Solvent-free synthesis of aryl ethers promoted by tetrabutylammonium fluoride

Wei Xiong[a], Quansheng Ding[a], Jiuxi Chen[a]*, Jinchang Ding[a,b] and Huayue Wu[a]

[a]*College of Chemistry and Materials Engineering, Wenzhou University, Wenzhou 325035, P. R. China*

[b]*Wenzhou Vocational and Technical College, Wenzhou 325035, P. R. China*

The reaction of aryl fluorides with silyl reagents has been shown to be efficiently promoted by tetrabutylammonium fluoride, providing aryl ethers with good to excellent yields under solvent-free conditions. The efficiency of this reaction was demonstrated by the compatibility with aldehyde, nitro, cyano and acetyl groups. Moreover, the rigorous exclusion of air/moisture was not required in these transformations which proceeded under milder conditions than previously reported reactions of this type.

Keywords: tetrabutylammonium fluoride, aryl ethers, solvent-free conditions

Aryl ethers are important functional groups. The recent publication of several total syntheses of vancomycin aglycon has further focused attention on the importance of new technology for the construction of the aryl ether unit.[1,2] The classical Ullmann ether synthesis remains an important tool in aryl ether synthesis.[3,4] In addition, several other methods have also been reported.[5,6] Recently, KF/Al_2O_3-mediated S_NAr addition of aryl alcohols to electron-deficient aryl halides in the presence of 18-crown-6 was shown to be an efficient alternative to ether synthesis.[7,8] Potentially efficient methods for the synthesis of aryl ethers from protected alcohols rather than from the parent alcohols have also been described.[9–13] A variety of reagents have been used to promote the deprotection of the silyl group, including CsF,[9] anhydrous TBAF[10,11] phosphazenes[12] or proazaphosphatranes.[13] These protocols all use trialkylsilyl-protected aryl alcohols. As such, these reactions produce one equivalent of silicon by-product relative to the amount of aryl ether formed. Love[14] reported that tetraphenoxysilane and tetraalkoxysilanes react readily with aryl fluorides in the presence of anhydrous TBAF in THF and acetone in a dry N_2 atmosphere. However the novel method was only successful with the highly reactive 2,4-dinitrofluorobenzene, 2-fluorobenzonitrile, 5-chloro-2,4,6-trifluoropyrimidine. Aldehydes were unreactive. Additionally, it was carried out in volatile organic solvent for long reaction time in a dry N_2 atmosphere. Therefore, the development of facile and environment friendly methods for the synthesis of aryl ether is a useful aspect of organic synthesis.

Compared with the reactions in organic solvents, solvent-free reactions are often rapid, regio- or chemoselective, occur in high yields and have environmental and economic advantages.[15–17] In continuation of our interest in solvent-free synthetic methodologies,[18–25] we report here solvent-free synthesis of aryl ethers from the reaction of aryl fluorides with tetra-alkoxysilanes in the presence of $TBAF{\cdot}3H_2O$ under mild conditions.

The model reaction of 4-fluorobenzaldehyde (**1a**) with tetramethoxysilane (**2a**) was conducted to identify the optimal reaction conditions. The results are listed in Table 1. Firstly, the efficacy of various promoters was investigated. Entry 1 shows the blank reaction without the addition of any promoters. In this case no desired product was obtained after 24 h. Among all the promoters screened (tetrabutylammonium bromide (TBAB), tetrabutylammonium iodide (TBAI), CsF, LiF, CuF_2, NH_4F, $KF{\cdot}2H_2O$ and CeF_3), $TBAF{\cdot}3H_2O$ was found to be the most effective reagent in terms of reaction yields. The desired product 4-methoxybenzaldehyde (**3a**) was obtained in 62% yield (entries 2–10). Encouraged by this result, we next planned to determine the influence of solvent on the model reaction. Obviously, the presence of additional solvents makes the reaction rate lower (entries11–17). So we choose to perform this reaction under solvent-free conditions. The yield was decreased to some extent when the amount of $TBAF{\cdot}3H_2O$ was decreased to 0.6 equiv. or 0.8 equiv. (entries 18–19).

