Tetrahedron Letters 53 (2012) 5338–5342

Contents lists available at SciVerse ScienceDirect

Tetrahedron Letters

journal homepage: www.elsevier.com/locate/tetlet

A mild, efficient, and selective deprotection of *tert*-butyldimethylsilyl (TBDMS) ethers using dicationic ionic liquid as a catalyst

Arvind H. Jadhav, Hern Kim *

Department of Environmental Engineering and Energy, Energy and Environment Fusion Technology Center, Myongji University, Yongin, Kyonggi-do 449-728, Republic of Korea

ARTICLE INFO

Article history:
Received 3 June 2012
Revised 21 July 2012
Accepted 25 July 2012
Available online 2 August 2012

Keywords:
Selective deprotection
Desilylation
tert-Butyldimethylsilyl group
Dicationic ionic liquid
Catalytic cleavage

ABSTRACT

Selective deprotection of alkyl TBDMS ether in the presence of phenolic TBDMS ether using dicationic ionic liquid [tetraEG(mim)$_2$][OMs]$_2$ as a homogeneous catalyst showed significant catalytic activity in methanol at ambient temperature to produce respective alcohol in excellent yield. The present environmentally benign catalytic system is found to be very convenient, fast, high yielding, and clean method for selective desilylation of alkyl silyl ethers even in the existence of other sensitive organic functional groups such as aldehyde, methoxy, and acetate were also achieved.

The strategy of hydroxyl functionality protection and its subsequent deprotection at a later stage is usual practice in multistep synthesis of complex organic and bioorganic molecules.[1] The *tert*-butyldimethylsilyl (TBDMS) group is used widely for the protection of hydroxyl group in synthetic organic chemistry due to their ease of installation, stability toward various conditions such as Grignard reaction, Wittig reaction, reduction reaction, etc., and finally deprotect easily without affecting other functional groups.[1,2]

The enormous research in silylation chemistry has been resulted in the development of numerous methods of desilylation. Mainly, the acidic reagents such as CF_3COOH/H_2O,[3] CH_3COOH,[4] HF,[5] $Zn(BF_4)_2$,[6] $Sc(OTf)_3$,[7] $SbCl_5$,[8] $SnCl_2$,[9] $ZrCl_4$,[10] BCl_3,[11] $InCl_3$,[12] $ZnBr_2$,[13] $Ce(OTf)_4$,[14] $Ni(II)Cl_2{\cdot}6H_2O$[15] have been reported for desilylation. The basic reagents like TBAF,[2a] $NaOH/Bu_4NHSO_4$,[16] TBATB,[17] K_2CO_3,[18] Cs_2CO_3,[19] were also utilized for the deprotection of silyl ethers. Oxidizing reagents DDQ,[20] $KMnO_4$,[21] $BaMnO_4$,[21] and reducing agents DIBAL-H,[22] $LiAlH_4$,[23] NaH,[24] have also been reported for desilylation. However, some of these procedures have difficulties such as longer reaction time, harsh reaction conditions, use of expensive and corrosive reagents as well as cumbersome work-up procedures. Although, discrimination between two silyl ethers derived from alcohols is common, relatively very few techniques have been developed to deprotect alkyl silyl ethers in the presence of phenolic silyl ethers such as oxone,[25] *N*-iodosuccinimide,[26] 1-chloroethyl chloroformate,[27] and Iron(III) tosylate.[28] While, these protocols are quite moisture sensitive, environmentally hazardous, and required long reaction time for the completion of reaction. Therefore, it is essential to develop a mild, eco-friendly, fast, and selective method for the deprotection of alkyl silyl ethers.

Ionic liquids (ILs) have received great attention in various fields of science, due to their unique properties such as extremely low vapor pressure, non-flammability, tunability, and high thermal stability.[29] Especially, imidazolium based ILs played important role in various organic reactions as a catalyst or alternative for conventional solvents.[29,30] More recently, tailor-made ILs [hexaEGmim][OMs] or [dihexaEGmim][OMs] with alkali-metal fluoride in *tert*-amyl alcohol has been reported as effective catalytic system for selective deprotection of phenolic silyl ethers.[31] However, the use of basic reagent and harsh reaction condition for desilylation restrict the use of this protocol in complex organic synthesis. Recently, we reported short oligo ethylene glycol functionalized imidazolium dicationic ILs as a catalyst for dehydration of fructose and sucrose into 5-hydroxymethylfurfural (HMF).[32] In continuation of this work, herein we disclose the selective deprotection of alkyl silyl ethers using [tetraEG(mim)$_2$][OMs]$_2$ as a catalyst in methanol at mild reaction condition whereas the phenolic silyl ethers were unaffected.

