Sodium Bisulfite: An Efficient Catalyst for Ether Formation via Dehydration of Aromatic/Aliphatic Alcohol

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Straightforward etherification of benzyl alcohols (**1**) via intermolecular dehydration can be efficiently catalyzed by sodium bisulfite under solvent-free conditions. In the presence of 0.3 mol% or 0.6 mol% amount of sodium bisulfite, symmetric and unsymmetric ethers are prepared from the corresponding alcohols in high yields (up to 95%). Etherification of benzhydryl alcohols is also discussed.

Keywords sodium bisulfite, etherification, dehydration, aromatic alcohol, aliphatic alcohol

Introduction

Preparation of ethers is one of the most fundamental reactions in synthetic organic chemistry. One of the most common methods for synthesis of ethers, Williamson ether synthesis, requires initial transformation of alcohols to halides or tosylates.[1] Direct dehydration of alcohols promoted by acids,[2] metal complexes,[3] dimethyl sulfoxide,[4] iodine,[5] Y-zeolite,[6] or ionic liquid[7] provides a straightforward method for the preparation of ethers. For example, Das *et al*.[8] reported that silica-supported sodium hydrogen sulfate ($NaHSO_4$•SiO_2) can catalyze the transformation of *p*-hydroxybenzyl alcohols to *p*-hydroxybenzyl ethers. Aslam *et al*.[9] reported synthesis of a bisarylalkyl ether using a substoichiometric amount of potassium bisulfate ($KHSO_4$) as a catalyst. However, strong acidity of $NaHSO_4$ and $KHSO_4$ (pK_a 1.92) often leads to undesired side-reactions during etherification of alcohols.

As the cheap sodium bisulfite ($NaHSO_3$) is a weaker acid (pK_a 7.21) when compared with $NaHSO_4$ and $KHSO_4$, we decided to investigate its reactivity as a catalyst in etherification of alcohols.[10] Herein, we would like to report the $NaHSO_3$ catalyzed direct etherification of aromatic/aliphatic alcohols, as well as benzhydryl alcohols.[11]

Results and discussion

First, a straightforward etherification of phenylethyl alcohol **1a** was carried out using $NaHSO_3$ as a catalyst under various reaction conditions (Scheme 1 and Table 1). It was found that **1a** could undergo intermolecular dehydration with 0.3 mol% amount of $NaHSO_3$ at 110 ℃ to give a symmetrical ether **3a** in up to 90% yield (Table 1, Entry 3). A decrease in temperature led to an incomplete reaction, while an increase in temperature produced a relatively large amount of intramolecular dehydration product **3a'** (Table 1, Entries 4 and 5).

Scheme 1

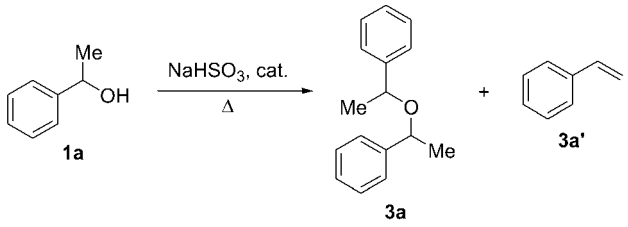

Table 1 Etherification of **1a** catalyzed with sodium bisulfite under various conditions

Entry	Catalyst amount	Temp./℃	Time	Yield[a] (**3a**)	Yield[a] (**3a'**)
1	0.6 mol%	110	1 h	88%	4%
2	0.3 mol%	110	10 min	5%	—
3	0.3 mol%	110	1 h	90%	3%
4	0.3 mol%	100	1 h	18%	17%
5	0.3 mol%	150	1 h	79%	16%

[a] NMR yields.

