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COMMUNICATION

Chemoselective Deoxygenation of Ether-substituted Alcohols and Carbonyl Compounds by $B(C_6F_5)_3$-catalyzed Reduction with $(HMe_2SiCH_2)_2$

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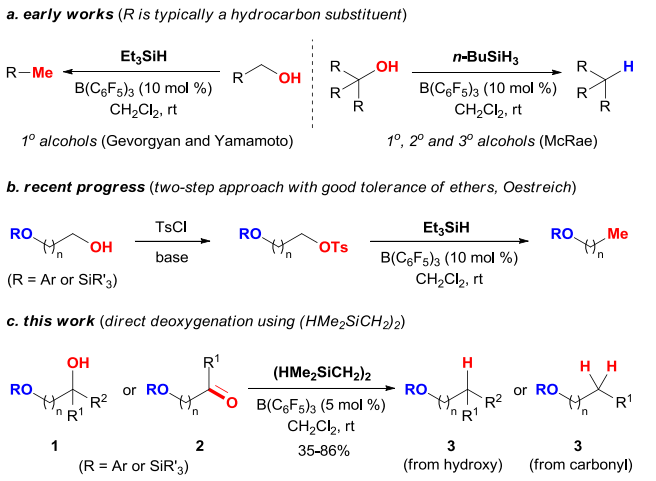
$B(C_6F_5)_3$-catalyzed deoxygenation of ether-substituted alcohols and carbonyl compounds has been developed using $(HMe_2SiCH_2)_2$ as the reductant. This unique reagent shows distinct superiority to traditional one silicon-centered hydrosilanes, giving the corresponding alkanes in high yields with good tolerance of ethers, aryl halides and alkenes. The control experiments suggest that $(HMe_2SiCH_2)_2$ might facilitate the approach by an intramolecular Si/O activation manner.

Cleavage of the Csp^3-O bond in alcohols to give alkanes is an important transformation in organic synthesis.[1] For example, it allows a promising process to convert readily available cellulosic biomass into hydrocarbons or partially oxygenated hydrocarbons, which are major components in many feedstocks and fuels.[2] Alcohol deoxygenation would be particularly useful in synthetic chemistry if the hydroxy groups could be removed selectively in the presence of other functional groups such as ethers and alkenes. However, achieving such selectivity issue still remains as a long-standing challenge.

Traditional approaches for directly removing hydroxyl groups using hydrosilanes as the reductant requires at least stoichiometric amount of strong Lewis acids such as BF_3•OEt_2.[3] The substrates are usually limited to benzylic, allylic, tertiary alcohols or what can generate a stable carbocation species. Thus, the S_N1 mechanism has been recognized as a representative model for this approach. Gevorgyan, Yamamoto and co-workers[4] made an important breakthrough on developing the catalytic process. They found that 10 mol % of tri(pentafluorophenyl)-borane $[B(C_6F_5)_3]$[5, 6, 7] showed excellent catalytic efficiency for direct cleavage of the Csp^3-O bond in alcohols using Et_3SiH at room temperature (Scheme 1a, left). The reaction was suggested to proceed by a S_N2-like mechanism based on the ability of $B(C_6F_5)_3$ to activate Si–H bonds through η^1



Scheme 1. a) Early works: R is typically a hydrocarbon substituent; b) Oestreich' two-step approach via tosylates; c) direct deoxygenation using $(HMe_2SiCH_2)_2$ with good tolerance of ethers.

coordination.[8] While Gevorgyan and Yamamoto's protocol shows good efficiency for primary alcohols, deoxygenation of secondary and tertiary alcohols proved ineffective to give the corresponding alkanes. The reaction suspended at formation of the triethylsilyl ethers, which are too sterically hindered to react further with Et_3SiH–$B(C_6F_5)_3$. McRae and co-workers[9] improved this methodology by using less hindered *n*-$BuSiH_3$, leading to facile deoxygenation of secondary and tertiary alcohols (Scheme 1a, right). However, few examples showed the functional group tolerance in both Gevorgyan/Yamamoto's and McRae's approaches. To this end, Oestreich and co-workers[10] recently made an elegant improvement, despite by a two-step deoxygenation process (Scheme 1b). The key of their approach relied on transforming the primary hydroxyl group first into the more reactive tosylate. This group shows preference to react with Et_3SiH–$B(C_6F_5)_3$ in the presence of primary and secondary silyl ethers and aryl ethers, enabling chemoselective defunctionalization of 1,n-diols and deoxygenation of the hydroxy methyl group of an orthogonally protected carbohydrate.