The task specific dicationic IL [tetraEG(mim)$_2$][OMs]$_2$ was prepared using simple pathway as shown in Scheme 1. In short, tetra ethylene glycol (**1**) was mesylated to obtain tetraethylene glycol dimesylate (**2**) followed by the addition of *N*-methylimidazole in **2** to afford tetra ethylene glycol-bis (3-methylimidazolium) dimesylate ([tetraEG(mim)$_2$][OMs]$_2$) (**3**) as a colorless thick liquid.[32]

Table 1 demonstrates the deprotection of benzyl silyl ether (**4**) as a model compound using various reaction conditions. Initially,

* Corresponding author. Tel.: +82 31 330 6688; fax: +82 31 336 6336.
E-mail address: hernkim@mju.ac.kr (H. Kim).

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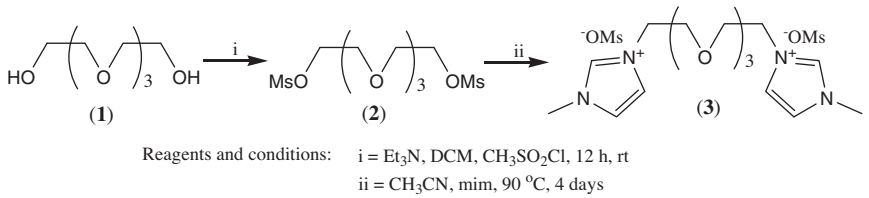
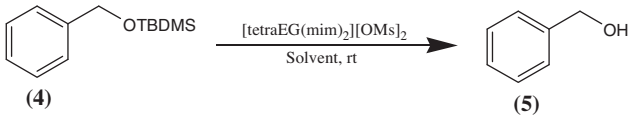
http://dx.doi.org/10.1016/j.tetlet.2012.07.108



Scheme 1. Preparation of tetra ethylene glycol-bis (3-methylimidazolium) dimesylate ([tetraEG(mim)$_2$][OMs]$_2$).

Table 1
Deprotection of benzyl silyl ether in presence of [tetraEG(mim)$_2$][OMs]$_2$ using various reaction conditions[a]



Entry	Solvent	[tetraEG(mim)$_2$][OMs]$_2$ (equiv)	Temperature	Time	Yield[b](%)
1	Methanol	–	rt	24 h	1
2	Methanol	2.0	rt	60 min	95
3	Methanol	1.0	rt	70 min	95
4	Methanol	0.5	rt	80 min	95
5	Methanol	0.2	rt	80 min	94
6	Dichloromethane	0.2	rt	12 h	28
7	Acetonitrile	0.2	rt	12 h	15
8	Benzene	0.2	rt	12 h	3
9	Tetrahydrofuran	0.2	rt	12 h	6
10	1,4-Dioxane	0.2	rt	12 h	3

[a] All reactions were carried out on 1.0 mmol scale of substrate with 0.2–2.0 equiv of [tetraEG(mim)$_2$][OMs]$_2$, accordingly in 4.0 ml solvent.
[b] Yield refers to the isolated product.