Under the optimized reaction conditions, straightforward etherifications of benzyl alcohols possessing *β*-hydrogens were carried out (Scheme 2 and Table 2). It was found that etherifications of **1** proceeded smoothly

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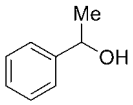
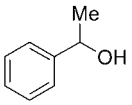
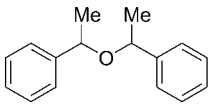
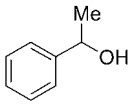
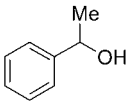
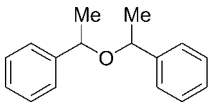
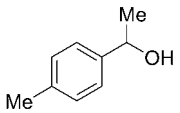
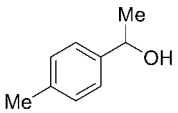
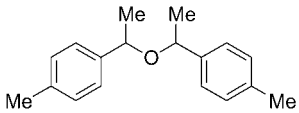
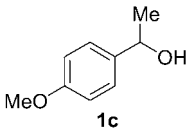
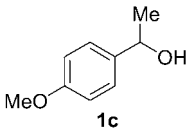
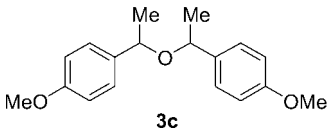
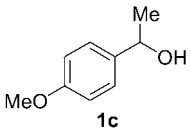
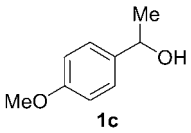
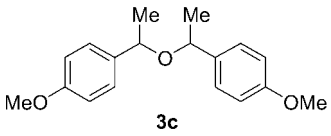
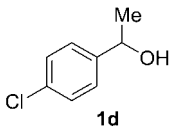
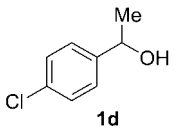
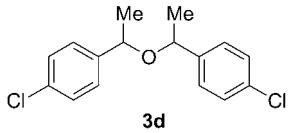
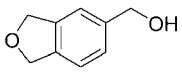
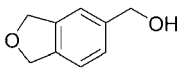
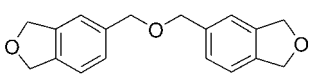
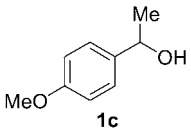
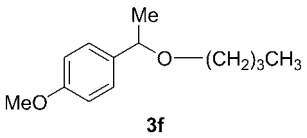
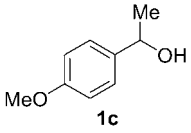
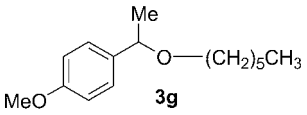
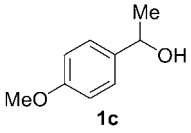
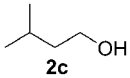
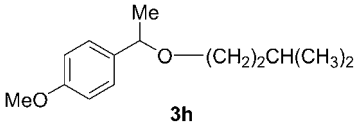
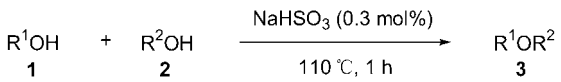
Table 2 Dehydration of arylalkyl alcohols **1a—1e** and **1c** with alkyl alcohol **2a—2c** catalyzed with sodium bisulfite under solvent-free conditions

Entry	R^1OH	R^2OH	Product	Yield[a]/% (**3**)	Yield[a]/% (**3'**)
1	 **1a**	 **1a**	 **3a**	90 (88)[b]	3
2[c]	 **1a**	 **1a**	 **3a**	4	94[b]
3	 **1b**	 **1b**	 **3b**	92 (89)[b]	4
4	 **1c**	 **1c**	 **3c**	95 (90)[b]	5
5[d]	 **1c**	 **1c**	 **3c**	36[b]	19
6	 **1d**	 **1d**	 **3d**	95 (92)[b]	4
7	 **1e**	 **1e**	 **3e**	88 (79)	-
8	 **1c**	$CH_3(CH_2)_2CH_2OH$ **2a**	 **3f**	84 (80)	16
9	 **1c**	$CH_3(CH_2)_5CH_2OH$ **2b**	 **3g**	90 (85)	10
10	 **1c**	 **2c**	 **3h**	91 (83)	9

[a] NMR yields. Isolated yields are given in the parentheses. [b] The product is about 1∶1 mixtures of *dl* enantiomers and *meso* compounds. [c] $NaHSO_4$ (0.6 mol%) was used as a catalyst. [d] $KHSO_4$ (0.6 mol%) was used as a catalyst.