As part of our continuing interests in bis(silyl) chemistry,[11] we recently launched an investigation to explore the synthetic values of

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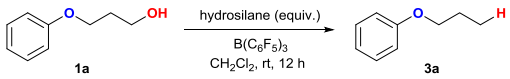
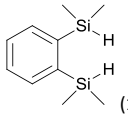
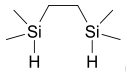
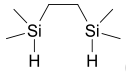
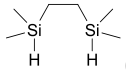
$(HMe_2SiCH_2)_2$.[12] This reagent can be easily accessible from commercially available $(ClMe_2SiCH_2)_2$ by reduction with $LiAlH_4$ (83% yield on gram-scale).[13] We discovered that $(HMe_2SiCH_2)_2$showed superiority to traditional one silicon-centered hydrosilanes (R_nSiH_{4-n}) in the $B(C_6F_5)_3$-catalyzed reduction–lactonization of keto acids to form γ- and δ-lactones. Herein, we report a new utility of $(HMe_2SiCH_2)_2$ for directly removing the hydroxyl group, which does not require pre-activation, in the presence of a range of silyl ethers, aryl ethers, aryl halides and alkenes (Scheme 1c). The approach is also expanded to directly reduce carbonyl compounds to alkanes with equally good chemoselectivity.

Table 1 Screening of Reaction Conditions.[a]



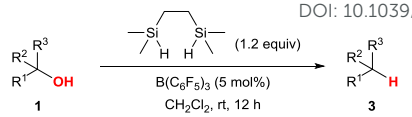
1a 3a

Entry	$B(C_6F_5)_3$	hydrosilanes (equiv.)	Yield[b] % (conv.%)
1	5 mol %	Et_3SiH (2.5)	0 (100)
2	5 mol %	$EtMe_2SiH$ (2.5)	0 (90)
3	5 mol %	Et_2SiH_2 (1.2)	58 (100)
4	5 mol %	$PhSiH_3$(0.8)	45 (100)
5	5 mol %	$HMe_2SiSiMe_2H$ (1.2)	0 (30)
6	5 mol %	$HMe_2SiOSiMe_2H$ (1.2)	0 (90)
7	5 mol %	 (1.2)	0 (25)
8	5 mol %	 (1.2)	70 (100)
9	1 mol %	 (1.2)	43 (67)
10	10 mol %	 (1.2)	57 (100)

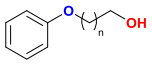
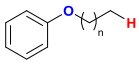
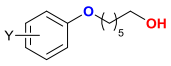
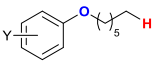
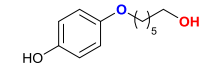
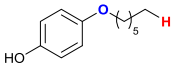
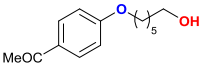
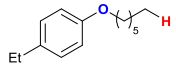
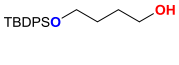
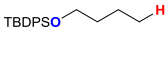
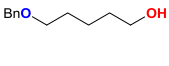
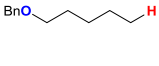
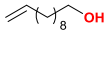
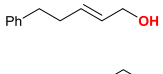
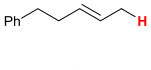
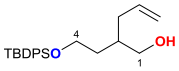
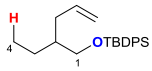
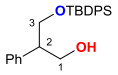
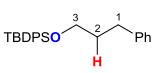
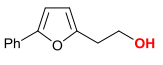
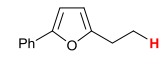
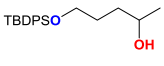
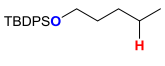
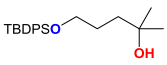
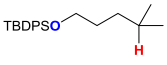
[a]Reaction conditions: 0.2 mmol of **1a**, $B(C_6F_5)_3$ and hydrosilane in dry CH_2Cl_2 (4 mL) at rt for 12 h. [b] Yield was determined by ^1H NMR analysis using dibromomethane as an internal standard.

