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The First Polymer-Supported and Recyclable Chiral Catalyst for Enantioselective Olefin Metathesis

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General. Infrared (IR) spectra were recorded on a Nicolet 210 spectrophotometer, ν_{max} in cm^{-1}. Bands are characterized as broad (br), strong (s), medium (m), and weak (w). ^{1}H NMR spectra were recorded on Varian GN-400 (400 MHz), Varian Gemini 2000 (400 MHz), and Unity 300 (300 MHz) spectrometers. Chemical shifts are reported in ppm from tetramethylsilane with the solvent resonance as the internal standard ($CHCl_3$: δ 7.26, C_6D_6: δ 7.15). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, br = broad, m = multiplet), coupling constants (Hz), integration, and assignment. ^{13}C NMR spectra were recorded on Varian GN-400 (100 MHz), Varian Gemini 2000 (400 MHz), and Unity 300 (75.4 MHz) spectrometers with complete proton decoupling. Chemical shifts are reported in ppm from tetramethylsilane with the solvent as the internal reference ($CDCl_3$: δ 77.7 ppm, C_6D_6: δ 128.1 ppm). Enantiomer ratios were determined by chiral GLC (Supelco alphadex 120 column (30 m x 0.25 mm), Supelco betadex 120 column (30 m x 0.25 mm), Alltech Associates chiraldex GTA column (30 m x 0.25 mm)) or chiral HPLC analysis (Chiral Technologies chiralcel OD (0.46 cm x 25 cm), chiracel OJ (0.46 cm x 25 cm), or chiralpak AD (0.46 cm x 25 cm)) in comparison with authentic racemic materials. Microanalyses were performed by Robertson Microlit Laboratories (Madison, NJ). High resolution mass spectrometry was performed by the University of Illinois Mass Spectrometry Laboratories (Urbana-Champaign, IL). All chemicals were purchased from Strem Chemicals, Inc., Aldrich Chemical Company, or Lancaster Synthesis and purified before use.

All reactions were conducted in oven- (135 C) and flame-dried glassware under an inert atmosphere of dry nitrogen. All solvents were purified under a positive pressure of dry argon by a modified Innovative Technologies purification system. Benzene and toluene were sparged with argon and are passed through activated copper and alumina columns. Tetrahydrofuran, diethyl ether are passed through activated alumina columns. Olefin-free pentane was generated by stirring commercial grade pentane over concentrated sulfuric acid 24 h. The pentane is then poured over fresh concentrated sulfuric acid. The process was repeated until the acid layer remained colorless for 48 h. The pentane was then separated, washed with water, dried over Na_2SO_4, filtered, sparged with nitrogen and then passed through an activated alumina column.

All handling of the molybdenum catalysts was peformed in a glovebox under inert atmosphere. All substrates were vigorously dried by repeated azeotropic distillation using anhydrous benzene (3x) prior to use.

5,5'-Bis-bromomethyl-3,3'-di-*tert*-butyl-6,6'-dimethyl-biphenyl-2,2'-diol (*R*)-7. To a suspension of (R)-3,3'-di-*tert*-butyl-5,6,5',6'-tetramethyl-biphenyl-2,2'-diol ((***R***)-**5**)[1] (18.1 g, 51.1 mmol) and sodium acetate (16.9 g 206 mmol) in glacial acetic acid (450 mL) was added a solution of bromine (17.9 g, 112 mmol) in glacial acetic acid (40 mL) over 30 min in a dropwise fashion. During the addition, the ligand gradually dissolved. The resulting yellow-orange solution was stirred for 15 min, after which ice-water (1000 mL) was added to the mixture. The resulting suspension was stirred for 15 min and then filtered. The pale yellow solid was washed with water (5 x 100 mL). The solid was then collected and dissolved in Et_2O (500 mL), the organic layer was separated from the residual water and dried over anhydrous $MgSO_4$. Removal of the solvent *in vacuo* gave ring-brominated cyclohexadienone (25.1 g) as a pale yellow solid ((*R*)-**6**), which was stored in a sealed flask for five days. During this period the solid turned brown and then off-white. The resulting off-white solid was recrystallized from hexane at 0 °C to give (*R*)-**7** as an off-white powder in two crops (22.4 g, 86%). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.32 (s, 2H, aryl-**H**), 5.01 (s, 2H, aryl-O**H**), 4.57 (s, 4H, aryl-C**H**$_2$), 1.94 (s, 6H, aryl-C**H**$_3$), 1.39 (s, 18H, C(C**H**$_3$)$_3$); ^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$): δ 152.9, 135.8, 134.6, 130.1, 128.1, 121.1 (aryl **C**), 34.7 (**C**(CH$_3$)$_3$), 34.1 (**C**H$_2$Br), 29.4 (C(**C**H$_3$)$_3$), 15.2 (aryl-**C**H$_3$). HRMS calcd. for $C_{24}H_{32}Br_2O_2$: 510.0769. Found: 510.0759.