Scheme 2



under solvent-free conditions to afford the corresponding symmetric bisarylalkyl ethers **3** in high yields. For example, etherification of alcohol **1b** with 0.3 mol% amount of $NaHSO_3$ as a catalyst leads to the desired symmetric ether **3b** in up to 92% yield with a small amount of intramolecular dehydration by-product styrene **3b'** (Table 2, Entry 3). The same selectivity was also observed in the case of **1a**, **1c** and **1d** which bear electron-donating or withdrawing substituents on the aromatic ring (Table 2, Entries 1, 4 and 6). Interestingly when $NaHSO_4$ was applied as a catalyst for etherification of alcohol **1a**, styrene **3a'** was obtained in 94% yield, and ether **3a** was only in 4% yield (Table 2, Entry 2). And $KHSO_4$ gave similar results when it was applied as a catalyst for etherification of alcohol **1c** (Table 2, Entry 5).[9] These results suggested that $NaHSO_3$ showed a unique property for direct etherification of benzyl alcohols possessing *β*-hydrogens. It is also noteworthy that dehydration of piperonyl alcohol (**1e**) with $NaHSO_3$ as a catalyst provided the corresponding symmetric ether **3e** (Table 2, Entry 7), while polymerization of **1e** was observed in the case of $NaHSO_4$. Such opposite reaction selectivity could possibly due to the relative weaker acidity of $NaHSO_3$ compared with $NaHSO_4$/$KHSO_4$.[12,13]

Subsequently we investigated direct etherifications of **1** with aliphatic alcohols catalyzed with $NaHSO_3$. To our delight, we found that **1c** and primary aliphatic alcohol **2a**, **2b** or **2c** underwent efficient etherification to afford unsymmetrical ether **3f**, **3g** or **3h** in good yields and high selectivities (Table 2, Entries 8, 9 and 10). For instance, **1c** reacted with 4.0 equiv. of 3-methyl-1-butanol (**2c**) catalyzed by $NaHSO_3$ at 110 ℃ to give **3h** in 95% yield, without formation of **3c** or **3c'**.

To extend the scope of this methodology, $NaHSO_3$ was also applied to catalyze etherification of various benzhydrol derivatives (Scheme 3 and Table 3). Both activated and deactivated benzhydryl alcohols demonstrated good reactivities. For example, the symmetrical ether **5b** can be efficiently synthesized from (4-methoxyphenyl)(phenyl)methanol (**4b**) in the presence of a catalytic amount of $NaHSO_3$. In the absence of $NaHSO_3$, we observed no etherification reaction.[14] Interestingly, bis(4-fluorophenyl)methanol (**4d**) can also smoothly undergo dehydration to afford ether **5d** in a similar yield as **4b**, which suggests that substituents on the aromatic ring have little effect on the reaction rate.

Scheme 3

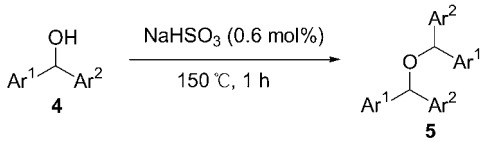
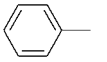
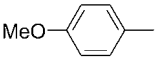
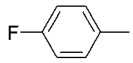
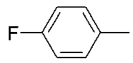
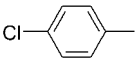


Table 3 Dehydration of benzhydryl alcohols **4a—4e** catalyzed with sodium bisulfite under solvent-free conditions

Entry	Ar^1	Ar^2	Product	Yield[a,b]/%
1		**4a**	**5a**	92 (90)
2		**4b**	**5b**	99 (95)
3		**4c**	**5c**	95 (92)
4		**4d**	**5d**	96 (95)
5		**4e**	**5e**	95 (94)

[a] NMR yields. Isolated yields are given in the parentheses. [b] All products were about 1∶1 mixtures of *dl* enantiomers and *meso* compounds.

Experimental

General

$NaHSO_3$, $NaHSO_4$, $KHSO_4$ and aliphatic alcohols were purchased from commercial source and were used as received. Benzyl alcohols and benzhydryl alcohols were obtained by reduction of aromatic ketones with $NaBH_4$ in MeOH. ^1H NMR spectra were measured on a JEOL 400 MHz spectrometer with TMS as an internal standard in $CDCl_3$. ^{13}C NMR spectra were measured on a Bruker 500 MHz spectrometer with TMS as an internal standard in $CDCl_3$. High-resolution mass spectra (HRMS) were obtained on a Bruker microTOF II.

