# Lewis Base Activation of Lewis Acids: Catalytic, Enantioselective Addition of Glycolate-Derived Silyl Ketene Acetals to Aldehydes

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SUPPORTING INFORMATION

## General Experimental

All reactions were performed in oven-dried (140 °C) or flame-dried glassware under an atmosphere of dry $N_2$. All reaction temperatures correspond to internal temperatures measured by Teflon-coated thermocouples unless otherwise noted. Reaction solvents including dichloromethane (HPLC Grade), diethyl ether (BHT stabilized HPLC Grade), and tetrahydrofuran (THF, HPLC Grade) were dried by percolation through a column packed with neutral alumina and a column packed with Q5 reactant, a supported copper catalyst for scavenging oxygen, under a positive pressure of argon. Ethanol was freshly distilled from $CaH_2$. *n*-Butanethiol was freshly distilled from $CaCl_2$. *N*,*N*-Dimethylformamide (DMF, ACS grade) and dimethylsulfoxide (DMSO, ACS grade) were distilled and then stored over 3 Å molecular sieves prior to use. Solvents for chromatography and filtration were: hexane (ACS Grade), pentane (ACS Grade), ethyl acetate (ACS Grade), dichloromethane (ACS Grade), and methanol (distilled from Mg). Column chromatography was performed using EM Science 230-400-mesh silica gel. Benzaldehyde, 4-anisaldehyde, 4-tolualdehyde, 4-trifluoromethyl-benzaldehyde, 2-tolualdehyde, (*E*)-cinnamaldehyde, α -methyl-*E*-cinnamaldehyde, 3-methylcrotonaldehyde, hydrocinnamaldehyde, cyclohexanecarboxaldehyde, isovaleraldehyde, *tert*-butyldimethylsilyl chloride (TBSCl), α-methoxyacetic acid, and methyl glycolate (TCI) were freshly distilled prior to use. 4-Chlorobenzaldehyde and 2-naphthaldehyde were sublimed prior to use. 1-naphthaldehyde was purified according to the previously published method.[1] Silicon tetrachloride was heated at reflux for 24 h and then distilled prior to use. *neo*-Pentanol, *N*,*N*-dimethylaminopyridine (DMAP), *N*,*N*'-dicyclohexylcarbodiimide, imidazole, aluminum chloride, *N*-methyl-*N*-nitroso-*p*-toluenesulfonamide (Diazald®), sodium hydride, platinum oxide, diisobutylaluminum hydride (DIBAL), 3,5-dinitrobenzoyl chloride, 2,2-dimethoxypropane, oxalyl chloride, potassium hydroxide, and potassium hexamethyldisilazide (KHMDS) were used

without further purification. Diisopropylethylamine, triethylamine, pyridine, trimethylsilyl chloride (TMSCl), and *tert*-butyldiphenylsilyl chloride (TBDPSCl) were freshly distilled from $CaH_2$ prior to use. *iso*-Propanol, *tert*-butanol, α,α-dimethylbenzyl alcohol, 3-methyl-3-pentanol, 3-ethyl-3-pentanol, and 2,4-dimethyl-3-pentanol were distilled from $K_2CO_3$ prior to use. Chloroacetic acid was recrystallized from $CH_2Cl_2$ prior to use. Potassium iodide was dried in the oven (140 $^oC$) for 24h prior to use. Lithium aluminum hydride (LAH) was dissolved in THF, filtered, and used as a solution.

$^1H$ NMR, $^{13}C$ NMR, $^{19}F$ NMR spectra were recorded at 500 MHz, $^1H$; 126 MHz, $^{13}C$; 376 MHz, $^{19}F$. Spectra were referenced to residual chloroform (7.26 ppm, $^1H$, 77.00 ppm, $^{13}C$) and $^{19}F$ spectra were referenced to residual $CFCl_3$ (0.00 ppm). Chemical shifts are reported in ppm, multiplicities are indicated by s (singlet), d (doublet), t (triplet), q (quartet), p (pentet), h (hextet), hept. (heptet), m (multiplet), bs (broad singlet), and bd (broad doublet). Coupling constants, *J*, are reported in Hertz. $^1H$ NMR and $^{13}C$ NMR assignments are corroborated by 2D experiments (COSY, HMQC, HMBC, and NOESY1D). Mass spectroscopy was on a 70-VSE spectrometer. ESI mass spectra were performed on a Q-Tof spectrometer. Data are reported in the form of (m/z). Infrared spectra (IR) were recorded in NaCl cells. Peaks are reported in $cm^{-1}$ with indicated relative intensities: s (strong, 67-100%); m (medium, 34-66%); w (weak, 0-33%).. Melting points (mp) were determined in sealed tubes and are corrected. Boiling points (bp) were head temperatures during distillations unless otherwise noted. Kugelrohr distillation temperatures are air bath temperatures (ABT). Analytical thin-layer chromatography was performed on silica gel plates with QF-254 indicator or silica gel 60 $F_{254}$ TLC plates. Visualization was accomplished with UV(254), and potassium permanganate ($KMnO_4$) staining solutions. Optical rotation data are reported as follows: concentration (c = g/100 mL), and solvent. Analytical supercritical fluid chromatography (CSP-SFC) was performed using Daicel Chiralcel OD and OJ,

Daicel Chiralpak AD and AS columns as well as a Regis Whelk-O1 column. Fourier transform IR analysis was performed using a ReactIR 1000 purchased from ASI Applied Systems INC., 8223 Coverleaf Drive, Suite 120, Millersvile, MD 21108. Reactions were monitored using 5/8" Dicomp probe fitted to an MCT detector. Acquisitions were recorded using software version 3.0.

**Literature Preparations**

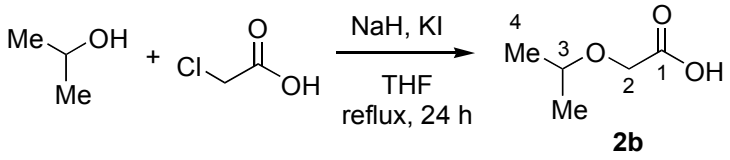
(*R*,*R*)-*N*,*N*'-bis[4,5-dihydro-3,5-dimethyl-4-(3Hdinaphtho[2,1-d:1',2'-f][1,3,2]-2-oxo-diazaphosphephepino)]-*N*,*N*'-dimethyl-1,5-pentanediamine ((*R*,*R*)-**1**)[2], 2-(phenylmethoxy)acetic acid[3], 2-methoxyacetic acid methyl ester[4], 2-methoxyacetic acid 1,1-dimethylethyl ester[5], 2-[2-(trimethylsilyl)ethoxy]acetic acid[6], 2-benzofuraldehyde (**5i**)[7], 3-(phenylmethoxy)propanal (**5l**)[8], and 2-(phenylmethoxy)acetaldehyde (**5m**)[9] were prepared according to established procedures.

**Experimental Procedures**

**Preparations of 2-Alkoxyacetic Acids**

**General Procedure 1. Alkoxylation of Chloroacetic Acid.**

**Preparation of 2-(1-Methylethoxy)acetic Acid[10] (2b) (Scheme 4)**




**2b**

To a flame-dried, 250-mL, 2-necked round-bottomed flask fitted with a magnetic stir bar, a reflux condenser with a gas inlet tube, and a septum were added NaH (2.4 g, 60% in mineral oil, 60 mmol, 3.0 equiv), KI (166 mg, 1 mmol, 0.05 equiv), and THF (180 mL). The slurry was cooled to 0 $^{o}$C in an ice-water bath and then *i*-PrOH (2.297 mL, 30 mmol, 1.5 equiv) was added dropwise. After 15 min, a solution of chloroacetic acid (1.890 g, 20 mmol) in THF (20 mL) was added slowly over 15 min. The resulting slurry was vigorously stirred and heated to reflux in an oil-bath. After 24 h, the reaction mixture was cooled to rt and quenched by the slow addition of $H_2O$ (50 mL) then was transferred to a 500-mL separatory funnel where the aqueous layer was separated. The organic layer was extracted with $H_2O$ (2 ×100 mL). The aqueous layers were combined and washed with ethyl acetate (3 × 100 mL). The resulting aqueous layer was acidified with 1*N* HCl (20 mL) and extracted with ethyl acetate (50 mL). The acidification and extraction was repeated until no more material was visible. The combined organic layers were dried over $MgSO_4$ (8 g), filtered, and concentrated *in vacuo* (23 $^{o}$C, 30 mmHg). The residue was purified by column chromatography (22 mm diam., $CH_2Cl_2$/MeOH, 10/1) on silica gel (20 g) and then distilled through a 5-cm Vigreux column under reduced pressure to give **2b** (1.585 g, 67%) as a colorless oil.

Data for **2b**:

bp: 58-59 °C (1.0 mmHg)

$^1$H NMR: (500 MHz, $CDCl_3$)

10.19 (bs, 1 H, $CO_2H$), 4.11 (s, 2 H, HC(2)), 3.72 (hept., $J$ = 6.1, 1 H, HC(3)), 1.22 (d, $J$ = 6.1, 6 H, HC(4))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

175.0 (C(1)), 73.1 (C(3)), 65.2 (C(2)), 21.7 (C(4))

IR: (neat)

3451 (br), 2977 (s), 2933 (s), 2642 (m), 2555 (m), 1737 (s), 1644 (m), 1462 (m), 1433 (s), 1374 (s), 1335 (s), 1234 (s), 1179 (s), 1124 (s), 1018 (m), 910 (s), 832 (s), 801 (s), 673 (s)

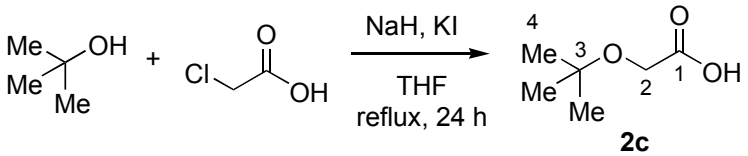
MS: (ESI)

141.0 ($M^+$+Na, 15), 163.0 (100), 259.1 (18), 303.0 (77), 361.0 (8), 443.1 (61), 444.1 (4), 501.0 (11)

HRMS: calcd for $C_5H_{10}O_3Na$: 141.0528, found 141.0533

TLC: $R_f$ 0.33 ($CH_2Cl_2$/MeOH, 10/1) [$KMnO_4$]

**Preparation of 2-(1,1-Dimethylethoxy)acetic Acid[10a,11] (2c) (Scheme 4)**




**2c**

Following General Procedure 1, NaH (2.4 g, 60% in mineral oil, 60 mmol, 3.0 equiv) was combined with KI (166 mg, 1 mmol, 0.05 equiv), *t*-BuOH (2.869 mL, 30 mmol, 1.5 equiv), and chloroacetic acid (1.890 g, 20 mmol) to yield, after column chromatography (22 mm diam.,

$CH_2Cl_2$/MeOH, 10/1) on silica gel (20 g) and distillation through a 5-cm Vigreux column under reduced pressure, **2c** (1.764 g, 67%) as a colorless oil.

Data for **2c**:

bp: 62-63 °C (1.0 mmHg)

$^1$H NMR: (500 MHz, $CDCl_3$)

9.91 (bs, 1 H, $CO_2H$), 4.04 (s, 2 H, HC(2)), 1.25 (s, 9 H, HC(4))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

173.6 (C(1)), 75.5 (C(3)), 60.1 (C(2)), 27.2 (C(4))

IR: (neat)

3454 (br), 2978 (s), 2939 (m), 2762 (m), 2646 (m), 2563 (m), 1738 (s), 1639 (m), 1466 (m), 1434 (m), 1393 (s), 1369 (s), 1240 (s), 1191 (s), 1114 (s), 1029 (m), 1013 (m), 878 (s), 764 (s), 741 (s), 659 (s)

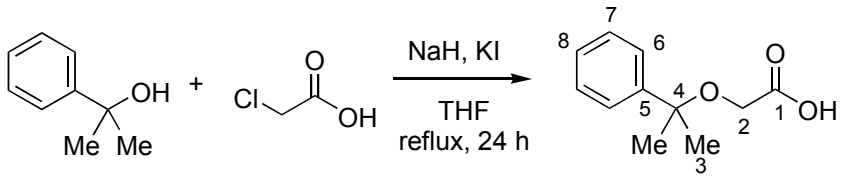
MS: (ESI)

155.1 ($M^+$+Na, 100), 177.0 (72), 211.1 (66), 331.1 (50)

HRMS: calcd for $C_6H_{12}O_3Na$: 155.0684, found 155.0684

TLC: $R_f$ 0.35 ($CH_2Cl_2$/MeOH, 10/1) [$KMnO_4$]

**Preparation of 2-(1-Methyl-1-phenylethoxy)acetic Acid (2d) (Scheme 4)**




**2d**

Following General Procedure 1, NaH (2.4 g, 60% in mineral oil, 60 mmol, 3.0 equiv) was combined with KI (166 mg, 1 mmol, 0.05 equiv), α,α-dimethylbenzylalcohol (4.199 mL, 30

mmol, 1.5 equiv), and chloroacetic acid (1.890 g, 20 mmol) to yield, after column chromatography (22 mm diam., $CH_2Cl_2$/MeOH, 10/1) on silica gel (20 g), **2d** (3.006 g, 77%) as pale-yellow crystals. An analytical sample was obtained as white crystals after recrystallization from $Et_2O$/hexane.

Data for **2d**:

mp: 69-70 °C ($Et_2O$/hexane)

$^1$H NMR: (500 MHz, $CDCl_3$)

10.84 (bs, 1 H, $CO_2H$), 7.42-7.40 (m, 2 H, HC(6)), 7.38-7.35 (m, 2 H, HC(7)), 7.30-7.27 (m, 1 H, HC(8)), 3.85 (s, 2 H, HC(2)), 1.62 (s, 6 H, HC(3))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

174.6 (C(1)), 144.2 (C(5)), 128.6 (C(7)), 127.6 (C(8)), 125.6 (C(6)), 78.7 (C(4)), 61.2 (C(2)), 28.0 (C(3))

IR: (neat)

3088 (m), 3061 (m), 3028 (m), 2981 (s), 2933 (m), 2656 (m), 2568 (m), 1736 (s), 1495 (m), 1448 (m), 1383 (m), 1366 (m), 1338 (m), 1262 (s), 1200 (s), 1154 (s), 1113 (s), 1076 (m), 1030 (m), 934 (m), 913 (m), 861 (m), 766 (s), 702 (s)

MS: (ESI)

99.0 (12), 119.1 (8), 217.1 ($M^+$+Na, 100), 218.1 (5), 239.0 (22), 335.1 (33), 337.1 (4)

HRMS: calcd for $C_{11}H_{14}O_3Na$: 217.0841, found 217.0841

TLC: $R_f$ 0.29 ($CH_2Cl_2$/MeOH, 10/1) [UV(254)/$KMnO_4$]

Analysis: $C_{11}H_{14}O_3$ (194.23)

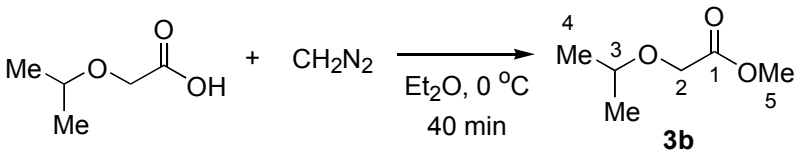
Calcd: C, 68.02%; H, 7.27%

Found: C, 67.71%; H, 7.28%

**Preparations of 2-Alkoxyacetic Acid Methyl Ester**

**General Procedure 2. Esterification of 2-Alkoxyacetic Acid with Diazomethane.**

**Preparation of 2-(1-Methylethoxy)acetic Acid Methyl Ester[12] (3b) (Scheme 4)**




The receiving flask of an Aldrich mini diazald® apparatus was charged with 2-(1-methylethoxy)acetic acid (1.432 g, 12.122 mmol) and $Et_2O$ (12 mL). The solution was cooled to 0 °C in an ice-water bath. Diazomethane (ca. 612 mg, 14.547 mmol, 1.2 equiv), which was generated according to Aldrich tech note AL-180 (70% yield was assumed.), was distilled to the receiving flask over 40 min. The excess $CH_2N_2$ was quenched by acetic acid (1*N* in $Et_2O$, 5 mL). The resulting solution was transferred to a 125-mL separatory funnel and was washed with sat. aq. $NaHCO_3$ solution (3 × 20 mL), then was dried over $MgSO_4$ (4 g), and filtered. The solvent was removed by fractional distillation under $N_2$. The residue was distilled through a 5-cm Vigreux column under reduced pressure to give **3b** (1.325 g, 83%) as a colorless oil.

Data for **3b**:

bp: 56-57 °C (20 mmHg)

$^1$H NMR: (500 MHz, $CDCl_3$)

4.09 (s, 2 H, HC(2)), 3.75 (s, 3 H, HC(5)), 3.68 (hept., $J$ = 6.1, 1 H, HC(3)), 1.21 (d, $J$ = 6.1, 6 H, HC(4))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

171.3 (C(1)), 72.6 (C(3)), 65.7 (C(2)), 51.8 (C(5)), 21.7 (C(4))

IR: (neat)

2975 (m), 1759 (s), 1739 (s), 1458 (m), 1438 (m), 1382 (m), 1372 (m), 1334

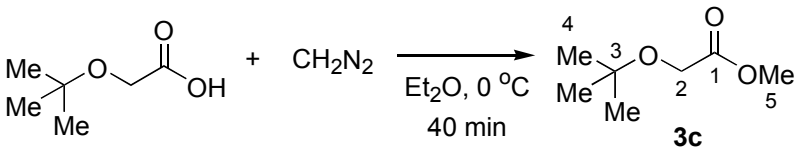
(m), 1281 (m), 1210 (s), 1179 (s), 1130 (s), 990 (m), 909 (m), 886 (m), 846 (m), 827 (m), 801 (m), 668 (m), 655 (m)

MS: (ESI)

155.1 ($M^+$+Na, 100), 169.1 (11)

HRMS: calcd for $C_6H_{12}O_3Na$: 155.0684, found 155.0679

**Preparation of 2-(1,1-Dimethylethoxy)acetic Acid Methyl Ester[11d] (3c) (Scheme 4)**




**3c**

Following General Procedure 2, 2-(1,1-dimethylethoxy)acetic acid (1.564 g, 11.834 mmol) was combined with $CH_2N_2$ (ca. 597 mg, 14.201 mmol, 1.2 equiv) to yield, after distillation through a 5-cm Vigreux column under reduced pressure, **3c** (1.464 g, 85%) as a colorless oil.

Data for **3c**

bp: 62-63 °C (20 mmHg)

$^1$H NMR: (500 MHz, $CDCl_3$)

4.04 (s, 2 H, HC(2)), 3.75 (s, 3 H, HC(5)), 1.24 (s, 9 H, HC(4))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

171.7 (C(1)), 74.5 (C(3)), 60.8 (C(2)), 51.9 (C(5)), 27.2 (C(4))

IR: (neat)

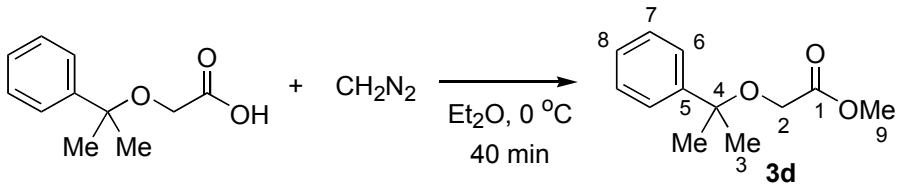
2978 (s), 1766 (s), 1738 (s), 1463 (m), 1438 (s), 1390 (s), 1366 (s), 1290 (s), 1238 (s), 1192 (s), 1122 (s), 1029 (m), 996 (m), 924 (m), 890 (m), 857 (s), 762 (m), 739 (m), 698 (m)

MS: (ESI)

169.1 ($M^+$+Na, 100)

HRMS: calcd for $C_7H_{14}O_3Na$: 169.0841, found 169.0835

**Preparation of 2-(1-Methyl-1-phenylethoxy)acetic Acid Methyl Ester (3d) (Scheme 4)**




Following General Procedure 2, 2-(1-methyl-1-phenylethoxy)acetic acid (2.900 g, 14.931 mmol) was combined with $CH_2N_2$ (ca. 753 mg, 17.917 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., pentane/$Et_2O$, 5/1) on silica gel (20 g) and distillation through a 5-cm Vigreux column under reduced pressure, **3d** (2.888 g, 93%) as a colorless oil.

Data for **3d**:

bp: 78 °C (1 mmHg)

$^1$H NMR: (500 MHz, $CDCl_3$)

7.44-7.42 (m, 2 H, HC(6)), 7.36-7.33 (m, 2 H, HC(7)), 7.28-7.25 (m, 1 H, C(8)), 3.82 (s, 2 H, HC(2)), 3.71 (s, 3 H, HC(9)), 1.59 (s, 6 H, HC(3))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

171.2 (C(1)), 144.9 (C(5)), 128.4 (C(7)), 127.2 (C(8)), 125.8 (C(6)), 78.2 (C(4)), 61.8 (C(2)), 51.7 (C(9)), 28.1 (C(3))

IR: (neat)

3061 (m), 3027 (m), 2980 (s), 2953 (s), 1763 (s), 1738 (s), 1602 (m), 1495 (s), 1447 (s), 1438 (s), 1382 (s), 1364 (s), 1289 (s), 1261 (s), 1199 (s), 1160 (s), 1116 (s), 1076 (s), 1030 (s), 1004 (m), 934 (m), 850 (s), 767 (s), 702 (s)

MS: (ESI)

215.1 (5), 231.1 ($M^+$+Na, 100), 232.1 (6)

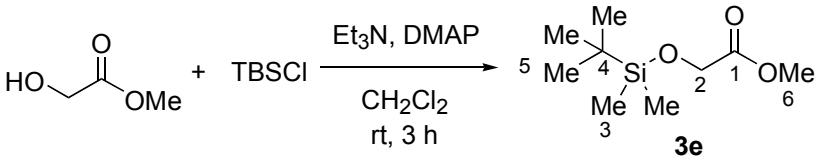
HRMS: calcd for $C_{12}H_{16}O_3Na$: 231.0997, found 231.0990

TLC: $R_f$ 0.35 (pentane/$Et_2O$, 5/1) [UV(254)/$KMnO_4$]

Analysis: $C_{12}H_{16}O_3$ (208.25)

| | | |
|---|---|---|
| Calcd: | C, 69.21%; | H, 7.74% |
| Found: | C, 69.12%; | H, 7.87% |

**Preparation of [[(1,1-Dimethylethyl)dimethylsilyl]oxy]acetic Acid Methyl Ester[13] (3e) (Scheme 4)**




To a flame-dried, 100-mL, 2-necked round-bottomed flask fitted with a magnetic stir bar, a gas inlet tube, and a septum were added DMAP (183 mg, 1.5 mmol, 0.1 equiv), $CH_2Cl_2$ (20 mL), methyl glycolate (1.158 mL, 15 mmol), and $Et_3N$ (2.300 mL, 16.5 mmol, 1.1 equiv). The solution was cooled to 0 °C in an ice-water bath and then a solution of TBSCl (2.487 g, 16.5 mmol, 1.1 equiv) in $CH_2Cl_2$ (10 mL) was added dropwise over 10 min. The ice-water bath was removed and the reaction mixture was stirred at rt. After 3 h, the reaction mixture was transferred to a 125-mL separatory funnel and was washed with $H_2O$ (30 mL) and sat. aq. $NH_4Cl$ solution (30 mL). The combined aqueous layers were extracted with $CH_2Cl_2$ (30 mL). The combined organic layers were dried over $MgSO_4$ (4 g), filtered, and concentrated *in vacuo* (23 °C, 30 mmHg). The residue was purified by column chromatography (22 mm diam., pentane/$Et_2O$, 30/1) on silica gel (20 g) and then was distilled through a 5-cm Vigreux column under reduced

pressure to give **3e** (2.585 g, 84%) as a colorless oil.

Data for **3e**:

bp: 39-42 °C (1 mmHg)

$^1$H NMR: (500 MHz, $CDCl_3$)

4.24 (s, 2 H, HC(2)), 3.73 (s, 3 H, HC(6)), 0.91 (s, 9 H, HC(5)), 0.10 (s, 6 H, HC(3))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

172.2 (C(1)), 61.7 (C(2)), 51.7 (C(6)), 25.7 (C(5)), 18.4 (C(4)), –5.5 (C(3))

IR: (neat)

2955 (s), 2931 (s), 2896 (s), 2859 (s), 1765 (s), 1742 (s), 1473 (s), 1464 (s), 1437 (s), 1409 (m), 1389 (m), 1362 (m), 1257 (s), 1213 (s), 1189 (s), 1150 (s), 1007 (m), 992 (m), 939 (m), 888 (m), 839 (s), 816 (s), 780 (s), 696 (m), 663 (m)

MS: (ESI)

227.1 ($M^+$+Na, 100), 228.1 (4)

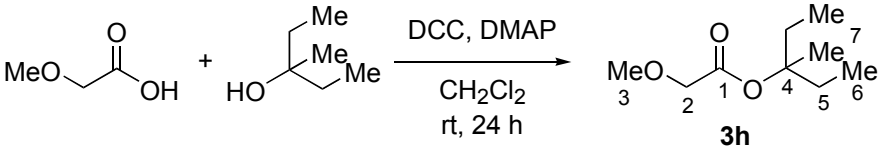
HRMS: calcd for $C_9H_{20}O_3SiNa$: 227.1079, found 227.1073

TLC: $R_f$ 0.34 (pentane/$Et_2O$, 30/1) [$KMnO_4$]

**Preparations of 2-Methoxyacetic Acid Alkyl Ester**

**General Procedure 3. DCC-DMAP coupling of 2-Methoxyacetic Acid with Alcohols.**

**Preparation of 2-Methoxyacetic Acid 1-Ethyl-1-methylpropyl Ester (3h) (Scheme 5)**




**3h**

To a flame-dried, 100-mL, 2-necked round-bottomed flask fitted with a magnetic stir bar,

a gas inlet tube, and a septum were added DCC (4.746 g, 23 mmol, 1.15 equiv), $CH_2Cl_2$ (50 mL), and 3-methyl-3-pentanol (2.480 mL, 20 mmol). The solution was cooled to 0 °C in an ice-water bath and then a solution of 2-methoxyacetic acid (1.841 mL, 24 mmol, 1.2 equiv) and DMAP (611 mg, 5 mmol, 0.25 equiv) in $CH_2Cl_2$ (10 mL) was added dropwise over 10 min. After 20 min, the ice-water bath was removed and the reaction mixture was stirred at rt. After 24 h, the reaction mixture was filtered through a glass frit. The filtrate was transferred to a 125-mL separatory funnel and was washed with sat. aq. $NaHCO_3$ solution (2 × 50 mL) and sat. aq. $NH_4Cl$ solution (2 × 50 mL). The resulting organic layer was dried over $MgSO_4$ (8 g), filtered, and concentrated *in vacuo* (23 °C, 30 mmHg). The residue was diluted with $Et_2O$ (10 mL), filtered, and concentrated *in vacuo* (23 °C, 30 mmHg). The residue was purified by column chromatography (22 mm diam., pentane/$Et_2O$, 10/1) on silica gel (20 g) and then was distilled from $K_2CO_3$ (10 mg) through a 5-cm Vigreux column under reduced pressure to give **3h** (2.803 g, 80%) as a colorless oil.

Data for **3h**:

bp: 66 °C (10 mmHg)

$^1$H NMR: (500 MHz, $CDCl_3$)

3.91 (s, 2 H, HC(2)), 3.42 (s, 3 H, HC(3)), 1.89 (dq, $J$ = 14.2, 7.6, 2 H, HC(5)), 1.76 (dq, $J$ = 14.2, 7.6, 2 H, HC(5)), 1.39 (s, 3 H, HC(7)), 0.84 (dd, $J$ = 7.6, 7.6, 6 H, HC(6))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

169.3 (C(1)), 86.8 (C(4)), 70.0 (C(2)), 59.1 (C(3)), 30.4 (C(5)), 22.8 (C(7)), 7.9 (C(6))

IR: (neat)

2977 (s), 2943 (m), 2885 (m), 2827 (m), 1749 (s), 1726 (s), 1462 (m), 1422 (m),

1376 (m), 1349 (m), 1293 (m), 1221 (s), 1199 (s), 1154 (m), 1128 (s), 1072 (m), 1019 (m), 1007 (m), 975 (m), 967 (m), 946 (m), 927 (m), 896 (m), 852 (m)

MS: (ESI)

113.0 (12), 149.0 (6), 197.1 ($M^+$+Na, 100), 198.1 (5)

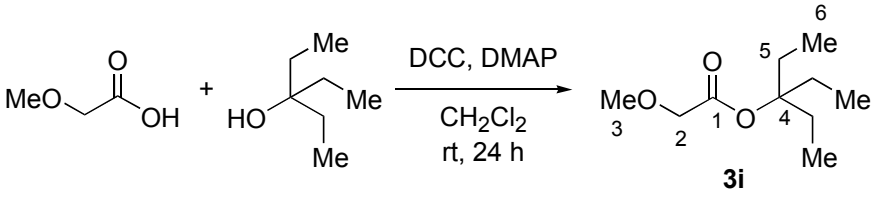
HRMS: calcd for $C_9H_{18}O_3Na$: 197.1154, found 197.1152

TLC: $R_f$ 0.35 (pentane/$Et_2O$, 10/1) [$KMnO_4$]

Analysis: $C_9H_{18}O_3$ (174.24)

| | | |
|---|---|---|
| Calcd: | C, 62.04%; | H, 10.41% |
| Found: | C, 61.81%; | H, 10.71% |

**Preparation of 2-Methoxyacetic Acid 1,1-Diethylpropyl Ester (3i) (Scheme 5)**




Following General Procedure 3, DCC (3.560 g, 17.25 mmol, 1.15 equiv) was combined with 3-ethyl-3-pentanol (2.115 mL, 15 mmol), 2-methoxyacetic acid (1.381 mL, 18 mmol, 1.2 equiv), and DMAP (458 mg, 3.75 mmol, 0.25 equiv) to yield, after column chromatography (30 mm diam., pentane/$Et_2O$, 10/1) on silica gel (40 g) and short path distillation under reduced pressure, **3i** (1.258 g, 45%) as a colorless oil.

Data for **3i**:

bp: 83-85 $^oC$ (10 mmHg)

$^1H$ NMR: (500 MHz, $CDCl_3$)

3.93 (s, 2 H, HC(2)), 3.42 (s, 3 H, HC(3)), 1.84 (q, $J$ = 7.5, 6 H, HC(5)), 0.81

(t, $J$ = 7.5, 9 H, HC(6))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

169.1 (C(1)), 89.6 (C(4)), 69.9 (C(2)), 59.1 (C(3)), 26.8 (C(5)), 7.6 (C(6))

IR: (neat)

2974 (s), 2945 (s), 2884 (s), 2827 (m), 1748 (s), 1724 (s), 1459 (s), 1422 (m), 1382 (m), 1361 (m), 1337 (w), 1288 (m), 1219 (s), 1197 (s), 1128 (s), 1080 (m), 1041 (w), 1019 (m), 996 (w), 952 (s), 937 (m), 912 (m), 877 (m), 732 (m)

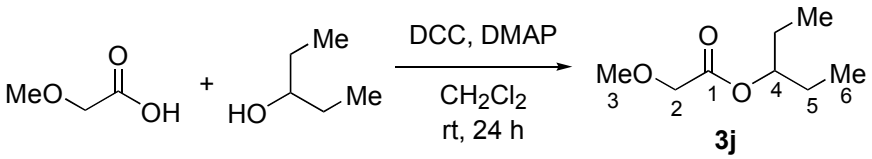
MS: (ESI)

113.0 (12), 211.1 ($M^+$+Na, 100), 212.1 (11), 285.2 (4)

HRMS: calcd for $C_{10}H_{20}O_3Na$: 211.1310, found 211.1318

TLC: $R_f$ 0.38 (pentane/$Et_2O$, 10/1) [$KMnO_4$]

**Preparation of 2-Methoxyacetic Acid 1-Ethylpropyl Ester[14] (3j) (Scheme 5)**




Following General Procedure 3, DCC (2.856 g, 13.841 mmol, 1.05 equiv) was combined with 3-pentanol (1.162 g, 13.182 mmol), 2-methoxyacetic acid (1.113 mL, 14.5 mmol, 1.1 equiv), and DMAP (322 mg, 2.636 mmol, 0.2 equiv) to yield, after column chromatography (30 mm diam., pentane/$Et_2O$, 10/1) on silica gel (30 g) and short path distillation under reduced pressure, **3j** (1.684 g, 80%) as a colorless oil.

Data for **3j**:

bp: 64-66 $^{o}$C (10.0 mmHg)

$^{1}$H NMR: (500 MHz, $CDCl_3$)

4.87 (tt, $J$ = 7.1, 5.4, 1 H, HC(4)), 4.02 (s, 2 H, HC(2)), 3.44 (s, 3 H, HC(3)), 1.65-1.52 (m, 4 H, HC(5)), 0.88 (dd, $J$ = 7.5, 7.5, 6 H, HC(6))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

170.2 (C(1)), 77.4 (C(4)), 69.9 (C(2)), 59.3 (C(3)), 26.4 (C(5)), 9.6 (C(6))

IR: (neat)

2971 (s), 2940 (s), 2882 (m), 2827 (m), 1753 (s), 1731 (s), 1461 (m), 1423 (m), 1386 (m), 1353 (m), 1282 (s), 1273 (s), 1196 (s), 1131 (s), 1050 (m), 1033 (m), 1020 (m), 998 (m), 955 (s), 924 (m), 904 (m), 864 (m), 778 (w), 722 (m)

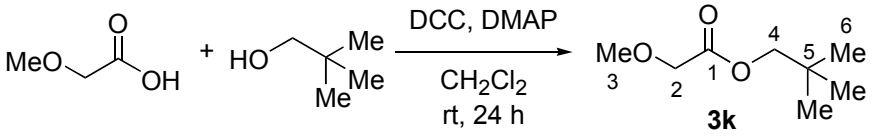
MS: (ESI)

183.1 ($M^+$+Na, 100), 184.1 (8)

HRMS: calcd for $C_8H_{16}O_3Na$: 183.0997, found 183.1004

TLC: $R_f$ 0.31 (pentane/$Et_2O$, 10/1) [$KMnO_4$]

**Preparation of 2-Methoxyacetic Acid 2,2-Dimethylpropyl Ester (3k) (Scheme 5)**




Following General Procedure 3, DCC (3.250 g, 15.75 mmol, 1.05 equiv) was combined with *neo*-pentanol (1.322 g, 15 mmol), 2-methoxyacetic acid (1.266 mL, 16.5 mmol, 1.1 equiv), and DMAP (367 mg, 3 mmol, 0.2 equiv) to yield, after column chromatography (22 mm diam., pentane/$Et_2O$, 10/1) on silica gel (20 g) and short path distillation under reduced pressure, **3k** (1.480 g, 62%) as a colorless oil.

Data for **3k**:

bp: 61-62 °C (10 mmHg)

$^1$H NMR: (500 MHz, $CDCl_3$)

4.05 (s, 2 H, HC(2)), 3.86 (s, 2 H, HC(4)), 3.45 (s, 3 H, HC(3)), 0.93 (s, 9 H, HC(6))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

170.4 (C(1)), 73.9 (C(4)), 69.7 (C(2)), 59.3 (C(3)), 31.3 (C(5)), 26.3 (C(6))

IR: (neat)

2960 (s), 2908 (m), 2873 (m), 2827 (m), 1760 (s), 1737 (s), 1480 (m), 1467 (m), 1453 (m), 1423 (m), 1382 (m), 1367 (m), 1286 (m), 1261 (m), 1191 (s), 1131 (s), 1038 (m), 1021 (m), 992 (m), 933 (m), 765 (w), 711 (w)

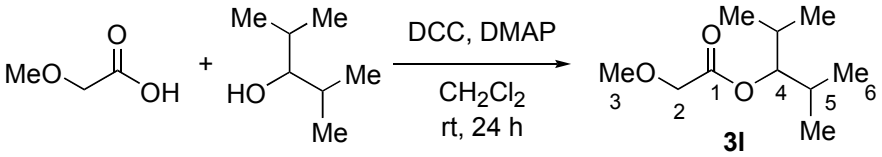
MS: (ESI)

167.1 (8), 183.1 ($M^+$+Na, 100), 184.1 (9)

HRMS: calcd for $C_8H_{16}O_3Na$: 183.0997, found 183.1000

TLC: $R_f$ 0.33 (pentane/$Et_2O$, 10/1) [$KMnO_4$]

**Preparation of 2-Methoxyacetic Acid 2-Methyl-1-(1-methylethyl)propyl Ester (3l) (Scheme 5)**




Following General Procedure 3, DCC (3.250 g, 15.75 mmol, 1.05 equiv) was combined with 2,4-dimethyl-3-pentanol (2.103 mL, 15 mmol), 2-methoxyacetic acid (1.266 mL, 16.5 mmol, 1.1 equiv), and DMAP (367 mg, 3 mmol, 0.2 equiv) to yield, after column

chromatography (22 mm diam., pentane/$Et_2O$, 10/1) on silica gel (20 g) and distillation through a 5-cm Vigreux column under reduced pressure, **3l** (2.436 g, 86%) as a colorless oil.

Data for **3l**:

bp: 81-82 $^oC$ (10 mmHg)

$^1H$ NMR: (500 MHz, $CDCl_3$)

4.69 (t, *J* = 6.2, 1 H, HC(4)), 4.04 (s, 2 H, HC(2)), 3.44 (s, 3 H, HC(3)), 1.90 (qqd, *J* = 6.6, 6.6, 6.6, 2 H, HC(5)), 0.87 (d, *J* = 6.8, 6 H, HC(6)), 0.85 (d, *J* = 6.8, 6 H, HC(6))

$^{13}C$ NMR: (126 MHz, $CDCl_3$)

170.3 (C(1)), 83.3 (C(4)), 69.6 (C(2)), 59.2 (C(3)), 29.3 (C(5)), 19.4 (C(6)), 17.1 (C(6))

IR: (neat)

2967 (s), 2937 (s), 2878 (s), 2827 (m), 1753 (s), 1734 (s), 1467 (s), 1423 (m), 1389 (s), 1372 (m), 1348 (m), 1322 (m), 1285 (s), 1268 (s), 1195 (s), 1128 (s), 1099 (s), 1020 (m), 1000 (m), 966 (s), 956 (s), 935 (m), 897 (m), 721 (m)

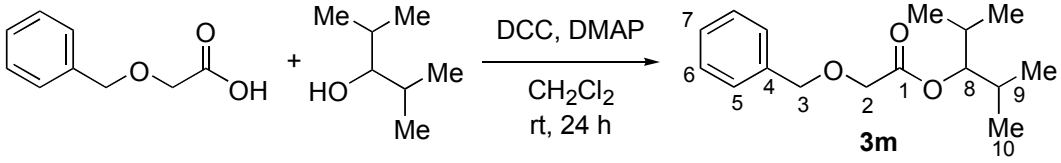
MS: (ESI)

211.1 ($M^+$+Na, 100), 212.1 (11)

HRMS: calcd for $C_{10}H_{20}O_3Na$: 211.1310, found 211.1320

TLC: $R_f$ 0.41 (pentane/$Et_2O$, 10/1) [$KMnO_4$]

**Preparation of 2-(Phenylmethoxy)acetic Acid 2-Methyl-1-(1-methylethyl)propyl Ester (3m) (Scheme 5)**




Following General Procedure 3, DCC (3.250 g, 15.75 mmol, 1.05 equiv) was combined with 2,4-dimethyl-3-pentanol (2.103 mL, 15 mmol), 2-(phenylmethoxy)acetic acid (2.742 g, 16.5 mmol, 1.1 equiv), and DMAP (367 mg, 3 mmol, 0.2 equiv) to yield, after column chromatography (22 mm diam., pentane/$Et_2O$, 10/1) on silica gel (20 g) and distillation through a 5-cm Vigreux column under reduced pressure, **3m** (3.217 g, 81%) as a colorless oil.

Data for **3m**:

bp: 88-89 °C (0.3 mmHg)

$^1$H NMR: (500 MHz, $CDCl_3$)

7.40-7.38 (m, 2 H, HC(5)), 7.37-7.34 (m, 2 H, HC(6)), 7.32-7.29 (m, 1 H, HC(7)), 4.72 (t, $J$ = 6.2, 1 H, HC(8)), 4.65 (s, 2 H, HC(3)), 4.14 (s, 2 H, HC(2)), 1.92 (m, 2 H, HC(9)), 0.90 (d, $J$ = 7.0, 6 H, HC(10)), 0.87 (d, $J$ = 6.9, 6 H, HC(10))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

170.5 (C(1)), 137.2 (C(4)), 128.4 (C(6)), 128.0 (C(5)), 127.9 (C(7)), 83.3 (C(8)), 73.2 (C(3)), 66.9 (C(2)), 29.3 (C(9)), 19.5 (C(10)), 17.2 (C(10))

IR: (neat)

3066 (w), 3032 (w), 2967 (s), 2937 (s), 2877 (m), 1751 (s), 1732 (s), 1497 (m), 1464 (m), 1456 (m), 1427 (m), 1389 (m), 1371 (m), 1282 (m), 1203 (s), 1127 (s), 1099 (m), 1030 (m), 1000 (m), 966 (m), 955 (m), 925 (m), 897 (m), 738 (m),

698 (m)

MS: (ESI)

91.0 (45), 99.1 (9), 167.1 (71), 168.1 (5), 265.2 (17), 282.2 (93), 287.1 ($M^+$+Na, 100), 288.2 (13)

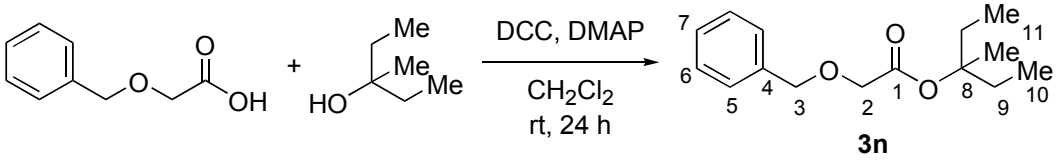
HRMS: calcd for $C_{16}H_{24}O_3Na$: 287.1623, found 287.1610

TLC: $R_f$ 0.42 (pentane/$Et_2O$, 10/1) [UV(254)/$KmnO_4$]

Analysis: $C_{16}H_{24}O_3$ (264.36)

| | | |
|---|---|---|
| Calcd: | C, 72.69%; | H, 9.15% |
| Found: | C, 72.61%; | H, 9.21% |

**Preparation of 2-(Phenylmethoxy)acetic Acid 1-Ethyl-1-methylpropyl Ester (3n) (Scheme 5)**




**3n**

Following General Procedure 3, DCC (3.560 g, 17.25 mmol, 1.15 equiv) was combined with 3-methyl-3-pentanol (1.860 mL, 15 mmol), 2-(phenylmethoxy)acetic acid (2.991 g, 18 mmol, 1.2 equiv), and DMAP (458 mg, 3.75 mmol, 0.25 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 10/1) on silica gel (20 g) and distillation through a 5-cm Vigreux column under reduced pressure, **3n** (2.570 g, 68%) as a colorless oil.

Data for **3n**:

bp: 90-91 $^oC$ (0.4 mmHg)

$^1H$ NMR: (500 MHz, $CDCl_3$)

7.39-7.34 (m, 4 H, HC(5), HC(6)), 7.32-7.28 (m, 1 H, HC(7)), 4.67 (s, 2 H,

HC(3)), 4.01 (s, 2 H, HC(2)), 1.91 (dq, $J$ = 14.2, 7.5, 2 H, HC(9)), 1.78 (dq, $J$ = 14.2, 7.5, 2 H, HC(9)), 1.41 (s, 3 H, HC(11)), 0.86 (dd, $J$ = 7.5, 7.5, 6 H, HC(10))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

169.4 (C(1)), 137.4 (C(4)), 128.4 (C(6)), 128.0 (C(5)), 127.9 (C(7)), 86.9 (C(8)), 73.2 (C(3)), 67.5 (C(2)), 30.4 (C(9)), 22.8 (C(11)), 8.0 (C(10))

IR: (neat)

3090 (w), 3065 (w), 3032 (w), 2975 (s), 2942 (m), 2883 (m), 1747 (s), 1725 (m), 1497 (w), 1456 (m), 1428 (w), 1384 (m), 1348 (w), 1294 (m), 1213 (s), 1127 (s), 1073 (w), 1030 (w), 1006 (w), 966 (w), 907 (w), 852 (m), 738 (m), 698 (m)

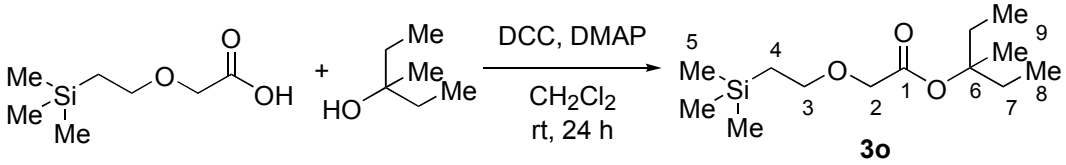
MS: (ESI)

189.1 (22), 273.1 ($M^+$+Na, 100), 274.2 (7)

HRMS: calcd for $C_{15}H_{22}O_3Na$: 273.1467, found 273.1460

TLC: $R_f$ 0.46 (hexane/EtOAc, 10/1) [UV(254)/$KMnO_4$]

**Preparation of 2-[2-(trimethylsilyl)ethyl]-acetic Acid 1-Ethyl-1-methylpropyl Ester (3o) (Scheme 5)**




Following General Procedure 3, DCC (959 mg, 4.648 mmol, 1.1 equiv) was combined with 3-methyl-3-pentanol (576 μL, 4.648 mmol, 1.1 equiv), 2-[2-(trimethylsilyl)ethyl]acetic acid

(745 mg, 4.226 mmol), and DMAP (103 mg, 0.845 mmol, 0.2 equiv) to yield, after filtration through silica gel (5 g), **3o** (897 mg, 81%) as a colorless oil.

Data for **3o**:

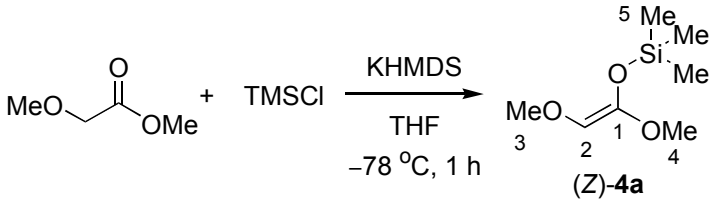
$^1$H NMR: (400 MHz, $CDCl_3$)

3.96 (s, 2 H, HC(2)), 3.61-3.57 (m, 2 H, HC(3)), 1.90 (dq, *J* = 14.0, 7.5, 2 H, HC(7)), 1.77 (dq, *J* = 14.0, 7.5, 2 H, HC(7)), 1.40 (s, 3 H, HC(9)), 1.02-0.98 (m, 2 H, HC(4)), 0.85 (dd, *J* = 7.5, 7.5, 6 H, HC(8)), 0.02 (s, 9 H, HC(5))

**Preparations of Trimethylsilyl Ketene Acetals**

**General Procedure 4. Enolization of Glycolates to Trimethylsilyl Ketene Acetals.**

**Preparation of [[(*Z*)-1,2-Dimethoxyethenyl]oxy]trimethylsilane[15] ((*Z*)-4a) (Scheme 6)**




To a flame-dried, 250-mL, 3-necked round-bottomed flask fitted with a magnetic stir bar, a thermocouple, a gas inlet tube, and a septum were added KHMDS (3.292 g, 16.5 mmol, 1.1 equiv) and THF (90 mL). The solution was cooled to –78 °C (internal temp.) in a dry ice-acetone bath and then 2-methoxyacetic acid methyl ester (1.486 mL, 15 mmol) was added dropwise via syringe over 5 min. The internal temperature was kept below –70 °C during addition. After 25 min, TMSCl (2.094 mL, 16.5 mmol, 1.1 equiv) was added dropwise via syringe over 5 min while vigorous strring was maintained. After 1 h, the dry ice-acetone bath was removed and pentane (60 mL) was added. The reaction mixture was filtered through a glass frit at rt and was concentrated *in vacuo* (23 °C, 30 mmHg). The residue was distilled through a 5-cm Vigreux

column under reduced pressure to give **4a** (1.092 g, 41%, 96/4, *Z*/*E*) as a colorless oil.

Data for (*Z*)-**4a**:

bp: 58-59 °C (20 mmHg)

$^1$H NMR: (500 MHz, $CDCl_3$)

5.30 (s, 1 H, HC(2)), 3.469 (s, 3 H, HC(4)), 3.467 (s, 3 H, HC(3)), 0.23 (s, 9 H, HC(5))

NOESY1D: (500 MHz, $CDCl_3$)

Irradiation at 5.30 ppm (HC(2)) enhanced signal at 3.469 ppm (HC(4)) in major isomer.

$^{13}$C NMR: (126 MHz, $CDCl_3$)

150.0 (C(1)), 112.4 (C(2)), 60.2 (C(3)), 55.8 (C(4)), 0.2 (C(5))

IR: (neat)

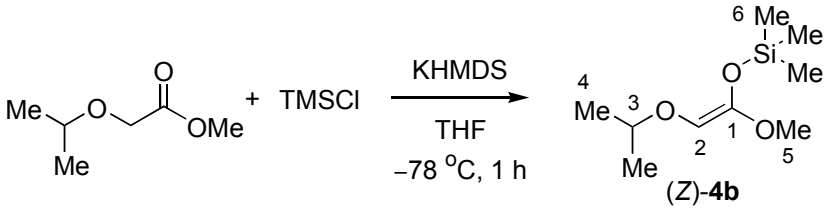
2959 (m), 2903 (m), 2830 (m), 1742 (m), 1708 (m), 1631 (w), 1463 (m), 1444 (m), 1364 (m), 1326 (s), 1252 (s), 1217 (s), 1175 (s), 1132 (s), 1088 (m), 1057 (m), 998 (m), 959 (m), 921 (m), 847 (s), 759 (m)

MS: (CI, $CH_4$)

75.0 (41), 103.1 (35), 161.1 (81), 177.1 (67, $M^+$+H), 217.1 (100)

HRMS: calcd for $C_7H_{17}O_3Si$: 177.0947, found 177.0942

**Preparation of [[(*Z*)-1-Methoxy-2-(1-methylethoxy)ethenyl]oxy]trimethylsilane ((*Z*)-4b) (Scheme 6)**




Following General Procedure 4, KHMDS (1.963 g, 9.838 mmol, 1.1 equiv) was combined with 2-(1-methylethoxy)acetic acid methyl ester (1.182 g, 8.944 mmol) and TMSCl (1.249 mL, 9.838 mmol, 1.1 equiv) to yield, after distillation through a 5-cm Vigreux column under reduced pressure, **4b** (1.312 g, 72%, 99/1, *Z*/*E*) as a colorless oil.

Data for (*Z*)-**4b**:

bp: 48-49 °C (5 mmHg)

$^1$H NMR: (500 MHz, $CDCl_3$)

5.31 (s, 1 H, HC(2)), 3.71 (hept., *J* = 6.1, 1 H, HC(3)), 3.47 (s, 3 H, HC(5)), 1.19 (d, *J* = 6.1, 6 H, HC(4)), 0.23 (s, 9 H, HC(6))

NOESY1D: (500 MHz, $CDCl_3$)

Irradiation at ppm 5.31 (HC(2)) enhanced signal at 3.47 ppm (HC(5)) in major isomer.

$^{13}$C NMR: (126 MHz, $CDCl_3$)

149.9 (C(1)), 109.4 (C(2)), 73.9 (C(3)), 56.1 (C(5)), 22.1 (C(4)), 0.3 (C(6))

IR: (neat)

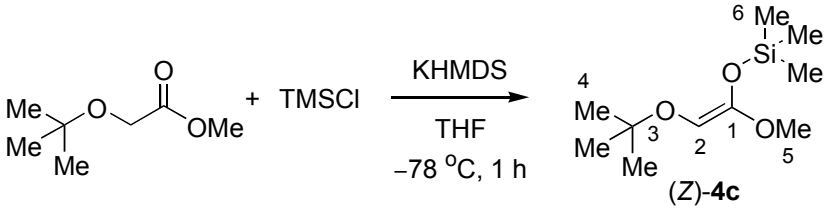
3082 (w), 2974 (s), 2938 (m), 2903 (m), 2877 (m), 2837 (m), 1758 (m), 1743 (m), 1708 (m), 1466 (m), 1443 (m), 1372 (s), 1333 (m), 1317 (s), 1252 (s), 1232 (s), 1200 (s), 1174 (s), 1148 (s), 1133 (s), 1050 (s), 973 (s), 850 (s), 758 (s)

MS: (ESI)

155.1 (14), 163.0 (25), 211.1 (14), 227.1 ($M^+$+Na, 12), 243.1 (29), 255.1 (36), 303.1 (17), 317.1 (19), 325.2 (13), 341.2 (30)

HRMS: calcd for $C_9H_{20}O_3SiNa$: 227.1079, found 227.1075

**Preparation of [[(*Z*)-2-(1,1-Dimethylethoxy)-1-methoxyethenyl]oxy]trimethylsilane[16] ((*Z*)-4c) (Scheme 6)**




Following General Procedure 4, KHMDS (2.024 g, 10.144 mmol, 1.1 equiv) was combined with 2-(1,1-dimethylethoxy)acetic acid methyl ester (1.348 g, 9.222 mmol) and TMSCl (1.287 mL, 10.144 mmol, 1.1 equiv) to yield, after distillation through a 5-cm Vigreux column under reduced pressure, **4c** (1.387 g, 69%, 99/1, *Z*/*E*) as a colorless oil.

Data for (*Z*)-**4c**:

bp: 31-32 °C (0.5 mmHg)

$^1$H NMR: (500 MHz, $CDCl_3$)

5.34 (s, 1 H, HC(2)), 3.49 (s, 3 H, HC(5)), 1.22 (s, 9 H, HC(4)), 0.23 (s, 9 H, HC(6))

NOESY1D: (500 MHz, $CDCl_3$)

Irradiation at 5.34 ppm (HC(2)) enhanced signal at 3.49 ppm (HC(5)) in major isomer.

$^{13}$C NMR: (126 MHz, $CDCl_3$)

150.9 (C(1)), 104.5 (C(2)), 76.0 (C(3)), 56.1 (C(5)), 27.6 (C(4)), 0.3 (C(6))

IR: (neat)

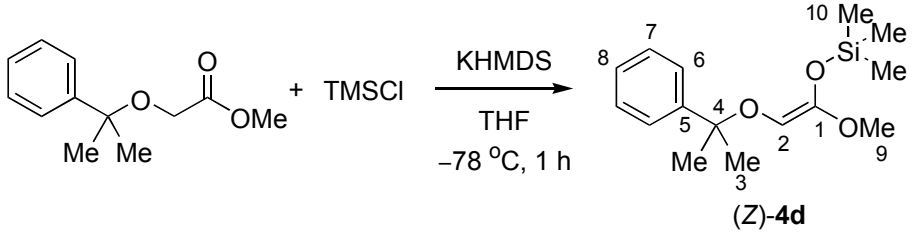
2977 (s), 2937 (s), 1906 (m), 1837 (m), 1768 (m), 1743 (m), 1699 (s), 1687 (s), 1466 (m), 1443 (s), 1389 (s), 1367 (s), 1330 (s), 1252 (s), 1232 (s), 1189 (s), 1171 (s), 1141 (s), 1089 (m), 1053 (s), 965 (s), 921 (m), 849 (s), 759 (s)

MS: (ESI)

169.0 (100), 169.2 (91), 169.3 (35), 170.1 (27), 211.1 (42), 212.1 (4), 241.1 ($M^+$+Na, 7), 257.1 (48), 258.1 (10), 301.1 (13), 313.1 (5), 387.2 (19), 388.2 (9)

HRMS: calcd for $C_{10}H_{22}O_3SiNa$: 241.1236, found 241.1235

**Preparation of [[(*Z*)-1-Methoxy-2-(1-methyl-1-phenylethoxy)ethenyl]oxy]trimethylsilane ((*Z*)-4d) (Scheme 6)**




(*Z*)-**4d**

Following General Procedure 4, KHMDS (2.923 g, 14.653 mmol, 1.1 equiv) was combined with 2-(1-methyl-1-phenylethoxy)acetic acid methyl ester (2.774 g, 13.321 mmol) and TMSCl (1.860 mL, 14.653 mmol, 1.1 equiv) to yield, after distillation through a 8-cm Vigreux column under reduced pressure, **4d** (3.059 g, 82%, >99/1, *Z*/*E*) as a colorless oil.

Data for (*Z*)-**4d**:

bp: 71-72 °C (0.4 mmHg)

$^{1}$H NMR: (500 MHz, $CDCl_3$)

7.44-7.42 (m, 2 H, HC(6)), 7.35-7.32 (m, 2 H, HC(7)), 7.26-7.23 (m, 1 H, HC(8)), 5.07 (s, 1 H, HC(2)), 3.38 (s, 3 H, HC(9)), 1.58 (s, 6 H, HC(3)), 0.28 (s, 9 H, HC(10))

NOESY1D: (500 MHz, $CDCl_3$)

Irradiation at 5.07 ppm (HC(2)) enhanced signal at 3.38 ppm (HC(9)) in major isomer.

