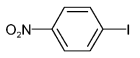
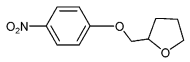
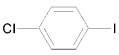
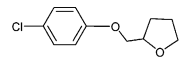
FIGURE 1. Molecular structure of the cation of **1** with thermal ellipsoids displayed at 50% probability. Selected bond lengths (Å) and angles (deg): Cu(1)−S(l) = 2.2737(5), Cu(1)−S(2) = 2.2791(5), Cu(1)−Cl = 2.6840(2), Cu(2)−Cl = 2.6769(4), S(1)−Cu(1)−S(2) = 121.319(18), S(1)−Cu(1)−Cl = 94.985(13), S(2)−Cu(1)−Cl = 94.467(13).

was attractive since it avoided the use of NMP as the solvent. These robust solids are stable in light and air for extended periods of time and exhibit a single resonance in the ^{31}P NMR spectrum. Additionally, the core remains intact in solution as evidenced by electrospray-MS.[9] The molecular structure of **1** was determined by single-crystal X-ray diffraction and is shown in Figure 1. These and related cubes have been the subject of several structural and theoretical investigations due to the presence of a halogen, sulfur, or selenium atom that is simultaneously bridging eight copper atoms.[9,10] Aside from the interesting structural characteristics, these complexes have great potential as catalysts due to the possibility that adjacent metals could act cooperatively.

Tetrahydrofurfuryl aryl ethers are an important class of compounds used in the synthesis of biologically active pharmaceuticals.[11] To devise a simple synthesis for these compounds, the coupling of aryl iodides with racemic tetrahydrofurfuryl alcohol was carried out (Table 1). After screening several solvents and reagent ratios, best results were obtained using 0.20 g (0.75−0.92 mmol) of the aryl iodide in neat alcohol (1.5 mL) with **2** as the catalyst (4.1 μmol) and Cs_2CO_3 (2 equiv) as the base. The efficacy of different inorganic bases was investigated, and Cs_2CO_3 was found to be superior. Most reactions proceeded smoothly at 110 °C (4−8 h) using conventional heating (oil bath). Moisture must be excluded from these reactions since significant amounts of phenols and diaryl ethers were formed when wet tetrahydrofurfuryl alcohol was used. When needed, powdered molecular sieves were added to guard against adventitious water and from moisture generated due to the decomposition of $HCsCO_3$ (entries 3−5 were most sensitive).[12] The presence of oxygen (atmosphere) slowed but did not quench the coupling chemistry, although reduced yields of the alkyl aryl ethers were obtained when reactions were carried out under an atmosphere of air. A significant advantage of this system was that, in some cases, pure alkyl aryl ether was obtained by a simple filtration and removal of the volatiles (no chromatography). The coupling of ortho-substituted and naphthyl iodides is often problematic; however, high yields of the corresponding alkyl aryl ethers were obtained using **2** as the precatalyst (entries 3−5), although longer reaction times were needed. Functionalized aryl iodides were also readily employed (entries 2, 5−8). The aryl iodide, 4-nitroiodobenzene, gave the lowest yield of the desired ether due to the formation of 4-tetrahydrofurfuryloxyaniline (40%).

TABLE 1. Tetrahydrofurfuryl Ether Formation Using Microwave and Conventional Heating



entry	aryl halide	product	isolated yield[a]
1			T: 91 (85) M: 88 (85)
2			T: 84 (78) M: 78 (73)
3			T: 90 (84) M: 84 (80)
4			T: 93 (90) M: 93 (87)
5			T: 88 (80) M: 77 (63)
6			T: 74 (68) M: 68 (63)
7			T: 55 (60)[b] M: 0 (0)[b]
8			T: 79 (73) M: 76 (73)

[a] T = reactions carried out using conventional heating; M = microwave-irradiated reactions. Yields in parentheses refer to reactions carried out in air. [b] GC yields.

Microwave irradiation is an efficient and convenient way to accelerate transition-metal-catalyzed organic transformations.[13] To investigate whether this technology could be extended to the coupling chemistry, reactions were carried out in a focused microwave reactor. Generally, the reactions were faster in the microwave (1−3 h),

(8) The second protocol in Sheme 1 was adapted from: Liu, C. W.; Stubbs, T.; Staples, R. J.; Fackler, J. P. Jr. *J. Am. Chem. Soc.* **1995**, *117*, 9778.

(9) Electrospray MS of an acetonitrile solution of **2** gives *m/z* =1821 which corresponds to the cation of **2**. Other reports of mass spectral data for related compounds include: ref 8 and Liu, C. W.; Hung, C.-M.; Santra, B. K.; Chen, H.-C.; Hsueh, H.-H. Wang, J.-C. *Inorg. Chem.* **2003**, *42*, 3216.

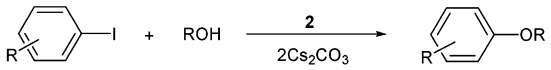
(10) (a) Liu, C. W.; Hung, C.-M.; Santra, B. K.; Wang, J.-C.; Kao, H.-M.; Lin, Z. *Inorg. Chem.* **2003**, *42*, 8551. (b) Garland, M. T.; Halet, J.-F.; Saillard, J.-Y. *Inorg. Chem.* **2001**, *40*, 3342. (c) Matsumoto, K.; Tanaka, R.; Shimomura, R.; Nakao, Y. *Inorg. Chim. Acta* **2000**, *304*, 293.