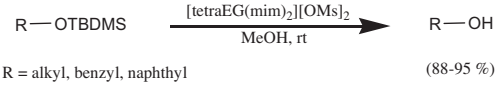
we performed catalyst free desilylation of **4** in methanol at room temperature and we observed that, this reaction proceeded very slowly and provide only 1% of **5** after 24 h (entry 1). Further, to determine the catalytic activity of [tetraEG(mim)$_2$][OMs]$_2$ at room temperature, a number of reactions were performed in methanol. Firstly, we discovered the minimum amount of [tetraEG(mim)$_2$][OMs]$_2$ required for rapid desilylation by carrying out the deprotection using 2, 1, 0.5, and 0.2 equiv of [tetraEG(mim)$_2$][OMs]$_2$ at uniform reaction condition (entries 2–5) and we found that all reactions were proceeded smoothly and provide excellent yield of benzyl alcohol within 80 min (94–95%) showing the significant catalytic activity of [tetraEG(mim)$_2$][OMs]$_2$. Considering the mole economy ratio of catalyst we selected to use 0.2 equiv of [tetraEG(mim)$_2$][OMs]$_2$ for further study of this protocol. In addition, we carried out desilylation using various solvents to study the effect of these solvents on desilylation of **4**. Entries 6 and 7 show desilylation in dichloromethane and acetonitrile were observed very sluggish and provide only 28% and 15% of desired product even extended time period after 12 h. However, solvents such as benzene, tetrahydrofuran, and 1, 4-dioxane were found to be inactive for this protocol (entries 8–10).

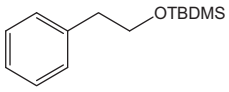
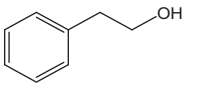
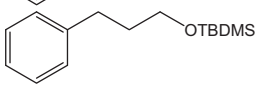
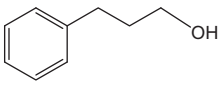
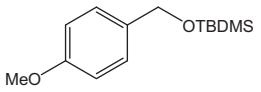
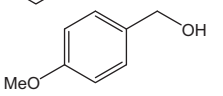
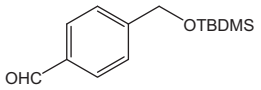
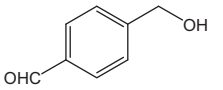
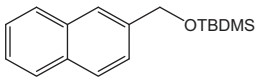
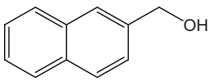
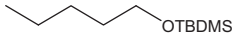
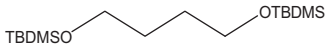
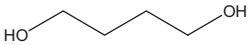
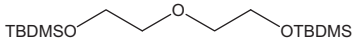
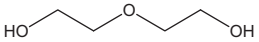
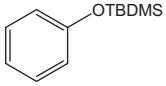
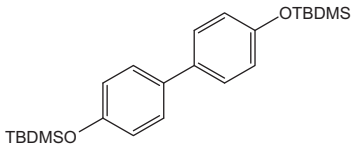
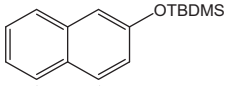
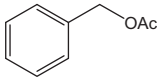
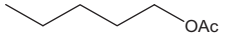
Table 2 shows desilylation of structurally varied silyl ethers of various substrates using optimized reaction condition. Deprotection of *tert*-butyl-dimethyl-phenethyloxy-silane and *tert*-butyl-dimethyl-(3-phenyl-propoxy)-silane was cleaved very cleanly and gave excellent yield of respective alcohols (entry 1, 2). Entries 3 and 4 indicate desilylation of substituted benzyl silyl ethers without affecting the substituted functionality such as methoxy and aldehyde to provide 4-methoxy benzyl alcohol and 4-hydroxymethyl-benzaldehyde in sufficient yield (92% and 89%). However, in entry 5 naphthyl silyl ether was deprotected very smoothly to get naphthyl alcohol in quantitative yield. The [tetraEG(mim)$_2$][OMs]$_2$ deprotects all aliphatic silyl ethers excellently to provide desired alcohols in significant yields (entries 6–8). Furthermore, we tried to deprotect phenolic silyl ethers using [tetraEG(mim)$_2$][OMs]$_2$ at uniform reaction condition surprisingly, all phenolic silyl ethers remain intact with phenol moieties even after prolonged reaction time 3 h (entries 9–11). In addition, acetate of benzyl alcohol and pentyl alcohol were unable to produce respective alcohol in the presence of IL at similar reaction condition (entries 12, 13). These observations encourage us to study the possibility of selective deprotection of alkyl silyl ether in the presence of phenolic silyl ether using [tetraEG(mim)$_2$][OMs]$_2$ IL as catalyst.

