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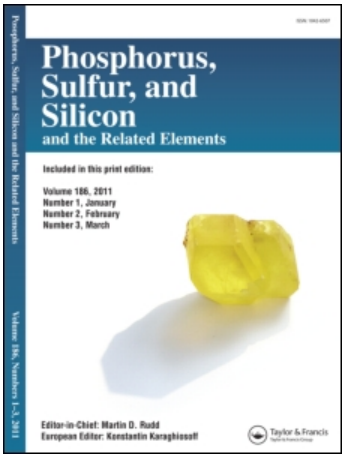


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# A Highly Efficient Method for the Silylation of Alcohols, Phenols, and Naphthols Using HMDS in the Presence of Zinc Oxide (ZnO) as Economical Heterogeneous Catalyst

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# A Highly Efficient Method for the Silylation of Alcohols, Phenols, and Naphthols Using HMDS in the Presence of Zinc Oxide (ZnO) as Economical Heterogeneous Catalyst

**Hamid Reza Shaterian and Majid Ghashang**
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*Variety alcohols, phenols, and naphthols were effectively converted into their corresponding trimethylsilyl ether with hexamethyldisilazane in the presence of zinc oxide under very mild and ambient conditions with short reaction time in good to excellent yields.*

**Keywords** Alcohols; hexamethyldisilazane; phenols; trimethylsilylation; zinc oxide

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## INTRODUCTION

Catalytic technologies play a key role in the economic development and development of the chemicals industry and add to around 20% of world Gross national product (GNP).[1] A major emerging and challenging part of heterogeneous catalysis is that of environmental pollution control, with contraction legislation on the release of waste and toxic emissions having serious implications for the chemical industry.[1] Many of these processes were focused on product yield, disregarding the environmental impact of inorganic waste and toxic by-products formed during the reaction.[1] Tightening legislation on the emission of hazardous pollutants is driving the industry toward the implementation of innovative "clean technology," including the use of alternative heterogeneously catalyzed processes.[2] In this research, ZnO as the solid heterogeneous Lewis acid catalyst deserve special mention, this catalyst is safe, easy to handle, environmentally benign, presents fewer disposal problems.

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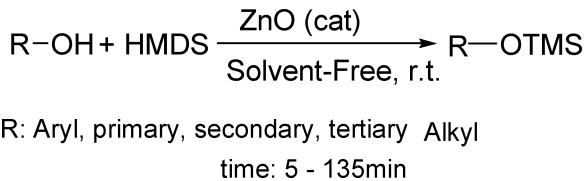
Zinc oxide has been demonstrated to be efficient catalyst for several important reactions such as Friedel-Crafts Acylation Reaction [3] and one-pot preparation of $\beta$-acetamido ketones.[4]

The preparation of silyl ethers could carry out by treatment of alcohols with silyl chlorides or silyl triflates in the presence of an organic base.[5–12] However, some of these methods frequently suffered from drawbacks such as lack of reactivity or the difficulty in removal of amine salts. 1,1,1,3,3,3-Hexamethyldisilazane (HMDS) is a stable, commercially available, and cheap reagent for trimethylsilylation of hydrogen-labile substrates, giving $NH_3$ as the only byproduct.[5–9] Even though the handling of this reagent is easy, the low silylation power of HMDS is the main drawback to its application; therefore, there are a variety of catalysts for activating of this reagent, such as $(CH_3)_3SiCl$,[13] K-10 montmorillonite,[14] sulfonic acids,[15] zirconium sulfophenyl phosphonate,[16] $ZnCl_2$,[17]Envirocat EPZGO,[18] tungstophosphoric acid,[19] iodine,[20] lithium perchlorate,[21] cupric sulfate pentahydrate,[22] H–$\beta$ zeolite,[23] $MgBr_2$,[24] lithium perchlorate supported on silica gel,[25] $Al(HSO_4)_3$,[26] $Al(OTF)_3$,[27] magnesium triflate,[28] copper triflate,[29] silica chloride,[30] and silica-$HClO_4$.[31]