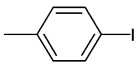
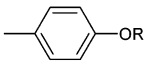
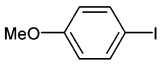
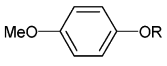
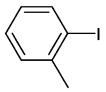
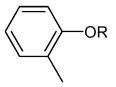
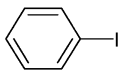
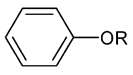
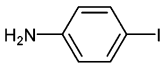
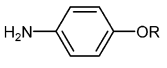
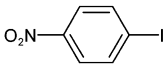
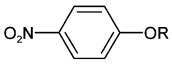
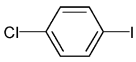
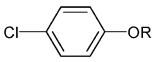
(11) Culshaw, A. J.; Dziadulewicz, E. K.; Hallett, A.; Hart, T. W. WO 2004033435, **2004**; *Chem. Abstr.* **2004**, 333702.

(12) Greiner, A. *Synthesis* **1989**, 312.

(13) *Microwaves in Organic Synthesis;* Loupy, A., Ed.; Wiley-VCH: Weinheim, Germany, 2003.

TABLE 2. Coupling of Aryl Iodides with Simple Alcohols Using Conventional and Microwave Heating[a]



entry	aryl halide	product	isolated yield
1			T: 1-Et: 80 (71) M: 1-Et: 75 (70) T: 1-Pr: 87 (80) M: 1-Pr: 83 (72)
2			T: 1-hex: 84 (82) M: 1-hex: 87 (79) T: 1-Pr: 92 (85) M:1-Pr: 78 (75)
3			T: 1-hex: 84 (78) M: 1-hex: 78 (72) T: 1-Pr: 81 (74) M:1-Pr: 78 (71)
4			T: 1-Bu: 95 (81) M:1-Bu: 88 (74) T: 1-Pr 90 (82) M:1-Pr 85 (75)
5			T: 1-hept: 80 (69) M: 1-hept: 74 (69) T: 1-Pr: 73 (55) M:1-Pr: 69 (65)
6			T: 1-hex: 60 (52)[b] M: 1-hex: 40 (20)[b] T: 1-hept: 63 (40)[b] M:1-hept: 32 (25)[b]
7			T: 1-hex: 89 (84) M: 1-hex: 84 (79) T: 1-Pr: 84 (77) M:1-Pr: 77 (70)

[a] T = reactions carried out using conventional heating; M = microwave-irradiated reactions. Yields in parentheses refer to reactions carried out in air. [b] GC yields.

but slightly lower yielding (Table 1) due to an increase in the amount of the dehalogenation products. When high microwave power (300 W) was used for short reaction times (5 min), complete dehalogenation of the iodoarene was observed. Additionally, when 4-nitroiodobenzene was used as the substrate, 4-tetrahydrofurfuryloxyaniline was the sole product. This is in contrast to the conventionally heated reactions where a mixture of the desired ether and the aniline was obtained. Wang has recently reported that microwave irradiation was able to promote the coupling of phenols with aryl halides to generate diaryl ethers in the absence of a catalyst.[14] To test whether an uncatalyzed microwave-induced process was occurring, control reactions were carried out in the absence of **2**. Under the same reaction conditions, only small amounts of alkyl aryl ethers were observed (4−8%) when the catalyst was removed. The rapid rates of the microwave-assisted reactions was attributed to core heating of the reaction mixture.

The coupling of simple alcohols and aryl iodides was investigated using **2** as the single component catalyst (Table 2). Similar to reactions summarized in Table 1, Cs_2CO_3 was found to be the most effective inorganic base. Reactions involving the alcohols listed in Table 2 were typically slower than with tetrahydrofurfuryl alcohol, but high yields were obtained in most cases. Although the reactions were moderately stable to moisture and oxygen, best results were obtained when the oxygen and moisture were removed. Most reactions carried out under an atmosphere of nitrogen were typically complete within 11 h (110 °C), whereas analogous reactions carried out under air required 36 h (110 °C) to reach completion. Reactions carried out in the microwave were typically complete in 1−2 h under nitrogen and 2−4 h under an atmosphere of air. Reactions involving ortho-substituted aryl iodides were successful although longer reaction times were needed. The precise identity of the active catalyst has not been determined in these reactions. Analysis of the crude reaction mixture by ^{31}P NMR revealed a single peak in the ^{31}P NMR spectrum at δ 92.5 ppm, which suggested the presence of a bridged $S_2P(O^iPr)_2^-$ ligand.

In summary, the coupling of aryl iodides with tetrahydrofurfuryl alcohol as well as simple alcohols has been accomplished using a single component octanuclear copper cluster. The clusters are attractive catalysts due to their ability to manipulate the steric and electronic nature of the ligand architecture by simply changing the alkyl or aryl group on the $S_2P(OR)_2^-$ bridging group. In contrast to other copper-based systems, the supporting ligand/Cu ratio in **1** and **2** is less than 1. Similar yields of the alkyl aryl ethers were obtained using conventional and microwave heating. Functionalized and halogen-containing aryl iodides were successfully coupled using the catalyst system. In some cases, pure alkyl aryl ether was obtained by a simple filtration and drying. Work is currently underway to determine the effectiveness of these clusters in other carbon−heteroelement bond forming reactions.

Experimental Section

The synthesis of the copper clusters was carried out under nitrogen using standard Schlenk techniques. Coupling reactions were carried out under an atmosphere of nitrogen or air. The $K[S_2P(OR)_2]$ salts used in this study were prepared by treatment of P_4S_{10} with the appropriate alcohol followed by addition of 1 equiv of KOH.[15] All yields are based upon isolated material with greater than 95% purity as determined by ^1H NMR spectroscopy and GC unless specified otherwise. Spectroscopic data of known compounds were compared with literature values or authentic samples. The connectivity of the compounds was established using $^1H-^1H$ (COSY) and $^1H-^{13}C$ (HETCOR) experiments.