$^{13}$C NMR: (126 MHz, $CDCl_3$)

150.4 (C(1)), 146.1 (C(5)), 128.1 (C(7)), 126.8 (C(8)), 125.7 (C(6)), 105.4 (C(2)), 79.1 (C(4)), 56.0 (C(9)), 28.3 (C(3)), 0.4 (C(10))

IR: (neat)

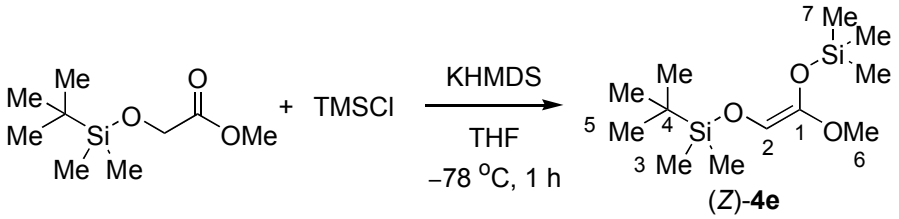
3089 (w), 3062 (w), 2980 (m), 2904 (w), 2837 (w), 1764 (w), 1742 (m), 1697 (m), 1496 (m), 1447 (m), 1380 (m), 1364 (m), 1328 (m), 1251 (s), 1233 (s), 1205 (s), 1170 (m), 1134 (s), 1102 (m), 1075 (m), 1047 (m), 971 (s), 848 (s), 764 (s), 700 (s)

MS: (ESI)

231.1 (100), 232.1 (6), 303.1 ($M^+$+Na, 4)

HRMS: calcd for $C_{15}H_{24}O_3SiNa$: 303.1392, found 303.1393

**Preparation of (4*Z*)-4-Methoxy-2,2,7,7,8,8-hexamethyl-3,6-dioxa-2,7-disilanon-4-ene[17] ((*Z*)-4e) (Scheme 6)**




Following General Procedure 4, KHMDS (2.613 g, 13.097 mmol, 1.1 equiv) was combined with [[(1,1-dimethylethyl)dimethylsilyl]oxy]acetic acid methyl ester (2.433 g, 11.907 mmol) and TMSCl (1.662 mL, 13.097 mmol, 1.1 equiv) to yield, after distillation through a 5-cm Vigreux column under reduced pressure, **4e** (2.847 g, 86%, 99/1, *Z*/*E*) as a colorless oil.

Data for (*Z*)-**4e**:

bp: 51-52 °C (0.4 mmHg)

$^1$H NMR: (500 MHz, $CDCl_3$)

5.51 (s, 1 H, HC(2)), 3.47 (s, 3 H, HC(6)), 0.92 (s, 9 H, HC(5)), 0.22 (s, 9 H, HC(7)), 0.10 (s, 6 H, HC(3))

NOESY1D: (500 MHz, $CDCl_3$)

Irradiation at 5.51 ppm (HC(2)) enhanced signal at 3.47 ppm (HC(6)) in major isomer.

$^{13}$C NMR: (126 MHz, $CDCl_3$)

149.9 (C(1)), 106.2 (C(2)), 56.4 (C(6)), 25.8 (C(5)), 18.4 (C(4)), 0.3 (C(7)), −5.3 (C(3))

IR: (neat)

2959 (s), 2932 (s), 2899 (s), 2859 (s), 1765 (m), 1745 (m), 1701 (s), 1473 (s), 1464 (s), 1443 (s), 1407 (s), 1390 (m), 1370 (s), 1332 (m), 1253 (s), 1232 (s),

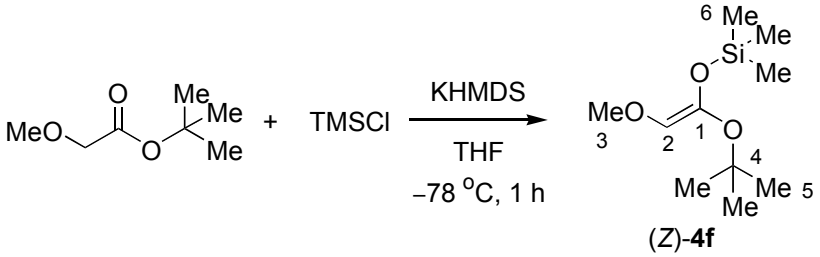
1158 (s), 1088 (m), 1043 (m), 1006 (m), 963 (s), 843 (s), 781 (s), 758 (s)

MS: (ESI)

227.1 (100), 228.1 (12), 299.1 ($M^+$+Na, 2), 315.1 (24), 316.1 (3), 459.2 (2), 503.2 (4), 504.2 (2)

HRMS: calcd for $C_{12}H_{28}O_3Si_2Na$: 299.1475, found 299.1469

**Preparation of [[(*Z*)-1-(1,1-Dimethylethoxy)-2-methoxyethenyl]oxy]trimethylsilane[18] ((*Z*)-4f) (Scheme 6)**




Following General Procedure 4, KHMDS (2.194 g, 11 mmol, 1.1 equiv) was combined with 2-methoxyacetic acid 1-ethyl-1-methylpropyl ester (1.529 mL, 10 mmol) and TMSCl (1.396 mL, 11 mmol, 1.1 equiv) to yield, after distillation through a 5-cm Vigreux column under reduced pressure, **4f** (1.652 g, 76%, >99/1, *Z*/*E*) as a colorless oil.

Data for (*Z*)-**4f**:

bp: 50-52 °C (5 mmHg)

$^1$H NMR: (500 MHz, $CDCl_3$)

5.38 (s, 1 H, HC(2)), 3.47 (s, 3 H, HC(3)), 1.26 (s, 9 H, HC(5)), 0.22 (s, 9 H, HC(6))

NOESY1D: (500 MHz, $CDCl_3$)

Irradiation at 5.38 ppm (HC(2)) enhanced signal at 1.26 ppm (HC(5)) in major isomer.

$^{13}$C NMR: (126 MHz, $CDCl_3$)

143.2 (C(1)), 121.3 (C(2)), 78.5 (C(4)), 59.6 (C(3)), 28.5 (C(5)), 0.4 (C(6))

IR: (neat)

3062 (w), 2978 (s), 2934 (m), 2904 (m), 2828 (m), 1754 (w), 1736 (w), 1699 (m), 1476 (m), 1459 (m), 1390 (m), 1367 (m), 1322 (s), 1252 (s), 1220 (s), 1155 (s), 1130 (s), 1041 (m), 1020 (m), 1000 (s), 921 (s), 848 (s), 763 (m), 688 (m)

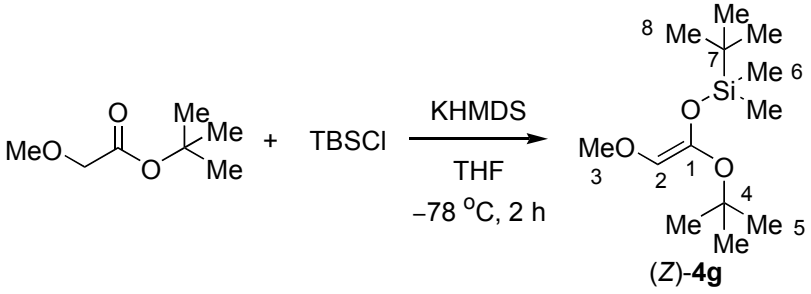
MS: (ESI)

113.0 (5), 169.1 (100), 201.1 (5), 241.1 ($M^+$+Na, 54), 257.1 (25)

HRMS: calcd for $C_{10}H_{22}O_3SiNa$: 241.1236, found 241.1248

**Preparations of *tert*-Butyldimethylsilyl Ketene Acetals**

**General Procedure 5. Enolization of Glycolates to *tert*-Butyldimethylsilyl Ketene Acetals. Preparation of [[(*Z*)-1-(1,1-Dimethylethoxy)-2-methoxyethenyl]oxy](1,1-dimethylethyl) dimethylsilane ((*Z*)-4g) (Scheme 7)**




(*Z*)-**4g**

To a flame-dried, 250-mL, 3-necked round-bottomed flask fitted with a magnetic stir bar, a thermocouple, a gas inlet tube, and a septum were added KHMDS (3.072 g, 15.4 mmol, 1.1 equiv) and THF (90 mL). The solution was cooled to −78 °C (internal temp.) in a dry ice-acetone bath and then 2-methoxyacetic acid 1-ethyl-1-methylpropyl ester (2.141 mL, 14 mmol) was added dropwise via syringe over 15 min. The internal temperature was kept below −70 °C during

the addition of the ester. After 15 min, a solution of TBSCl (2.321 g, 15.4 mmol, 1.1 equiv) in THF (20 mL) was added dropwise via syringe over 15 min. After 2 h, the dry ice-acetone bath was removed and pentane (80 mL) was added. The reaction mixture was filtered through a glass frit at rt and concentrated *in vacuo* (23 $^{o}$C, 30 mmHg). The residue was distilled through a 5-cm Vigreux column under reduced pressure to give **4g** (2.829 g, 78%, 98/2, *Z*/*E*) as a colorless oil.

Data for (*Z*)-**4g**:

bp: 55-58 $^{o}$C (1 mmHg)

$^{1}$H NMR: (500 MHz, $CDCl_3$)

5.36 (s, 1 H, HC(2)), 3.47 (s, 3 H, HC(3)), 1.27 (s, 9 H, HC(5)), 0.95 (s, 9 H, HC(8)), 0.17 (s, 6 H, HC(6))

NOESY1D: (500 MHz, $CDCl_3$)

Irradiation at 5.36 ppm (HC(2)) enhanced signal at 1.27 ppm (HC(5)) in major isomer.

$^{13}$C NMR: (126 MHz, $CDCl_3$)

143.0 (C(1)), 121.9 (C(2)), 78.6 (C(4)), 59.5 (C(3)), 28.5 (C(5)), 25.6 (C(8)), 18.2 (C(7)), –4.6 (C(6))

IR: (neat)

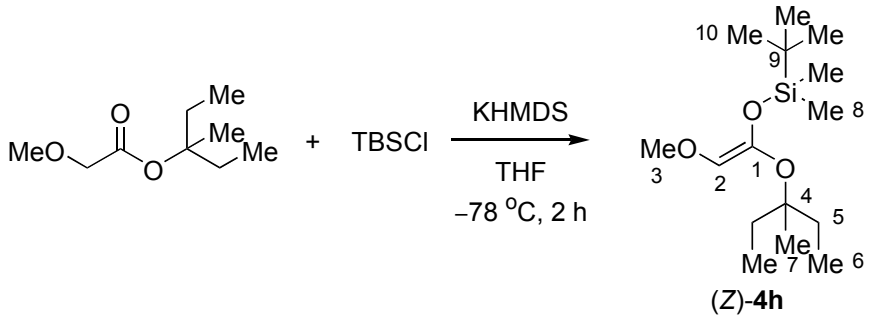
3061 (w), 2978 (s), 2959 (s), 2932 (s), 2900 (m), 2859 (s), 2828 (m), 1698 (m), 1474 (m), 1464 (m), 1390 (m), 1366 (s), 1321 (s), 1254 (s), 1222 (s), 1153 (s), 1130 (s), 1041 (m), 1021 (m), 940 (m), 855 (s), 840 (s), 828 (s), 813 (s), 785 (s)

MS: (ESI)

211.1 (15), 227.1 (90), 228.1 (16), 243.1 (19), 267.2 (57), 268.2 (12), 283.2 ($M^{+}$+Na, 100), 284.2 (23), 285.2 (14), 299.2 (76), 300.2 (14), 315.2 (35)

HRMS: calcd for $C_{13}H_{28}O_3SiNa$: 283.1705, found 283.1703

**Preparation of (1,1-Dimethylethyl)[[(*Z*)-1-(1-ethyl-1-methylpropoxy)-2-methoxyethenyl] oxy]dimethylsilane ((*Z*)-4h) (Scheme 7)**




(*Z*)-**4h**

Following General Procedure 5, KHMDS (3.395 g, 17.020 mmol, 1.1 equiv) was combined with 2-methoxyacetic acid 1-ethyl-1-methylpropyl ester (2.696 g, 15.473 mmol) and TBSCl (2.565 g, 17.020 mmol, 1.1 equiv) to yield, after distillation through a 8-cm Vigreux column under reduced pressure, **4h** (3.609 g, 81%, 96/4, *Z*/*E*) as a colorless oil.

Data for (*Z*)-**4h**:

bp: 59-60 °C (0.4 mmHg)

$^1$H NMR: (500 MHz, $CDCl_3$)

5.32 (s, 1 H, HC(2)), 3.46 (s, 3 H, HC(3)), 1.64-1.49 (m, 4 H, HC(5)), 1.17 (s, 3 H, HC(7)), 0.94 (s, 9 H, HC(10)), 0.86 (dd, *J* = 7.6, 7.6, 6 H, HC(6)), 0.16 (s, 6 H, HC(8))

NOESY1D: (500 MHz, $CDCl_3$)

Irradiation at 5.32 ppm (HC(2)) enhanced signal at 1.64-1.49 ppm (HC(5)), 1.17 ppm (HC(7)), and 0.86 ppm (HC(6)) in major isomer.

$^{13}$C NMR: (126 MHz, $CDCl_3$)

143.0 (C(1)), 121.6 (C(2)), 83.5 (C(4)), 59.5 (C(3)), 30.7 (C(5)), 25.7 (C(10)), 23.0 (C(7)), 18.2 (C(9)), 8.4 (C(6)), –4.5 (C(8))

IR: (neat)

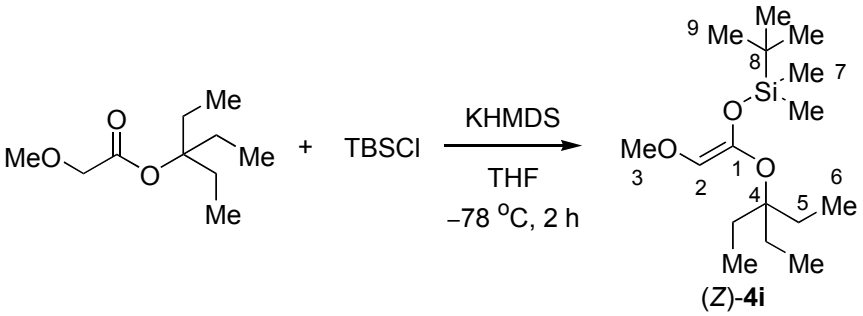
3062 (w), 2967 (s), 2932 (s), 2885 (m), 2859 (m), 2827 (m), 1746 (w), 1697 (m), 1463 (m), 1375 (m), 1362 (m), 1320 (s), 1251 (m), 1213 (s), 1130 (s), 1026 (m), 1000 (m), 971 (m), 854 (s), 840 (s), 828 (s), 813 (m), 784 (s), 735 (m), 694 (m)

MS: (ESI)

113.0 (9), 197.1 (100), 198.1 (5), 227.1 (5), 243.1 (7), 311.2 ($M^+$+Na, 1), 327.2 (14), 337.1 (4)

HRMS: calcd for $C_{15}H_{32}O_3SiNa$: 311.2018, found 311.2021

**Preparation of [[(*Z*)-1-(1,1-Diethylpropoxy)-2-methoxyethenyl]oxy](1,1-dimethylethyl) dimethylsilane ((*Z*)-4i) (Scheme 7)**




(*Z*)-**4i**

Following General Procedure 5, KHMDS (3.149 g, 15.787 mmol, 1.1 equiv) was combined with 2-methoxyacetic acid 1,1-diethylpropyl ester (2.701 g, 14.352 mmol) and TBSCl (2.380 g, 15.787 mmol, 1.1 equiv) to yield, after short path distillation under reduced pressure, **4i** (3.405 g, 78%, 91/9, *Z*/*E*) as a colorless oil.

Data for (*Z*)-**4i**:

bp: 78-79 °C (0.2 mmHg)

$^1$H NMR: (500 MHz, $CDCl_3$)

5.30 (s, 1 H, HC(2)), 3.45 (s, 3 H, HC(3)), 1.57 (q, *J* = 7.5, 6 H, HC(5)), 0.93 (s,

9 H, HC(9)), 0.85 (t, $J$ = 7.5, 9 H, HC(6)), 0.16 (s, 6 H, HC(7))

NOESY1D: (500 MHz, $CDCl_3$)

Irradiation at 5.30 ppm (HC(2)) enhanced signal at 1.57 ppm (HC(5)) and 0.85 ppm (HC(6)) in major isomer.

$^{13}$C NMR: (126 MHz, $CDCl_3$)

143.0 (C(1)), 121.2 (C(2)), 85.9 (C(4)), 59.4 (C(3)), 27.6 (C(5)), 25.7 (C(9)), 18.3 (C(8)), 8.1 (C(6)), –4.4 (C(7))

IR: (neat)

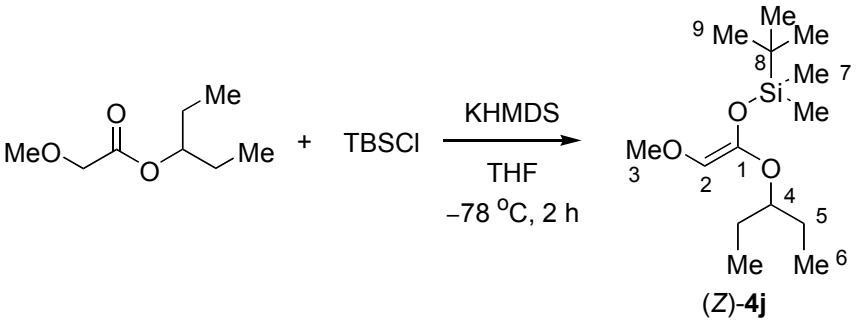
3063 (w), 2967 (s), 2932 (s), 2885 (m), 2859 (m), 2827 (m), 1749 (w), 1721 (w), 1695 (m), 1463 (m), 1390 (m), 1381 (m), 1362 (m), 1320 (s), 1253 (s), 1213 (s), 1130 (s), 1027 (m), 999 (m), 938 (m), 884 (m), 840 (s), 828 (s), 814 (m), 784 (s)

MS: (CI, $CH_4$)

143.2 (27), 205.1 (100), 303.3 (56, $M^+$+H), 319.2 (35)

HRMS: calcd for $C_{16}H_{35}O_3Si$: 303.2356, found 303.2349

**Preparation of (1,1-Dimethylethyl)[[(*Z*)-1-(1-ethylpropoxy)-2-methoxyethenyl]oxy] dimethylsilane ((*Z*)-4j) (Scheme 7)**




(*Z*)-**4j**

Following General Procedure 5, KHMDS (2.112 g, 10.587 mmol, 1.1 equiv) was combined with 2-methoxyacetic acid 1-ethylpropyl ester (1.542 g, 9.625 mmol) and TBSCl

(1.596 g, 10.587 mmol, 1.1 equiv) to yield, after short path distillation under reduced pressure, **4j** (2.182 g, 83%, 99/1, *Z*/*E*) as a colorless oil.

Data for (*Z*)-**4j**:

bp: 54-55 °C (0.3 mmHg)

$^1$H NMR: (500 MHz, $CDCl_3$)

5.35 (s, 1 H, HC(2)), 3.76 (tt, *J* = 5.8, 5.8, 1 H, HC(4)), 3.44 (s, 3 H, HC(3)), 1.57-1.52 (m, 4 H, HC(5)), 0.95 (s, 9 H, HC(9)), 0.88 (dd, *J* = 7.5, 7.5, 6 H, HC(6)), 0.18 (s, 6 H, HC(7))

NOESY1D: (500 MHz, $CDCl_3$)

Irradiation at 5.35 ppm (HC(2)) enhanced signal at 3.76 ppm (HC(4)), 1.57-1.52 ppm (HC(5)), 0.88 ppm (HC(6)) in major isomer.

$^{13}$C NMR: (126 MHz, $CDCl_3$)

145.8 (C(1)), 117.0 (C(2)), 79.4 (C(4)), 59.8 (C(3)), 25.7 (C(9)), 24.9 (C(5)), 18.3 (C(8)), 9.2 (C(6)), –4.6 (C(7))

IR: (neat)

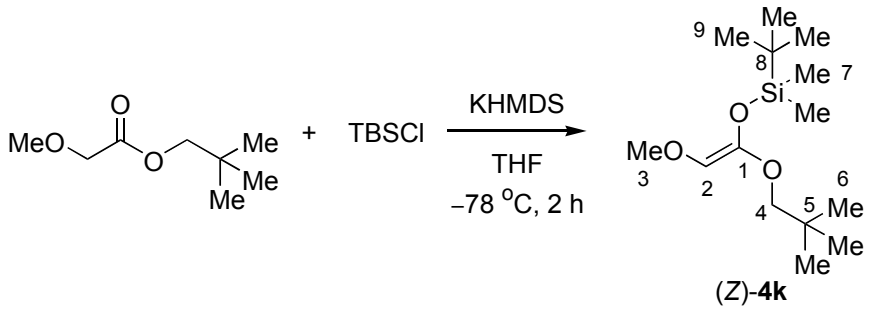
3062 (w), 2962 (s), 2932 (s), 2898 (s), 2884 (s), 2859 (s), 2827 (m), 1704 (m), 1472 (m), 1464 (s), 1389 (m), 1362 (m), 1321 (s), 1252 (s), 1209 (s), 1130 (s), 1105 (m), 1045 (m), 1027 (m), 999 (s), 921 (m), 840 (s), 828 (s), 814 (s), 785 (s)

MS: (ESI)

189.1 (12) 227.1 (15), 281.2 (57), 282.2 (12), 297.2 ($M^+$+Na, 94), 298.2 (21), 299.2 (10), 313.2 (100), 314.2 (23), 329.2 (9)

HRMS: calcd for $C_{14}H_{30}O_3SiNa$: 297.1862, found 297.1857

**Preparation of (1,1-Dimethylethyl)[[(*Z*)-1-(2,2-dimethylpropoxy)-2-methoxyethenyl]oxy] dimethylsilane ((*Z*)-4k) (Scheme 7)**




(*Z*)-**4k**

Following General Procedure 5, KHMDS (1.961 g, 9.832 mmol, 1.1 equiv) was combined with 2-methoxyacetic acid 2,2-dimethylpropyl ester (1.432 g, 8.938 mmol) and TBSCl (1.482 g, 8.938 mmol, 1.1 equiv) to yield, after distillation through a 5-cm Vigreux column under reduced pressure, **4k** (2.043 g, 83%, 98/2, *Z*/*E*) as a colorless oil.

Data for (*Z*)-**4k**:

bp: 60-62 °C (0.4 mmHg)

$^1$H NMR: (500 MHz, $CDCl_3$)

5.26 (s, 1 H, HC(2)), 3.45 (s, 3 H, HC(3)), 3.25 (s, 2 H, HC(4)), 0.96 (s, 9 H, HC(9)), 0.93 (s, 9 H, HC (6)), 0.18 (s, 6 H, HC(7))

NOESY1D: (500 MHz, $CDCl_3$)

Irradiation at 5.26 ppm (HC(2)) enhanced signal at 3.25 ppm (HC(4)) in major isomer.

$^{13}$C NMR: (126 MHz, $CDCl_3$)

150.2 (C(1)), 112.4 (C(2)), 78.4 (C(4)), 60.3 (C(3)), 31.6 (C(5)), 26.6 (C(6)), 25.6 (C(9)), 18.2 (C(8)), –4.6 (C(7))

IR: (neat)

2957 (s), 2932 (s), 2903 (s), 2860 (s), 2827 (m), 1695 (m), 1474 (m), 1464 (m),

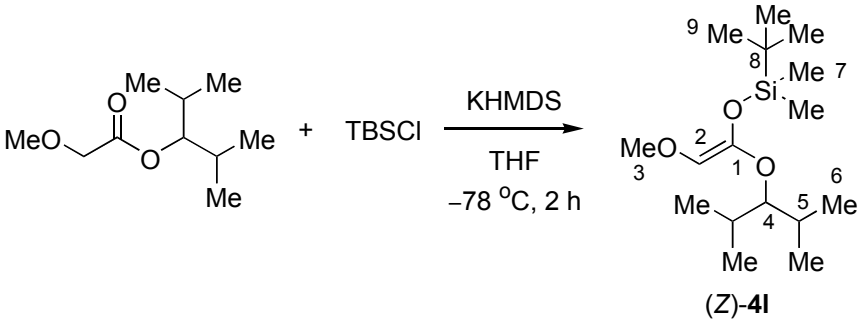
1402 (m), 1375 (s), 1364 (s), 1323 (s), 1251 (s), 1206 (s), 1137 (s), 1086 (m), 1052 (m), 1037 (m), 1000 (s), 978 (s), 840 (s), 828 (s), 813 (s), 786 (s)

MS: (ESI)

102.1 (7), 183.1 (6), 212.1 (6), 227.1 (15), 297.2 ($M^+$+Na, 16), 313.2 (100), 314.2 (23), 329.2 (20), 369.2 (5), 385.3 (6)

HRMS: calcd for $C_{14}H_{30}O_3SiNa$: 297.1862, found 297.1860

**Preparation of (1,1-Dimethylethyl)[[(*Z*)-2-methoxy-1-[2-methyl-1-(1-methylethyl)propoxy] ethenyl]oxy]dimethylsilane ((*Z*)-4l) (Scheme 7)**




(*Z*)-**4l**

Following General Procedure 5, KHMDS (2.784 g, 13.954 mmol, 1.1 equiv) was combined with 2-methoxyacetic acid 2-methyl-1-(1-methylethyl)propyl ester (2.388 g, 12.685 mmol) and TBSCl (2.103 g, 13.954 mmol, 1.1 equiv) to yield, after short path distillation under reduced pressure, **4l** (3.284 g, 86%, 86/14, *Z*/*E*) as a colorless oil.

Data for (*Z*)-**4l**:

bp: 66-69 °C (0.2 mmHg)

$^1$H NMR: (500 MHz, $CDCl_3$)

5.34 (s, 1 H, HC(2)), 3.45 (t, *J* = 5.5, 1 H, HC(4)), 3.42 (s, 3 H, HC(3)), 1.88 (qqd, *J* = 6.9, 6.9, 5.5, 2 H, HC(5)), 0.96 (s, 9 H, HC(9)), 0.93 (d, *J* = 6.9, 6 H, HC(6)), 0.92 (d, *J* = 6.9, 6 H, HC(6)), 0.19 (s, 6 H, HC(7))

NOESY1D: (500 MHz, $CDCl_3$)

Irradiation at 5.34 ppm (HC(2)) enhanced signal at 3.45 ppm (HC(4)), 0.93 ppm (HC(6)), and 0.92 ppm (HC(6)) in major isomer.

$^{13}$C NMR: (126 MHz, $CDCl_3$)

149.6 (C(1)), 114.0 (C(2)), 87.0 (C(4)), 60.0 (C(3)), 30.1 (C(5)), 25.8 (C(9)), 19.8 (C(6)), 18.0 (C(8)), –4.4 (C(7))

IR: (neat)

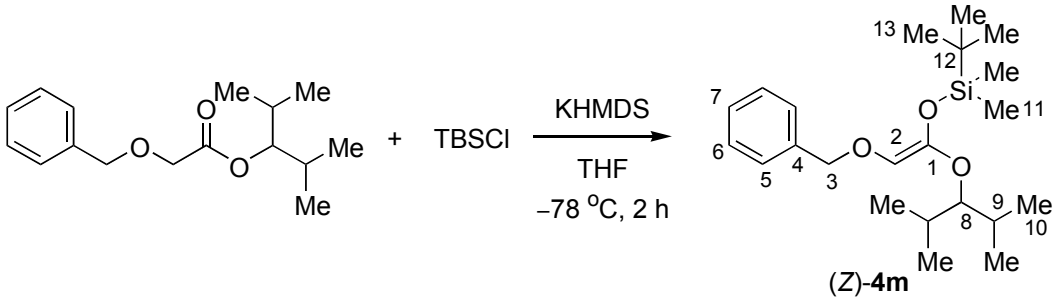
3064 (m), 2960 (s), 2932 (s), 2901 (s), 2860 (s), 2826 (m), 1702 (m), 1472 (s), 1464 (m), 1388 (m), 1363 (m), 1348 (m), 1324 (s), 1251 (s), 1210 (s), 1130 (s), 1110 (m), 1046 (m), 999 (s), 941 (s), 908 (m), 840 (s), 828 (s), 813 (s), 785 (s)

MS: (ESI)

211.1 (67), 227.1 (12), 309.2 (45), 310.2 (10), 325.2 ($M^+$+Na, 78), 326.2 (18), 341.2 (100), 357.2 (23), 383.3 (22), 399.3 (12)

HRMS: calcd for $C_{16}H_{34}O_3SiNa$: 325.2175, found 325.2191

**Preparation of (1,1-Dimethylethyl)[[(*Z*)-1-[2-methyl-1-(1-methylethyl)propoxy]-2-(phenyl methoxy)ethenyl]oxy]dimethylsilane ((*Z*)-4m) (Scheme 7)**




Following General Procedure 5, KHMDS (2.194 g, 11 mmol, 1.1 equiv) was combined with 2-(phenylmethoxy)acetic acid 2-methyl-1-(1-methylethyl)propyl ester (2.644 g, 10 mmol)

and TBSCl (1.658 g, 11 mmol, 1.1 equiv) to yield, after bulb-to-bulb distillation under reduced pressure, **4m** (3.623 g, 96%, 89/11, *Z*/*E*) as a colorless oil.

Data for (*Z*)-**4m**:

bp: 200-210 °C (0.2 mmHg, ABT)

$^1$H NMR: (500 MHz, $CDCl_3$)

7.39-7.32 (m, 4 H, HC(5), HC(6)), 7.30-7.26 (m, 1 H, HC(7)), 5.42 (s, 1 H, HC(2)), 4.61 (s, 2 H, HC(3)), 3.40 (t, *J* = 5.4, 1 H, HC(8)), 1.86 (qqd, *J* = 6.8, 6.8, 5.4, 2 H, HC(9)), 0.96 (s, 9 H, HC(13)), 0.92 (d, *J* = 6.8, 6 H, HC(10)), 0.90 (d, *J* = 6.8, 6 H, HC(10))

NOESY1D: (500 MHz, $CDCl_3$)

Irradiation at 5.42 ppm (HC(2)) enhanced signal at 3.40 ppm (HC(8)), 1.86 ppm (HC(9)), 0.92 ppm (HC(10)), and 0.90 ppm (HC(10)) in major isomer.

$^{13}$C NMR: (126 MHz, $CDCl_3$)

150.5 (C(1)), 138.1 (C(4)), 128.2 (C(6)), 127.9 (C(5)), 127.5 (C(7)), 111.7 (C(2)), 87.3 (C(8)), 74.5 (C(3)), 30.1 (C(9)), 25.8 (C(13)), 19.8 (C(10)), 18.3 (C(12)), 18.0 (C(10)), –4.3 (C(11))

IR: (neat)

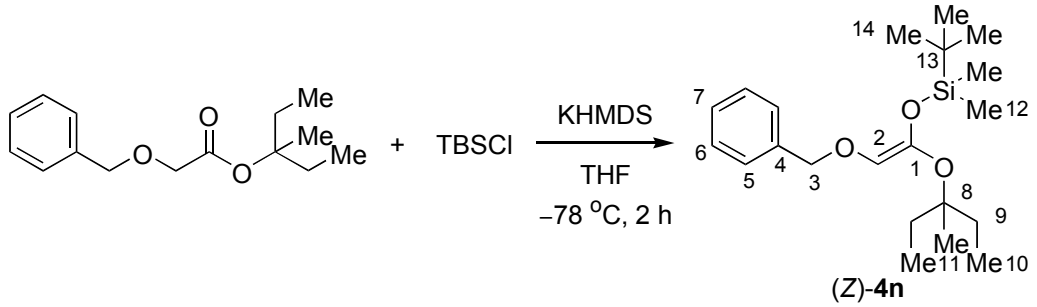
3066 (w), 3032 (w), 2960 (s), 2931 (s), 2858 (m), 1751 (w), 1700 (m), 1472 (m), 1386 (m), 1370 (m), 1324 I(m), 1252 (s), 1217 (s), 1150 (s), 1129 (s), 1046 (m), 1004 (m), 941 (m), 908 (m), 839 (s), 812 (m), 785 (s), 734 (m), 697 (m)

MS: (ESI)

167.1 (5), 189.0 (52), 190.1 (7), 287.1 (100), 287.6 (92), 288.2 (56), 288.4 (20), 401.3 ($M^+$ + Na, 22), 417.2 (42), 418.3 (15)

HRMS: calcd for $C_{22}H_{38}O_3SiNa$: 401.2488, found 401.2487

**Preparation of (1,1-Dimethylethyl)[[(*Z*)-1-(1-ethyl-1-methylpropoxy)-2-(phenylmethoxy) ethenyl]oxy]dimethylsilane ((*Z*)-4n) (Scheme 7)**




(*Z*)-**4n**

Following General Procedure 5, KHMDS (682 mg, 3.419 mmol, 1.1 equiv) was combined with 2-(phenylmethoxy)acetic acid 1-ethyl-1-methylpropyl ester (778 mg, 3.108 mmol) and TBSCl (515 mg, 3.419 mmol, 1.1 equiv) to yield, after bulb-to-bulb distillation under reduced pressure, **4n** (870 mg, 77%,97/3, *Z*/*E*) as a colorless oil. The product contained 6% of 2-(phenylmethoxy)acetic acid (1,1-dimethylethyl)dimethylsilyl ester as by-product.

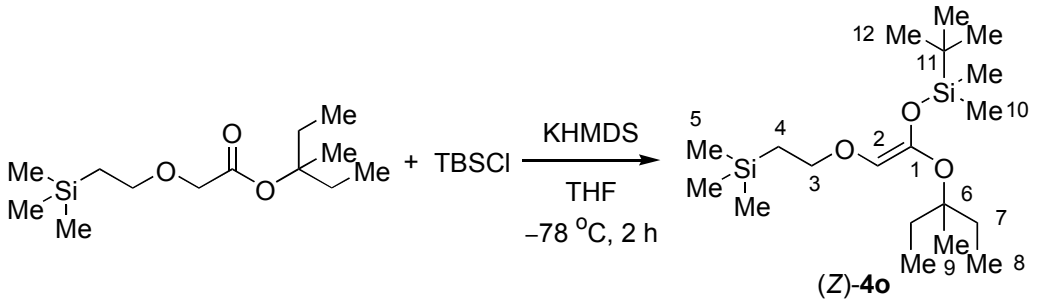
Data for (*Z*)-**4n**:

bp: 170-200 °C (0.04 mmHg, ABT)

$^1$H NMR: (400 MHz, $CDCl_3$)

7.37-7.33 (m, 4 H, HC(5), HC(6)), 7.32-7.27 (m, 1 H, HC(7)), 5.42 (s, 1 H, HC(2)), 4.66 (s, 2 H, HC(3)), 1.59-1.44 (m, 4 H, HC(9)), 1.12 (s, 3 H, HC(11)), 0.93 (s, 9 H, HC(14)), 0.83 (dd, *J* = 7.6, 7.6, 6 H, HC(10)), 0.16 (s, 6 H, HC(12))

**Preparation of (1,1-Dimethylethyl)[[(*Z*)-1-(1-ethyl-1-methylpropoxy)-2-[2-(trimethylsilyl) ethoxy]ethenyl]oxy]dimethylsilane ((*Z*)-4o) (Scheme 7)**




(*Z*)-**4o**

Following General Procedure 5, KHMDS (755 mg, 3.788 mmol, 1.1 equiv) was combined with 2-[2-(trimethylsilyl)ethoxy]acetic acid 1-ethyl-1-methylpropyl ester (897 mg, 3.444 mmol) and TBSCl (571 mg, 3.788 mmol, 1.1 equiv) to yield, after bulb-to-bulb distillation under reduced pressure, **4o** (914 mg, 71%, 98/2, *Z*/*E*) as a colorless oil. The product contained 13% of 2-(phenylmethoxy)acetic acid (1,1-dimethylethyl)dimethylsilyl ester as by-product.

Data for (*Z*)-**4o**:

bp: 200-220 °C (0.2 mmHg, ABT)

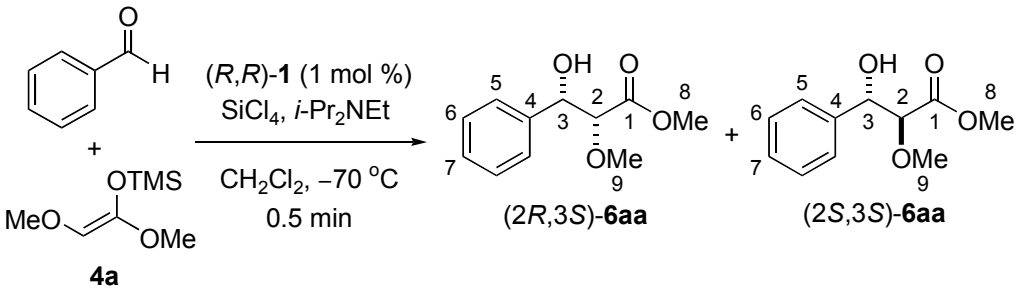
$^1$H NMR: (500 MHz, $CDCl_3$)

5.34 (s, 1 H, HC(2)), 3.65-3.62 (m, 2 H, HC(3)), 1.63-1.50 (m, 4 H, HC(7)), 1.17 (s, 3 H, HC(9)), 1.03-0.99 (m, 2 H, HC(4)), 0.94 (s, 9 H, HC(12)), 0.86 (dd, *J* = 7.5, 7.5, 6 H, HC(8)), 0.17 (s, 9 H, HC(12)), 0.02 (s, 9 H, HC(5))

**Aldol Additions of Silyl Ketene Acetals to Aromatic Aldehydes**

**General Procedure 6. *In situ*-Monitored Addition of Silyl Ketene Acetals to Benzaldehyde**

**Preparation of (2*R*,3*S*)-3-Hydroxy-2-methoxy-3-phenylpropanoic Acid Methyl Ester[19] ((2*R*,3*S*)-6aa) and (2*S*,3*S*)-3-Hydroxy-2-methoxy-3-phenylpropanoic Acid Methyl Ester ((2*S*,3*S*)-6aa) (Table 1, entry 1)**




To a flame-dried, 10-mL, ReactIR flask fitted with a magnetic stir bar, a thermocouple, a gas inlet tube, and a septum were added (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv), $CH_2Cl_2$ (5 mL), and diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv). The solution was cooled to –70 °C (internal temp.) in a dry ice-acetone bath. After the background scan was taken, benzaldehyde (101.6 μL, 1.0 mmol) and $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv) were added to the flask. The ReactIR was initialized. After 1 min, a solution of **4a** (212 mg, 1.2 mmol, 1.2 equiv) in $CH_2Cl_2$ (0.5 mL) was added. The carbonyl peak of the benzaldehyde (1701 $cm^{-1}$) was monitored. After the carbonyl peak disappeared, a mixture of MeOH (1 mL), $Et_3N$ (1 mL), and $CH_2Cl_2$ (5 mL) was added at -70 °C. The resulting solution was transferred into a 125-mL Erlenmeyer flask containing a sat. aq. $NaHCO_3$ solution (10 mL) and a sat. aq. KF solution (10 mL). The biphasic mixture was stirred vigorously for 2 h at room temperature. The mixture was filtered through a glass frit and the filtrate was transferred to a 125-mL separatory funnel where the organic layer was separated. The aqueous layer was extracted with $CH_2Cl_2$ (2 × 20 mL). The combined organic extracts were dried over $Na_2SO_4$ (15 g), filtered, and concentrated *in vacuo* (23 °C, 30

mmHg). The residue was purified by column chromatography (22 mm diam., hexane/EtOAc, 5/1 to 2/1) on silica gel (20 g) to give **6aa** (206 mg, 98%) as a colorless oil. The *syn*/*anti* ratio was determined to be 57/43 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*R*,3*S*)-**6aa**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.37-7.32 (m, 4 H, HC(5), HC(6)), 7.31-7.27 (m, 1 H, HC(7)), 4.90 (d, $J$ = 5.6, 1 H, HC(3)), 3.90 (d, $J$ = 5.6, 1 H, HC(2)), 3.63 (s, 3 H, HC(8)), 3.40 (s, 3 H, HC(9)), 2.95 (bs, 1 H, OH)

$^{13}$C NMR: (126 MHz, $CDCl_3$)

170.7 (C(1)), 139.0 (C(4)), 128.3 (C(6)), 128.1 (C(7)), 126.4 (C(5)), 85.2 (C(2)), 74.6 (C(3)), 58.8 (C(9)), 51.9 (C(8))

IR: (neat)

3478 (br), 3064 (w), 3032 (m), 3000 (m), 2952 (m), 2905 (m), 2833 (m), 1745 (s), 1496 (m), 1455 (s), 1437 (m), 1395 (m), 1354 (m), 1270 (s), 1202 (s), 1121 (s), 1086 (m), 1061 (s), 1025 (s), 916 (m), 847 (m), 833 (m), 771 (m), 753 (m), 702 (s)

MS: (ESI)

233.1 ($M^+$+Na, 100), 234.1 (5)

HRMS: calcd for $C_{11}H_{14}O_4Na$: 233.0790, found 233.0798

TLC: $R_f$ 0.28 (hexane/EtOAc, 2/1) [UV(254)/$KMnO_4$]

SFC: (2*R*,3*S*)-**6aa**, $t_R$ 3.46 min (73.6%); (2*S*,3*R*)-**6aa**, $t_R$ 4.39 min (26.4%)

(Chiralpak AS, 125 bar, 40 $^o$C, 2.0% MeOH in $CO_2$, 2.5 mL/min, 220 nm)

Data for (2*S*,3*S*)-**6aa**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.37-7.32 (m, 4 H, HC(5), HC(6)), 7.31-7.27 (m, 1 H, HC(7)), 4.97 (d, $J$ = 5.9, 1 H, HC(3)), 3.98 (d, $J$ = 5.9, 1 H, HC(2)), 3.67 (s, 3 H, HC(8)), 3.37 (s, 3 H, HC(9)), 2.95 (bs, 1 H, OH)

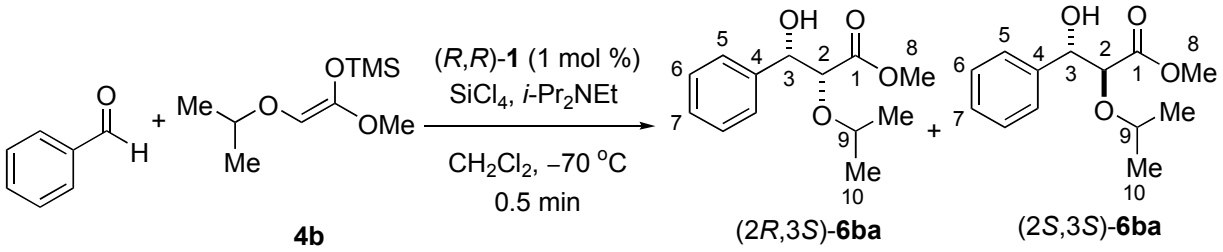
$^{13}$C NMR: (126 MHz, $CDCl_3$)

170.8 (C(1)), 139.4 (C(4)), 128.2 (C(6)), 128.0 (C(7)), 126.5 (C(5)), 84.5 (C(2)), 74.0 (C(3)), 58.8 (C(9)), 51.8 (C(8))

SFC: (2*S*,3*S*)-**6aa**, $t_R$ 12.73 min (81.5%); (2*R*,3*R*)-**6aa**, $t_R$ 15.44 min (18.5%)

(Chiralcel OD, 125 bar, 40 °C, 4.0% MeOH in $CO_2$, 1.0 mL/min, 220 nm)

**Preparation of (2*R*,3*S*)-3-Hydroxy-2-(1-methylethoxy)-3-phenylpropanoic Acid Methyl Ester ((2*R*,3*S*)-6ba) and (2*S*,3*S*)-3-Hydroxy-2-(1-methylethoxy)-3-phenylpropanoic Acid Methyl Ester ((2*S*,3*S*)-6ba) (Table 1, entry 2)**




Following General Procedure 6, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined with diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), benzaldehyde (101.6 μL, 1.0 mmol), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4b** (245 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 5/1 to 2/1) on silica gel (20 g), **6ba** (226 mg, 95%) as a colorless oil. The *syn*/*anti* ratio was determined to be 86/14 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*R*,3*S*)-**6ba**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.40-7.32 (m, 4 H, HC(5), HC(6)), 7.31-7.28 (m, 1 H, HC(7)), 4.90 (d, *J* = 5.6, 1 H, HC(3)), 4.00 (d, *J* = 5.6, 1 H, HC(2)), 3.63 (s, 3 H, HC(8)), 3.60 (qq, *J* = 6.1, 6.1, 1 H, HC(9)), 2.98 (bs, 1 H, OH), 1.18 (d, *J* = 6.1, 3 H, HC(10)), 1.05 (d, *J* = 6.1, 3 H, HC(10))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

171.6 (C(1)), 139.2 (C(4)), 128.2 (C(6)), 128.0 (C(7)), 126.4 (C(5)), 81.7 (C(2)), 74.7 (C(3)), 73.2 (C(9)), 51.9 (C(8)), 22.5 (C(10)), 21.3 (C(10))

IR: (neat)

3491 (br), 3032 (m), 2975 (s), 2933 (m), 1747 (s), 1496 (m), 1455 (s), 1436 (m), 1384 (s), 1333 (m), 1272 (s), 1197 (s), 1176 (s), 1123 (s), 1086 (s), 1058 (s), 1027 (m), 984 (m), 935 (m), 915 (m), 857 (m), 770 (m), 752 (m), 702 (s)

MS: (ESI)

179.1 (7), 261.1 ($M^+$+Na, 100), 262.1 (7), 338.4 (9)

HRMS: calcd for $C_{13}H_{18}O_4Na$: 261.1103, found 261.1116

TLC: $R_f$ 0.21 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

SFC: (2*R*,3*S*)-**6ba**, $t_R$ 3.32 min (79.8%); (2*S*,3*R*)-**6ba**, $t_R$ 4.35 min (20.2%)

(Chiralpak AS, 125 bar, 40 $^o$C, 1.0% MeOH in $CO_2$, 2.5 mL/min, 220 nm)

Data for (2*S*,3*S*)-**6ba**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.40-7.32 (m, 4 H, HC(5), HC(6)), 7.31-7.28 (m, 1 H, HC(7)), 4.92 (d, *J* = 6.6, 1 H, HC(3)), 4.07 (d, *J* = 6.6, 1 H, HC(2)), 3.68 (s, 3 H, HC(8)), 3.51 (qq, *J* = 6.1,

6.1, 1 H, HC(9)), 2.98 (bs, 1 H, OH), 1.14 (d, $J$ = 6.1, 3 H, HC(10)), 0.97 (d, $J$ = 6.1, 3 H, HC(10))

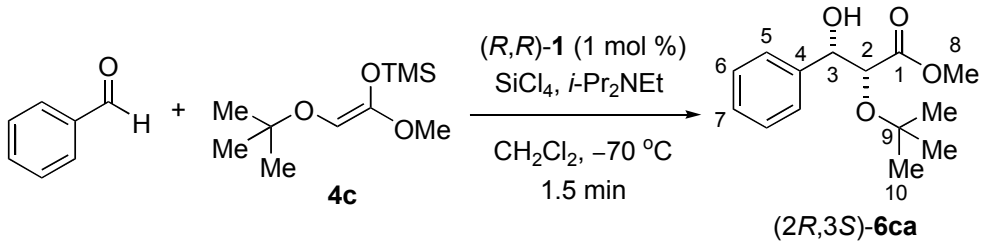
$^{13}$C NMR: (126 MHz, $CDCl_3$)

172.0 (C(1)), 139.9 (C(4)), 128.2 (C(6)), 128.0 (C(7)), 126.7 (C(5)), 80.7 (C(2)), 74.3 (C(3)), 73.1 (C(9)), 51.9 (C(8)), 22.4 (C(10)), 21.3 (C(10))

SFC: (2*R*,3*R*)-**6ba**, $t_R$ 7.03 min (23.0%); (2*S*,3*S*)-**6ba**, $t_R$ 8.21 min (77.0%)

(Chiralpak AS, 125 bar, 40 °C, 1.0% MeOH in $CO_2$, 2.5 mL/min, 220 nm)

**Preparation of (2*R*,3*S*)-2-(1,1-Dimethylethoxy)-3-hydroxy-3-phenylpropanoic Acid Methyl Ester ((2*R*,3*S*)-6ca) (Table 1, entry 3)**




Following General Procedure 6, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined with diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), benzaldehyde (101.6 μL, 1.0 mmol), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4c** (262 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 5/1 to 2/1) on silica gel (20 g), **6ca** (235 mg, 93%) as a colorless oil. The *syn*/*anti* ratio was determined to be 99/1 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*R*,3*S*)-**6ca**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.36-7.32 (m, 4 H, HC(5), HC(6)), 7.31-7.27 (m, 1 H, HC(7)), 4.82 (d, $J$ = 5.8, 1 H, HC(3)), 4.04 (d, $J$ = 5.8, 1 H, HC(2)), 3.59 (s, 3 H, HC(8)), 3.01 (bs, 1 H,

OH), 1.11 (s, 9 H, HC(10))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

172.7 (C(1)), 139.1 (C(4)), 128.2 (C(6)), 128.1 (C(7)), 126.5 (C(5)), 76.9 (C(2)), 76.0 (C(9)), 75.1 (C(3)), 51.8 (C(8)), 27.6 (C(10))

IR: (neat)

3501 (br), 3064 (w), 3032 (m), 2977 (s), 1748 (s), 1496 (m), 1455 (s), 1436 (m), 1393 (s), 1369 (s), 1333 (m), 1274 (s), 1192 (s), 1121 (s), 1088 (s), 1060 (s), 1026 (s), 985 (m), 898 (m), 776 (m), 758 (m), 735 (m), 701 (m)

MS: (ESI)

179.1 (6), 275.1 ($M^+$+Na, 100), 276.1 (6), 338.3 (11)

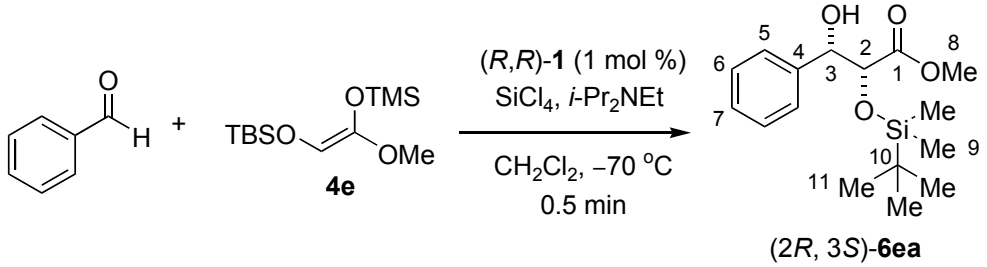
HRMS: calcd for $C_{14}H_{20}O_4Na$: 275.1259, found 275.1264

TLC: $R_f$ 0.21 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

SFC: (2*R*,3*S*)-**6ca**, $t_R$ 2.81 min (93.4%); (2*S*,3*R*)-**6ca**, $t_R$ 3.25 min (6.6%)

(Chiralcel OD, 125 bar, 40 °C, 3.0% MeOH in $CO_2$, 3.0 mL/min, 220 nm)

**Preparation of (2*R*,3*S*)-2-[[(1,1-Dimethylethyl)dimethylsilyl]oxy]-3-hydroxy-3-phenyl propanoic Acid Methyl Ester[20] ((2*R*,3*S*)-6ea) (Table 1, entry 5)**




(2*R*, 3*S*)-**6ea**

Following General Procedure 6, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined with diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), benzaldehyde (101.6 μL, 1.0 mmol), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4e** (332 mg, 1.2 mmol, 1.2 equiv) to yield, after

column chromatography (22 mm diam., hexane/EtOAc, 10/1 to 5/1) on silica gel (20 g), **6ea** (302 mg, 97%) as a colorless oil. The *syn*/*anti* ratio was determined to be 98/2 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*R*,3*S*)-**6ea**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.37-7.33 (m, 4 H, HC(5), HC(6)), 7.30-7.26 (m, 1 H, HC(7)), 5.02 (d, $J$ = 3.3, 1 H, HC(3)), 4.31 (d, $J$ = 3.3, 1 H, HC(2)), 3.74 (s, 3 H, HC(8)), 2.98 (bs, 1 H, OH), 0.79 (s, 9 H, HC(11)), –0.08 (s, 3 H, HC(9)), –0.23 (s, 3 H, HC(9))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

171.9 (C(1)), 140.3 (C(4)), 128.2 (C(6)), 127.7 (C(7)), 126.0 (C(5)), 76.9 (C(2)), 75.0 (C(3)), 52.1 (C(8)), 25.5 (C(11)), 18.2 (C(10)), –5.5 (C(9)), –5.9 (C(9))

IR: (neat)

3502 (br), 3065 (m), 3032 (m), 2953 (s), 2930 (s), 2887 (s), 2858 (s), 1760 (s), 1606 (m), 1472 (s), 1453 (s), 1437 (s), 1391 (s), 1362 (s), 1256 (s), 1198 (s), 1144 (s), 1084 (s), 1057 (s), 871 (s), 838 (s), 808 (s), 781 (s), 736 (s), 701 (s)

MS: (ESI)

249.2 (5), 293.2 (44), 294.2 (5), 311.2 (4), 333.1 ($M^+$+Na, 100)

HRMS: calcd for $C_{16}H_{26}O_4SiNa$: 333.1498, found 333.1497

TLC: $R_f$ 0.32 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

SFC: (2*R*,3*S*)-**6ea**, $t_R$ 3.01 min (95.3%); (2*S*,3*R*)-**6ea**, $t_R$ 4.15 min (4.7%)

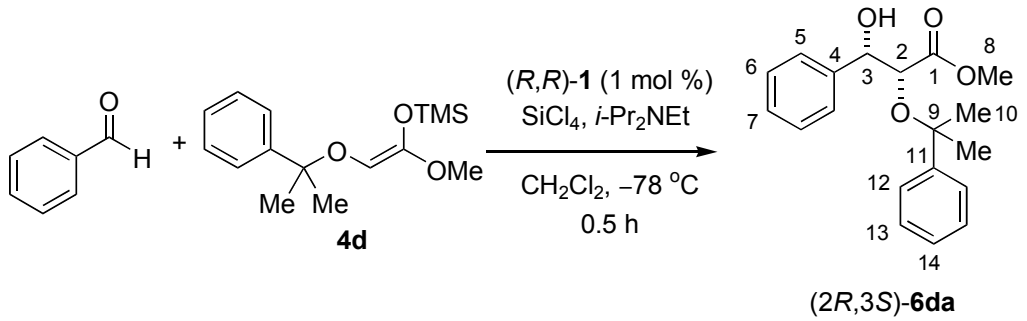
(Chiralpak AS, 125 bar, 40 $^o$C, 1.0% MeOH in $CO_2$, 2.5 mL/min, 220 nm)

Opt. Rot.: $[\alpha]_D^{24}$ 33.9 (c = 1.6, $CHCl_3$), $[\alpha]_D^{24}$ 34.3 (c = 3.5, EtOH)

lit. $[\alpha]_D^{24}$ 27.5 (c =1.6, $CHCl_3$) for (2*R*,3*S*)-**6ea**

**General Procedure 7. Addition of Silyl Ketene Acetals to Aromatic Aldehydes**

**Preparation of (2*R*,3*S*)-3-Hydroxy-2-(1-methyl-1-phenylethoxy)-3-phenylpropanoic Acid Methyl Ester ((2*R*,3*S*)-6da) (Table 2, entry 1)**




To a flame-dried, 10-mL, Schlenk flask fitted with a magnetic stir bar, a thermocouple, a gas inlet tube, and a septum were added (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv), $CH_2Cl_2$ (5 mL), and benzaldehyde (101.6 μL, 1.0 mmol). The solution was cooled to –78 °C (internal temp.) in a dry ice-acetone bath. After diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv) and $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv) were added to the flask via syringe, a solution of **4d** (336.5 mg, 1.2 mmol, 1.2 equiv) in $CH_2Cl_2$ (5 mL) was added dropwise via syringe over 15 min. The internal temperature was kept below –70 °C during the addition of **4d**. The reaction mixture was stirred for additional 15 min at –78 °C before a mixture of MeOH (1 mL), $Et_3N$ (1 mL), and $CH_2Cl_2$ (5 mL) was added. The resulting solution was transferred into a 125-mL Erlenmeyer flask containing a sat. aq. $NaHCO_3$ solution (10 mL) and a sat. aq. KF solution (10 mL). The biphasic mixture was stirred vigorously for 2 h at room temperature. The mixture was filtered through a glass frit and the filtrate was transferred to a 125-mL separatory funnel where the organic layer was separated. The aqueous layer was extracted with $CH_2Cl_2$ (2 × 20 mL). The combined organic extracts were dried over $Na_2SO_4$ (15 g), filtered, and concentrated *in vacuo* (23 °C, 30 mmHg). The residue was purified by column chromatography (18 mm diam., hexane/EtOAc, 5/1 to 1/1) on silica gel (10 g) to give **6da** (275 mg, 87%) as a colorless oil. The *syn*/*anti* ratio was

determined to be 99/1 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*R*,3*S*)-**6da**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.36-7.33 (m, 2 H, HC(12)), 7.31-7.22 (m, 8 H, HC(5), HC(6), HC(7), HC(13), HC(14)), 4.79 (d, *J* = 6.1, 1 H, HC(3)), 3.86 (d, *J* = 6.1, 1 H, HC(2)), 3.40 (s, 3 H, HC(8)), 2.98 (bs, 1 H, OH), 1.53 (s, 3 H, HC(10)), 1.52 (s, 3 H, HC(10))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