5,5'-di-*tert*-butyl-6,6'-bis-ethoxymethoxy-2,2'-dimethyl-3,3'-bis-[2-(4-vinyl-phenyl)-ethyl]-biphenyl (*R*)-8. To a cooled (0 C) filtered solution of *p*-vinylbenzyl magnesium chloride, prepared from freshly distilled *p*-vinylbenzyl chloride (6.56 g, 43.0 mmol) and magnesium powder (4.18 g, 172 mmol) in ether (80 mL), was added a cold solution of (*R*)-**7** (5.12 g, 10.0 mmol) in THF (40 mL) in a dropwise fashion. The resulting mixture was warmed to 22 C and stirred for 90 min. At this time the reaction was quenched by sequential addition of methanol (20 mL) and saturated aqueous NH_4Cl (40 mL). The resulting aqueous layer was washed with Et_2O (2 x 40 mL), the combined organic layers were dried over anhydrous $MgSO_4$ and solvents were removed *in vacuo*. Silica gel chromatography with pentane gave the desired alkylation product (3,3'-di-*tert*-butyl-6,6'-dimethyl-5,5'-bis-[2-(4-vinyl-phenyl)-ethyl]-biphenyl-2,2'-diol) as a white solid (3.01 g, 51%; $R_f = 0.05$). (Oligomeric sideproducts were not eluted under these conditions.) ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31 (d, $^3J_{HH} = 8.0$ Hz, 4H, aryl-**H**), 7.07 (d, $^3J_{HH} = 8.0$ Hz, 4H, aryl-**H**), 7.02 (s, 2H, aryl-**H**), 6.68 (dd, $^3J_{HH,trans} = 17.6$ Hz, $^3J_{HH,cis} = 10.9$ Hz, 2H, aryl-C**H**=CH$_2$), 5.69 (d, $^3J_{HH,trans} = 17.6$ Hz, 2H, aryl-CH=C**H**$_2$), 5.18 (d, $^3J_{HH,cis} = 10.9$ Hz, 2H, aryl-CH=C**H**$_2$), 4.78 (s, 2H, aryl-O**H**), 2.87 (m, 4H, C**H**$_2$C**H**$_2$), 1.74 (s, 6H, aryl-C**H**$_3$), 1.36 (s, 18H, C(C**H**$_3$)$_3$); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 150.5, 141.6 (aryl-**C**), 136.6 (aryl-**C**H=CH$_2$), 135.3, 133.6, 131.5, 128.7, 128.5, 126.1, 121.0 (aryl-**C**), 113.0 (aryl-CH=**C**H$_2$), 37.2, 35.6 (aryl-**C**H$_2$), 34.5 (**C**(CH$_3$)$_3$), 29.5 (C(**C**H$_3$)$_3$), 15.3 (aryl-**C**H$_3$). HRMS (FAB) calcd. for $C_{42}H_{50}O_2$: 586.3811. Found: 586.3827. Anal. calcd. for $C_{42}H_{50}O_2$: C 85.96, H 8.59. Found: C, 85.95; H, 8.56.