Synthesis of $[Cu_8(S_2P(OMe)_2)_6(\mu_8\text{-}Cl)][PF_6]$, 1: Method 1. A round-bottomed flask (100 mL) was charged with CuCl (0.30 g, 3.0 mmol), $K[S_2P(OMe)_2]$ (0.51 g, 2.6 mmol), $[Cu(CH_3CN)_4][PF_6]$ (0.16 g, 0.43 mmol), and NMP (50 mL). The solution was stirred for 20 h at 25 °C. The reaction mixture was diluted with CH_2Cl_2 (200 mL), extracted with water (500 mL × 3), filtered, and dried over $MgSO_4$. The solution was concentrated under vacuum, layered with hexane, and placed in the refrigerator (5 °C, 48 h). Colorless cuboids formed and were separated by filtration (0.42 g, 60%). Anal. Calcd for $C_{12}H_{36}ClCu_8F_6O_{12}P_7S_{12}$: C, 8.83; H, 2.21. Found: C, 9.03; H, 2.19. ^1H NMR ($CDCl_3$, 25 °C): δ 3.90 (d, 36H, J = 14.2 Hz, −O*Me*).

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(15) Chen, H. W.; Paparizos, C.; Fackler, J. P., Jr. *Inorg. Chim. Acta* **1985**, *96*, 137.

$^{13}C\{^1H\}$ NMR ($CDCl_3$, 25 °C): δ 77.1 (d, J = 11.3 Hz, −O*Me*). $^{31}P\{^1H\}$ NMR ($CDCl_3$, 25 °C): δ 91.5 (s).

Method 2. A round-bottomed flask was charged with $K[S_2P(OMe)_2]$ (0.39 g, 2.0 mmol), $[Cu(CH_3CN)_4][PF_6]$ (1.0 g, 2.7 mmol), NBu_4Cl (0.19 g, 0.68 mmol), and CH_2Cl_2 (50 mL). The solution was stirred for 20 h at 25 °C. The reaction mixture was diluted with CH_2Cl_2 (200 mL) and extracted with water (500 mL × 3), and dried over $MgSO_4$. The CH_2Cl_2 solution was filtered, concentrated, layered with hexane, and placed in the refrigerator (5 °C) overnight. Colorless cuboids formed and were separated by filtration (0.31 g, 57%).

Synthesis of $[Cu_8(S_2P(O^iPr)_2)_6(\mu_8\text{-}Cl)][PF_6]$, 2. Method 1. A round-bottomed flask (100 mL) was charged with CuCl (0.30 g, 3.0 mmol), $K[S_2P(O^iPr)_2]$ (0.65 g, 2.6 mmol), $[Cu(CH_3CN)_4][PF_6]$ (0.16 g, 0.43 mmol), and NMP (50 mL). The solution was stirred for 20 h at 25 °C. The reaction mixture was diluted with CH_2Cl_2 (200 mL), extracted with water (500 mL × 3), filtered, and dried over $MgSO_4$. The solution was concentrated under vacuum, layered with hexane, and placed in the refrigerator (5 °C, 48 h). Colorless cuboids formed and were separated by filtration (0.61 g, 72%). Anal. Calcd for $C_{36}H_{84}ClCu_8F_6O_{12}P_7S_{12}$: C, 21.96; H, 4.27. Found: C, 22.13; H, 4.24. ^1H NMR ($CDCl_3$, 25 °C): δ 4.86 (dsept, 6H, −C*H*Me$_2$), 1.42 (d, 36H, J = 6.2 Hz, −CH*Me*$_2$). $^{13}C\{^1H\}$ NMR ($CDCl_3$, 25 °C): δ 76.8 (d, J = 10.7 Hz, −*C*HMe$_2$), 24.0 (br s, −*C*H$_3$). $^{31}P\{^1H\}$ NMR ($CDCl_3$, 25 °C): δ 91.3 (s).

Method 2. A round-bottomed flask was charged with $K[S_2P(O^iPr)_2]$ (0.51 g, 2.0 mmol), $[Cu(CH_3CN)_4][PF_6]$ (1.0 g, 2.7 mmol), NBu_4Cl (0.19 g, 0.68 mmol), and CH_2Cl_2 (50 mL). The solution was stirred for 20 h at 25 °C. The reaction mixture was diluted with CH_2Cl_2 (200 mL) and extracted with water (500 mL × 3). The CH_2Cl_2 solution was dried over $MgSO_4$, layered with hexane, and placed in the refrigerator (5 °C) overnight. Colorless cuboids formed and were separated by filtration (0.43 g, 65%).

Synthesis of Aryl Ethers. Method A: Conventional Heating. A test tube was charged (in air) with $[Cu_8(S_2P(O^iPr)_2)_6(\mu_8\text{-}Cl)][PF_6]$ (0.008 g, 4.1 μmol), aryl halide (1.0 equiv), Cs_2CO_3 (2.0 equiv), alcohol (1.5 mL), and a magnetic stirring bar. Where appropriate, the test tube was evacuated and refilled three times with N_2. Molecular sieves were added to degassed samples. The test tube was capped with a septum (secured with copper wire) and placed in an oil bath at 110 °C. The reaction was monitored by gas chromatography until the aryl halide was consumed (5−36 h). The test tube was cooled to room temperature and filtered through a plug of silica. The volatiles were removed under vacuum, and the resulting residue was purified by column chromatography. In several cases the alkyl aryl ether was isolated (>95 % pure) by simply removing the volatiles, extracting the residue with hexane or ether, and filtering though a short column of silica gel (1 × 1 cm: no chromatography). ***Caution:*** *for reactions involving low boiling alcohols, a significant amount of pressure builds up in the test tube. Cooling the flask to room temperature before opening the flask or cutting the copper wire is highly recommended.*

