

# The Efficient Desymmetrization of Glycerol using Scaffolding Catalysis

Zachary X. Giustra, Kian L. Tan

*Department of Chemistry, Merkert Chemistry Center, Boston College, Chestnut Hill, MA,  
02467*

## Supporting Information

### *Table of Contents*

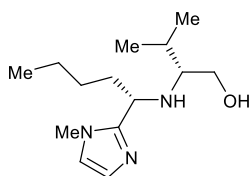
General Considerations.....	S-2
Catalyst Synthesis.....	S-2
General Procedure for Table 1.....	S-3
Procedure for Time Course (Fig. 2).....	S-4
Silylation Product Characterization.....	S-5
References.....	S-9
<sup>1</sup> H and <sup>13</sup> C NMR spectra.....	S-10

### General Considerations:

Unless otherwise noted, all reagents were obtained from commercial suppliers and used without further purification. Lithium reagents were titrated against 2-pentanol using 1,10-phenanthroline as the indicator. Flash column chromatography was performed using EMD Silica Gel 60 (230-400 mesh) and ACS grade solvents as received from Fisher Scientific. All experiments were performed in oven or flame dried glassware under an atmosphere of nitrogen or argon using standard syringe and cannula techniques, except where otherwise noted. All reactions were run with dry, degassed solvents dispensed from a Glass Contour Solvent Purification System (SG Water, USA LLC). Deuterated solvents were purchased from Cambridge Isotope Labs and stored over 3 Å molecular sieves. C<sub>6</sub>D<sub>6</sub> was degassed by three successive freeze-pump-thaw cycles and stored over 3 Å molecular sieves in a dry box under a nitrogen atmosphere. Glycerol and 2-methyl-1,3-propanediol were distilled and stored over 3 Å molecular sieves in a dry box under a nitrogen atmosphere prior to use. 1-Naphthoyl chloride was heated to reflux in the presence of thionyl chloride for 2 h before being distilled and stored over 3 Å molecular sieves under a nitrogen atmosphere. Triisopropylsilyl chloride and *tert*-butyldiphenyl silyl chloride were distilled and stored over 3 Å molecular sieves under a nitrogen atmosphere prior to use. <sup>1</sup>H and <sup>13</sup>C NMR were performed on either a Varian Gemini 400 MHz, Varian Gemini 500 MHz, or a Varian Unity Inova 500 MHz spectrometer. All NMR chemical shifts are reported in ppm relative to residual solvent for <sup>1</sup>H and <sup>13</sup>C NMR. Coupling constants are reported in Hz. All IR spectra were gathered on a Bruker Alpha FT-IR equipped with a single crystal diamond ATR module and values are reported in cm<sup>-1</sup>. HRMS data were generated in Boston College facilities. Analytical chiral high-performance liquid chromatography (HPLC) was performed on either a Shimadzu-LC-2010A HT or an Agilent 1120 Compact LC.

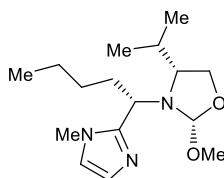
The following compounds were made according to literature procedure: (*R*)-2-amino-3-methylbutan-1-ol,<sup>1</sup> **4b**,<sup>2</sup> **4c**,<sup>3</sup> **5**<sup>3</sup>

### Catalyst Synthesis:



**(*R*)-3-Methyl-2-(((*S*)-2-methyl-1-(1-methyl-1H-imidazol-2-yl)propyl)amino)butan-1-ol**<sup>4</sup>. To a solution of (*R*)-2-amino-3-methylbutan-1-ol (2.1 g, 2.0×10<sup>1</sup> mmol) in anhydrous THF (20 mL) was added a solution of pentanal (1.7 g, 2.0×10<sup>1</sup> mmol) in anhydrous THF (20 mL) and MgSO<sub>4</sub> (4.0 g). The mixture was allowed to stir at 22 °C for 5 h. <sup>1</sup>H NMR analysis confirmed the formation of the oxazolidene intermediate. In a separate flask, to a solution of *N*-methylimidazole (5.8 g, 7.0×10<sup>1</sup> mmol) in anhydrous THF (40 mL) was added *n*-butyllithium (7.0 mL, 1.0×10<sup>1</sup> M in hexanes, 7.0×10<sup>1</sup> mmol) dropwise at -78 °C. The solution was allowed to stir at -78 °C for 30 min, and then slowly cannula transferred to the pre-formed oxazolidene solution at -78 °C. The combined solution was

allowed to slowly warm to 22 °C and was allowed to stir overnight. Saturated aqueous NH<sub>4</sub>Cl was added slowly to quench the reaction. The organic layer was separated, and the aqueous layer was extracted with diethyl ether (3×50 mL). The combined organic layers were dried over MgSO<sub>4</sub>, filtered to remove solids, and concentrated under reduced pressure. Silica column chromatography (50-100% EtOAc in hexanes) afforded pure product as a yellow oil (1.8 g, 35%). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz) δ 6.94 (d, 1H, *J* = 1.0), 6.80 (d, 1H, *J* = 1.0), 3.84 (dd, 1H, *J* = 7.9, 5.2), 3.61 (s, 3H), 3.48 (dd, 1H, *J* = 11.1, 3.5), 3.38 (dd, 1H, *J* = 11.1, 8.0), 2.30 (appdq, 1H, *J* = 8.1, 3.7), 1.77-1.56 (m, 4H), 1.53-1.40 (m, 1H), 1.39-1.23 (m, 4H), 0.94 (d, 3H, *J* = 6.8), 0.90 (d, 6H, *J* = 6.8); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 126 MHz) δ 151.3, 126.5, 121.1, 63.3, 62.5, 54.6, 36.0, 32.6, 31.2, 28.2, 22.6, 19.1, 19.0, 13.9; IR 2958, 2873, 2243, 1467, 1412, 1281, 1044, 904, 724 cm<sup>-1</sup>; HRMS (DART-TOF) calcd. for C<sub>14</sub>H<sub>28</sub>N<sub>3</sub>O [M+H<sup>+</sup>]: 254.2188, found: 254.2236. [α]<sub>D</sub><sup>20</sup> = -39.7 (*c* = 0.70, CHCl<sub>3</sub>, *l* = 50 mm).



**(2*S*,4*R*)-4-Isopropyl-2-methoxy-3-((*S*)-1-(1-methyl-1*H*-imidazol-2-yl)pentyl)oxazolidine (91:9 dr), (4a)**<sup>2,3</sup>. To a solution of (*R*)-3-methyl-2-(((*S*)-2-methyl-1-(1-methyl-1*H*-imidazol-2-yl)propyl)amino)butan-1-ol (1.75 g, 6.91 mmol) in anhydrous MeOH (14 mL) was added *N,N*-dimethylformamide dimethyl acetal (2.76 mL, 20.7 mmol). The solution was allowed to stir at 50 °C overnight. Volatiles were removed under reduced pressure, the residue was reconstituted in MeOH (14 mL), and the solution was allowed to stir at 50 °C for 2 h. Volatiles were removed under reduced pressure and Kugelrohr distillation of the residue (150 °C @ 0.05 mmHg) afforded the product as a yellow oil (1.39 g, 68%). <sup>1</sup>H NMR (C<sub>6</sub>D<sub>6</sub>, 500 MHz) δ 7.07 (d, 1H, *J* = 1.2), 6.31 (d, 0.09H, *J* = 1.2), 6.29 (d, 0.91H, *J* = 1.2), 5.76 (s, 0.92H), 5.28 (s, 0.08H), 4.06 (dd, 0.08H, *J* = 10.8, 3.2), 3.84 (dd, 0.92H, *J* = 10.0, 4.6), 3.71 (d, 1.80H, *J* = 7.1), 3.20 (s, 3H), 3.11 (t, 1H, *J* = 3.3), 3.07 (s, 2.73H), 2.99 (s, 0.27H), 2.83-2.77 (m, 0.12H), 2.56-2.49 (m, 0.88H), 2.38 (d, 0.2H, *J* = 4.2), 2.11-2.05 (m, 0.09H), 1.88-1.81 (m, 0.91H), 1.55-1.48 (m, 1H), 1.50-1.37 (m, 1H), 1.34-1.27 (m, 2H), 1.23-1.16 (m, 1H), 0.87 (t, 0.38H, *J* = 7.2), 0.84 (t, 2.62H, *J* = 7.3), 0.66 (d, 0.28H, *J* = 6.8), 0.56 (d, 2.72H, *J* = 6.8), 0.49 (d, 3H, *J* = 7.1); <sup>13</sup>C NMR (C<sub>6</sub>D<sub>6</sub>, 126 MHz) δ 147.1, 127.2, 120.3, 113.9, 67.2, 62.5, 55.2, 51.6, 31.4, 30.3, 29.4, 29.3, 22.8, 19.1, 16.7, 13.9; IR: 2279, 1618, 1453, 1330, 1162, 811, 516, 490 cm<sup>-1</sup>; HRMS (DART-TOF) calcd. for C<sub>15</sub>H<sub>26</sub>N<sub>3</sub>O [M-OMe]: 264.2076, found: 264.2172. [α]<sub>D</sub><sup>20</sup> = +35.9 (*c* = 0.87, CHCl<sub>3</sub>, *l* = 50 mm).