172.3 (C(1)), 143.8 (C(11)), 138.8 (C(4)), 128.1 (C(6) or C(13)), 128.03 (C(6) or C(13)), 127.99 (C(7)), 127.4 (C(14)), 126.5 (C(5)), 126.1 (C(12)), 78.8 (C(9)), 77.4 (C(2)), 75.0 (C(3)), 51.5 (C(8)), 27.9 (C(10)), 27.1 (C(10))

IR: (neat)

3490 (br), 3062 (w), 3031 (w), 2981 (m), 2950 (w), 1745 (s), 1496 (m), 1451 (m), 1435 (m), 1385 (m), 1368 (m), 1264 (m), 1197 (m), 1170 (m), 1151 (m), 1086 (s), 1076 (s), 1059 (s), 1028 (m), 983 (m), 914 (m), 879 (m), 767 (s), 722 (m), 700 (s)

MS: (ESI)

119.1 (86), 120.1 (5), 136.1 (15), 179.1 (35), 214.1 (8), 332.2 (100), 337.1 ($M^+$+Na, 58), 338.1 (6), 353.1 (23)

HRMS: calcd for $C_{19}H_{22}O_4Na$: 337.1416, found 337.1413

TLC: $R_f$ 0.27 (hexane/EtOAc, 4/1) [UV(254)/$KMnO_4$]

SFC: (2*R*,3*S*)-**6da**, $t_R$ 7.61 min (96.6%); (2*S*,3*R*)-**6da**, $t_R$ 8.95 min (3.4%)

(Chiralpak AS, 125 bar, 40 $^o$C, 1.6% MeOH in $CO_2$, 2.5 mL/min, 220 nm)

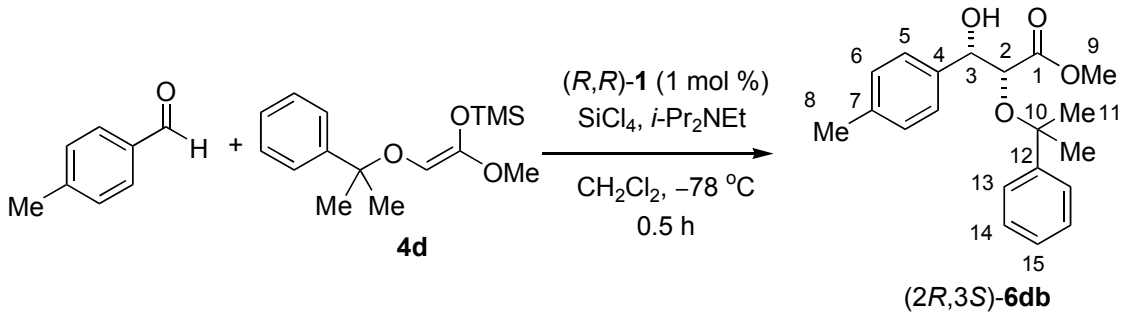
Opt. Rot.: $[\alpha]_D^{24}$ 42.2 (c = 1.0, EtOH)

Analysis: $C_{19}H_{22}O_4$ (314.38)

Calcd: C, 72.59%; H, 7.05%

Found: C, 72.59%; H, 7.08%

**Preparation of (2*R*,3*S*)-3-Hydroxy-3-(4-methylphenyl)-2-(1-methyl-1-phenylethoxy) propanoic Acid Methyl Ester ((2*R*,3*S*)-6db) (Table 2, entry 2)**




Following General Procedure 7, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined with diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), 4-tolualdehyde (118 μL, 1.0 mmol), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4d** (336.5 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 5/1 to 2/1) on silica gel (15 g), **6db** (319 mg, 97%) as a colorless oil. The *syn*/*anti* ratio was determined to be >99/1 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*R*,3*S*)-**6db**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.37-7.35 (m, 2 H, HC(13)), 7.29-7.22 (m, 3 H, HC(14), HC(15)), 7.14-7.12 (m, 2 H, HC(5)), 7.10-7.08 (m, 2 H, HC(6)), 4.75 (d, *J* = 6.2, 1 H, HC(3)), 3.84 (d, *J* = 6.2, 1 H, HC(2)), 3.40 (s, 3 H, HC(9)), 2.95 (bs, 1 H, OH), 2.32 (s, 3 H, HC(8)), 1.54 (s, 3 H, HC(11)), 1.53 (s, 3 H, HC(11))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

172.3 (C(1)), 144.0 (C(12)), 137.7 (C(7)), 135.7 (C(4)), 128.9 (C(6)), 128.1 (C(14)), 127.5 (C(15)), 126.5 (C(5)), 126.1 (C(13)), 78.9 (C(10)), 77.6 (C(2)), 74.9 (C(3)), 51.6 (C(9)), 28.1 (C(11)), 27.2 (C(11)), 21.1 (C(8))

IR: (neat)

3498 (br), 3059 (w), 3026 (m), 2981 (m), 2950 (m), 1745 (s), 1516 (m), 1496 (m), 1448 (m), 1435 (m), 1385 (m), 1368 (m), 1264 (s), 1196 (s), 1169 (s), 1152 (s), 1111 (s), 1067 (s), 1030 (m), 1020 (m), 912 (m), 818 (s), 766 (s), 732 (m), 701 (s)

MS: (ESI)

119.1 (68), 120.1 (5), 136.1 (12), 193.1 (64), 194.1 (5), 228.1 (6), 335.2 (5), 346.2 (100), 347.2 (13), 351.2 ($M^+$+Na, 19)

HRMS: calcd for $C_{20}H_{24}O_4Na$: 351.1572, found 351.1581

TLC: $R_f$ 0.33 (hexane/EtOAc, 4/1) [UV(254)/$KMnO_4$]

SFC: (2*R*,3*S*)-**6db**, $t_R$ 7.46 min (97.5%); (2*S*,3*R*)-**6db**, $t_R$ 9.31 min (2.5%)

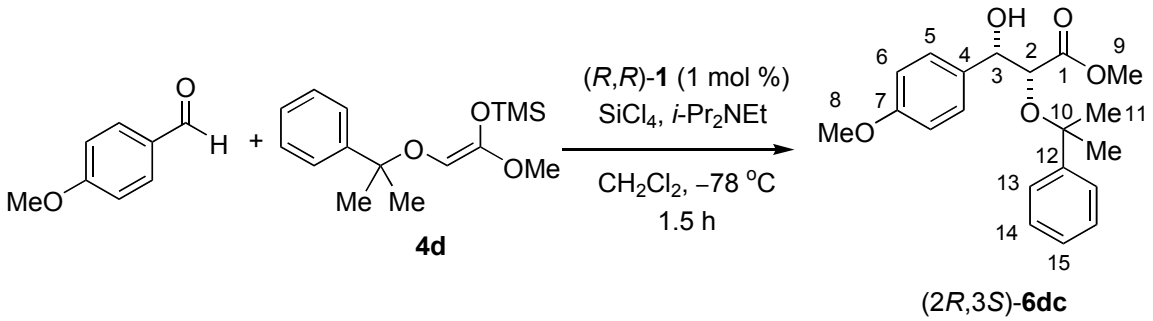
(Chiralpak AS, 125 bar, 40 $^oC$, 1.5% MeOH in $CO_2$, 2.5 mL/min, 220 nm)

Opt. Rot.: $[\alpha]_D^{24}$ 32.2 (c = 1.5, EtOH)

Analysis: $C_{20}H_{24}O_4$ (328.40)

| | | |
|---|---|---|
| Calcd: | C, 73.15%; | H, 7.37% |
| Found: | C, 72.96%; | H, 7.37% |

**Preparation of (2*R*,3*S*)-3-Hydroxy-3-(4-methoxyphenyl)-2-(1-methyl-1-phenylethoxy) propanoic Acid Methyl Ester ((2*R*,3*S*)-6dc) (Table 2, entry 3)**




(2*R*,3*S*)-**6dc**

Following General Procedure 7, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined with diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), 4-anisaldehyde (121.7 μL, 1.0 mmol), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4d** (336.5 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 5/1 to 1/1) on silica gel (15 g), **6dc** (336 mg, 98%) as a colorless oil. The *syn*/*anti* ratio was determined to be 99/1 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*R*,3*S*)-**6dc**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.39-7.37 (m, 2 H, HC(13)), 7.31-7.27 (m, 2 H, HC(14)), 7.26-7.23 (m, 1 H, HC(15)), 7.18-7.15 (m, 2 H, HC(5)), 6.84-6.81 (m, 2 H, HC(6)), 4.73 (d, *J* = 6.5, 1 H, HC(3)), 3.82 (d, *J* = 6.5, 1 H, HC(2)), 3.78 (s, 3 H, HC(8)), 3.38 (s, 3 H, HC(9)), 2.93 (bs, 1 H, OH), 1.55 (s, 3 H, HC(11)), 1.54 (s, 3 H, HC(11))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

172.3 (C(1)), 159.4 (C(7)), 144.0 (C(12)), 130.7 (C(4)), 128.1 (C(14)), 127.9 (C(5)), 127.6 (C(15)), 126.2 (C(13)), 113.6 (C(6)), 78.9 (C(10)), 77.7 (C(2)), 74.7 (C(3)), 55.2 (C(8)), 51.6 (C(9)), 28.0 (C(11)), 27.3 (C(11))

IR: (neat)

3500 (br), 2981 (m), 2951 (m), 2250 (w), 1744 (s), 1614 (m), 1587 (w), 1515 (s), 1448 (m), 1385 (m), 1368 (m), 1304 (m), 1251 (s), 1198 (m), 1173 (s), 1111 (m), 1198 (m), 1067 (s), 1032 (s), 912 (m), 881 (w), 833 (m), 766 (m), 733 (m), 702 (s)

MS: (ESI)

119.1 (26), 136.1 (13), 209.1 (100), 210.1 (7), 362.2 (9), 367.2 ($M^+$+Na, 78), 368.2 (8), 383.1 (15)

HRMS: calcd for $C_{20}H_{24}O_5Na$: 367.1521, found 367.1514

TLC: $R_f$ 0.37 (hexane/EtOAc, 2/1) [UV(254)/$KMnO_4$]

SFC: (2*R*,3*S*)-**6dc**, $t_R$ 9.09 min (97.7%); (2*S*,3*R*)-**6dc**, $t_R$ 10.48 min (2.3%)

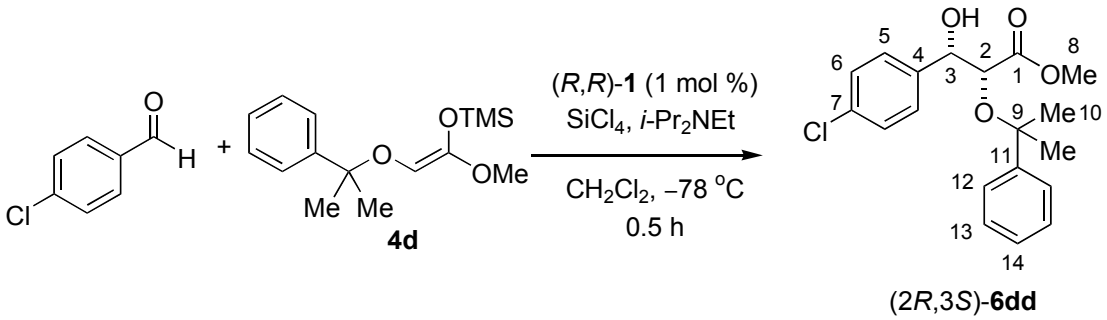
(Chiralpak AS, 125 bar, 40 $^oC$, 2.5% MeOH in $CO_2$, 2.0 mL/min, 220 nm)

Opt. Rot.: $[\alpha]_D^{24}$ 32.4 (c = 3.0, EtOH)

Analysis: $C_{20}H_{24}O_5$ (344.40)

| | | |
|---|---|---|
| Calcd: | C, 69.75%; | H, 7.02% |
| Found: | C, 69.51%; | H, 7.07% |

**Preparation of (2*R*,3*S*)-3-(4-Chlorophenyl)-3-hydroxy-2-(1-methyl-1-phenylethoxy) propanoic Acid Methyl Ester ((2*R*,3*S*)-6dd) (Table 2, entry 4)**




Following General Procedure 7, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined with diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), 4-chlorobenzaldehyde (140.6 mg, 1.0 mmol), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4d** (336.5 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 5/1 to 1/1) on silica gel (15 g), **6dd** (325 mg, 93%) as a colorless oil. The *syn*/*anti* ratio was determined to be >99/1 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*R*,3*S*)-**6dd**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.34-7.31 (m, 2 H, HC(12)), 7.29-7.23 (m, 5 H, HC(6), HC(13), HC(14)), 7.20-7.17 (m, 2 H, HC(5)), 4.77 (d, *J* = 5.9, 1 H, HC(3)), 3.81 (d, *J* = 5.9, 1 H, HC(2)), 3.44 (s, 3 H, HC(8)), 3.03 (bs, 1 H, OH), 1.54 (s, 3 H, HC(10)), 1.53 (s, 3 H, HC(10))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

172.1 (C(1)), 143.6 (C(11)), 137.5 (C(4)), 133.8 (C(7)), 128.3 (C(6) or C(13)), 128.1 (C(6) or C(13)), 127.9 (C(5)), 127.6 (C(14)), 126.1 (C(12)), 79.0 (C(9)), 77.2 (C(2)), 74.4 (C(3)), 51.7 (C(8)), 28.1 (C(10)), 27.0 (C(10))

IR: (neat)

3485 (br), 2981 (m), 2950 (w), 1744 (s), 1600 (w), 1494 (s), 1448 (m), 1436 (m), 1409 (m), 1385 (m), 1368 (m), 1264 (s), 1196 (s), 1170 (s), 1151 (m), 1090 (s), 1069 (s), 1030 (m), 1015 (s), 911 (m), 880 (m), 829 (m), 767 (s), 744 (m), 701 (s)

MS: (ESI)

119.1 (11), 285.0 (4), 326.0 (8), 330.9 (12), 333.0 (4), 355.1 (4), 371.1 ($M^+$+Na, 100), 372.1 (12), 373.1 (24)

HRMS: calcd for $C_{19}H_{21}ClO_4Na$: 371.1026, found 371.1016

TLC: $R_f$ 0.25 (hexane/EtOAc, 4/1) [UV(254)/$KMnO_4$]

SFC: (2*R*,3*S*)-**6dd**, $t_R$ 9.58 min (97.2%); (2*S*,3*R*)-**6dd**, $t_R$ 11.81 min (2.8%)

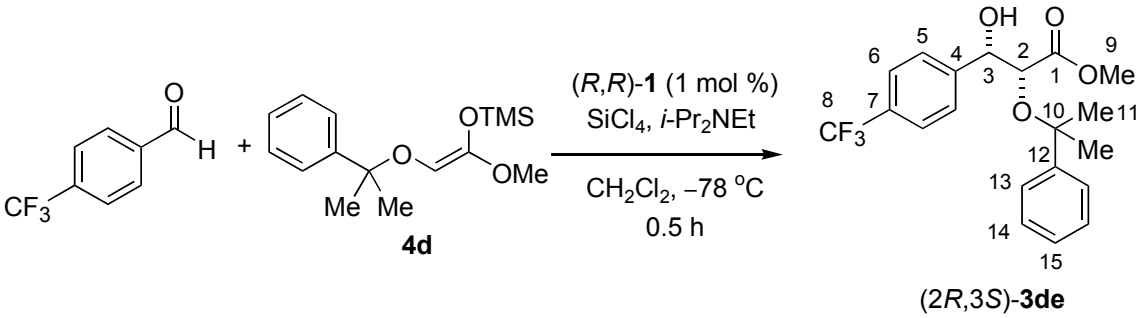
(Chiralpak AS, 125 bar, 40 $^oC$, 1.5% MeOH in $CO_2$, 2.5 mL/min, 220 nm)

Opt. Rot.: $[\alpha]_D^{24}$ 29.3 (c = 2.5, EtOH)

Analysis: $C_{19}H_{21}ClO_4$ (348.82)

| | | |
|---|---|---|
| Calcd: | C, 65.42%; | H, 6.07% |
| Found: | C, 65.06%; | H, 5.68% |

**Preparation of (2*R*,3*S*)-3-Hydroxy-2-(1-methyl-1-phenylethoxy)-3-[4-(trifluoromethyl) phenyl]propanoic Acid Methyl Ester ((2*R*,3*S*)-6de) (Table 2, entry 5)**




Following General Procedure 7, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined with diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), 4-trifluoromethylbenzaldehyde (136.6 μL, 1.0 mmol), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4d** (336.5 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 5/1 to 2/1) on silica gel (15 g), **6de** (380 mg, 99%) as a colorless oil. The *syn*/*anti* ratio was determined to be 99/1 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*R*,3*S*)-**6de**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.55-7.54 (m, 2 H, HC(6)), 7.36-7.35 (m, 2 H, HC(5)), 7.27-7.21 (m, 5 H, HC(13), HC(14), HC(15)), 4.84 (dd, *J* = 5.4, 4.9, 1 H, HC(3)), 3.84 (d, *J* = 5.4, 1 H, HC(2)), 3.48 (s, 3 H, HC(9)), 3.07 (d, *J* = 4.9, 1 H, OH), 1.53 (s, 3 H, HC(11)), 1.51 (s, 3 H, HC(11))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

172.1 (C(1)), 143.31 (C(4) or C(12)), 143.29 (C(4) or (C(12)), 130.1 (q, *J* = 32.5, C(7)), 128.1 (C(14)), 127.6 (C(15)), 126.8 (C(5)), 126.1 (C(13)), 125.1 (q, *J* = 3.6, C(6)), 124.1 (q, *J* =271.8, C(8)), 79.1 (C(10)), 76.9 (C(2)), 74.4 (C(3)), 51.9 (C(9)), 28.3 (C(11)), 26.7 (C(11))

$^{19}$F NMR: (376 MHz, $CDCl_3$)

−62.99 (FC(8))

IR: (neat)

3483 (br), 2983 (m), 2953 (w), 2251 (w), 1746 (s), 1621 (m), 1496 (m), 1449 (m), 1437 (m), 1418 (m), 1386 (m), 1369 (m), 1327 (s), 1265 (m), 1198 (m), 1166 (s), 1125 (s), 1068 (s), 1018 (s), 911 (m), 881 (m), 843 (m), 768 (m), 735 (m), 701 (s)

MS: (ESI)

119.1 (100), 136.1 (21), 282.1 (7), 400.2 (28), 405.1 ($M^+$+Na, 46), 421.1 (8)

HRMS: calcd for $C_{20}H_{21}F_3O_4Na$: 405.1290, found 405.1284

TLC: $R_f$ 0.22 (hexane/EtOAc, 4/1) [UV(254)/$KMnO_4$]

SFC: (2*R*,3*S*)-**6de**, $t_R$ 5.75 min (98.0%); (2*S*,3*R*)-**6de**, $t_R$ 6.62 min (2.0%)

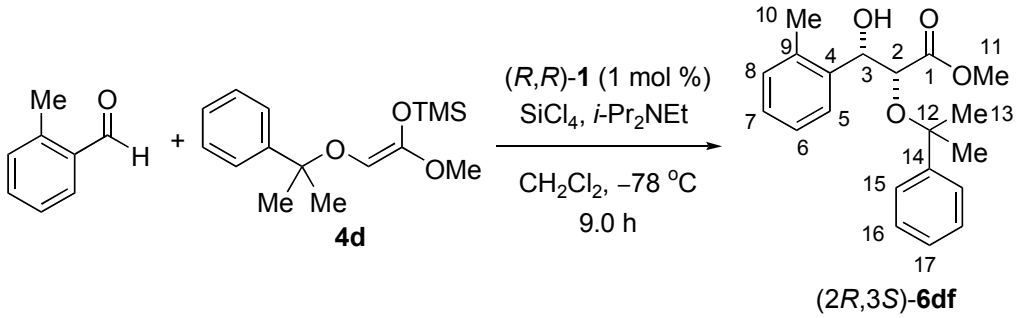
(Chiralpak AS, 125 bar, 40 °C, 1.6% MeOH in $CO_2$, 1.8 mL/min, 220 nm)

Opt. Rot.: $[\alpha]_D^{24}$ 34.3 (c = 8.3, EtOH)

Analysis: $C_{20}H_{21}F_3O_4$ (382.37)

| | | |
|---|---|---|
| Calcd: | C, 62.82%; | H, 5.54% |
| Found: | C, 62.50%; | H, 5.49% |

**Preparation of (2*R*,3*S*)-3-Hydroxy-3-(2-methylphenyl)-2-(1-methyl-1-phenylethoxy) propanoic Acid Methyl Ester ((2*R*,3*S*)-6df) (Table 2, entry 6)**




Following General Procedure 7, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined with diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), 2-tolualdehyde (116 μL, 1.0 mmol), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4d** (336.5 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 5/1 to 1/1) on silica gel (15 g), **6df** (305 mg, 93%) as a colorless oil. The *syn*/*anti* ratio was determined to be 98/2 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*R*,3*S*)-**6df**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.39-7.38 (m, 1 H, HC(5)), 7.26-7.16 (m, 7 H, HC(6), HC(7), HC(15), HC(16), HC(17)), 7.06-7.05 (m, 1 H, HC(8)), 5.04 (d, *J* = 5.3, 1 H, HC(3)), 3.91 (d, *J* = 5.3, 1 H, HC(2)), 3.47 (s, 3 H, HC(11)), 2.89 (bs, 1 H, OH), 2.16 (s, 3 H, HC(10)), 1.50 (s, 3 H, HC(13)), 1.47 (s, 3 H, HC(13))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

172.5 (C(1)), 143.6 (C(14)), 137.1 (C(4)), 135.0 (C(9)), 130.4 (C(8)), 128.0 (C(16)), 127.8 (C(7)), 127.4 (C(17)), 127.1 (C(5)), 126.1 (C(15)), 125.8 (C(6)), 78.9 (C(12)), 75.4 (C(2)), 71.8 (C(3)), 51.7 (C(11)), 28.2 (C(13)), 26.8 (C(13)), 18.8 (C(10))

IR: (neat)

3492 (br), 3061 (w), 3027 (w), 2980 (m), 2951 (m), 1746 (s), 1495 (m), 1448 (m), 1436 (m), 1384 (m), 1368 (m), 1264 (s), 1197 (s), 1171 (s), 1151 (s), 1113 (s), 1100 (s), 1075 (s), 1057 (s), 1030 (m), 911 (m), 766 (s), 733 (s), 701 (s)

MS: (ESI)

119.1 (24), 193.1 (16), 346.2 (14), 351.1 ($M^+$+Na, 100), 352.1 (12), 367.1 (5)

HRMS: calcd for $C_{20}H_{24}O_4Na$: 351.1572, found 351.1564

TLC: $R_f$ 0.24 (hexane/EtOAc, 4/1) [UV(254)/$KMnO_4$]

SFC: (2*S*,3*R*)-**6df**, $t_R$ 4.46 min (3.2%); (2*R*,3*S*)-**6df**, $t_R$ 5.09 min (96.8%)

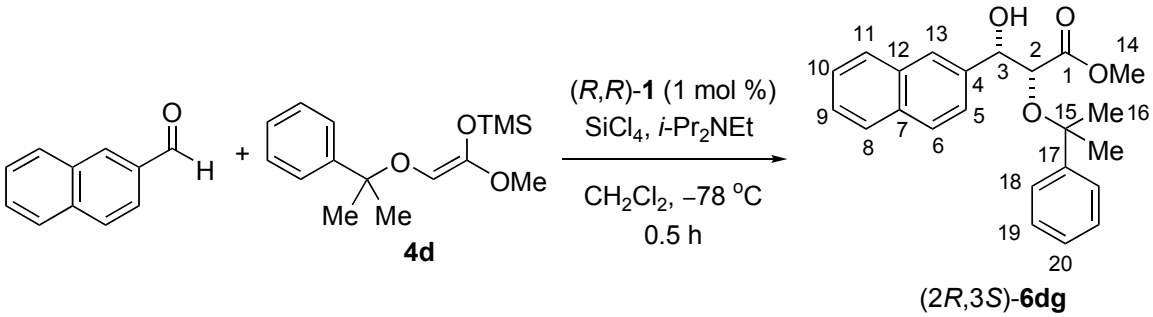
(Chiralcel OD, 125 bar, 40 °C, 5.0% MeOH in $CO_2$, 3.0 mL/min, 220 nm)

Opt. Rot.: $[\alpha]_D^{24}$ 68.5 (c = 2.0, EtOH)

Analysis: $C_{20}H_{24}O_4$ (328.40)

| | | |
|---|---|---|
| Calcd: | C, 73.15%; | H, 7.37% |
| Found: | C, 73.13%; | H, 7.33% |

**Preparation of (2*R*,3*S*)-3-Hydroxy-2-(1-methyl-1-phenylethoxy)-3-(2-naphthyl)propanoic Acid Methyl Ester ((2*R*,3*S*)-6dg) (Table 2, entry 7)**




Following General Procedure 7, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined with diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), 2-naphthaldehyde (156 mg, 1.0

mmol), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4d** (336.5 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 5/1 to 2/1) on silica gel (15 g), **6dg** (342 mg, 94%) as a colorless oil. The *syn*/*anti* ratio was determined to be >99/1 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*R*,3*S*)-**6dg**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.83-7.79 (m, 2 H, HC(8), HC(11)), 7.77 (d, *J* = 8.3, 1 H, HC(6)), 7.72 (bs, 1 H, HC(13)), 7.49-7.45 (m, 2 H, HC(9), HC(10)), 7.36 (dd, *J* = 8.3, 1.7, 1 H, HC(5)), 7.32-7.30 (m, 2 H, HC(18)), 7.21-7.15 (m, 3 H, HC(19), HC(20)), 4.97 (dd, *J* = 5.7, 4.2, 1 H, HC(3)), 3.96 (d, *J* = 5.7, 1 H, HC(2)), 3.39 (s, 3 H, HC(14)), 3.15 (d, *J* = 4.2, 1 H, OH), 1.54 (s, 3 H, HC(16)), 1.521 (s, 3 H, HC(16))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

172.4 (C(1)), 143.7 (C(17)), 136.4 (C(4)), 133.2 (C(7) or C(12)), 133.0 (C(7) or C(12)), 128.03 (C(19)), 128.00 (C(6), C(8), C(11), or C(20)), 127.96 (C(6), C(8), C(11), or C(20)), 127.6 (C(6), C(8), C(11), or C(20)), 127.5 (C(6), C(8), C(11), or C(20)), 126.1 (C(18)), 126.05 (C(9) or C(10)), 125.97 (C(9) or C(10)), 125.7 (C(13)), 124.2 (C(5)), 79.0 (C(15)), 77.3 (C(2)), 75.1 (C(3)), 51.7 (C(14)), 28.1 (C(16)), 27.0 (C(16))

IR: (neat)

3464 (br), 2984 (m), 2257 (m), 1741 (s), 1450 (m), 1439 (s), 1382 (m), 1368 (s), 1356 (m), 1315 (m), 1286 (m), 1200 (m), 1148 (s), 1113 (s), 1073 (s), 991 (m), 902 (s), 881 (m), 859 (s), 827 (s), 766 (s), 743 (m), 718 (s), 701 (s), 644 (m)

MS: (ESI)

119.1 (4), 229.1 (4), 287.2 (5), 387.1 ($M^+$+Na, 100), 388.1 (26)

HRMS: calcd for $C_{23}H_{24}O_4Na$: 387.1572, found 387.1574

TLC: $R_f$ 0.29 (hexane/EtOAc, 4/1) [UV(254)/$KMnO_4$]

SFC: (2*S*,3*R*)-**6dg**, $t_R$ 5.60 min (1.5%); (2*R*,3*S*)-**6dg**, $t_R$ 6.25 min (98.5%)

(Chiralcel OD, 125 bar, 40 °C, 10.0% MeOH in $CO_2$, 3.0 mL/min, 220 nm)

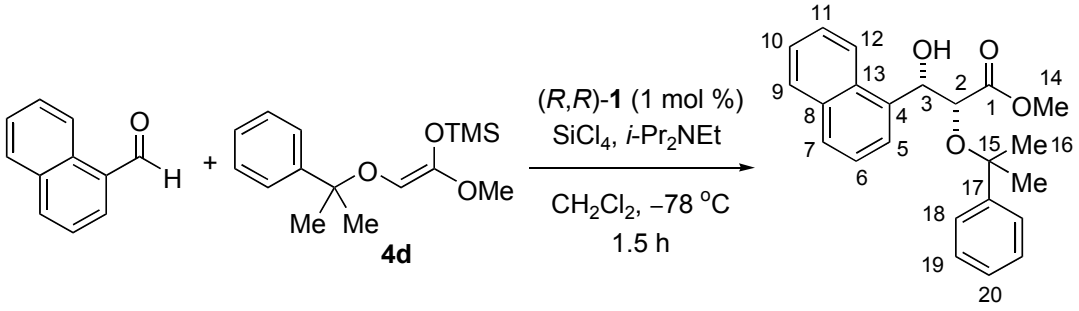
Opt. Rot.: $[\alpha]_D^{24}$ 30.7 (c = 2.0, EtOH)

Analysis: $C_{23}H_{24}O_4$ (364.43)

Calcd: C, 75.80%; H, 6.64%

Found: C, 75.67%; H, 6.37%

**Preparation of (2*R*,3*S*)-3-Hydroxy-2-(1-methyl-1-phenylethoxy)-3-(1-naphthyl)propanoic Acid Methyl Ester ((2*R*,3*S*)-6dh) (Table 2, entry 8)**




Following General Procedure 7, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined with diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), 1-naphthaldehyde (136 μL, 1.0 mmol), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4d** (336.5 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 5/1 to 2/1) on silica gel (15 g), **6dh** (338 mg, 93%) as a colorless oil. The *syn*/*anti* ratio was determined to be 99/1 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*R*,3*S*)-**6dh**:

$^{1}$H NMR: (500 MHz, $CDCl_3$)

7.88-7.87 (m, 1 H, HC(12)), 7.85-7.84 (m, 1 H, HC(9)), 7.81 (d, *J* = 8.0, 1 H, HC(7)), 7.63 (bd, *J* = 7.1, 1 H, HC(5)), 7.49 (dd, *J* = 8.0, 7.1, 1 H, HC(6)), 7.46-7.40 (m, 2 H, HC(10), HC(11)), 7.18-7.14 (m, 2 H, HC(18)), 7.10-7.07 (m, 3 H, HC(19), HC(20)), 5.60 (d, *J* = 4.5, 1 H, HC(3)), 4.16 (d, *J* = 4.5, 1 H, HC(2)), 3.47 (s, 3 H, HC(14)), 3.19 (bs, 1 H, OH), 1.364 (s, 3 H, HC(16)), 1.35 (s, 3 H, HC(16))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

172.7 (C(1)), 143.5 (C(17)), 134.9 (C(4)), 133.6 (C(8)), 130.3 (C(13)), 128.8 (C(9)), 128.5 (C(7)), 127.8 (C(19)), 127.3 (C(20)), 126.1 (C(11)), 125.9 (C(18)), 125.4 (C(10)), 125.1 (C(6)), 124.8 (C(5)), 122.5 (C(12)), 78.9 (C(15)), 75.3 (C(2)), 72.4 (C(3)), 51.8 (C(14)), 28.0 (C(16)), 26.5 (C(16))

IR: (neat)

3490 (br), 3059 (w), 2980 (m), 2950 (m), 2249 (w), 1746 (s), 1512 (m), 1496 (m), 1448 (m), 1435 (m), 1384 (m), 1368 (m), 1267 (s), 1198 (s), 1165 (s), 1112 (s), 1088 (s), 1017 (m), 911 (m), 803 (m), 780 (s), 767 (s), 732 (s), 701 (s)

MS: (ESI)

119.1 (6), 211.1 (6), 287.2 (4), 382.2 (7), 387.1 ($M^+$+Na, 100), 388.1 (25)

HRMS: calcd for $C_{23}H_{24}O_4Na$: 387.1572, found 387.1589

TLC: $R_f$ 0.20 (hexane/EtOAc, 4/1) [UV(254)/$KMnO_4$]

SFC: (2*R*,3*S*)-**6dh**, $t_R$ 13.50 min (96.1%); (2*S*,3*R*)-**6dh**, $t_R$ 14.51 min (3.9%)

(Chiralpak AD, 125 bar, 40 $^{o}$C, 5.0% MeOH in $CO_2$, 2.5 mL/min, 220 nm)

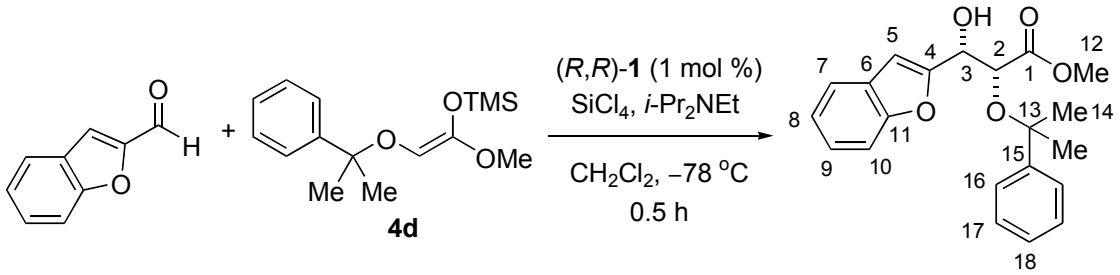
Opt. Rot.: $[\alpha]_D^{24}$ 55.5 (c = 1.4, EtOH)

Analysis: $C_{23}H_{24}O_4$ (364.43)

Calcd: C, 75.80%; H, 6.64%

Found: C, 75.83%; H, 6.56%

**Preparation of (2*R*,3*S*)-3-(2-Benzofuryl)-3-hydroxy-2-(1-methyl-1-phenylethoxy)propanoic Acid Methyl Ester ((2*R*,3*R*)-6di) (Table 2, entry 9)**




Following General Procedure 7, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined with diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), 2-benzofuraldehyde (121 μL, 1.0 mmol), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4d** (336.5 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 5/1 to 2/1) on silica gel (15 g), **6di** (336 mg, 95%) as a colorless oil. Analytical sample was obtained after recrystallization from $Et_2O$/hexane. Enantiomeric ratio was measured prior to recrystallization. The *syn*/*anti* ratio was determined to be 98/2 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*R*,3*R*)-**6di**:

mp: 94-98 °C ($Et_2O$/hexane)

$^1$H NMR: (500 MHz, $CDCl_3$)

7.55-7.53 (m, 1 H, HC(7)), 7.36-7.35 (m, 1 H, HC(10)), 7.30-7.28 (m, 2 H, HC(16)), 7.28-7.21 (m, 2 H, HC(8), HC(9)), 7.15-7.12 (m, 1 H, HC(18)), 7.09-7.06 (m, 2 H, HC(17)), 6.73 (bs, 1 H, HC(5)), 4.99 (ddd, *J* = 7.9, 4.2, 0.5, 1 H,

HC(3)), 4.27 (d, $J$ = 4.2, 1 H, HC(2)), 3.61 (s, 3 H, HC(12)), 3.21 (d, $J$ = 7.9, 1 H, OH), 1.51 (s, 3 H, HC(14)), 1.50 (s, 3 H, HC(14))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

172.0 (C(1)), 155.2 (C(4)), 154.7 (C(11)), 143.8 (C(15)), 127.94 (C(6)), 127.91 (C(17)), 127.4 (C(18)), 125.9 (C(16)), 124.0 (C(9)), 122.7 (C(8)), 121.0 (C(7)), 111.3 (C(10)), 104.5 (C(5)), 79.2 (C(13)), 73.9 (C(2)), 70.2 (C(3)), 52.0 (C(12)), 28.4 (C(14)), 26.4 (C(14))

IR: (neat)

3484 (br), 3036 (w), 2982 (m), 2952 (m), 2251 (w), 1748 (s), 1604 (w), 1496 (m), 1454 (s), 1436 (m), 1385 (m), 1369 (m), 1257 (s), 1199 (s), 1174 (s), 1151 (s), 1117 (s), 1074 (s), 911 (m), 882 (m), 811 (m), 767 (s), 751 (s), 701 (s)

MS: (ESI)

119.1 (100), 136.1 (22), 149.1 (33), 163.1 (8), 219.1 (46), 254.1 (9), 279.1 (16), 309.0 (9), 337.0 (9), 361.2 (8), 372.2 (23), 377.1 ($M^+$+Na, 28), 378.2 (8)

HRMS: calcd for $C_{21}H_{22}O_5Na$: 377.1365, found 377.1370

TLC: $R_f$ 0.29 (hexane/EtOAc, 4/1) [UV(254)/$KMnO_4$]

SFC: (2*S*,3*S*)-**6di**, $t_R$ 11.87 min (27.8%); (2*R*,3*R*)-**6di**, $t_R$ 12.89 min (72.2%)

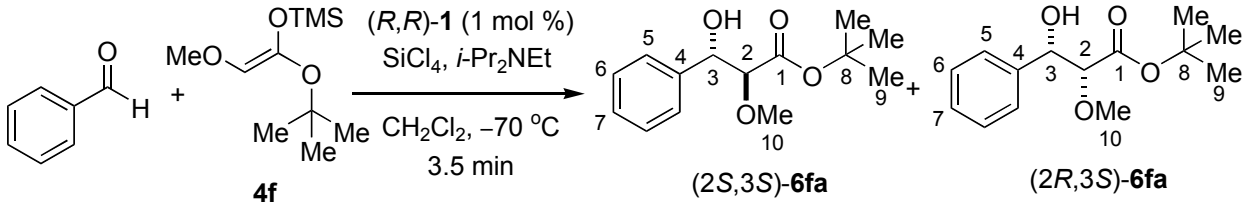
(Chiralcel OD, 125 bar, 40 $^oC$, 3.0% MeOH in $CO_2$, 3.0 mL/min, 220 nm)

Opt. Rot.: $[\alpha]_D^{24}$ 18.1 (c = 2.1, EtOH)

Analysis: $C_{21}H_{22}O_5$ (354.40)

| | | |
|---|---|---|
| Calcd: | C, 71.17%; | H, 6.26% |
| Found: | C, 71.27%; | H, 6.26% |

**Preparation of (2*S*,3*S*)-3-Hydroxy-2-methoxy-3-phenylpropanoic Acid 1,1-Dimethylethyl Ester [21] ((2*S*,3*S*)-6fa) and (2*R*,3*S*)-3-Hydroxy-2-methoxy-3-phenylpropanoic Acid 1,1-Dimethylethyl Ester ((2*R*,3*S*)-6fa) (Table 3, entry 1)**




Following General Procedure 6, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined with diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), benzaldehyde (101.6 μL, 1.0 mmol), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4f** (262 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 5/1 to 2/1) on silica gel (20 g), **6fa** (235 mg, 93%) as a colorless oil. The *syn*/*anti* ratio was determined to be 4/96 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture. For clear SFC anaylsis, the diastereomers were partially separated by column chromatography (18 mm diam., pentane/$Et_2O$, 1/1) on silica gel (10 g). The fractions which contained a single diastereomer were used for the SFC anaylsis.

Data for (2*S*,3*S*)-**6fa**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.41-7.39 (m, 2 H, HC(5)), 7.35-7.32 (m, 2 H, HC(6)), 7.30-7.27 (m, 1 H, HC(7)), 4.99 (d, *J* = 5.4, 1 H, HC(3)), 3.90 (d, *J* = 5.4, 1 H, HC(2)), 3.42 (s, 3 H, HC(10)), 2.94 (bs, 1 H, OH), 1.33 (s, 9 H, HC(9))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

169.2 (C(1)), 139.4 (C(4)), 128.1 (C(6)), 127.8 (C(7)), 126.7 (C(5)), 84.5 (C(2)), 82.1 (C(8)), 73.9 (C(3)), 58.6 (C(10)), 27.8 (C(9))

IR: (neat)

3474 (br), 3064 (m), 3032 (m), 2980 (s), 2934 (m), 2831 (m), 1737 (w), 1694 (s), 1599 (m), 1582 (w), 1495 (m), 1454 (s), 1394 (s), 1369 (s), 1251 (s), 1157 (s), 1124 (s), 1065 (m), 1026 (m), 990 (m), 919 (m), 841 (m), 762 (m), 700 (s)

MS: (ESI)

275.1 ($M^+$+Na, 100), 276.1 (11)

HRMS: calcd for $C_{14}H_{20}O_4Na$: 275.1259, found 275.1271

TLC: $R_f$ 0.25 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

$R_f$ 0.51 (pentane/$Et_2O$, 1/1) [UV(254)/$KMnO_4$]

SFC: (2*S*,3*S*)-**6fa**, $t_R$ 2.22 min (81.3%); (2*R*,3*S*)-**6fa**, $t_R$ 2.57 min (18.7%)

(Chiralcel OD , 125 bar, 40 °C, 6.0% MeOH in $CO_2$, 3.0 mL/min, 220 nm)

Data for (2*R*,3*S*)-**6fa**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.41-7.39 (m, 2 H, HC(5)), 7.35-7.32 (m, 2 H, HC(6)), 7.30-7.27 (m, 1 H, HC(7)), 4.82 (d, *J* = 6.5, 1 H, HC(3)), 3.79 (d, *J* = 6.5, 1 H, HC(2)), 3.44 (s, 3 H, HC(10)), 2.94 (bs, 1 H, OH), 1.30 (s, 9 H, HC(9))

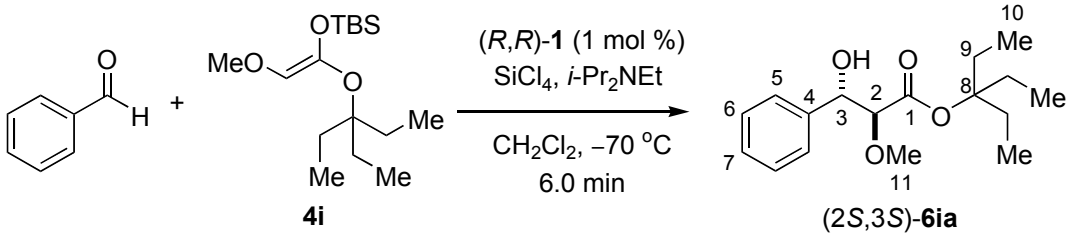
SFC: (2*S*,3*R*)-**6fa**, $t_R$ 3.31 min (9.6%); (2*R*,3*S*)-**6fa**, $t_R$ 3.85 min (90.4%)

(Chiralpak AS , 125 bar, 40 °C, 2.0% MeOH in $CO_2$, 2.0 mL/min, 220 nm)

TLC: $R_f$ 0.25 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

$R_f$ 0.37 (pentane/$Et_2O$, 1/1) [UV(254)/$KMnO_4$]

**Preparation of (2*S*,3*S*)-3-Hydroxy-2-methoxy-3-phenylpropanoic Acid 1,1-Diethylpropyl Ester ((2*S*,3*S*)-6ia) (Table 3, entry 4)**




Following General Procedure 6, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined with diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), benzaldehyde (101.6 μL, 1.0 mmol), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4i** (363 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 5/1) on silica gel (20 g), **6ia** (273 mg, 93%) as a colorless oil. The *syn*/*anti* ratio was determined to be 2/98 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*S*,3*S*)-**6ia**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.42-7.39 (m, 2 H, HC(5)), 7.34-7.31 (m, 2 H, HC(6)), 7.29-7.25 (m, 1 H, HC(7)), 4.93 (d, *J* = 5.9, 1 H, HC(3)), 3.86 (d, *J* = 5.9, 1 H, HC(2)), 3.38 (s, 3 H, HC(11)), 2.95 (bs, 1 H, OH), 1.81 (dq, *J* = 14.4, 7.6, 3 H, HC(9)), 1.74 (dq, *J* = 14.4, 7.6, 3 H, HC(9)), 0.74 (dd, *J* = 7.6, 7.6, 9 H, HC(10))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

169.5 (C(1)), 139.8 (C(4)), 128.1 (C(6)), 127.8 (C(7)), 126.9 (C(5)), 90.7 (C(8)), 84.8 (C(2)), 74.0 (C(3)), 58.9 (C(11)), 26.8 (C(9)), 7.6 (C(10))

IR: (neat)

3484 (br), 3064 (m), 3032 (m), 2973 (s), 2943 (s), 2884 (m), 2830 (m), 1736 (s), 1599 (m), 1581 (m), 1495 (m), 1457 (s), 1383 (m), 1357 (m), 1284 (s), 1196 (s),

1124 (s), 1064 (m), 1002 (m), 983 (m), 928 (m), 880 (m), 753 (m), 699 (s)

MS: (ESI)

130.2 (8), 219.1 (100), 220.1 (10), 317.2 ($M^+$+Na, 96), 318.2 (14)

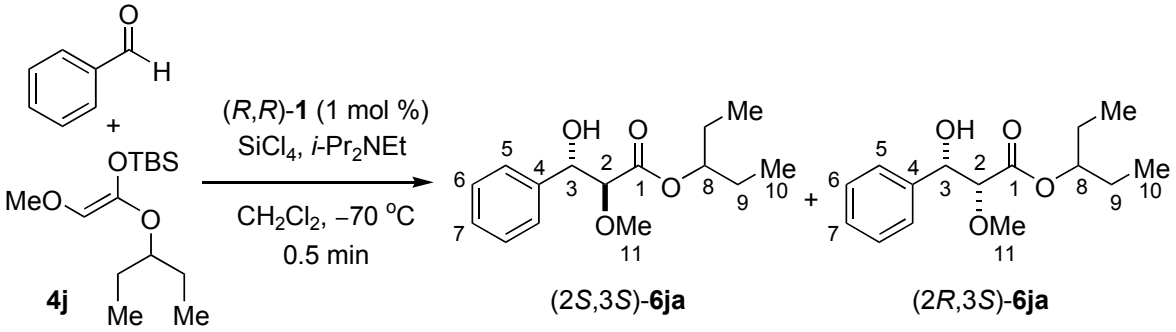
HRMS: calcd for $C_{17}H_{26}O_4Na$: 317.1729, found 317.1732

TLC: $R_f$ 0.35 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

SFC: (2*R*,3*R*)-**6ia**, $t_R$ 9.01 min (7.2%); (2*S*,3*S*)-**6ia**, $t_R$ 9.87 min (92.8%)

(Chiralpak AD, 125 bar, 40 °C, 2.0% MeOH in $CO_2$, 2.5 mL/min, 220 nm)

**Preparation of (2*S*,3*S*)-3-Hydroxy-2-methoxy-3-phenylpropanoic Acid 1-Ethylpropyl Ester ((2*S*,3*S*)-6ja) and (2*R*,3*S*)-3-Hydroxy-2-methoxy-3-phenylpropanoic Acid 1-Ethylpropyl Ester ((2*R*,3*S*)-6ja) (Table 3, entry 5)**




Following General Procedure 6, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined with diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), benzaldehyde (101.6 μL, 1.0 mmol), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4j** (329 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 5/1) on silica gel (20 g), **6ja** (259 mg, 97%) as a colorless oil. The *syn*/*anti* ratio was determined to be 8/92 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*S*,3*S*)-**6ja**:

$^{1}$H NMR: (500 MHz, $CDCl_3$)

7.40-7.37 (m, 2 H, HC(5)), 7.34-7.29 (m, 2 H, HC(6)), 7.29-7.25 (m, 1 H, HC(7)), 4.96 (d, *J* = 5.9, 1 H, HC(3)), 4.79 (dddd, *J* = 6.2, 6.2, 6.2, 6.2, 1 H, HC(8)), 3.96 (d, *J* = 5.9, 1 H, HC(2)), 3.38 (s, 3 H, HC(11)), 2.97 (bs, 1 H, OH), 1.54-1.44 (m, 4 H, HC(9)), 0.81 (dd, *J* = 7.5, 7.5, 3 H, HC(10)), 0.77 (dd, *J* = 7.5, 7.5, 3 H, HC(10))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

170.4 (C(1)), 139.6 (C(4)), 128.2 (C(6)), 127.9 (C(7)), 126.7 (C(5)), 84.6 (C(2)), 78.0 (C(8)), 74.0 (C(3)), 58.7 (C(11)), 26.1 (C(9)), 26.0 (C(9)), 9.39 (C(10)), 9.36 (C(10))

IR: (neat)

3484 (br), 3064 (m), 3033 (m), 2970 (s), 2938 (s), 2881 (m), 2830 (m), 1736 (s), 1600 (m), 1582 (w), 1495 (m), 1456 (s), 1386 (m), 1344 (m), 1281 (s), 1194 (s), 1122 (s), 1052 (s), 1027 (s), 984 (s), 911 (m), 764 (m), 751 (m), 700 (s), 612 (m)

MS: (ESI)

289.1 ($M^+$+Na, 100), 290.1 (13)

HRMS: calcd for $C_{15}H_{22}O_4Na$: 289.1416, found 289.1414

TLC: $R_f$ 0.28 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

SFC: (2*S*,3*S*)-**6ja**, $t_R$ 8.11 min (82.9%); (2*R*,3*R*)-**6ja**, $t_R$ 10.32 min (17.1%)

(Chiralpak AS, 125 bar, 40 $^{o}$C, 1.0% MeOH in $CO_2$, 2.5 mL/min, 220 nm)

Data for (2*R*,3*S*)-**6ja**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.40-7.37 (m, 2 H, HC(5)), 7.34-7.29 (m, 2 H, HC(6)), 7.29-7.25 (m, 1 H, HC(7)), 4.89 (d, *J* = 6.1, 1 H, HC(3)), 4.74 (dddd, *J* = 6.1, 6.1, 6.1, 6.1, 1 H, HC(8)), 3.89 (d, *J* = 6.1, 1 H, HC(2)), 3.42 (s, 3 H, HC(11)), 2.97 (bs, 1 H, OH), 1.58-1.55 (m, 2 H, HC(9)), 1.38-1.33 (m, 2 H, HC(9)), 0.83 (dd, *J* = 7.5, 7.5, 3 H, HC(10)), 0.65 (dd, *J* = 7.5, 7.5, 3 H, HC(10))

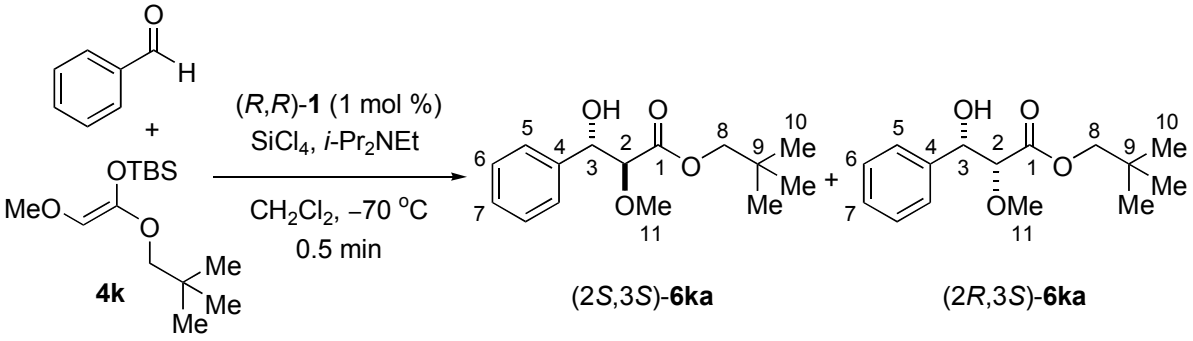
$^{13}$C NMR: (126 MHz, $CDCl_3$)

170.1 (C(1)), 138.9 (C(4)), 128.3 (C(6)), 127.9 (C(7)), 126.9 (C(5)), 85.3 (C(2)), 77.9 (C(8)), 74.7 (C(3)), 58.6 (C(11)), 26.1 (C(9)), 25.9 (C(9)), 9.5 (C(10)), 9.1 (C(10))

SFC: (2*S*,3*R*)-**6ja**, $t_R$ 4.76 min (37.8%); (2*R*,3*S*)-**6ja**, $t_R$ 7.31 min (62.2%)

(Chiralpak AS, 125 bar, 40 °C, 1.0% MeOH in $CO_2$, 2.5 mL/min, 220 nm)

**Preparation of (2*S*,3*S*)-3-Hydroxy-2-methoxy-3-phenylpropanoic Acid 2,2-Dimethylpropyl Ester ((2*S*,3*S*)-6ka) and (2*R*,3*S*)-3-Hydroxy-2-methoxy-3-phenylpropanoic Acid 2,2-Dimethylpropyl Ester ((2*R*,3*S*)-6ka) (Table 3, entry 6)**




Following General Procedure 6, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined with diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), benzaldehyde (101.6 μL, 1.0 mmol),

$SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4k** (329 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 5/1) on silica gel (20 g), **6ka** (261 mg, 98%) as a colorless oil. The *syn*/*anti* ratio was determined to be 17/83 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*S*,3*S*)-**6ka**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.38-7.35 (m, 2 H, HC(5)), 7.34-7.31 (m, 2 H, HC(6)), 7.30-7.25 (m, 1 H, HC(7)), 5.00 (d, $J$ = 5.6, 1 H, HC(3)), 4.02 (d, $J$ = 5.6, 1 H, HC(2)), 3.82 (d, $J$ = 10.5, 1 H, HC(8)), 3.67 (d, $J$ = 10.5, 1 H, HC(8)), 3.40 (s, 3 H, HC(11)), 2.93 (bs, 1 H, OH), 0.84 (s, 9 H, HC(10))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

170.5 (C(1)), 139.3 (C(4)), 128.2 (C(6)), 128.0 (C(7)), 126.6 (C(5)), 84.4 (C(2)), 74.4 (C(8)), 73.9 (C(3)), 58.8 (C(11)), 31.0 (C(9)), 26.3 (C(10))

IR: (neat)

3485 (br), 3064 (m), 3033 (m), 2959 (s), 2906 (s), 2872 (m), 2831 (m), 1740 (s), 1600 (m), 1495 (m), 1479 (m), 1456 (s), 1368 (s), 1341 (m), 1281 (s), 1257 (s), 1191 (s), 1124 (s), 1061 (s), 1006 (s), 937 (m), 761 (m), 743 (m), 700 (s)

MS: (ESI)

133.1 (7), 273.2 (12), 289.1 ($M^+$+Na, 100), 290.1 (16), 305.1 (8), 362.2 (9)

HRMS: calcd for $C_{15}H_{22}O_4Na$: 289.1416, found 289.1424

TLC: $R_f$ 0.33 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

SFC: (2*S*,3*S*)-**6ka**, $t_R$ 17.64 min (79.7%); (2*R*,3*R*)-**6ka**, $t_R$ 23.53 min (20.3%)

(Chiralpak AS, 125 bar, 40 °C, 1.0% MeOH in $CO_2$, 1.0 mL/min, 220 nm)

Data for (2*R*,3*S*)-**6ka**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.38-7.35 (m, 2 H, HC(5)), 7.34-7.31 (m, 2 H, HC(6)), 7.30-7.25 (m, 1 H, HC(7)), 4.90 (d, $J$ = 6.0, 1 H, HC(3)), 3.92 (d, $J$ = 6.0, 1 H, HC(2)), 3.76 (d, $J$ = 10.5, 1 H, HC(8)), 3.64 (d, $J$ = 10.5, 1 H, HC(8)), 3.43 (s, 3 H, HC(11)), 2.93 (bs, 1 H, OH), 0.81 (s, 9 H, HC(10))

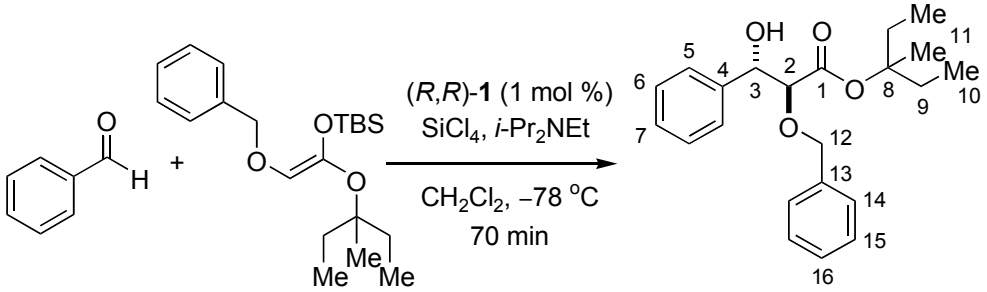
$^{13}$C NMR: (126 MHz, $CDCl_3$)

170.4 (C(1)), 138.9 (C(4)), 128.3 (C(6)), 128.2 (C(7)), 126.6 (C(5)), 85.2 (C(2)), 74.8 (C(3)), 74.4 (C(8)), 58.7 (C(11)), 31.0 (C(9)), 26.2 (C(10))

SFC: (2*S*,3*R*)-**6ka**, $t_R$ 11.18 min (15.3%); (2*R*,3*S*)-**6ka**, $t_R$ 16.53 min (84.7%)

(Chiralpak AS, 125 bar, 40 °C, 1.0% MeOH in $CO_2$, 2.5 mL/min, 220 nm)

**Preparation of (2*S*,3*S*)-3-Hydroxy-3-phenyl-2-(phenylmethoxy)propanoic Acid 1-Ethyl-1-methylpropyl Ester ((2*S*, 3*S*)-6na) (Scheme 8)**




**4n** (2*S*,3*S*)-**6na**

Following General Procedure 6, (*R*,*R*)-**1** (2.1 mg, 0.025 mmol, 0.01 equiv) was combined with diisopropylethylamine (4.4 μL, 0.025 mmol, 0.1 equiv), benzaldehyde (25.4 μL, 0.25 mmol), $SiCl_4$ (31.5 μL, 0.275 mmol, 1.1 equiv), and **4n** (109.4 mg, 0.3 mmol, 1.2 equiv) to yield, after column chromatography (18 mm diam., hexane/EtOAc, 5/1 to 1/1) on silica gel (5 g), **6na** (30 mg, 34%) as a colorless oil. The *syn*/*anti* ratio was determined to be 5/95 by $^1$H NMR

(500 MHz) analysis of the crude reaction mixture.