A solution of the styryl functionalized biphenol (3,3'-Di-*tert*-butyl-6,6'-dimethyl-5,5'-bis-[2-(4-vinyl-phenyl)-ethyl]-biphenyl-2,2'-diol) (1.93 g, 3.29 mmol) in THF (40 mL) was treated with

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[1] a) J. B. Alexander, D. S. La, D. R. Cefalo, A. H. Hoveyda, R. R. Schrock, *J. Am. Chem. Soc.* **1998**, *120*, 4041-4042 b) A. H. Hoveyda, R. R. Schrock, *Chemistry – Eur. J.*, **2001**, *7*, 945-950.

small portions of solid KH (500 mg, 12.5 mmol). The suspension was allowed to stir for 5 h, then cooled to –20 C and treated with chloromethyl ethyl ether (1.25 g, 13.2 mmol). The resulting mixture was stirred for 2 h at 22 C and was then cooled –20 C and stirred for an additional 1 h. Solvent was removed *in vacuo* and the remaining solid was washed with pentane (3 x 20 mL). The combined pentane washes were filtered to remove KCl. Evaporation of solvents *in vacuo* afforded **(*R*)-8** as a pale yellow oil (2.20 g, 95%). The product was of sufficient purity for subsequent polymerization, but may be further purified by column chromatography with pentane/EtOAc (50:1) (1.85 g, 80% yield, $R_f = 0.3$). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31 (d, $^3J_{HH}$ = 8.0 Hz, 4H, aryl-**H**), 7.10 (d, $^3J_{HH}$ = 8.0 Hz, 4H, aryl-**H**), 7.00 (s, 2H, aryl-**H**), 6.68 (dd, $^3J_{HH,cis}$ = 10.9 Hz, $^3J_{HH,trans}$ = 17.6 Hz, 2H, aryl-C**H**=CH$_2$), 5.68 (d, $^3J_{HH,trans}$ = 17.6 Hz, 2H, aryl-CH=C**H**$_2$), 5.18 (d, $^3J_{HH,cis}$ = 10.9 Hz, 2H, aryl-CH=C**H**$_2$), 4.37 (d, $^2J_{HH}$ = 4.1 Hz, 2H, OC**H**$_2$O), 3.96 (d, $^2J_{HH}$ = 4.1 Hz, 2H, OC**H**$_2$O), 3.55 (m, 2H, OC**H**$_2$CH$_3$), 3.02 (m, 2H, OC**H**$_2$CH$_3$), 2.83 (m, 4H, C**H**$_2$C**H**$_2$), 1.88 (s, 6H, aryl-C**H**$_3$), 1.35 (s, 18H, C(C**H**$_3$)$_3$); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 152.4, 141.5, 140.1 (aryl-**C**), 136.6 (aryl-**C**H=CH$_2$), 135.4, 134.0, 133.2, 128.7, 128.5, 126.1 (aryl-**C**), 113.0 (aryl-CH=**C**H$_2$), 96.0 (O**C**H$_2$O), 64.7 (O**C**H$_2$CH$_3$), 36.7, 35.8 (aryl-**C**H$_2$), 34.8 (**C**(CH$_3$)$_3$), 30.6 (C(**C**H$_3$)$_3$), 16.1 (OCH$_2$**C**H$_3$), 15.0 (aryl-**C**H$_3$). HRMS (ESI) calcd. for $C_{48}H_{62}O_4$+Na: 725.4540. Found: 725.4561.