#### General Procedure for Table 1:

In a dry box, a solution of 1,2,2,6,6-pentamethylpiperidine hydrochloride (1.2 mg, 6.3×10<sup>-3</sup> mmol) and catalyst (20 mol %) in anhydrous THF (0.5 mL) was added to glycerol (18 mg, 0.20 mmol) in an oven-dried glass vial. The solution was allowed to stir at 22 °C for 15 min before 1,2,2,6,6-pentamethylpiperidine (45 μL, 0.25 mmol) was

added. The reaction was cooled to 4 °C and *tert*-butyldimethylsilyl chloride ( $5.0 \times 10^1$  mg, 0.33 mmol) in anhydrous THF (0.5 mL) was added. After 12 h, the reaction was quenched with 15  $\mu$ L MeOH and 60  $\mu$ L 1,2,2,6,6-pentamethylpiperidine. Solids were removed by flushing the reaction mixture through a silica plug with EtOAc; 1,3,5-trimethoxybenzene ( $5.0 \times 10^1$   $\mu$ L, 0.40 M solution in EtOAc, 0.020 mmol) was added as an internal standard, and volatiles were removed under reduced pressure.  $^1\text{H}$  NMR analysis of the crude residue was used to determine the yields of products **2** and **3**. To determine er values, the NMR solution was concentrated under reduced pressure and the residue reconstituted in anhydrous  $\text{CH}_2\text{Cl}_2$  (2 mL) before 1,2,2,6,6-pentamethylpiperidine (2 equiv) and 1-naphthoyl chloride<sup>5</sup> (1 equiv) were added. After 1 h at room temperature, the reaction was quenched with 5  $\mu$ L MeOH and 20  $\mu$ L 1,2,2,6,6-pentamethylpiperidine. Silica column chromatography (3-10% EtOAc in hexanes) afforded the 1-naphthoate derivative as a yellow oil. The er of the derivative was determined by chiral HPLC analysis (OD-H, 1.0 mL/min, 3:97 (*i*-PrOH: hexanes), 230 nm,  $t_{\text{major}} = 18.9$  and  $t_{\text{minor}} = 15.3$  min).

Entry	Catalyst	Yield <b>2</b> (%) <sup>a</sup>	Yield <b>3</b> (%) <sup>a</sup>	er <b>2</b> <sup>b</sup>
1	<b>4a</b>	68	14	97:3
2	<b>4b</b>	74	12	98:2
3	<b>4c</b>	82	12	>99:1
4	<b>4c</b> <sup>c</sup>	66	20	99:1
5	<b>4c</b> <sup>d</sup>	66 (65) <sup>e</sup>	10	99:1 (96:4) <sup>e</sup>
6	<b>5</b>	52	8	50:50

<sup>a</sup> Yields determined by  $^1\text{H}$  NMR analysis using 1,3,5-trimethoxybenzene as an internal standard.

<sup>b</sup> Enantiomeric ratios (er) determined by chiral HPLC analysis of the 1-naphthoate derivative of **2** (*vide supra*).

<sup>c</sup> Using 10 mol % catalyst for 26 h.

<sup>d</sup> Using 5 mol % catalyst for 26 h.

<sup>e</sup> 1.0 mmol scale, isolated yield and er an average of two runs (run 1: 140 mg, 67% yield, 96:4 er; run 2: 130 mg, 63% yield, 96:4 er).

#### Procedure for Time Course (Fig. 2):

The general procedure for Table 1 was followed using catalyst **4c**, and reactions were quenched with 15  $\mu$ L MeOH and 60  $\mu$ L 1,2,2,6,6-pentamethylpiperidine after 1, 2, 4, 6, 8, 12, and 22 h.

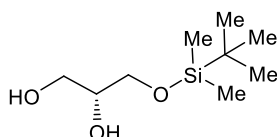
Entry	Time (h)	Yield <b>2</b> (%) <sup>a</sup>	Yield <b>3</b> (%) <sup>a</sup>	er <b>2</b> <sup>b</sup>
1	1	45	2	90:10
2	2	56	4	94:6
3	4	70	6	97:3
4	6	78	9	98.5:1.5
5	8	80	9	99:1

6	12	82	12	>99:1
7	22	75	16	>99:1

<sup>a</sup> Yields determined by <sup>1</sup>H NMR analysis using 1,3,5-trimethoxybenzene as an internal standard.

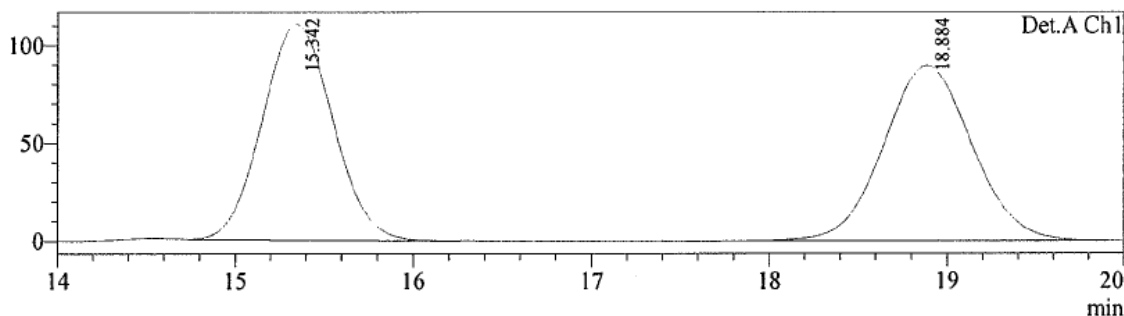
<sup>b</sup> Enantiomeric ratios (er) determined by chiral HPLC analysis of the 1-naphthoate derivative of **2** (*vide supra*).