In addition, to go further insight to study the selective cleavage of alkyl silyl ether we performed desilylation of benzyl silyl ether (**4**) and phenolic silyl ether (**6**) in equimolar ratio in competitive reaction with 0.2 equiv of [tetraEG(mim)$_2$][OMs]$_2$ in methanol at room temperature for 3 h and this reaction results in the desilylation of benzyl silyl ether selectively and provides 100% of benzyl alcohol (**5**) whereas phenolic silyl ether (**6**) was unaffected and unable to produce phenol (**7**). Table 3 shows the results of selective cleavage of alkyl silyl ether in the presence of phenolic silyl ether of various substrates using standard condition of this protocol. Entries 1–3 provide phenolic silyl ether of different benzyl alcohols from their di-TBDMS ethers by desilylation of benzyl silyl ethers selectively in excellent yields (92–94%). Finally, Aromatic silylated kojic acid was obtained from di-silyl kojic acid by selective cleavage of benzylic silyl ether using [tetraEG(mim)$_2$][OMs]$_2$ in high yield whereas, phenolic silyl ether was unaffected (entry 4).

To compare the remarkable selectivity of dicationic IL with reported monocationic mesylate ILs, we performed desilylation of *di*-TBDMS silyl ether **8** using optimized reaction condition and the results are shown in Table 4. Entries 1 and 2 show deprotection using [hexaEGmim][OMs] and [dihexaEGim][OMs] monocationic ILs in methanol proceeded very sluggishly and provide only 42% and 36% of **9**, respectively after 12 h. Next, we carried out desilylation using potassium fluoride (KF) in MeOH to demonstrate the role of KF in

Table 2
Desilylation of various substrates using [tetraEG(mim)$_2$][OMs]$_2$ as catalyst[a]



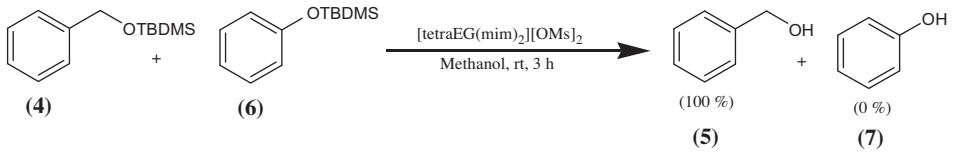
Entry	Substrate	Time	Product	Yield[b] (%)
1		80 min		91
2		80 min		95
3		80 min		92
4		80 min		89
5		80 min		89
6		75 min		94
7		90 min		88
8		85 min		91
9		3 h	No reaction	–
10		3 h	No reaction	–
11		3 h	No reaction	–
12		3 h	No reaction	–
13		3 h	No reaction	–

[a] All reactions were carried out on 1.0 mmol scale of substrate with 0.2 equiv of [tetraEG(mim)$_2$][OMs]$_2$ in 4.0 ml of methanol.
[b] Yield refers to the isolated product. All products were compared with authentic samples.

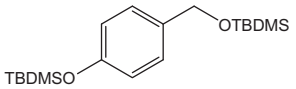
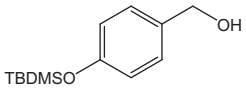
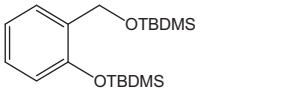
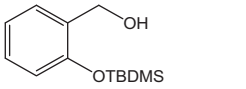
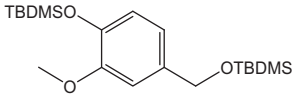
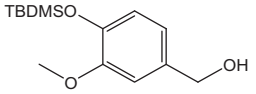
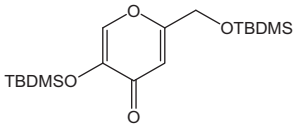
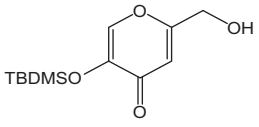
desilylation under optimized condition of this protocol, we observed that only KF (0.2 equiv) was unable to cleave any silyl ether even after prolonged reaction time 12 h (entry 3). While, KF in the presence of IL [hexaEGmim][OMs] provides 58% of **10** (entry 4). To determine the possibility of acidic pathway of silyl ether cleavage we carried out desilylation using methanesulfonic acid (MsOH) in methanol, this reaction cleaved both silyl ethers efficiently in 80 min and provides 94% of **11**. Finally, to confirm the role of MeOH in optimized condition, we carried out deprotection using IL [tetraEG(mim)$_2$][OMs]$_2$ as a solvent in the absence of MeOH and this reaction was unable to produce any alcohol even after 12 h. These reactions results clearly indicate that the desilylation occurs by acidic pathway and [tetraEG(mim)$_2$][OMs]$_2$/methanol system controlled acidity to result in the selective deprotection of alkyl silyl ether in the presence of phenolic silyl ether.