The reaction was examined using a terminal phenyl ether-substituted alcohol **1a** as the model substrate and 5 mol % of $B(C_6F_5)_3$ as catalyst in CH_2Cl_2 at room temperature. **1a** was consumed completely by reduction with Et_3SiH, but the phenyl ether moiety rather than the free hydroxyl group was removed (Table 1, entry 1). Formation of the triethylsilyl phenol ether was evident by the ^1H NMR of the crude product. This result was consistent with Gevorgyan and Yamamoto's observation that the initially formed triethylsilyl ether was too bulky to remove by further reaction with Et_3SiH–$B(C_6F_5)_3$.[4] Using sterically less hindered $EtMe_2SiH$ provided similar results (entry 2). Multi-hydride-substituted hydrosilanes such as Et_2SiH_2 and $PhSiH_3$ appeared more effective than Et_3SiH to provide the desired **3a**, but the yields were only moderate (entries 3 and 4). Then we turned our attention to hydrosilanes containing two silicon centers. While $HMe_2SiSiMe_2H$ provided only 30% conversion affording the silyl ether without further reduction (entry 5), $HMe_2SiOSiMe_2H$ showed similar reactivity as Et_3SiH leading to the undesired phenyl ether cleavage (entry 6). Next we tested the disilane containing a phenyl ring as the linkage (entry 7). Unfortunately, the reaction was retarded as that using HMe2SiSiMe2H. To our delight, the conformationally more flexible $(HMe_2SiCH_2)_2$ led to complete consumption of **1a**, generating the desired deoxygenation product **3a** in 70% yield (entry 8). The loading of $B(C_6F_5)_3$ also showed great impact on the

Table 2 Scope of Alcohols.[a]

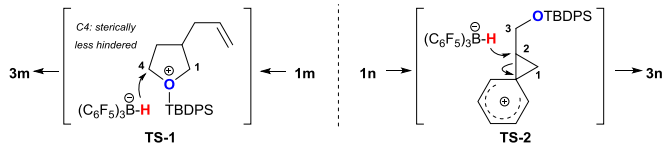


1 3

Entry	Substrate		Product		Yield (%)[c]
1		**1a** (n = 2)		**3a**	70[d]
2		**1b** (n = 1)		**3b**	62[d]
3		**1c** (Y = 4-Cl)		**3c**	81
4		**1d** (Y = 3-Br)		**3d**	82
5		**1e** (Y = 4-CF_3)		**3e**	76
6		**1f** (Y = 4-OMe)		**3f**	59
7[b]		**1g**		**3g**	36[d]
8[b]		**1h**		**3h**	80
9		**1i**		**3i**	84
10		**1j**		**3j**	N.D.
11		**1k**		**3k**	73[e]
12		**1l**		**3l**	80[d]
13		**1m**		**3m**	73
14		**1n**		**3n**	70[d]
15		**1o**		**3o**	N.D.
16		**1p**		**3p**	35
17		**1q**		**3q**	65
18		**1r**		**3r**	71

[a] Reactions conditions: 0.2 mmol of **1**, 0.24 mmol of $(HMe_2SiCH_2)_2$ and $B(C_6F_5)_3$ (5 mol%) in dry CH_2Cl_2 (4 mL) at rt for 12 h. [b] $(HMe_2SiCH_2)_2$(2.4 equiv). [c] Isolated yields after purification by silica gel column chromatography. [d] Yields were determined by ^1H NMR analysis using dibromomethane as an internal standard. [e] Yields were determined by ^1H NMR analysis using 1,3,5-trimethoxybenzene as an internal standard.

reduction efficiency. While 1 mol % of $B(C_6F_5)_3$ was insufficient to give a complete conversion (entry 9), 10 mol % of $B(C_6F_5)_3$ appeared to cause some side-reactions such as elimination of hydroxyl or phenyl ether, leading to a lower yield of 57% (entry 10).