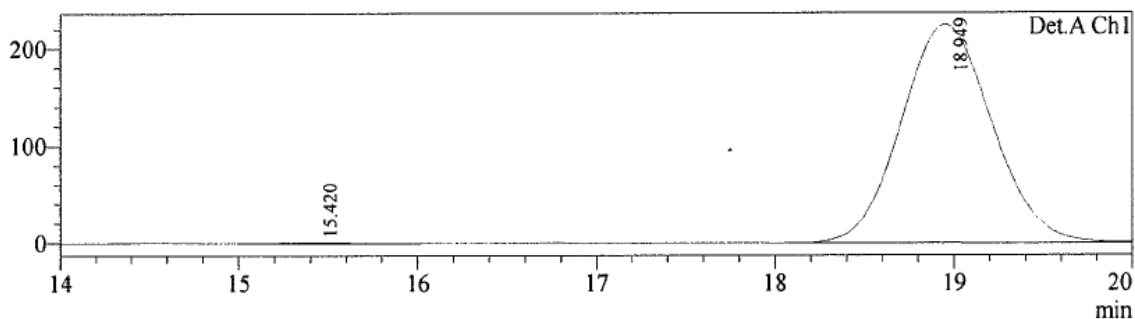
#### Silylation Product Characterization:



**(R)-3-((tert-Butyldimethylsilyloxy)propane-1,2-diol (2)**<sup>6</sup>. In a dry box, a solution of 1,2,2,6,6-pentamethylpiperidine hydrochloride (5.8 mg, 0.030 mmol) and catalyst **4c** (61 mg, 0.20 mmol) in anhydrous THF (2.5 mL) was added to glycerol (92 mg, 1.0 mmol) in an oven-dried glass vial. The solution was allowed to stir at 22 °C for 15 min before 1,2,2,6,6-pentamethylpiperidine (0.22 mL, 1.2 mmol) was added. The reaction was cooled to 4 °C and *tert*-butyldimethylsilyl chloride (0.25 g, 1.6 mmol) in anhydrous THF (2.5 mL) was added. After 12 h, the reaction was quenched with 75 μL MeOH and 3.0 × 10<sup>2</sup> μL 1,2,2,6,6-pentamethylpiperidine. Solids were removed by flushing the reaction mixture through a silica plug with EtOAc; volatiles were removed under reduced pressure. Silica column chromatography (5-25% EtOAc in hexanes) afforded product (0.16 g, 78%) as a colorless oil. The product's enantiomeric ratio was determined for the 1-naphthoate derivative, prepared according to the general procedure for Table 1. **Chiral HPLC Analysis** (OD-H, 1.0 mL/min, 3:97 (*i*-PrOH: hexanes), 230 nm, *t*<sub>major</sub> = 18.9 and *t*<sub>minor</sub> = 15.3 min) >99:1 er. **<sup>1</sup>H NMR** (CDCl<sub>3</sub>, 500 MHz) δ 3.76-3.61 (m, 5H), 2.70 (br s, 1H), 2.34 (br s, 1H), 0.90 (s, 9H), 0.07 (s, 6H); **<sup>13</sup>C NMR** (CDCl<sub>3</sub>, 126 MHz) δ 71.6, 64.7, 64.0, 25.8, 18.2, -5.47, -5.50. **[α]<sub>D</sub><sup>20</sup>** = +3.51 (*c* = 0.73, CHCl<sub>3</sub>, *l* = 50 mm).

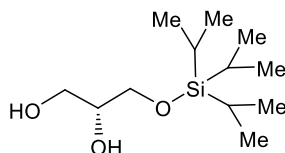
A second run of this experiment afforded product (0.16 g, 78%) in >99:1 er.





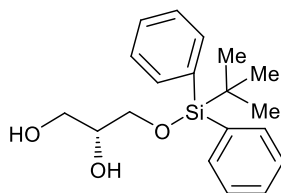
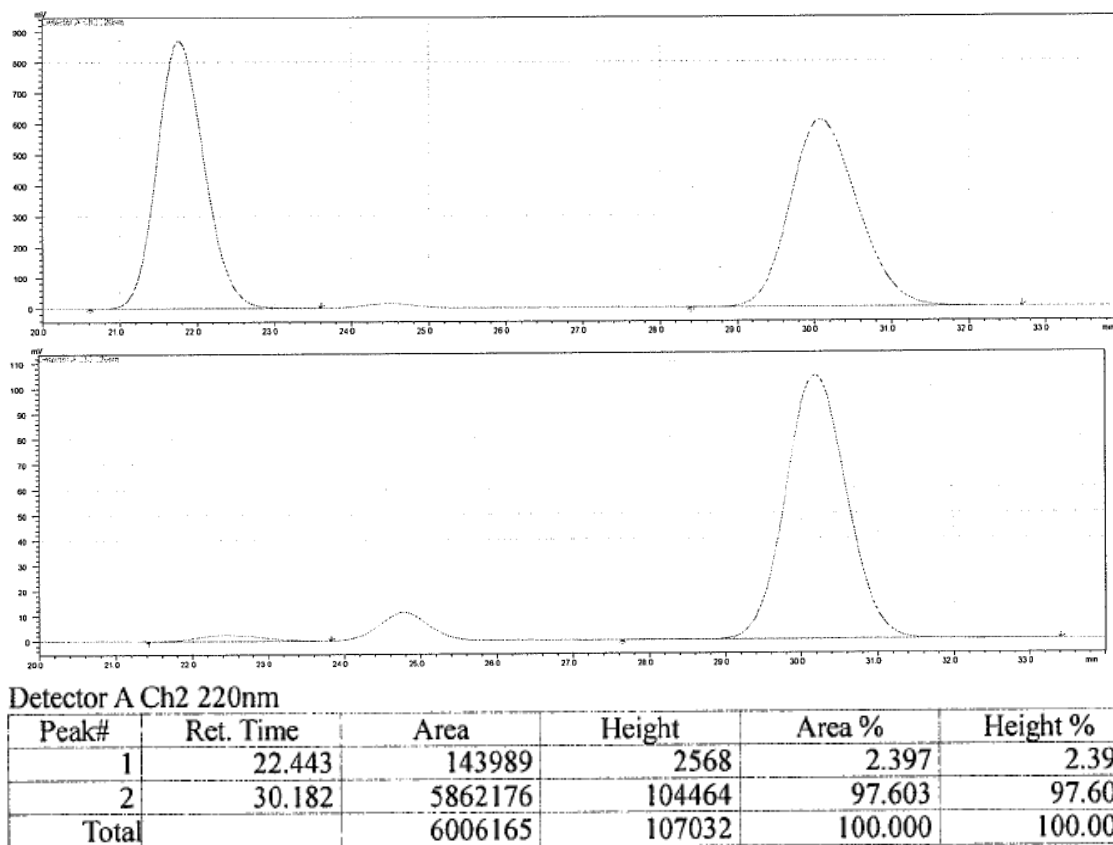
Detector A Ch1 230nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.420	27084	1041	0.341	0.462
2	18.949	7904353	224297	99.659	99.538
Total		7931438	225338	100.000	100.000