Synthesis of Alkyl Aryl Ethers. Method B: Microwave Heating. A 10 mL reactor tube was charged (in air) with $[Cu_8(S_2P(O^iPr)_2)_6(\mu_8\text{-}Cl)][PF_6]$ (0.008 g, 4.1 μmol), aryl halide (1.0 equiv), Cs_2CO_3 (2.0 equiv), alcohol (1.5 mL), and magnetic stirring bar. The reactor tube was crimped closed, evacuated and refilled with nitrogen (when appropriate), placed in the microwave reactor, and connected to the monitoring system. The reaction was heated to the desired temperature using the appropriate microwave power for 1−4 h. For reactions involving tetrahydrofurfuryl alcohol, the maximum power setting (50 W) was maintained until the desired temperature was reached. For the remainder of the experiments involving tetrahydrofurfuryl alcohol, a power level of 9 W (pressure = 19 psi) was used to maintain the temperature. In rare cases a trace of the aryl halide remained, and the reactor vial was placed in the microwave reactor for an additional 0.25−0.50 h. The vessel was cooled to room temperature, filtered through a small plug of silica gel, and dried under vacuum. The aryl ether was then purified by column chromatography. In several cases, the alkyl aryl ether was isolated (>95% pure) by simply removing the volatiles, extracting the residue with hexane or ether, and filtering though a short plug of silica gel (1 × 1 cm: no chromatography). *Caution: for reactions involving low boiling alcohols, a significant amount of pressure builds up in the microwave reactor vial. Cooling the cooling the vial to room temperature before opening the flask is highly recommended.*

Preparation of 4-tetrahydrofurfuryloxytoluene (Table 1 entry 1): Method A. The general procedure was followed using **2** (0.008 g, 4.1 μmol), 4-iodotoluene (0.20 g, 0.92 mmol), crushed molecular sieves (0.5 g), and Cs_2CO_3 (0.60 g, 1.84 mmol). The test tube was evacuated and refilled with nitrogen before addition of the tetrahydrofurfuryl alcohol (1.50 mL, 15.5 mmol). The reaction mixture was stirred at 110 °C for 4 h, cooled to room temperature, and filtered through a short plug of silica gel eluting with ethyl acetate. The excess tetrahydrofurfuryl alcohol was removed under vacuum with gentle heating. Purification of the residue by column chromatography (silica: hexane/ethyl acetate 3:1) afforded 0.16 g (91%) of the title compound as a dull yellow oil. Anal. Calcd for $C_{12}H_{16}O_2$: C, 74.97; H, 8.39. Found: C, 74.79; H, 8.41. ^1H NMR ($CDCl_3$, 25 °C): δ 7.09 (d, 2H, J = 8.4 Hz, −C$_6$*H*$_4$Me), 6.85 (d, 2H, J = 8.4 Hz, −C$_6$*H*$_4$Me), 4.28 (m, 1H, −C*H*−), 3.95 (m, 3H, −C*H*$_2$−), 3.86 (m, 1H, −C*H*$_2$−), 2.30 (s, 3H, −C$_6$H$_4$*Me*), 2.09 (m, 1H, −C*H*$_2$−), 1.95 (m, 2H, −C*H*$_2$−), 1.78 (m, 1H, −C*H*$_2$−). $^{13}C\{^1H\}$ NMR ($CDCl_3$, 25 °C): δ 156.7 (s, quat), 129.8 (s, quat), 129.7 (s, −*C*$_6$H$_4$Me), 114.3 (s, *C*$_6$H$_4$Me), 77.00 (s, −*C*H−), 70.4 (s, −*C*H$_2$−), 68.4 (s, −*C*H$_2$−), 28.1 (s, −*C*H$_2$−), 25.6 (s, −*C*H$_2$−), 20.3 (s, −C$_6$H$_4$*Me*).

The above procedure was repeated in the presence of air without deoxygenating the reagents or solvent. Molecular sieves were not added to this reaction. The reaction was stirred at 110 °C for 9 h, and the title compound was isolated in 85% yield (0.15 g).

Preparation of 4-Tetrahydrofurfuryloxytoluene (Table 1, Entry 1): Method B. The general procedure was followed using **2** (0.008 g, 4.1 μmol), 4-iodotoluene (0.20 g, 0.92 mmol), crushed molecular sieves (0.5 g), and Cs_2CO_3 (0.60 g, 1.84 mmol). The reactor vial was evacuated and refilled with nitrogen before addition of the tetrahydrofurfuryl alcohol (1.50 mL, 15.5 mmol). The microwave settings were as follows: temperature = 125 °C, maximum microwave power = 50 W, maximum pressure = 300 psi, and reaction time = 2 h. The reaction mixture was cooled to room temperature, filtered through a small plug of silica gel, and dried under vacuum with gentle heating. Purification by column chromatography (silica gel hexane/ethyl acetate 3:1) afforded 0.155 g (88%) of the title compound as a dull yellow oil.

The above procedure was repeated in the presence of air without deoxygenating the reagents or solvent. Molecular sieves were not added to this reaction. The microwave power and pressure settings were the same in these experiments, but the reaction time was increased to 3 h. The title compound was isolated in 85% yield (0.15 g).