Representative procedure for solid supported Mo-catalyzed metathesis. In a dry box, to a solution of substrate **15** (20.0 mg, 0.100 mmol), and styrene (20.8 mg, 0.200 mmol) in benzene (1.0 mL), **4** (16.2 mg, 0.0500 mmol) was added in one portion. The vessel was secured with a teflon-lined cap, and the reaction was agitated on an automated shaker plate for 30 min. The reaction was then removed from the dry box. The reaction was then filtered through a Pasteur pipette fitted with a cotton plug and concentrated *in vacuo* to afford a yellow oil. Purification by silica gel chromatography to remove excess styrene (50:1 hexanes:Et_2O) gave **(1*S*, 2*S*, 5*R*)-1-(4-Benzyloxy)-2-styryl-5-vinyl-cyclopentane (16)** as a colorless oil (28.0 mg, 92%). IR (neat): 3609 (m), 2949 (s), 2873 (s), 1344 (m), 1099 (s) cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35-7.19 (m, 10H), 6.44 (d, *J* = 16.0 Hz, 1H), 6.39 (dd, *J* = 16.4, 6.8 Hz, 1H), 6.06 (ddd, *J* = 17.2, 10.0, 8.8 Hz, 1H), 5.12 (dd, *J* = 17.2, 1.2 Hz, 1H), 5.05 (d, *J* = 10.4 Hz, 1H), 4.60 (d, *J* = 11.6 Hz, 1H), 4.55 (d, *J* = 12.0 Hz, 1H), 3.85 (t, *J* = 4.4 Hz, 1H), 2.86-2.62 (m, 2H), 1.94-1.83 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.9, 139.1, 138.3, 132.1, 130.3, 129.0, 128.8, 128.3, 127.9, 127.4, 126.6, 115.4, 88.1, 74.4, 50.9, 50.1, 30.0, 29.7. Anal. calcd. for $C_{22}H_{24}O$: C, 86.80; H, 7.95. Found: C, 86.76; H, 8.03.

Representative procedure for recycling the supported chiral Mo-catalyst. In the dry box, substrate **15** (20.0 mg, 0.100 mmol), and styrene (20.8 mg, 0.200 mmol) were dissolved in benzene (1.0 mL). Solid **4** (16.2 mg, 0.0500 mmol) was then added to the reaction vessel in one portion. The vessel was secured with a teflon-lined cap, and the reaction was agitated on an automated shaker plate for 30 minutes. The reaction was filtered through a Pasteur pipette fitted with a cotton plug. The vessel was then rinsed with an additional 1 mL benzene, and the rinse was filtered through the same cotton plug. The contents of the pipette were then transferred to a fresh solution of substrate **15** (20.0 mg, 0.100 mmol) and styrene (20.8 mg, 0.200 mmol) in benzene (1.0 mL), and the cycle (agitate / filter / rinse) was repeated twice.

2-*iso*-Propenyl-3-methyl-2,5-dihydrofuran (12). IR (neat): 3074 (s), 2973 (s), 2946 (s), 2917 (s), 2845 (s), 2673 (w), 1802 (w), 1669 (w), 1648 (s), 1478 (w), 1447 (s (br)), 1381 (s), 1370 (s), 1346 (s), 1250 (s), 1184 (s), 1070 (br), 1015 (s), 940 (s), 921 (s), 900 (s), 830 (s), 774 (w), 759 (w) cm^{-1}; ^{1}H NMR (300 MHz, C_6D_6): δ 5.17 (septet, *J* = 1.7 Hz, 1H, C=C**H**CH$_2$), 5.02 (t, *J* = 4.2 Hz, 1H, C=C**H**CH$_2$), 4.89 (t, *J* = 1.7 Hz, 1H, CH$_3$C=CH**H**), 4.82 (quintet, *J* = 1.7 Hz, 1H, CH$_3$C=C**H**H), 4.50 (d, *J* = 1.8 Hz, 2H, OC**H**$_2$CH=C), 1.64 (t (br), *J* = 1.3 Hz, C**H**$_3$C=CH$_2$), 1.38 (m, 1H, C**H**$_3$C=CHCH$_2$). ^{13}C NMR (125 MHz, C$_6$D$_6$): δ 146.2, 137.2, 122.4, 113.4, 93.5, 76.1, 16.4, 12.4. HRMS calcd. for C$_8$H$_{12}$O: 124.0888; Found: 124.0888.

2-*iso*-Butyl-3-methyl-2,5-dihydrofuran (14). IR (neat): 2962 (s), 2936 (s), 2861 (m), 1256 (m), 1080 (s), 1029 (s), 897 (w), 797 (m) cm^{-1}; ^{1}H NMR (400 MHz, CDCl$_3$): δ 5.43 (t, *J* = 1.6 Hz, 1H, C=C**H**), 4.65-4.49 (m, 3H, **H**$_2$COC**H**), 1.85 (tqq, *J* = 2.8, 2.8, 2.8 Hz, 1H, C**H**(CH$_3$)$_2$), 1.68 (d, *J* = 1.2 Hz, 3H, CC**H**$_3$), 1.35 (dd, *J* = 6.8, 6.8 Hz, 2H, OCHC**H**$_2$), 0.94 (d, *J* = 6.8 Hz, 6H, CH(C**H**$_3$)$_2$); ^{13}C NMR (100 MHz, CDCl$_3$): δ 139.6, 120.6, 86.6, 74.7, 44.1, 25.6, 24.7, 22.5, 13.1. Anal. calcd. for C$_9$H$_{16}$O: C, 77.09; H, 11.50. Found: C, 76.89; H, 11.20.