The general efficiency of this protocol was then studied for the synthesis of a variety of aryl ethers. As shown in Table 2, the reaction proceeded smoothly in the presence of a variety of functional groups including aldehyde, nitro, cyano and acetyl groups under the optimised conditions. Nitro-substituted aryl fluorides reacted with silyl reagents easily and gave aryl ethers in high yield because the electron withdrawing group increased the reactivity of the aryl fluorides. For example, 4-nitrofluorobenzene (**1b**) reacted quickly to generate the corresponding aryl ether, 1-methoxy-4-nitrobenzene (**3b**), in almost quantitative yield in 0.5 hour (Table 2, entries 2 and 7).

Steric hindrance of an aryl fluoride was also well accommodated. The reaction of *ortho*-substituted aryl fluoride, such as 2-fluorobenzaldehyde (**1d**) with **2a** produced 2-methoxybenzaldehyde (**3d**) in 85% isolated yield (Table 2, entry 4). Unfortunately, attempts to use less activating *meta*-substituted aryl fluorides such as 3-fluorobenzaldehyde with tetramethyl orthosilicate produced 3-methoxybenzaldehyde (**3i**) in 41% yield. However, the reaction of electron-rich and electron- neutral aryl fluoride with tetramethyl orthosilicate was unsuccessful under the standard conditions. Note that the 2,4-dimethoxy-1-nitrobenzene (**3j**) was obtained in 92% yield when the 2,4-difluoro-1-nitrobenzene (**1f**) was employed (Table 2, entry 9).

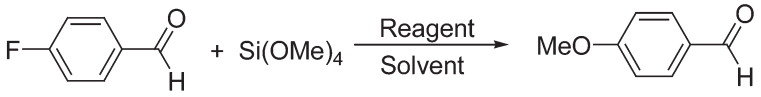
Furthermore, the present route to aryl ethers was successfully applied to a large scale reaction. For instance, the reaction of 1**a** (1.24 g) with **2a** (1.52 g) promoted by $TBAF{\cdot}3H_2O$ provided the desired product **3a** in 93% (Table 2, Entry 1).

Finally, to extend the scope of $TBAF{\cdot}3H_2O$-promoted method of aryl ethers formation under solvent-free condition, trimethoxy(phenyl)silane (**2c**),methyltri(ethylmethylketoximo) silane (**2d**) and vinyltri(methylethylketoxime)silane (**2e**) were employed. As shown in Scheme 1, the reactions were also performed smoothly and gave the corresponding products **3b** and butan-2-one-*O*-4-nitrophenyl oxime (**3k**) using the present protocol with excellent yields.

In summary, a new protocol to synthesise aryl ethers has been developed. Compared to previous reported methodologies, the presented protocol is not only very simple, quick but also greatly decreases environmental pollution. The reaction did not need the protection of nitrogen nor argon, and $TBAF{\cdot}3H_2O$ was much cheaper and safer than solution of TBAF in THF.[14] This protocol avoids the use of a hazardous solvent, toxic metallic catalysts, and is of low cost. Furthermore, the present protocol can tolerate a wide variety of

* Correspondent. E-mail: jiuxichen@wzu.edu.cn

Table 1 Screening conditions[a]



1a **2a** **3a**

Entry	Reagent /equiv.)	Solvent	Yield[b]/%
1	none	none	NR[c]
2	TBAF·3H_2O (1.0)	THF	62
3	TBAB (1.0)	THF	NR
4	TBAI (1.0)	THF	NR
5	CsF (1.0)	THF	45
6	LiF (1.0)	THF	NR
7	CuF_2 (1.0)	THF	NR
8	NH_4F (1.0)	THF	15
9	KF·2H_2O (1.0)	THF	NR
10	CeF_3 (1.0)	THF	NR
11	TBAF·3H_2O (1.0)	dioxane	58
12	TBAF·3H_2O (1.0)	DMF	75
13	TBAF·3H_2O (1.0)	toluene	60
14	TBAF·3H_2O (1.0)	CH_2ClCH_2Cl	NR
15	TBAF·3H_2O (1.0)	acetone	NR
16	TBAF·3H_2O (1.0)	CH_3CN	NR
17	TBAF·3H_2O (1.0)	none	92
18	TBAF·3H_2O (0.6)	none	61
19	TBAF·3H_2O (0.8)	none	82
20	TBAF·3H_2O (1.2)	none	92

[a]Reaction conditions: 4-fluorobenzaldehyde **1a** (1.0 mmol), tetramethyl orthosilicate **2a** (0.6 equiv.), reagent, solvent (3 mL), 9 h, 80 °C. [b]Isolated yield. [c]NR = No reaction.

functional groups, including aldehyde, nitro, cyano and acetyl groups.

