An Efficient and Highly Selective Deprotecting Method for *β*-(Trimethylsilyl)ethoxymethyl Ethers

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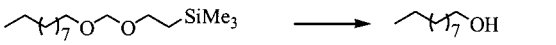
Abstract: A series of *β*-(trimethylsilyl)ethoxymethyl ethers were hydrolyzed to their corresponding alcohols in high yields by using a catalytic amount of CBr_4 (15%) in MeOH under refluxing reaction conditions. The chemoselective deprotection between trialkylsilyl and *β*-(trimethylsilyl)-ethoxymethyl-protected alcohols can be achieved by using an alcohol with steric hindrance such as *i*PrOH. The selectivity also can be achieved in the CBr_4/MeOH reaction mixture under ultrasonic reaction conditions.

Selective introduction and removal of protective groups is an important tool in organic synthesis.[1] Transformation of alcohols into their corresponding ethers has been recognized as a common and useful method for protection of the hydroxy group.[2,3] The *β*-(trimethylsilyl)ethoxymethyl ($Me_3SiCH_2CH_2OCH_2$; SEM) ether has been widely employed as an effective protective group for alcohols since 1980.[4−6] The *β*-(trimethylsilyl)ethoxymethyl group is typically deprotected by using a fluoride ion[7] such as nBu$_4$NF,[4a,b,8] $LiBF_4$,[9] and CsF,[10] by strong acids such as H_2SO_4[11] and CF_3CO_2H,[12] or by Lewis acids such as BF_3[13] and $MgBr_2$.[14]

Our previous studies showed that acetals/ketals,[15] tetrahydropyranyl ethers,[16] trialkylsilyl ethers,[17] methoxymethyl ethers, and methoxyethoxymethyl ethers[18] can be hydrolyzed by using a catalytic amount of CBr_4 in methanol or 2-propanol under thermal or ultrasonic reaction conditions. The success of such deprotections relies on the in situ generation of HBr which provides a mild but sufficient anhydrous acidic reaction condition.[18−20] Therefore, we expected that this reaction system can be applied to the deprotection of SEM-protected alcohols. Herewith, we wish to report a simple and an efficient hydrolyzing method for *β*-(trimethylsilyl)ethoxymethyl ethers by using the protocol of a CBr_4/ROH reaction system. A highly selective deprotection method between trialkylsilyl and *β*-(trimethylsilyl)ethoxymethyl functionalities also was investigated and explored.

Scheme 1



Entry	Reaction Conditions	Yields	Recovery of S.M.
1	CBr_4 / MeOH (10% / 10 mL), reflux, 2 h	55%	40%
2	CBr_4 / MeOH (15% / 10 mL), reflux, 2 h	64%	33%.
3	CBr_4 / MeOH (20% / 10 mL), reflux, 2 h	72%	24%
4	CBr_4 / MeOH (20% / 10 mL), reflux, 10 h	93%	<3%
5	CBr_4 / *i*-PrOH (10% / 10 mL), reflux, 2 h	90%	<5%
6	CBr_4 / *i*-PrOH (15% / 10 mL), reflux, 2 h	91%	<3%
7	CBr_4 / *i*-PrOH (20% / 10 mL), reflux, 2 h	92%	<3%
8	CBr_4 / MeOH (10% / 10 mL), *hν*, 1 h ; rt, 19 h	86%	10%
9	CBr_4 / MeOH (15% / 10 mL), *hν*, 1 h ; rt, 19 h	94%	<3%
10	CBr_4 / MeOH (20% / 10 mL), *hν*, 1 h ; rt, 19 h	95%	
11	CBr_4 / *i*-PrOH (15% / 10 mL), *hν*, 1 h ; rt, 19 h	74%	22%
12	CBr_4 / MeOH (10% / 10 mL), ultasound, 1.5 h	<5%	93%
13	CBr_4 / MeOH (15% / 10 mL), ultrasound, 2 h	15%	82%
14	CBr_4 / MeOH (15% / 10 mL), ultrasound, 6 h	43%	21%