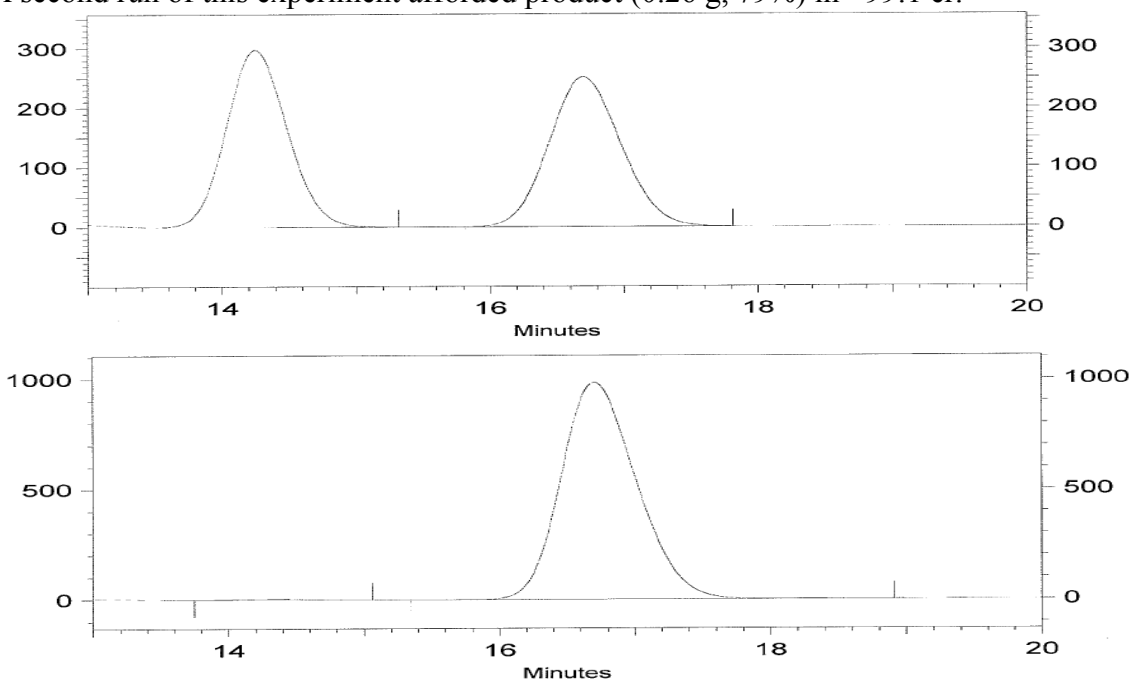
**(R)-3-((Triisopropylsilyl)oxy)propane-1,2-diol.** In a dry box, a solution of 1,2,2,6,6-pentamethylpiperidine hydrochloride (5.8 mg, 0.030 mmol) and catalyst **4c** (61 mg, 0.20 mmol) in anhydrous THF (5.0 mL) was added to glycerol (92 mg, 1.0 mmol) in an oven-dried glass vial. The solution was allowed to stir at 22 °C for 15 min before 1,2,2,6,6-pentamethylpiperidine (0.22 mL, 1.2 mmol) and triisopropylsilyl chloride (0.34 mL, 1.6 mmol) were added dropwise. After 24 h, the reaction was quenched with 75  $\mu$ L MeOH and  $3.0 \times 10^2$   $\mu$ L 1,2,2,6,6-pentamethylpiperidine. Solids were removed by flushing the reaction mixture through a silica plug with EtOAc; volatiles were removed under reduced pressure. Silica column chromatography (0-40% EtOAc in hexanes) afforded product (0.16 g, 64%) as a colorless oil. The product's enantiomeric ratio was determined for the 1-naphthoate derivative, prepared according to the general procedure for Table 1. **Chiral HPLC Analysis** (OD-H, 1.0 mL/min, 2:98 (*i*-PrOH: hexanes), 220 nm,  $t_{\text{major}} = 30.2$  and  $t_{\text{minor}} = 22.4$  min) 98:2 er.  **$^1\text{H NMR}$**  ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  3.79-3.60 (m, 5H) 2.68 (d, 1H,  $J = 4.4$ ), 2.22 (t, 1H,  $J = 6.0$ ), 1.14-0.94 (m, 21H);  **$^{13}\text{C NMR}$**  ( $\text{CDCl}_3$ , 126 MHz)  $\delta$  72.2, 65.5, 64.5, 18.4, 12.3; **IR**: 3392, 2942, 2892, 2867, 1463, 1121, 1065, 1014, 882, 793, 682, 660  $\text{cm}^{-1}$ ; **HRMS** (DART-TOF) calcd. for  $\text{C}_{12}\text{H}_{29}\text{O}_3\text{Si}$  [ $\text{M}+\text{H}^+$ ]: 249.1841, found: 249.1886.  $[\alpha]_{\text{D}}^{20} = +0.60$  ( $c = 1.0$ ,  $\text{CH}_2\text{Cl}_2$ ,  $l = 50$  mm).

A second run of this experiment afforded product (0.17 g, 68%) in 98:2 er. The reported average result was thus calculated as 66% yield, 98:2 er.



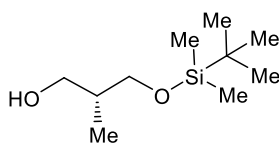
**(*R*)-3-((*tert*-Butyldiphenylsilyl)oxy)propane-1,2-diol<sup>7</sup>.** In a dry box, a solution of 1,2,2,6,6-pentamethylpiperidine hydrochloride (5.8 mg, 0.030 mmol) and catalyst **4c** (61 mg, 0.20 mmol) in anhydrous THF (5 mL) was added to glycerol (92 mg, 1.0 mmol) in an oven-dried glass vial. The solution was allowed to stir at 22 °C for 15 min before 1,2,2,6,6-pentamethylpiperidine (0.22 mL, 1.2 mmol) was added. The reaction was cooled to 4 °C and *tert*-butyldiphenylsilyl chloride (1.3 mL, 5.0 mmol) was added. After 24 h, the reaction was quenched with 25  $\mu$ L MeOH and  $1.0 \times 10^2$   $\mu$ L 1,2,2,6,6-pentamethylpiperidine. Solids were removed by flushing the reaction mixture through a silica plug with EtOAc; volatiles were removed under reduced pressure. Silica column chromatography (5-40% EtOAc in hexanes) afforded product (0.26 g, 79%) as a white solid. **Chiral HPLC Analysis** (OD-H, 1.5 mL/min, 2.98 (*i*-PrOH: hexanes), 220 nm,  $t_{\text{major}} = 16.7$  and  $t_{\text{minor}} = 14.4$  min) >99:1 er. **<sup>1</sup>H NMR** (CDCl<sub>3</sub>, 500 MHz)  $\delta$  7.67-7.65 (m, 4H), 7.46-7.43 (m, 2H), 7.41-7.38 (m, 4H), 3.86-3.79 (m, 1H), 3.76-3.70 (m, 3H), 3.67-3.64 (m, 1H), 2.61 (br s, 1H), 2.04 (br s, 1H), 1.08 (s, 9H); **<sup>13</sup>C NMR** (CDCl<sub>3</sub>, 126 MHz)  $\delta$  135.5, 132.9, 129.9, 127.8, 71.8, 65.3, 63.8, 26.9, 19.2.  $[\alpha]_{\text{D}}^{20} = +3.1$  ( $c = 0.86$ , CHCl<sub>3</sub>,  $l = 50$  mm).

A second run of this experiment afforded product (0.26 g, 79%) in >99:1 er.



VWD: Signal A,  
220 nm Results

Retention Time	Area	Area %	Height	Height %
14.367	2672598	0.42	83404	0.50
16.700	637354567	99.58	16495265	99.50
Totals	640027165	100.00	16578669	100.00