In most of these cases, however, a long reaction time, drastic reaction conditions, or tedious workup is needed. In addition, many of these reagents are moisture sensitive or expensive. The lack of a facile and effective synthetic methodology for the silylation of hydroxyl groups prompted us to develop a convenient and practical procedure for the protection of hydroxyl groups in the presence of heterogeneous catalyst conditions. The use of heterogeneous catalysts under solvent-free conditions is becoming very popular as it has many advantages: reduced pollution, reusability, high selectivity, low cost, and simplicity in process and in handling. In the present research for functional group transformation, we wish to describe a new protocol for the mild and rapid trimethylsilylation of a wide variety of hydroxyl groups using HMDS and a catalytic amount of zinc oxide as solid Lewis acid catalyst. The trimethylsilylation is easily carried out at room temperature (Scheme 1).




**SCHEME 1**

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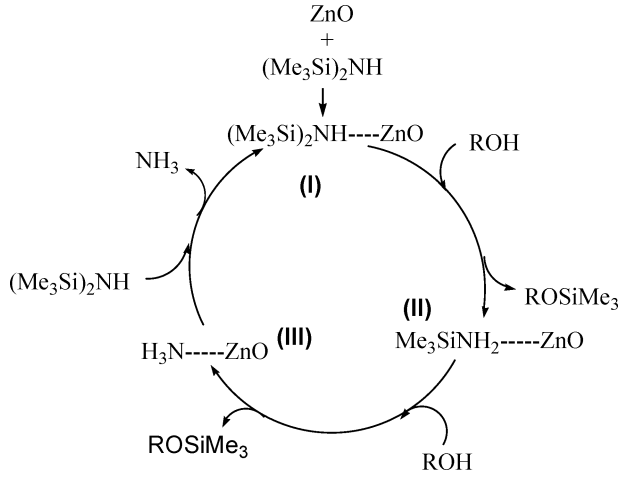
## RESULTS AND DISCUSSION

A wide range of structurally diverse and functionalized phenols and alcohols underwent silylation by this process to provide the corresponding TMS ethers in good to excellent isolated yields as shown in Table I (Entries 1–22). Phenols and benzylic alcohols mostly reacted faster than aliphatic alcohols whereas primary alcohols generally are faster than secondary and tertiary alcohols. Generally, in the all cases of benzyl, primary, secondary and tertiary alcohols the reactions were completed within less than 135 min in solvent-free condition accompanied by evolution of $NH_3$ gas from the reaction mixture. Inspection of the data in Table I clearly shows that different types of hindered secondary and tertiary alcohols were successfully converted to the corresponding silyl ethers at ambient conditions (Table I).

The suggested mechanism of the ZnO catalyzed silylation of hydroxyl groups is shown in Scheme 2.

In this mechanism, however operation process chart (OPC) of catalyst in this works is unknown nonetheless according to the observations such as evolution of $NH_3$ in the reaction conditions, we have suggested that an acid-base interaction between empty $\pi$ orbital of Zn in ZnO as catalyst and nitrogen in HMDS polarizes N—Si bond of HMDS to produce a reactive silylating agent (**I**). A rapid reaction with alcohol then ensues, leading to the ammonium silylating species (**II**) with concomitant release of the corresponding silyl ether. Irreversible cleavage of (**II**) with alcohol leading to the fast evolution of ROTMS and also formation




**SCHEME 2**

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of the unstable complexes of ZnO with ammonia (**III**). Irreversible cleavage of this complex with HMDS, leading to the fast evolution of $NH_3$. Release of ZnO as catalyst from intermediate (**III**), re-enters catalytic cycle (Scheme 2).

In conclusion, we have demonstrated that zinc oxide is a new, efficient catalyst for trimethylsilylation of a variety of hydroxyl groups using HMDS under mild and efficient conditions. This method is important from an environmental point-of-view and economic considerations because it produces little waste. It also has excellent activity on an industrial scale. Simple work-up procedure, including filtering the catalyst and purification of products using short pad column of silica gel followed evaporation of the solvent is another advantage of this method.