We first investigated the reaction condition for hydrolysis of *β*-(trimethylsilyl)ethoxymethyl ether. The deprotection proceeded smoothly by treatment of CH_3-$(CH_2)_7$O-SEM with a catalytic amount of CBr_4 in MeOH or *i*PrOH under thermal or photochemical reaction conditions (Scheme 1). Our investigations showed that 2-propanol was the most effective solvent for SEM-ether hydrolysis under refluxing conditions and methanol is the solvent of choice for photochemical reaction condi-

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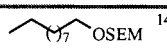
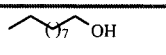
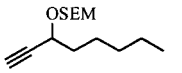
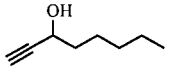
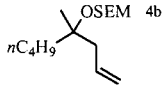
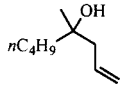
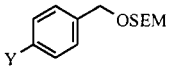
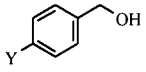
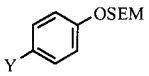
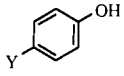
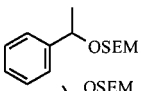
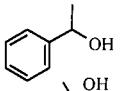
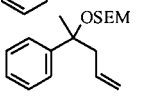
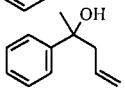
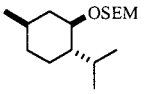
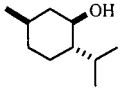
Table 1. Hydrolysis of SEM-ethers in a CBr_4/ROH Reaction Mixture

Entry	Substrate	Product	Methods[a]	Y	Time(h)	Yield(%)[b]
1	 14a		A		2	91
			B		19	94
2			A		2	91
			B		20	82
3	 4b		A		1	64[c]
			B		22	81
4a			A	H	1.5	94
			B		19	95
4b			A	NO_2	1	90
			B		19	87
4c			A	Cl	2	93
			B		21	90
4d			A	MeS	1.5	85
			B		23	83
4e			A	NMe_2	1	50(42)[d]
			B		21	43(40)[e]
4f			A	MeO	1	12(85)[d]
			B		22	5(90)[e]
5a			A	MeO	1	95
			B		24	99
5b			A	NO_2	1	96
			B		20	92
5c			A	CHO	1	91
			B		20	86
6			A		2	90
			B		21	84
7			A		1	68[c]
			B		23	80
8			A		2	91
			B		22	93

[a] Method A: CBr_4/*i*PrOH, reflux. Method B: CBr_4/MeOH, *hv*; stirred at room temperature without irradiation. [b] The isolated yield after chromatographic purification. [c] The yield was low due to the formation of elimination product. [d] The number in parentheses indicates the yield of isopropyl ether. [e] The number in parentheses indicates the yield of methyl ether.

tions. It should be noted that β-(trimethylsilyl)ethoxymethyl ether was resistant with a 10% CBr_4/MeOH reaction mixture when ultrasound was used as energy source (entry 12, Scheme 1).

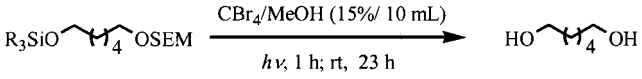
A series of β-(trimethylsilyl)ethoxymethyl ethers were prepared[4a] and investigated under the method A or method B reaction conditions (Table 1). The results showed that primary, secondary, benzyl, and phenolic SEM-ethers were successfully deprotected to their corresponding alcohols in high yields. Deprotection of tertiary SEM-ether was accompanied with the elimination compound under thermal reaction conditions. The undesirable elimination compound such as a conjugated olefin, 4-phenyl-1,3-pentadiene, was generated under method A reaction conditions (entry 7, Table 1). However, this problem can be overcome by using photochemical reaction conditions (method B, referring to entries 3 and 7 in Table 1). The SEM-protected 2-phenyl-4-penten-2-ol (entry 7, Table 1) was deprotected to its corresponding alcohol in 80% yield under method B reaction conditions.