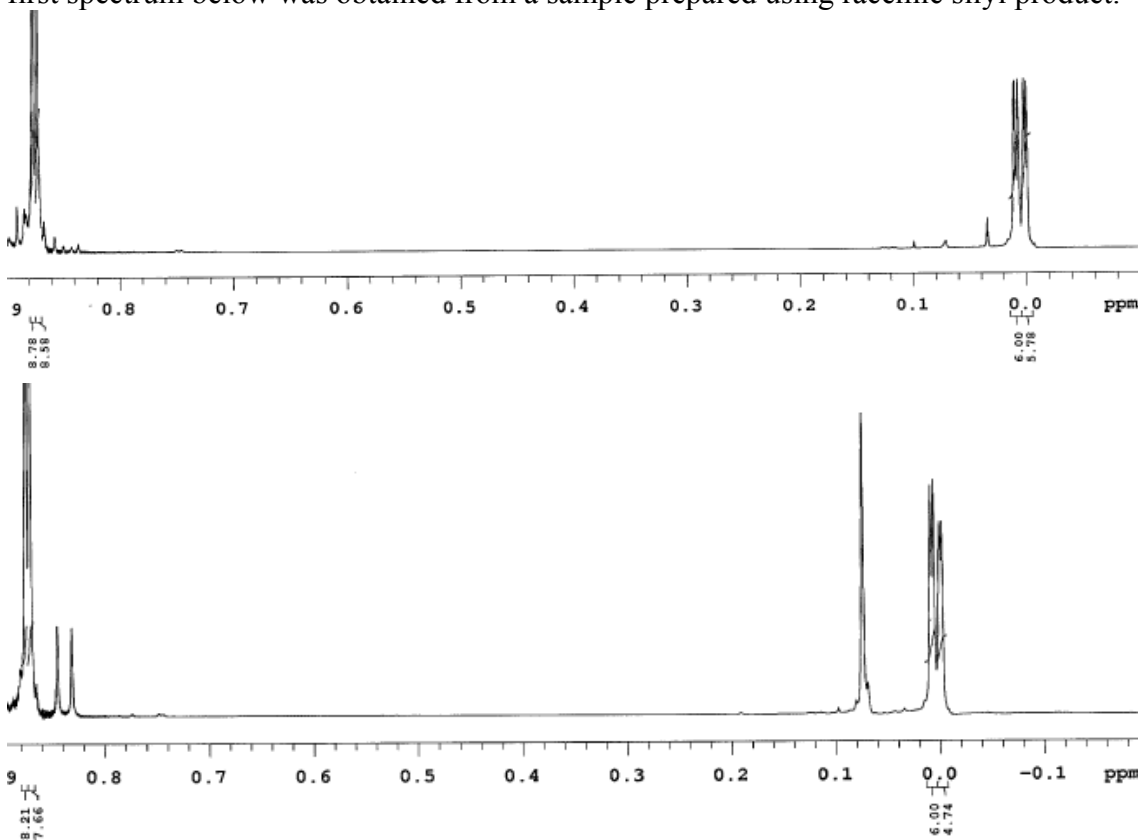


**(R)-3-((tert-Butyldimethylsilyl)oxy)-2-methylpropan-1-ol**<sup>8</sup>. The general procedure for Table 1 was followed, using catalyst **4c** and 18 mg 2-methyl-1,3-propanediol. Silica column chromatography (5-10% EtOAc in hexanes) of the crude reaction mixture afforded product (15 mg, 36%). The product's enantiomeric ratio was determined by <sup>1</sup>H NMR analysis of the crude Mosher ester derivative<sup>9,10</sup> using (*S*)-MTPA (55:45 dr). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz) δ 3.73 (ddd, 1H, *J* = 9.9, 4.4, 0.9), 3.65-3.58 (m, 2H), 3.54 (dd, 1H, *J* = 9.9, 8.0), 2.81 (br s, 1H), 2.04-1.89 (m, 1H), 0.90 (s, 9H), 0.83 (d, 3H, *J* = 7.1), 0.07 (s, 6H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 126 MHz) δ 68.7, 68.2, 37.1, 25.8, 18.2, 13.1, -5.57, -5.63. [ $\alpha$ ]<sub>D</sub><sup>20</sup> = +6.7 (*c* = 0.28, CHCl<sub>3</sub>, *l* = 50 mm).

A second run of this experiment afforded product (13 mg, 32%) whose Mosher ester derivative was found by <sup>1</sup>H NMR analysis to exhibit a dr of 56:44.



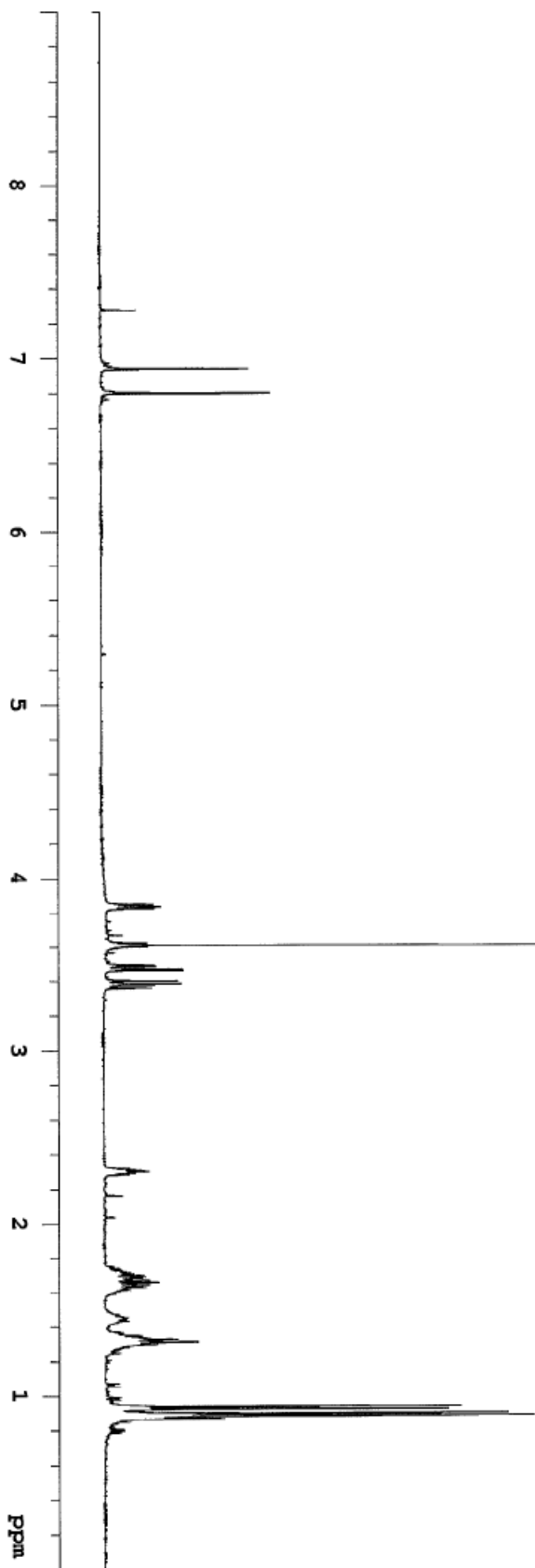
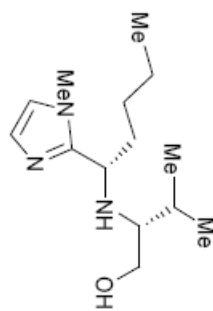
The diastereomeric ratios of the Mosher ester derivatives were calculated based on the integration of peaks corresponding to the TBS group's methyl and *tert*-butyl protons. The first spectrum below was obtained from a sample prepared using racemic silyl product.