Data for (2*S*,3*S*)-**6na**:

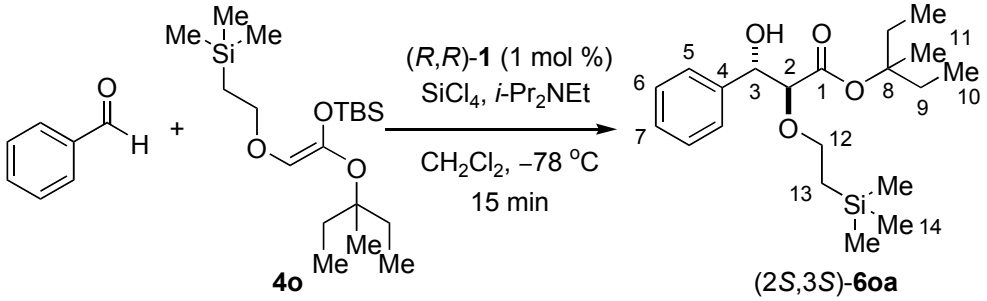
$^1$H NMR: (500 MHz, $CDCl_3$)

7.40-7.38 (m, 2 H, Aryl), 7.34-7.31 (m, 2 H, Aryl), 7.30-7.27 (m, 4 H, Aryl), 7.18-7.16 (m, 2 H, Aryl), 4.97 (dd, *J* = 5.6, 5.6, 1 H, HC(3)), 4.71 (d, *J* = 11.5, 1 H, HC(12)), 4.36 (d, *J* = 11.5, 1 H, HC(12)), 4.04 (d, *J* = 6.1, 1 H, HC(2)), 3.02 (d, *J* = 4.8, 1 H, OH), 1.84-1.72 (m, 3 H, HC(9)), 1.65 (dq, *J* = 14.2, 7.5, 1 H, HC(9)), 1.32 (s, 3 H, HC(11)), 0.79 (dd, *J* = 7.5, 7.5, 3 H, HC(10)), 0.77 (dd, *J* = 7.6, 7.6, 3 H, HC(10))

SFC: (2*R*,3*R*)-**6na**, $t_R$ 8.93 min (42.7%); (2*S*,3*S*)-**6na**, $t_R$ 10.14 min (57.3%)

(Chiralpak AD, 125 bar, 40 °C, 5.0% MeOH in $CO_2$, 2.0 mL/min, 220 nm)

**Preparation of (2*S*,3*S*)-3-Hydroxy-3-phenyl-2-(phenylmethoxy)propanoic Acid 1-Ethyl-1-methylpropyl Ester ((2*S*, 3*S*)-6oa) (Scheme 8)**




Following General Procedure 6, (*R*,*R*)-**1** (2.1 mg, 0.025 mmol, 0.01 equiv) was combined with diisopropylethylamine (4.4 μL, 0.025 mmol, 0.1 equiv), benzaldehyde (25.4 μL, 0.25 mmol), $SiCl_4$ (31.5 μL, 0.275 mmol, 1.1 equiv), and **4o** (140.5 mg, 0.375 mmol, 1.5 equiv) to yield, after column chromatography (18 mm diam., hexane/EtOAc, 5/1) on silica gel (5 g), **6oa** (23 mg, 25%) as a colorless oil. The *syn*/*anti* ratio was determined to be 4/96 by $^1$H NMR

(400 MHz) analysis of the crude reaction mixture.

Data for (2*S*,3*S*)-**6oa**:

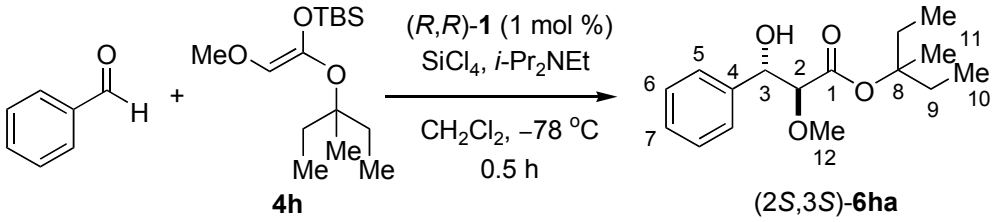
$^1$H NMR: (500 MHz, $CDCl_3$)

7.42-7.40 (m, 2 H, HC(5)), 7.34-7.30 (m, 2 H, HC(6)), 7.28-7.25 (m, 1 H, HC(7)), 4.94 (dd, *J* = 4.9, 4.9, 1 H, HC(3)), 3.96 (d, *J* = 5.6, 1 H, HC(2)), 3.73-3.68 (m, 1 H, HC(12)), 3.42-3.36 (m, 1 H, HC(12)), 3.05 (d, *J* = 4.6, 1 H, OH), 1.79-1.72 (m, 3 H, HC(9)), 1.59 (dq, *J* = 14.4, 7.4, 1 H, HC(9)), 1.28 (s, 3 H, HC(11)), 0.92 (dd, *J* = 8.4, 8.4, 2 H, HC(13)), 0.77 (dd, *J* = 7.6, 7.6, 3 H, HC(10)), 0.74 (dd, *J* = 7.5, 7.5, 3 H, HC(10)), –0.03 (s, 9 H, HC(14))

SFC: (2*S*,3*S*)-**6oa**, $t_R$ 5.83 min (60.7%); (2*R*,3*R*)-**6oa**, $t_R$ 6.83 min (39.3%)

(Chiralpak AD, 125 bar, 40 °C, 2.0% MeOH in $CO_2$, 2.5 mL/min, 220 nm)

**Preparation of (2*S*,3*S*)-3-Hydroxy-2-methoxy-3-phenylpropanoic Acid 1-Ethyl-1-methylpropyl Ester ((2*S*, 3*S*)-6ha) (Table 4, entry 1)**




Following General Procedure 7, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined with diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), benzaldehyde (101.6 μL, 1.0 mmol), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4h** (346.2 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (30 mm diam., hexane/EtOAc, 5/1) on silica gel (30 g), **6ha** (255 mg, 91%) as a colorless oil. The *syn*/*anti* ratio was determined to be >1/99 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*S*,3*S*)-**6ha**:

$^{1}$H NMR: (500 MHz, $CDCl_3$)

7.41-7.40 (m, 2 H, HC(5)), 7.35-7.32 (m, 2 H, HC(6)), 7.29-7.26 (m, 1 H, HC(7)), 4.96 (d, $J$ = 5.6, 1 H, HC(3)), 3.89 (d, $J$ = 5.6, 1 H, HC(2)), 3.40 (s, 3 H, HC(12)), 2.96 (bs, 1 H, OH), 1.81-1.73 (m, 3 H, HC(9)), 1.60 (dq, $J$ = 13.9, 7.5, 1 H, HC(9)), 1.30 (s, 3 H, HC(11)), 0.77 (dd, $J$ = 7.5, 7.5, 3 H, HC(10)), 0.74 (dd, $J$ = 7.5, 7.5, 3 H, HC(10))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

169.4 (C(1)), 139.6 (C(4)), 128.2 (C(6)), 127.9 (C(7)), 126.8 (C(5)), 87.7 (C(8)), 84.6 (C(2)), 74.0 (C(3)), 58.8 (C(12)), 30.3 (C(9)), 30.2 (C(9)), 22.6 (C(11)), 7.9 (C(10)), 7.8 (C(10))

IR: (neat)

3474 (br), 3064 (w), 3033 (w), 2976 (m), 2941 (m), 2884 (m), 2829 (w), 1734 (s), 1495 (w), 1456 (m), 1376 (m), 1266 (m), 1196 (s), 1151 (m), 1124 (s), 1065 (m), 980 (m), 846 (m), 752 (m), 700 (s), 613 (m)

MS: (ESI)

179.1 (100), 180.1 (3), 197.1 (18), 298.2 (36), 303.2 ($M^+$+Na, 7)

HRMS: calcd for $C_{16}H_{24}O_4Na$: 303.1572, found 303.1559

TLC: $R_f$ 0.27 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

SFC: (2*S*,3*S*)-**6ha**, $t_R$ 10.81 min (95.1%); (2*R*,3*R*)-**6ha**, $t_R$ 11.40 min (4.9%)

(Chiralpak AD, 125 bar, 40 $^{o}$C, 2.0% MeOH in $CO_2$, 2.0 mL/min, 220 nm)

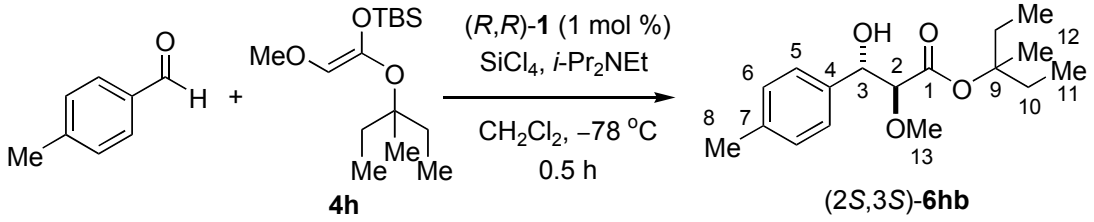
Opt. Rot.: $[\alpha]_D^{24}$ –5.6 (c = 4.0, EtOH)

Analysis: $C_{16}H_{24}O_4$ (280.36)

Calcd: C, 68.54%; H, 8.63%

Found: C, 68.35%; H, 8.72%

**Preparation of (2*S*,3*S*)-3-Hydroxy-2-methoxy-3-(4-methylphenyl)propanoic Acid 1-Ethyl-1-methylpropyl Ester ((2*S*, 3*S*)-6hb) (Table 4, entry 2)**




Following General Procedure 7, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined with diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), 4-tolualdehyde (118 μL, 1.0 mmol), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4h** (346.2 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 5/1) on silica gel (15 g), **6hb** (272 mg, 92%) as a colorless oil. The *syn*/*anti* ratio was determined to be 1/99 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*S*,3*S*)-**6hb**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.29-7.28 (m, 2 H, HC(5)), 7.14-7.13 (m, 2 H, HC(6)), 4.90 (d, *J* = 5.6, 1 H, HC(3)), 3.86 (d, *J* = 5.6, 1 H, HC(2)), 3.39 (s, 3 H, HC(13)), 2.88 (bs, 1 H, OH), 2.33 (s, 3 H, HC(8)), 1.80 (dq, *J* = 14.1, 7.5, 1 H, HC(10)), 1.78 (m, 2 H, HC(10)), 1.61 (dq, *J* = 14.1, 7.5, 1 H, HC(10)), 1.32 (s, 3 H, HC(12)), 0.79 (dd, *J* = 7.5, 7.5, 3 H, HC(11)), 0.77 (dd, *J* = 7.5, 7.5, 3 H, HC(11))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

169.5 (C(1)), 137.5 (C(7)), 136.7 (C(4)), 128.8 (C(6)), 126.7 (C(5)), 87.6 (C(9)), 84.7 (C(2)), 73.9 (C(3)), 58.7 (C(13)), 30.30 (C(10)), 30.28 (C(10)), 22.6 (C(12)), 21.1 (C(8)), 7.9 (C(11)), 7.8 (C(11))

IR: (neat)

3478 (br), 2976 (s), 2942 (s), 2884 (s), 2830 (m), 1738 (s), 1732 (s), 1516 (m), 1462 (s), 1382 (s), 1359 (s), 1272 (s), 1197 (s), 1151 (s), 1126 (s), 1072 (s), 1042 (s), 1006 (s), 981 (s), 901 (m), 847 (s), 821 (s), 806 (m), 766 (m), 732 (s)

MS: (ESI)

193.1 (7), 233.1 (100), 234.1 (8), 317.2 ($M^+$+Na, 98), 318.2 (10)

HRMS: calcd for $C_{17}H_{26}O_4Na$: 317.1729, found 317.1729

TLC: $R_f$ 0.35 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

SFC: (2*R*,3*R*)-**6hb**, $t_R$ 5.19 min (3.9%); (2*S*,3*S*)-**6hb**, $t_R$ 5.61 min (96.1%)

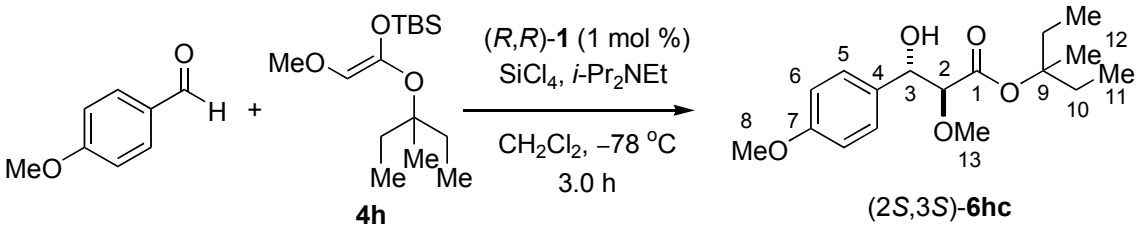
(Chiralpak AD, 125 bar, 40 $^oC$, 3.0% MeOH in $CO_2$, 3.0 mL/min, 220 nm)

Opt. Rot.: $[\alpha]_D^{24}$ –1.7 (c = 6.5, EtOH)

Analysis: $C_{17}H_{26}O_4$ (294.39)

| | | |
|---|---|---|
| Calcd: | C, 69.36%; | H, 8.90% |
| Found: | C, 69.30%; | H, 8.91% |

**Preparation of (2*S*,3*S*)-3-Hydroxy-2-methoxy-3-(4-methoxyphenyl)propanoic Acid 1-Ethyl-1-methylpropyl Ester ((2*S*,3*S*)-6hc) (Table 4, entry 3)**




Following General Procedure 7, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined with diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), 4-anisaldehyde (121.7 μL, 1.0 mmol), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4h** (346.2 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 5/1 to 1/1) on silica gel (15 g), **6hc** (289 mg, 93%) as a colorless oil. The *syn*/*anti* ratio was determined to be >1/99 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*S*,3*S*)-**6hc**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.35-7.32 (m, 2 H, HC(5)), 6.88-6.85 (m, 2 H, HC(6)), 4.89 (dd, *J* = 5.6, 5.6, 1 H, HC(3)), 3.86 (d, *J* = 5.8, 1 H, HC(2)), 3.80 (s, 3 H, HC(8)), 3.40 (s, 3 H, HC(13)), 2.86 (d, *J* = 5.4, 1 H, OH), 1.80 (dq, *J* = 14.1, 7.5, 1 H, HC(10)), 1.78 (m, 2 H, HC(10)), 1.63 (dq, *J* = 14.1, 7.5, 1 H, HC(10)), 1.32 (s, 3 H, HC(12)), 0.79 (dd, *J* = 7.5, 7.5, 3 H, HC(11)), 0.77 (dd, *J* = 7.5, 7.5, 3 H, HC(11))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

169.6 (C(1)), 159.3 (C(7)), 131.9 (C(4)), 128.1 (C(5)), 113.5 (C(6)), 87.6 (C(9)), 84.7 (C(2)), 73.6 (C(3)), 58.8 (C(13)), 55.2 (C(8)), 30.3 (C(10)), 30.2 (C(10)), 22.6 (C(12)), 7.9 (C(11)), 7.8 (C(11))

IR: (neat)

3482 (br), 2975 (m), 2940 (m), 2884 (m), 2835 (w), 1733 (s), 1613 (m), 1587 (w), 1514 (s), 1462 (m), 1376 (m), 1355 (w), 1302 (m), 1250 (s), 1197 (m), 1180 (m), 1125 (s), 1071 (m), 1034 (m), 1010 (w), 981 (m), 899 (w), 835 (m), 767 (w), 734 (w)

MS: (ESI)

98.5 (4), 165.1 (5), 209.1 (100), 210.1 (6), 249.1 (20), 265.0 (3), 328.2 (4), 333.2 ($M^+$+Na, 39), 349.1 (7)

HRMS: calcd for $C_{17}H_{26}O_5Na$: 333.1678, found 333.1669

TLC: $R_f$ 0.36 (hexane/EtOAc, 3/1) [UV(254)/$KMnO_4$]

SFC: (2*S*,3*S*)-**6hc**, $t_R$ 10.21 min (98.3%); (2*R*,3*R*)-**6hc**, $t_R$ 11.65 min (1.7%)

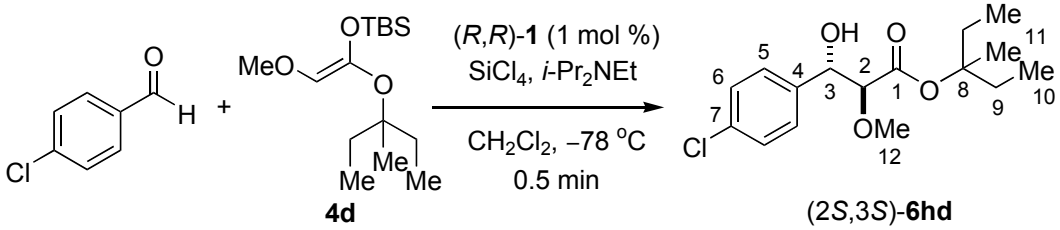
(Chiralcel OD, 125 bar, 40 °C, 2.0% MeOH in $CO_2$, 3.0 mL/min, 220 nm)

Opt. Rot.: $[\alpha]_D^{24}$ –2.8 (c = 8.4, EtOH)

Analysis: $C_{17}H_{26}O_5$ (310.39)

| | | |
|---|---|---|
| Calcd: | C, 65.78%; | H, 8.44% |
| Found: | C, 65.39%; | H, 8.55% |

**Preparation of (2*S*,3*S*)-3-(4-Chlorophenyl)-3-hydroxy-2-methoxypropanoic Acid 1-Ethyl-1-methylpropyl Ester ((2*S*,3*S*)-6hd) (Table 4, entry 4)**




Following General Procedure 7, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined

with diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), 4-chlorobenzaldehyde (140.6 mg, 1.0 mmol), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4h** (346.2 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 5/1 to 1/1) on silica gel (15 g), **6hd** (290 mg, 92%) as a colorless oil. The *syn*/*anti* ratio was determined to be 1/99 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*S*,3*S*)-**6hd**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.35-7.32 (m, 2 H, HC(5)), 7.30-7.28 (m, 2 H, HC(6)), 4.91 (d, *J* = 5.6, 1 H, HC(3)), 3.82 (d, *J* = 5.6, 1 H, HC(2)), 3.38 (s, 3 H, HC(12)), 3.10 (bs, 1 H, OH), 1.78 (dq, *J* = 14.1, 7.5, 1 H, HC(9)), 1.76 (m, 2 H, HC(9)), 1.61 (dq, *J* = 14.1, 7.5, 1 H, HC(9)), 1.30 (s, 3 H, HC(11)), 0.77 (dd, *J* = 7.5, 7.5, 3 H, HC(10)), 0.76 (dd, *J* = 7.5, 7.5, 3 H, HC(10))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

169.2 (C(1)), 138.2 (C(4)), 133.6 (C(7)), 128.24 (C(5) or C(6)), 128.22 (C(5) or C(6)), 87.9 (C(8)), 84.4 (C(2)), 73.3 (C(3)), 58.8 (C(12)), 30.3 (C(9)), 30.2 (C(9)), 22.6 (C(11)), 7.84 (C(10)), 7.81 (C(10))

IR: (neat)

3462 (br), 2976 (m), 2941 (m), 2884 (m), 2830 (w), 1731 (s), 1598 (w), 1492 (m), 1461 (m), 1376 (m), 1361 (m), 1271 (m), 1193 (s), 1150 (m), 1124 (s), 1089 (s), 1015 (m), 981 (m), 900 (w), 838 (m), 743 (m)

MS: (ESI)

196.0 (4), 213.0 (25), 215.0 (6), 235.0 (15), 237.0 (4), 253.0 (53), 255.0 (12), 321.1 (4), 330.9 (18), 337.1 ($M^+$+Na, 100), 338.1 (11), 339.1 (29), 375.0 (9)

HRMS: calcd for $C_{16}H_{23}ClO_4Na$: 337.1183, found 337.1169

TLC: $R_f$ 0.45 (hexane/EtOAc, 3/1) [UV(254)/$KMnO_4$]

SFC: (2*S*,3*S*)-**6hd**, $t_R$ 5.37 min (95.1%); (2*R*,3*R*)-**6hd**, $t_R$ 6.08 min (4.9%)

(Chiralcel OD, 125 bar, 40 °C, 3.0% MeOH in $CO_2$, 3.0 mL/min, 220 nm)

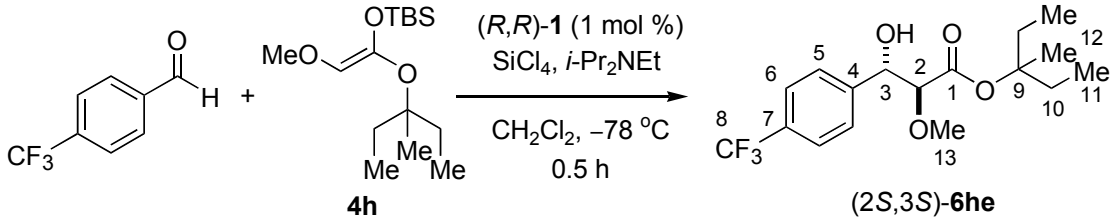
Opt. Rot.: $[\alpha]_D^{24}$ –0.5 (c = 7.0, EtOH)

Analysis: $C_{16}H_{23}ClO_4$ (314.80)

Calcd: C, 61.04%; H, 7.36%

Found: C, 61.20%; H, 7.45%

**Preparation of (2*S*,3*S*)-3-Hydroxy-2-methoxy-3-[4-(trifluoromethyl)phenyl]propanoic Acid 1-Ethyl-1-methylpropyl Ester ((2*S*,3*S*)-6he) (Table 4, entry 5)**




Following General Procedure 7, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined with diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), 4-trifluoromethylbenzaldehyde (136.6 μL, 1.0 mmol), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4h** (346.2 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 5/1) on silica gel (15 g), **6he** (334 mg, 96%) as a colorless oil. The *syn*/*anti* ratio was determined to be >1/99 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*S*,3*S*)-**6he**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.60 (m, 2 H, HC(7)), 7.54 (m, 2 H, HC(6)), 5.03 (d, *J* = 5.5, 1 H, HC(3)), 3.87 (d, *J* = 5.5, 1 H, HC(2)), 3.42 (s, 3 H, HC(13)), 3.10 (bs, 1 H, OH), 1.76 (dq, *J* =

14.0, 7.5, 1 H, HC(10)), 1.74 (q, *J* = 7.5, 2 H, HC(10)), 1.58 (dq, *J* = 14.0, 7.5, 1 H, HC(10)), 1.29 (s, 3 H, HC(12)), 0.75 (dd, *J* = 7.5, 7.5, 3 H, HC(11)), 0.72 (dd, *J* =7.5, 7.5, 3 H, HC(11))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

169.0 (C(1)), 143.6 (C(4)), 130.0 (q, *J* = 32.2, C(7)), 127.1 (C(5)), 125.0 (q, *J* = 3.9, C(6)), 124.1 (q, *J* = 272.8, C(8)), 88.1 (C(9)), 84.3 (C(2)), 73.5 (C(3)), 58.8 (C(13)), 30.3 (C(10)), 30.2 (C(10)), 22.6 (C(12)), 7.8 (C(11)), 7.8 (C(11))

$^{19}$F NMR: (376 MHz, $CDCl_3$)

−63.06 (FC(8))

IR: (neat)

3461 (br), 2798 (m), 2944 (m), 2886 (m), 2883 (w), 1729 (s), 1621 (m), 1462 (m), 1417 (m), 1376 (m), 1327 (s), 1265 (m), 1196 (m), 1165 (s), 1126 (s), 1088 (m), 1068 (s), 1018 (m), 982 (m), 909 (w), 846 (m), 798 (w), 764 (w), 734 (m)

MS: (ESI)

119.1 (35), 131.1 (21), 149.0 (9), 163.1 (7), 191.0 (5), 247.1 (100), 248.1 (8), 265.1 (77), 266.1 (6), 282.1 (21), 287.0 (37), 365.0 (17), 371.1 ($M^+$+Na, 52), 397.0 (5), 405.1 (5)

HRMS: calcd for $C_{17}H_{23}F_3O_4Na$: 371.1446, found 371.1437

TLC: $R_f$ 0.30 (hexane/EtOAc, 4/1) [UV(254)/$KMnO_4$]

SFC: (2*S*,3*S*)-**6he**, $t_R$ 4.12 min (98.3%); (2*R*,3*R*)-**6he**, $t_R$ 4.68 min (1.7%)

(Chiralcel OD, 125 bar, 40 °C, 2.0% MeOH in $CO_2$, 3.0 mL/min, 220 nm)

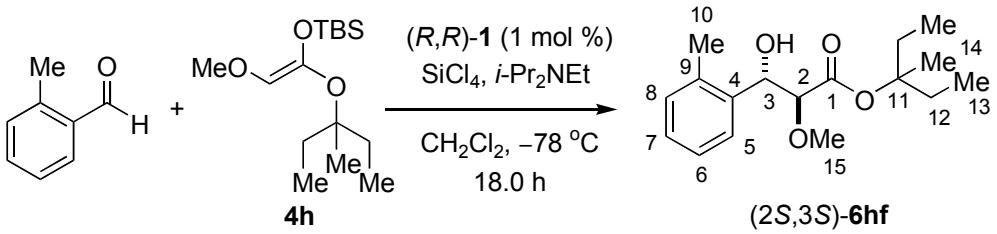
Opt. Rot.: $[\alpha]_D^{24}$ −1.4 (c = 0.75, EtOH)

Analysis: $C_{17}H_{23}F_3O_4$ (348.36)

Calcd: C, 58.61%; H, 6.65%

Found: C, 58.58%; H, 6.69%

**Preparation of (2*S*,3*S*)-3-Hydroxy-2-methoxy-3-(2-methylphenyl)propanoic Acid 1-Ethyl-1-methylpropyl Ester ((2*S*,3*S*)-6hf) (Table 4, entry 6)**




Following General Procedure 7, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined with diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), 2-tolualdehyde (116 μL, 1.0 mmol), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4h** (346.2 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 5/1 to 2/1) on silica gel (15 g), **6hf** (269 mg, 91%) as a colorless oil. The *syn*/*anti* ratio was determined to be 2/98 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*S*,3*S*)-**6hf**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.55-7.53 (m, 1 H, HC(5)), 7.22-7.16 (m, 2 H, HC(6), HC(7)), 7.14-7.12 (m, 1 H, HC(8)), 5.19 (d, *J* = 6.0, 1 H, HC(3)), 3.88 (d, *J* = 6.0, 1 H, HC(2)), 3.38 (s, 3 H, HC(15)), 2.78 (bs, 1 H, OH), 2.37 (s, 3 H, HC(10)), 1.78 (q, *J* = 7.5, 2 H, HC(12)), 1.77 (dq, *J* = 14.0, 7.5, 1 H, HC(12)), 1.59 (dq, *J* = 14.0, 7.5, 1 H, HC(12)), 1.32 (s, 3 H, HC(14)), 0.78 (dd, *J* = 7.5, 7.5, 3 H, HC(13)), 0.73 (dd, *J* = 7.5, 7.5, 3 H, HC(13))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

169.7 (C(1)), 137.9 (C(4)), 135.7 (C(9)), 130.2 (C(8)), 127.7 (C(7)), 126.3 (C(5)), 126.0 (C(6)), 87.7 (C(11)), 84.0 (C(2)), 70.5 (C(3)), 58.7 (C(15)), 30.29 (C(12)), 30.27 (C(12)), 22.6 (C(14)), 19.3 (C(10)), 7.9 (C(13)), 7.8 (C(13))

IR: (neat)

3477 (br), 3024 (w), 2975 (s), 2941 (m), 2884 (m), 2829 (w), 1732 (s), 1491 (w), 1462 (m), 1377 (m), 1272 (m), 1199 (s), 1129 (s), 1109 (s), 1072 (m), 1048 (m), 1006 (m), 981 (m), 899 (w), 846 (m), 803 (w), 756 (m), 730 (m)

MS: (ESI)

233.1 (100), 234.1 (8), 317.2 ($M^+$+Na, 81), 318.2 (8), 338.3 (12)

HRMS: calcd for $C_{17}H_{26}O_4Na$: 317.1729, found 317.1719

TLC: $R_f$ 0.45 (hexane/EtOAc, 4/1) [UV(254)/$KMnO_4$]

SFC: (2*R*,3*R*)-**6hf**, $t_R$ 6.09 min (6.9%); (2*S*,3*S*)-**6hf**, $t_R$ 6.60 min (93.1%)

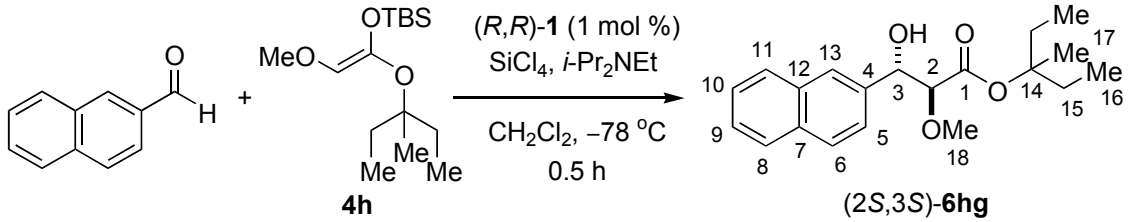
(Chiralpak AD, 125 bar, 40 $^oC$, 2.0% MeOH in $CO_2$, 3.0 mL/min, 220 nm)

Opt. Rot.: $[\alpha]_D^{24}$ 4.9 (c = 2.2, EtOH)

Analysis: $C_{17}H_{26}O_4$ (294.39)

| | | |
|---|---|---|
| Calcd: | C, 69.36%; | H, 8.90% |
| Found: | C, 69.59%; | H, 9.07% |

**Preparation of (2*S*,3*S*)-3-Hydroxy-2-methoxy-3-(2-naphthyl)propanoic Acid 1-Ethyl-1-methylpropyl Ester ((2*S*,3*S*)-6hg) (Table 4, entry 7)**




Following General Procedure 7, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined with diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), 2-naphthaldehyde (156.2 mg, 1.0 mmol), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4h** (346.2 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 5/1 to 2/1) on silica gel (15 g), **6hg** (307 mg, 93%) as a colorless oil. The *syn*/*anti* ratio was determined to be >1/99 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*S*,3*S*)-**6hg**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.88 (d, *J* = 1.7, 1 H, HC(13)), 7.83-7.81 (m, 3 H, HC(6), HC(8), HC(11)), 7.52 (dd, *J* = 8.6, 1.7, 1 H, HC(5)), 7.48-7.45 (m, 2 H, HC(9), HC(10)), 5.13 (d, *J* = 5.6, 1 H, HC(3)), 3.97 (d, *J* = 5.6, 1 H, HC(2)), 3.41 (s, 3 H, HC(18)), 3.08 (bs, 1 H, OH), 1.74 (dq, *J* = 14.0, 7.5, 1 H, HC(15)), 1.73 (q, *J* = 7.5, 2 H, HC(15)), 1.55 (dq, *J* = 14.0, 7.5, 1 H, HC(15)), 1.27 (s, 3 H, HC(17)), 0.72 (dd, *J* = 7.5, 7.5, 3 H, HC(16)), 0.67 (dd, *J* = 7.5, 7.5, 3 H, HC(16))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

169.5 (C(1)), 137.1 (C(4)), 133.11 (C(7) or C(12)), 133.10 (C(7) or C(12)), 128.0 (C(6), C(8), or C(11)), 127.8 (C(6), C(8), or C(11)), 127.6 (C(6), C(8), or C(11)), 126.0 (C(9), C(10), or C(13)), 125.88 (C(9), C(10), or C(13)), 125.86

(C(9), C(10), or C(13)), 124.6 (C(5)), 87.8 (C(14)), 84.7 (C(2)), 74.2 (C(3)), 58.8 (C(18)), 30.3 (C(15)), 30.2 (C(15)) 22.5 (C(17)), 7.8 (C(16)), 7.7 (C(16))

IR: (neat)

3464 (br), 3057 (m), 2976 (s), 2941 (s), 2883 (m), 2830 (m), 2249 (w), 1731 (s), 1603 (w), 1509 (m), 1461 (s), 1376 (s), 1359 (s), 1270 (s), 1202 (s), 1151 (s), 1126 (s), 1072 (s), 1007 (m), 982 (m), 910 (s), 857 (s), 821 (s), 774 (m), 734 (s)

MS: (ESI)

169.1 (5), 185.1 (3), 229.1 (28), 269.1 (100), 270.1 (8), 348.2 (7), 353.1 ($M^+$+Na, 85), 354.2 (9)

HRMS: calcd for $C_{20}H_{26}O_4Na$: 353.1729, found 353.1723

TLC: $R_f$ 0.26 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

SFC: (2*R*,3*R*)-**6hg**, $t_R$ 8.84 min (2.6%); (2*S*,3*S*)-**6hg**, $t_R$ 9.76 min (97.4%)

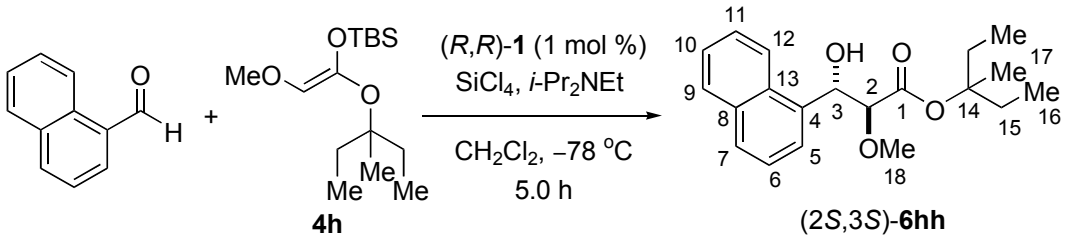
(Chiralcel OJ, 125 bar, 40 $^oC$, 3.0% MeOH in $CO_2$, 3.0 mL/min, 220 nm)

Opt. Rot.: $[\alpha]_D^{24}$ 7.3 (c = 3.8, EtOH)

Analysis: $C_{20}H_{26}O_4$ (330.42)

| | | |
|---|---|---|
| Calcd: | C, 72.70%; | H, 7.93% |
| Found: | C, 72.63%; | H, 8.01% |

**Preparation of (2*S*,3*S*)-3-Hydroxy-2-methoxy-3-(1-naphthyl)propanoic Acid 1-Ethyl-1-methylpropyl Ester ((2*S*,3*S*)-6hh) (Table 4, entry 8)**




Following General Procedure 7, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined with diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), 1-naphthaldehyde (136 μL, 1.0 mmol), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4h** (346.2 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 5/1 to 2/1) on silica gel (15 g), **6hh** (310 mg, 94%) as a colorless oil. The *syn*/*anti* ratio was determined to be >1/99 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*S*,3*S*)-**6hh**:

$^1$H NMR: (500 MHz, $CDCl_3$)

8.08 (d, *J* = 8.5, 1 H, HC(12)), 7.88-7.86 (m, 1 H, HC(9)), 7.80 (d, *J* = 8.3, 1 H, HC(7)), 7.76 (bd, *J* = 7.1, 1 H, HC(5)), 7.53 (ddd, *J* = 8.5, 6.7, 1.4, 1 H, HC(11)), 7.50-7.45 (m, 2 H, HC(6), HC(10)), 5.78 (d, *J* = 4.9, 1 H, HC(3)), 4.18 (d, *J* = 4.9, 1 H, HC(2)), 3.44 (s, 3 H, HC(18)), 3.14 (bs, 1 H, OH), 1.74 (dq, *J* = 14.2, 7.5, 1 H, HC(15)), 1.67 (dq, *J* = 14.2, 7.5, 1 H, HC(15)), 1.66 (dq, *J* = 14.1, 7.5, 1 H, HC(15)), 1.43 (dq, *J* = 14.1, 7.5, 1 H, HC(15)), 0.67 (dd, *J* = 7.5, 7.5, 3 H, HC(16)), 0.57 (dd, *J* = 7.5, 7.5, 3 H, HC(16))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

169.2 (C(1)), 135.0 (C(4)), 133.6 (C(8)), 130.8 (C(13)), 128.9 (C(9)), 128.4 (C(7)), 126.1 (C(11)), 125.4 (C(6) or C(10)), 125.3 (C(6) or C(10)), 124.4 (C(5)),

122.9 (C(11)), 87.6 (C(14)), 84.0 (C(2)), 71.1 (C(3)), 58.5 (C(18)), 30.20 (C(15)), 30.19 (C(15)), 22.5 (C(17)), 7.8 (C(16)), 7.6 (C(16))

IR: (neat)

3475 (br), 2975 (s), 2941 (m), 2883 (m), 2829 (w), 2250 (w), 1731 (s), 1598 (w), 1512 (m), 1461 (m), 1375 (m), 1354 (m), 1265 (m), 1202 (s), 1150 (s), 1120 (s), 1095 (s), 1004 (m), 976 (m), 910 (m), 850 (m), 801 (s), 778 (s), 733 (m)

MS: (ESI)

197.0 (8), 229.1 (7), 269.1 (100), 270.1 (8), 353.1 ($M^+$+Na, 69), 354.2 (9)

HRMS: calcd for $C_{20}H_{26}O_4Na$: 353.1729, found 353.1714

TLC: $R_f$ 0.28 (hexane/EtOAc, 4/1) [UV(254)/$KMnO_4$]

SFC: (2*R*,3*R*)-**6hh**, $t_R$ 8.29 min (21.8%); (2*S*,3*S*)-**6hh**, $t_R$ 9.83 min (78.2%)

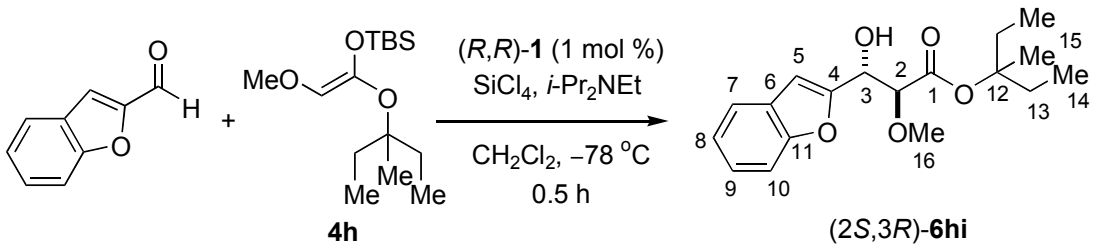
(Chiralpak AD, 125 bar, 40 °C, 5.0% MeOH in $CO_2$, 2.5 mL/min, 220 nm)

Opt. Rot.: $[\alpha]_D^{24}$ 22.2 (c = 2.0, EtOH)

Analysis: $C_{20}H_{26}O_4$ (330.42)

| | | |
|---|---|---|
| Calcd: | C, 72.70%; | H, 7.93% |
| Found: | C, 72.83%; | H, 8.04% |

**Preparation of (2*S*,3*R*)-3-(2-Benzofuryl)-3-hydroxy-2-methoxypropanoic Acid 1-Ethyl-1-methylpropyl Ester ((2*S*,3*R*)-6hi) (Table 4, entry 9)**




Following General Procedure 7, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined

with diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), 2-benzofuraldehyde (121.2 μL, 1.0 mmol), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4h** (346.2 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 5/1 to 1/1) on silica gel (15 g), **6hi** (311 mg, 97%) as a colorless oil. The *syn*/*anti* ratio was determined to be 1/99 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*S*,3*R*)-**6hi**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.54-7.52 (m, 1 H, HC(7)), 7.44-7.43 (m, 1 H, HC(10)), 7.27-7.24 (m, 1 H, HC(9)), 7.22-7.18 (m, 1 H, HC(8)), 6.76 (bs, 1 H, HC(5)), 5.15 (bd, *J* = 5.1, 1 H, HC(3)), 4.15 (d, *J* = 5.1, 1 H, HC(2)), 3.51 (s, 3 H, HC(16)), 3.00 (bs, 1 H, OH), 1.81 (dq, *J* = 14.2, 7.6, 1 H, HC(13)), 1.78 (m, 2 H, HC(13)), 1.64 (dq, *J* = 14.2, 7.6, 1 H, HC(13)), 1.34 (s, 3 H, HC(15)), 0.78 (dd, *J* = 7.6, 7.6, 3 H, HC(14)), 0.76 (dd, *J* = 7.6, 7.6, 3 H, HC(14))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

168.7 (C(1)), 155.2 (C(4)), 154.8 (C(11)), 128.0 (C(6)), 124.2 (C(9)), 122.7 (C(8)), 121.1 (C(7)), 111.2 (C(11)), 104.7 (C(5)), 88.1 (C(12)), 82.4 (C(2)), 68.9 (C(3)), 59.2 (C(16)), 30.32 (C(13)), 30.30 (C(13)), 22.6 (C(15)), 7.9 (C(14)), 7.8 (C(14))

IR: (neat)

3458 (br), 2975 (s), 2941 (m), 2883 (m), 2832 (w), 1740 (s), 1603 (w), 1586 (w), 1455 (s), 1376 (m), 1255 (s), 1199 (s), 1150 (s), 1124 (s), 1073 (m), 1037 (m), 1007 (m), 977 (m), 952 (m), 882 (m), 850 (m), 826 (m), 807 (m), 743 (s)

MS: (ESI)

175.1 (11), 219.1 (20), 259.0 (96), 260.0 (5), 338.2 (6), 343.1 ($M^+$+Na, 100),

344.1 (9)

HRMS: calcd for $C_{18}H_{24}O_5Na$: 343.1521, found 343.1512

TLC: $R_f$ 0.22 (hexane/EtOAc, 4/1) [UV(254)/$KMnO_4$]

SFC: (2*S*,3*R*)-**6hi**, $t_R$ 31.16 min (94.9%); (2*R*,3*S*)-**6hi**, $t_R$ 34.29 min (5.1%)

(Chiralcel OD, 125 bar, 40 °C, 2.0% MeOH in $CO_2$, 1.5 mL/min, 220 nm)

Opt. Rot.: $[\alpha]_D^{24}$ 13.2 (c = 2.5, EtOH)

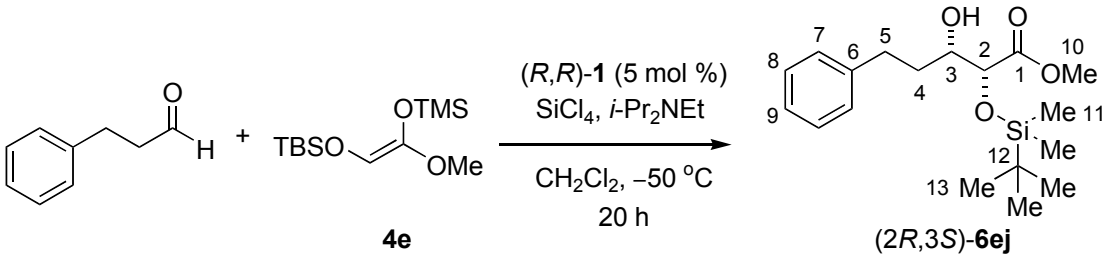
Analysis: $C_{18}H_{24}O_5$ (320.38)

| | | |
|---|---|---|
| Calcd: | C, 67.48%; | H, 7.55% |
| Found: | C, 67.17%; | H, 7.62% |

**Aldol Additions of Silyl Ketene Acetals to Aliphatic Aldehydes**

**General Procedure 8. Addition of Silyl Ketene Acetals to Aliphatic Aldehydes**

**Preparation of (2*R*,3*S*)-2-[[(1,1-Dimethylethyl)dimethylsilyl]oxy]-3-hydroxy-5-phenyl pentanoic Acid Methyl Ester ((2*R*,3*S*)-6ej) (Table 5, entry 2)**




To a flame-dried, 10-mL, Schlenk flask fitted with a magnetic stir bar, a thermocouple, a gas inlet tube, and a septum were added (*R*,*R*)-**1** (42 mg, 0.05 mmol, 0.05 equiv), $CH_2Cl_2$ (1.5 mL), and hydrocinnamaldehyde (132 μL, 1.0 mmol). The solution was cooled to –50 °C (bath temp.) in an acetone bath of Cryocooler®. After diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv) and $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv) were added to the flask via syringe, a solution of **4e** (331.8 mg, 1.2 mmol, 1.2 equiv) in $CH_2Cl_2$ (1 mL) was added dropwise via syringe over 10

min. The reaction mixture was stirred for 20 h at –50 °C before a mixture of MeOH (1 mL), $Et_3N$ (1 mL), and $CH_2Cl_2$ (5 mL) was added. The resulting solution was transferred into a 125-mL Erlenmeyer flask containing a sat. aq. $NaHCO_3$ solution (10 mL) and a sat. aq. KF solution (10 mL). The biphasic mixture was stirred vigorously for 2 h at room temperature. The mixture was filtered through a glass frit and the filtrate was transferred to a 125-mL separatory funnel where the organic layer was separated. The aqueous layer was extracted with $CH_2Cl_2$ (2 × 20 mL). The combined organic extracts were dried over $Na_2SO_4$ (15 g), filtered, and concentrated *in vacuo* (23 °C, 30 mmHg). The residue was purified by column chromatography (22 mm diam., hexane/EtOAc, 10/1 to 5/1) on silica gel (20 g) to give **6ej** (165 mg, 49%) as a colorless oil. The *syn*/*anti* ratio was determined to be 90/10 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*R*,3*S*)-**6ej**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.30-7.27 (m, 2 H, HC(8)), 7.21-7.18 (m, 3 H, HC(7), HC(9)), 4.14 (d, *J* = 3.4, 1 H, HC(2)), 3.84-3.81 (m, 1 H, HC(3)), 3.74 (s, 3 H, HC(10)), 2.86 (ddd, *J* = 13.9, 9.4, 5.6, 1 H, HC(5)), 2.69 (ddd, *J* = 13.9, 9.2, 7.5, 1 H, HC(5)), 2.25 (bs, 1 H, OH), 1.88-1.76 (m, 2 H, HC(4)), 0.93 (s, 9 H, HC(13)), 0.11 (s, 3 H, HC(11)), 0.07 (s, 3 H, HC(11))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

172.5 (C(1)), 141.6 (C(6)), 128.42 (C(7)), 128.38 (C(8)), 125.9 (C(9)), 74.8 (C(2)), 72.5 (C(3)), 52.0 (C(10)), 35.3 (C(4)), 31.8 (C(5)), 25.6 (C(13)), 18.2 (C(12)), –5.0 (C(11)), –5.5 (C(11))

IR: (neat)

3490 (br), 3063 (w), 3028 (m), 2953 (s), 2930 (s), 2896 (m), 2858 (s), 1759 (s),

1604 (m), 1497 (m), 1472 (m), 1456 (m), 1438 (m), 1390 (m), 1362 (m), 1255 (s), 1206 (m), 1137 (s), 1053 (m), 1008 (m), 859 (s), 838 (s), 780 (s), 748 (m), 700 (s)

MS: (ESI)

129.1 (7), 189.1 (7), 339.2 (8), 361.2 ($M^+$+Na, 100), 362.2 (17)

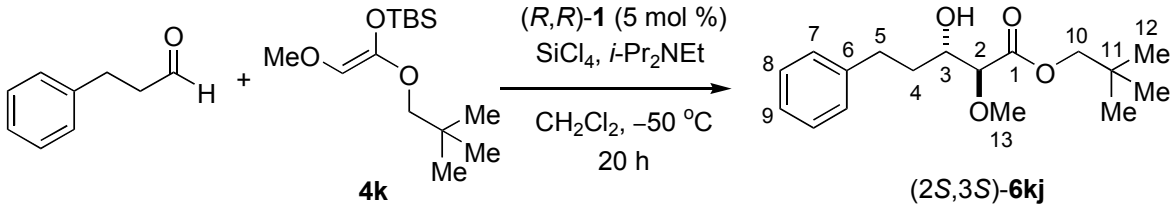
HRMS: calcd for $C_{18}H_{30}O_4SiNa$: 361.1811, found 361.1811

TLC: $R_f$ 0.42 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

SFC: (2*S*,3*R*)-**6ej**, $t_R$ 8.04 min (12.7%); (2*R*,3*S*)-**6ej**, $t_R$ 8.69 min (87.3%)

(Chiralpak AD, 125 bar, 40 °C, 2.5% MeOH in $CO_2$, 1.0 mL/min, 220 nm)

**Preparation of (2*S*,3*S*)-3-Hydroxy-2-methoxy-5-phenylpentanoic Acid 2,2-Dimethylpropyl Ester ((2*S*,3*S*)-6kj) (Table 6, entry 2)**




Following General Procedure 8, (*R*,*R*)-**1** (42 mg, 0.05 mmol, 0.05 equiv) was combined with hydrocinnamaldehyde (132 μL, 1.0 mmol), diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4k** (329 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 5/1 to 2/1) on silica gel (20 g), **6kj** (239 mg, 81%) as a colorless oil. The *syn*/*anti* ratio was determined to be 9/91 by $^1H$ NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*S*,3*S*)-**6kj**:

$^{1}$H NMR: (500 MHz, $CDCl_3$)

7.29-7.25 (m, 2 H, HC(8)), 7.21-7.16 (m, 3 H, HC(7), HC(9)), 3.92 (ddd, *J* = 9.6, 4.3, 3.3, 1 H, HC(3)), 3.85 (s, 2 H, HC(10)), 3.83 (d, *J* = 4.3, 1 H, HC(2)), 3.46 (s, 3 H, HC(13)), 2.87 (ddd, *J* = 13.8, 9.4, 5.0, 1 H, HC(5)), 2.68 (ddd, *J* = 13.8, 9.2, 7.4, 1 H, HC(5)), 2.20 (bs, 1 H, OH), 1.86 (dddd, *J* = 14.0, 9.4, 9.4, 5.0, 1 H, HC(4)), 1.78 (dddd, *J* = 14.0, 9.4, 7.4, 3.3, 1 H, HC(4)), 0.93 (s, 9 H, HC(12))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

170.6 (C(1)), 141.6 (C(6)), 128.4 (C(7)), 128.3 (C(8)), 125.8 (C(9)), 83.8 (C(2)), 74.3 (C(10)), 71.3 (C(3)), 58.8 (C(13)), 33.8 (C(4)), 31.8 (C(5)), 31.2 (C(11)), 26.4 (C(12))

IR: (neat)

3484 (br), 3086 (m), 3063 (m), 3027 (m), 2958 (s), 2871 (s), 2832 (m), 1748 (s), 1604 (m), 1497 (m), 1479 (m), 1455 (s), 1367 (s), 1344 (m), 1276 (s), 1260 (s), 1190 (s), 1091 (s), 1051 (s), 994 (s), 937 (m), 749 (s), 700 (s)

MS: (ESI)

147.1 (25), 225.1 (12), 295.2 (10), 317.2 ($M^+$+Na, 100), 318.2 (13)

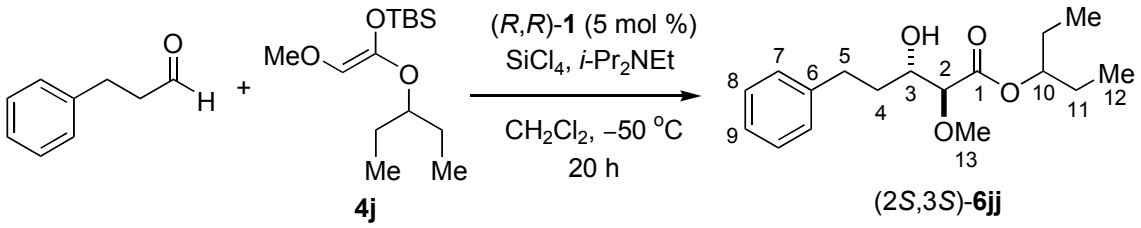
HRMS: calcd for $C_{17}H_{26}O_4Na$: 317.1729, found 317.1735

TLC: $R_f$ 0.31 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

SFC: (2*S*,3*S*)-**6kj**, $t_R$ 6.07 min (94.7%); (2*R*,3*R*)-**6kj**, $t_R$ 7.09 min (5.3%)

(Chiralcel OD, 125 bar, 40 $^{o}$C, 2.0% MeOH in $CO_2$, 3.0 mL/min, 220 nm)

**Preparation of (2*S*,3*S*)-3-Hydroxy-2-methoxy-5-phenylpentanoic Acid 1-Ethylpropyl Ester ((2*S*,3*S*)-6jj) (Table 6, entry 3)**




Following General Procedure 8, (*R*,*R*)-**1** (42 mg, 0.05 mmol, 0.05 equiv) was combined with hydrocinnamaldehyde (132 μL, 1.0 mmol), diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4j** (329 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 5/1 to 2/1) on silica gel (20 g), **6jj** (253 mg, 86%) as a colorless oil. The *syn*/*anti* ratio was determined to be 3/97 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*S*,3*S*)-**6jj**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.29-7.25 (m, 2 H, HC(8)), 7.20-7.16 (m, 3 H, HC(7), HC(9)), 4.89-4.84 (m, 1 H, HC(10)), 3.90 (ddd, *J* = 9.4, 4.4, 3.5, 1 H, HC(3)), 3.79 (d, *J* = 4.4, 1 H, HC(2)), 3.45 (s, 3 H, HC(13)), 2.87 (ddd, *J* = 13.8, 9.6, 5.3, 1 H, HC(5)), 2.68 (ddd, *J* = 13.8, 9.4, 7.5, 1 H, HC(5)), 2.31 (bs, 1 H, OH), 1.86 (dddd, *J* = 14.1, 9.4, 9.4, 5.3, 1 H, HC(4)), 1.79 (dddd, *J* = 14.1, 9.6, 7.5, 3.5, 1 H, HC(4)), 1.66-1.52 (m, 4 H, HC(11)), 0.89 (dd, *J* = 7.6, 7.6, 3 H, HC(12)), 0.86 (dd, *J* = 7.5, 7.5, 3 H, HC(12))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

170.4 (C(1)), 141.6 (C(6)), 128.4 (C(7)), 128.3 (C(8)), 125.8 (C(9)), 84.0 (C(2)),

77.9 (C(10)), 71.4 (C(3)), 58.7 (C(13)), 33.9 (C(4)), 31.8 (C(5)), 26.29 (C(11)), 26.28 (C(11)), 9.58 (C(12)), 9.56 (C(12))

IR: (neat)

3483 (br), 3063 (m), 3027 (m), 2969 (s), 2937 (s), 2881 (m), 2832 (m), 1744 (s), 1604 (m), 1497 (m), 1456 (s), 1385 (m), 1346 (m), 1273 (s), 1196 (s), 1124 (s), 1091 (s), 1050 (m), 1032 (m), 968 (m), 940 (m), 911 (m), 748 (s), 700 (s)

MS: (ESI)

147.1 (5), 317.2 ($M^+$+Na, 100), 318.2 (14)

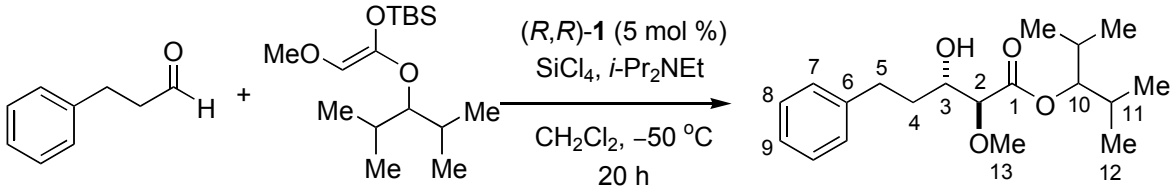
HRMS: calcd for $C_{17}H_{26}O_4Na$: 317.1729, found 317.1730

TLC: $R_f$ 0.29 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

SFC: (2*R*,3*R*)-**6jj**, $t_R$ 16.68 min (3.7%); (2*S*,3*S*)-**6jj**, $t_R$ 17.44 min (96.3%)

(Chiralcel OD, 110 bar, 40 °C, 5.0% MeOH in $CO_2$, 0.8 mL/min, 220 nm)

**Preparation of (2*S*,3*S*)-3-Hydroxy-2-methoxy-5-phenylpentanoic Acid 2-Methyl-1-(1-methylethyl)propyl Ester ((2*S*,3*S*)-6lj) (Table 6, entry 4)**




**4l** (2*S*,3*S*)-**6lj**

Following General Procedure 8, (*R*,*R*)-**1** (42 mg, 0.05 mmol, 0.05 equiv) was combined with hydrocinnamaldehyde (132 μL, 1.0 mmol), diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4l** (363 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 5/1 to 2/1) on silica gel (20 g), **6lj** (283 mg, 88%) as a colorless oil. The *syn*/*anti* ratio was determined to be >1/99 by $^1$H NMR (500

MHz) analysis of the crude reaction mixture.