The absolute configuration of (1*S*,2*R*,5*R*)-menthyl SEM-ether was retained under this type of hydrolyzing reaction condition. Hydrolysis of 4-methoxybenzyl SEM-ether and 4-(*N*,*N*-dimethylamino)benzyl SEM-ether (entries 4e and 4f in Table 1) was somewhat complicated due to the partial formation of isopropyl and methyl ethers.

The hydrolyses for trialkylsilyl ethers such as triethylsilyl, *tert*-butyldimethylsilyl, triisopropylsilyl, and *tert*-butyldiphenylsilyl ethers were also investigated with the CBr_4/MeOH reaction mixture under photochemical reaction conditions (method B). The removal of β-(trimethylsilyl)ethoxymethyl and trialkylsilyl groups was achieved under method B reaction conditions to afford the corresponding 1,6-hexanediol in moderate to high yields (Scheme 2). The 1,6-hexanediol was obtained in high yield by using a 20% CBr_4/MeOH reaction mixture under refluxing reaction conditions (Scheme 3).

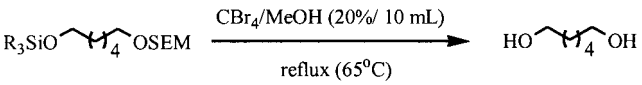
In contrast to the photochemical reaction condition, we observed that trialkylsilyl and β-(trimethylsilyl)ethoxymethyl groups can be selectively deprotected by treat-

Scheme 2



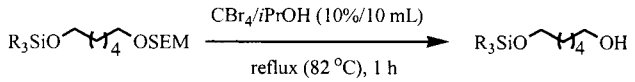
R_3Si	Yield (%)
Et_3Si	92
t-$BuMe_2Si$	87
(*i*-$Pr)_3Si$	81
t-$BuPh_2Si$	72

Scheme 3



R_3Si	Time	Yield (%)
Et_3Si	10h	98
t-$BuMe_2Si$	10h	95
(*i*-$Pr)_3Si$	18h	89
t-$BuPh_2Si$	18h	88

Scheme 4



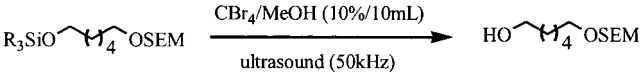
R_3Si	Yield (%)
(*i*-$Pr)_3Si$	84 (10)[a]
t-$BuPh_2Si$	90 (3)[a]

(a) The number in parenthesis indicates the yield of 1,6-hexanediol.

ment of a catalytic amount of CBr_4 in MeOH or *i*PrOH under thermal or sonochemical reaction conditions. As shown in Scheme 4, the *β*-(trimethylsilyl)ethoxymethyl group was removed selectively by treatment with 10% CBr_4 in *i*PrOH under refluxing conditions and the corresponding 6-triisopropylsilyloxy-1-hexanol and 6-(*tert*-butyldiphenyl)silyloxy-1-hexanol were produced in 84% and 90% yields individually. The *β*-(trimethylsilyl)ethoxymethyl ether was rather stable under sonochemical reaction conditions within 1.5 h (entries 12 and 13 in Scheme 1). Thus, the selective hydrolysis between *β*-(trimethylsilyl)ethoxymethyl and trialkylsilyl groups was investigated under sonication and the results are shown in Scheme 5.

In conclusion, our current studies indicated that the CBr_4/ROH reaction system provides a simple and highly efficient deprotection method for *β*-(trimethylsilyl)-ethoxymethyl ether. A simple and selective deprotecting method for multifunctionalized compounds is valuble in organic synthesis and our CBr_4/ROH reaction system for selective removal of trialkylsilyl or *β*-(trimethylsilyl)-ethoxymethyl functionalities, in a complementary manner, fulfilled this requirement. This reaction system manipulates a highly selective deprotecting reaction between trialkylsilyl or *β*-(trimethylsilyl)ethoxymethyl ethers by introducing different energy sources such as heat or ultrasound.