Preparation of 4-Tetrahydrofurfuryloxyanisole (Table 1, Entry 2): Method A. The general procedure was followed using **2** (0.008 g, 4.1 μmol), 4-iodoanisole (0.20 g, 0.85 mmol), and Cs_2CO_3 (0.56 g, 1.7 mmol). The test tube was evacuated and refilled with nitrogen before addition of the tetrahydrofurfuryl alcohol (1.50 mL, 15.5 mmol). The reaction mixture was stirred at 110 °C for 4 h, cooled to room temperature, and filtered through a short plug of silica gel eluting with ethyl acetate. The excess tetrahydrofurfuryl alcohol was removed under vacuum with gentle heating. Purification of the residue by column chromatography (silica gel hexane/ethyl acetate 2:1) afforded 0.15 g (84%) of the title compound as a pale brown oil. Anal. Calcd for $C_{12}H_{16}O_3$: C, 69.23; H, 7.69. Found: C, 68.99; H, 7.92. ^1H NMR ($CDCl_3$, 25 °C): δ 6.85 (m, 4H, −C$_6$*H*$_4$OMe), 4.26 (m, 1H, −C*H*−), 3.92 (m, 3H, −C*H*$_2$−), 3.83 (m,

1H, $-CH_2-$), 3.77 (s, 3H, $-OMe$), 2.13−1.89 (m, 3H, $-CH_2-$), 1.76 (m, 1H, $-CH_2-$). $^{13}C\{^1H\}$ NMR ($CDCl_3$, 25 °C): δ 153.6 (s, 1C, quat), 152.8 (s, 1C, quat), 115.2 (s, 2C, $-C_6H_4OMe$), 114.3 (s, 2C, $-C_6H_4OMe$), 76.9 (s, 1C, $-CH-$), 70.8 (s, 1C, $-CH_2-$), 68.3 (s, 1C, $-CH_2-$), 55.4 (s, 1C, $-OMe$), 27.9 (s, 1C, $-CH_2-$), 25.4 (s, 1C, $-CH_2-$).

The above procedure was repeated in the presence of air without deoxygenating the reagents or solvent. Molecular sieves were not added to this reaction. The reaction mixture was stirred at 110 °C for 8 h, and the title compound was isolated in 78% yield (0.14 g).

Preparation of 4-Tetrahydrofurfuryloxyanisole (Table 1, Entry 2): Method B. The general procedure was followed using **2** (0.008 g, 4.1 μmol), 4-iodoanisole (0.20 g, 0.85 mmol), molecular sieves (0.5 g), and Cs_2CO_3 (0.56 g, 1.7 mmol). The reactor vial was evacuated and refilled with nitrogen before addition of the tetrahydrofurfuryl alcohol (1.50 mL, 15.5 mmol). The microwave settings were as follows: temperature = 125 °C, maximum microwave power = 50 W, maximum pressure = 300 psi, and reaction time = 3 h. The reaction mixture was cooled to room temperature, filtered through a small plug of silica gel, and dried under vacuum with gentle heating. Purification by column chromatography (silica gel hexane/ethyl acetate 2:1) afforded 0.14 g (78%) of the title compound as a pale brown oil.

The above procedure was repeated in the presence of air without deoxygenating the reagents or solvent. Molecular sieves were not added to this reaction. The microwave power and pressure settings were the same in these experiments, but the reaction time was increased to 4 h. The title compound was isolated in 73% yield (0.13 g).

Preparation of 2-Tetrahydrofurfuryloxytoluene (Table 1, Entry 3): Method A. The general procedure was followed using **2** (0.008 g, 4.1 μmol), 2-iodotoluene (0.11 mL, 0.86 mmol), molecular sieves (0.5 g), and Cs_2CO_3 (0.56 g, 1.7 mmol). The reaction flask was evacuated and refilled with nitrogen before addition of tetrahydrofurfuryl alcohol (1.5 mL, 15.5 mmol). After being stirred at 100 °C for 10 h, the reaction mixture was transferred to a round-bottom flask, and the excess alcohol was removed under vacuum with gentle heating. The residue was extracted with ethyl acetate, filtered through a short column of silica gel (1 cm), and purified by column chromatography (silica hexane/ethyl acetate 2:1) to afford 0.15 g (90%) of the title compound as a dull yellow oil. Anal. Calcd for $C_{12}H_{16}O_2$: C, 74.97; H, 8.39. Found: C, 75.12; H, 8.22. ^1H NMR ($CDCl_3$, 25 °C): δ 7.20 (m, 2H, $-C_6H_4Me$), 6.91 (t, 1H, J = 7.4 Hz, $-C_6H_4Me$), 6.86 (d, 1H, J = 8.4 Hz, $-C_6H_4Me$), 4.37 (m, 1H, $-CH-$), 4.02 (m, 3H, $-CH_2-$), 3.90 (m, 1H, $-CH_2-$), 2.32 (s, 3H, $-C_6H_4Me$), 2.20−1.85 (m, 4H, $-CH_2-$). $^{13}C\{^1H\}$ NMR ($CDCl_3$, 25 °C): δ 156.9 (s, 1C, quat), 130.5 (s, 1C, $-C_6H_4Me$), 126.7 (s, 1C, quat), 126.6 (s, 1C, $-C_6H_4Me$), 120.3 (s, 1C, $-C_6H_4Me$), 110.8 (s, 1C, $-C_6H_4Me$), 77.0 (s, 1C, $-CH-$), 70.3 (s, 1C, $-CH_2-$), 68.5 (s, 1C, $-CH_2-$), 28.1 (s, 1C, $-CH_2-$), 25.7 (s, 1C, $-CH_2-$), 16.1 (s, 1C, $-C_6H_4Me$).

The above procedure was repeated in the presence of air without deoxygenating the reagents or solvent. Molecular sieves were not added to this reaction. The reaction was stirred at 110 °C for 24 h, and the title compound was isolated in 84% yield (0.14 g).

Preparation of 2-Tetrahydrofurfuryloxytoluene (Table 1, Entry 3): Method B. The general procedure was followed using **2** (0.008 g, 4.1 μmol), 2-iodotoluene (0.11 mL, 0.86 mmol), molecular sieves (0.5 g), and Cs_2CO_3 (0.56 g, 1.7 mmol). The reaction flask was evacuated and refilled with nitrogen before the addition of tetrahydrofurfuryl alcohol (1.5 mL, 15.5 mmol). The microwave settings were as follows: temperature = 125 °C, maximum microwave power = 50 W, maximum pressure = 300 psi, and reaction time = 5 h. The reaction mixture was cooled to room temperature, filtered through a short plug of silica, and dried under vacuum with gentle heating. The residue was purified by column chromatography (silica: hexane/ethyl acetate 2:1), affording 0.14 g (84%) of the title compound as a dull yellow oil.