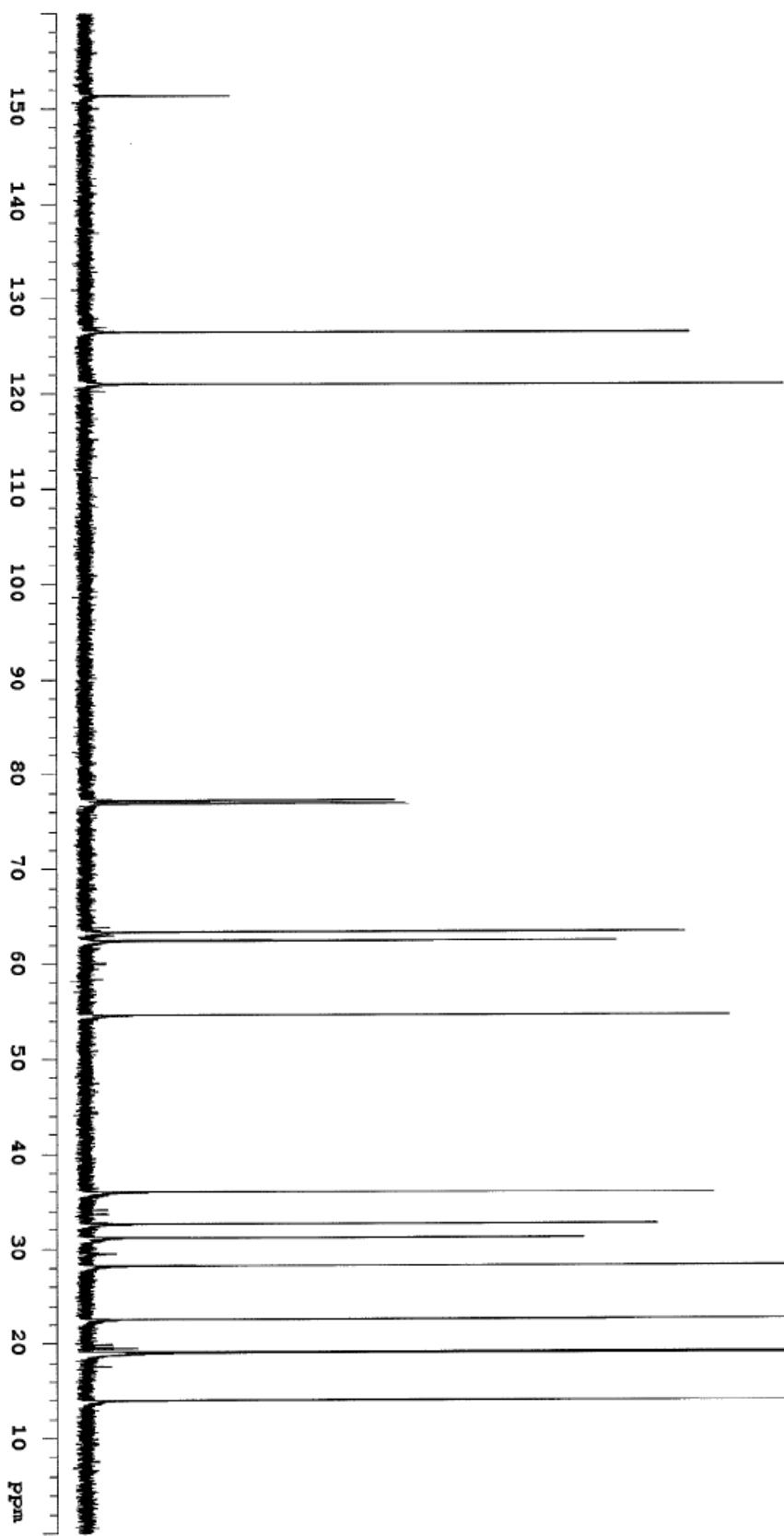
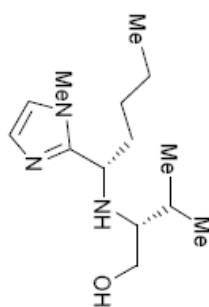
#### References:

- (1) D. A. Dickman, A. I. Meyers, G. A. Smith, and R. E. Gawley, *Org. Synth.*, 1990, **Coll. Vol. 7**, 530-533.
- (2) X. Sun, A. D. Worthy, and K. L. Tan, *Angew. Chem. Int. Ed.*, 2011, **50**, 8167-8171.
- (3) A. D. Worthy, X. Sun, and K. L. Tan, *J. Am. Chem. Soc.*, 2012, **734**, 7321-7324.
- (4) Y. Suzuki and H. Takahashi, *Chem. Pharm. Bull.*, 1983, **31**, 2895-2898.
- (5) H. J. Bryant, C. Y. Dardonville, T. J. Hodgkinson, M. B. Hursthouse, K. M. A. Malik, and M. Shipman, *J. Chem Soc., Perkins Trans. 1*, 1998, 1249-1255.
- (6) S. E. Schaus, B. D. Brandes, J. F. Larrow, M. Tokunaga, K. B. Hansen, A. E. Gould, M. E. Furrow, and E. N. Jacobsen, *J. Am. Chem. Soc.*, 2002, **124**, 1307-1315.
- (7) L. T. Kliman, S. N. Mlynarski, and J. P. Morken, *J. Am. Chem. Soc.*, 2009, **131**, 13210-13211.
- (8) J. Cossy, D. Bauer, and V. Bellosta, *Tetrahedron*, 2002, **58**, 5909-5922.
- (9) E. Santaniello, P. Ferraboschi, and P. Grisenti, *Tetrahedron Lett.*, 1990, **31**, 5657-5660.
- (10) J. Patterson and S. T. Sigurdsson, *J. Chem. Educ.*, 2005, **82**, 1049-1050.

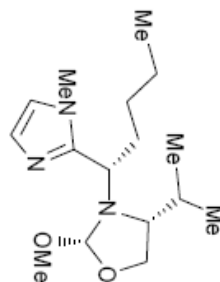
Sample Name :  
ZXG-TI-13-column3-f-9-12-si  
Archive directory :  
Sample directory :  
FidFile : ZXG-TI-13-column3-f-9-12-si-2  
Pulse Sequence : Proton (szpu1)  
Solvent : cdcl3  
Data collected on: May 8 2012



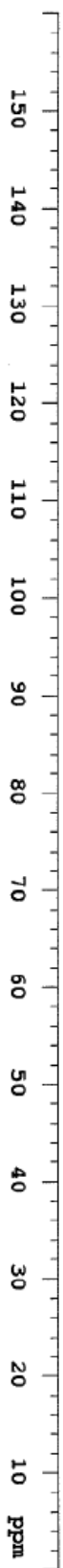
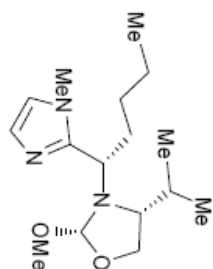
Sample Name:  
ZXG-II-13-column3-f-9-12-si-cl3  
Archive directory:  
Sample directory:  
PdfFile: ZXG-II-13-column3-f-9-12-si-cl3  
Pulse Sequence: Carbon (zgpg1)  
Solvent: cdcl3  
Data collected on: May 8 2012



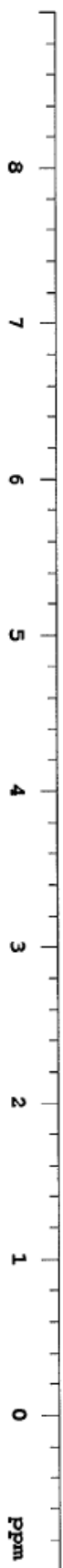
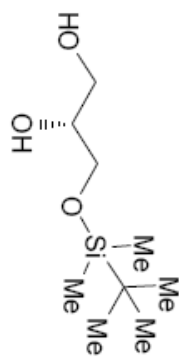
Sample Name:  
ZXG-II-XS-3-294  
Archive directory:  
Sample directory:  
FidFile: ZXG-II-XS-3-294  
Pulse Sequence: Proton (s2pul)  
Solvent: e6d6  
Data collected on: May 7 2012



Sample Name:  
ZXG-II-XS-3-294  
Archive directory:  
Sample directory:  
FidFile: ZXG-II-XS-3-294-c13  
Pulse sequence: Carbon (zgpg3)  
Solvent: c6d6  
Data collected on: May 7 2012



Sample Name:  
ZXG-II-15-1  
Archive directory:  
Sample directory:  
FidFile: ZXG-II-15-1  
Pulse Sequence: Proton (zgpg30)  
Solvent: cdcl3  
Data collected on: May 10 2012

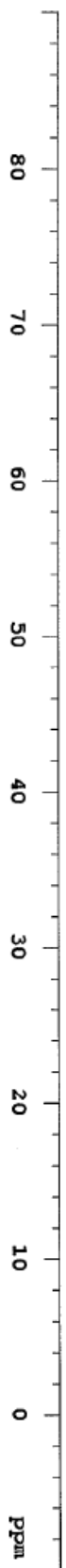
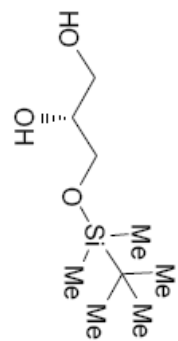