Scheme 5



R_3Si	Time	Yield (%)
Et_3Si	1.5h	91 (3)[a]
t-$BuMe_2Si$	1.5h	86 (6)[a]
(*i*-$Pr)_3Si$	6h	42 (31)[a]
t-$BuPh_2Si$	6h	45 (27)[a]

(a) The number in parenthesis indicates the yield of 1,6-hexanediol.

Experimental Section

Experimental Procedure for Deprotection of *β*-(Trimethylsilyl)ethoxymethyl Ether. Method A. A mixture of *β*-(trimethylsilyl)ethoxymethyl ether (1.0 equiv) and CBr_4 (0.15 equiv) in anhydrous *i*PrOH (10 mL/1.0 equiv of SEM-ether) was refluxed at 82 °C for 1−2 h. After the completion of hydrolysis which was indicated by TLC analysis, the reaction mixture was concentrated directly under reduced pressure. The desired alcohol was isolated by flash chromatography on a silica gel column (Merck 230−400 mesh) with elution of gradients of ethyl acetate/hexane. **Method B.** Alternatively, a solution of *β*-(trimethylsilyl)ethoxymethyl ether (1.0 equiv), CBr_4 (0.15 equiv), and anhydrous MeOH (10 mL/1.0 equiv of SEM-ether) in a Pyrex culture tube was irradiated by a TLC lamp (Uvltec Limited, 254 nm, 8 W) for 1 h, followed by stirring overnight without irradiation at room temperature. After the reaction was complete (by TLC), the organic solvent was removed directly under reduced pressure. Further purification was achieved by flash chromatography on a silica gel column with ethyl acetate/hexane as eluant.

Decyl *β*-(Trimethylsilyl)ethoxymethyl ether (Table 1, Entry 1). ^{1}H NMR: δ 0.00 (9H, s), 0.87 (3H, t, J = 7.0), 0.94 (2H, t, J = 8.6), 1.26−1.29 (16H, m), 1.55−1.60 (2H, m), 3.52 (2H, t, J = 6.6), 3.61 (2H, t, J = 8.6), 4.66 (2H, s). ^{13}C NMR: δ −1.4, 14.1, 18.1, 22.7, 26.2, 29.3, 29.6, 29.8, 31.9, 64.9, 67.9, 94.8.

1-Octyn-3-*β*-(trimethylsilyl)ethoxymethyl Ether (Table 1, Entry 2). ^{1}H NMR: δ 0.03 (9H, s), 0.87 (3H, t, J = 7.0), 0.88−0.94 (2H, m), 1.27−1.31 (4H, m), 1.40−1.52 (2H, m), 1.68−1.22 (2H, m), 2.35 (1H,s), 3.54 (1H, m), 3.67 (1H, m), 4.30 (1H, t, J = 6.5), 4.64 (1H, d, J = 6.9), 4.91 (1H, d, J = 6.9). ^{13}C NMR: δ −1.5, 13.9, 18.0, 22.5, 24.9, 31.4, 35.6, 65.2, 65.3, 73.1, 82.8, 92.4.

4-Methyl 5-*β*-(Trimethylsilyl)ethoxymethyl-1-octene (Table 1, Entry 3). ^{1}H NMR: δ 0.00 (9H, s), 0.87−0.93 (2H, m), 0.88 (3H, t, J = 7.0), 1.17 (3H, s), 1.26−1.33 (4H, m), 1.45−1.54 (2H, m), 2.27 (2H, d, J = 7.2), 3.61 (2H, t, J = 8.5), 4.75 (2H, s), 5.03 (2H, brd, J = 14), 5.80 (1H, m). ^{13}C NMR: δ −1.4, 14.1, 18.1, 23.1, 23.7, 38.9, 44.0, 65.0, 77.8, 89.0, 117.3, 134.5.