The above procedure was repeated in the presence of air without deoxygenating the reagents or solvent. Molecular sieves were not added to this reaction. The reaction was irradiated for 7 h with the same microwave settings as above, and the title compound was isolated in 80% yield (0.133 g) yield.

Preparation of 1-Tetrahydrofurfuryloxynaphthalene (Table 1, Entry 4): Method A. The general procedure was followed using **2** (0.008 g, 4.1 μmol), 1-iodonaphthalene (0.11 mL, 0.75 mmol), and Cs_2CO_3 (0.50 g, 1.5 mmol). The test tube was evacuated and refilled with nitrogen, and tetrahydrofurfuryl alcohol (1.50 mL, 15.5 mmol) was added by syringe. The reaction mixture was stirred at 110 °C for 4 h, cooled to room temperature, and filtered through a short plug of silica gel eluting with ethyl acetate. The excess alcohol was removed under vacuum with gentle heating. Purification of the residue by column chromatography (silica gel: hexane/ethyl acetate 2:1) afforded 0.16 g (93%) of the title compound as a pale brown oil. Anal. Calcd for $C_{15}H_{16}O_2$: C, 78.92; H, 7.06. Found: C, 78.88; H, 7.10. ^1H NMR ($CDCl_3$, 25 °C): δ 8.18 (m, 1H, $-OC_{10}H_7$), 7.64 (m, 1H, $-OC_{10}H_7$), 7.37−7.19 (m, 4H, $-OC_{10}H_7$), 6.64 (d, 1H, J = 7.5, $-OC_{10}H_7$), 4.28 (m, 1H, $-CH-$), 3.95 (m, 2H, $-CH_2-$), 3.84 (m, 1H, $-CH_2-$), 3.72 (m, 1H, $-CH_2-$), 2.00−1.70 (m, 4H, $-CH_2-$). $^{13}C\{^1H\}$ NMR ($CDCl_3$, 25 °C): δ 154.4 (s, 1C, quat), 134.3 (s, 1C, quat), 127.2 (s, 1C, $-C_{10}H_7$), 126.1 (s, 1C, $-C_{10}H_7$), 125.6 (s, 1C, $-C_{10}H_7$), 125.4 (s, quat), 124.9 (s, 1C, $-C_{10}H_7$), 121.9 (s, 1C, $-C_{10}H_7$), 120.1 (s, 1C, $-C_{10}H_7$), 104.5 (s, 1C, $-C_{10}H_7$), 76.8 (s, 1C, $-CH-$), 70.3 (s, 1C, $-CH_2-$), 68.4 (s, 1C, $-CH_2-$), 28.1 (s, 1C, $-CH_2-$), 25.6 (s, 1C, $-CH_2-$).

The above procedure was repeated in the presence of air without deoxygenating the reagents or solvent. Molecular sieves were not added to this reaction. The reaction was stirred at at 110 °C for 5 h, and the title compound was isolated in 90% yield (0.155 g).

Preparation of 1-Tetrahydrofurfuryloxynaphthalene (Table 1, Entry 4): Method B. The general procedure was followed using **2** (0.008 g, 4.1 μmol), 1-iodonaphthalene (0.11 mL, 0.75 mmol), and Cs_2CO_3 (0.50 g, 1.5 mmol). The test tube was evacuated and refilled with nitrogen, and tetrahydrofurfuryl alcohol (1.50 mL, 15.5 mmol) was added by syringe. The microwave settings were as follows: temperature = 125 °C, maximum microwave power = 50 W, maximum pressure = 300 psi, and reaction time = 1 h. The reaction mixture was cooled to room temperature, filtered through a small plug of silica gel, and dried under vacuum with gentle heating. Purification by column chromatography (silica: hexane/ethyl acetate 2:1) afforded 0.16 g (93%) of the title compound as a pale brown oil.

The above procedure was repeated in the presence of air without deoxygenating the reagents or solvent. Molecular sieves were not added to this reaction. The reaction was irradiated for 2 h with the same microwave settings as above, and the title compound was isolated in 87% yield (0.15 g).

Preparation of 2-Tetrahydrofurfuryloxyanisole (Table 1, Entry 5): Method A. The general procedure was followed using **2** (0.008 g, 4.1 μmol), 2-iodoanisole (0.11 g, 0.85 mmol), crushed molecular sieves (0.5 g), and Cs_2CO_3 (0.55 g, 1.7 mmol). The test tube was evacuated and refilled with nitrogen before addition of the tetrahydrofurfuryl alcohol (1.50 mL, 15.5 mmol). The reaction mixture was stirred at 110 °C for 15 h, cooled to room temperature, and filtered through a short plug of silica gel eluting with ethyl acetate. The excess tetrahydrofurfuryl alcohol was removed under vacuum with gentle heating. Purification of the residue by column chromatography (silica: hexane/ethyl acetate 2:1) afforded 0.155 g (88%) of the title compound as a dull yellow oil. Anal. Calcd for $C_{12}H_{16}O_3$: C, 69.23; H, 7.69. Found: C, 69.05; H, 7.91. ^1H NMR ($CDCl_3$, 25 °C): δ 6.89 (m, 4H, $-C_6H_4OMe$), 4.31 (m, 1H, $-CH-$), 3.97 (m, 3H, $-CH_2-$), 3.83 (s, 3H, $-C_6H_4OMe$), 3.81 (m, 1H,