Data for (2*S*,3*S*)-**6lj**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.28-7.24 (m, 2 H, HC(8)), 7.19-7.15 (m, 3 H, HC(7), HC(9)), 4.70 (dd, *J* = 6.1, 6.1, 1 H, HC(10)), 3.91 (ddd, *J* = 9.4, 4.4, 3.5, 1 H, HC(3)), 3.81 (d, *J* = 4.4, 1 H, HC(2)), 3.46 (s, 3 H, HC(13)), 2.88 (ddd, *J* = 13.7, 9.5, 5.5, 1 H, HC(5)), 2.68 (ddd, *J* = 13.7, 9.2, 7.4, 1 H, HC(5)), 2.20 (bs, 1 H, OH), 1.96-1.86 (m, 3 H, HC(4), HC(11)), 1.83 (dddd, *J* = 14.0, 9.5, 7.4, 3.5, 1 H, HC(4)), 0.90 (d, *J* = 6.8, 3 H, HC(12)), 0.870 (d, *J* = 6.9, 3 H, HC(12)), 0.866 (d, *J* = 6.8, 3 H, HC(12)), 0.83 (d, *J* = 6.6, 3 H, HC(12))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

170.6 (C(1)), 141.6 (C(6)), 128.5 (C(7)), 128.3 (C(8)), 125.8 (C(9)), 84.3 (C(2)), 84.0 (C(10)), 71.4 (C(3)), 58.9 (C(13)), 33.9 (C(4)), 31.8 (C(5)), 29.4 (C(11)), 29.2 (C(11)), 19.6 (C(12)), 19.5 (C(12)), 17.4 (C(12)), 17.2 (C(12))

IR: (neat)

3484 (br), 3027 (m), 2966 (s), 2936 (s), 2877 (m), 2832 (m), 1745 (s), 1604 (m), 1497 (m), 1464 (s), 1455 (s), 1389 (m), 1372 (m), 1272 (s), 1194 (s), 1130 (s), 1091 (s), 1050 (m), 1000 (m), 970 (m), 943 (m), 896 (m), 749 (m), 700 (s)

MS: (ESI)

345.2 ($M^+$+Na, 100), 346.2 (18)

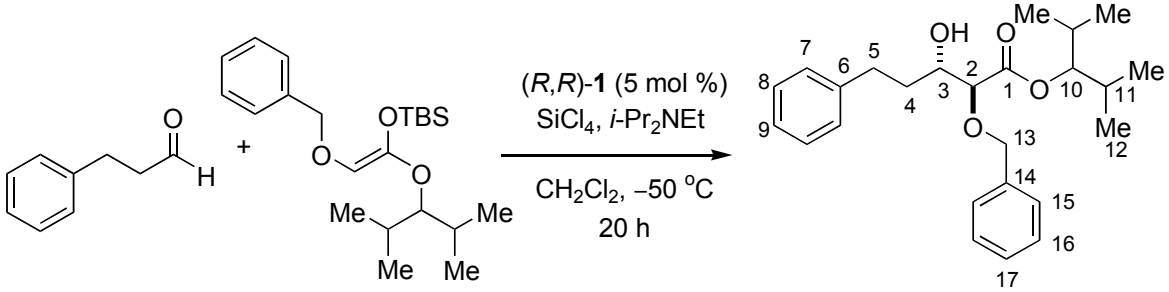
HRMS: calcd for $C_{19}H_{30}O_4Na$: 345.2042, found 345.2043

TLC: $R_f$ 0.34 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

SFC: (2*R*,3*R*)-**6lj**, $t_R$ 4.98 min (3.0%); (2*S*,3*S*)-**6lj**, $t_R$ 5.79 min (97.0%)

(Chiralcel OD, 125 bar, 40 $^oC$, 3.0% MeOH in $CO_2$, 3.0 mL/min, 220 nm)

**Preparation of (2*S*,3*S*)-3-Hydroxy-5-phenyl-2-(phenylmethoxy)pentanoic Acid 2-Methyl-1-(1-methylethyl)propyl Ester ((2*S*,3*S*)-6mj) (Table 7, entry 1)**




**4m** (2*S*,3*S*)-**6mj**

Following General Procedure 8, (*R*,*R*)-**1** (42 mg, 0.05 mmol, 0.05 equiv) was combined with hydrocinnamaldehyde (132 μL, 1.0 mmol), diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4m** (454 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 10/1 to 5/1) on silica gel (20 g), **6mj** (325 mg, 82%) as a colorless oil. The *syn*/*anti* ratio was determined to be 2/98 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*S*,3*S*)-**6mj**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.38-7.37 (m, 4 H, HC(15), HC(16)), 7.36-7.31 (m, 1 H, HC(17)), 7.29-7.27 (m, 2 H, HC(8)), 7.20-7.17 (m, 3 H, HC(7), HC(9)), 4.85 (d, *J* = 11.4, 1 H, HC(13)), 4.75 (dd, *J* = 6.1, 6.1, 1 H, HC(10)), 4.45 (d, *J* = 11.4, 1 H, HC(13)), 4.06 (d, *J* = 4.7, 1 H, HC(2)), 3.99-3.95 (m, 1 H, HC(3)), 2.87 (ddd, *J* = 13.7, 9.3, 5.5, 1 H, HC(5)), 2.69 (ddd, *J* = 13.7, 9.0, 7.6, 1 H, HC(5)), 2.28 (bs, 1 H, OH), 1.98-1.85 (m, 4 H, HC(4), HC(11)), 0.93 (d, *J* = 6.9, 3 H, HC(12)), 0.903 (d, *J* = 6.8, 3 H, HC(12)), 0.901 (d, *J* = 6.8, 3 H, HC(12)), 0.86 (d, *J* = 6.8, 3 H, HC(12))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

170.7 (C(1)), 141.7 (C(6)), 137.2 (C(14)), 128.52 (Aryl), 128.49 (Aryl), 128.3

(Aryl), 128.2 (Aryl), 128.1 (C(17)), 125.8 (C(9)), 84.1 (C(10)), 81.4 (C(2)), 72.9 (C(13)), 71.5 (C(3)), 33.9 (C(4)), 31.8 (C(5)), 29.4 (C(11)), 29.2 (C(11)), 19.7 (C(12)), 19.5 (C(12)), 17.4 (C(12)), 17.2 (C(12))

IR: (neat)

3484 (br), 3063 (w), 3028 (m), 2966 (s), 2935 (s), 2876 (m), 1743 (s), 1603 (w), 1497 (m), 1455 (s), 1389 (m), 1371 (m), 1271 (m), 1200 (s), 1128 (s), 1095 (s), 1045 (m), 1029 (m), 1000 (m), 967 (m), 949 (m), 897 (m), 748 (s), 699 (s)

MS: (ESI)

91.1 (7), 219.1 (10), 237.1 (55), 238.1 (7), 287.2 (18), 301.1 (23), 399.2 (11), 421.2 ($M^+$+Na, 100), 422.2 (22)

HRMS: calcd for $C_{25}H_{34}O_4Na$: 421.2355, found 421.2361

TLC: $R_f$ 0.30 (hexane/EtOAc, 10/1) [UV(254)/$KMnO_4$]

SFC: (2*R*,3*R*)-**6mj**, $t_R$ 6.63 min (3.6%); (2*S*,3*S*)-**6mj**, $t_R$ 8.46 min (96.4%)

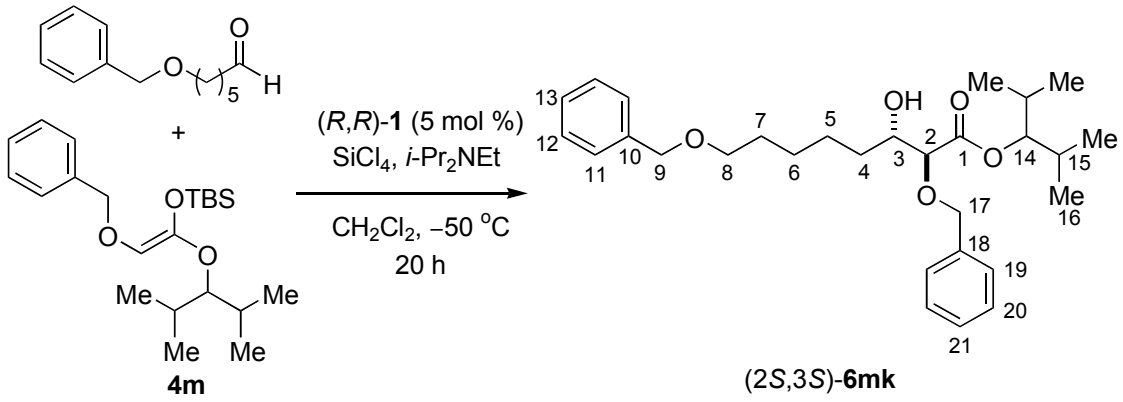
(Chiralcel OD, 125 bar, 40 $^oC$, 5.0% MeOH in $CO_2$, 3.0 mL/min, 220 nm)

Opt. Rot.: $[\alpha]_D^{24}$ –47.6 (c = 5.0, EtOH)

Analysis: $C_{25}H_{34}O_4$ (398.54)

| | | |
|---|---|---|
| Calcd: | C, 75.34%; | H, 8.60% |
| Found: | C, 75.26%; | H, 8.57% |

**Preparation of (2*S*,3*S*)-2,8-Di(phenylmethoxy)-3-hydroxyoctanoic Acid 2-Methyl-1-(1-methylethyl)propyl Ester ((2*S*,3*S*) -6mk) (Table 7, entry 2)**




Following General Procedure 8, (*R*,*R*)-**1** (42 mg, 0.05 mmol, 0.05 equiv) was combined with 6-benzyloxyhexanal (206 mg, 1.0 mmol), diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4m** (454 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 10/1 to 5/1) on silica gel (20 g), **6mk** (420 mg, 89%) as a colorless oil. The *syn*/*anti* ratio was determined to be 2/98 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*S*,3*S*)-**6mk**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.38-7.26 (m, 10 H, Aryl), 4.84 (d, *J* = 11.5, 1 H, HC(17)), 4.75 (dd, *J* = 6.1, 6.1, 1 H, HC(14)), 4.49 (s, 2 H, HC(9)), 4.45 (d, *J* = 11.5, 1 H, HC(17)), 4.01 (d. *J* = 4.3, 1 H, HC(2)), 3.94-3.91 (m, 1 H, HC(3)), 3.45 (dd, *J* = 6.7, 6.7, 2 H, HC(8)), 2.19 (bs, 1 H, OH), 1.96 (qqd, *J* = 6.8, 6.8, 6.1, 1 H, HC(15)), 1.95 (qqd, *J* = 6.6, 6.6, 6.1, 1 H, HC(15)), 1.64-1.31 (m, 8 H, HC(4), HC(5), HC(6), HC(7)), 0.93 (d, *J* = 6.8, 6 H, HC(16)), 0.90 (d, *J* = 6.6, 6 H, HC(16))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

170.8 (C(1)), 138.6 (C(10)), 137.2 (C(18)), 128.4 (Aryl), 128.3 (Aryl), 128.1

(Aryl), 128.0 (C(21)), 127.6 (C(11)), 127.4 (C(13)), 84.0 (C(14)), 81.5 (C(2)), 72.85 (C(9) or C(17)), 72.79 (C(9) or C(17)), 72.3 (C(3)), 70.3 (C(8)), 32.2 (C(4)), 29.6 (C(7)), 29.4 (C(15)), 29.3 (C(15)), 26.1 (C(6)), 25.5 (C(5)), 19.7 (C(16)), 19.6 (C(16)), 17.4 (C(16)), 17.3 (C(16))

IR: (neat)

3475 (br), 3064 (m), 3031 (m), 2965 (s), 2937 (s), 2874 (s), 2247 (w), 1743 (s), 1605 (w), 1587 (w), 1496 (m), 1455 (s), 1389 (m), 1370 (s), 1270 (s), 1199 (s), 1099 (s), 1028 (s), 1000 (m), 968 (m), 898 (m), 735 (s), 698 (s), 611 (m)

MS: (ESI)

181.1 (7), 193.1 (8), 373.2 (8), 493.3 ($M^+$+Na, 100), 494.3 (15)

HRMS: calcd for $C_{29}H_{42}O_5Na$: 493.2930, found 493.2934

TLC: $R_f$ 0.35 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

SFC: (2*S*,3*S*)-**6mk**, $t_R$ 7.23 min (98.5%); (2*R*,3*R*)-**6mk**, $t_R$ 8.29 min (1.5%)

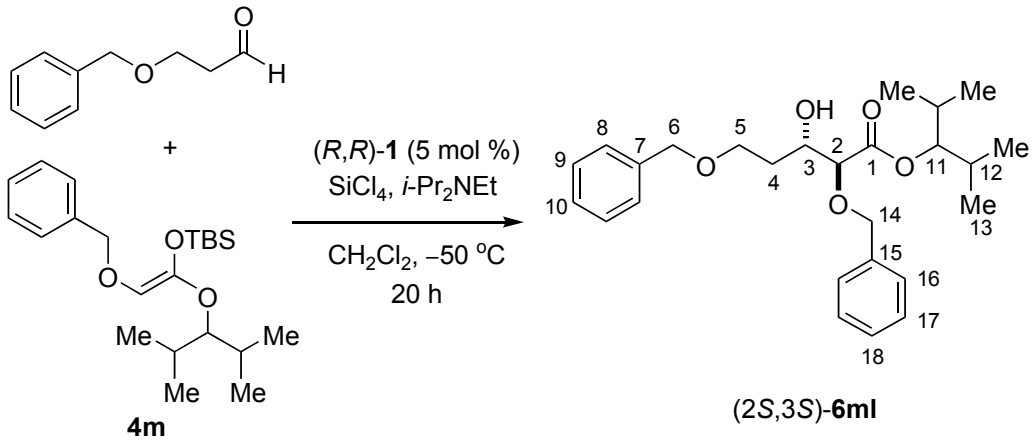
(Chiralcel OJ, 125 bar, 40 $^oC$, 5.0% MeOH in $CO_2$, 3.0 mL/min, 220 nm)

Opt. Rot.: $[\alpha]_D^{24}$ –41.0 (c = 2.5, EtOH)

Analysis: $C_{29}H_{42}O_5$ (470.64)

| | | |
|---|---|---|
| Calcd: | C, 74.01%; | H, 8.99% |
| Found: | C, 73.93%; | H, 8.93% |

**Preparation of (2*S*,3*S*)-2,5-Di(phenylmethoxy)-3-hydroxypentanoic Acid 2-Methyl-1-(1-methylethyl)propyl Ester ((2*S*,3*S*)-6ml) (Table 7, entry 3)**




Following General Procedure 8, (*R*,*R*)-**1** (42 mg, 0.05 mmol, 0.05 equiv) was combined with 3-benzyloxypropanal (164 mg, 1.0 mmol), diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4m** (454 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 10/1 to 5/1) on silica gel (20 g), **6ml** (344 mg, 80%) as a colorless oil. The *syn*/*anti* ratio was determined to be 1/99 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*S*,3*S*)-**6ml**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.37-7.27 (m, 10 H, Aryl), 4.79 (d, *J* = 11.5, 1 H, HC(14)), 4.74 (dd, *J* = 6.1, 6.1, 1 H, HC(11)), 4.49 (s, 2 H, HC(6)), 4.44 (d, *J* = 11.5, 1 H, HC(14)), 4.17 (ddd, *J* = 9.0, 5.4, 2.6, 1 H, HC(3)), 4.00 (d, *J* = 5.4, 1 H, HC(2)), 3.69 (ddd, *J* = 9.4, 7.0, 4.8, 1 H, HC(5)), 3.63 (ddd, *J* = 9.4, 6.7, 4.9, 1 H, HC(5)), 3.02 (bs, 1 H, OH), 1.99-1.85 (m, 4 H, HC(4), HC(12)), 0.92 (d, *J* = 6.9, 3 H, HC(13)), 0.91 (d, *J* = 6.9, 3 H, HC(13)), 0.90 (d, *J* = 6.6, 3 H, HC(13)), 0.89 (d, *J* = 6.8, 3 H, HC(13))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

170.9 (C(1)), 138.0 (C(7)), 137.3 (C(15)), 128.38 (Aryl), 128.35 (Aryl), 128.1 (Aryl), 127.9 (C(18)), 127.61 (C(10)), 127.60 (C(8)), 83.9 (C(11)), 81.6 (C(2)), 73.1 (C(6)), 72.7 (C(14)), 71.3 (C(3)), 68.1 (C(5)), 32.0 (C(4)), 29.4 (C(12)), 29.3 (C(12)), 19.63 (C(13)), 19.59 (C(13)), 17.4 (C(13)), 17.3 (C(13))

IR: (neat)

3495 (br), 3064 (m), 3032 (m), 2965 (s), 2935 (s), 2875 (s), 2248 (w), 1962 (w), 1873 (w), 1809 (w), 1742 (s), 1497 (m), 1455 (s), 1390 (s), 1370 (s), 1269 (s), 1199 (s), 1098 (s), 1028 (s), 1000 (m), 971 (m), 898 (m), 736 (s), 698 (s)

MS: (ESI)

87.0 (6), 91.1 (8), 133.0 (22), 151.1 (26), 182.1 (7), 331.2 (81), 332.2 (11), 385.1 (9), 429.3 (12), 451.2 ($M^+$+Na, 100), 452.3 (19)

HRMS: calcd for $C_{26}H_{36}O_5Na$: 451.2460, found 451.2444

TLC: $R_f$ 0.32 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

SFC: (2*R*,3*R*)-**6ml**, $t_R$ 10.89 min (7.5%); (2*S*,3*S*)-**6ml**, $t_R$ 12.46 min (92.5%)

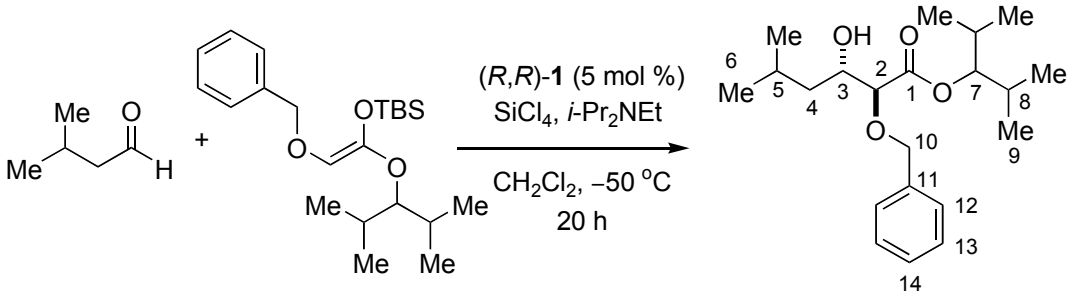
(Chiralcel OJ, 125 bar, 40 $^oC$, 2.0% MeOH in $CO_2$, 3.0 mL/min, 220 nm)

Opt. Rot.: $[\alpha]_D^{24}$ –39.6 (c = 2.0, EtOH)

Analysis: $C_{26}H_{36}O_5$ (428.56)

| | | |
|---|---|---|
| Calcd: | C, 72.87%; | H, 8.47% |
| Found: | C, 72.96%; | H, 8.46% |

**Preparation of (2*S*,3*S*)-3-Hydroxy-5-methyl-2-(phenylmethoxy)hexanoic Acid 2-Methyl-1-(1-methylethyl)propyl Ester ((2*S*,3*S*)-6mn) (Table 7, entry 5)**




**4m** (2*S*,3*S*)-**6mn**

Following General Procedure 8, (*R*,*R*)-**1** (42 mg, 0.05 mmol, 0.05 equiv) was combined with *iso*-valeraldehyde (107 μL, 1.0 mmol), diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4m** (454 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 10/1) on silica gel (15 g), **6mn** (291 mg, 83%) as a colorless oil. The *syn*/*anti* ratio was determined to be >1/99 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*S*,3*S*)-**6mn**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.39-7.34 (m, 4 H, HC(12), HC(13)), 7.33-7.29 (m, 1 H, HC(14)), 4.84 (d, *J* = 11.4, 1 H, HC(10)), 4.74 (dd, *J* = 6.1, 6.1, 1 H, HC(7)), 4.44 (d, *J* = 11.4, 1 H, HC(10)), 4.05-4.01 (m, 2 H, HC(2), HC(3)), 2.08 (bs, 1 H, OH), 2.00-1.89 (m, 2 H, HC(8)), 1.87-1.79 (m, 1 H, HC(5)), 1.61-1.55 (m, 1 H, HC(4)), 1.29 (ddd, *J* = 14.4, 9.6, 2.6, 1 H, HC(4)), 0.93-0.88 (m, 18 H, HC(6), HC(9))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

170.6 (C(1)), 137.3 (C(11)), 128.5 (C(13)), 128.1 (C(12)), 128.0 (C(14)), 84.0 (C(7)), 81.9 (C(2)), 72.9 (C(10)), 70.5 (C(3)), 40.9 (C(4)), 29.4 (C(8)), 29.3 (C(8)), 24.4 (C(5)), 23.7 (C(6)), 21.4 (C(6)), 19.7 (C(9)), 19.6 (C(9)), 17.5

(C(9)), 17.3 (C(9))

IR: (neat)

3491 (br), 3066 (w), 3032 (w), 2962 (s), 2937 (s), 2874 (m), 1745 (s), 1467 (m), 1456 (m), 1389 (m), 1369 (m), 1279 (m), 1199 (s), 1146 (m), 1129 (s), 1099 (s), 1078 (s), 1028 (m), 1001 (m), 966 (m), 953 (m), 898 (m), 737 (m), 698 (m)

MS: (ESI)

149.0 (6), 175.1 (8), 253.1 (19), 354.3 (13), 373.2 ($M^+$+Na, 100), 374.2 (12), 413.3 (12)

HRMS: calcd for $C_{21}H_{34}O_4Na$: 373.2355, found 373.2343

TLC: $R_f$ 0.27 (hexane/EtOAc, 10/1) [UV(254)/$KMnO_4$]

SFC: (2*S*,3*S*)-**6mn**, $t_R$ 13.44 min (95.3%); (2*R*,3*R*)-**6mn**, $t_R$ 15.29 min (4.7%)

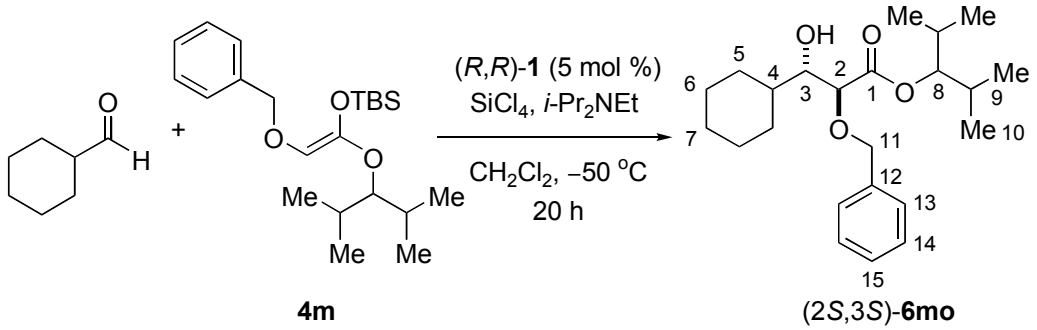
(Chiralcel OD, 125 bar, 40 °C, 2.0% MeOH in $CO_2$, 1.0 mL/min, 220 nm)

Opt. Rot.: $[\alpha]_D^{24}$ −55.5 (c = 2.0, EtOH)

Analysis: $C_{21}H_{34}O_4$ (350.49)

| | | |
|---|---|---|
| Calcd: | C, 71.96%; | H, 9.78% |
| Found: | C, 71.92%; | H, 9.93% |

**Preparation of (2*S*,3*S*)-3-Cyclohexyl-3-hydroxy-2-(phenylmethoxy)hexanoic Acid 2-Methyl-1-(1-methylethyl)propyl Ester ((2*S*,3*S*)-6mo) (Table 7, entry 6)**




Following General Procedure 8, (*R*,*R*)-**1** (12.6 mg, 0.015 mmol, 0.05 equiv) was combined with cyclohexanal (36.3 μL, 0.3 mmol), diisopropylethylamine (52 μL, 0.3 mmol, 1.0 equiv), $SiCl_4$ (37.8 μL, 0.33 mmol, 1.1 equiv), and **4m** (136.3 mg, 0.36 mmol, 1.2 equiv) to yield, after column chromatography (18 mm diam., hexane/EtOAc, 10/1 to 5/1) on silica gel (5 g), **6mo** (10 mg, 9%) as a colorless oil. The *syn*/*anti* ratio was determined to be 2/98 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*S*,3*S*)-**6mo**:

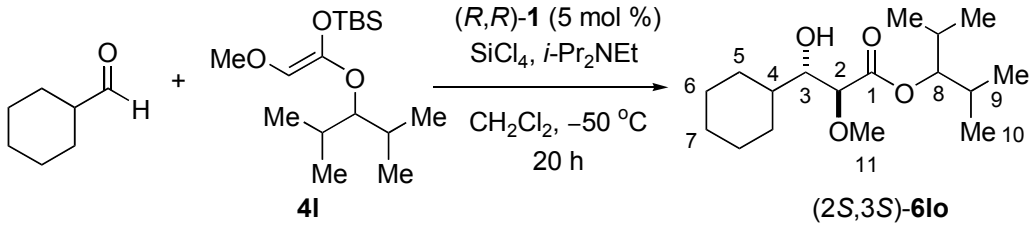
$^1$H NMR: (500 MHz, $CDCl_3$)

7.37-7.34 (m, 4 H, HC(13), HC(14)), 7.33-7.29 (m, 1 H, HC(15)), 4.81 (d, *J* = 11.7, 1 H, HC(11)), 4.77 (dd, *J* = 6.0, 6.0, 1 H, HC(8)), 4.44 (d, *J* = 11.2, 1 H, HC(11)), 4.03 (d, *J* = 5.8, 1 H, HC(2)), 3.70 (ddd, *J* = 5.1, 5.1, 5.1, 1 H, HC(3)), 2.33 (d, *J* = 4.7, 1 H, OH), 2.00-1.93 (m, 2 H, HC(9)), 1.74-1.59 (m, 6 H, Cy), 1.29-0.99 (m, 5 H, Cy), 0.95-0.91 (m, 12 H, HC(10))

SFC: (2*S*,3*S*)-**6mo**, $t_R$ 4.75 min (68.0%); (2*R*,3*R*)-**6mo**, $t_R$ 5.27 min (32.0%)

(Chiralcel OD, 125 bar, 40 °C, 3.0% MeOH in $CO_2$, 3.0 mL/min, 220 nm)

**Preparation of (2*S*,3*S*)-3-Cyclohexyl-3-hydroxy-2-methoxyhexanoic Acid 2-Methyl-1-(1-methylethyl)propyl Ester ((2*S*,3*S*)-6lo) (Table 7, entry 7)**



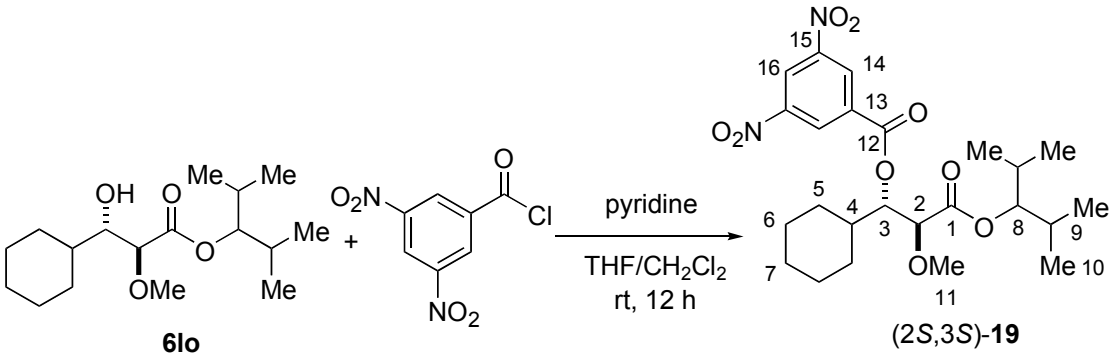

Following General Procedure 8, (*R*,*R*)-**1** (12.6 mg, 0.015 mmol, 0.05 equiv) was combined with cyclohexanal (36.3 μL, 0.3 mmol), diisopropylethylamine (52 μL, 0.3 mmol, 1.0 equiv), $SiCl_4$ (37.8 μL, 0.33 mmol, 1.1 equiv), and **4l** (108.9 mg, 0.36 mmol, 1.2 equiv) to yield, after column chromatography (18 mm diam., hexane/EtOAc, 10/1 to 5/1) on silica gel (5 g), **6lo** (41 mg, 46%) as a colorless oil. The *syn*/*anti* ratio was determined to be >1/99 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*S*,3*S*)-**6lo**:

$^1$H NMR: (500 MHz, $CDCl_3$)

4.74 (dd, *J* = 6.1, 6.1, 1 H, HC(8)), 3.84 (d, *J* = 5.6, 1 H, HC(2)), 3.66 (ddd, *J* = 5.5, 5.5, 5.5, 1 H, HC(3)), 3.45 (s, 3 H, HC(11)), 2.27 (d, *J* = 5.1, 1 H, OH), 1.99-1.92 (m, 4 H, HC(9)), 1.92-1.88 (m, 1 H, Cy), 1.78-1.60 (m, 5 H, Cy), 1.31-1.04 (m, 5 H, Cy), 0.93 (d, *J* = 6.9, 3 H, HC(10)), 0.92 (d, *J* = 6.8, 3 H, HC(10)), 0.92 (d, *J* = 6.6, 3 H, HC(10)), 0.90 (d, *J* = 6.6, 3 H, HC(10))

**Preparation of (2*S*,3*S*)-3-Cyclohexyl-3-(3,5-dinitrobenzoyloxy)-2-methoxyhexanoic Acid 2-Methyl-1-(1-methylethyl)propyl Ester ((2*S*,3*S*)-19) (Table 7, entry 7)**




**6lo** (2*S*,3*S*)-**19**

To a flame-dried, 5-mL, 2-necked round-bottomed flask fitted with a magnetic stir bar, a gas inlet tube, and a septum were added **6lo** (40 mg, 0.133 mmol), THF (0.5 mL), and pyridine (0.5 mL). Then, a solution of 3,5-dinitrobenzoyl chloride (101.4 mg, 0.438 mmol, 3.3 equiv) in $CH_2Cl_2$ (1.5 mL) was added dropwise via syringe at rt. After 12 h, the reaction mixture was transferred to a 50-mL separatory funnel where the reaction mixture was diluted with $Et_2O$ (10 mL) and washed with sat. aq. $NH_4Cl$ solution (15 mL). The organic layer was dried over $MgSO_4$ (1 g), filtered, and concentrated *in vacuo* (23 $^oC$, 30 mmHg). The residue was purified by column chromatography (18 mm diam., hexane/EtOAc, 10/1) on silica gel (5 g) to give **19** (59 mg, 87%) as a white foam.

Data for (2*S*,3*S*)-**19**:

$^1$H NMR: (500 MHz, $CDCl_3$)

9.21 (t, *J* = 2.0, 1 H, HC(16)), 9.13 (d, *J* = 2.2, 2 H, HC(14)), 5.42 (dd, *J* = 8.5, 3.7, 1 H, HC(3)), 4.82 (dd, *J* = 6.1, 6.1, 1 H, HC(8)), 4.13 (d, *J* = 3.4, 1 H, HC(2)), 3.44 (s, 3 H, HC(11)), 2.10-1.96 (m, 2 H, HC(9)), 1.94-1.90 (m, 1 H, Cy), 1.83-1.68 (m, 5 H, Cy), 1.36-1.06 (m, 5 H, Cy), 0.98 (d, *J* = 6.6, 3 H, HC(10)), 0.97 (d, *J* = 6.3, 3 H, HC(10)), 0.92 (d, *J* = 6.8, 3 H, HC(10)), 0.90 (d,

$J$ = 6.8, 3 H, HC(10))

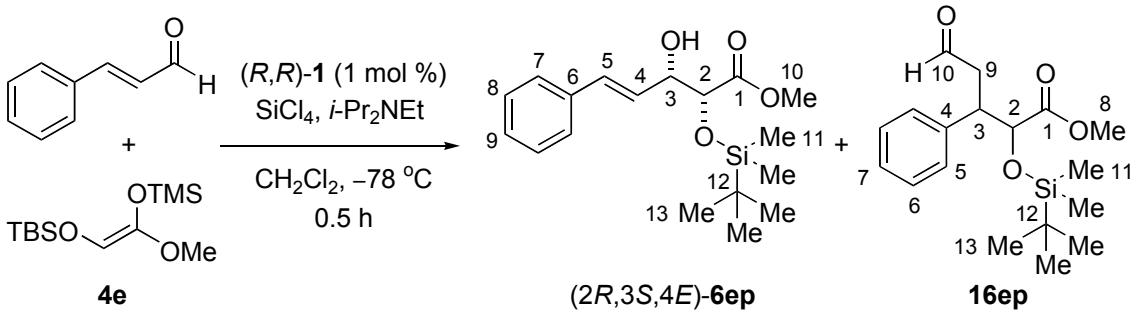
SFC: (2*S*,3*S*)-**19**, $t_R$ 2.61 min (87.7%); (2*R*,3*R*)-**19**, $t_R$ 4.76 min (23.3%)

(Chiralcel OD, 125 bar, 40 °C, 15.0% MeOH in $CO_2$, 3.0 mL/min, 220 nm)

**Aldol Additions of Silyl Ketene Acetals to Olefinic Aldehydes**

**General Procedure 9. Addition of Silyl Ketene Acetals to Olefinic Aldehydes**

**Preparation of (2*R*,3*S*,4*E*)-2-[[(1,1-Dimethylethyl)dimethylsilyl]oxy]-3-hydroxy-5-phenyl-4-pentenoic Acid Methyl Ester ((2*R*,3*S*,4*E*)-6ep) and 2-[[(1,1-Dimethylethyl)dimethylsilyl] oxy]-5-oxo-3-phenylpentanoic Acid Methyl Ester (16ep) (Table 8, entry 1)**




To a flame-dried, 10-mL, Schlenk flask fitted with a magnetic stir bar, a thermocouple, a gas inlet tube, and a septum were added (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv), $CH_2Cl_2$ (5 mL), and (*E*)-cinnamaldehyde (126 μL, 1.0 mmol). The solution was cooled to −78 °C (internal temp.) in a dry ice-acetone bath. After diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv) and $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv) were added to the flask via syringe, a solution of **4e** (332 mg, 1.2 mmol, 1.2 equiv) in $CH_2Cl_2$ (5 mL) was added dropwise via syringe over 15 min. The internal temperature was kept below −70 °C during the addition of **4e**. The reaction mixture was stirred for additional 15 min at −78 °C before a mixture of MeOH (1 mL), $Et_3N$ (1 mL), and $CH_2Cl_2$ (5 mL) was added. The resulting solution was transferred into a 125-mL Erlenmeyer flask containing a sat. aq. $NaHCO_3$ solution (10 mL) and a sat. aq. KF solution (10 mL). The biphasic

mixture was stirred vigorously for 2 h at room temperature. The mixture was filtered through a glass frit and the filtrate was transferred to a 125-mL separatory funnel where the organic layer was separated. The aqueous layer was extracted with $CH_2Cl_2$ (2 × 20 mL). The combined organic extracts were dried over $Na_2SO_4$ (15 g), filtered, and concentrated *in vacuo* (23 °C, 30 mmHg). The residue was purified by column chromatography (22 mm diam., hexane/EtOAc, 5/1) on silica gel (20 g) to give **6ep** (302 mg, 90%) as a colorless oil and **16ep** (20 mg, 6%) as a colorless oil. The *syn*/*anti* ratio of **6ep** was determined to be 99/1 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture. The diastereomeric ratio of **16ep** was determined to be 63/37 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*R*,3*S*,4*E*)-**6ep**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.37-7.36 (m, 2 H, HC(7)), 7.33-7.30 (m, 2 H, HC(8)), 7.26-7.23 (m, 1 H, HC(9)), 6.68 (dd, *J* = 15.9, 1.2, 1 H, HC(5)), 6.22 (dd, *J* = 15.9, 5.9, 1 H, HC(4)), 4.56 (ddd, *J* = 5.9, 3.4, 1.2, 1 H, HC(3)), 4.28 (d, *J* = 3.4, 1 H, HC(2)), 3.76 (s, 3 H, HC(10)), 2.64 (bs, 1 H, OH), 0.92 (s, 9 H, HC(13)), 0.12 (s, 3 H, HC(11)), 0.06 (s, 3 H, HC(11))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

171.9 (C(1)), 136.4 (C(6)), 131.8 (C(5)), 128.6 (C(8)), 128.0 (C(4) or C(9)), 127.8 (C(4) or C(9)), 126.5 (C(7)), 75.4 (C(2)), 74.1 (C(3)), 52.1 (C(10)), 25.6 (C(13)), 18.3 (C(12)), −5.0 (C(11)), −5.4 (C(11))

IR: (neat)

3505 (br), 3027 (w), 2953 (m), 2930 (m), 2887 (m), 2857 (m), 1759 (s), 1496 (m), 1472 (m), 1463 (m), 1449 (m), 1437 (m), 1389 (m), 1362 (m), 1257 (s), 1203 (m), 1143 (s), 1044 (m), 1007 (m), 968 (m), 864 (s), 837 (s), 781 (s), 746

(m), 694 (m)

MS: (ESI)

177.1 (10), 187.1 (8), 319.2 (100), 320.2 (9), 354.2 (19), 359.2 ($M^+$+Na, 19)

HRMS: calcd for $C_{18}H_{28}O_4SiNa$: 359.1655, found 359.1646

TLC: $R_f$ 0.28 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

SFC: (2*R*,3*S*,4*E*)-**6ep**, $t_R$ 4.29 min (97.4%); (2*S*,3*R*,4*E*)-**6ep**, $t_R$ 5.46 min (2.6%) (Chiralpak AS, 125 bar, 40 °C, 1.0% MeOH in $CO_2$, 2.5 mL/min, 220 nm)

Opt. Rot.: $[\alpha]_D^{24}$ 5.7 (c = 4.5, EtOH)

Analysis: $C_{18}H_{28}O_4Si$ (336.50)

| | | |
|---|---|---|
| Calcd: | C, 64.25%; | H, 8.39% |
| Found: | C, 64.31%; | H, 8.52% |

Data for **16ep**:

$^1$H NMR: (500 MHz, $CDCl_3$)

9.67 (dd, *J* = 1.7, 1.7, 1 H, HC(10)), 7.31-7.20 (m, 5 H, HC(5), HC(6), HC(7)), 4.26 (d, *J* = 4.9, 1 H, HC(2)), 3.74-3.67 (m, 1 H, HC(3)), 3.61 (s, 3 H, HC(8)), 2.98 (ddd, *J* = 17.6, 5.9, 1.5, 1 H, HC(9)), 2.90 (ddd, *J* = 17.6, 8.3, 2.0, 1 H, HC(9)), 0.85 (s, 9 H, HC(13)), –0.10 (s, 3 H, HC(11)), –0.20 (s, 3 H, HC(11))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

200.9 (C(10)), 172.7 (C(1)), 139.8 (C(4)), 128.5 (C(5) or C(6)), 128.3 (C(5) or C(6)), 127.3 (C(7)), 76.3 (C(2)), 51.8 (C(8)), 43.9 (C(3)), 43.7 (C(9)), 25.6 (C(13)), 18.2 (C(12)), –5.5 (C(11)), –5.7 (C(11))

IR: (neat)

3031 (w), 2953 (s), 2930 (s), 2895 (m), 2858 (s), 1754 (s), 1728 (s), 1604 (w),

1496 (m), 1472 (m), 1463 (m), 1455 (m), 1436 (m), 1390 (m), 1362 (m), 1258 (s), 1203 (m), 1138 (s), 1007 (m), 913 (m), 838 (s), 780 (s), 734 (s), 700 (s)

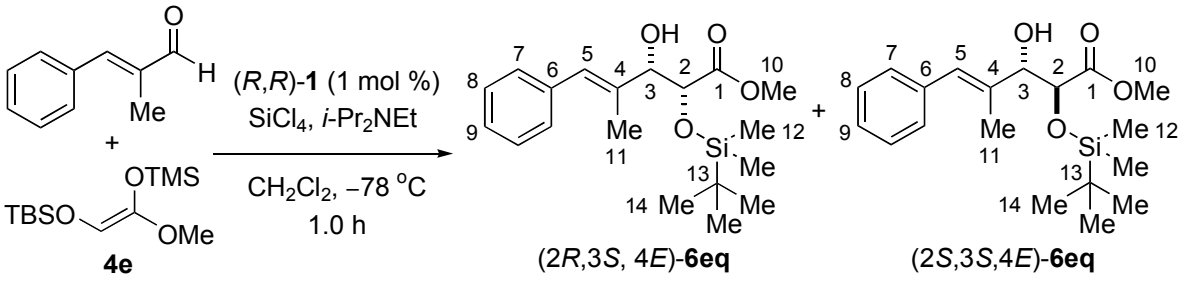
MS: (ESI)

177.1 (56), 205.1 (45), 277.2 (11), 337.2 ($M^+$+Na, 100), 338.2 (12)

HRMS: calcd for $C_{18}H_{28}O_4SiNa$: 337.1835, found 337.1819

TLC: $R_f$ 0.45 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

**Preparation of (2*R*,3*S*,4*E*)-2-[[(1,1-Dimethylethyl)dimethylsilyl]oxy]-3-hydroxy-4-methyl-5-phenyl-4-pentenoic Acid Methyl Ester ((2*R*,3*S*,4*E*)-6eq) and (2*S*,3*S*,4*E*)-2-[[(1,1-Dimethylethyl)dimethylsilyl]oxy]-3-hydroxy-4-methyl-5-phenyl-4-pentenoic Acid Methyl Ester ((2*S*,3*S*,4*E*)-6eq) (Table 8, entry 2)**




Following General Procedure 9, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined with (*E*)-α-methylcinnamaldehyde (140 μL, 1.0 mmol), diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4e** (332 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 10/1 to 5/1) on silica gel (30 g), (2*R*,3*S*,4*E*)-**6eq** (272 mg, 78%) as a colorless oil and (2*S*,3*S*,4*E*)-**6eq** (56 mg, 16%) as white crystals. The *syn*/*anti* ratio of **6ep** was determined to be 83/17 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*R*,3*S*,4*E*)-**6eq**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.34-7.31 (m, 2 H, HC(8)), 7.25-7.24 (m, 2 H, HC(7)), 7.23-7.20 (m, 1 H, HC(9)), 6.56 (bs, 1 H, HC(5)), 4.37-4.35 (m, 2 H, HC(2), HC(3)), 3.76 (s, 3 H, HC(10)), 2.82 (bs, 1 H, OH), 1.89 (d, $J$ = 1.2, 3 H, HC(11)), 0.91 (s, 9 H, HC(14)), 0.10 (s, 3 H, HC(12)), 0.05 (s, 3 H, HC(12))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

172.3 (C(1)), 137.4 (C(6)), 135.8 (C(4)), 128.9 (C(7)), 128.1 (C(8)), 126.8 (C(5)), 126.5 (C(9)), 77.9 (C(3)), 73.7 (C(2)), 52.1 (C(10)), 25.6 (C(14)), 18.3 (C(13)), 14.8 (C(11)), −5.0 (C(12)), −5.4 (C(12))

IR: (neat)

3546 (br), 3057 (m), 3025 (m), 2953 (s), 2930 (s), 2896 (m), 2858 (s), 1760 (s), 1600 (m), 1493 (m), 1472 (m), 1464 (m), 1437 (m), 1390 (m), 1362 (m), 1256 (s), 1200 (s), 1142 (s), 1074 (s), 1009 (m), 985 (m), 915 (m), 839 (s), 781 (s), 699 (s)

MS: (ESI)

189.1 (11), 216.9 (7), 254.8 (8), 256.8 (10), 273.1 (18), 373.2 ($M^+$+Na, 100), 374.2 (8)

HRMS: calcd for $C_{19}H_{30}O_4SiNa$: 373.1811, found 373.1807

TLC: $R_f$ 0.37 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

SFC: (2*S*,3*R*,4*E*)-**6eq**, $t_R$ 2.73 min (24.7%); (2*R*,3*S*,4*E*)-**6eq**, $t_R$ 3.33 min (75.3%) (Chiralcel OJ, 125 bar, 40 °C, 0.5% MeOH in $CO_2$, 3.0 mL/min, 220 nm)

Opt. Rot.: $[\alpha]_D^{24}$ 10.3 (c = 2.0, EtOH)

Analysis: $C_{19}H_{30}O_4Si$ (350.52)

Calcd: C, 65.10%; H, 8.63%

Found: C, 65.03%; H, 8.76%

Data for (2*S*,3*S*,4*E*)-**6eq**:

mp: 70-71 °C (hexane)

$^1$H NMR: (500 MHz, $CDCl_3$)

7.34-7.31 (m, 2 H, HC(8)), 7.26-7.24 (m, 2 H, HC(7)), 7.23-7.21 (m, 1 H, HC(9)), 6.57 (bs, 1 H, HC(5)), 4.39 (s, 2 H, HC(2), HC(3)), 3.72 (s, 3 H, HC (10)), 2.74 (bs, 1 H, OH), 1.90 (d, $J$ = 1.5, 3 H, HC(11)), 0.91 (s, 9 H, HC(14)), 0.10 (s, 3 H, HC(12)), 0.08 (s, 3 H, HC(12))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

171.9 (C(1)), 137.3 (C(6)), 135.6 (C(4)), 129.0 (C(7)), 128.1 (C(8)), 128.0 (C(5)), 126.5 (C(9)), 78.4 (C(3)), 74.4 (C(2)), 51.8 (C(10)), 25.6 (C(14)), 18.2 (C(13)), 14.1 (C(11)), −5.1 (C(12)), −5.4 (C(12))

IR: (neat)

3484 (br), 3057 (w), 3025 (m), 2953 (s), 2930 (s), 2895 (m), 2858 (s), 1745 (s), 1600 (m), 1493 (s), 1472 (m), 1463 (m), 1438 (m), 1390 (m), 1362 (m), 1254 (s), 1199 (s), 1173 (s), 1131 (s), 1045 (m), 918 (m), 839 (s), 780 (s), 699 (s)

MS: (ESI)

201.1 (12), 373.2 ($M^+$+Na, 100), 374.2 (18)

HRMS: calcd for $C_{19}H_{30}O_4SiNa$: 373.1811, found 373.1824

TLC: $R_f$ 0.22 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

SFC: (2*R*,3*R*,4*E*)-**6eq**, $t_R$ 4.12 min (3.6%); (2*S*,3*S*,4*E*)-*anti*-**6eq**, $t_R$ 4.45 min (96.4%)

(Chiralpak AD, 125 bar, 40 °C, 3.0% MeOH in $CO_2$, 3.0 mL/min, 220 nm)

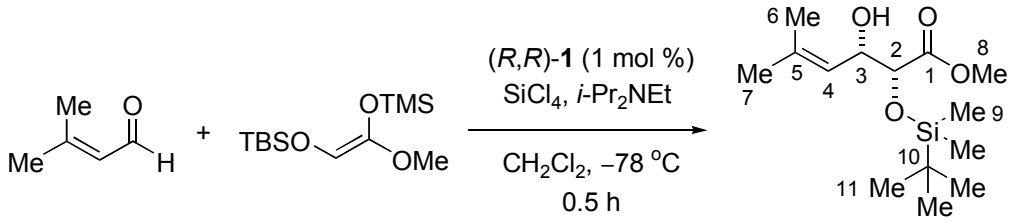
Opt. Rot.: $[\alpha]_D^{24}$ 73.1 (c = 2.2, EtOH)

Analysis: $C_{19}H_{30}O_4Si$ (350.52)

Calcd: C, 65.10%; H, 8.63%

Found: C, 65.11%; H, 8.87%

**Preparation of (2*R*,3*S*)-2-[[(1,1-Dimethylethyl)dimethylsilyl]oxy]-3-hydroxy-5-methyl-4-hexenoic Acid Methyl Ester ((2*R*,3*S*)-6er) (Table 8, entry 3)**




**4e** (2*R*,3*S*)-**6er**

Following General Procedure 9, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined with β-methylcrotonaldehyde (96.5 μL, 1.0 mmol), diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4e** (332 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 5/1) on silica gel (25 g), **6er** (238 mg, 82%) as a colorless oil. The *syn*/*anti* ratio of **6er** was determined to be >99/1 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture. The 1,4-adduct was not isolated.

Data for (2*R*,3*S*)-**6er**:

$^1$H NMR: (500 MHz, $CDCl_3$)

5.22 (m, 1 H, HC(4)), 4.55 (dd, *J* = 8.8, 4.4, 1 H, HC(3)), 4.08 (d. *J* = 4.4, 1 H, HC(2)), 3.73 (s, 3 H, HC(8)), 2.39 (bs, 1 H, OH), 1.73 (d, *J* = 1.2, 3 H, HC(7)), 1.69 (d, *J* = 1.5, 3 H, HC(6)), 0.92 (s, 9 H, HC(11)), 0.10 (s, 3 H, HC(9)), 0.07 (s, 3 H, HC(9))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

172.0 (C(1)), 137.3 (C(5)), 123.1 (C(4)), 76.0 (C(2)), 70.3 (C(3)), 51.9 (C(8)), 25.8 (C(7)), 25.7 (C(11)), 18.37 (C(6)), 18.35 (C(10)), −5.0 (C(9)), −5.5 (C(9))

IR: (neat)

3491 (br), 2954 (s), 2931 (s), 2888 (m), 2858 (s), 1759 (s), 1677 (w), 1473 (m), 1464 (m), 1437 (m), 1390 (m), 1378 (m), 1362 (m), 1256 (s), 1202 (s), 1142 (s), 1044 (s), 1007 (m), 939 (m), 864 (s), 838 (s), 807 (m), 781 (s), 672 (m)

MS: (ESI)

227.2 (8), 271.2 (51), 311.2 ($M^+$+Na, 100), 312.2 (8)

HRMS: calcd for $C_{14}H_{28}O_4SiNa$: 311.1655, found 311.1669

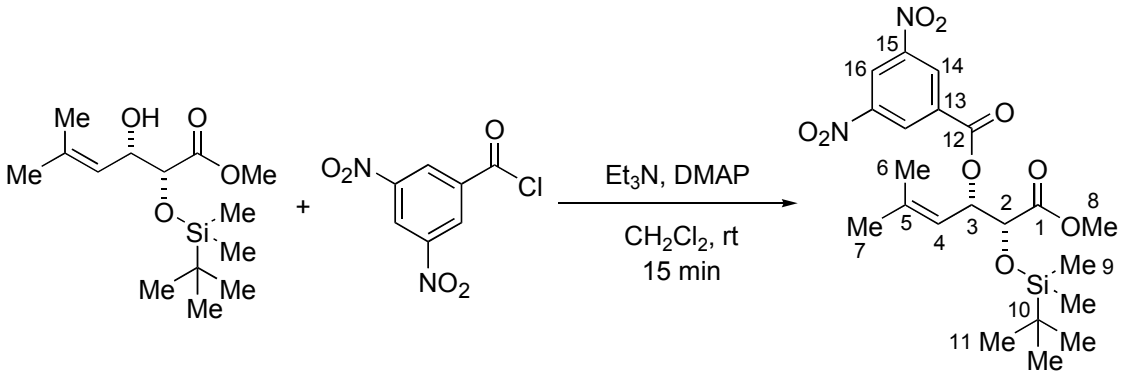
TLC: $R_f$ 0.36 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

Opt. Rot.: $[\alpha]_D^{24}$ 16.5 (c = 1.0, EtOH)

Analysis: $C_{14}H_{28}O_4Si$ (288.46)

| | | |
|---|---|---|
| Calcd: | C, 58.29%; | H, 9.78% |
| Found: | C, 58.31%; | H, 9.90% |

**(2*R*,3*S*)-2-[[(1,1-Dimethylethyl)dimethylsilyl]oxy]-3-(3,5-dinitrobenzoyloxy)-5-methyl-4-hexenoic Acid Methyl Ester (2*R*,3*S*)-20 (Table 8, entry 3)**




(2*R*,3*S*)-**6er** (2*R*,3*S*)-**19**

To a flame-dried, 25-mL, 2-necked round-bottomed flask fitted with a magnetic stir bar, a gas inlet tube, and a septum were added **6er** (144.2 mg, 0.5 mmol), DMAP (12.2 mg, 0.1 mmol, 0.2 equiv), $CH_2Cl_2$ (3 mL), $Et_3N$ (139 μL, 1.0 mmol, 2.0 equiv). Then, a solution of 3,5-dinitrobenzoyl chloride (230.6 mg, 1.0 mmol, 2.0 equiv) in $CH_2Cl_2$ (7 mL) was added dropwise via syringe at rt. After 15 min, the reaction mixture was washed with sat. aq. $NH_4Cl$ slution (15 mL) and sat. aq. $NaHCO_3$ solution (15 mL). The organic layer was dried over $MgSO_4$ (1 g), then was filtered, and concentrated *in vacuo* (23 $^oC$, 30 mmHg). The residue was purified by column chromatography (22 mm diam., packed with hexane, loaded with $CH_2Cl_2$, eluted with hexane/EtOAc, 10/1) on silica gel (15 g) and recrystallized from hot hexane to give **20** (220 mg, 97%) as white crystals. The enantiomeric ratio was measured prior to recrystallization.