Benzyl *β*-(Trimethylsilyl)ethoxymethyl Ether (Table 1, Entry 4a). ^{1}H NMR: δ 0.02 (9H, s), 0.95 (2H, t, J = 8.5), 3.66 (2H, t, J = 8.5), 4.59 (2H, s) 4.73 (2H, s), 7.26−7.38 (5H, m). ^{13}C NMR: δ −1.5, 18.0, 65.1, 69.2, 94.0, 127.5, 127.8, 128.3, 138.0.

4-Nitrobenzyl *β*-(Trimethylsilyl)ethoxymethyl Ether (Table 1, Entry 4b). ^{1}H NMR: δ 0.02 (9H, s), 0.92 (2H, t, J = 8.4), 3.64 (2H, t, J = 8.4), 4.68 (2H, s) 4.75 (2H, s), 7.48 (2H, d, J = 8.6), 8.16 (2H, d, J = 8.6). ^{13}C NMR: δ −1.4, 18.0, 65.4, 68.0, 94.5, 123.4, 127.7, 145.8, 147.2.

4-Chlorobenzyl *β*-(Trimethylsilyl)ethoxymethyl Ether (Table 1, Entry 4c). ^{1}H NMR: δ 0.00 (9H, s), 0.92 (2H, t, J = 8.4), 3.62 (2H, t, J = 8.4), 4.54 (2H, s), 4.71(2H, s), 7.22−7.31

(4H, m). ^{13}C NMR: δ −1.5, 18.0, 65.2, 68.4, 94.1, 128.4, 129.1, 133.3, 136.7.

4-(Methylthio)benzyl β-(Trimethylsilyl)ethoxymethyl Ether (Table 1, Entry 4d). ^{1}H NMR: δ 0.00 (9H, s), 0.85 (2H, t, J = 8.4), 2.38 (3H, s), 3.56 (2H, t, J = 8.4), 4.46 (2H, s) 4.64 (2H, s), 7.15−7.17 (4H, m). ^{13}C NMR: δ −1.4, 16.0, 18.1, 65.2, 68.8, 94.5, 126.7, 128.5, 133.0, 135.0.

4-(*N*,*N*-Dimethylamino)benzyl β-(Trimethylsilyl)ethoxymethyl Ether (Table 1, Entry 4e). ^{1}H NMR: δ 0.00 (9H, s), 0.94 (2H, t, J = 8.4), 2.90 (6H, s), 3.63 (2H, t, J = 8.4), 4.47 (2H, s) 4.69 (2H, d, J = 14), 6.68 (2H, d, J = 9), 7.20 (2H, d, J = 9). ^{13}C NMR: δ −1.5, 18.0, 40.6, 65.0, 68.2, 94.0, 112.6, 128.4, 129.0, 150.1.

4-Methoxybenzyl β-(Trimethylsilyl)ethoxymethyl Ether (Table 1, Entry 4f). ^{1}H NMR: δ 0.00 (9H, s), 0.93 (2H, t, J = 8.4), 3.62 (2H, t, J = 8.4), 3.77 (3H, s), 4.51 (2H, s) 4.70 (2H, d, J = 14), 6.85 (2H, d, J = 11), 7.24 (2H, d, J = 11). ^{13}C NMR: δ −1.5, 18.0, 55.2, 65.1, 68.3, 93.8, 113.7, 129.5, 130.0, 159.1.

4-Methoxyphenyl β-(Trimethylsilyl)ethoxymethyl Ether (Table 1, Entry 5a). ^{1}H NMR: δ 0.02 (9H, s), 0.98 (2H, t, J = 8.4), 3.28 (3H, s), 3.28 (2H, t, J = 8.4), 5.16 (2H, s), 6.83 (2H, d, J = 14), 7.00 (2H, d, J = 14). ^{13}C NMR: δ −1.5, 18.0, 55.6, 66.0, 93.7, 114.5, 117.4, 151.5, 154.5.