In conclusion, we have developed a mild, efficient, simple, and selective method for the deprotection of alkyl TBDMS ether in the presence of phenolic TBDMS ether using [tetraEG(mim)$_2$][OMs]$_2$ dicationic IL as a catalyst in methanol at room temperature. Furthermore, other functional groups such as aldehyde, acetate, and methoxy were found to be unaffected using this protocol. The present catalytic system [tetraEG(mim)$_2$][OMs]$_2$/methanol showed higher yield of alcohols in short reaction period, green catalytic system, easy work up environmentally favorable reaction procedure, etc., and therefore, this method has advantages over other desilylation methods.

Table 3
Selective desilylation of various substrates using [tetraEG(mim)$_2$][OMs]$_2$ as catalyst[a]

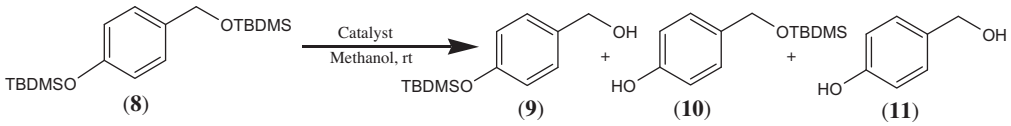


(4) (6) (5) (7)

Entry	Substrate	Time	Product	Yield[b](%)
1		85 min		94
2		85 min		92
3		80 min		94
4		70 min		91

[a] All reactions were carried out on 1.0 mmol scale of substrate with 0.2 equiv of [tetraEG(mim)$_2$][OMs]$_2$ in 4.0 ml of methanol.
[b] Yield refers to the isolated product.

Table 4
Comparison of dicationic [tetraEG(mim)$_2$][OMs]$_2$ IL with reported monocationic mesylate IL in TBDMS deprotection[a]



(8) (9) (10) (11)

Entry	Solvent	Catalyst	Time	Yield[b] (%)		
				9	**10**	**11**
1	Methanol	[hexaEGmim][OMs]	12 h	42	—	—
2	Methanol	[dihexaEGim][OMs]	12 h	36	—	—
3	Methanol	KF	12 h	—	—	—
4	Methanol	[hexaEGmim][OMs]+KF	12 h	—	58[c]	—
5	Methanol	MsOH	80 min	—	—	94
6	[tetraEG(mim)$_2$][OMs]$_2$	—	12 h	—	—	—

[a] All reactions were carried out on 1.0 mmol scale of substrate with 0.2 equiv of catalyst in 4.0 ml of methanol at room temperature.
[b] Yield refers to the isolated product.
[c] 0.2 equiv of KF.

Preparation of tetra ethylene glycol-bis (3-methylimidazolium) dimesylate ([tetraEG(mim)$_2$][OMs]$_2$)

A mixture of tetra ethylene glycol dimesylate (1.0 mmol) and *N*-methylimidazole (2.0 mmol) in acetonitrile was refluxed magnetically for 4 days in a two necked round bottomed flask. The reaction progress was monitored by TLC. After completion of the reaction as indicated by TLC, the reaction mixture was allowed to cool at room temperature and the acetonitrile was evaporated under reduced pressure on rotary evaporator at 55 °C. The reaction mixture was washed three times using ethyl acetate to remove unreacted starting materials and the resulting quaternized tetra ethylene glycol-bis (3-methylimidazolium) dimesylate was obtained.