General procedure for compounds 3a—3e

A mixture of 1-phenylethanol (**1a**) (244 mg, 2.0 mmol) and $NaHSO_3$ (0.6 mg, 0.006 mmol) was stirred at 110 ℃ in oil bath for 1 h. After the reaction finished, the residue was purified by column chromatography on silica gel to give the corresponding ether **3a** in 88% isolated yield.

1-(1-(1-Phenylethoxy)ethyl)benzene (3a)[2j] Colorless oil, 88% yield; ^1H NMR ($CDCl_3$, 400 MHz) δ: 1.38 (d, J=6.4 Hz, 6H), 4.24 (q, J=6.4 Hz, 2H), 7.32—7.40 (m, 10H).

1-Methyl-4-(1-(1-*p*-tolylethoxy)ethyl)benzene (3b)[2j] Colorless oil, 89% yield; ^1H NMR ($CDCl_3$, 400 MHz) δ: 1.36 (d, J=6.0 Hz, 6H), 2.36 (s, 6H), 4.21 (q, J=6.4 Hz, 2H), 7.09—7.23 (m, 8H).

1-Methoxy-4-(1-(1-(4-methoxyphenyl)ethoxy)-ethyl)benzene (3c)[2j] Colorless oil, 90% yield; ^1H NMR ($CDCl_3$, 400 MHz) δ: 1.35 (d, J=6.4 Hz, 6H), 3.78 (s, 6H), 4.18 (q, J=6.8 Hz, 2H), 6.84—6.90 (m, 4H), 7.18—7.25 (m, 4H).

1-Chloro-4-(1-(1-(4-chlorophenyl)ethoxy)ethyl)-benzene (3d)[2j] Colorless oil, 92% yield; ^1H NMR

($CDCl_3$, 400 MHz) δ: 1.34 (d, J=6.4 Hz, 6H), 4.17 (q, J=6.8 Hz, 2H), 7.16—7.33 (m, 8H).

5-(((1,3-Dihydroisobenzofuran-5-yl)methoxy)-methyl)-1,3-dihydroisobenzofuran (3e) White solid, m.p. 40—41 ℃ (Lit.[15] 41—43 ℃), 79% yield; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 4.42 (s, 4H), 5.94 (s, 4H), 6.76—6.79 (m, 4H), 6.86 (s, 2H).

General procedure for compounds 3f—3h

A mixture of 1-(4-methoxyphenyl)ethanol (**1c**) (310 mg, 2.0 mmol), 1-butanol (590 mg, 8.0 mmol) and $NaHSO_3$ (0.6 mg, 0.006 mmol) was stirred at 110 ℃ for 1 h. After the reaction finished, the residue was purified by column chromatography on silica gel to give the corresponding ether **3f** in 80% isolated yield.

1-(1-Butoxyethyl)-4-methoxybenzene (3f)[2i] Colorless oil, 80% yield; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 0.88 (t, J=7.2 Hz, 3H), 1.35—1.42 (m, 2H), 1.41 (d, J=6.4 Hz, 3H), 1.51—1.60 (m, 2H), 3.26 (t, J=6.8 Hz, 2H), 3.80 (s, 3H), 4.34 (q, J=6.4 Hz, 1H), 6.88 (d, J=8.4 Hz, 2H), 7.23 (d, J=8.4 Hz, 2H).

1-(1-(Heptyloxy)ethyl)-4-methoxybenzene (3g) Colorless oil, 85% yield; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 0.90 (t, J=6.4 Hz, 3H), 1.27—1.35 (m, 6H), 1.42 (d, J=6.4 Hz, 3H), 1.47—1.59 (m, 4H), 3.27 (t, J=6.4 Hz, 2H), 3.82 (s, 3H), 4.36 (q, J=6.4 Hz, 1H), 6.89 (d, J=8.8 Hz, 2H), 7.26 (d, J=8.8 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 13.82, 22.40, 23.93, 25.98, 28.95, 29.78, 21.63, 54.67, 68.18, 77.25, 113.41, 126.96, 135.06, 158.65. HRMS calcd for $[C_{16}H_{26}O_2Na]^+$ 273.1825, found 273.1810.