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## EXPERIMENTAL

All reagents were purchased from Merck and Aldrich and used without further purification. All yields refer to isolated products after purification. Products were characterized by comparison with authentic samples and by spectroscopy data (IR, $^1$H NMR spectra) and melting point. The NMR spectra were recorded on a Bruker Avance DPX 500 and 300 MHz instrument. The spectra were measured in $CDCl_3$ relative to TMS (0.00 ppm). IR spectra were recorded on a Perkin-Elmer 781 spectrophotometer. Melting points were determined in open capillaries with a BUCHI 510 melting point apparatus and are uncorrected. TLC was performed on Silica-gel polygram SIL G/UV 254 plates.

### General Procedure for Silylation of Alcohols Using HMDS Catalyzed with ZnO

To a stirred solution of alcohol (1 mmol), HMDS (0.7 mmol) ZnO (0.05 g) was added at room temperature and the mixture was stirred at appropriate time specified in Table I. The reaction was followed by TLC (n-hexane–EtOAc, 9:1). After completion of the reaction, the catalyst was filtrated and residue was passed through a short pad of silica gel. Then, the pad column was washed with n-hexane (2 × 10 mL). Evaporation of the solvent under reduced pressure gave pure product(s) (Table I).

The desired pure product(s) was characterized by comparison of their physical data with those of known compounds.[14–31] The spectral data of some representative Trimethylsilyl ethers are given below:

**TABLE I Silylation of Alcohols, Phenols and Naphthols with HMDS in the Presence of ZnO as Catalyst under Solvent-Free Condition at Ambient Conditions**

| Entry | Substrate | Product | Molar ratio Substrate/HMDS | Time (min) | Yield (%) |
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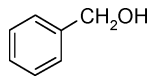
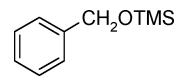
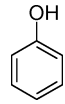
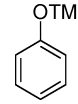
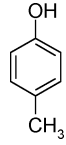
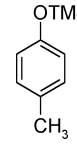
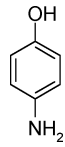
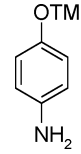
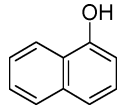
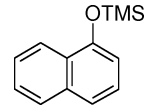
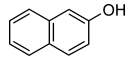
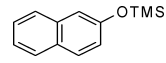
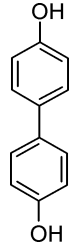
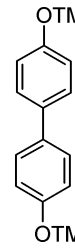
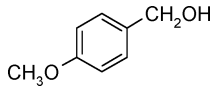
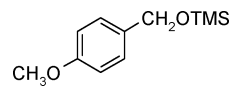
| 1 |  |  | 1/0.7 | 5 | 95 |
| 2 |  |  | 1/0.7 | 5 | 95 |
| 3 |  |  | 1/0.7 | 5 | 91 |
| 4 |  |  | 1/1.3 | 6 | 93 |
| 5 |  |  | 1/0.7 | 5 | 90 |
| 6 |  |  | 1/0.7 | 5 | 93 |
| 7 |  |  | 1/1.3 | 5 | 88 |
| 8 |  |  | 1/0.7 | 5 | 96 |

*(Continued on next page)*

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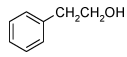
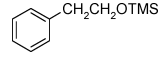
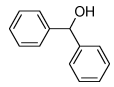
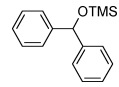
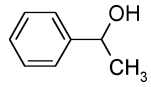
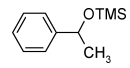
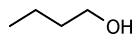
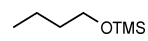
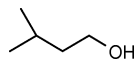
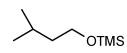
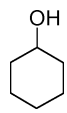
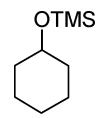
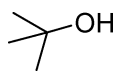
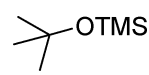
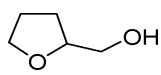
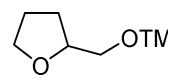
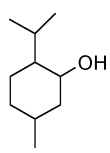
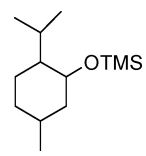
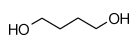
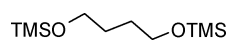
**TABLE I Silylation of Alcohols, Phenols and Naphthols with HMDS in the Presence of ZnO as Catalyst under Solvent-Free Condition at Ambient Conditions *(Continued)***