Data for (2*R*,3*S*)-**20**:

$^1$H NMR: (500 MHz, $CDCl_3$)

9.22-9.21 (m, 1 H, HC(16)), 9.14-9.14 (m, 2 H, HC(14)), 6.03 (dd, *J* = 9.7, 5.4, 1 H, HC(3)), 5.32 (m, 1 H, HC(4)), 4.40 (d, *J* = 5.4, 1 H, HC(2)), 3.72 (s, 3 H, HC(8)), 1.85 (d, *J* = 1.3, 3 H, HC(6)), 1.78 (d, *J* = 1.0, 3 H, HC(7)), 0.89 (s, 9 H,

HC(11)), 0.10 (s, 3 H, HC(9)), 0.05 (s, 3 H, HC(9))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

170.8 (C(1)), 161.5 (C(12)), 148.6 (C(15)), 141.9 (C(5)), 134.1 (C(13)), 129.5 (C(14)), 122.3 (C(16)), 117.7 (C(4)), 75.0 (C(3)), 74.3 (C(2)), 52.1 (C(8)), 25.9 (C(7)), 25.5 (C(11)), 18.7 (C(6)), 18.2 (C(10)), −5.1 (C(9)), −5.4 (C(9))

IR: (neat)

3442 (br), 3102 (m), 2952 (m), 2933 (m), 2857 (m), 1737 (s), 1629 (m), 1548 (s), 1459 (m), 1445 (m), 1345 (s), 1274 (s), 1210 (m), 1165 (s), 1136 (m), 1076 (m), 1023 (m), 1005 (m), 956 (m), 920 (m), 867 (m), 838 (s), 782 (s), 731 (s), 721 (s)

MS: (ESI)

271.2 (30), 293.2 (8), 311.2 (25), 312.2 (7), 349.0 (6), 500.2 (34), 505.2 ($M^+$+Na, 100), 506.2 (26), 507.2 (8), 536.2 (6)

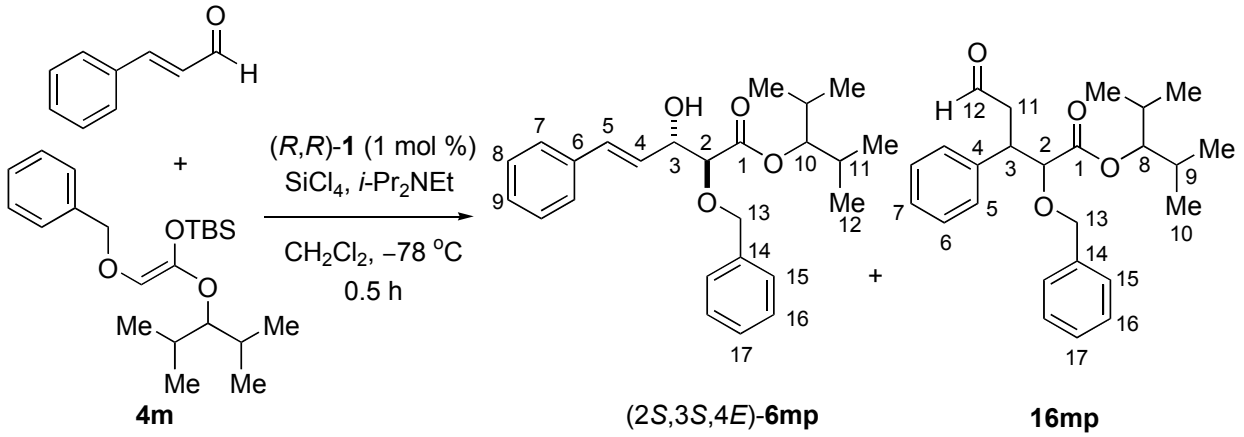
HRMS: calcd for $C_{21}H_{30}N_2O_9SiNa$: 505.1618, found 505.1597

TLC: $R_f$ 0.32 (hexane/EtOAc, 10/1) [UV(254)/$KMnO_4$]

SFC: (2*S*,3*R*)-**20**, $t_R$ 3.78 min (4.7%); (2*R*,3*S*)-**20**, $t_R$ 4.55 min (95.3%)

(Chiralcel OJ, 125 bar, 40 $^oC$, 0.5% MeOH in $CO_2$, 2.0 mL/min, 220 nm)

**Preparation of (2*S*,3*S*,4*E*)-3-Hydroxy-5-phenyl-2-(phenylmethoxy)-4-pentenoic Acid 2-Methyl-1-(1-methylethyl)propyl Ester ((2*S*,3*S*,4*E*)-6mp) and 5-Oxo-3-phenyl-2-(phenylmethoxy)pentanoic Acid 2-Methyl-1-(1-methylethyl)propyl Ester (16mp) (Table 9, entry 1)**




Following General Procedure 9, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined with (*E*)-cinnamaldehyde (126 μL, 1.0 mmol), diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4m** (454 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 10/1 to 5/1) on silica gel (20 g), **6mp** (358 mg, 90%) as a colorless oil and **16mp** (15 mg, 4%) as white crystals. The *syn*/*anti* ratio of **6mp** was determined to be 1/99 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture. The diastereomeric ratio of **16mp** was determined to be 96/4 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*S*,3*S*,4*E*)-**6mp**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.38-7.29 (m, 9 H, HC(7), HC(8), HC(15), HC(16), HC(17)), 7.25-7.22 (m, 1 H, HC(9)), 6.69 (dd, *J* = 15.9, 1.2, 1 H, HC(5)), 6.30 (dd, *J* = 15.9, 6.6, 1 H, HC(4)), 4.89 (d, *J* = 11.6, 1 H, HC(13)), 4.74 (dd, *J* = 6.2, 6.2, 1 H, HC(10)), 4.65 (ddd, *J* = 6.6, 4.8, 1.2, 1 H, HC(3)), 4.51 (d, *J* = 11.6, 1 H, HC(13)), 4.14 (d, *J* = 4.8, 1

H, HC(2)), 2.59 (bs, 1 H, OH), 1.98-1.89 (m, 2 H, HC(11)), 0.90 (d, $J$ = 6.8, 6 H, HC(12)), 0.88 (d, $J$ = 6.8, 6 H, HC(12))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

170.3 (C(1)), 137.1 (C(14)), 136.5 (C(6)), 132.9 (C(5)), 128.5 (C(8), C(15), or C(16)), 128.4 (C(8), C(15), or C(16)), 128.2 (C(8), C(15), or C(16)), 128.1 (C(9) or C(17)), 127.7 (C(9) or C(17)), 126.7 (C(4)), 126.6 (C(7)), 84.3 (C(10)), 81.1 (C(2)), 73.03 (C(3) or C(13)), 73.00 (C(3) or C(13)), 29.4 (C(11)), 29.3 (C(11)), 19.6 (C(12)), 19.5 (C(12)), 17.4 (C(12)), 17.3 (C(12))

IR: (neat)

3447 (br), 3027 (m), 2965 (s), 2935 (s), 2875 (m), 1734 (s), 1496 (m), 1453 (m), 1338 (m), 1371 (m), 1272 (m), 1202 (s), 1129 (s), 1097 (s), 1028 (m), 1000 (m), 967 (s), 893 (m), 749 (s), 695 (s)

MS: (ESI)

281.1 (17), 419.2 ($M^+$+Na, 100), 420.2 (27), 498.3 (15)

HRMS: calcd for $C_{25}H_{32}O_4Na$: 419.2198, found 419.2192

TLC: $R_f$ 0.32 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

SFC: (2*S*,3*S*,4*E*)-**6mp**, $t_R$ 4.21 min (98.0%); (2*R*,3*R*,4*E*)-**6mp**, $t_R$ 4.89 min (2.0%) (Chiralcel OD, 125 bar, 40 $^oC$, 10.0% MeOH in $CO_2$, 3.0 mL/min, 220 nm)

Opt. Rot.: $[\alpha]_D^{24}$ –9.9 (c = 2.0 , EtOH)

Analysis: $C_{25}H_{32}O_4$ (396.52)

| | | |
|---|---|---|
| Calcd: | C, 75.73%; | H, 8.13% |
| Found: | C, 75.74%; | H, 8.03% |

Data for **16mp**:

$^{1}$H NMR: (500 MHz, $CDCl_3$)

9.64 (dd, *J* = 1.8, 1.8, 1 H, HC(12)), 7.36-7.20 (m, 10 H, Aryl), 4.72 (d, *J* = 11.4, 1 H, HC(13)), 4.61 (dd, *J* = 6.0, 6.0, 1 H, HC(8)), 4.37 (d, *J* = 11.4, 3 H, HC(13)), 4.14 (d, *J* = 6.1, 1 H, HC(2)), 3.80-3.76 (m, 1 H, HC(3)), 2.94 (ddd, *J* = 17.4, 8.8, 1.8, 1 H, HC(11)), 2.88 (ddd, *J* = 17.4, 5.5, 1.8, 1 H, HC(11)), 1.92-1.78 (m, 2 H, HC(9)), 0.83 (d, *J* = 6.8, 3 H, HC(10)), 0.80 (d, *J* = 6.6, 3 H, HC(10)), 0.74 (d, *J* = 6.9, 3 H, HC(10)), 0.71 (d, *J* = 6.5, 3 H, HC(10))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

200.7 (C(12)), 170.9 (C(1)), 139.9 (C(4)), 136.9 (C(14)), 128.7 (Aryl), 128.43 (Aryl), 128.36 (Aryl), 128.2 (Aryl), 127.9 (C(7) or C(17)), 127.4 (C(7) or C(17)), 84.2 (C(8)), 81.9 (C(2)), 72.7 (C(13)), 45.6 (C(11)), 42.7 (C(3)), 29.3 (C(9)), 29.2 (C(9)), 19.5 (C(10)), 19.4 (C(10)), 17.5 (C(10)), 17.0 (C(10))

IR: (neat)

3064 (w), 3032 (w), 2966 (s), 2936 (m), 2876 (m), 2725 (w), 1726 (s), 1604 (w), 1497 (m), 1455 (m), 1389 (m), 1371 (m), 1340 (m), 1269 (m), 1237 (m), 1200 (s), 1127 (m), 1095 (m), 1028 (m), 1000 (m), 970 (m), 895 (m), 746 (m), 699 (s)

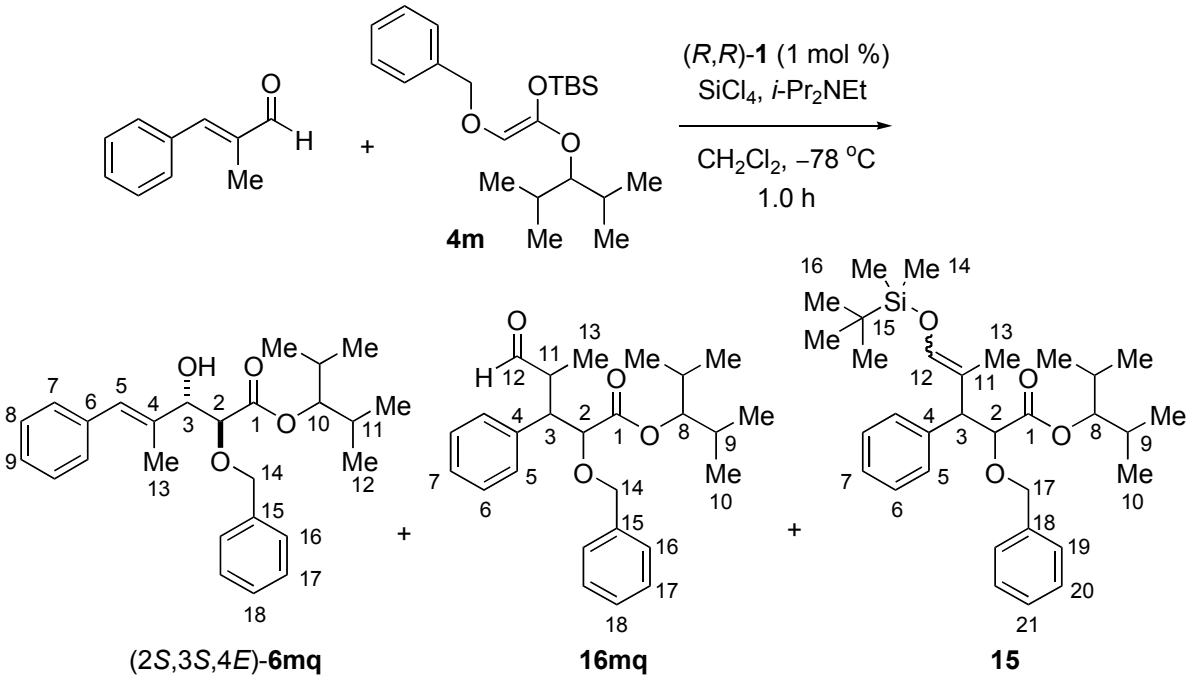
MS: (ESI)

91.1 (11), 130.2 (11), 191.1 (100), 192.1 (8), 217.1 (8), 363.1 (8), 281.1 (13), 289.2 (43), 297.1 (12), 397.2 (9), 413.2 (8), 414.3 (11), 419.2 ($M^+$+Na, 14), 440.2 (12), 446.3 (56), 451.2 (30), 451.2 (30)

HRMS: calcd for $C_{25}H_{32}O_4Na$: 419.2198, found 419.2191

TLC: $R_f$ 0.49 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

**Preparation of (2*S*,3*S*,4*E*)-3-Hydroxy-4-methyl-5-phenyl-2-(phenylmethoxy)-4-pentenoic Acid 2-Methyl-1-(1-methylethyl)propyl Ester ((2*S*,3*S*,4*E*)-6mq), 4-Methyl-5-oxo-3-phenyl-2-(phenylmethoxy)pentanoic Acid 2-Methyl-1-(1-methylethyl)propyl Ester (16mq), and 5-[[(1,1-Dimethylethyl)dimethylsilyl]oxy]-4-methyl-3-phenyl-2-(phenylmethoxy)-4-pentenoic Acid 2-Methyl-1-(1-methylethyl)propyl Ester (Table 9, entry 2)**




Following General Procedure 9, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined with (*E*)-α-methylcinnamaldehyde (139.6 μL, 1.0 mmol), diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4m** (454 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 20/1 to 10/1 to 5/1) on silica gel (25 g), **6mq** (286 mg, 70%) as a colorless oil, **16mq** (19 mg, 5%) as a colorless oil, and **15** (75 mg, 14%) as a colorless oil. The *syn*/*anti* ratio of **6mq** was determined to be >1/99 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture. The diastereomeric ratio of **16mq** was determined to be 87/6/4/3 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture. The diastereomeric ratio of **15** was determined to be 72/24/4 by $^1$H NMR (500 MHz) analysis of the

crude reaction mixture.

Data for (2*S*,3*S*,4*E*)-**6mq**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.35-7.29 (m, 7 H, HC(8), HC(16), HC(17), HC(18)), 7.28-7.25 (m, 2 H, HC(7)), 7.24-7.21 (m, 1 H, HC(9)), 6.65 (bs, 1 H, HC(5)), 4.84 (d, *J* = 11.5, 1 H, HC (14)), 4.76 (dd, *J* = 6.1, 6.1, 1 H, HC(10)), 4.50 (dd, *J* = 6.5, 0.7, 1 H, HC(3)), 4.48 (d, *J* = 11.5, 1 H, HC(14)), 4.13 (d, *J* = 6.5, 1 H, HC(2)), 2.75 (bs, 1 H, OH), 2.02-1.89 (m, 2 H, HC(11)), 1.82 (d, *J* = 1.3, 3 H, HC(13)), 0.932 (d, *J* = 6.8, 3H, HC(12)), 0.927 (d, *J* = 6.8, 3 H, HC(12)), 0.91 (d, *J* = 7.1, 3 H, HC(12)), 0.90 (d, *J* = 6.8, 3 H, HC(12))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

171.2 (C(1)), 137.3 (C(6) or C(15)), 137.1 (C(6) or C(15)), 135.5 (C(4)), 129.1 (C(7)), 128.6 (C(5)), 128.4 (C(8), C(16), or C(17)), 128.1 (C(8), C(16), or C(17)), 127.98 (C(8), C(16), or C(17)), 127.97 (C(18)), 126.5 (C(9)), 84.4 (C(10)), 79.7 (C(2)), 76.7 (C(3)), 72.9 (C(14)), 29.42 (C(11)), 29.36 (C(11)), 19.7 (C(12)), 19.5 (C(12), 17.6 (C(12)), 17.3 (C(12)), 14.0 (C(13))

IR: (neat)

3484 (br), 3062 (w), 2966 (s), 2936 (s), 2876 (m), 1736 (s), 1600 (w), 1495 (m), 1464 (m), 1454 (m), 1390 (s), 1372 (s), 1340 (m), 1323 (m), 1264 (s), 1235 (s), 1199 (s), 1117 (s), 1098 (s), 1028 (s), 1000 (s), 973 (s), 894 (m), 747 (s), 698 (s)

MS: (ESI)

102.1 (10), 277.1 (20), 295.1 (100), 296.1 (9), 393.2 (29), 433.2 ($M^+$+Na, 48), 434.2 (7), 512.4 (18)

HRMS: calcd for $C_{26}H_{34}O_4Na$: 433.2355, found 433.2342

TLC: $R_f$ 0.41 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

SFC: (2*S*,3*S*,4*E*)-**6mq**, $t_R$ 4.48 min (89.1%); (2*R*,3*R*,4*E*)-**6mq**, $t_R$ 5.50 min (10.9%) (Chiralcel OJ, 125 bar, 40 °C, 5.0% MeOH in $CO_2$, 3.0 mL/min, 220 nm)

Opt. Rot.: $[\alpha]_D^{24}$ 42.2 (c = 2.0, EtOH)

Analysis: $C_{26}H_{34}O_4$ (410.55)

| | | |
|---|---|---|
| Calcd: | C, 76.06%; | H, 8.35% |
| Found: | C, 76.14%; | H, 8.25% |

Data for **16mq**:

$^1$H NMR: (500 MHz, $CDCl_3$)

9.73 (d, *J* = 0.5, 1 H, HC(12)), 7.39-7.27 (m, 7 H, Aryl), 7.25-7.18 (m, 3 H, Aryl), 4.72 (d, *J* = 11.0, 1 H, HC(14)), 4.53 (dd, *J* = 6.0, 6.0, 1 H, HC(8)), 4.46 (d, *J* = 9.8, 1 H, HC(2)), 4.46 (d, *J* = 11.0, 1 H, HC(14)), 3.75 (dd, *J* = 9.8, 3.7, 1 H, HC(3)), 2.69 (ddd, *J* = 14.1, 7.0, 3.7, 1 H, HC(11)), 1.83-1.69 (m, 2 H, HC(9)), 0.99 (d, *J* = 7.0, 3 H, HC(13)), 0.69 (d, *J* = 6.6, 3 H, HC(10)), 0.652 (d, *J* = 6.9, 3 H, HC(10)), 0.648 (d, *J* = 6.8, 3 H, HC(10)), 0.63 (d, *J* = 6.5, 3 H, HC(10))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

203.2 (C(12)), 170.8 (C(1)), 138.1 (C(4)), 136.5 (C(15)), 129.0 (Aryl), 128.7 (Aryl), 128.6 (Aryl), 128.4 (Aryl), 128.1 (C(7) or C(18)), 127.4 (C(7) or C(18)), 84.3 (C(8)), 79.1 (C(2)), 72.9 (C(14)), 49.4 (C(3) or C(11)), 49.2 (C(3) or C(11)), 29.3 (C(9)), 29.2 (C(9)), 19.3 (C(10)), 19.2 (C(10)), 17.1 (2 × (C(10)), 9.5 (C(13))

IR: (neat)

3031 (w), 2966 (s), 2935 (m), 2876 (m), 1737 (s), 1722 (s), 1497 (w), 1463 (m), 1454 (m), 1389 (m), 1371 (m), 1338 (w), 1269 (m), 1201 (m), 1181 (m), 1127 (m), 1097 (m), 1073 (m), 1029 (m), 999 (m), 967 (m), 945 (m), 892 (m), 754 (m), 700 (s)

MS: (ESI)

91.1 (12), 131.1 (8), 159.1 (11), 205.1 (66), 206.1 (8), 231.1 (6), 295.1 (16), 303.2 (57), 311.1 (11), 417.3 (30), 428.3 (100), 433.2 ($M^+$+Na, 50), 434.2 (13), 449.2 (10)

HRMS: calcd for $C_{26}H_{34}O_4Na$: 433.2355, found 433.2365

TLC: $R_f$ 0.48 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

Data for **15**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.39-7.19 (m, 10 H, Aryl), 6.45 (bs, 1 H, HC(12)), 4.76 (d, *J* = 11.6, 1 H, HC(17)), 4.62 (dd, *J* = 6.0, 6.0, 1 H, HC(8)), 4.58 (d, *J* = 11.6, 1 H, HC(17)), 4.47 (dd, *J* = 7.6, 0.8, 1 H, HC(3)), 4.11 (d, *J* = 7.6, 1 H, HC(2)), 1.96-1.86 (m, 2 H, HC(9)), 1.90 (d, *J* = 1.2, 3 H, HC(13)), 0.89 (s, 9 H, HC(16)), 0.86 (d, *J* = 6.8, 3 H, HC(10)), 0.85 (d, *J* = 6.6, 3 H, HC(10)), 0.79 (d, *J* = 6.8, 3 H, HC(10)), 0.77 (d, *J* = 6.8, 3 H, HC(10)), 0.07 (s, 3 H, HC(14)), 0.05 (s, 3 H, HC(14))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

170.6 (C(1)), 137.7 (C(18)), 137.2 (C(4)), 136.5 (C(11)), 129.2 (C(12)), 129.0 (C(5)), 128.2 (C(6), C(19), or C(20)), 127.99 (C(6), C(19), or C(20)), 127.95 (C(6), C(19), or C(20)), 127.6 (C(7) or C(21)), 126.5 (C(7) or C(21)), 84.0

(C(8)), 82.6 (C(2)), 80.3 (C(3)), 73.2 (C(17)), 29.5 (C(9)), 29.3 (C(9)), 25.8 (C(16)), 19.7 (C(10)), 19.5 (C(10)), 18.2 (C(15)), 17.7 (C(10)), 17.6 (C(10)), 13.8 (C(13)), –4.7 (C(14)), –4.8 (C(14))

IR: (neat)

3028 (m), 2962 (s), 2930 (s), 2879 (s), 2857 (s), 1743 (s), 1496 (m), 1472 (s), 1463 (s), 1338 (m), 1371 (m), 1361 (m), 1256 (s), 1176 (s), 1115 (s), 1096 (s), 1078 (s), 1028 (m), 1004 (m), 971 (m), 882 (s), 837 (s), 778 (s), 735 (s), 698 (s)

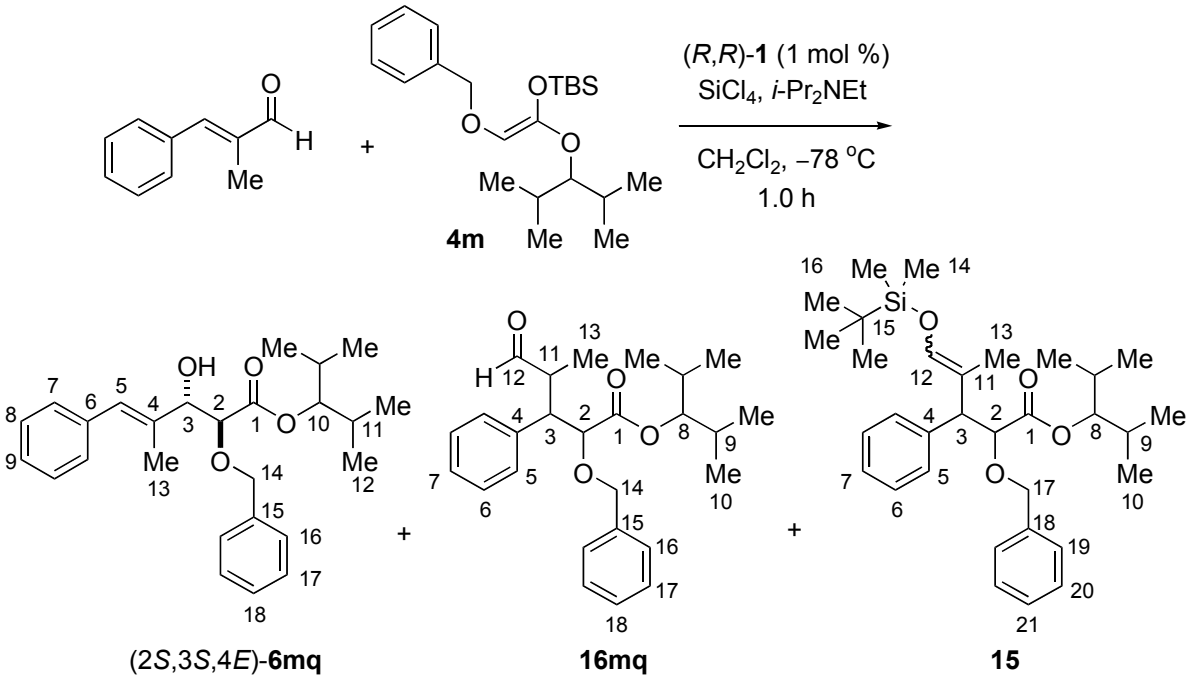
MS: (ESI)

91.1 (16), 102.1 (34), 212.1 (20), 231.1 (30), 249.1 (12), 277.1 (69), 295.1 (27), 401.2 (11), 542.3 (16), 547.3 ($M^+$+Na, 100), 548.3 (51), 563.3 (14)

HRMS: calcd for $C_{32}H_{48}O_4SiNa$: 547.3220, found 547.3238

TLC: $R_f$ 0.57 (hexane/EtOAc, 10/1) [UV(254)/$KMnO_4$]

**Preparation of (2*S*,3*S*)-3-Hydroxy-5-methyl-2-(phenylmethoxy)-4-hexenoic Acid 2-Methyl-1-(1-methylethyl)propyl Ester ((2*S*,3*S*)-6mr), (2*R*,3*S*)-3-Hydroxy-5-methyl-2-(phenyl methoxy)-4-hexenoic Acid 2-Methyl-1-(1-methylethyl)propyl Ester ((2*R*,3*S*)-6mr), and 3,3-Dimethyl-5-oxo-2-(phenylmethoxy)pentanoic Acid 2-Methyl-1-(1-methylethyl)propyl Ester (16mr) (Table 9, entry 3)**




Following General Procedure 9, (*R*,*R*)-**1** (8.4 mg, 0.01 mmol, 0.01 equiv) was combined with β-methylcrotonaldehyde (96.5 μL, 1.0 mmol), diisopropylethylamine (17.4 μL, 0.1 mmol, 0.1 equiv), $SiCl_4$ (126 μL, 1.1 mmol, 1.1 equiv), and **4m** (454 mg, 1.2 mmol, 1.2 equiv) to yield, after column chromatography (22 mm diam., hexane/EtOAc, 10/1 to 5/1) on silica gel (20 g), **6mr** (275 mg, 79%) as a colorless oil and **16mr** (22 mg, 6%) as a colorless oil. The *syn*/*anti* ratio of **6mr** was determined to be 22/78 by $^1$H NMR (500 MHz) analysis of the crude reaction mixture.

Data for (2*S*,3*S*)-**6mr**:

$^{1}$H NMR: (500 MHz, $CDCl_3$)

7.39-7.34 (m, 4 H, HC(13), HC(14)), 7.33-7.29 (m, 1 H, HC(15)), 5.40 (qqd, *J* = 8.8, 1.3, 1.3, 1 H, HC(4)), 4.87 (d, *J* = 11.5, 1 H, HC(11)), 4.70 (dd, *J* = 6.2, 6.2, 1 H, HC(8)), 4.69 (dd, *J* = 8.8, 4.4, 1 H, HC(3)), 4.49 (d, *J* = 1.5, 1 H, HC(11)), 4.09 (d, *J* = 4.4, 1 H, HC(2)), 2.21 (bs, 1 H, OH), 1.97-1.88 (m, 2 H, HC(9)), 1.71 (d, *J* = 1.3, 3 H, HC(7)), 1.69 (d, *J* = 1.3, 3 H, HC(6)), 0.893 (d, *J* = 6.9, 3 H, HC(10)), 0.887 (d, *J* = 6.8, 3 H, HC(10)), 0.88 (d, *J* = 6.9, 3 H, HC(10)), 0.87 (d, *J* = 6.6, 3 H, HC(10))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

170.4 (C(1)), 138.7 (C(5)), 137.4 (C(12)), 128.4 (C(14)), 128.1 (C(13)), 127.9 (C(15)), 122.1 (C(4)), 83.9 (C(8)), 81.2 (C(2)), 73.1 (C(11)), 68.9 (C(3)), 29.4 (C(9)), 29.3 (C(9)), 25.8 (C(7)), 19.5 (2 × C(10)), 18.4 (C(6)), 17.3 (C(10)), 17.2 (C(10))

IR: (neat)

3477 (br), 3031 (w), 2966 (s), 2934 (s), 2876 (s), 1732 (s), 1673 (w), 1497 (m), 1463 (m), 1455 (s), 1389 (s), 1372 (s), 1338 (m), 1266 (s), 1203 (s), 1182 (s), 1098 (s), 1028 (s), 1000 (m), 968 (s), 932 (m), 897 (m), 737 (m), 698 (s)

MS: (ESI)

331.2 (6), 371.2 ($M^+$+Na, 100), 372.2 (24)

HRMS: calcd for $C_{21}H_{32}O_4Na$: 371.2198, found 371.2195

TLC: $R_f$ 0.39 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

SFC: (2*R*,3*R*)-**6mr**, $t_R$ 5.08 min (3.9%); (2*S*,3*S*)-**6mr**, $t_R$ 6.17 min (96.1%)

(Chiralpak AD, 150 bar, 40 °C, 2.0% MeOH in $CO_2$, 3.0 mL/min, 220 nm)

Opt. Rot.: $[\alpha]_D^{24}$ –18.7 (c = 2.0, EtOH)

Analysis: $C_{21}H_{32}O_4$ (348.48)

| | | |
|---|---|---|
| Calcd: | C, 72.38%; | H, 9.26% |
| Found: | C, 72.00%; | H, 9.25% |

Data for (2*R*,3*S*)-**6mr**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.39-7.34 (m, 4 H, HC(13), HC(14)), 7.33-7.29 (m, 1 H, HC(15)), 5.30 (qqd, *J* = 8.8, 1.3, 1.3, 1 H, HC(4)), 4.82 (d, *J* = 11.5, 1 H, HC(11)), 4.71 (dd, *J* = 5.6, 5.6, 1 H, HC(8)), 4.63 (dd, *J* = 8.8, 5.7, 1 H, HC(3)), 4.48 (d, *J* = 11.2, 1 H, HC(11)), 3.89 (d, *J* = 5.7, 1 H, HC(2)), 2.21 (bs, 1 H, OH), 1.97-1.88 (m, 2 H, HC(9)), 1.72 (d, *J* = 1.3, 3 H, HC(7))

HC(9) and 4 × HC(10) could not be clearly assigned because they were under the peaks of the major isomer.

$^{13}$C NMR: (126 MHz, $CDCl_3$)

170.5 (C(1)), 138.8 (C(5)), 137.0 (C(12)), 128.4 (C(14)), 128.2 (C(13)), 128.1 (C(15)), 122.5 (C(4)), 84.0 (C(8)), 81.8 (C(2)), 72.8 (C(11)), 69.0 (C(3)), 29.4 (C(9)), 29.3 (C(9)), 25.9 (C(7)), 19.5 (2 × C(10)), 18.4 (C(6)), 17.6 (C(10)), 16.9 (C(10))

Data for **16mr**:

$^1$H NMR: (500 MHz, $CDCl_3$)

9.78 (dd, *J* = 3.4, 2.2, 1 H, HC(9)), 7.38-7.27 (m, 5 H, HC(12), HC(13), HC (14)), 4.77 (dd, *J* = 5.9, 5.9, 1 H, HC(5)), 4.67 (d, *J* = 11.2, 1 H, HC(10)), 4.30

(d, $J$ = 11.2, 1 H, HC(10)), 3.84 (s, 1 H, HC(2)), 2.56 (dd, $J$ = 15.3, 2.2, 1 H, HC(8)), 2.34 (dd, $J$ = 15.3, 3.4, 1 H, HC(8)), 2.01-1.88 (m, 2 H, HC(6)), 1.18 (s, 3 H, HC(4)), 1.18 (s, 3 H, HC(4)), 0.95 (d, $J$ = 6.8, 3 H, HC(7)), 0.94 (d, $J$ = 7.3, 3 H, HC(7)), 0.923 (d, $J$ = 6.8, 3 H, HC(7)), 0.917 (d, $J$ = 6.8, 3 H, HC(7))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

202.2 (C(9)), 171.0 (C(1)), 137.2 (C(11)), 128.3 (C(12) or C(13)), 128.0 (C(12) or C(13)), 127.9 (C(14)), 85.3 (C(2)), 84.5 (C(5)), 72.5 (C(10)), 52.8 (C(8)), 37.7 (C(3)), 29.5 (2 × (C(6)), 25.4 (C(4)), 24.0 (C(4)), 19.9 (C(7)), 19.8 (C(7)), 17.8 (C(7)), 17.6 (C(7))

IR: (neat)

3031 (w), 2967 (s), 2935 (s), 2876 (s), 2733 (w), 1735 (s), 1719 (s), 1498 (m), 1470 (m), 1455 (m), 1389 (m), 1370 (m), 1340 (m), 1258 (m), 1197 (s), 1115 (s), 1056 (m), 1029 (m), 999 (m), 970 (m), 936 (m), 806 (m), 739 (m), 698 (m)

MS: (ESI)

91.1 (19), 115.1 (6), 143.1 (63), 233.1 (18), 241.2 (78), 249.1 (38), 267.1 (11), 347.2 (8), 355.2 (47), 371.2 ($M^+$+Na, 100), 387.2 (23)

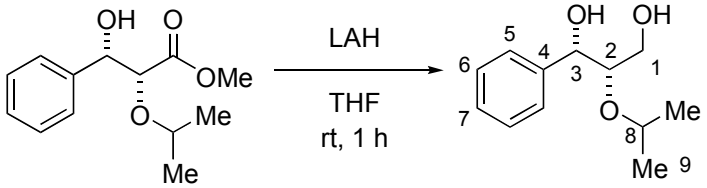
HRMS: calcd for $C_{21}H_{32}O_4Na$: 371.2198, found 371.2216

TLC: $R_f$ 0.54 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

**Determination of Relative Configurations**

**General Procedure 10. Lithium Aluminum Hydride Reduction of Aldol Products to Diols.**

**Preparation of (1*S*, 2*S*)-2-(1-Methylethoxy)-1-phenyl-1,3-propanediol ((1*S*,2*S*)-7ba)**




**6ba** (1*S*,2*S*)-**7ba**

To a flame-dried, 10-mL, 2-necked round-bottomed flask fitted with a magnetic stir bar, a gas inlet tube, and a septum were added **6ba** (119.1 mg, 0.5 mmol) and THF (1.5 mL). The solution was cooled to 0 °C in an ice-water bath and then a solution of LAH (254 μL, 1.97 M in THF, 0.5 mmol, 1.0 equiv) was added dropwise via syringe over 5 min. The ice-water bath was removed and the reaction mixture was stirred at rt for 1 h. The reaction mixture was diluted with $Et_2O$ (2 mL) prior to sequencial addition of $H_2O$ (20 μL), 3*N* aq. NaOH solution (20 μL), and $H_2O$ (60 μL). After 15 min, $MgSO_4$ (1 g) was added to the heterogeneous mixture prior to filtration through a glass frit. The white solid on the filter was rinsed with EtOAc (4 × 2 mL). The combined filtrates were concentrated *in vacuo* (23 °C, 30 mmHg). The residue was purified by column chromatography (18 mm diam., hexane/EtOAc, 1/1) on silica gel (10 g) to give **7ba** (95 mg, 90%) as a colorless oil.

Data for (1S,2S)-**7ba**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.42-7.38 (m, 2 H, HC(5)), 7.37-7.34 (m, 2 H, HC(6)), 7.31-7.27 (m, 1 H, HC(7)), 4.74 (d, *J* = 6.8, 1 H, HC(3)), 3.68 (qq, *J* = 6.1, 6.1, 1 H, HC(8)), 3.66 (dd, *J* = 11.7, 3.9, 1 H, HC(1)), 3.50 (ddd, *J* = 6.8, 3.9, 3.9, 1 H, HC(2)), 3.43 (dd, *J* = 11.7, 3.9, 1 H, HC(1)), 2.32 (bs, 2 H, OH), 1.20 (d, *J* = 6.1, 3 H, HC(9)),

1.161(d, $J$ = 6.1, 3 H, HC(9))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

140.7 (C(4)), 128.2 (C(6)), 127.7 (C(7)), 126.6 (C(5)), 81.5 (C(2)), 73.3 (C(3)), 72.0 (C(8)), 61.4 (C(1)), 22.9 (C(9)), 22.2 (C(9))

IR: (neat)

3402 (br), 3064 (m), 3032 (m), 2973 (s), 2931 (m), 2889 (m), 1646 (br), 1604 (w), 1495 (m), 1454 (m), 1383 (m), 1331 (m), 1198 (m), 1176 (m), 1143 (s), 1122 (s), 1079 (s), 1049 (s), 975 (m), 914 (m), 877 (m), 795 (m), 763 (m), 702 (s), 626 (m)

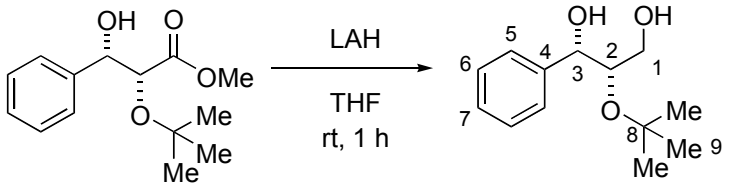
MS: (ESI)

133.1 (6), 151.1 (21), 193.1 (100), 194.1 (4), 228.2 (4), 230.1 (8), 233.1 ($M^+$+Na, 18), 335.2 (13), 344.2 (4)

HRMS: calcd for $C_{12}H_{18}O_3Na$: 233.1154, found 233.1159

TLC: $R_f$ 0.32 (hexane/EtOAc, 1/1) [UV(254)/$KMnO_4$]

**Preparation of (1*S*, 2*S*)-2-(1,1-Dimethylethoxy)-1-phenyl-1,3-propanediol ((1*S*,2*S*)-7ca)**




**6ca** (1*S*,2*S*)-**7ca**

Following General Procedure 10, **6ca** (126.2 mg, 0.5 mmol) was combined with a solution of LAH (254 μL, 1.97 M in THF, 0.5 mmol, 1.0 equiv) to yield, after column chromatography (18 mm diam., hexane/EtOAc, 1/1) on silica gel (10 g), **7ca** (99 mg, 88%) as a colorless oil.

Data for (1*S*,2*S*)-**7ca**:

$^{1}$H NMR: (500 MHz, $CDCl_3$)

7.42-7.40 (m, 2 H, HC(5)), 7.37-7.34 (m, 2 H, HC(6)), 7.30-7.27 (m, 1 H, HC(7)), 4.79-4.75 (m, 1 H, HC(3)), 3.66-3.62 (m, 2 H, HC(1), HC(2)), 3.46-3.42 (m, 1 H, HC(1)), 2.49 (bs, 2 H, OH), 1.21 (s, 9 H, HC(9))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

141.1 (C(4)), 128.1 (C(6)), 127.6 (C(7)), 126.9 (C(5)), 75.6 (C(2)), 75.1 (C(8)), 73.2 (C(3)), 62.5 (C(1)), 28.5 (C(9))

IR: (neat)

3401 (br), 3063 (m), 3032 (m), 2975 (s), 2936 (m), 1645 (br), 1605 (w), 1495 (m), 1453 (m), 1392 (s), 1367 (s), 1256 (m), 1236 (m), 1190 (s), 1080 (s), 1047 (s), 1024 (s), 981 (m), 900 (m), 873 (m), 811 (m), 747 (s), 701 (s), 667 (m),

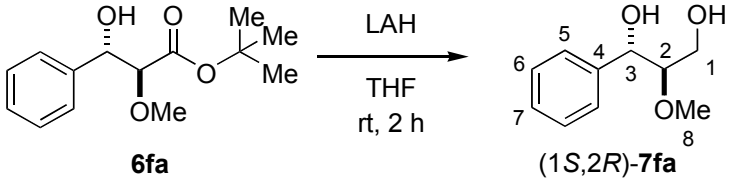
MS: (ESI)

133.1 (14), 151.1 (74), 225.2 (10), 231.2 (13), 242.2 (14), 244.1 (21), 247.1 ($M^+$+Na, 100), 248.1 (6), 282.3 (8), 338.3 (28), 356.2 (16), 356.7 (7)

HRMS: calcd for $C_{13}H_{20}O_3Na$: 247.1310, found 247.1319

TLC: $R_f$ 0.41 (hexane/EtOAc, 1/1) [UV(254)/$KMnO_4$]

**Preparation of (1*S*, 2*R*)-2-Methoxy-1-phenyl-1,3-propanediol[22] ((1*S*,2*R*)-7fa)**




**6fa** (1*S*,2*R*)-**7fa**

Following General Procedure 10, **6fa** (126 mg, 0.5 mmol) was combined with a solution of LAH (254 μL, 1.97 M in THF, 0.5 mmol, 1.0 equiv) to yield, after column chromatography

(18 mm diam., EtOAc/hexane, 1/1 to 2/1) on silica gel (10 g), **7fa** (80 mg, 88%) as a white crystal.

Data for (1S,2R)-**7fa**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.40-7.35 (m, 4 H, HC(5), HC(6)), 7.31-7.28 (m, 1 H, HC(7)), 4.98 (d, $J$ = 4.9, 1 H, HC(3)), 3.72 (dd, $J$ = 12.0, 5.1, 1 H, HC(1)), 3.66 (dd, $J$ = 12.0, 3.9, 1 H, HC(1)), 3.41 (s, 3 H, HC(8)), 3.39 (ddd, $J$ = 5.0, 5.0, 3.9, 1 H, HC(2)), 2.38 (bs, 2 H, OH)

$^{13}$C NMR: (126 MHz, $CDCl_3$)

140.8 (C(4)), 128.3 (C(6)), 127.5 (C(7)), 126.1 (C(5)), 84.5 (C(2)), 73.4 (C(3)), 60.7 (C(1)), 57.8 (C(8))

IR: (neat)

3392 (br), 3063 (m), 3032 (m), 2934 (m), 2889 (m), 2832 (m), 1638 (br), 1604 (w), 1495 (m), 1454 (m), 1408 (m), 1335 (m), 1191 (m), 1156 (m), 1114 (s), 1062 (s), 923 (m), 873 (m), 831 (m), 763 (m), 702 (s), 633 (m)

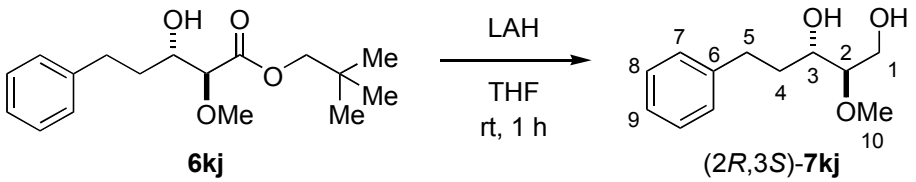
MS: (ESI)

205.1 ($M^+$+Na, 100), 206.1 (16), 263.0 (22), 265.0 (12)

HRMS: calcd for $C_{10}H_{14}O_3Na$: 205.0841, found: 205.0835

TLC: $R_f$ 0.27 (EtOAc/hexane, 2/1) [UV(254)/$KMnO_4$]

**Preparation of (2*R*,3*S*)-2-Methoxy-5-phenyl-1,3-propanediol[23] ((2*R*,3*S*)-7kj)**




**6kj** (2*R*,3*S*)-**7kj**

Following General Procedure 10, **6kj** (147.2 mg, 0.5 mmol) was combined with a solution of LAH (254 μL, 1.97 M in THF, 0.5 mmol, 1.0 equiv) to yield, after column chromatography (18 mm diam., loaded with $CH_2Cl_2$, packed and eluted with EtOAc/hexane, 2/1) on silica gel (8 g), **7kj** (87 mg, 83%) as white crystals.

Data for (2*R*,3*S*)-**7kj**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.31-7.27 (m, 2 H, HC(8)), 7.23-7.22 (m, 2 H, HC(7)), 7.21-7.18 (m, 1 H, HC(9)), 3.85 (ddd, *J* = 9.5, 5.1, 3.3, 1 H, HC(3)), 3.82-3.81 (m, 2 H, HC(1)), 3.43 (s, 3 H, HC(10)), 3.15-3.09 (m, 3 H, HC(2), OH), 2.91 (ddd, *J* = 13.7, 10.1, 5.3, 1 H, HC(5)), 2.68 (ddd, *J* = 13.7, 9.7, 6.8, 1 H, HC(5)), 1.86 (dddd, *J* = 13.6, 10.1, 6.8, 3.3, 1 H, HC(4)), 1.80 (dddd, *J* = 13.6, 9.6, 9.6, 5.3, 1 H, HC(4))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

141.8 (C(6)), 128.34 (C(7) or C(8)), 128.31 (C(7) or C(8)), 125.8 (C(9)), 83.6 (C(2)), 70.5 (C(1)), 60.4 (C(3)), 57.5 (C(10)), 34.8 (C(4)), 32.1 (C(5))

IR: (neat)

3399 (br), 3085 (m), 3061 (m), 3026 (m), 2933 (s), 2830 (m), 2245 (w), 1948 (w), 1868 (w), 1810 (w), 1653 (br), 1603 (m), 1496 (s), 1454 (s), 1332 (m), 1192 (m), 1154 (m), 1092 (s), 1057 (s), 1030 (s), 973 (m), 910 (m), 843 (m), 733 (s), 701 (s)

MS: (ESI)

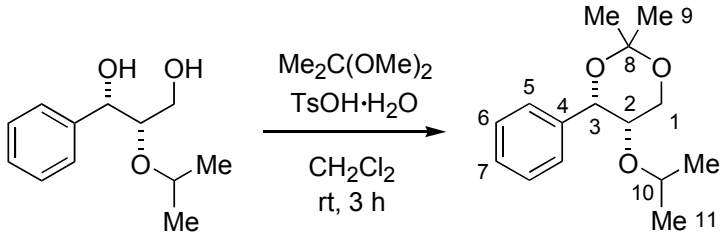
230.1 (15), 233.1 ($M^+$+Na, 100), 234.1 (20), 291.1 (26)

HRMS: calcd for $C_{12}H_{18}O_3Na$: 233.1154, found 233.1152

TLC: $R_f$ 0.24 (EtOAc/hexane, 2/1) [UV(254)/$KMnO_4$]

**General Procedure 11. Acetalization of Diols to Acetonides**

**Preparation of (4*S*,5*S*)-2,2-Dimethyl-5-(1-methylethoxy)-4-phenyl-1,3-dioxane ((4*S*,5*S*)-8ba)**




**7ba** (4*S*,5*S*)-**8ba**

To a flame-dried, 5-mL, 2-necked round-bottomed flask fitted with a magnetic stir bar, a gas inlet tube, and a septum were added **7ba** (88 mg, 0.419 mmol) and $CH_2Cl_2$ (1 mL). To the stirred solution were added 2,2-dimethoxypropane (515 μL, 4.19 mmol, 10.0 equiv) and a solution of TsOH monohydrate (8 mg, 0.042 mmol, 0.1 equiv) in $CH_2Cl_2$ (0.5 mL) via syringe at rt. After 3 h, the reaction mixture was transferred to a 50-mL separatory funnel and was diluted with $CH_2Cl_2$ (10 mL) and sat. aq. $NaHCO_3$ solution (10 mL). The organic layer was separated. The aqueous layer was extracted with $CH_2Cl_2$ (10 mL). The combined organic layers were dried over $K_2CO_3$ (1 g), filtered, and concentrated *in vacuo* (23 °C, 30 mmHg). The residue was purified by column chromatography (18 mm diam., hexane/EtOAc, 10/1) on silica gel (10 g) to give **8ba** (83 mg, 79%) as a colorless oil.

Data for (4*S*,5*S*)-**8ba**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.44-7.41 (m, 2 H, HC(5)), 7.35-7.31 (m, 2 H, HC(6)), 7.27-7.24 (m, 1 H,

HC(7)), 5.00 (d, *J* = 2.2, 1 H, HC(3)), 4.18 (dd, *J* = 12.2, 2.5, 1 H, HC(1)), 3.89 (dd, *J* = 12.2, 2.5, 1 H, HC(1)), 3.31-3.30 (m, 1 H, HC(2)), 2.91 (qq, *J* = 6.1, 6.1, 1 H, HC(10)), 1.56 (s, 3 H, HC(9)), 1.53 (s, 3 H, HC(9)), 0.97 (d, *J* = 6.1, 3 H, HC(11)), 0.70 (d, *J* = 6.1, 3 H, HC(11))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

138.9 (C(4)), 127.7 (C(6)), 127.3 (C(7)), 126.9 (C(5)), 98.9 (C(8)), 73.7 (C(3)), 72.24 (C(2) or C(10)), 72.22 (C(2) or C(10)), 64.7 (C(1)), 28.9 (C(9)), 22.0 (C(11)), 21.8 (C(11)), 19.2 (C(9))

IR: (neat)

3445 (br), 3028 (w), 2971 (s), 2937 (m), 2876 (m), 1664 (br), 1451 (s), 1379 (s), 1330 (m), 1271 (m), 1238 (s), 1198 (s), 1170 (s), 1134 (s), 1084 (s), 1060 (s), 1030 (m), 1006 (m), 964 (m), 939 (m), 854 (m), 769 (m), 738 (m), 700 (s)

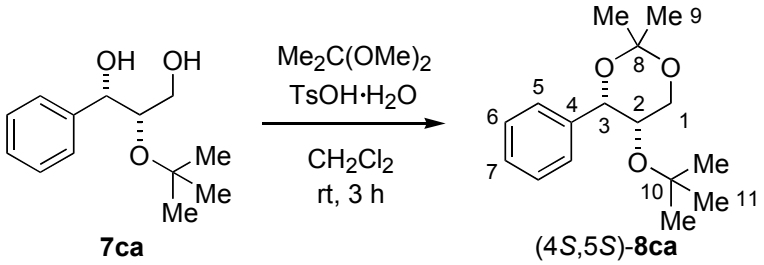
MS: (ESI)

233.1 (9), 273.1 ($M^+$+Na, 100), 274.1 (21), 331.1 (5)

HRMS: calcd for $C_{15}H_{22}O_3Na$: 273.1467, found 273.1478

TLC: $R_f$ 0.25 (hexane/EtOAc, 10/1) [UV(254)/$KMnO_4$]

**Preparation of (4*S*,5*S*)-2,2-Dimethyl-5-(1,1-dimethylethoxy)-4-phenyl-1,3-dioxane ((4*S*,5*S*)-8ca)**




**7ca** (4*S*,5*S*)-**8ca**

Following General Procedure 11, **7ca** (94 mg, 0.419 mmol) was combined with 2,2-

dimethoxypropane (515 μL, 4.19 mmol, 10.0 equiv) and TsOH monohydrate (8 mg, 0.042 mmol, 0.1 equiv) to yield, after column chromatography (18 mm diam., hexane/EtOAc, 10/1) on silica gel (10 g), **8ca** (81 mg, 90%) as a colorless oil.

Data for (4*S*,5*S*)-**8ca**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.42-7.35 (m, 2 H, HC(5)), 7.34-7.28 (m, 2 H, HC(6)), 7.26-7.22 (m, 1 H, HC(7)), 4.97 (d, *J* = 2.4, 1 H, HC(3)), 4.16 (dd, *J* = 12.1, 2.9, 1 H, HC(1)), 3.83 (dd, *J* = 12.1, 2.7, 1 H, HC(1)), 3.41-3.39 (m, 1 H, HC(2)), 1.54 (s, 3 H, HC(9)), 1.52 (s, 3 H, HC(9)), 0.74 (s, 9 H, HC(11))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

139.3 (C(4)), 127.9 (C(5)), 127.5 (C(6)), 127.4 (C(7)), 99.0 (C(8)), 74.6 (C(3)), 73.4 (C(10)), 66.3 (C(2)), 66.0 (C(1)), 28.5 (C(9)), 27.5 (C(11)), 19.6 (C(9))

IR: (neat)

3521 (br), 3028 (m), 2978 (s), 2938 (s), 2869 (m), 1495 (m), 1471 (m), 1451 (m), 1390 (s), 1379 (s), 1364 (s), 1269 (s), 1238 (s), 1193 (s), 1168 (s), 1129 (s), 1086 (s), 1061 (s), 1026 (m), 1003 (s), 962 (s), 935 (m), 911 (m), 882 (m), 858 (s), 845 (m), 764 (m), 754 (s), 700 (s), 656 (m)

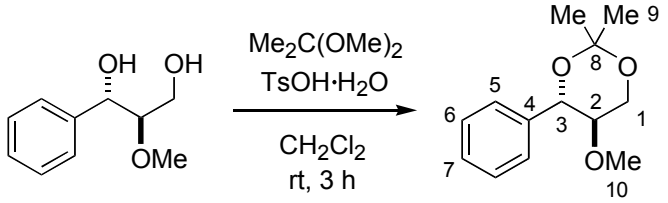
MS: (ESI)

247.1 (8), 271.2 (7), 287.2 ($M^+$+Na, 100), 288.2 (14)

HRMS: calcd for $C_{16}H_{24}O_3Na$: 287.1623, found 287.1620

TLC: $R_f$ 0.29 (hexane/EtOAc, 10/1) [UV(254)/$KMnO_4$]

**Preparation of (4*S*,5*R*)-2,2-Dimethyl-5-methoxy-4-phenyl-1,3-dioxane[24] ((4*S*,5*R*)-8fa)**




**7fa** (4*S*,5*R*)-**8fa**

Following General Procedure 11, **7fa** (69 mg, 0.379 mmol) was combined with 2,2-dimethoxypropane (466 μL, 3.79 mmol, 10.0 equiv) and TsOH monohydrate (7.2 mg, 0.038 mmol, 0.1 equiv) to yield, after column chromatography (18 mm diam., hexane/EtOAc, 5/1) on silica gel (10 g), **8fa** (70 mg, 83%) as a colorless oil.

Data for (4*S*,5*R*)-**8fa**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.47-7.45 (m, 2 H, HC(5)), 7.39-7.35 (m, 2 H, HC(6)), 7.33-7.29 (m, 1 H, HC(7)), 4.60 (d, *J* = 9.3, 1 H, HC(3)), 4.06 (dd, *J* = 11.5, 5.2, 1 H, HC(1)), 3.77 (dd, *J* = 11.5, 9.0, 1 H, HC(1)), 3.31 (ddd, *J* = 9.2, 9.2, 5.2, 1 H, HC(2)), 3.08 (s, 3 H, HC(10)), 1.57 (s, 3 H, HC(9)), 1.49 (s, 3 H, HC(9))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

139.8 (C(4)), 128.3 (C(6)), 128.0 (C(7)), 127.3 (C(5)), 99.1 (C(8)), 77.9 (C(2)), 75.4 (C(3)), 62.7 (C(1)), 58.1 (C(10)), 28.5 (C(9)) 19.4 (C(9))

IR: (neat)

3472 (br), 3033 (m), 2992 (s), 2938 (m), 2888 (m), 2829 (m), 1495 (m), 1454 (m), 1380 (s), 1308 (m), 1264 (s), 1224 (s), 1197 (s), 1164 (s), 1100 (s), 1061 (m), 1027 (s), 987 (m), 936 (m), 879 (s), 829 (m), 767 (s), 752 (s), 699 (s)

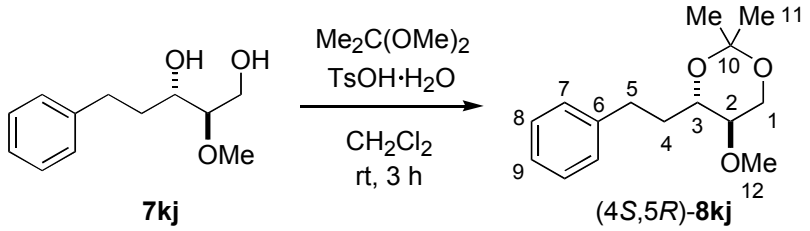
MS: (ESI)

102.1 (18), 205.1 (100), 206.1 (15), 245.1 ($M^+$+Na, 19), 301.1 (12)

HRMS: calcd for $C_{13}H_{18}O_3Na$: 245.1154, found 245.1160

TLC: $R_f$ 0.47 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

**Preparation of (4*S*,5*R*)-2,2-Dimethyl-5-methoxy-4-(2-phenylethyl)-1,3-dioxane ((4*S*,5*R*)-8kj)**




**7kj** (4*S*,5*R*)-**8kj**

Following General Procedure 11, **7kj** (85 mg, 0.404 mmol) was combined with 2,2-dimethoxypropane (497 μL, 4.042 mmol, 10.0 equiv) and TsOH monohydrate (7.6 mg, 0.04 mmol, 0.1 equiv) to yield, after column chromatography (18 mm diam., hexane/EtOAc, 5/1) on silica gel (10 g), **8kj** (91 mg, 90%) as a colorless oil.

Data for (4*S*,5*R*)-**8kj**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.32-7.27 (m, 2 H, HC(8)), 7.24-7.21 (m, 2 H, HC(7)), 7.20-7.17 (m, 1 H, HC(9)), 3.98 (dd, *J* = 11.5, 5.1, 1 H, HC(1)), 3.60 (dd, *J* = 11.5, 8.3, 1 H, HC(1)), 3.60-3.55 (m, 1 H, HC(3)), 3.35 (s, 3 H, HC(12)), 3.08 (ddd, *J* = 8.5, 8.5, 5.1, 1 H, HC(2)), 2.84 (ddd, *J* = 13.7, 10.0, 4.9, 1 H, HC(5)), 2.63 (ddd, *J* = 13.7, 9.6, 7.0, 1 H, HC(5)), 2.11 (dddd, *J* = 13.9, 10.0, 7.0, 3.0, 1 H, HC(4)), 1.77 (dddd, *J* = 13.9, 9.3, 9.3, 4.9, 1 H, HC(4)), 1.44 (s, 3 H, HC(11)), 1.42 (s, 3 H, HC(11))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

142.1 (C(6)), 128.5 (C(7)), 128.3 (C(8)), 125.7 (C(9)), 98.8 (C(10)), 77.2 (C(2)), 71.5 (C(3)), 62.1 (C(1)), 57.6 (C(12)), 34.3 (C(4)), 31.2 (C(5)), 28.2 (C(11)),

19.8 (C(11))

IR: (neat)

3450 (br), 3026 (w), 2992 (m), 2937 (m), 2879 (m), 2825 (w), 1652 (br), 1604 (w), 1496 (m), 1455 (m), 1379 (m), 1369 (m), 1267 (m), 1227 (m), 1202 (s), 1163 (m), 1142 (m), 1125 (s), 1105 (s), 1067 (m), 1040 (m), 988 (m), 859 (m), 750 (m), 700 (s)

MS: (ESI)

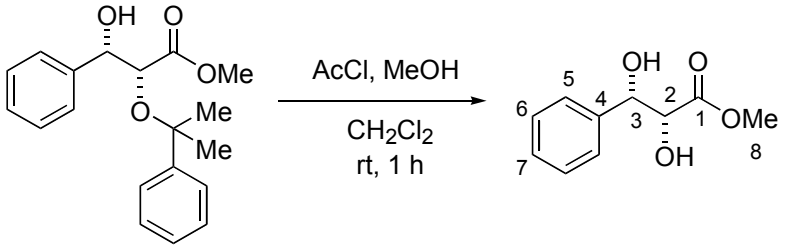
143.1 (12), 233.1 (52), 234.1 (8), 273.1 ($M^+$+Na, 100), 274.1 (13), 301.1 (10), 385.2 (13)

HRMS: calcd for $C_{15}H_{22}O_3Na$: 273.1467, found 273.1469

TLC: $R_f$ 0.48 (hexane/EtOAc, 5/1) [UV(254)/$KMnO_4$]

**Determination of Absolute Configurations**

**Preparation of (2*R*,3*S*)-1,2-Dihydroxy-3-phenylpropanoic Acid Methyl Ester[10] ((2*R*,3*S*)-9)**




**6da** (2*R*,3*S*)-**9**

To a flame-dried, 5-mL, 2-necked round-bottomed flask fitted with a magnetic stir bar, a gas inlet tube, and a septum were added $CH_2Cl_2$ (1 mL), acetyl chloride (69 μL, 0.966 mmol, 1.1 equiv), and MeOH (39 mL, 0.966 mmol 1.1 equiv). The resulting solution was transferred via cannula to a stirred solution of **6da** (276 mg, 0.878 mmol) in $CH_2Cl_2$ (3 mL) in 10-mL 2-necked round-bottomed flask fitted with a magnetic stir bar, a gas inlet tube, and a septum at rt. After 1h,

the reaction mixture was transferred to a 125-mL separatory funnel where the reaction was quenched with sat. aq. $NaHCO_3$ solution (20 mL). The organic layer was separaed. The aqueous layer was extracted with EtOAc (2 × 20 mL). The combined organic extracts were dried over $Na_2SO_4$ (5 g), filtered, and concentrated *in vacuo* (23 °C, 30 mmHg). The residue was purified by column chromatography (22 mm diam., hexane/EtOAc, 1/1) on silica gel (15 g) to give **9** (161 mg, 93%) as white crystals.