4-Nitrophenyl β-(Trimethylsilyl)ethoxymethyl Ether (Table 1, Entry 5b). ^{1}H NMR: δ 0.00 (9H, s), 0.94 (2H, t, J = 8.3), 3.76 (2H, t, J = 8.3), 5.30 (2H, s), 7.10 (2H, d, J = 8.6), 8.18 (2H, d, J = 8.6). ^{13}C NMR: δ −1.4, 18.0, 67.0, 92.8, 116.0, 125.7, 143.0, 163.0.

4-β-(trimethylsilyl)ethoxymethylbenzaldehyde (Table 1, Entry 5c). ^{1}H NMR: δ 0.00 (9H, s), 0.91 (2H, t, J = 8.3), 3.45 (2H, t, J = 8.3), 5.27 (2H, s), 7.00 (2H, d, J = 8.5), 7.71 (2H, d, J = 8.5). ^{13}C NMR: δ −1.4, 18.0, 64.2, 93.5, 117.8, 129.0, 130.7, 166.0, 190.1.

2-Phenylethyl β-(Trimethylsilyl)ethoxymethyl Ether (Table 1, Entry 6). ^{1}H NMR: δ 0.03 (9H, s), 0.88−0.95 (2H, m), 1.48 (3H, d, J = 6.6), 3.54 (1H, m), 3.74 (1H, m), 4.57 (1H, d, J = 7.0), 4.67 (1H, d, J = 7.0), 4.78 (1H, q, J = 6.6), 7.25−7.35 (5H, m). ^{13}C NMR: δ −1.5, 23.6, 18.0, 23.6, 65.0, 73.9, 92.5, 126.3, 127.4, 128.3, 143.5.

1-Phenyl-2-β-(trimethylsilyl)ethoxymethyl-4-pentene (Table 1, Entry 7). ^{1}H NMR: δ 0.03 (9H, s), 0.91 (2H, t, J = 7.0), 1.64 (3H, s), 2.55−2.62 (2H, m), 3.58 (1H, m), 3.74 (1H, m), 4.66 (2H, s), 5.01 (2H, brd, J = 13), 5.65 (1H, m), 7.25−7.44 (5H, m). ^{13}C NMR: δ −1.5, 18.0, 24.0, 48.3, 65.3, 79.3, 90.3, 117.6, 126.1, 128.0, 134.0, 145.2.

(1*S*,2*R*,5*R*)-Menthyl β-(Trimethylsilyl)ethoxymethyl Ether (Table 1, Entry 8). ^{1}H NMR: δ 0.00 (9H, s), 0.76 (3H, d, J = 6.1), 0.85−1.09 (4H, m), 0.92 (6H, d, J = 6.2), 1.15−1.25 (2H, m), 1.36 (1H, m), 1.60−1.66 (2H, m), 2.08 (1H, m), 2.19 (1H, m), 3.34 (1H, ddd, J = 10.5, 10.5, 4.3), 3.56−3.68 (2H, m), 4.62 (1H, d, J = 7.1), 4.80 (1H, d, J = 7.1). ^{13}C NMR: δ −1.5, 15.9, 18.0, 21.1, 22.3, 23.0, 25.4, 31.5, 34.4, 41.4, 48.1, 65.0, 76.7, 95.2.

1-Triethylsilyloxy-6-[β-(trimethylsilyl)ethoxymethyl]hexane (Schemes 2−4). ^{1}H NMR: δ 0.00 (9H, s), 0.58 (6H, q, J = 8.1), 0.90−0.97 (2H, m), 0.93 (9H, t, J = 8.1), 1.34−1.36 (4H, m), 1.48−1.66 (4H, m), 3.51 (2H, t, J = 6.2), 3.56−3.62 (4H, m), 4.64 (2H, s). ^{13}C NMR: δ −1.5, 4.4, 6.7, 18.1, 25.7, 26.1, 29.7, 32.8, 62.8, 64.9, 67.8, 94.7.