Experimental

Chemicals were purchased and used without further purification. All the melting points were uncorrected. NMR spectroscopy was performed on a Bruker-300 spectrometer using $CDCl_3$ as the solvent with tetramethylsilane (TMS) as an internal standard at room temperature. Chemical shifts are given in δ relative to TMS, the coupling constants J are given in Hz. Mass spectra (EI, 70 eV) were measured with SHIMADZU GCMS-QP2010 Plus. Elemental analyses were carried out using a Carlo-Erba EA1108 instrument. Column chromatography was performed using EM Silica gel 60 (300–400 mesh).

Preparation of aryl ethers; general procedure

The aryl fluorides (1.0 mmol) and tetraalkoxysilanes (0.6 equiv.), and TBAF·3H_2O (1.0 equiv.) were placed in a Schlenk reaction tube and stirred for given time at 80 °C. After completion of the reaction, as indicated by TLC, the crude products were directly separated by column chromatography using petroleum ether/EtOAc as eluent to afford pure corresponding products.

4-Methoxybenzaldehyde (**3a**):[26] Colourless oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 3.80 (s, 3H, CH_3), 6.92 (d, 2H, J = 8.7 Hz, ArH), 7.76 (d, 2H, J = 8.7 Hz, ArH), 9.80 (s, 1H, CHO); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 55.5, 114.3, 129.9, 131.9, 164.6, 190.8; IR (KBr): ν_{max}/cm^{-1} 3068, 2938, 2838, 2739, 1689, 1598, 1511, 1454, 1313, 1259, 1161, 1108, 1025, 835, 602, 512.

1-Methoxy-4-nitrobenzene (**3b**):[27] White solid, m.p. 51–52 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 3.91 (s, 3H, CH_3), 6.96 (d, 2H, J = 9.2 Hz, ArH), 8.20 (d, 2H, J = 9.2 Hz, ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 55.8, 113.9, 125.7, 141.3, 164.5; IR (KBr): ν_{max}/cm^{-1} 3025, 2973, 2938, 2842, 1591, 1501, 1459, 1334, 1261, 1173, 1105, 1017, 847, 749, 690, 618.

4-Methoxybenzonitrile (**3c**):[28] White solid, m.p. 56 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 3.86 (s, 3H, CH_3), 6.95 (d, 2H, J = 8.9 Hz, ArH), 7.59 (d, 2H, J = 8.9 Hz, ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 55.3, 103.8, 114.5, 119.0, 133.8, 162.6; IR (KBr): ν_{max}/cm^{-1} 3072, 2938, 2842, 2221, 1602, 1505, 1453, 1300, 1257, 1170, 1023, 832, 680, 547.

2-Methoxybenzaldehyde (**3d**):[29] Colourless oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 3.89 (s, 3H, CH_3), 6.94–6.99 (m, 2H, ArH), 7.51–7.52 (m, 1H, ArH), 7.78–7.80 (m, 1H, ArH), 10.44 (s, 1H, CHO); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 55.4, 111.4, 120.4, 124.6, 128.2, 135.8, 161.6, 189.6.

1-(4-Methoxyphenyl)ethanone (**3e**):[30] Pale yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 2.55 (s, 3H, CH_3CO), 3.88 (s, 3H, CH_3O), 6.93 (d, 2H, J = 8.9 Hz, ArH), 7.94 (d, 2H, J = 8.9 Hz, ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 26.1, 55.3, 113.5, 130.1, 130.4, 163.3, 196.6; IR (KBr): ν_{max}/cm^{-1} 3065, 3002, 2950, 2843, 1675, 1597, 1510, 1425, 1359, 1258, 1175, 1115, 1026, 958, 834, 579.