1-(1-(Heptyloxy)ethyl)-4-methoxybenzene (3h) Colorless oil, 83% yield; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 0.92 (d, J=6.4 Hz, 6H), 1.41 (d, J=6.4 Hz, 3H), 1.52—1.57 (m, 1H), 1.64—1.75 (m, 2H), 3.44 (dt, J=6.4, 10.8 Hz, 1H), 3.50 (dt, J=6.0, 10.6 Hz, 1H), 3.80 (s, 3H), 4.32 (q, J=6.4 Hz, 1H), 6.88 (d, J=8.4 Hz, 2H), 7.23 (d, J=8.4 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 22.17, 22.52, 23.90, 24.74, 38.60, 54.66, 66.45, 77.30, 113.41, 127.06, 136.02, 158.59. HRMS calcd for $[C_{14}H_{22}O_2Na]^+$ 245.1512, found 245.1502.

General procedure for compounds 5a—5e

A mixture of diphenylmethanol **5a** (184 mg, 1.0 mmol) and $NaHSO_3$ (0.6 mg, 0.006 mmol) was stirred at 150 ℃ in oil bath for 1 h. After the reaction finished, the residue was purified by column chromatography on silica gel to give the corresponding ether **5a** in 90% isolated yield.

Benzhydryloxydiphenylmethane (5a) White solid, m.p. 104—105 ℃ (Lit.[5a] 108—110 ℃), 90% yield; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 5.40 (s, 2H), 7.23—7.37 (m,20H).

1-((4-Methoxyphenyl)((4-methoxyphenyl)(phenyl) methoxy)methyl)benzene (5b) White solid, m.p. 120—122 ℃ (Lit.[16] 122—123 ℃), 95% yield; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 3.77 (s, 6H), 5.34 (s, 2H), 6.84 (d, J=7.8 Hz, 4H), 7.24—7.34 (m, 14H).

1-((4-Fluorophenyl)((4-fluorophenyl)(4-methoxyphenyl)methoxy)methyl)-4-methoxybenzene (5c) Yellow oil, 92% yield; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 3.78 (s, 3H), 3.79 (s, 3H), 5.29 (s, 2H), 6.84—6.88 (m, 4H), 6.96—7.02 (m, 4H), 7.20—7.23 (m, 4H), 7.27—7.31 (m, 4H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 54.93, 78.84, 113.78, 113.86, 114.92, 115.02, 115.09, 115.20, 128.49, 128.33, 133.69, 133.97, 138.14, 138.41, 159.02, 159.10, 160.93, 161.00, 162.88, 162.96. HRMS calcd for $[C_{28}H_{24}F_2O_3Na]^+$ 469.1586, found 469.1590.

(Bis(4-fluorophenyl)methoxy)bis(4-fluorophenyl)methane (5d) White solid, m.p. 78—79 ℃, 95% yield; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 5.30 (s, 2H), 6.99—7.04 (m, 8H), 7.25—7.28 (m, 8H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 78.88, 115.32, 115.50, 128.70, 128.76, 137.45, 161.25, 163.21. HRMS calcd for $[C_{28}H_{18}F_4ONa]^+$ 469.1186, found 469.1199.

1-((4-Chlorophenyl)((4-chlorophenyl)(*p*-tolyl)-methoxy)methyl)-4-methylbenzene (5e) Yellow oil, 94% yield; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 2.32 (s, 3H), 2.33 (s, 3H), 5.29 (s, 2H), 7.12—7.20 (m, 8H), 7.24—7.27 (m, 8H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 21.02, 79.20, 127.00, 127.18, 128.24, 128.38, 128.49, 129.15, 129.23, 132.94, 133.07, 137.27, 137.40, 138.28, 138.53, 140.72, 140.96. HRMS calcd for $[C_{28}H_{24}Cl_2ONa]^+$ 469.1096, found 469.1094.

Conclusions

In conclusion, we have developed a simple and efficient protocol for the synthesis of symmetric and unsymmetric ethers via direct dehydration of benzyl alcohols containing β-hydrogens by applying cheap $NaHSO_3$ as a catalyst under solvent-free conditions. The etherification of benzhydryl alcohols catalyzed with $NaHSO_3$ is also explored. Further studies on $NaHSO_3$ catalyzing C—C, C—O and C—N bond formations are ongoing in our laboratory.

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