Data for (2*R*,3*S*)-**9**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.42-7.36 (m, 4 H, HC(5), HC(6)), 7.34-7.30 (m, 1 H, HC(7)), 5.03 (d, $J$ = 2.9, 1 H, HC(3)), 4.38 (d, $J$ = 2.9, 1 H, HC(2)), 3.82 (s, 3 H, HC(8)), 3.08 (bs, 1 H, OH), 2.70 (bs, 1 H, OH)

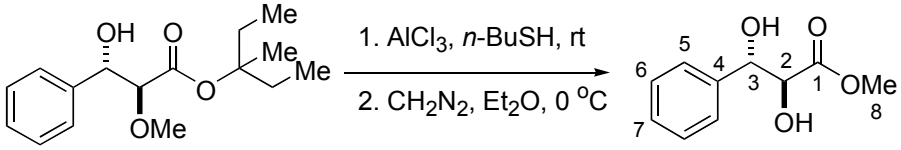
TLC: $R_f$ 0.31 (hexane/EtOAc,1/1) [UV(254)/$KMnO_4$]

Opt. Rot.: $[\alpha]_D^{24}$ 7.9 (c = 1.0, EtOH)

lit. $[\alpha]_D^{24}$ 8.2 (c = 1.1, EtOH) for (2*R*,3*S*)-**9**[20]

lit. $[\alpha]_D^{24}$ –10.3 (c = 1.0, EtOH) for (2*S*,3*R*)-**9**[25]

**Preparation of (2*S*,3*S*)-1,2-Dihydroxy-3-phenylpropanoic Acid Methyl Ester[26] ((2*S*,3*S*)-10)**




**6ha** (2*S*,3*S*)-**10**

To a flame-dried, 5-mL, 2-necked round-bottomed flask fitted with a magnetic stir bar, a gas inlet tube, and a septum were added $AlCl_3$ (309 mg, 2.318 mmol, 5.0 equiv) and *n*-BuSH (1 mL, 9.336 mmol, 20 equiv). To the solution was added a solution of **6ha** (130 mg, 0.464 mmol) in *n*-BuSH (1 mL, 9.336 mmol, 20 equiv) via syringe at rt. After 24 h, the reaction mixture was

transferred to a 50-mL separatory funnel and was diluted with $CH_2Cl_2$ (10 mL) and 1*N* aq. HCl solution (10 mL). The organic layer was separaed. The aqueous layer was extracted with EtOAc (8 × 10 mL). The combined organic extracts were dried over $Na_2SO_4$ (3 g), filtered, and concentrated *in vacuo* (23 °C, 30 mmHg). The residue dissolved in $Et_2O$ (1 mL) and placed in the receiving flask of Aldrich mini diazald® apparatus. The solution was cooled to 0 °C in an ice-water bath. $CH_2N_2$ (ca. 23.4 mg, 0.557 mmol, 1.2 equiv), which was generated according to Aldrich tech note AL-180 (70% yield was assumed), was distilled to the receiving flask over 10 min. The excess $CH_2N_2$ was quenched by acetic acid (1*N* in $Et_2O$, 0.5 mL). The resulting solution was transferred to a 50-mL separatory funnel and was washed with sat. aq. $NaHCO_3$ solution (3 × 5 mL), dried over $Na_2SO_4$ (2 g), filtered, and concentrated *in vacuo* (23 °C, 30 mmHg). The residue was purified by column chromatography (18 mm diam., hexane/EtOAc, 2/1 to 1/1) on silica gel (10 g) to give **10** (48 mg, 53% for two steps) as white crystals.

Data for (2*S*,3*S*)-**10**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.37-7.34 (m, 2 H, HC(5)), 7.32-7.29 (m, 3 H, HC(6), HC(7)), 5.01 (d, *J* = 4.3, 1 H, HC(3)), 4.50 (d, *J* = 4.3, 1 H, HC(2)), 3.69 (s, 3 H, HC(8)), 2.94 (bs, 2 H, OH)

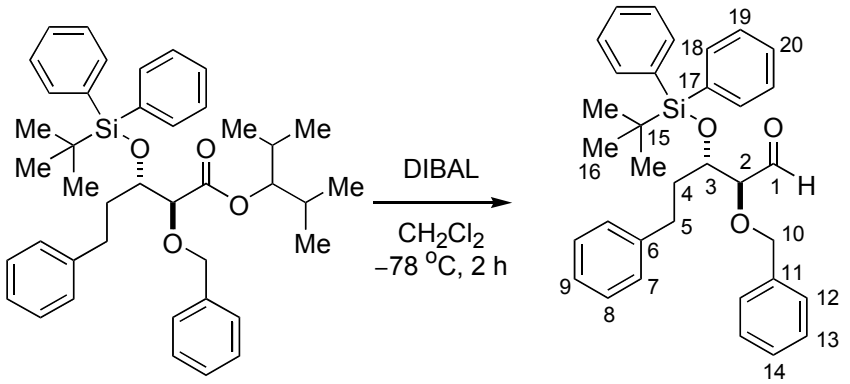
TLC: $R_f$ 0.27 (hexane/EtOAc, 1/1) [UV(254)/$KMnO_4$]

Opt. Rot.: $[\alpha]_D^{24}$ 51.0 (c = 1.0, $CHCl_3$)

lit. $[\alpha]_D^{24}$ 36.1 (c = 0.72, $CHCl_3$) for (2*S*,3*S*)-**10**[26]

lit. $[\alpha]_D^{24}$ –41.3 (c = 0.48, $CHCl_3$) for (2*R*,3*R*)-**10**[27]

**Preparation of (2*S*,3*S*)-3-[[(1,1-Dimethylethyl)diphenylsilyl]oxy]-5-phenyl-2-(phenyl methoxy)pentanoic Acid 2-Methyl-1-(1-methylethyl)propyl Ester ((2*S*,3*S*)-12)**




**12** (2*S*,3*S*)-**13**

To a flame-dried, 5-mL, 2-necked round-bottomed flask fitted with a magnetic stir bar, a gas inlet tube, and a septum were added **6mj** (136 mg, 0.341 mmol) and DMF (1 mL). To the solution were added TBDPSCl (266 μL, 1.023 mmol, 3.0 equiv) and a solution of imidazole (70 mg, 1.023 mmol, 3.0 equiv) in DMF (0.5 mL) via syringe at rt. After 24 h, the reaction mixture was transferred to a 125-mL separatory funnel and was diluted with $Et_2O$ (20 mL). The resulting solution was washed with sat. aq. $NaHCO_3$ solution (20 mL) and sat. aq. $NH_4Cl$ solution (2 × 20 mL). The resulting organic layer was dried over $Na_2SO_4$ (1 g), filtered, and concentrated *in vacuo* (23 °C, 30 mmHg). The residue was purified by column chromatography (22 mm diam., hexane/EtOAc, 20/1) on silica gel (20 g) to give **12** (206 mg, 95%) as a colorless oil.

Data for (2*S*,3*S*)-**12**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.73-7.71 (m, 4 H, HC(21)), 7.45-7.40 (m, 2 H, HC(23)), 7.39-7.34 (m, 4 H, HC(15), HC(22)), 7.33-7.27 (m, 5 H, HC(16), HC(17), HC(22)), 7.20-7.17 (m, 2 H, HC(8)), 7.13-7.10 (m, 1 H, HC(9)), 6.99-6.97 (m, 2 H, HC(7)), 4.67 (d, *J* = 11.5, 1 H, HC(13)), 4.60 (dd, *J* = 6.1, 6.1, 1 H, HC(10)), 4.25 (d, *J* = 11.5, 1 H,

HC(13)), 4.18 (ddd, *J* = 8.3, 3.9, 2.0, 1 H, HC(3)), 4.00 (d, *J* = 2.0, 1 H, HC(2)), 2.74 (ddd, *J* = 13.4, 11.5, 5.1, 1 H, HC(5)), 2.38 (ddd, *J* = 13.4, 11.4, 5.5, 1 H, HC(5)), 2.10 (dddd, *J* = 14.0, 11.4, 8.3, 5.1, 1 H, HC(4)), 1.85-1.76 (m, 3 H, HC(4), HC(11)), 1.08 (s, 9 H, HC(19)), 0.82 (d, *J* = 6.8, 3 H, HC(12)), 0.80 (d, *J* = 7.1, 3 H, HC(12)), 0.74 (d, *J* = 6.5, 3 H, HC(12)), 0.70 (d, *J* = 6.9, 3 H, HC (12))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

170.1 (C(1)), 142.1 (C(6)), 138.0 (C(14)), 136.13 (C(21)), 136.05 (C(21)), 133.7 (C(20)), 133.6 (C(20)), 129.7 (C(23)), 129.6 (C(23)), 128.3 (Aryl), 128.2 (2 × Aryl), 127.64 (C(15) or C(22)), 127.60 (C(15) or C(22)), 127.45 (C(15) or C(22)), 127.42 (C(17)), 125.6 (C(9)), 83.5 (C(10)), 82.2 (C(2)), 74.8 (C(3)), 72.1 (C(13)), 34.5 (C(4)), 32.6 (C(5)), 29.3 (C(11)), 29.1 (C(11)), 27.0 (C(19)), 19.7 (C(12)), 19.54 (C(18)), 19.45 (C(12)), 17.6 (C(12)), 17.1 (C(12))

IR: (neat)

3069 (w), 3028 (w), 2964 (m), 2933 (m), 2858 (m), 1745 (s), 1604 (w), 1590 (w), 1497 (m), 1472 (m), 1455 (m), 1428 (m), 1389 (m), 1371 (m), 1274 (m), 1199 (s), 1112 (s), 969 (m), 969 (m), 936 (m), 908 (m), 822 (m), 737 (s), 700 (s), 611 (s)

MS: (ESI)

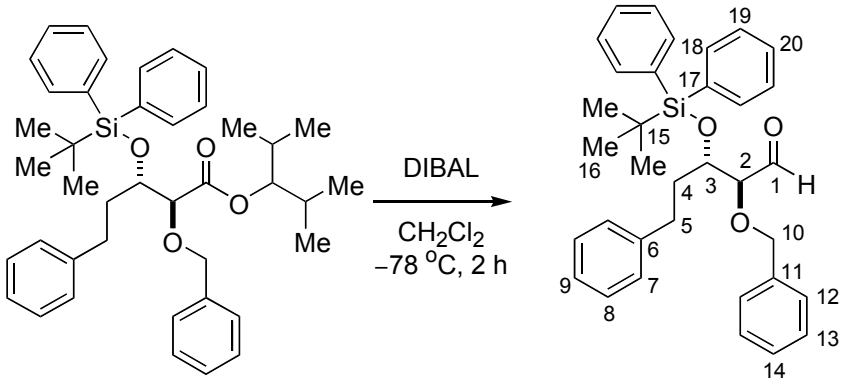
91.1 (24), 102.1 (34), 119.0 (14), 181.1 (13), 219.1 (34), 293.1 (25), 305.1 (14), 355.1 (35), 383.1 (30), 415.2 (12), 461.2 (100), 462.2 (54), 463.2 (15), 559.3 (12), 659.3 ($M^+$+Na, 57), 660.3 (29)

HRMS: calcd for $C_{41}H_{52}O_4SiNa$: 659.3533, found 659.3564

TLC: $R_f$ 0.34 (hexane/EtOAc, 20/1) [UV(254)/$KMnO_4$]

Opt. Rot.: $[\alpha]_D^{24}$ –4.3 (c = 2.0, EtOH)

**Preparation of (2*S*,3*S*)-3-[[(1,1-Dimethylethyl)diphenylsilyl]oxy]-5-phenyl-2-(phenyl methoxy)pentanal[28] ((2*S*,3*S*)-13)**




**12** (2*S*,3*S*)-**13**

To a flame-dried, 10-mL, 2-necked round-bottomed flask fitted with a magnetic stir bar, a gas inlet tube, and a septum were added **12** (206 mg, 0.323 mmol) and $CH_2Cl_2$ (2 mL). The solution was cooled to –78 °C in a dry ice-acetone bath. To the solution was added DIBAL (766 μL, 0.464 M in hexane, 0.355 mmol, 1.1 equiv) dropwise via syringe over 10 min. The reaction mixture was stirred at –78 °C for 2 h prior to dropwise addition of MeOH (0.5 mL) and sat. Rochelle's salt (*aq*, 4 mL). The resulting heterogeneous mixure was stirred vigorously for 1 h at rt and then transferred to a 50-mL separatory funnel prior to dilution with $H_2O$ (10 mL) and $CH_2Cl_2$ (10 mL). The organic layer was separated. The aqueous layer was extracted with $CH_2Cl_2$ (2 × 10 mL). The combined organic layers were dried over $Na_2SO_4$ (5 g), filtered, and concentrated *in vacuo* (23 °C, 30 mmHg). The residue was purified by column chromatography (22 mm diam., hexane/EtOAc, 40/1 to 20/1) on silica gel (20 g) to give **13** (153 mg, 91%) as a colorless oil.

Data for (2*S*,3*S*)-**13**:

$^1$H NMR: (500 MHz, $CDCl_3$)

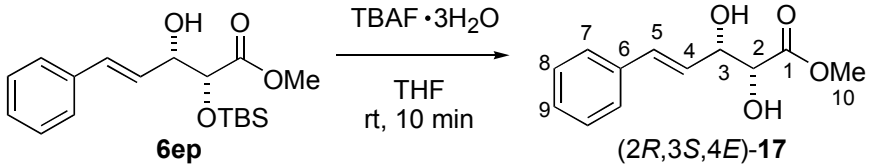
9.70 (d, *J* = 2.2, 1 H, HC(1)), 7.71-7.67 (m, 4 H, HC(18)), 7.47-7.42 (m, 2 H, HC(20)), 7.39-7.36 (m, 2 H, Aryl), 7.36-7.32 (m, 5 H, Aryl), 7.31-7.28 (m, 2 H, Aryl), 7.19-7.16 (m, 2 H, HC(8)), 7.14-7.11 (m, 1 H, HC(9)), 6.88-6.86 (m, 2 H, HC(7)), 4.57 (d, *J* = 11.7, 1 H, HC(10)), 4.50 (d, *J* = 11.7, 1 H, HC(10)), 4.17 (ddd, *J* = 6.0, 6.0, 3.3, 1 H, HC(3)), 3.76 (dd, *J* = 3.3, 2.3, 1 H, HC(2)), 2.45-2.36 (m, 2 H, HC(5)), 1.91-1.79 (m, 2 H, HC(4)), 1.07 (s, 9 H, HC(16))

TLC: $R_f$ 0.25 (hexane/EtOAc, 20/1) [UV(254)/$KMnO_4$]

Opt. Rot.: $[\alpha]_D^{24}$ 21.7 (c = 1.0, $CHCl_3$)

lit. $[\alpha]_D^{24}$ -27.1 (c = 1.0, $CHCl_3$) for (2*R*,3*R*)-**13**[28]

**Preparation of (2*R*,3*S*,4*E*)-2,3-Dihydroxy-5-phenyl-4-pentenoic Acid Methyl Ester ((2*R*,3*S*,4*E*)-17)**




**6ep** (2*R*,3*S*,4*E*)-**17**

To a flame-dried, 5-mL, 2-necked round-bottomed flask fitted with a magnetic stir bar, a gas inlet tube, and a septum were added **6ep** (90 mg, 0.267 mmol) and THF (0.5 mL). To the solution was added TBAF trihydrate (101 mg, 0.320 mmol, 1.2 equiv) at rt. After 10 min, the reaction mixture was transferred to a 50-mL separatory funnel ans was diluted with $Et_2O$ (10 mL) and $H_2O$ (10 mL). The organic layer was separated. The aqueous layer was extracted with $Et_2O$ (2 × 10 mL). The combined organic layers were dried over $Na_2SO_4$ (2 g), filtered, and concentrated *in vacuo* (23 $^oC$, 30 mmHg). The residue was purified by column chromatography

(18 mm diam., hexane/EtOAc, 1/1) on silica gel (10 g) to give **17** (54 mg, 91%) as a colorless oil.

Data for (2*R*,3*S*,4*E*)-**17**:

$^1$H NMR: (500 MHz, $CDCl_3$)

7.40-7.38 (m, 2 H, HC(7)), 7.32-7.29 (m, 2 H, HC(8)), 7.27-7.23 (m, 1 H, HC(9)), 6.69 (bd, *J* = 15.9, 1 H, HC(5)), 6.34 (dd, *J* = 15.9, 6.6, 1 H, HC(4)), 4.60 (ddd, *J* = 6.6, 2.7, 1.4, 1 H, HC(3)), 4.270 (d, *J* = 2.7, 1 H, HC(2)), 3.82 (s, 3 H, HC(10)), 3.39 (bs, 1 H, OH), 2.89 (bs, 1 H, OH)

$^{13}$C NMR: (126 MHz, $CDCl_3$)

173.1 (C(1)), 136.1 (C(6)), 132.5 (C(5)), 128.5 (C(8)), 127.9 (C(9)), 127.3 (C(4)), 126.6 (C(7)), 73.9 (C(2)), 73.5 (C(3)), 52.8 (C(10))

IR: (neat)

3417 (br), 3083 (m), 3059 (m), 3026 (m), 2954 (m), 1738 (s), 1731 (s), 1650 (m), 1599 (m), 1577 (m), 1495 (m), 1449 (s), 1440 (s), 1397 (m), 1283 (s), 1226 (s), 1120 (s), 1039 (s), 969 (s), 912 (m), 838 (m), 795 (m), 755 (s), 734 (s), 693 (s)

MS: (ESI)

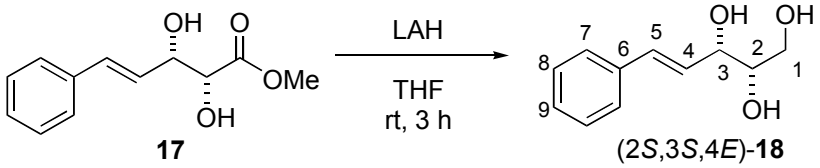
245.1 ($M^+$+Na, 100), 246.1 (15)

HRMS: calcd for $C_{12}H_{14}O_4Na$: 245.0790, found 245.0802

TLC: $R_f$ 0.29 (hexane/EtOAc, 1/1) [UV(254)/$KMnO_4$]

Opt. Rot.: $[\alpha]_D^{24}$ -24.6 (c = 1.0, EtOH)

**Preparation of (2*S*,3*S*,4*E*)-5-Phenyl-4-penten-1,2,3-triol[29] ((2*S*,3*S*,4*E*)-18)**




To a flame-dried, 10-mL, 2-necked round-bottomed flask fitted with a magnetic stir bar, a gas inlet tube, and a septum were added a solution of LAH (682 μL, 1.32 M in THF, 0.9 mmol, 2.0 equiv) and THF (1 mL). The solution was cooled to 0 °C in an ice-water bath and then a solution of **17** (100 mg, 0.45 mmol) in THF (1 mL) was added dropwise via syringe. The ice-water bath was removed and the reaction mixture was stirred at rt for 3 h. The reaction mixture was diluted with $Et_2O$ (4 mL) prior to sequential addition of $H_2O$ (30 μL), 3*N* aq. NaOH solution (30 μL), and $H_2O$ (90 μL). After 15 min, the heterogeneous mixture was transferred to a 50-mL flask where the mixture was mixed with 1*N* aq. HCl solution (20 mL) and stirred until the solution became clear. The organic layer was separated. The aqueous layer was extracted with EtOAc (4 × 20 mL). The combined organic extracts were concentrated *in vacuo* (23 °C, 30 mmHg). The residue was purified by column chromatography (18 mm diam., EtOAc) on silica gel (10 g) to give **18** (68 mg, 78%) as a colorless oil. The product contained 18% of by-product whose double bond was saturated ($^1$H NMR analysis).

Data for (2*S*,3*S*)-**18**:

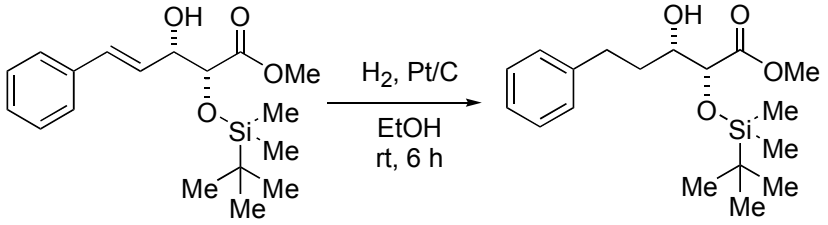
$^1$H NMR: (500 MHz, $CDCl_3$)

7.29-7.27 (m, 2 H, HC(7)), 7.23-7.19 (m, 2 H, HC(8)), 7.15-7.10 (m, 1 H, HC(9)), 6.58 (d, *J* = 15.9, 1 H, HC(5)), 6.16 (dd, *J* = 15.9, 7.0, 1 H, HC(4)), 4.26-4.23 (m, 4, HC(3), OH), 3.72 (dd, *J* = 11.2, 2.4, 1 H, HC(1)), 3.69-3.66 (m, 1 H, HC(2)), 3.61 (dd, *J* = 11.2, 6.0, 1 H, HC(1))

TLC: $R_f$ 0.27 (EtOAc) [UV(254)/$KMnO_4$]

Opt. Rot.: $[\alpha]_D^{24}$ -18.2 (c = 1.36, EtOH) lit. $[\alpha]_D^{24}$ 10 (c = 0.05, EtOH) for (2*R*,3*R*)-**18**[29]

**Hydrogenation of (2*R*,3*S*,4*E*)-2-[[(1,1-Dimethylethyl)dimethylsilyl]oxy]-3-hydroxy-5-phenyl-4-pentenoic Acid Methyl Ester ((2*S*,3*S*,4*E*)-6ep) to (2*R*,3*S*)-2-[[(1,1-Dimethyl ethyl)dimethylsilyl]oxy]-3-hydroxy-5-phenylpentanoic Acid Methyl Ester ((2*S*,3*S*)-6ej)**



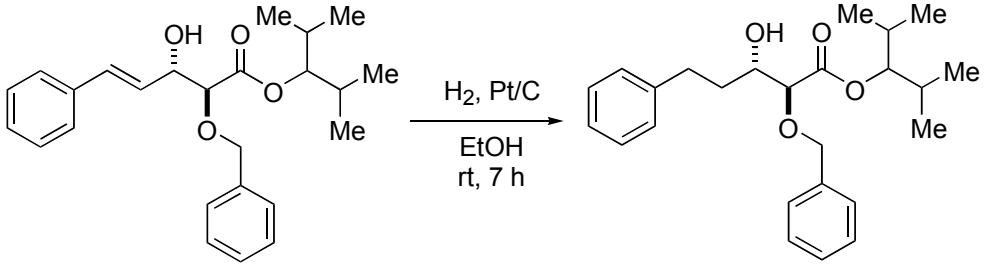

(2*R*,3*S*,4*E*)-**6ep** (2*R*,3*S*)-**6ej**

To a flame-dried, 5-mL, 2-necked round-bottomed flask fitted with a magnetic stir bar, a gas inlet tube, and a septum were added (2*R*,3*S*,4*E*)-**6ep** (45 mg, 0.134 mmol), EtOH (0.5 mL) and Pt/C (1.4 mg, 5 % on C, 0.0067 mmol, 0.05 equiv). The suspension was stirred for 6 h at rt under 1 atm of $H_2$ on a manifold. The suspension was then filtered through Celite (1 g) and the filtrate was concentrated *in vacuo* (23 $^oC$, 30 mmHg). The residue was purified by column chromatography (18 mm diam., hexane/EtOAc, 10/1 to 5/1) on silica gel (10 g) to give (2*R*,3*S*)-**6ej** (44 mg, 97%) as a colorless oil.

Data for (2*R*,3*S*)-**6ej**:

See page *S92*.

**Hydrogenation of (2*S*,3*S*,4*E*)-3-Hydroxy-5-phenyl-2-(phenylmethoxy)-4-pentenoic Acid 2-Methyl-1-(1-methylethyl)propyl Ester ((2*S*,3*S*,4*E*)-6mp) to (2*S*,3*S*)-3-Hydroxy-5-phenyl-2-(phenylmethoxy)pentanoic Acid 2-Methyl-1-(1-methylethyl)propyl Ester ((2*S*,3*S*)-6mj)**




(2*S*,3*S*,4*E*)-**6mp** (2*S*,3*S*)-**6mj**

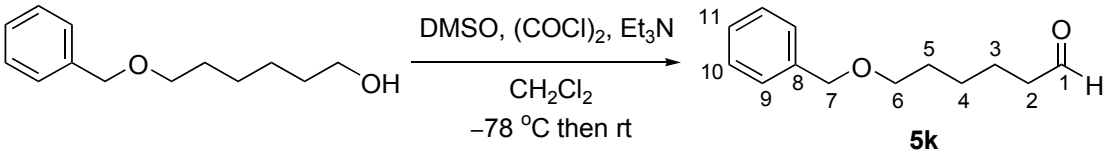
To a flame-dried, 5-mL, 2-necked round-bottomed flask fitted with a magnetic stir bar, a gas inlet tube, and a septum were added (2*S*,3*S*,4*E*)-**6mp** (114 mg, 0.288 mmol), EtOH (1 mL) and Pt/C (3 mg, 5 % on C, 0.014 mmol, 0.05 equiv). The suspension was stirred for 7 h at rt under 1 atm of $H_2$ on a manifold. The suspension was then filtered through Celite (1 g) and the filtrate was concentrated *in vacuo* (23 $^{o}$C, 30 mmHg). The residue was purified by column chromatography (22 mm diam., hexane/EtOAc, 10/1 to 5/1) on silica gel (20 g) to give (2*S*,3*S*)-**6mj** (101 mg, 88%) as a colorless oil.

Data for (2*S*,3*S*)-**6mj**:

See page *S98*.

**Miscellaneous**

**Preparation of 6-(Phenylmethoxy)hexanal[30] (5k)**




**5k**

To a flame-dried, 500-mL, 3-necked round-bottomed flask fitted with a magnetic stir bar,

a thermocouple, a gas inlet tube, and a septum was added $CH_2Cl_2$ (100 mL). The solution was cooled to −78 °C (internal temp.) in a dry ice-acetone bath prior to addition of $(COCl)_2$ (2.058 mL, 24 mmol, 1.2 equiv). While the internal temperature was maintained below −65 °C, DMSO (3.409 mL, 48 mmol, 2.4 equiv) was added dropwise via syringe over 10 min. To the resulting solution was added 6-(phenylmethoxy)hexanol (4.166 g, 20 mmol) dropwise via syringe over 5 min while the internal temperature was maintained below −70 °C. After 30 min, $Et_3N$ (11.15 mL, 80 mmol, 4.0 equiv) was added dropwise via syringe over 5 min while the internal temperature was maintained below -68 °C. The dry ice-acetone bath was removed and the reaction mixture was allowed to warm to rt over 15 min. The reaction mixture was diluted with $Et_2O$ (100 mL), filtered through $MgSO_4$ (10 g), and concentrated *in vacuo* (23 °C, 30 mmHg). The resulting material was diluted with $Et_2O$ (10 mL), filtered, and concentrated *in vacuo* (23 °C, 30 mmHg). The residue was purified by column chromatography (55 mm diam., hexane/EtOAc, 10/1 to 5/1) on silica gel (100 g) and distillation through a 5-cm Vigreux column under reduced pressure to give **5k** (3.345 g, 81%) as a colorless oil.

Data for **5k**:

bp: 95-96 °C (0.2 mmHg)

$^1$H NMR: (500 MHz, $CDCl_3$)

9.75 (t, *J* = 1.7, 1 H, HC(1)), 7.37-7.32 (m, 4 H, HC(9), HC(10)), 7.31-7.27 (m, 1 H, HC(11)), 4.50 (s, 2 H, HC(7)), 3.47 (t, *J* = 6.5, 2 H, HC(6)), 2.43 (td, *J* = 7.3, 1.7, 2 H, HC(2)), 1.68-1.61 (m, 4 H, HC(3), HC(5)), 1.45-1.39 (m, 2 H, HC(4))

$^{13}$C NMR: (126 MHz, $CDCl_3$)

202.7 (C(1)), 138.5 (C(8)), 128.3 (C(10)), 127.6 (C(9)), 127.5 (C(11)), 72.8 (C(7)), 69.9 (C(6)), 43.7 (C(2)), 29.4 (C(5)), 25.7 (C(4)), 21.8 (C(3))

IR: (neat)

3426 (w), 3064 (m), 3031 (m), 2938 (s), 2860 (s), 2721 (m), 1724 (s), 1496 (m), 1479 (m), 1454 (s), 1410 (m), 1391 (m), 1364 (m), 1310 (m), 1250 (m), 1205 (m), 1175 (m), 1100 (s), 1028 (m), 1001 (m), 909 (m), 819 (m), 737 (s), 699 (s), 611 (m)

MS: (EI, 70 eV)

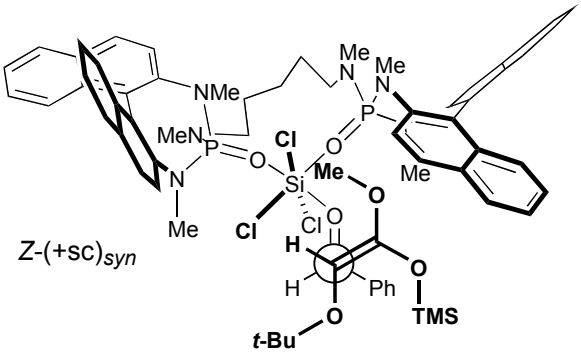
55.0 (7), 57.0 (3), 65.0 (9), 67.1 (2), 69.1 (7), 77.0 (4), 79.0 (9), 81.0 (3), 82.0 (3), 91.0 (100), 92.1 (27), 97.1 (5), 99.1 (3), 100.1 (4), 105.1 (2), 107.1 (41), 108.1 (7), 115.1 (4), 206.1 ($M^+$, 2)

HRMS: calcd for $C_{13}H_{18}O_2$: 206.1307, found 206.1297

TLC: $R_f$ 0.27 (hexane/EtOAc, 10/1) [UV(254)/$KMnO_4$]

**Computational Results**

**Atom Coordinates of Calculated Transition Structures (Figures 3)**



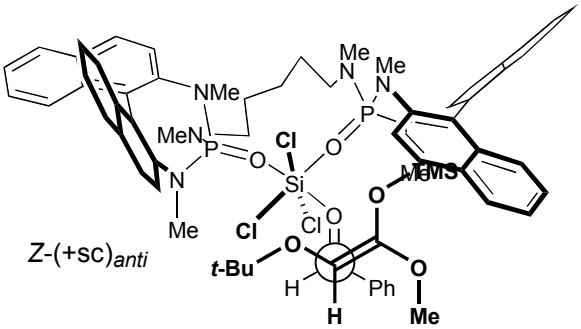

| Atom | Partial Charge | X Coordinate | Y Coordinate | Z Coordinate |
|---|---|---|---|---|
| C1 | -0.085 | -4.962 | 7.721 | -3.204 |
| C2 | -0.085 | -3.589 | 7.729 | -2.868 |
| C3 | -0.097 | -2.920 | 6.555 | -2.649 |
| C4 | -0.019 | -3.592 | 5.305 | -2.751 |
| C5 | -0.040 | -4.968 | 5.305 | -3.063 |
| C6 | -0.090 | -5.638 | 6.535 | -3.300 |
| C7 | -0.070 | -5.678 | 4.075 | -3.126 |
| C8 | -0.108 | -5.050 | 2.900 | -2.826 |
| C9 | -0.050 | -3.667 | 2.893 | -2.485 |
| C10 | -0.011 | -2.922 | 4.060 | -2.517 |
| H11 | 0.112 | -5.475 | 8.672 | -3.383 |
| H12 | 0.111 | -3.066 | 8.688 | -2.784 |
| H13 | 0.121 | -1.853 | 6.561 | -2.394 |
| H14 | 0.112 | -6.705 | 6.516 | -3.555 |
| H15 | 0.114 | -6.738 | 4.088 | -3.404 |
| H16 | 0.117 | -5.603 | 1.953 | -2.856 |
| N17 | -0.407 | -3.123 | 1.601 | -2.089 |
| C18 | -0.029 | -1.465 | 4.041 | -2.280 |
| C19 | -0.056 | -0.937 | 3.588 | -1.080 |
| C20 | -0.022 | -0.570 | 4.452 | -3.321 |
| C21 | -0.131 | 0.467 | 3.626 | -0.840 |
| C22 | -0.056 | 1.318 | 4.079 | -1.805 |
| C23 | -0.048 | 0.818 | 4.484 | -3.073 |
| C24 | -0.091 | -0.153 | 5.169 | -5.603 |
| N25 | -0.336 | -1.832 | 3.085 | -0.047 |
| C26 | -0.095 | 1.234 | 5.228 | -5.338 |
| C27 | -0.103 | -1.033 | 4.797 | -4.622 |
| P28 | 1.836 | -2.470 | 1.489 | -0.460 |
| H29 | 0.149 | 0.855 | 3.246 | 0.125 |

| | | | | |
|---|---|---|---|---|
| O30 | -0.767 | -1.377 | 0.383 | -0.320 |
| H31 | 0.117 | 2.400 | 4.110 | -1.624 |
| N32 | -0.412 | -3.840 | 1.114 | 0.578 |
| C33 | -0.083 | 1.711 | 4.894 | -4.100 |
| H34 | 0.111 | -0.517 | 5.434 | -6.602 |
| H35 | 0.111 | 1.918 | 5.544 | -6.134 |
| H36 | 0.122 | -2.111 | 4.767 | -4.825 |
| H37 | 0.114 | 2.785 | 4.930 | -3.882 |
| C38 | -0.065 | -4.399 | -0.255 | 0.382 |
| C39 | -0.069 | -4.880 | 2.152 | 0.735 |
| C40 | -0.056 | -2.639 | 0.743 | -3.187 |
| C41 | -0.075 | -1.368 | 3.279 | 1.343 |
| C42 | -0.119 | -5.120 | -0.729 | 1.643 |
| H43 | 0.053 | -5.098 | -0.281 | -0.482 |
| H44 | 0.092 | -3.554 | -0.944 | 0.134 |
| H45 | 0.048 | -5.339 | 2.459 | -0.219 |
| H46 | 0.058 | -4.441 | 3.030 | 1.226 |
| H47 | 0.063 | -5.677 | 1.750 | 1.385 |
| C48 | -0.094 | -5.505 | -2.201 | 1.551 |
| C49 | -0.114 | -4.531 | -3.115 | 2.282 |
| C50 | -0.050 | -3.193 | -3.252 | 1.547 |
| N51 | -0.472 | -2.088 | -3.505 | 2.512 |
| H52 | 0.047 | -1.768 | 1.169 | -3.711 |
| H53 | 0.076 | -2.366 | -0.244 | -2.779 |
| H54 | 0.053 | -3.450 | 0.614 | -3.917 |
| H55 | 0.082 | -0.327 | 2.929 | 1.499 |
| H56 | 0.047 | -2.037 | 2.743 | 2.029 |
| H57 | 0.061 | -1.412 | 4.352 | 1.576 |
| H58 | 0.058 | -6.018 | -0.091 | 1.802 |
| H59 | 0.065 | -4.484 | -0.555 | 2.544 |
| H60 | 0.057 | -6.513 | -2.338 | 1.991 |
| H61 | 0.061 | -5.602 | -2.517 | 0.492 |
| H62 | 0.057 | -4.384 | -2.740 | 3.318 |
| H63 | 0.057 | -4.990 | -4.117 | 2.403 |
| H64 | 0.084 | -2.957 | -2.336 | 0.955 |
| H65 | 0.065 | -3.245 | -4.074 | 0.800 |
| P66 | 1.837 | -0.748 | -2.372 | 2.656 |
| N67 | -0.327 | 0.689 | -3.241 | 3.194 |
| C68 | -0.035 | -1.915 | -4.936 | 2.836 |
| N69 | -0.326 | -1.136 | -1.353 | 4.055 |
| O70 | -0.748 | -0.645 | -1.445 | 1.389 |
| C71 | -0.066 | 1.271 | -4.272 | 2.306 |
| C72 | -0.052 | -2.114 | -0.259 | 3.919 |

| | | | | |
|---|---|---|---|---|
| C73 | -0.060 | -1.145 | -2.079 | 5.313 |
| C74 | -0.043 | 1.643 | -2.440 | 3.945 |
| C75 | -0.127 | -2.374 | -2.375 | 5.968 |
| C76 | 0.005 | 0.067 | -2.456 | 5.875 |
| C77 | -0.018 | 0.063 | -3.271 | 7.053 |
| C78 | -0.044 | -1.163 | -3.577 | 7.681 |
| C79 | -0.063 | -2.379 | -3.090 | 7.131 |
| C80 | -0.125 | 2.888 | -2.088 | 3.350 |
| C81 | -0.008 | 1.341 | -2.060 | 5.245 |
| C82 | -0.016 | 2.261 | -1.219 | 5.952 |
| C83 | -0.040 | 3.491 | -0.875 | 5.352 |
| C84 | -0.060 | 3.793 | -1.340 | 4.043 |
| C85 | -0.091 | 4.101 | 0.438 | 7.292 |
| C86 | -0.088 | 4.411 | -0.048 | 6.051 |
| C87 | -0.087 | -0.006 | -4.885 | 9.357 |
| Si88 | 1.412 | -0.105 | -0.750 | -0.083 |
| Cl89 | -0.491 | 1.060 | 0.829 | 1.046 |
| Cl90 | -0.616 | 1.678 | -2.302 | 0.044 |
| Cl91 | -0.567 | -1.449 | -2.253 | -1.232 |
| O92 | -0.340 | 0.650 | -0.229 | -1.590 |
| H93 | 0.051 | -2.892 | -5.354 | 3.119 |
| H94 | 0.035 | -1.529 | -5.519 | 1.986 |
| H95 | 0.055 | -1.234 | -5.052 | 3.689 |
| H96 | 0.082 | 2.250 | -3.972 | 1.888 |
| H97 | 0.051 | 1.404 | -5.186 | 2.900 |
| H98 | 0.053 | 0.596 | -4.481 | 1.464 |
| H99 | 0.061 | -2.223 | 0.261 | 4.880 |
| H100 | 0.056 | -1.726 | 0.458 | 3.182 |
| H101 | 0.036 | -3.119 | -0.592 | 3.594 |
| H102 | 0.111 | -3.322 | -2.031 | 5.532 |
| H103 | 0.111 | -3.321 | -3.313 | 7.646 |
| H104 | 0.142 | 3.092 | -2.423 | 2.315 |
| H105 | 0.108 | 4.756 | -1.070 | 3.593 |
| C106 | 0.398 | 0.936 | -0.967 | -2.605 |
| C107 | -0.173 | 0.739 | -0.414 | -3.963 |
| C108 | -0.056 | 0.660 | 0.957 | -4.223 |
| C109 | -0.047 | 0.597 | -1.327 | -5.017 |
| C110 | -0.117 | 0.362 | -0.869 | -6.307 |
| C111 | -0.064 | 0.270 | 0.497 | -6.559 |
| C112 | -0.129 | 0.422 | 1.407 | -5.517 |
| C113 | -0.349 | 3.116 | -1.214 | -2.654 |
| C114 | -0.100 | 1.964 | -0.686 | 7.238 |
| C115 | -0.096 | 1.259 | -3.815 | 7.599 |

| | | | | |
|---|---|---|---|---|
| C116 | -0.090 | -1.174 | -4.384 | 8.850 |
| C117 | 0.372 | 3.758 | 0.027 | -2.845 |
| H118 | 0.118 | 1.002 | -0.937 | 7.704 |
| C119 | -0.089 | 2.859 | 0.120 | 7.887 |
| O120 | -0.206 | 3.299 | -2.106 | -3.705 |
| H121 | 0.122 | 2.213 | -3.587 | 7.107 |
| C122 | -0.086 | 1.223 | -4.602 | 8.718 |
| H123 | 0.110 | -2.134 | -4.601 | 9.334 |
| H124 | 0.111 | -0.010 | -5.511 | 10.256 |
| H125 | 0.108 | 2.622 | 0.526 | 8.877 |
| H126 | 0.110 | 2.148 | -5.019 | 9.132 |
| H127 | 0.109 | 4.806 | 1.077 | 7.834 |
| H128 | 0.108 | 5.367 | 0.198 | 5.576 |
| H129 | 0.123 | 0.876 | -2.074 | -2.467 |
| H130 | 0.121 | 0.793 | 1.686 | -3.411 |
| H131 | 0.125 | 0.695 | -2.410 | -4.817 |
| H132 | 0.112 | 0.250 | -1.585 | -7.128 |
| H133 | 0.108 | 0.083 | 0.856 | -7.577 |
| H134 | 0.119 | 0.357 | 2.488 | -5.709 |
| C135 | 0.099 | 3.425 | -3.505 | -3.354 |
| O136 | -0.347 | 4.167 | 0.623 | -3.931 |
| Si137 | 0.714 | 5.106 | -0.002 | -5.236 |
| C138 | -0.163 | 4.243 | -3.748 | -2.086 |
| C139 | -0.163 | 2.024 | -4.110 | -3.206 |
| C140 | -0.136 | 4.140 | -4.123 | -4.562 |
| C141 | -0.291 | 4.006 | -0.800 | -6.539 |
| C142 | -0.268 | 5.873 | 1.590 | -5.903 |
| C143 | -0.297 | 6.440 | -1.187 | -4.625 |
| O144 | -0.198 | 3.897 | 1.014 | -1.933 |
| C145 | 0.007 | 3.781 | 0.682 | -0.559 |
| H146 | 0.188 | 3.107 | -1.626 | -1.612 |
| H147 | 0.047 | 5.268 | -3.367 | -2.189 |
| H148 | 0.076 | 3.781 | -3.251 | -1.213 |
| H149 | 0.058 | 4.305 | -4.821 | -1.866 |
| H150 | 0.049 | 1.394 | -3.873 | -4.082 |
| H151 | 0.076 | 1.508 | -3.707 | -2.315 |
| H152 | 0.056 | 2.073 | -5.200 | -3.108 |
| H153 | 0.051 | 3.551 | -4.015 | -5.483 |
| H154 | 0.058 | 5.118 | -3.644 | -4.737 |
| H155 | 0.054 | 4.313 | -5.195 | -4.400 |
| H156 | 0.066 | 4.018 | -1.893 | -6.451 |
| H157 | 0.070 | 2.963 | -0.473 | -6.441 |
| H158 | 0.062 | 4.347 | -0.539 | -7.546 |

| | | | | |
|---|---|---|---|---|
| H159 | 0.059 | 5.107 | 2.338 | -6.128 |
| H160 | 0.060 | 6.572 | 2.032 | -5.187 |
| H161 | 0.062 | 6.422 | 1.384 | -6.827 |
| H162 | 0.072 | 6.044 | -2.204 | -4.492 |
| H163 | 0.059 | 6.866 | -0.862 | -3.672 |
| H164 | 0.067 | 7.257 | -1.241 | -5.353 |
| H165 | 0.034 | 4.795 | 0.629 | -0.152 |
| H166 | 0.085 | 3.239 | -0.260 | -0.358 |
| H167 | 0.075 | 3.231 | 1.522 | -0.120 |




*Z*-(+sc)$_{anti}$

| Atom | Partial Charge | X Coordinate | Y Coordinate | Z Coordinate |
|---|---|---|---|---|
| C1 | -0.088 | -1.981 | 8.968 | -2.769 |
| C2 | -0.084 | -0.711 | 8.553 | -2.309 |
| C3 | -0.097 | -0.384 | 7.223 | -2.275 |
| C4 | -0.015 | -1.317 | 6.235 | -2.694 |
| C5 | -0.051 | -2.591 | 6.654 | -3.131 |
| C6 | -0.087 | -2.903 | 8.039 | -3.172 |
| C7 | -0.054 | -3.555 | 5.685 | -3.523 |
| C8 | -0.146 | -3.284 | 4.355 | -3.394 |
| C9 | -0.055 | -2.018 | 3.918 | -2.903 |
| C10 | -0.013 | -1.006 | 4.835 | -2.655 |
| H11 | 0.112 | -2.218 | 10.037 | -2.796 |
| H12 | 0.110 | 0.011 | 9.309 | -1.982 |
| H13 | 0.121 | 0.605 | 6.901 | -1.926 |
| H14 | 0.111 | -3.893 | 8.349 | -3.527 |
| H15 | 0.113 | -4.521 | 6.025 | -3.916 |
| H16 | 0.120 | -4.026 | 3.598 | -3.695 |
| N17 | -0.362 | -1.773 | 2.508 | -2.674 |
| C18 | -0.014 | 0.369 | 4.394 | -2.358 |
| C19 | -0.038 | 0.675 | 3.609 | -1.255 |
| C20 | -0.014 | 1.414 | 4.742 | -3.279 |
| C21 | -0.130 | 2.023 | 3.232 | -0.991 |
| C22 | -0.068 | 3.030 | 3.625 | -1.822 |
| C23 | -0.044 | 2.746 | 4.376 | -2.993 |

| | | | | |
|---|---|---|---|---|
| C24 | -0.089 | 2.171 | 5.748 | -5.356 |
| N25 | -0.315 | -0.370 | 3.205 | -0.330 |
| C26 | -0.093 | 3.507 | 5.408 | -5.045 |
| C27 | -0.100 | 1.154 | 5.426 | -4.499 |
| P28 | 1.833 | -1.577 | 2.081 | -0.969 |
| H29 | 0.133 | 2.245 | 2.599 | -0.113 |
| O30 | -0.749 | -1.137 | 0.601 | -0.804 |
| H31 | 0.118 | 4.070 | 3.332 | -1.605 |
| N32 | -0.463 | -3.055 | 2.402 | -0.065 |
| C33 | -0.089 | 3.789 | 4.733 | -3.888 |
| H34 | 0.109 | 1.960 | 6.276 | -6.293 |
| H35 | 0.109 | 4.306 | 5.688 | -5.739 |
| H36 | 0.120 | 0.119 | 5.698 | -4.745 |
| H37 | 0.107 | 4.819 | 4.456 | -3.637 |
| C38 | -0.065 | -4.179 | 1.435 | -0.201 |
| C39 | -0.067 | -3.499 | 3.805 | 0.082 |
| C40 | -0.091 | -2.459 | 1.539 | -3.553 |
| C41 | -0.089 | 0.077 | 2.870 | 1.042 |
| C42 | -0.119 | -5.045 | 1.426 | 1.059 |
| H43 | 0.042 | -4.801 | 1.688 | -1.087 |
| H44 | 0.103 | -3.759 | 0.412 | -0.380 |
| H45 | 0.042 | -3.791 | 4.264 | -0.877 |
| H46 | 0.059 | -2.696 | 4.398 | 0.538 |
| H47 | 0.062 | -4.375 | 3.832 | 0.754 |
| C48 | -0.094 | -6.164 | 0.396 | 0.949 |
| C49 | -0.111 | -5.898 | -0.854 | 1.774 |
| C50 | -0.046 | -4.705 | -1.669 | 1.256 |
| N51 | -0.474 | -3.811 | -2.061 | 2.382 |
| H52 | 0.058 | -1.749 | 1.221 | -4.336 |
| H53 | 0.087 | -2.762 | 0.638 | -2.987 |
| H54 | 0.052 | -3.351 | 1.967 | -4.041 |
| H55 | 0.083 | 0.936 | 2.169 | 1.060 |
| H56 | 0.057 | -0.753 | 2.408 | 1.608 |
| H57 | 0.058 | 0.372 | 3.799 | 1.548 |
| H58 | 0.057 | -5.463 | 2.445 | 1.223 |
| H59 | 0.063 | -4.413 | 1.227 | 1.958 |
| H60 | 0.054 | -7.114 | 0.850 | 1.295 |
| H61 | 0.060 | -6.343 | 0.119 | -0.110 |
| H62 | 0.061 | -5.757 | -0.558 | 2.836 |
| H63 | 0.055 | -6.806 | -1.489 | 1.771 |
| H64 | 0.095 | -4.112 | -1.087 | 0.506 |
| H65 | 0.056 | -5.060 | -2.575 | 0.723 |
| P66 | 1.834 | -2.061 | -1.880 | 2.294 |

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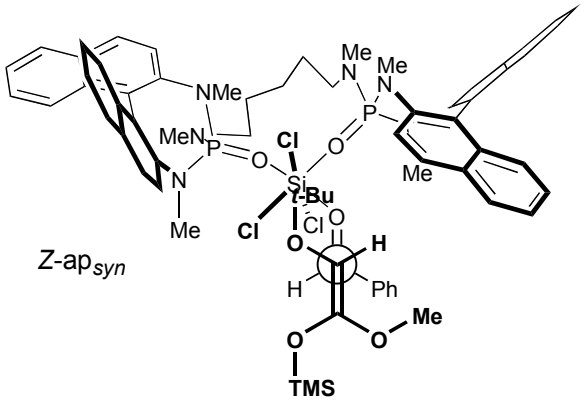
|---|---|---|---|---|
| N67 | -0.337 | -1.301 | -3.379 | 2.835 |
| C68 | -0.045 | -4.366 | -3.140 | 3.225 |
| N69 | -0.324 | -1.647 | -0.724 | 3.576 |
| O70 | -0.739 | -1.639 | -1.236 | 0.924 |
| C71 | -0.060 | -1.620 | -4.638 | 2.125 |
| C72 | -0.062 | -2.057 | 0.681 | 3.382 |
| C73 | -0.051 | -1.708 | -1.248 | 4.928 |
| C74 | -0.040 | 0.079 | -3.214 | 3.257 |
| C75 | -0.134 | -2.730 | -0.806 | 5.819 |
| C76 | 0.002 | -0.747 | -2.157 | 5.353 |
| C77 | -0.016 | -0.867 | -2.737 | 6.659 |
| C78 | -0.046 | -1.889 | -2.297 | 7.526 |
| C79 | -0.063 | -2.805 | -1.304 | 7.087 |
| C80 | -0.120 | 1.132 | -3.714 | 2.440 |
| C81 | -0.012 | 0.350 | -2.585 | 4.463 |
| C82 | -0.015 | 1.716 | -2.325 | 4.813 |
| C83 | -0.035 | 2.752 | -2.789 | 3.973 |
| C84 | -0.078 | 2.433 | -3.509 | 2.790 |
| C85 | -0.094 | 4.411 | -1.798 | 5.431 |
| C86 | -0.094 | 4.105 | -2.514 | 4.306 |
| C87 | -0.089 | -1.154 | -3.852 | 9.228 |
| Si88 | 1.398 | -0.789 | -1.072 | -0.569 |
| Cl89 | -0.508 | 1.066 | -0.409 | 0.584 |
| Cl90 | -0.598 | -0.539 | -3.412 | -0.387 |
| Cl91 | -0.533 | -2.763 | -1.469 | -1.701 |
| O92 | -0.354 | 0.129 | -1.087 | -2.064 |
| H93 | 0.053 | -5.405 | -2.896 | 3.489 |
| H94 | 0.036 | -4.367 | -4.119 | 2.720 |
| H95 | 0.053 | -3.786 | -3.216 | 4.155 |
| H96 | 0.080 | -0.744 | -5.065 | 1.603 |
| H97 | 0.049 | -1.977 | -5.360 | 2.872 |
| H98 | 0.054 | -2.405 | -4.474 | 1.373 |
| H99 | 0.059 | -1.905 | 1.262 | 4.302 |
| H100 | 0.058 | -1.414 | 1.125 | 2.599 |
| H101 | 0.040 | -3.114 | 0.797 | 3.070 |
| H102 | 0.112 | -3.463 | -0.062 | 5.485 |
| H103 | 0.111 | -3.582 | -0.957 | 7.778 |
| H104 | 0.150 | 0.874 | -4.242 | 1.502 |
| H105 | 0.114 | 3.234 | -3.869 | 2.120 |
| C106 | 0.373 | 0.580 | -2.085 | -2.752 |
| C107 | -0.170 | 0.295 | -2.105 | -4.217 |
| C108 | -0.066 | -0.358 | -1.063 | -4.879 |
| C109 | -0.090 | 0.611 | -3.271 | -4.928 |

| | | | | |
|---|---|---|---|---|
| C110 | -0.107 | 0.299 | -3.380 | -6.276 |
| C111 | -0.070 | -0.339 | -2.331 | -6.933 |
| C112 | -0.116 | -0.669 | -1.177 | -6.231 |
| C113 | -0.289 | 2.667 | -2.036 | -2.773 |
| C114 | -0.095 | 2.068 | -1.575 | 5.969 |
| C115 | -0.098 | -0.003 | -3.775 | 7.106 |
| C116 | -0.089 | -2.009 | -2.863 | 8.823 |
| C117 | 0.422 | 3.241 | -0.721 | -2.778 |
| H118 | 0.118 | 1.266 | -1.206 | 6.622 |
| C119 | -0.095 | 3.379 | -1.321 | 6.270 |
| O120 | -0.200 | 2.999 | -2.736 | -1.612 |
| H121 | 0.122 | 0.784 | -4.134 | 6.431 |
| C122 | -0.087 | -0.146 | -4.317 | 8.355 |
| H123 | 0.110 | -2.801 | -2.498 | 9.488 |
| H124 | 0.110 | -1.244 | -4.294 | 10.226 |
| H125 | 0.107 | 3.641 | -0.744 | 7.163 |
| H126 | 0.109 | 0.522 | -5.118 | 8.690 |
| H127 | 0.106 | 5.453 | -1.585 | 5.692 |
| H128 | 0.104 | 4.895 | -2.875 | 3.636 |
| H129 | 0.132 | 0.624 | -3.090 | -2.256 |
| H130 | 0.128 | -0.659 | -0.152 | -4.334 |
| H131 | 0.120 | 1.094 | -4.116 | -4.407 |
| H132 | 0.112 | 0.545 | -4.298 | -6.822 |
| H133 | 0.110 | -0.590 | -2.420 | -7.995 |
| H134 | 0.109 | -1.188 | -0.356 | -6.738 |
| C135 | 0.098 | 3.401 | -4.117 | -1.780 |
| O136 | -0.337 | 3.733 | -0.012 | -1.816 |
| Si137 | 0.746 | 5.027 | -0.342 | -0.715 |
| C138 | -0.161 | 4.483 | -4.279 | -2.851 |
| C139 | -0.142 | 3.962 | -4.493 | -0.403 |
| C140 | -0.181 | 2.193 | -4.995 | -2.117 |
| C141 | -0.318 | 4.466 | -1.223 | 0.834 |
| C142 | -0.309 | 6.372 | -1.299 | -1.628 |
| C143 | -0.284 | 5.610 | 1.413 | -0.334 |
| O144 | -0.195 | 3.258 | 0.168 | -3.793 |
| C145 | 0.032 | 2.995 | -0.266 | -5.112 |
| H146 | 0.124 | 2.794 | -2.603 | -3.721 |
| H147 | 0.041 | 4.133 | -3.893 | -3.823 |
| H148 | 0.058 | 5.403 | -3.732 | -2.587 |
| H149 | 0.064 | 4.746 | -5.336 | -2.983 |
| H150 | 0.067 | 3.201 | -4.421 | 0.397 |
| H151 | 0.056 | 4.787 | -3.822 | -0.105 |
| H152 | 0.054 | 4.346 | -5.521 | -0.410 |

| | | | | |
|---|---|---|---|---|
| H153 | 0.100 | 1.280 | -4.638 | -1.600 |
| H154 | 0.042 | 1.972 | -4.990 | -3.199 |
| H155 | 0.059 | 2.366 | -6.037 | -1.824 |
| H156 | 0.117 | 3.384 | -1.086 | 0.990 |
| H157 | 0.068 | 4.656 | -2.305 | 0.774 |
| H158 | 0.065 | 4.982 | -0.837 | 1.719 |
| H159 | 0.067 | 6.064 | -2.329 | -1.858 |
| H160 | 0.058 | 6.656 | -0.815 | -2.567 |
| H161 | 0.071 | 7.270 | -1.356 | -1.003 |
| H162 | 0.069 | 4.866 | 2.162 | -0.642 |
| H163 | 0.059 | 6.545 | 1.643 | -0.853 |
| H164 | 0.065 | 5.780 | 1.536 | 0.740 |
| H165 | 0.062 | 1.945 | -0.552 | -5.245 |
| H166 | 0.035 | 3.652 | -1.094 | -5.405 |
| H167 | 0.066 | 3.224 | 0.627 | -5.699 |