| Entry | Substrate | Product | Molar ratio Substrate/HMDS | Time (min) | Yield (%) |
|---|---|---|---|---|---|
| 9 |  |  | 1/0.7 | 11 | 92 |
| 10 |  |  | 1/0.7 | 15 | 89 |
| 11 |  |  | 1/0.7 | 7 | 89 |
| 12 |  |  | 1/0.7 | 85 | 85 |
| 13 |  |  | 1/0.7 | 89 | 87 |
| 14 |  |  | 1/0.7 | 120 | 91 |
| 15 |  |  | 1/0.8 | 131 | 89 |
| 16 |  |  | 1/0.8 | 110 | 95 |
| 17 |  |  | 1/0.7 | 125 | 95 |
| 18 |  |  | 1/1.3 | 91 | 90 |

*(Continued on next page)*

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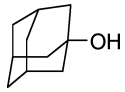
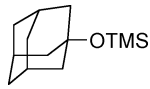
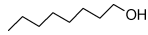
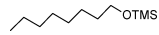
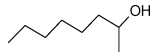
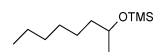
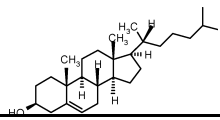
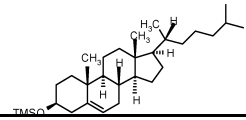
**TABLE I Silylation of Alcohols, Phenols and Naphthols with HMDS in the Presence of ZnO as Catalyst under Solvent-Free Condition at Ambient Conditions *(Continued)***

| Entry | Substrate | Product | Molar ratio Substrate/HMDS | Time (min) | Yield (%) |
|---|---|---|---|---|---|
| 19 |  |  | 1/0.7 | 135 | 94 |
| 20 |  |  | 1/0.7 | 89 | 95 |
| 21 |  |  | 1/0.7 | 121 | 93 |
| 22 |  |  | 1/0.7 | 120 | 97 |

[a]Yields refer to the pure isolated products. All known products have been reported previously in the literature and were characterized by comparison of IR and $^1$H NMR spectra with authentic samples.[14–31]

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### *Trimethyl(benzyloxy) Silane (Entry 1, Table I)*

$^1$H NMR ($CDCl_3$ 500 MHz): $\delta = 7.36$–$7.35$ (5H, m), 4.72 (2H, s), 0.19 (9H, s), ppm; IR ($CCl_4$): 3050, 2957, 1496, 1454, 1377, 1250, 1207, 1096, 1027, 842, 727, 695 $cm^{-1}$.

### *Trimethylphenoxy Silane (Entry 2, Table I)*

$^1$H NMR ($CDCl_3$, 500 MHz): $\delta = 7.31$ (t, 2H, $J = 8.0$ Hz), 7.02 (t, 1H, $J = 7.3$ Hz), 6.92 (d, 2H, $J = 7.8$ Hz), 0.34 (s, 9H) ppm; IR ($CCl_4$): 3039, 2960, 1596, 1492, 1252, 1164, 1070, 1024, 1002, 918, 843, 759, 692 $cm^{-1}$.

### *Trimethyl(4-methyl phenoxy) Silane (Entry 3, Table I)*

$^1$H NMR ($CDCl_3$, 500 MHz): $\delta = 7.08$ (d, 2H,$J = 8.0$ Hz), 6.80 (d, 2H,$J = 8.1$ Hz), 2.33 (s, 3H), 0.32 (s, 9H) ppm; IR ($CCl_4$): 2960, 1613, 1509, 1251, 1168, 1103, 916, 846, 754 $cm^{-1}$.