4-Ethoxybenzonitrile (**3f**):[31] Pale yellow solid, m.p. 51–53 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 1.44 (t, 3H, J = 7.0 Hz, CH_3), 4.08 (q, 2H, J = 7.0 Hz, CH_2), 6.93 (d, 2H, J = 8.7 Hz, ArH), 7.58 (d, 2H, J = 8.7 Hz, ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 14.6, 63.9, 103.7, 115.2, 119.3, 134.0, 162.3; IR (KBr): ν_{max}/cm^{-1} 3074, 2983, 2935, 2895, 2221, 1603, 1504, 1394, 1299, 1255, 1168, 1112, 1036, 917, 833, 702, 499.

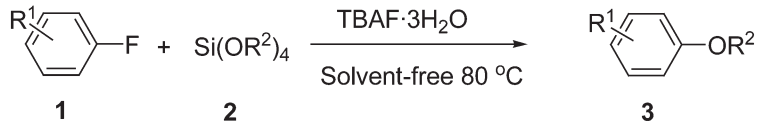
4-Ethoxybenzaldehyde (**3g**):[32] Colourless oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 1.43 (t, 3H, J = 7.0 Hz, CH_3), 4.09 (q, 2H, J = 7.0 Hz, CH_2), 6.96 (d, 2H, J = 8.7 Hz, ArH), 7.80 (d, 2H, J = 8.7 Hz, ArH), 9.85 (s, 1H, CHO); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 14.5, 63.8, 114.6, 129.7, 131.9, 164.0, 190.7; IR (KBr): ν_{max}/cm^{-1} 3069, 2926, 2858, 2736, 1690, 1599, 1508, 1255, 1161, 1112, 1040, 834.

1-Ethoxy-4-nitrobenzene (**3h**):[31] Pale yellow solid, m.p. 55–56 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 1.45 (t, 3H, J = 7.0 Hz, CH_3), 4.12 (q, 2H, J = 7.0 Hz, CH_2), 6.93 (d, 2H, J = 9.0 Hz, ArH), 8.16 (d, 2H, J = 9.0 Hz, ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 14.5, 64.4, 114.4, 125.8, 141.3, 164.0; IR (KBr): ν_{max}/cm^{-1} 3111, 2987, 2938, 1592, 1497, 1397, 1333, 1254, 1173, 1103, 1034, 911, 852, 755, 645.

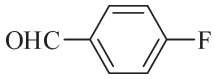
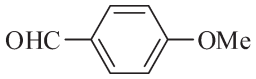
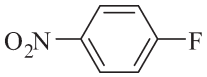
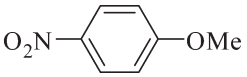
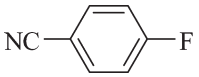
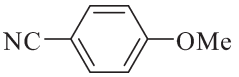
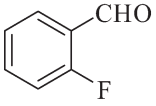
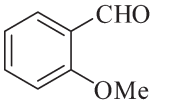
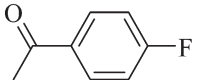
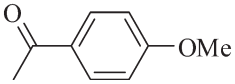
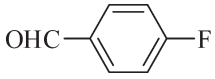
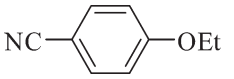
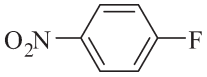
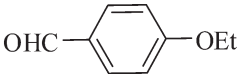
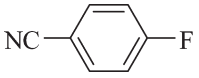
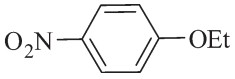
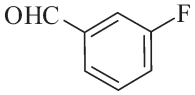
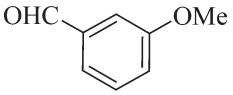
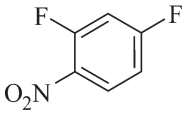
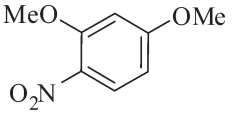
3-Methoxybenzaldehyde (**3i**)[23]: Colourless oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 3.85 (s, 3H, CH_3), 7.17–7.18 (m, 1H, ArH), 7.37–7.44 (m, 3H, ArH), 9.96 (s, 1H, CHO); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 55.3, 111.8, 121.3, 123.3, 129.8, 137.6, 159.9, 192.0

2,4-Dimethoxy-1-nitrobenzene (**3j**):[27] Pale yellow solid, m.p. 69 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 3.90 (s, 3H, CH_3), 3.96 (s, 3H, CH_3), 6.50–6.54 (m, 2H, ArH), 8.01 (d, 1H, J = 8.8 Hz, ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 55.9, 56.5, 99.6, 104.7, 128.5, 132.9, 155.7, 164.8; IR (KBr): ν_{max}/cm^{-1} 3035, 2986, 2850, 1609, 1545, 1449, 1344, 1301, 1251, 1153, 1105, 847, 749.