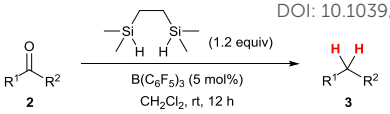


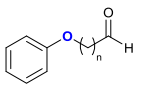
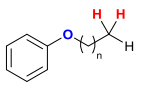
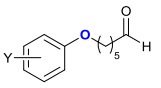
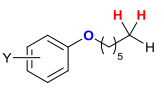
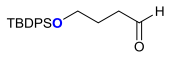
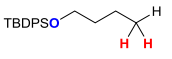
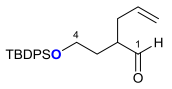
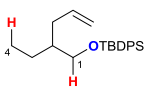
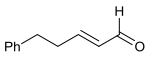
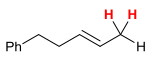
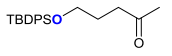
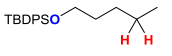
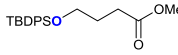
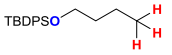
Scheme 2. Mechanism of the rearrangement during transformation of **1m** and **1n** into **3m** and **3n**, respectively.

With the optimal reaction conditions in hand, the scope of our approach was explored. The reaction tolerated a range of terminal aryl ether-substituted alcohol **1a-1h**, giving deoxygenated products **3a-3h** in generally good yields (Table 2, entry 1-8). The aryl bromide moiety in **1d**, which typically cannot survive in transition-metal catalyzed[14] or radical reduction, was tolerated. Reaction of **1e** possessing an electron-withdrawing CF_3 group afforded **3e** in 76% yield. In a sharp contrast, Oestreich showed that reducing the tosylate of **1e** with Et_3SiH only provided a low yield of 30%.[10] In addition, the methoxy group, which is prone to undergo facile Me-O bond cleavage,[10] did not interfere with our approach (entry 6). The free hydroxyl group in **1g** was also compatible, despite **3g** was delivered in 36% yield (entry 7). It was noteworthy that the aryl ether in **1h** was fully untouched, even though the hydroxyl and ketone groups were reduced to methyl and ethyl groups, respectively (entry 8). TBDPSO-substituted **1i** functioned well to give **3i** in 84% yield. But the reaction of **1j** was complex, indicating that the Bn-O bond was vulnerable to the approach. Both terminal and internal alkenes were tolerated (entries 11 and 12); no double bond shift was observed in the reduction of allylic alcohol **1l**. Rearrangement was observed in the reactions of two branched substrates **1m** and **1n**, respectively (entries 13 and 14). Cyclic silyloxonium ion **TS-1**[2e] was proposed to form by intramolecular attack of the C4–OTBDPS group onto the activated hydroxyl at C1 in **1m**. The subsequent reduction at the sterically less hindered C4 position led to **3m**. Formation of **3n** might proceed by C2-reduction of the phenonium ion **TS-2**[10], which was generated by a semi-pinacol like rearrangement of **1n** (Scheme 2). While **1o** containing a tetrahydrofuran ring decomposed (entry 15), reaction of **1p** possessing a furan gave the desired **3p** despite in a moderate yield of 35% (entry 16). These results indicate the limitation of our approach that substrates containing a saturated *O*-heterocycle might not be a good choice. The approach works well with secondary and tertiary alcohols (entries 17 and 18), but the yields (65% and 71%) were lower than that obtained using primary alcohol (84%, entry 9). We observed 10-15% of the corresponding silyl ether, which did not undergo further deoxygenation.

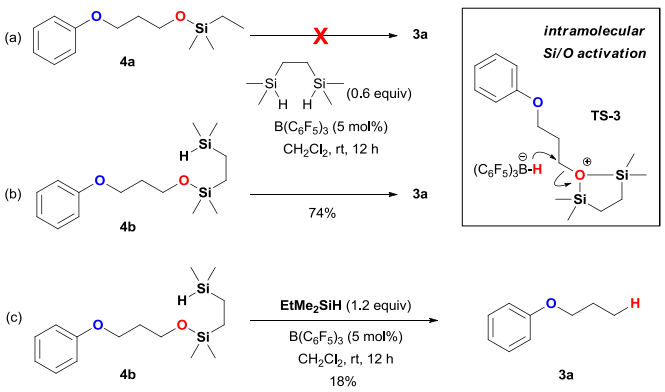
The success of deoxygenation of ether-substituted alcohols led to us to examine the corresponding carbonyl compounds. As shown in Table 3, the aldehyde moiety in all of the substrates was reduced chemoselectively into the methyl group in high yields without touching either ether or alkene (entries 1-10). Ketone **2k** served as a good substrate as well (entry 11). Thus the approach could be complementary to traditional methods for aldehyde reduction such as Wolf-Kishner-Huang reduction,[15] which generally requires basic and hash conditions. In addition, the ester group in **2l** can be also fully deoxygenated with good chemoselectivity, despite 1.8 equiv of $(HMe_2SiCH_2)_2$ were required to give **3i** in 81% yield (entry 12).