Sample Name:  
ZXG-II-15-1-cl3  
Archive directory:

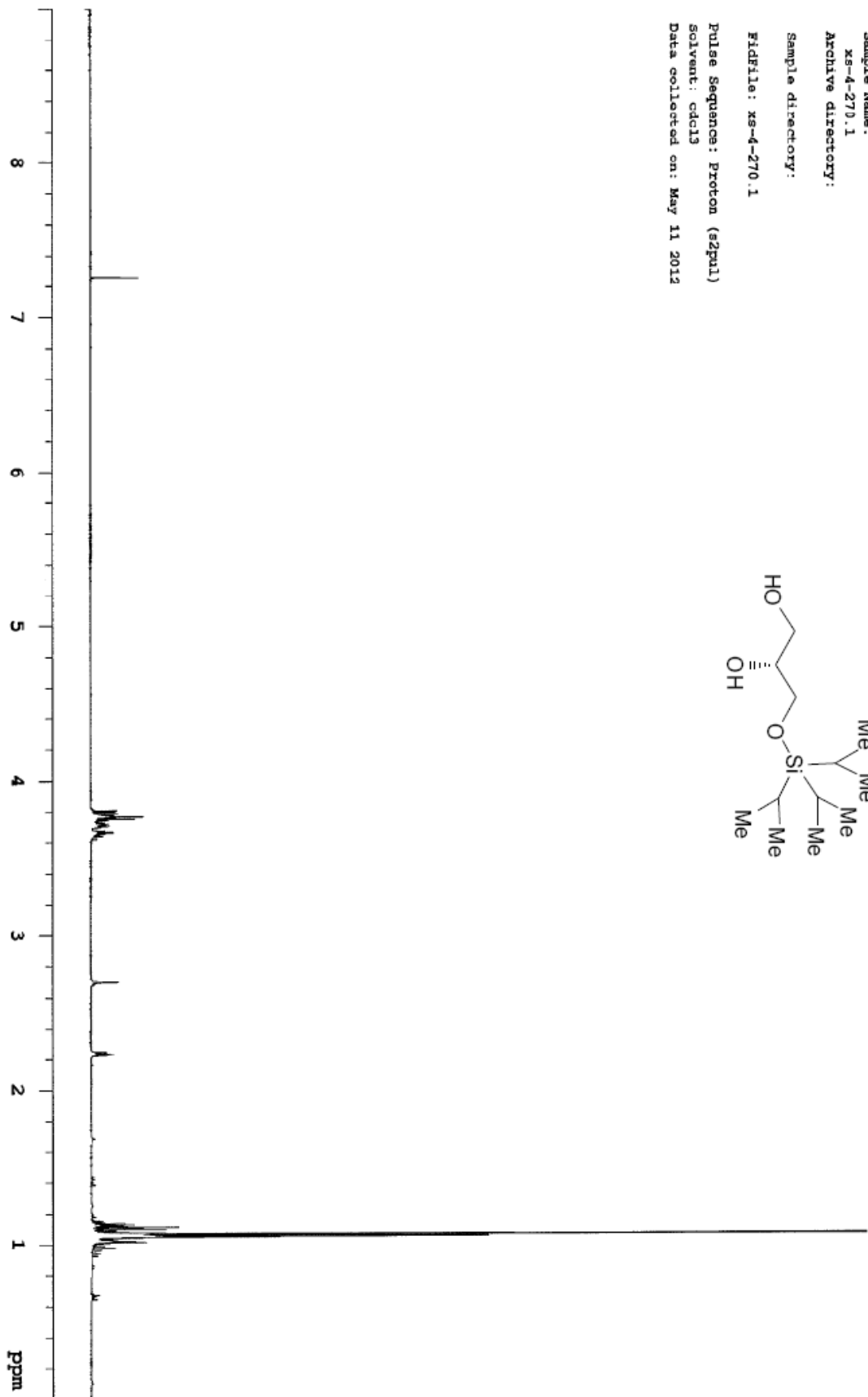
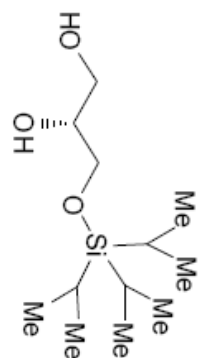
Sample directory:

FidFile: ZXG-II-15-1-cl3

Pulse Sequence: Carbon (zgpg1)  
Solvent: cdcl3  
Data collected on: May 10 2012

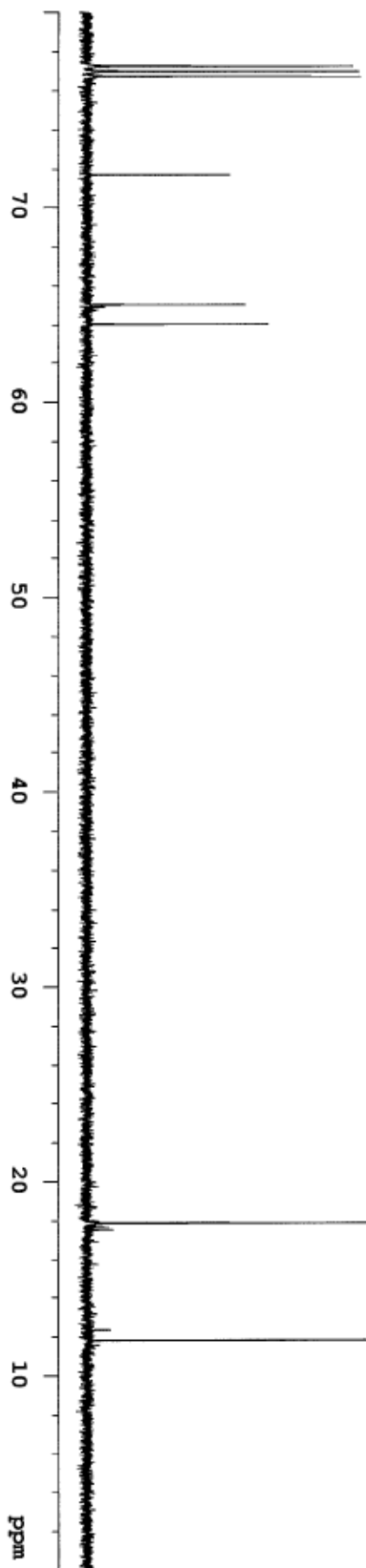
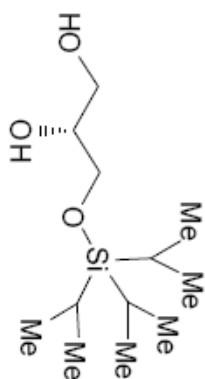


Sample Name: xs-4-270.1  
Archive directory:  
Sample directory:  
FidFile: xs-4-270.1  
Pulse Sequence: Proton (szpul)  
Solvent: cdcl3  
Data collected on: May 11 2012