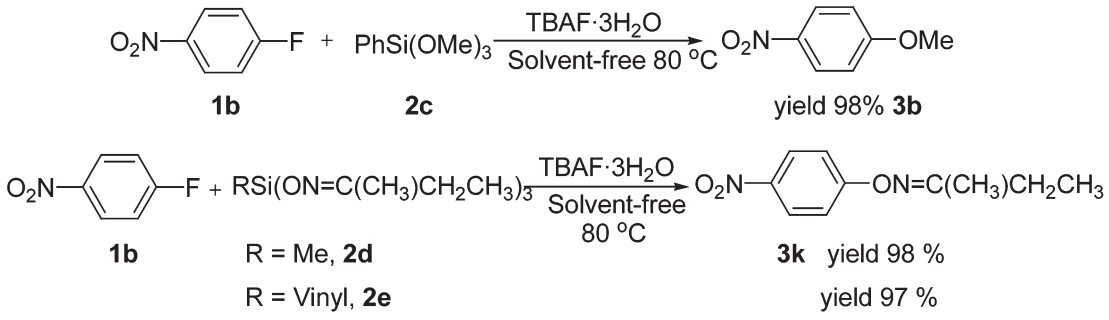
Table 2 Solvent-free synthesis of aryl ethers[a]



1 **2** **3**

Entry	Ar-F	R^2	Time /h	Product		Yield[b] /%
1		OMe	9		**3a**	92 (93)[c]
2		OMe	0.5		**3b**	99
3		OMe	2		**3c**	94
4		OMe	9		**3d**	85
5		OMe	12		**3e**	84
6		OEt	9		**3f**	89
7		OEt	0.5		**3g**	98
8		OEt	2		**3h**	91
9		OMe	12		**3i**	41
10		OMe	2		**3j**	94[d]

[a] Reactions conditions: ArF 1 (1.0 mmol), Silane 2 (0.6 equiv.), TBAF·3H2O (1.0 equiv.), 80 °C, solvent-free. [b] Isolated yield.
[c] 4-Fluorobenzaldehyde (20 mmol), aldehydes (12 mmol), TBAF·$3H_2O$ (1.0 equiv.), 80 °C, solvent-free.
[d] 0.8 equiv. silane was used.



Scheme 1

Butan-2-one-O-4-nitrophenyl oxime (**3k**): Yellow solid, m.p. 35–36 °C; *anti*:*syn* = 1:4 (ratio based on its ^{1}H NMR spectrum), ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 1.16 (t, 3H, J = 7.7 Hz, *anti*, CH_2CH_3), 1.20 (t, 3H, J = 7.5 Hz, *syn*, CH_2CH_3), 2.04 (s, 3H, *anti*, CH_3), 2.07 (s, 3H, *syn*, CH_3), 2.38 (q, 2H, J = 7.5 Hz, *syn*, CH_2), 2.55 (q, 2H, J = 7.7 Hz, *anti,* CH_2), 7.26 (d, 2H, J = 9.3 Hz, ArH), 8.19 (d, 2H, J = 9.3 Hz, ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 10.2, 10.6, 14.9, 19.3, 23.4, 29.3 (6C, CH_2CH_3, CH_3, *anti* + *syn*), 114.2, 125.7 (2C, Ph), 142.0 (C-NO_2, Ph), 164.3 (C=N), 164.8 (C-O, Ph); IR (KBr): ν_{max}/cm^{-1} 3106, 2977, 2932, 1649, 1593, 1510, 1339, 1255, 1160, 1106, 898, 847, 751, 679; MS (EI, 70 eV): *m/z* (%) = 208 (M^+, 26), 139 (30), 109 (39), 93 (15), 81 (18), 70 (100), 63 (63), 54 (18); Anal. Calcd for $C_{10}H_{12}N_2O_3$: C, 57.68; H, 5.81; N, 13.45. Found: C, 57.58; H, 5.90; N, 13.22%.

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