−CH_2−), 2.05 (m, 1H, −CH_2−), 1.91 (m, 2H, −CH_2−), 1.80 (m, 1H, −CH_2−). $^{13}C\{^1H\}$ NMR ($CDCl_3$, 25 °C): δ 149.7 (s, quat), 148.6 (s, quat), 121.5 (s, 1C, −C_6H_4OMe), 120.9 (s, 1C, −C_6H_4OMe), 113.9 (s, 1C, −C_6H_4OMe), 112.1 (s, 1C, −C_6H_4OMe), 77.1 (s, 1C, −CH−), 71.7 (m, 1C, −CH$_2$−), 68.6 (s, 1C, −CH$_2$−), 56.0 (s, 1C, −C_6H_4O*Me*), 28.5 (s, 1C, −CH$_2$−), 25.7 (s, 1C, −CH$_2$).

The above procedure was repeated in the presence of air without deoxygenating the reagents or solvent. Molecular sieves were not added to this reaction. The reaction was stirred at 110 °C for 48 h, and the title compound was isolated in 80% yield (0.14 g).

Preparation of 2-Tetrahydrofurfuryloxyanisole Ether (Table 1, Entry 5): Method B. The general procedure was followed using **2** (0.008 g, 4.1 μmol), 2-iodoanisole (0.11 g, 0.85 mmol), crushed molecular sieves (0.5 g), and Cs_2CO_3 (0.56 g, 1.7 mmol). The reactor vial was evacuated and refilled with nitrogen before addition of the tetrahydrofurfuryl alcohol (1.50 mL, 15.5 mmol). The microwave settings were as follows: temperature = 125 °C, maximum microwave power = 50 W, maximum pressure = 300 psi, and reaction time = 3 h. The reaction mixture was cooled to room temperature, filtered through a small plug of silica gel, and dried under vacuum with gentle heating. The excess tetrahydrofurfuryl alcohol was removed under vacuum with gentle heating. Purification by column chromatography (silica gel hexane/ethyl acetate 2:1) afforded 0.135 g (77%) of the title compound as a dull yellow oil.

The above procedure was repeated in the presence of air without deoxygenating the reagents or solvent. Molecular sieves were not added to this reaction. The reaction was irradiated for 4 h with the same microwave settings as above, and title compound was isolated in 63% yield (0.11 g).

Preparation of 4-Tetrahydrofurfuryloxyaniline (Table 1, Entry 6): Method A. The general procedure was followed using **2** (0.008 g, 4.1 μmol), 4-iodoaniline (0.20 g, 0.91 mmol), crushed molecular sieves (0.5 g), and Cs_2CO_3 (0.60 g, 1.8 mmol). The reaction flask was evacuated and refilled with nitrogen before the addition of tetrahydrofurfuryl alcohol (1.50 mL, 15.5 mmol). The reaction mixture was stirred at 110 °C for 5 h, cooled to room temperature, and filtered through a short plug of silica gel eluting with ethyl acetate. The excess tetrahydrofurfuryl alcohol was removed under vacuum with gentle heating. Purified by column chromatography (silica hexane/ethyl acetate 2:1) afforded 0.13 g (74%) of the title compound. Anal. Calcd for $C_{12}H_{16}O_3$: C, 68.39; H, 7.77. Found: C, 68.14; H, 7.91. ^1H NMR ($CDCl_3$, 25 °C): δ 6.77 (d, 2H, J = 8.8, −$C_6H_4NH_2$), 6.64 (d, 2H, J = 8.8, −$C_6H_4NH_2$), 4.24 (m, 1H, −CH−), 3.88 (m, 4H, −CH_2−), 3.45 (m, 2H, −NH_2), 2.05 (m, 1H, −CH_2−), 1.95 (m, 2H, −CH_2−), 1.75 (m, 1H, −CH_2−). $^{13}C\{^1H\}$ NMR ($CDCl_3$, 25 °C): δ 151.8 (s, 1C, quat), 140.0 (s, 1C, quat), 116.1 (s, 2C, −$C_6H_4NH_2$), 115.5 (s, 2C, −$C_6H_4NH_2$), 77.0 (s, 1C, −CH−), 71.0 (s, 1C, −CH$_2$−), 68.3 (s, 1C, −CH$_2$−), 28.0 (s, 1C, −CH$_2$−), 25.5 (s, 1C, −CH$_2$−).

The above procedure was repeated in the presence of air without deoxygenating the reagents or solvent. Molecular sieves were not added to this reaction. The reaction was stirred at 110 °C for 7 h, and the title compound was isolated in 68% yield (0.12 g).

Preparation of 4-Tetrahydrofurfuryloxyaniline (Table 1, Entry 6): Method B. The general procedure was followed using **2** (0.008 g, 4.1 μmol), 4-iodoaniline (0.20 g, 0.91 mmol), crushed molecular sieves (0.5 g), and Cs_2CO_3 (0.60 g, 1.8 mmol). The reactor vial was evacuated and refilled with nitrogen before addition of tetrahydrofurfuryl alcohol (1.50 mL, 15.5 mmol). The microwave settings were as follows: temperature = 125 °C, maximum microwave power = 50 W, maximum pressure = 300 psi, and reaction time = 2 h. After the reaction was cooled to room temperature, the excess alcohol was removed under vacuum with gentle heating. Purification of the residue by column chromatography (silica hexane/ethyl acetate 2:1) afforded 0.12 g (68%) of the title compound.

The above procedure was repeated in the presence of air without deoxygenating the reagents or solvent. Molecular sieves were not added to this reaction. The reaction was irradiated for 3 h with the same microwave settings as above, and title compound was isolated in 63% yield (0.11 g).