1-*tert*-Butyldimethylsilyloxy-6-[β-(trimethylsilyl)ethoxymethyl]hexane (Schemes 2−4). ^{1}H NMR: δ 0.00 (9H, s), 0.04 (6H, s), 0.89 (9H, s), 0.96 (3H, t, J = 8.4), 1.32−1.37 (4H, m), 1.50−1.61 (4H, m), 3.52 (2H, t, J = 6.5), 3.60−3.64 (4H, m), 4.65 (2H, s). ^{13}C NMR: δ −1.5, 18.1, 18.2, 25.7, 26.0 (2C), 26.1 (3C), 29.8, 32.8 (2C), 63.2, 64.9, 67.8, 94.8.

1-Triisopropylsilyloxy-6-[β-(trimethylsilyl)ethoxymethyl]hexane (Schemes 2−4). ^{1}H NMR: δ 0.02 (9H, s), 0.94 (3H, t, J = 8.2), 1.04−1.08 (3H, m), 1.06 (18H, s), 1.36−1.40 (4H, m), 1.49−1.65 (4H, m), 3.52 (2H, t, J = 6.5), 3.58−3.69 (4H, m), 4.65 (2H, s).^{13}C NMR: δ −1.5, 12.0 (3C), 18.0 (6C), 18.1, 25.7, 26.1, 29.8, 33.0, 63.4, 64.9, 67.8, 94.8.

1-*tert*-Butyldiphenylsilyloxy-6-[β-(trimethylsilyl)ethoxymethyl]hexane (Schemes 2−4). ^{1}H NMR: δ 0.03 (9H, s), 0.96 (2H, t, J = 8.4), 1.32−1.39 (4H, m), 1.54−1.60 (4H, m), 3.52 (2H, t, J = 6.5), 3.60−3.69 (4H, m), 4.67 (2H, s), 7.26−7.42 (6H, m), 7.66−7.69 (4H, m).^{13}C NMR: δ −1.5, 14.1, 18.1, 25.7, 26.0, 26.9, 29.8, 32.5, 63.9, 64.9, 67.5, 95.1, 127.6 (4C), 129.5 (2C), 134.5 (2C), 135.6 (4C).

6-*tert*-Butyldiphenylsilyloxyyhexanol (Scheme 4).: ^{1}H NMR: δ 1.05 (9H, Si*t*BuPh$_2$, s), 1.19 (1H, br s, OH), 1.29−1.46 (4H, m), 1.50−1.65 (4H, m), 3.61 (2H, t, J = 6.6), 3.67 (2H, t, J = 6.5), 7.31−7.45 (6H, m), 7.64−7.70 (4H, m). ^{13}C NMR: δ 19.0, 25.3, 25.4, 26.7, 32.3, 32.4, 62.2, 63.7, 127.4, 129.3, 133.8, 135.3.

6-Triisopropylsilyloxyyhexanol (Scheme 4). ^{1}H NMR: δ 1.09−1.15 (21H, Si*i*Pr$_3$), 1.36−1.52 (4H, m), 1.55−1.60 (4H, m), 1.94 (1H, br s, OH), 3.54 (2H, t, J = 6.5), 3.67 (2H, t, J = 6.6) ^{13}C NMR: δ 12.3, 18.0, 26.3, 26.6, 33.2, 34.1, 63.0, 65.7.

6-[β-(Trimethylsilyl)ethoxymethyl]hexan-1-ol (Scheme 5). ^{1}H NMR: δ 0.02 (9H, s), 0.92 (2H, t, J = 8.4), 1.34−1.37 (4H, m), 1.52−1.59 (4H, m), 1.92 (1H, brs, OH), 3.51 (2H, t, J = 6.5), 3.58−3.62 (4H, m), 4.63 (2H, s). ^{13}C NMR: δ −1.5, 18.0, 25.5, 26.0, 29.6, 32.6, 62.7, 64.9, 67.7, 94.7.

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Supporting Information Available: NMR spectra for all new compounds. This material is available free of charge via the Internet at http://pubs.acs.org.

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