### *Trimethyl(4-amino phenoxy) Silane (Entry 4, Table I)*

$^1$H NMR ($CDCl_3$, 500 MHz): $\delta = 6.61$ (d, 2H, $J = 8.5$ Hz), 6.51 (d, 2H, $J = 6.5$ Hz), 3.34 (s, 2H), 0.22 (s, 9H) ppm; $^{13}$C NMR ($CDCl_3$, 125 MHz):

$\delta$ = 147.6, 140.4, 120.6, 116.7, 0.2 ppm; IR ($CCl_4$): 3357, 2952, 1624, 1509, 1244, 1120, 911, 846, 754 $cm^{-1}$.

### *Trimethyl(1-naphtaleneoxy) Silane (Entry 5, Table I)*

$^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ = 8.25 (dd, 1H, $J$ = 6.12 and 3.4 Hz), 7.88 (dd, 1H, $J$ = 6.01 and 3.4 Hz), 7.56–7.54 (m, 3H), 7.41 (t, 1H, $J$ = 7.7 Hz), 6.97 (d, 1H, $J$ = 7.4 Hz), 0.44(s, 9H) ppm; IR ($CCl_4$): 3050, 2959, 1579, 1507, 1461, 1390, 1272, 1154, 1093, 1051, 1015, 914, 848, 796, 771 $cm^{-1}$.

### *Trimethyl(2-naphtaleneoxy) Silane (Entry 6, Table I)*

$^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ = 7.85–7.78 (m, 3H), 7.50, ($J$ = 5.0 Hz), 7.41 (t, 1H, $J$ = 6.9 Hz), 7.31 (d, 1H, $J$ = 2.1 Hz), 7.18 (dd, 1H, $J$ = 8.8 and 2.3 Hz), 0.41(s, 9H) ppm; IR ($CCl_4$): 3057, 2959, 1631, 1590, 1508, 1460, 1349, 1254, 1173, 1122, 978, 926, 855, 746 $cm^{-1}$.

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### *Bistrimethyl(4,4′-biphenoxy) Silane (Entry 7, Table I)*

$^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ = 7.44 (d, 4H, $J$ = 8.6 Hz), 6.91 (d, 4H, $J$ = 8.5 Hz), 0.32 (s, 18H) ppm; $^{13}C$ NMR ($CDCl_3$, 125 MHz): $\delta$ = 154.3, 134.2, 127.7, 120.2, 0.28 ppm; IR ($CCl_4$): 3060, 2960, 1603, 1490, 1255, 1169, 1102, 923, 841, 759 $cm^{-1}$.

### *Trimethyl(4-methoxybenzyloxy) Silane (Entry 8, Table I)*

$^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ = 7.29 (d, 2H, $J$ = 8.4 Hz), 6.91 (d, 2H, $J$ = 8.4 Hz), 4.66 (s, 2H), 3.81 (s, 3H), 0.18 (s, 9H) ppm; $^{13}C$ NMR ($CDCl_3$, 125 MHz): $\delta$ = 158.9, 133.1, 128.1, 113.7, 64.4, 55.2, 0.3 ppm; IR ($CCl_4$): 3040, 2956, 1613, 1587, 1512, 1464, 1376, 1300, 1248, 1171, 1085, 840, 751 $cm^{-1}$.

### *Trimethyl(2-phenylethoxy) Silane (Entry 9, Table I)*

$^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ = 7.33–7.23 (m, 5H), 3.81 (t, 2H, $J$ = 7.3 Hz), 2.87 (t, 2H, $J$ = 7.3 Hz), 0.11(s, 9H) ppm; IR ($CCl_4$): 3064, 3028, 2955, 2899, 1604, 1479, 1474, 1454, 1383, 1250, 1207, 1094, 1030, 928, 842, 740, 698 $cm^{-1}$.

### *Trimethyl(diphenylmethoxy) Silane (Entry 10, Table I)*

$^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ = 7.41–7.23 (m, 10H), 5.80 (s, 1H), 0.12 (s, 9H) ppm; IR ($CCl_4$): 3063, 3027, 2957, 2863, 1598, 1492, 1453, 1354, 1303, 1251, 1187, 1090, 1061, 1027, 917, 885, 740, 700 $cm^{-1}$.