Reaction of 4-Iodonitrobenzene with Tetrahydrofurfuryl Alcohol (Table 1, Entry 7): Method A. The general procedure was followed using **2** (0.008 g, 4.1 μmol), 4-iodonitrobenzene (0.20 g, 0.80 mmol), crushed molecular sieves (0.5 g), and Cs_2CO_3 (0.52 g, 1.6 mmol). The reaction flask was evacuated and refilled with nitrogen before the addition of tetrahydrofurfuryl alcohol (1.5 mL, 15.5 mmol). After stirring at 100 °C for 5 h, the reaction mixture was transferred to a round-bottom flask and the excess alcohol was removed under vacuum with gentle heating. Analysis by GC and NMR spectroscopy revealed a mixture of 4-tetrahydrofurufryloxynitrobenzene and 4-tetrahydrofurfuryloxyaniline.

The above procedure was repeated in the presence of air without deoxygenating the reagents or solvent. Molecular sieves were not added to this reaction. The reaction was stirred at 110 °C for 5 h, and a mixture of 4-tetrahydrofurufryloxynitrobenzene and 4-tetrahydrofurfuryloxyaniline was observed.

Reaction of 4-Iodonitrobenzene with Tetrahydrofurfuryl Alcohol (Table 1 entry 7): Method B. The general procedure was followed using **2** (0.008 g, 4.1 μmol), 4-iodonitrobenzene (0.20 g, 0.80 mmol), crushed molecular sieves (0.5 g), and Cs_2CO_3 (0.52 g, 1.6 mmol). The reactor vial was evacuated and refilled with nitrogen before addition of tetrahydrofurfuryl alcohol (1.5 mL, 15.5 mmol). The microwave settings were as follows: temperature = 125 °C, maximum microwave power = 50 W, maximum pressure = 300 psi, and reaction time = 2 h. After the reaction was cooled to room temperature, the excess alcohol was removed under vacuum with gentle heating. Analysis by GC and NMR spectroscopy revealed no 4-tetrahydrofurfuryloxynitrobenzene. The only aromatic-containing product was 4-tetrahydrofurfuryloxyaniline.

The above procedure was repeated in the presence of air without deoxygenating the reagents or solvent. Molecular sieves were not added to this reaction. The reaction was irradiated for 3 h with the same microwave settings as above which resulted in an intractable mixture of products.

Preparation of 4-Tetrahydrofurfuryloxychlorobenzene (Table 1, Entry 8): Method A. The general procedure was followed using **2** (0.008 g, 4.1 μmol), 4-iodochlorobenzene (0.20 g, 0.84 mmol), Cs_2CO_3 (0.56 g, 1.7 mmol), and crushed molecular sieves (0.5 g). The reaction flask was evacuated and refilled with nitrogen, and tetrahydrofurfuryl alcohol (1.50 mL, 15.5 mmol) was added by syringe. After being stirred at 100 °C for 5 h, the reaction mixture was transferred to a round-bottom flask and the excess alcohol was removed under vacuum with gentle heating. The residue was purified by column chromatography (silica gel: hexane/ethyl acetate 2:1) to afford 0.14 g (79%) of a pale yellow oil. Anal. Calcd for $C_{11}H_{13}ClO_2$: C, 62.13; H, 6.12. Found: C, 62.10; H, 6.49. ^1H NMR ($CDCl_3$, 25 °C): δ 7.20 (d, 2H, J = 8.9 Hz, C_6H_4Cl), 6.83 (d, 2H, J = 8.9 Hz, C_6H_4Cl), 4.24 (m, 1H, −CH−), 3.90 (m, 3H, −CH_2−), 3.83 (m, 1H, −CH_2−), 2.05 (m, 1H, −CH_2−), 1.95 (m, 2H, −CH_2−), 1.75 (m, 1H, −CH_2−). $^{13}C\{^1H\}$ NMR ($CDCl_3$, 25 °C): δ 157.3 (s, 1C, quat), 129.1 (s 2C, −C_6H_4Cl), 125.4 (s, 1C, quat), 115.7 (s, 2C, −C_6H_4Cl), 76.8 (s, 1C, −CH−), 70.5 (s, 1C, −CH$_2$−), 68.4 (s, 1C, −CH$_2$−), 27.9 (s, 1C, −CH$_2$−), 25.5 (s, 1C, −CH$_2$−).

The above procedure was repeated in the presence of air without deoxygenating the reagents or solvent. The reaction time was increased to 8 h, and the title compound was isolated in 73% yield (0.13 g).

Preparation of 4-Tetrahydrofurfuryloxychlorobenzene (Table 1, Entry 8): Method B. The general procedure was followed using **2** (0.008 g, 4.1 μmol), 4-iodochlorobenzene (0.20 g, 0.84 mmol), Cs_2CO_3 (0.56 g, 1.7 mmol), and molecular sieves (0.5 g). The reactor vial was evacuated and refilled with

nitrogen, and tetrahydrofurfuryl alcohol (1.50 mL, 15.5 mmol) was added by syringe. The microwave settings were as follows: temperature = 125 °C, maximum microwave power = 50 W, maximum pressure = 300 psi, and reaction time = 2 h. After the reaction was cooled to room temperature, the excess alcohol was removed under vacuum with gentle heating. Purification of the residue by column chromatography (silica gel: hexane/ethyl acetate 2:1) afforded 0.135 g (76%) of a pale yellow oil.

The above procedure was repeated in the presence of air without deoxygenating the reagents or solvent. The reaction was irradiated for 3 h with the same microwave settings as above, and the title compound was isolated in 73% (0.13 g) yield.

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Supporting Information Available: Experimental procedures for simple alcohols (Table 2, entries 1−7) and crystallographic data. This material is available free of charge via the Internet at http://pubs.acs.org.

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