Yield 87%; colorless thick liquid; ^{1}H NMR (500 MHz, $CDCl_3$): δ: 9.66 (s, 2 × H), 7.62 (s, 2 × H), 7.58 (s, 2 × H), 4.50 (t, 2 × 2H), 4.01 (s, 2 × 3H), 3.90 (t, 2 × 2H), 3.65 (t, 2 × 2H), 3.59 (t, 2 × 2H), 2.76 (s, 2 × 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ: 138.00, 123.32, 123.27, 70.43, 70.34, 69.11, 49.51, 39.83, 36.30; FT-IR (400–4000 cm^{-1}): 3096 [ν(Ar–H)]; 2973, 2829 [ν(C–H)]; 1612 [ν(C=N)]; 1568, 1526, 1458 [ν(C=C)]; 1183, 1073 [ν(S=O)]; 1136 [ν(C–N)]. LRMS-ESI: *m/z* [M-OMs]$^+$ calcd: 419.20; found: 419.19. Elem. Anal. Calcd (%) for $C_{18}H_{34}N_4O_9S_2$: C, 42.01; H, 6.66; N, 10.89. Found: C, 41.85; H, 6.42; N, 10.63.

General procedure for deprotection of TBDMS ether using [tetraEG(mim)$_2$][OMs]$_2$

A mixture of TBDMS ether (1.0 mmol) and [tetraEG(mim)$_2$][OMs]$_2$ IL (0.2 mmol) in methanol (4 mL) was stirred at room temperature up to the completion of reaction. Reaction progress was monitored by thin layer chromatography (TLC). After

completion of the reaction, methanol was removed under reduced pressure and the residue was extracted with diethyl ether and dried over sodium sulfate. The diethyl ether was evaporated under reduced pressure to afford respective products.

^{1}H and ^{13}C NMR data of products in Table 3

[4-(tert-Butyl-dimethyl-silanyloxy)-phenyl]-methanol (Table 3, entry 1)

^{1}H NMR (500 MHz, $CDCl_3$): δ: 0.19 (s, 6H), 0.98 (s, 9H), 1.72 (s, H), 4.59 (s, 2H), 6.82 (d, 2H), 7.23 (d, 2H). ^{13}C NMR (125 MHz, $CDCl_3$): δ: 4.33, 18.29, 25.76, 65.19, 120.26, 128.65, 133.77, 155.38.

[2-(tert-Butyl-dimethyl-silanyloxy)-phenyl]-methanol (Table 3, entry 2)

^{1}H NMR (500 MHz, $CDCl_3$): δ: 0.26 (s, 6H), 1.02 (s, 9H), 2.13 (s, H), 4.67 (s, 2H), 6.82 (d, H), 6.95 (m, H), 7.17 (m, H), 7.29 (m, H). ^{13}C NMR (125 MHz, $CDCl_3$): δ: −4.07, 18.26, 25.82, 62.11, 118.48, 121.41, 128.73, 128.96, 131.52, 153.60.

[4-(tert-Butyl-dimethyl-silanyloxy)-3-methoxy-phenyl]-methanol (Table 3, entry 3)

^{1}H NMR (500 MHz, $CDCl_3$): δ: 0.14 (s, 6H), 0.99 (s, 9H), 1.72 (s, H), 3.81 (s, 3H), 4.60 (s, 2H), 6.81 (m, 2H), 6.89 (s, H). ^{13}C NMR (125 MHz, $CDCl_3$): δ: −4.56, 18.54, 25.80, 55.52, 65.51, 111.25, 119.62, 120.89, 134.52, 144.69, 151.14.

5-(tert-Butyl-dimethyl-silanyloxy)-2-hydroxymethyl-pyran-4-one (Table 3, entry 4)

^{1}H NMR (500 MHz, $CDCl_3$): δ: 0.00 (s, 6H), 0.81 (s, 9H), 1.45 (s, H), 4.37 (s, 2H), 6.45 (s, H), 7.14 (S, H). ^{13}C NMR (125 MHz, $CDCl_3$): δ: −5.39, 18.33, 26.01, 61.46, 108.77, 136.94, 145.50, 168.73, 174.30.

Acknowledgements

This study was supported by a Grant (#10035574) from the "Platform Chemical Process Technology from Lignocellulosic Biomass Conversion" R&D Program funded by the Ministry of Knowledge Economy and by a grant support by priority Research Centers Program through the National Research Foundation of Korea (NRF) funded by the Ministry of Education, Science and Technology (2011-0022968), Republic of Korea.

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