Table 3 Scope of Carbonyl Compounds.[a]



Entry	Substrate		Product		Yield (%)[c]
1		**2a** n = 2		**3a**	68[d]
2		**2b** n = 1		**3b**	65[d]
3		**2c** (Y = 4-Cl)		**3c**	80
4		**2d** (Y = 3-Br)		**3d**	87
5		**2e** (Y = 4-CF_3)		**3e**	73
6[b]		**2f** (Y = 4-OMe)		**3f**	61
7		**2g** (Y = 4-COMe)		**3h** (Y = C_2H_5)	78
8		**2h**		**3i**	86
9		**2i**		**3m**	74
10		**2j**		**3l**	70[d]
11		**2k**		**3q**	69
12[e]		**2l**		**3i**	81

[a] Reactions conditions: 0.2 mmol of **2**, 0.24 mmol of $(HMe_2SiCH_2)_2$ and $B(C_6F_5)_3$ (5 mol%) in dry CH_2Cl_2 (4 mL) at rt for 12 h. [b] $(HMe_2SiCH_2)_2$ (2.4 equiv). [c] Isolated yields after purification by silica gel column chromatography. [d] Yields were determined by ^1H NMR analysis using dibromomethane as an internal standard. [e] $(HMe_2SiCH_2)_2$ (1.8 equiv).



Scheme 3. Control experiments using **4a** and **4b**.

In our previous work, we have suggested that $(HMe_2SiCH_2)_2$ might facilitate an intramolecular Si/ketone activation manner that makes $(HMe_2SiCH_2)_2$ superior to one silicon-centered hydrosilanes for reduction/lactonization of keto acids.[12] To examine whether a similar activation manner as shown by **TS-3** is involved in the deoxygenation of alcohols, we performed the control experiments using **4a** and **4b** (Scheme 3). Despite the silyl ether moieties in **4a** and **4b** are sterically similar to each other, only **4b** is potentially capable of an intramolecular Si/O activation. As expected, removal of the phenyl ether was observed as the predominant path in the reaction of **4a**, while that of **4b** gave the desired **3a** in 74% yield. In addition, the reaction of **4b** with $EtMe_2SiH$ indeed gave **3a**, but only in 18% yield. The contrasting results from these two reactions using **4b** suggested that in addition to the proposed intramolecular Si/O activation, $(HMe_2SiCH_2)_2$ should play other important roles for promoting the deoxygenation approach. More detailed studies are currently underway.

In conclusion, $(HMe_2SiCH_2)_2$ has been used as a useful reagent for $B(C_6F_5)_3$-catalyzed chemoselective deoxygenation of ether-substituted alcohols and carbonyl compounds. The approach shows good tolerance of ether, aryl halide and alkene, giving the corresponding alkane in good yields. $(HMe_2SiCH_2)_2$ has been proposed to facilitate an intramolecular Si/O activation, making this reagent superior to traditional one silicon-centered hydrosilanes. More detailed studies and applications of this approach are currently underway.

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Chemoselective Deoxygenation of Ether-substituted Alcohols and Carbonyl Compounds by $B(C_6F_5)_3$-catalyzed Reduction with $(HMe_2SiCH_2)_2$

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$B(C_6F_5)_3$-catalyzed deoxygenation of ether-substituted alcohols and carbonyl compounds has been developed using $(HMe_2SiCH_2)_2$ as the reductant. This unique reagent shows distinct superiority to traditional one silicon-centered hydrosilanes, giving the corresponding alkanes in high yields with good tolerance of ethers, aryl halides and alkenes. The control experiments suggest that $(HMe_2SiCH_2)_2$ might facilitate the approach by an intramolecular Si/O activation manner.