### *Trimethyl($\alpha$-methylbenzyloxy) Silane (Entry 11, Table I)*

$^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ = 7.40–7.34 (m, 4H), 7.28 (t, 1H, $J$ = 6.9 Hz), 4.93 (q, 1H, $J$ = 6.3 Hz), 1.51 (d, 3H, $J$ = 6.3 Hz), 0.15 (s, 9H)

ppm; IR ($CCl_4$): 3063, 3027, 2972, 2927, 2868, 1688, 1603, 1492, 1450, 1369, 1250, 1206, 1090, 1032, 999, 959, 841, 757, 699 $cm^{-1}$.

### *Trimethyl Butoxy Silane (Entry 12, Table I)*

$^1H$ NMR ($CDCl_3$, 500 MHz): $\delta = 3.58$ (t, 2H, $J = 6.5$ Hz), 1.52 (qui, 2H, $J = 5.9$ Hz), 1.33 (six, 2H, $J = 5.4$ Hz), 0.92 (t, 3H, $J = 7.3$ Hz), 0.11 (s, 9H) ppm; IR ($CCl_4$): 2959, 1384, 1250, 1095, 900, 792 $cm^{-1}$.

### *Trimethyl Isopentyloxy Silane (Entry 13, Table 1)*

$^1H$ NMR ($CDCl_3$, 500 MHz): $\delta = 3.59$ (t, 2H, $J = 6.8$ Hz), 1.73–1.64 (m, 1H), 1.42 (q, 2H, $J = 6.8$ Hz), 0.90 (d, 6H, $J = 6.6$ Hz), 0.10 (s, 9H) ppm; IR ($CCl_4$): 2959, 1464, 1385, 1250, 1092, 991, 794 $cm^{-1}$.

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### *Trimethyl Cyclohexyloxy Silane (Entry 14, Table I)*

$^1H$ NMR ($CDCl_3$, 500 MHz): $\delta = 3.57$–3.51 (m, 1H), 1.79–1.70 (m, 4H), 1.53–1.50 (m, 1H), 1.29–1.13 (m, 5H), 0.10 (s, 9H) ppm; IR ($CCl_4$): 2933, 2857, 1450, 1375, 1249, 1092, 1049, 996, 887, 839, 748 $cm^{-1}$.

### *Trimethyl (Tert-butoxy) Silane (Entry 15, Table I)*

$^1H$ NMR ($CDCl_3$, 300 MHz): $\delta = 1.23$ (s, 9H), 0.05 (s, 9H), ppm; $^{13}C$ NMR ($CDCl_3$, 75 MHz): $\delta = 58.1$, 31.1, 1.4 ppm; IR ($CCl_4$): 2977, 1363, 1250, 1051, 794 $cm^{-1}$.

### *Trimethyl(tetrahydro-2-furylmethoxy) Silane (Entry 16, Table I)*

$^1H$ NMR ($CDCl_3$, 500 MHz): $\delta = 3.91$–3.83 (m, 1H), 3.79–3.73 (m, 1H), 3.70–3.63 (m, 1H), 3.46 (t, 2H, $J = 4.9$ Hz), 1.84–1.73 (m, 3H), 1.59–1.50 (m, 1H), 0.02 (s, 9H) ppm; $^{13}C$ NMR ($CDCl_3$, 125 MHz): $\delta =$ 79.4, 68.1, 65.1, 27.6, 25.5, 0.6 ppm; IR ($CCl_4$): 2933, 1450, 1375, 1249, 1092, 1049, 996, 887, 839, 748 $cm^{-1}$.