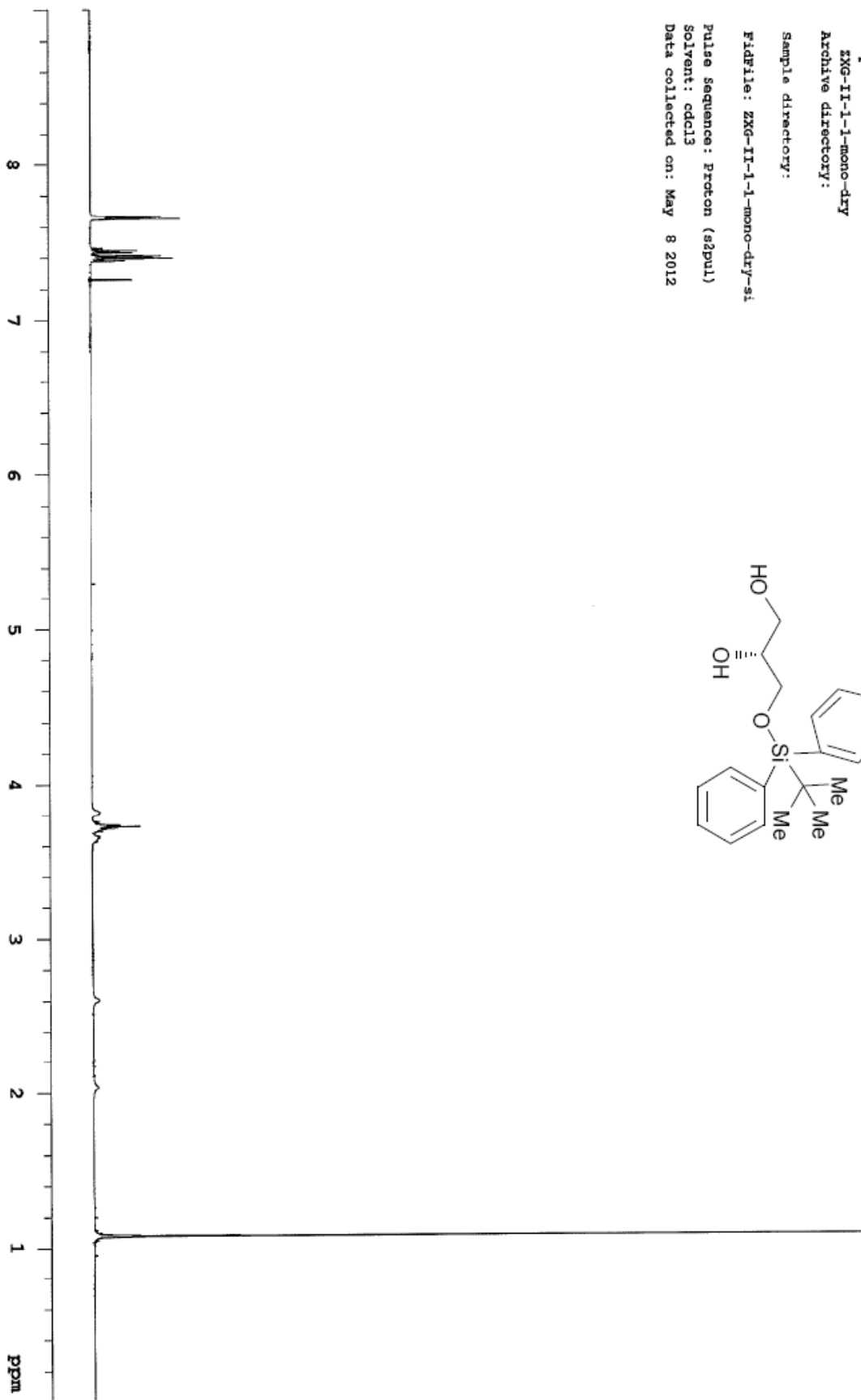
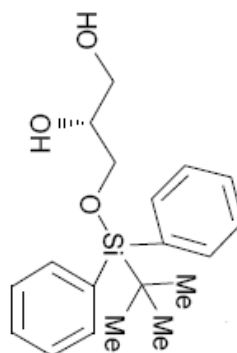




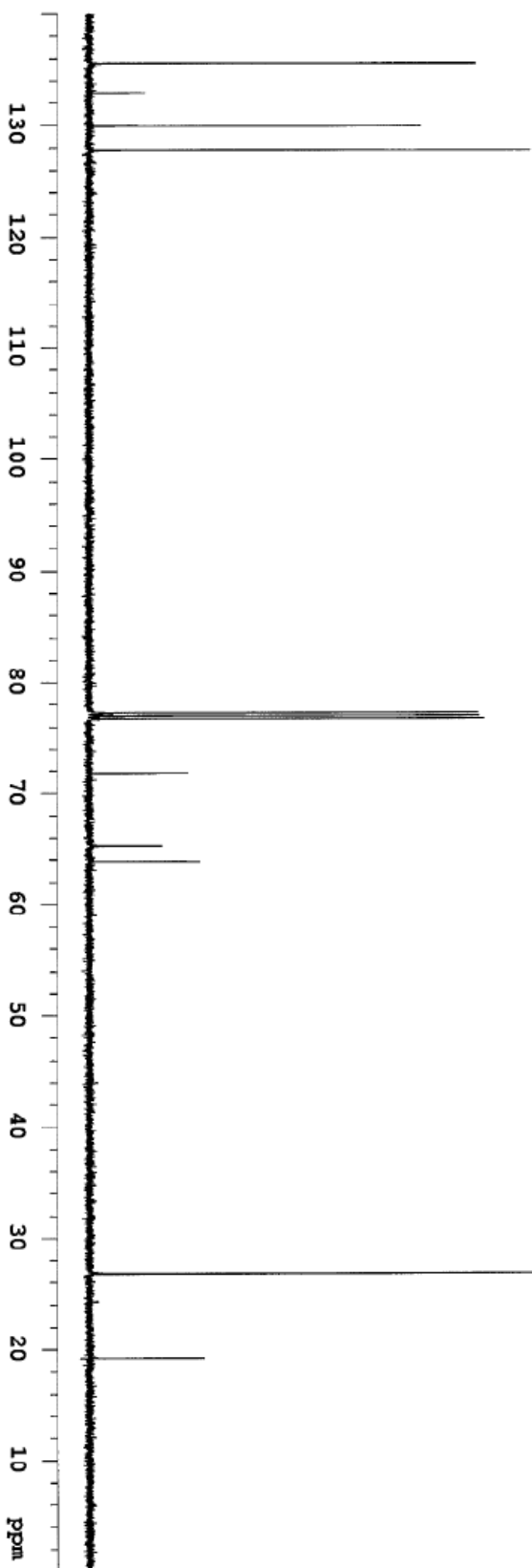
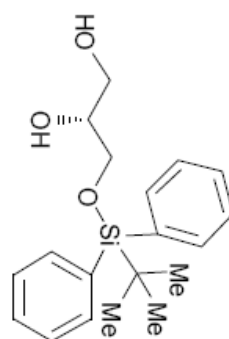
Sample Name:  
xs-4-270.1  
Archive directory:  
Sample directory:  
FidFile: xs-4-270.1-C  
Pulse Sequence: Carbon (zgpg3)  
Solvent: cdcl3  
Data collected on: May 11 2012



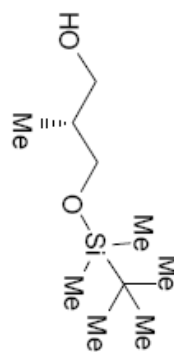
Sample Name:  
ZXG-II-1-1-mono-dry  
Archive directory:  
Sample directory:  
Fidfile: ZXG-II-1-1-mono-dry-si  
Pulse sequence: Proton (s2pu1)  
Solvent: cdcl3  
Data collected on: May 8 2012



Sample Name: ZKG-II-1-1-mono-dry-c13  
Archive directory:  
Sample directory:  
F1: File: ZKG-II-1-1-mono-dry-c13  
Pulse Sequence: Carbon (zgpg30)  
Solvent: cdcl3  
Data collected on: May 8 2012



Sample Name:  
ZKG-II-25-2-dry  
Archive directory:  
Sample directory:  
FIDFile: ZKG-II-25-2-dry  
Pulse Sequence: Proton (s2pul)  
Solvent: cdcl3  
Data collected on: Jun 5 2012



Sample Name :  
ZKG-II-25-2-dry  
Archive directory :

Sample directory :

Fidfile: ZKG-II-25-2-dry-c13

Pulse Sequence: Carbon (s2pul)

Solvent: cdcl3

Data collected on: Jun 5 2012