(1*S*,2*S*,3*R*)-2-Methoxymethoxy-1-(2-phenyl-vinyl)-3-vinyl-cyclopentane (18). IR (neat): 3075 (w), 2949 (s), 1640 (w), 1451 (w), 1036 (s) cm^{-1}; ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.36 (dd, *J* = 8.4, 1.2 Hz, 2H, Ar**H**), 7.29 (dd, *J* = 15.6, 7.6 Hz, 2H, Ar**H**), 7.20 (t, *J* = 7.6 Hz, 1H, Ar**H**), 6.44 (d, *J* = 16.0 Hz, 1H, CC**H**=CHCH), 6.35 (dd, *J* = 16.0, 8.0 Hz, 1H, CCH=C**H**CH), 5.97 (ddd, *J* = 18.0 10.4, 8.4 Hz, 1H, CH$_2$=CHCH), 5.11 (ddd, *J* = 17.2, 1.6, 0.8 Hz, 1H, C**H**H=CH), 5.06 (dd, *J* = 11.6, 1.2 Hz, 1H, CH**H**=CH), 4.65 (d, *J* = 7.2 Hz, 1H, OC**H**HO), 4.61 (d, *J* = 6.8 Hz, 1H, OCH**H**O), 4.05 (t, *J* = 4.0 Hz, 1H, OC**H**), 3.33 (s, 3H, OC**H**$_3$), 2.78 (ddd, *J* = 17,2, 8.8, 4.4 Hz, 1H, C**H**CH=CH$_2$), 2.65 (ddd, *J* = 16.8, 8.4, 4.4 Hz, 1H, C**H**CH=CH$_2$) 1.95-1.80 (m, 4H, C**HH**CH**H**). ^{13}C NMR (100 MHz, CDCl$_3$): δ 139.4, 138.2, 131.5, 130.6, 129.0, 127.5, 126.5, 115.7, 97.4, 85.7, 56.6, 50.4, 49.7, 29.6, 29.1. Anal. calcd. for C$_{17}$H$_{22}$O$_2$: C, 79.03; H, 8.58. Found: C, 78.88; H, 8.86.

(*R*)-2-Allyl-2-phenyl-3,6-dihydro-2H-pyran (20). IR (neat): 3077 (w), 3027 (w), 2935 (w), 2829 (w), 1442 (m), 1089 (s), 698 (s), 655 (s) cm^{-1}. ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.44-7.33 (m, 4H, Ar**H**) 7.28-7.24 (m, 1H, Ar**H**), 5.89–5.84 (m, 1H, CCH$_2$C**H**=CH), 5.63-5.59 (m, 1H, OCH$_2$C**H**=CH), 5.57-5.48 (m, 1H, **H**C=CH$_2$), 5.01-4.96 (m, 2H, HC=C**H**$_2$), 4.17 (ddd, *J* = 17.2, 2.0, 2.0 Hz, 1H, OC**H**H), 3.95 (ddd, *J* = 16.8, 2.8, 2.8 Hz, 1H, OCH**H**), 2.67-2.39 (m, 4H, C(C**H**$_2$)$_2$). ^{13}C NMR (100 MHz, CDCl$_3$): δ 143.7, 134.0, 128.6, 127.5, 126.9, 126.6, 123.4, 118.4, 76.5, 62.3, 47.7, 32.7. Anal. calcd. for C$_{14}$H$_{16}$O: C, 83.96; H, 8.05. Found: C, 83.77; H, 7.97.