### *Trimethyl[5-methyl-2-(1-methylethyl) cyclohexyl]oxy Silane (Entry 17, Table I)*

$^1H$ NMR ($CDCl_3$, 500 MHz): $\delta = 3.39$ (six, 1H, $J = 4.3$ Hz), 2.14 (six, 1H, $J = 2.2$ Hz), 1.85–1.83 (m, 1H), 1.64–1.56 (m, 2H), 1.43–1.32 (m, 1H), 1.17–1.11 (m, 1H), 1.06–0.93 (m, 2H), 0.89 (d, 6H, $J = 6.7$ Hz), 0.86–0.76 (m, 1H), 0.72 (d, 3H, $J = 6.8$ Hz), 0.10 (s, 9H) ppm; $^{13}C$ NMR ($CDCl_3$, 125 MHz): $\delta = 72.4$, 50.0, 45.4, 34.5, 31.6, 25.2, 22.9, 22.2, 21.2, 15.9, 0.5 ppm; IR (CCl4): 2955, 1455, 1249, 1069, 931, 886, 839 $cm^{-1}$.

### *Bistrimethyl(1,4-butanedioxy) Silane (Entry 18, Table I)*

$^1$H NMR ($CDCl_3$, 500 MHz): $\delta = 3.59$ (t, 4H, $J = 5.7$ Hz), 1.55 (qui, 4H, $J = 3.0$ Hz), 0.10(s, 18H) ppm; IR ($CCl_4$): 2956, 1250, 1094, 840 $cm^{-1}$.

### *Trimethyl(tricyclo[3,3,1,1$^{3,7}$] doc-1-yloxy) Silane (Entry 19, Table I)*

$^1$H NMR ($CDCl_3$, 500 MHz): $\delta = 2.10$ (s, 3H), 1.76 (d, 6H, $J = 2.9$ Hz), 1.60 (d, 6H, $J = 2.5$ Hz), 0.13(s, 9H), ppm; $^{13}$C NMR ($CDCl_3$, 125 MHz): $\delta = 71.3$, 46.0, 36.2, 30.9, 3.0 ppm; IR ($CCl_4$): 2908, 1453, 1353, 1304, 1240, 1132, 1093, 1016, 964, 871, 837, 752 $cm^{-1}$.

### *Trimethyl(1-octyloxy) Silane (Entry 20, Table I)*

$^1$H NMR ($CDCl_3$, 500 MHz): $\delta = 3.56$ (t, 2H, $J = 6.7$ Hz), 1.52 (qui, 2H, $J = 6.8$ Hz), 1.31–1.28 (m, 10H), 0.88 (t, 3H, $J = 6.5$ Hz), 0.10 (s, 9H) ppm; IR ($CCl_4$): 2928, 2857, 1250, 1099, 840, 746 $cm^{-1}$.

### *Trimethyl[(1-methylheptyl)oxy] Silane (Entry 21, Table I)*

$^1$H NMR ($CDCl_3$, 500 MHz): $\delta = 3.75$ (six, 1H, $J = 5.7$ Hz), 1.44–1.26 (m, 10H), 1.12 (d, 3H, $J = 6.0$ Hz), 0.88 (t, 3H, $J = 6.6$ Hz), 0.10 (s, 9H) ppm; IR ($CCl_4$): 2958, 2929, 2858, 1458, 1375, 1249, 1135, 1084, 1049, 956, 830, 794, 747 $cm^{-1}$.

### *Trimethyl[(3$\beta$)-Choest-4-en-3-yl]oxy Silane (Entry 22, Table I)*

$^1$H NMR ($CDCl_3$, 500 MHz): $\delta = 5.32$ (t, 1H, $J = 2.2$ Hz), 3.51–3.45 (m, 1H), 2.29–0.86 (m, 40H), 0.67 (s, 3H), 0.12 (s, 9H) ppm; $^{13}$C NMR ($CDCl_3$, 125 MHz): $\delta = 141.4$, 121.3, 72.3, 56.8, 56.1, 50.2, 42.7, 42.3, 39.8, 39.5, 37.4, 36.5, 36.2, 35.7, 32.0, 31.95, 31.9, 28.2, 28.0, 24.2, 23.8, 22.7, 22.5, 21.0, 19.3, 18.7, 11.8, 0.2 ppm; IR (KBr): 2950, 1466, 1380, 1249, 1085, 958, 897, 840, 754 $cm^{-1}$.

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