Anti-9-vinyl-4-methyl-*trans*-2-oxabicyclo[4.3.0]nona-4-ene (22). IR (neat) 2963 (s), 2872 (s), 1448 (m), 1127 (s), 909 (m) cm^{-1}. ^{1}H NMR (400 MHz, CDCl$_3$): δ 5.78 (ddd, *J* = 17.2, 10.0, 8.0 Hz, 1H, C**H**=CH$_2$), 5.64-5.60 (m, 1H, R$_2$C=C**H**R), 5.14-5.09 (m, 1H, CH=C**H**H), 5.05-5.01 (m, 1H, CH=CH**H**), 4.19 (s, 2H, OC**H**$_2$), 3.06 (t, *J* = 10.0 Hz, 1H, OC**H**), 2.52-2.41 (m, 1H), 2.29-2.17 (m, 1H), 2,10-1.98 (m, 1H), 1.83-1.73 (m, 1H), 1.58 (s, 3H, C**H**$_3$), 1.56-1.46 (m, 1H), 1.26-1.13 (m, 1H). ^{13}C NMR (100 MHz, CDCl$_3$): δ 141.5, 133.9, 124.0, 115.7, 85.5, 72.3, 46.3, 41.8, 28.2, 24.5, 19.0. HRMS calcd. for C$_{11}$H$_{16}$O: 164.1201. Found: 164.1200.

2,2,5-Trimethyl-7-(2-methyl-allyl)-7-phenyl-2,3,6,7-tetrahydro-[1,2]oxasilepine (24). IR (neat) 3071 (m), 3026 (m), 2964 (s), 2915 (s), 1646 (m), 1490 (m), 1446 (s), 1375 (m), 1321 (m), 1251 (s), 1144 (s), 1126 (s), 1075 (s), 1001 (m), 957 (s), 841(s), 888 (m), 788 (s), 700 (s) cm^{-1}. ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.37 (d, J = 3.6 Hz, 2H), 7.28 (m, 2H), 7.15 (m, 1H), 5.58 (m, 1H), 4.61 (s, 1H), 4.48 (s, 1H), 2.80 (d, J=14.0 Hz, 1H), 2.63 (s, 2H), 2.52 (d, J=14.0 Hz, 1H), 1.54 (m, 1H), 1.48 (s, 3H), 1.40 (m, 1H), 1.34 (s, 3H), 0.23 (s, 3H), 0.21 (s, 3H). ^{13}C NMR (100 MHz, CDCl$_3$): 146.8, 142.7, 132.8, 127.6, 126.2, 126.1, 123.0, 114.6, 80.1, 51.34, 47.8, 26.0, 24.5, 18.5, 1.9, 1.0. Anal. calcd. for C$_{18}$H$_{26}$OSi: C, 74.56; H, 9.15. Found: C, 75.35; H, 8.99.

(*R*)-1-Oxa-6-(2-propenyl)-2-sila-2,2,5-trimethylcyclohex-4-ene (26). IR (neat): 2974 (m), 2924 (m), 2836 (w), 1464 (w), 1275 (s), 1099 (s), 1055 (s) cm-1. ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.72-5.70 (m, 1H, **H**C=CCH_3), 4.92 (dd, *J* = 1.2, 1.2 Hz, 1H, **H**H=CCH3), 4.85 (t, *J* = 1.2 Hz, 1H, H**H**C=CCH_3), 4.62 (s, 1H, OC**H**), 1.66 (t, *J* = 0.8 Hz, 3H, HC=CC**H**$_3$), 1.55 (dd, *J* = 1.2, 1.2 Hz, 3H, CH_2=CC**H**$_3$), 1.35-1.14 (m, 2H, SiC**H**$_2$CH), 0.19 (s, 3H, SiC**H**$_3$), 0.12 (s, 3H, SiC**H**$_3$). ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.5, 136.0, 121.1, 113.8, 82.1, 22.4, 17.2, 13.0, 0.8, 0.0. HRMS calcd. for $C_{10}H_{18}OSi$: 182.1127. Found: 182.1129. Anal. calcd. for $C_{10}H_{18}OSi$: C, 65.87; H, 9.95. Found C, 65.80; H, 10.00.