*Z*-ap$_{syn}$

| Atom | Partial Charge | X Coordinate | Y Coordinate | Z Coordinate |
|---|---|---|---|---|
| C1 | -0.087 | 1.779 | 9.023 | -2.498 |
| C2 | -0.085 | 2.708 | 8.021 | -2.138 |
| C3 | -0.099 | 2.323 | 6.708 | -2.072 |
| C4 | -0.018 | 0.982 | 6.329 | -2.357 |
| C5 | -0.043 | 0.054 | 7.337 | -2.689 |
| C6 | -0.088 | 0.480 | 8.690 | -2.768 |
| C7 | -0.069 | -1.305 | 6.995 | -2.928 |
| C8 | -0.111 | -1.730 | 5.708 | -2.764 |
| C9 | -0.036 | -0.806 | 4.688 | -2.397 |
| C10 | -0.020 | 0.547 | 4.964 | -2.283 |
| H11 | 0.111 | 2.112 | 10.066 | -2.554 |
| H12 | 0.110 | 3.741 | 8.307 | -1.913 |
| H13 | 0.119 | 3.045 | 5.929 | -1.798 |
| H14 | 0.112 | -0.252 | 9.460 | -3.042 |

| | | | | |
|---|---|---|---|---|
| H15 | 0.114 | -2.007 | 7.783 | -3.226 |
| H16 | 0.118 | -2.785 | 5.449 | -2.922 |
| N17 | -0.421 | -1.381 | 3.381 | -2.115 |
| C18 | -0.025 | 1.534 | 3.888 | -2.069 |
| C19 | -0.041 | 1.487 | 3.071 | -0.949 |
| C20 | -0.020 | 2.538 | 3.649 | -3.065 |
| C21 | -0.123 | 2.461 | 2.048 | -0.756 |
| C22 | -0.087 | 3.449 | 1.851 | -1.676 |
| C23 | -0.051 | 3.509 | 2.649 | -2.850 |
| C24 | -0.093 | 3.569 | 4.148 | -5.208 |
| N25 | -0.341 | 0.442 | 3.271 | 0.044 |
| C26 | -0.091 | 4.562 | 3.172 | -4.966 |
| C27 | -0.094 | 2.585 | 4.381 | -4.285 |
| P28 | 1.854 | -1.141 | 2.706 | -0.510 |
| H29 | 0.160 | 2.383 | 1.388 | 0.133 |
| O30 | -0.754 | -1.212 | 1.159 | -0.512 |
| H31 | 0.112 | 4.198 | 1.062 | -1.525 |
| N32 | -0.420 | -2.365 | 3.413 | 0.534 |
| C33 | -0.107 | 4.529 | 2.433 | -3.815 |
| H34 | 0.112 | 3.601 | 4.722 | -6.140 |
| H35 | 0.110 | 5.353 | 3.018 | -5.708 |
| H36 | 0.124 | 1.822 | 5.148 | -4.471 |
| H37 | 0.107 | 5.295 | 1.674 | -3.613 |
| C38 | -0.069 | -3.733 | 2.824 | 0.446 |
| C39 | -0.070 | -2.370 | 4.885 | 0.671 |
| C40 | -0.039 | -1.698 | 2.527 | -3.273 |
| C41 | -0.083 | 0.797 | 2.908 | 1.436 |
| C42 | -0.119 | -4.487 | 3.035 | 1.759 |
| H43 | 0.050 | -4.303 | 3.275 | -0.394 |
| H44 | 0.104 | -3.645 | 1.728 | 0.225 |
| H45 | 0.046 | -2.546 | 5.412 | -0.281 |
| H46 | 0.057 | -1.411 | 5.212 | 1.093 |
| H47 | 0.064 | -3.175 | 5.168 | 1.372 |
| C48 | -0.094 | -5.842 | 2.338 | 1.745 |
| C49 | -0.112 | -5.844 | 1.033 | 2.530 |
| C50 | -0.047 | -5.041 | -0.076 | 1.839 |
| N51 | -0.474 | -4.251 | -0.851 | 2.838 |
| H52 | 0.030 | -0.801 | 2.075 | -3.735 |
| H53 | 0.080 | -2.365 | 1.714 | -2.941 |
| H54 | 0.050 | -2.214 | 3.124 | -4.038 |
| H55 | 0.085 | 1.383 | 1.970 | 1.501 |
| H56 | 0.054 | -0.119 | 2.773 | 2.040 |
| H57 | 0.057 | 1.389 | 3.726 | 1.865 |

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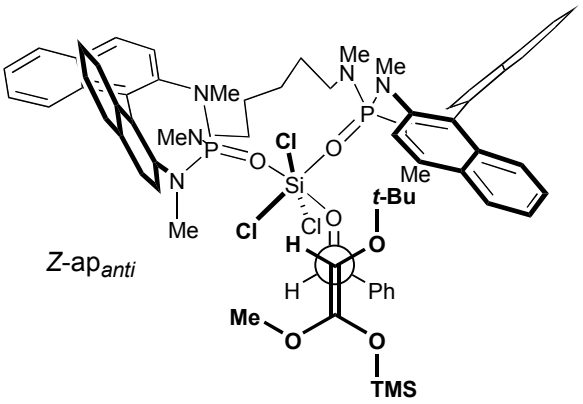
|---|---|---|---|---|
| H58 | 0.056 | -4.606 | 4.128 | 1.934 |
| H59 | 0.064 | -3.873 | 2.673 | 2.619 |
| H60 | 0.054 | -6.602 | 3.013 | 2.186 |
| H61 | 0.062 | -6.182 | 2.156 | 0.705 |
| H62 | 0.059 | -5.464 | 1.227 | 3.556 |
| H63 | 0.055 | -6.890 | 0.697 | 2.672 |
| H64 | 0.092 | -4.349 | 0.349 | 1.069 |
| H65 | 0.058 | -5.721 | -0.756 | 1.284 |
| P66 | 1.834 | -2.521 | -1.113 | 2.634 |
| N67 | -0.334 | -2.092 | -2.765 | 3.084 |
| C68 | -0.040 | -5.057 | -1.838 | 3.583 |
| N69 | -0.327 | -1.748 | -0.146 | 3.907 |
| O70 | -0.743 | -2.070 | -0.554 | 1.238 |
| C71 | -0.063 | -2.722 | -3.895 | 2.364 |
| C72 | -0.060 | -1.759 | 1.325 | 3.790 |
| C73 | -0.053 | -1.860 | -0.692 | 5.247 |
| C74 | -0.043 | -0.683 | -2.927 | 3.405 |
| C75 | -0.132 | -2.698 | -0.057 | 6.210 |
| C76 | 0.004 | -1.122 | -1.819 | 5.586 |
| C77 | -0.017 | -1.307 | -2.408 | 6.880 |
| C78 | -0.046 | -2.149 | -1.777 | 7.819 |
| C79 | -0.065 | -2.823 | -0.577 | 7.466 |
| C80 | -0.114 | 0.183 | -3.588 | 2.487 |
| C81 | -0.010 | -0.205 | -2.444 | 4.615 |
| C82 | -0.016 | 1.201 | -2.518 | 4.881 |
| C83 | -0.036 | 2.054 | -3.146 | 3.949 |
| C84 | -0.075 | 1.516 | -3.693 | 2.752 |
| C85 | -0.094 | 3.975 | -2.653 | 5.338 |
| C86 | -0.093 | 3.451 | -3.209 | 4.203 |
| C87 | -0.089 | -1.716 | -3.532 | 9.428 |
| Si88 | 1.388 | -1.316 | -0.554 | -0.314 |
| Cl89 | -0.543 | 0.701 | -0.403 | 0.792 |
| Cl90 | -0.579 | -1.647 | -2.869 | -0.258 |
| Cl91 | -0.500 | -3.355 | -0.323 | -1.328 |
| O92 | -0.359 | -0.473 | -0.727 | -1.840 |
| H93 | 0.051 | -5.948 | -1.336 | 3.988 |
| H94 | 0.035 | -5.396 | -2.675 | 2.952 |
| H95 | 0.053 | -4.475 | -2.236 | 4.425 |
| H96 | 0.083 | -2.005 | -4.445 | 1.727 |
| H97 | 0.049 | -3.130 | -4.582 | 3.117 |
| H98 | 0.053 | -3.537 | -3.538 | 1.719 |
| H99 | 0.059 | -1.379 | 1.787 | 4.712 |
| H100 | 0.057 | -1.082 | 1.620 | 2.967 |

| | | | | |
|---|---|---|---|---|
| H101 | 0.038 | -2.763 | 1.747 | 3.584 |
| H102 | 0.111 | -3.247 | 0.854 | 5.941 |
| H103 | 0.111 | -3.457 | -0.086 | 8.213 |
| H104 | 0.150 | -0.246 | -3.980 | 1.544 |
| H105 | 0.116 | 2.191 | -4.168 | 2.019 |
| C106 | 0.410 | -0.144 | -1.775 | -2.521 |
| C107 | -0.186 | -0.487 | -1.739 | -3.975 |
| C108 | -0.076 | -0.657 | -0.549 | -4.686 |
| C109 | -0.056 | -0.743 | -2.959 | -4.612 |
| C110 | -0.110 | -1.142 | -2.985 | -5.943 |
| C111 | -0.067 | -1.297 | -1.796 | -6.649 |
| C112 | -0.117 | -1.057 | -0.580 | -6.017 |
| C113 | -0.272 | 1.948 | -1.779 | -2.529 |
| C114 | -0.096 | 1.779 | -1.940 | 6.046 |
| C115 | -0.096 | -0.690 | -3.638 | 7.243 |
| C116 | -0.089 | -2.333 | -2.354 | 9.104 |
| C117 | 0.409 | 2.374 | -2.340 | -3.781 |
| H118 | 0.117 | 1.121 | -1.442 | 6.769 |
| C119 | -0.095 | 3.128 | -2.007 | 6.267 |
| O120 | -0.182 | 2.165 | -2.613 | -1.442 |
| H121 | 0.122 | -0.045 | -4.140 | 6.511 |
| C122 | -0.088 | -0.892 | -4.183 | 8.482 |
| H123 | 0.110 | -2.980 | -1.843 | 9.826 |
| H124 | 0.110 | -1.857 | -3.983 | 10.416 |
| H125 | 0.107 | 3.565 | -1.561 | 7.166 |
| H126 | 0.109 | -0.415 | -5.132 | 8.754 |
| H127 | 0.106 | 5.051 | -2.700 | 5.538 |
| H128 | 0.104 | 4.098 | -3.704 | 3.469 |
| H129 | 0.124 | -0.174 | -2.772 | -2.029 |
| H130 | 0.120 | -0.509 | 0.422 | -4.183 |
| H131 | 0.118 | -0.638 | -3.899 | -4.054 |
| H132 | 0.111 | -1.342 | -3.944 | -6.433 |
| H133 | 0.109 | -1.618 | -1.817 | -7.696 |
| H134 | 0.107 | -1.194 | 0.358 | -6.566 |
| C135 | 0.087 | 3.488 | -2.609 | -0.837 |
| O136 | -0.352 | 2.402 | -3.608 | -4.002 |
| Si137 | 0.756 | 3.461 | -4.649 | -4.882 |
| C138 | -0.136 | 3.665 | -4.049 | -0.336 |
| C139 | -0.166 | 4.643 | -2.249 | -1.777 |
| C140 | -0.169 | 3.498 | -1.641 | 0.347 |
| C141 | -0.303 | 5.155 | -3.858 | -5.091 |
| C142 | -0.286 | 3.544 | -6.089 | -3.670 |
| C143 | -0.281 | 2.640 | -5.080 | -6.510 |

| | | | | |
|---|---|---|---|---|
| O144 | -0.220 | 2.686 | -1.742 | -4.943 |
| C145 | 0.038 | 2.741 | -0.329 | -5.003 |
| H146 | 0.123 | 2.157 | -0.708 | -2.353 |
| H147 | 0.062 | 2.907 | -4.306 | 0.425 |
| H148 | 0.053 | 3.584 | -4.783 | -1.158 |
| H149 | 0.046 | 4.650 | -4.178 | 0.132 |
| H150 | 0.037 | 4.546 | -1.228 | -2.175 |
| H151 | 0.052 | 4.729 | -2.937 | -2.636 |
| H152 | 0.062 | 5.598 | -2.295 | -1.236 |
| H153 | 0.056 | 3.297 | -0.607 | 0.026 |
| H154 | 0.094 | 2.718 | -1.900 | 1.083 |
| H155 | 0.043 | 4.466 | -1.658 | 0.863 |
| H156 | 0.064 | 5.167 | -3.102 | -5.881 |
| H157 | 0.065 | 5.478 | -3.379 | -4.156 |
| H158 | 0.074 | 5.898 | -4.619 | -5.349 |
| H159 | 0.075 | 3.621 | -5.720 | -2.634 |
| H160 | 0.067 | 2.656 | -6.724 | -3.729 |
| H161 | 0.066 | 4.419 | -6.715 | -3.870 |
| H162 | 0.070 | 3.038 | -6.022 | -6.903 |
| H163 | 0.063 | 2.805 | -4.307 | -7.266 |
| H164 | 0.066 | 1.558 | -5.203 | -6.390 |
| H165 | 0.064 | 3.090 | -0.148 | -6.023 |
| H166 | 0.050 | 3.452 | 0.085 | -4.273 |
| H167 | 0.056 | 1.747 | 0.111 | -4.853 |




*Z*-ap$_{anti}$

| Atom | Partial Charge | X Coordinate | Y Coordinate | Z Coordinate |
|---|---|---|---|---|
| C1 | -0.084 | -3.480 | 8.849 | -2.599 |
| C2 | -0.086 | -2.142 | 8.482 | -2.871 |
| C3 | -0.095 | -1.758 | 7.169 | -2.819 |
| C4 | -0.022 | -2.694 | 6.151 | -2.485 |
| C5 | -0.039 | -4.022 | 6.525 | -2.191 |

| | | | | |
|---|---|---|---|---|
| C6 | -0.091 | -4.401 | 7.893 | -2.266 |
| C7 | -0.074 | -4.968 | 5.535 | -1.810 |
| C8 | -0.102 | -4.581 | 4.234 | -1.656 |
| C9 | -0.066 | -3.234 | 3.854 | -1.919 |
| C10 | -0.010 | -2.319 | 4.770 | -2.409 |
| H11 | 0.112 | -3.767 | 9.904 | -2.656 |
| H12 | 0.111 | -1.416 | 9.262 | -3.125 |
| H13 | 0.121 | -0.722 | 6.880 | -3.037 |
| H14 | 0.112 | -5.441 | 8.166 | -2.051 |
| H15 | 0.114 | -6.007 | 5.835 | -1.629 |
| H16 | 0.119 | -5.304 | 3.471 | -1.337 |
| N17 | -0.394 | -2.891 | 2.471 | -1.612 |
| C18 | -0.036 | -0.978 | 4.333 | -2.839 |
| C19 | -0.040 | -0.092 | 3.723 | -1.960 |
| C20 | -0.015 | -0.610 | 4.481 | -4.217 |
| C21 | -0.142 | 1.198 | 3.321 | -2.406 |
| C22 | -0.067 | 1.575 | 3.512 | -3.703 |
| C23 | -0.047 | 0.676 | 4.086 | -4.640 |
| C24 | -0.084 | -1.132 | 5.116 | -6.502 |
| N25 | -0.349 | -0.462 | 3.518 | -0.568 |
| C26 | -0.091 | 0.168 | 4.743 | -6.912 |
| C27 | -0.104 | -1.512 | 4.989 | -5.194 |
| P28 | 1.835 | -1.725 | 2.304 | -0.305 |
| H29 | 0.140 | 1.892 | 2.821 | -1.699 |
| O30 | -0.750 | -1.084 | 0.888 | -0.197 |
| H31 | 0.124 | 2.570 | 3.184 | -4.047 |
| N32 | -0.410 | -2.590 | 2.696 | 1.178 |
| C33 | -0.087 | 1.054 | 4.236 | -6.001 |
| H34 | 0.111 | -1.833 | 5.510 | -7.246 |
| H35 | 0.111 | 0.454 | 4.863 | -7.963 |
| H36 | 0.122 | -2.522 | 5.286 | -4.881 |
| H37 | 0.111 | 2.063 | 3.934 | -6.305 |
| C38 | -0.069 | -3.757 | 1.803 | 1.432 |
| C39 | -0.071 | -2.860 | 4.120 | 1.468 |
| C40 | -0.065 | -2.932 | 1.513 | -2.733 |
| C41 | -0.066 | 0.669 | 3.457 | 0.385 |
| C42 | -0.120 | -4.151 | 1.823 | 2.908 |
| H43 | 0.054 | -4.629 | 2.098 | 0.807 |
| H44 | 0.094 | -3.469 | 0.768 | 1.119 |
| H45 | 0.049 | -3.434 | 4.625 | 0.673 |
| H46 | 0.058 | -1.907 | 4.644 | 1.618 |
| H47 | 0.063 | -3.445 | 4.183 | 2.402 |
| C48 | -0.093 | -5.179 | 0.745 | 3.230 |

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|---|---|---|---|---|
| C49 | -0.113 | -4.558 | -0.487 | 3.875 |
| C50 | -0.048 | -3.744 | -1.324 | 2.881 |
| N51 | -0.474 | -2.586 | -1.965 | 3.564 |
| H52 | 0.041 | -2.026 | 1.547 | -3.368 |
| H53 | 0.087 | -3.034 | 0.498 | -2.312 |
| H54 | 0.058 | -3.805 | 1.725 | -3.363 |
| H55 | 0.074 | 1.429 | 2.700 | 0.104 |
| H56 | 0.049 | 0.280 | 3.219 | 1.384 |
| H57 | 0.058 | 1.155 | 4.440 | 0.422 |
| H58 | 0.057 | -4.540 | 2.835 | 3.161 |
| H59 | 0.065 | -3.249 | 1.698 | 3.553 |
| H60 | 0.056 | -5.935 | 1.162 | 3.924 |
| H61 | 0.061 | -5.744 | 0.455 | 2.321 |
| H62 | 0.057 | -3.935 | -0.171 | 4.740 |
| H63 | 0.056 | -5.360 | -1.113 | 4.314 |
| H64 | 0.087 | -3.370 | -0.701 | 2.032 |
| H65 | 0.063 | -4.387 | -2.099 | 2.412 |
| P66 | 1.847 | -0.935 | -1.632 | 3.053 |
| N67 | -0.339 | 0.067 | -3.050 | 3.364 |
| C68 | -0.036 | -2.934 | -3.240 | 4.224 |
| N69 | -0.332 | -0.300 | -0.466 | 4.230 |
| O70 | -0.744 | -0.913 | -0.961 | 1.632 |
| C71 | -0.059 | -0.224 | -4.329 | 2.679 |
| C72 | -0.051 | -0.682 | 0.954 | 4.133 |
| C73 | -0.053 | -0.139 | -1.001 | 5.570 |
| C74 | -0.039 | 1.478 | -2.761 | 3.569 |
| C75 | -0.128 | -1.032 | -0.618 | 6.612 |
| C76 | 0.001 | 0.914 | -1.871 | 5.823 |
| C77 | -0.018 | 1.011 | -2.488 | 7.113 |
| C78 | -0.045 | 0.109 | -2.114 | 8.131 |
| C79 | -0.064 | -0.898 | -1.148 | 7.863 |
| C80 | -0.134 | 2.419 | -3.095 | 2.555 |
| C81 | -0.003 | 1.890 | -2.190 | 4.765 |
| C82 | -0.014 | 3.274 | -1.855 | 4.932 |
| C83 | -0.038 | 4.198 | -2.193 | 3.921 |
| C84 | -0.066 | 3.745 | -2.831 | 2.735 |
| C85 | -0.090 | 6.001 | -1.212 | 5.204 |
| C86 | -0.090 | 5.572 | -1.870 | 4.084 |
| C87 | -0.088 | 1.146 | -3.680 | 9.658 |
| Si88 | 1.407 | -0.626 | -0.764 | -0.054 |
| Cl89 | -0.518 | 1.507 | -0.081 | 0.420 |
| Cl90 | -0.623 | -0.073 | -3.059 | -0.014 |
| Cl91 | -0.526 | -2.808 | -1.306 | -0.488 |

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|---|---|---|---|---|
| O92 | -0.340 | -0.221 | -0.721 | -1.758 |
| H93 | 0.051 | -3.821 | -3.081 | 4.854 |
| H94 | 0.035 | -3.169 | -4.039 | 3.503 |
| H95 | 0.053 | -2.110 | -3.571 | 4.869 |
| H96 | 0.074 | 0.550 | -4.594 | 1.935 |
| H97 | 0.049 | -0.267 | -5.115 | 3.445 |
| H98 | 0.059 | -1.189 | -4.276 | 2.157 |
| H99 | 0.059 | -0.183 | 1.528 | 4.925 |
| H100 | 0.058 | -0.337 | 1.336 | 3.161 |
| H101 | 0.036 | -1.772 | 1.129 | 4.217 |
| H102 | 0.111 | -1.833 | 0.104 | 6.405 |
| H103 | 0.111 | -1.574 | -0.848 | 8.672 |
| H104 | 0.140 | 2.049 | -3.548 | 1.610 |
| H105 | 0.102 | 4.472 | -3.086 | 1.954 |
| C106 | 0.408 | 0.088 | -1.634 | -2.609 |
| C107 | -0.149 | -0.199 | -1.377 | -4.043 |
| C108 | -0.042 | -0.467 | -0.107 | -4.559 |
| C109 | -0.077 | -0.193 | -2.475 | -4.911 |
| C110 | -0.118 | -0.371 | -2.296 | -6.276 |
| C111 | -0.062 | -0.620 | -1.025 | -6.786 |
| C112 | -0.118 | -0.685 | 0.063 | -5.922 |
| C113 | -0.307 | 2.230 | -1.645 | -2.610 |
| C114 | -0.098 | 3.750 | -1.158 | 6.077 |
| C115 | -0.098 | 1.974 | -3.496 | 7.396 |
| C116 | -0.089 | 0.202 | -2.719 | 9.413 |
| C117 | 0.390 | 2.563 | -2.992 | -2.951 |
| H118 | 0.119 | 3.033 | -0.877 | 6.860 |
| C119 | -0.090 | 5.076 | -0.845 | 6.208 |
| O120 | -0.183 | 2.563 | -0.675 | -3.543 |
| H121 | 0.120 | 2.669 | -3.798 | 6.602 |
| C122 | -0.087 | 2.036 | -4.076 | 8.634 |
| H123 | 0.110 | -0.497 | -2.407 | 10.197 |
| H124 | 0.110 | 1.220 | -4.151 | 10.644 |
| H125 | 0.108 | 5.434 | -0.308 | 7.093 |
| H126 | 0.109 | 2.778 | -4.854 | 8.844 |
| H127 | 0.108 | 7.059 | -0.959 | 5.334 |
| H128 | 0.106 | 6.279 | -2.152 | 3.295 |
| H129 | 0.095 | 0.064 | -2.694 | -2.280 |
| H130 | 0.116 | -0.524 | 0.765 | -3.888 |
| H131 | 0.116 | -0.029 | -3.495 | -4.519 |
| H132 | 0.113 | -0.267 | -3.165 | -6.944 |
| H133 | 0.107 | -0.764 | -0.883 | -7.862 |
| H134 | 0.109 | -0.897 | 1.063 | -6.317 |

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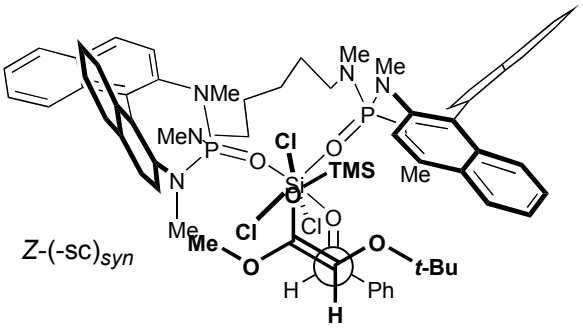
|---|---|---|---|---|
| C135 | 0.082 | 3.837 | -0.019 | -3.296 |
| O136 | -0.337 | 2.659 | -3.444 | -4.158 |
| Si137 | 0.732 | 2.472 | -5.056 | -4.769 |
| C138 | -0.176 | 3.800 | 0.764 | -1.982 |
| C139 | -0.145 | 5.004 | -1.013 | -3.271 |
| C140 | -0.127 | 4.011 | 0.926 | -4.488 |
| C141 | -0.284 | 4.037 | -6.052 | -4.474 |
| C142 | -0.297 | 0.937 | -5.870 | -4.037 |
| C143 | -0.287 | 2.202 | -4.698 | -6.602 |
| O144 | -0.224 | 2.717 | -4.057 | -2.143 |
| C145 | 0.021 | 3.091 | -3.862 | -0.790 |
| H146 | 0.158 | 2.383 | -1.326 | -1.551 |
| H147 | 0.054 | 3.096 | 1.613 | -2.028 |
| H148 | 0.082 | 3.464 | 0.121 | -1.148 |
| H149 | 0.051 | 4.789 | 1.166 | -1.731 |
| H150 | 0.055 | 5.037 | -1.622 | -4.184 |
| H151 | 0.034 | 4.933 | -1.698 | -2.414 |
| H152 | 0.056 | 5.962 | -0.484 | -3.190 |
| H153 | 0.054 | 4.027 | 0.380 | -5.440 |
| H154 | 0.051 | 3.198 | 1.669 | -4.545 |
| H155 | 0.048 | 4.955 | 1.479 | -4.403 |
| H156 | 0.063 | 4.930 | -5.490 | -4.765 |
| H157 | 0.061 | 4.148 | -6.336 | -3.424 |
| H158 | 0.068 | 4.019 | -6.973 | -5.067 |
| H159 | 0.069 | 1.095 | -6.208 | -3.009 |
| H160 | 0.063 | 0.091 | -5.168 | -4.034 |
| H161 | 0.069 | 0.646 | -6.740 | -4.634 |
| H162 | 0.067 | 3.009 | -4.087 | -7.016 |
| H163 | 0.068 | 1.255 | -4.162 | -6.774 |
| H164 | 0.066 | 2.166 | -5.634 | -7.168 |
| H165 | 0.099 | 2.234 | -3.557 | -0.162 |
| H166 | 0.039 | 3.905 | -3.132 | -0.694 |
| H167 | 0.054 | 3.437 | -4.857 | -0.500 |




*Z*-(-sc)$_{syn}$

| Atom | Partial Charge | X Coordinate | Y Coordinate | Z Coordinate |
|---|---|---|---|---|
| C1 | -0.085 | -1.662 | 9.084 | -2.860 |
| C2 | -0.086 | -0.382 | 8.516 | -2.666 |
| C3 | -0.096 | -0.230 | 7.158 | -2.587 |
| C4 | -0.020 | -1.355 | 6.293 | -2.692 |
| C5 | -0.040 | -2.633 | 6.867 | -2.861 |
| C6 | -0.090 | -2.764 | 8.279 | -2.955 |
| C7 | -0.072 | -3.780 | 6.031 | -2.923 |
| C8 | -0.104 | -3.660 | 4.680 | -2.756 |
| C9 | -0.055 | -2.378 | 4.096 | -2.556 |
| C10 | -0.009 | -1.230 | 4.869 | -2.600 |
| H11 | 0.111 | -1.758 | 10.173 | -2.929 |
| H12 | 0.110 | 0.488 | 9.176 | -2.581 |
| H13 | 0.120 | 0.763 | 6.714 | -2.443 |
| H14 | 0.112 | -3.762 | 8.708 | -3.101 |
| H15 | 0.114 | -4.762 | 6.488 | -3.094 |
| H16 | 0.117 | -4.549 | 4.036 | -2.784 |
| N17 | -0.401 | -2.372 | 2.663 | -2.284 |
| C18 | -0.029 | 0.103 | 4.239 | -2.527 |
| C19 | -0.055 | 0.480 | 3.488 | -1.423 |
| C20 | -0.021 | 0.994 | 4.333 | -3.645 |
| C21 | -0.138 | 1.761 | 2.870 | -1.364 |
| C22 | -0.078 | 2.623 | 2.981 | -2.415 |
| C23 | -0.042 | 2.253 | 3.701 | -3.581 |
| C24 | -0.088 | 1.510 | 5.064 | -5.903 |
| N25 | -0.341 | -0.447 | 3.326 | -0.312 |
| C26 | -0.087 | 2.772 | 4.431 | -5.829 |
| C27 | -0.101 | 0.642 | 5.012 | -4.845 |
| P28 | 1.832 | -1.731 | 2.179 | -0.716 |
| H29 | 0.152 | 2.037 | 2.249 | -0.481 |
| O30 | -0.760 | -1.197 | 0.715 | -0.695 |
| H31 | 0.132 | 3.604 | 2.463 | -2.383 |
| N32 | -0.411 | -3.070 | 2.366 | 0.411 |
| C33 | -0.105 | 3.134 | 3.759 | -4.694 |
| H34 | 0.110 | 1.234 | 5.596 | -6.820 |
| H35 | 0.110 | 3.448 | 4.482 | -6.689 |
| H36 | 0.122 | -0.337 | 5.505 | -4.907 |
| H37 | 0.119 | 4.099 | 3.230 | -4.619 |
| C38 | -0.068 | -4.199 | 1.427 | 0.150 |
| C39 | -0.071 | -3.511 | 3.742 | 0.724 |
| C40 | -0.061 | -2.259 | 1.781 | -3.459 |
| C41 | -0.073 | 0.183 | 3.185 | 1.018 |

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|---|---|---|---|---|
| C42 | -0.119 | -5.059 | 1.248 | 1.399 |
| H43 | 0.053 | -4.830 | 1.786 | -0.692 |
| H44 | 0.093 | -3.764 | 0.445 | -0.166 |
| H45 | 0.049 | -3.799 | 4.321 | -0.169 |
| H46 | 0.057 | -2.704 | 4.265 | 1.253 |
| H47 | 0.063 | -4.388 | 3.685 | 1.392 |
| C48 | -0.094 | -6.086 | 0.137 | 1.212 |
| C49 | -0.114 | -5.666 | -1.169 | 1.872 |
| C50 | -0.043 | -4.501 | -1.856 | 1.149 |
| N51 | -0.479 | -3.587 | -2.502 | 2.132 |
| H52 | 0.043 | -1.258 | 1.815 | -3.929 |
| H53 | 0.083 | -2.468 | 0.744 | -3.146 |
| H54 | 0.055 | -3.002 | 2.084 | -4.208 |
| H55 | 0.081 | 0.972 | 2.405 | 1.037 |
| H56 | 0.047 | -0.588 | 2.937 | 1.759 |
| H57 | 0.059 | 0.636 | 4.147 | 1.291 |
| H58 | 0.057 | -5.556 | 2.216 | 1.634 |
| H59 | 0.065 | -4.417 | 1.036 | 2.287 |
| H60 | 0.056 | -7.051 | 0.457 | 1.650 |
| H61 | 0.061 | -6.294 | -0.031 | 0.136 |
| H62 | 0.059 | -5.417 | -0.973 | 2.938 |
| H63 | 0.056 | -6.536 | -1.855 | 1.903 |
| H64 | 0.086 | -3.916 | -1.133 | 0.531 |
| H65 | 0.061 | -4.883 | -2.613 | 0.432 |
| P66 | 1.847 | -1.870 | -2.121 | 2.194 |
| N67 | -0.338 | -0.996 | -3.584 | 2.652 |
| C68 | -0.037 | -4.082 | -3.809 | 2.612 |
| N69 | -0.331 | -1.672 | -1.095 | 3.629 |
| O70 | -0.745 | -1.429 | -1.293 | 0.933 |
| C71 | -0.059 | -1.046 | -4.764 | 1.760 |
| C72 | -0.052 | -2.059 | 0.325 | 3.554 |
| C73 | -0.054 | -1.940 | -1.770 | 4.887 |
| C74 | -0.034 | 0.264 | -3.367 | 3.343 |
| C75 | -0.129 | -3.121 | -1.474 | 5.627 |
| C76 | -0.000 | -1.013 | -2.684 | 5.370 |
| C77 | -0.018 | -1.324 | -3.430 | 6.554 |
| C78 | -0.045 | -2.504 | -3.139 | 7.269 |
| C79 | -0.063 | -3.383 | -2.128 | 6.796 |
| C80 | -0.130 | 1.491 | -3.653 | 2.678 |
| C81 | -0.008 | 0.256 | -2.920 | 4.657 |
| C82 | -0.015 | 1.505 | -2.637 | 5.302 |
| C83 | -0.041 | 2.713 | -2.915 | 4.629 |
| C84 | -0.073 | 2.681 | -3.449 | 3.312 |

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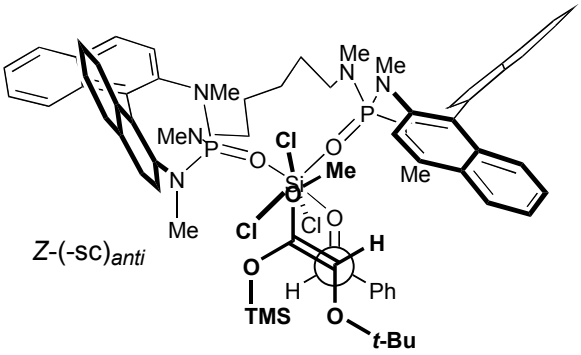
|---|---|---|---|---|
| C85 | -0.091 | 3.987 | -2.084 | 6.512 |
| C86 | -0.093 | 3.955 | -2.637 | 5.260 |
| C87 | -0.088 | -1.987 | -4.871 | 8.878 |
| Si88 | 1.410 | -0.624 | -0.903 | -0.545 |
| Cl89 | -0.508 | 1.151 | -0.098 | 0.633 |
| Cl90 | -0.619 | 0.127 | -3.135 | -0.440 |
| Cl91 | -0.523 | -2.506 | -1.506 | -1.708 |
| O92 | -0.335 | 0.288 | -0.726 | -2.044 |
| H93 | 0.051 | -5.138 | -3.707 | 2.900 |
| H94 | 0.035 | -4.014 | -4.596 | 1.844 |
| H95 | 0.054 | -3.514 | -4.123 | 3.498 |
| H96 | 0.075 | -0.061 | -5.002 | 1.316 |
| H97 | 0.048 | -1.383 | -5.621 | 2.358 |
| H98 | 0.057 | -1.752 | -4.596 | 0.935 |
| H99 | 0.059 | -1.884 | 0.813 | 4.523 |
| H100 | 0.057 | -1.420 | 0.813 | 2.804 |
| H101 | 0.036 | -3.119 | 0.485 | 3.276 |
| H102 | 0.111 | -3.824 | -0.718 | 5.254 |
| H103 | 0.111 | -4.284 | -1.895 | 7.377 |
| H104 | 0.146 | 1.460 | -4.022 | 1.630 |
| H105 | 0.109 | 3.628 | -3.668 | 2.801 |
| C106 | 0.416 | 0.666 | -1.594 | -2.909 |
| C107 | -0.161 | 0.490 | -1.296 | -4.354 |
| C108 | -0.051 | 0.239 | -0.016 | -4.850 |
| C109 | -0.065 | 0.538 | -2.377 | -5.245 |
| C110 | -0.110 | 0.333 | -2.177 | -6.603 |
| C111 | -0.066 | 0.079 | -0.897 | -7.090 |
| C112 | -0.121 | 0.032 | 0.179 | -6.212 |
| C113 | -0.309 | 2.851 | -1.677 | -2.979 |
| C114 | -0.096 | 1.573 | -2.049 | 6.596 |
| C115 | -0.098 | -0.492 | -4.486 | 7.020 |
| C116 | -0.089 | -2.814 | -3.869 | 8.448 |
| C117 | 0.405 | 3.250 | -2.393 | -1.811 |
| H118 | 0.119 | 0.637 | -1.817 | 7.120 |
| C119 | -0.091 | 2.781 | -1.782 | 7.182 |
| O120 | -0.193 | 3.385 | -0.409 | -3.141 |
| H121 | 0.121 | 0.420 | -4.726 | 6.459 |
| C122 | -0.086 | -0.818 | -5.187 | 8.150 |
| H123 | 0.110 | -3.727 | -3.619 | 9.000 |
| H124 | 0.110 | -2.223 | -5.438 | 9.785 |
| H125 | 0.108 | 2.824 | -1.330 | 8.179 |
| H126 | 0.109 | -0.174 | -6.000 | 8.502 |
| H127 | 0.108 | 4.942 | -1.868 | 7.003 |

| | | | | |
|---|---|---|---|---|
| H128 | 0.106 | 4.883 | -2.866 | 4.722 |
| H129 | 0.110 | 0.672 | -2.666 | -2.621 |
| H130 | 0.121 | 0.185 | 0.845 | -4.165 |
| H131 | 0.112 | 0.727 | -3.390 | -4.866 |
| H132 | 0.113 | 0.365 | -3.028 | -7.292 |
| H133 | 0.109 | -0.087 | -0.742 | -8.161 |
| H134 | 0.109 | -0.170 | 1.188 | -6.590 |
| C135 | 0.083 | 4.299 | -0.258 | -4.268 |
| O136 | -0.358 | 3.741 | -2.034 | -0.673 |
| Si137 | 0.745 | 4.879 | -0.856 | -0.143 |
| C138 | -0.159 | 3.502 | 0.114 | -5.520 |
| C139 | -0.154 | 5.219 | 0.906 | -3.886 |
| C140 | -0.159 | 5.141 | -1.506 | -4.549 |
| C141 | -0.277 | 5.046 | -1.352 | 1.670 |
| C142 | -0.305 | 6.502 | -1.172 | -1.058 |
| C143 | -0.312 | 4.322 | 0.929 | -0.272 |
| O144 | -0.210 | 2.966 | -3.711 | -1.813 |
| C145 | 0.057 | 3.056 | -4.487 | -0.631 |
| H146 | 0.129 | 2.805 | -2.279 | -3.911 |
| H147 | 0.058 | 2.783 | -0.673 | -5.794 |
| H148 | 0.059 | 2.926 | 1.040 | -5.367 |
| H149 | 0.052 | 4.168 | 0.273 | -6.377 |
| H150 | 0.058 | 5.822 | 0.676 | -2.996 |
| H151 | 0.061 | 4.644 | 1.830 | -3.672 |
| H152 | 0.054 | 5.909 | 1.135 | -4.708 |
| H153 | 0.055 | 5.773 | -1.773 | -3.687 |
| H154 | 0.053 | 4.498 | -2.371 | -4.776 |
| H155 | 0.061 | 5.802 | -1.344 | -5.409 |
| H156 | 0.062 | 5.707 | -0.653 | 2.193 |
| H157 | 0.078 | 4.073 | -1.336 | 2.175 |
| H158 | 0.052 | 5.462 | -2.358 | 1.781 |
| H159 | 0.062 | 7.093 | -1.945 | -0.558 |
| H160 | 0.065 | 6.335 | -1.500 | -2.094 |
| H161 | 0.065 | 7.106 | -0.260 | -1.094 |
| H162 | 0.099 | 3.259 | 1.042 | 0.007 |
| H163 | 0.068 | 4.430 | 1.339 | -1.288 |
| H164 | 0.062 | 4.914 | 1.552 | 0.406 |
| H165 | 0.076 | 2.358 | -4.139 | 0.150 |
| H166 | 0.031 | 4.080 | -4.524 | -0.242 |
| H167 | 0.055 | 2.753 | -5.471 | -1.000 |




| Atom | Partial Charge | X Coordinate | Y Coordinate | Z Coordinate |
|---|---|---|---|---|
| C1 | -0.084 | -1.605 | 9.295 | -2.933 |
| C2 | -0.084 | -0.343 | 8.658 | -2.900 |
| C3 | -0.098 | -0.255 | 7.299 | -2.760 |
| C4 | -0.020 | -1.427 | 6.502 | -2.640 |
| C5 | -0.039 | -2.683 | 7.146 | -2.647 |
| C6 | -0.089 | -2.751 | 8.556 | -2.809 |
| C7 | -0.070 | -3.869 | 6.383 | -2.478 |
| C8 | -0.104 | -3.799 | 5.039 | -2.247 |
| C9 | -0.057 | -2.535 | 4.384 | -2.214 |
| C10 | -0.011 | -1.366 | 5.079 | -2.480 |
| H11 | 0.112 | -1.652 | 10.382 | -3.056 |
| H12 | 0.111 | 0.563 | 9.266 | -2.988 |
| H13 | 0.120 | 0.724 | 6.803 | -2.740 |
| H14 | 0.112 | -3.735 | 9.040 | -2.829 |
| H15 | 0.114 | -4.839 | 6.893 | -2.522 |
| H16 | 0.118 | -4.715 | 4.453 | -2.098 |
| N17 | -0.393 | -2.573 | 2.966 | -1.872 |
| C18 | -0.027 | -0.070 | 4.378 | -2.558 |
| C19 | -0.051 | 0.423 | 3.669 | -1.472 |
| C20 | -0.022 | 0.670 | 4.376 | -3.786 |
| C21 | -0.138 | 1.684 | 3.010 | -1.543 |
| C22 | -0.072 | 2.413 | 3.045 | -2.696 |
| C23 | -0.044 | 1.918 | 3.720 | -3.844 |
| C24 | -0.087 | 0.903 | 4.970 | -6.128 |
| N25 | -0.353 | -0.359 | 3.612 | -0.245 |
| C26 | -0.087 | 2.162 | 4.330 | -6.173 |
| C27 | -0.102 | 0.175 | 4.992 | -4.969 |
| P28 | 1.829 | -1.738 | 2.514 | -0.387 |
| H29 | 0.153 | 2.049 | 2.430 | -0.665 |
| O30 | -0.761 | -1.325 | 1.007 | -0.390 |
| H31 | 0.110 | 3.379 | 2.524 | -2.757 |
| N32 | -0.407 | -2.867 | 2.813 | 0.929 |

| | | | | |
|---|---|---|---|---|
| C33 | -0.092 | 2.658 | 3.714 | -5.056 |
| H34 | 0.112 | 0.517 | 5.451 | -7.034 |
| H35 | 0.112 | 2.731 | 4.331 | -7.110 |
| H36 | 0.123 | -0.799 | 5.496 | -4.935 |
| H37 | 0.111 | 3.626 | 3.196 | -5.082 |
| C38 | -0.058 | -4.042 | 1.895 | 0.945 |
| C39 | -0.068 | -3.219 | 4.217 | 1.229 |
| C40 | -0.069 | -2.599 | 2.040 | -3.021 |
| C41 | -0.066 | 0.427 | 3.612 | 1.006 |
| C42 | -0.120 | -4.594 | 1.739 | 2.361 |
| H43 | 0.053 | -4.843 | 2.260 | 0.265 |
| H44 | 0.084 | -3.710 | 0.904 | 0.553 |
| H45 | 0.048 | -3.685 | 4.741 | 0.378 |
| H46 | 0.058 | -2.315 | 4.758 | 1.538 |
| H47 | 0.063 | -3.934 | 4.223 | 2.069 |
| C48 | -0.094 | -5.646 | 0.636 | 2.432 |
| C49 | -0.117 | -5.092 | -0.683 | 2.953 |
| C50 | -0.042 | -4.190 | -1.386 | 1.933 |
| N51 | -0.471 | -3.200 | -2.262 | 2.619 |
| H52 | 0.046 | -1.656 | 2.054 | -3.600 |
| H53 | 0.083 | -2.777 | 1.015 | -2.655 |
| H54 | 0.057 | -3.422 | 2.326 | -3.690 |
| H55 | 0.081 | 1.285 | 2.912 | 0.969 |
| H56 | 0.048 | -0.221 | 3.334 | 1.848 |
| H57 | 0.057 | 0.809 | 4.627 | 1.173 |
| H58 | 0.058 | -5.019 | 2.713 | 2.690 |
| H59 | 0.067 | -3.767 | 1.529 | 3.080 |
| H60 | 0.058 | -6.464 | 0.962 | 3.104 |
| H61 | 0.059 | -6.122 | 0.484 | 1.442 |
| H62 | 0.059 | -4.552 | -0.502 | 3.908 |
| H63 | 0.059 | -5.934 | -1.352 | 3.224 |
| H64 | 0.075 | -3.646 | -0.645 | 1.302 |
| H65 | 0.063 | -4.804 | -1.982 | 1.224 |
| P66 | 1.839 | -1.475 | -1.915 | 2.488 |
| N67 | -0.322 | -0.575 | -3.408 | 2.762 |
| C68 | -0.034 | -3.693 | -3.636 | 2.841 |
| N69 | -0.329 | -1.074 | -0.937 | 3.907 |
| O70 | -0.752 | -1.211 | -1.068 | 1.190 |
| C71 | -0.076 | -0.676 | -4.480 | 1.746 |
| C72 | -0.054 | -1.301 | 0.517 | 3.896 |
| C73 | -0.060 | -1.301 | -1.633 | 5.161 |
| C74 | -0.035 | 0.725 | -3.257 | 3.399 |
| C75 | -0.126 | -2.398 | -1.284 | 5.999 |

| | | | | |
|---|---|---|---|---|
| C76 | 0.003 | -0.414 | -2.634 | 5.533 |
| C77 | -0.019 | -0.707 | -3.425 | 6.691 |
| C78 | -0.044 | -1.815 | -3.092 | 7.497 |
| C79 | -0.065 | -2.636 | -1.986 | 7.146 |
| C80 | -0.139 | 1.908 | -3.528 | 2.654 |
| C81 | -0.014 | 0.800 | -2.891 | 4.736 |
| C82 | -0.016 | 2.089 | -2.693 | 5.332 |
| C83 | -0.042 | 3.252 | -2.962 | 4.581 |
| C84 | -0.057 | 3.136 | -3.399 | 3.234 |
| C85 | -0.093 | 4.646 | -2.309 | 6.449 |
| C86 | -0.087 | 4.533 | -2.773 | 5.167 |
| C87 | -0.087 | -1.340 | -4.958 | 8.962 |
| Si88 | 1.404 | -0.581 | -0.546 | -0.319 |
| Cl89 | -0.518 | 1.178 | 0.427 | 0.746 |
| Cl90 | -0.596 | 0.505 | -2.633 | -0.365 |
| Cl91 | -0.514 | -2.446 | -1.349 | -1.364 |
| O92 | -0.331 | 0.144 | -0.229 | -1.905 |
| H93 | 0.051 | -4.692 | -3.585 | 3.297 |
| H94 | 0.033 | -3.774 | -4.213 | 1.906 |
| H95 | 0.055 | -3.027 | -4.169 | 3.533 |
| H96 | 0.090 | 0.266 | -4.638 | 1.173 |
| H97 | 0.051 | -0.927 | -5.412 | 2.271 |
| H98 | 0.051 | -1.471 | -4.247 | 1.024 |
| H99 | 0.060 | -0.943 | 0.954 | 4.837 |
| H100 | 0.064 | -0.715 | 0.946 | 3.068 |
| H101 | 0.032 | -2.364 | 0.803 | 3.768 |
| H102 | 0.110 | -3.054 | -0.449 | 5.721 |
| H103 | 0.111 | -3.472 | -1.716 | 7.803 |
| H104 | 0.155 | 1.820 | -3.833 | 1.588 |
| H105 | 0.111 | 4.046 | -3.610 | 2.660 |
| C106 | 0.419 | 0.427 | -1.036 | -2.848 |
| C107 | -0.168 | 0.244 | -0.628 | -4.258 |
| C108 | -0.060 | 0.052 | 0.693 | -4.666 |
| C109 | -0.038 | 0.249 | -1.648 | -5.221 |
| C110 | -0.117 | 0.064 | -1.345 | -6.563 |
| C111 | -0.057 | -0.124 | -0.025 | -6.962 |
| C112 | -0.127 | -0.128 | 0.992 | -6.012 |
| C113 | -0.412 | 2.729 | -1.070 | -2.897 |
| C114 | -0.101 | 2.242 | -2.197 | 6.657 |
| C115 | -0.095 | 0.070 | -4.564 | 7.041 |
| C116 | -0.090 | -2.111 | -3.873 | 8.646 |
| C117 | 0.408 | 3.214 | -1.658 | -1.708 |
| H118 | 0.118 | 1.344 | -1.968 | 7.244 |

| | | | | |
|---|---|---|---|---|
| C119 | -0.090 | 3.486 | -2.011 | 7.198 |
| O120 | -0.190 | 2.866 | -1.818 | -4.056 |
| H121 | 0.121 | 0.928 | -4.830 | 6.410 |
| C122 | -0.087 | -0.241 | -5.310 | 8.145 |
| H123 | 0.110 | -2.968 | -3.593 | 9.270 |
| H124 | 0.110 | -1.566 | -5.565 | 9.846 |
| H125 | 0.107 | 3.594 | -1.628 | 8.219 |
| H126 | 0.110 | 0.359 | -6.188 | 8.408 |
| H127 | 0.108 | 5.631 | -2.161 | 6.906 |
| H128 | 0.108 | 5.425 | -3.001 | 4.572 |
| H129 | 0.131 | 0.480 | -2.131 | -2.641 |
| H130 | 0.117 | 0.019 | 1.508 | -3.925 |
| H131 | 0.126 | 0.441 | -2.692 | -4.910 |
| H132 | 0.113 | 0.073 | -2.147 | -7.310 |
| H133 | 0.109 | -0.268 | 0.214 | -8.021 |
| H134 | 0.112 | -0.269 | 2.034 | -6.321 |
| C135 | 0.079 | 3.960 | -1.416 | -4.922 |
| O136 | -0.324 | 3.332 | -2.897 | -1.348 |
| Si137 | 0.730 | 2.795 | -4.396 | -2.003 |
| C138 | -0.148 | 3.849 | 0.057 | -5.326 |
| C139 | -0.156 | 5.317 | -1.682 | -4.266 |
| C140 | -0.124 | 3.792 | -2.313 | -6.154 |
| C141 | -0.281 | 2.482 | -5.402 | -0.433 |
| C142 | -0.296 | 1.256 | -4.416 | -3.096 |
| C143 | -0.297 | 4.287 | -5.059 | -2.957 |
| O144 | -0.171 | 3.655 | -1.027 | -0.595 |
| C145 | 0.030 | 4.006 | 0.339 | -0.696 |
| H146 | 0.125 | 2.829 | 0.022 | -3.034 |
| H147 | 0.057 | 2.945 | 0.244 | -5.921 |
| H148 | 0.042 | 3.792 | 0.710 | -4.437 |
| H149 | 0.054 | 4.716 | 0.370 | -5.921 |
| H150 | 0.064 | 5.404 | -2.733 | -3.942 |
| H151 | 0.041 | 5.469 | -1.047 | -3.382 |
| H152 | 0.056 | 6.137 | -1.480 | -4.966 |
| H153 | 0.051 | 3.922 | -3.374 | -5.899 |
| H154 | 0.052 | 2.794 | -2.199 | -6.602 |
| H155 | 0.051 | 4.535 | -2.058 | -6.920 |
| H156 | 0.058 | 3.395 | -5.520 | 0.157 |
| H157 | 0.080 | 1.729 | -4.906 | 0.207 |
| H158 | 0.058 | 2.109 | -6.399 | -0.685 |
| H159 | 0.082 | 0.427 | -3.888 | -2.600 |
| H160 | 0.066 | 1.408 | -3.944 | -4.077 |
| H161 | 0.063 | 0.946 | -5.451 | -3.266 |

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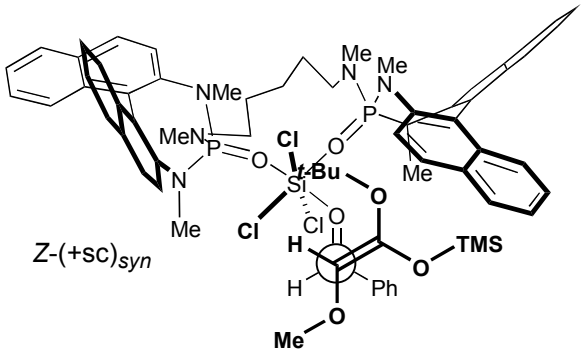
|---|---|---|---|---|
| H162 | 0.060 | 5.077 | -5.400 | -2.282 |
| H163 | 0.066 | 4.717 | -4.285 | -3.610 |
| H164 | 0.061 | 3.997 | -5.904 | -3.588 |
| H165 | 0.062 | 3.115 | 0.990 | -0.738 |
| H166 | 0.023 | 4.659 | 0.539 | -1.555 |
| H167 | 0.062 | 4.542 | 0.508 | 0.241 |

**Atom Coordinates of Calculated Transition Structures (Figures 4)**



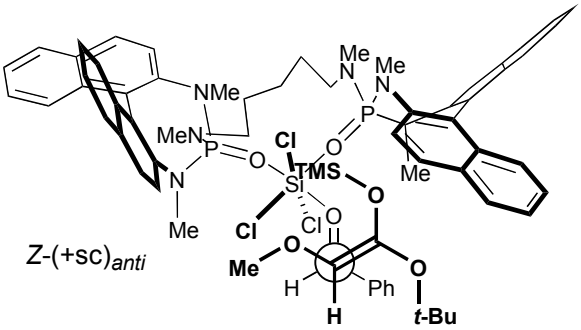

| Atom | Partial Charge | X Coordinate | Y Coordinate | Z Coordinate |
|---|---|---|---|---|
| C1 | -0.085 | -6.775 | 6.370 | -2.839 |
| C2 | -0.085 | -5.427 | 6.684 | -2.556 |
| C3 | -0.098 | -4.490 | 5.691 | -2.453 |
| C4 | -0.021 | -4.855 | 4.327 | -2.622 |
| C5 | -0.040 | -6.207 | 4.017 | -2.879 |
| C6 | -0.089 | -7.159 | 5.066 | -2.998 |
| C7 | -0.071 | -6.609 | 2.660 | -3.006 |
| C8 | -0.104 | -5.708 | 1.652 | -2.812 |
| C9 | -0.053 | -4.347 | 1.955 | -2.522 |
| C10 | -0.012 | -3.900 | 3.266 | -2.507 |
| H11 | 0.112 | -7.507 | 7.181 | -2.927 |
| H12 | 0.111 | -5.140 | 7.733 | -2.421 |
| H13 | 0.120 | -3.441 | 5.933 | -2.238 |
| H14 | 0.112 | -8.203 | 4.810 | -3.214 |
| H15 | 0.114 | -7.656 | 2.437 | -3.246 |
| H16 | 0.118 | -6.023 | 0.603 | -2.886 |
| N17 | -0.403 | -3.495 | 0.810 | -2.224 |
| C18 | -0.021 | -2.463 | 3.561 | -2.351 |
| C19 | -0.058 | -1.768 | 3.148 | -1.224 |
| C20 | -0.020 | -1.748 | 4.202 | -3.415 |
| C21 | -0.123 | -0.369 | 3.385 | -1.098 |
| C22 | -0.084 | 0.315 | 3.997 | -2.108 |
| C23 | -0.044 | -0.360 | 4.419 | -3.286 |

| | | | | |
|---|---|---|---|---|
| C24 | -0.090 | -1.672 | 5.187 | -5.634 |
| N25 | -0.345 | -2.479 | 2.453 | -0.160 |
| C26 | -0.089 | -0.283 | 5.407 | -5.496 |
| C27 | -0.097 | -2.384 | 4.596 | -4.626 |
| P28 | 1.824 | -2.816 | 0.769 | -0.597 |
| H29 | 0.151 | 0.154 | 3.021 | -0.191 |
| O30 | -0.764 | -1.519 | -0.088 | -0.494 |
| H31 | 0.123 | 1.407 | 4.139 | -2.040 |
| N32 | -0.397 | -4.083 | 0.115 | 0.437 |
| C33 | -0.111 | 0.360 | 5.027 | -4.350 |
| H34 | 0.112 | -2.172 | 5.494 | -6.560 |
| H35 | 0.110 | 0.269 | 5.881 | -6.315 |
| H36 | 0.124 | -3.463 | 4.425 | -4.737 |
| H37 | 0.120 | 1.449 | 5.169 | -4.239 |
| C38 | -0.072 | -4.418 | -1.298 | 0.092 |
| C39 | -0.071 | -5.251 | 0.990 | 0.664 |
| C40 | -0.054 | -2.786 | 0.213 | -3.372 |
| C41 | -0.075 | -1.969 | 2.709 | 1.204 |
| C42 | -0.105 | -5.252 | -1.987 | 1.171 |
| H43 | 0.052 | -4.957 | -1.352 | -0.879 |
| H44 | 0.098 | -3.446 | -1.839 | -0.043 |
| H45 | 0.048 | -5.893 | 1.098 | -0.226 |
| H46 | 0.058 | -4.906 | 1.983 | 0.982 |
| H47 | 0.061 | -5.861 | 0.569 | 1.476 |
| C48 | -0.096 | -5.167 | -3.506 | 1.052 |
| C49 | -0.108 | -4.175 | -4.121 | 2.029 |
| C50 | -0.049 | -2.716 | -3.918 | 1.606 |
| N51 | -0.449 | -1.856 | -3.591 | 2.780 |
| H52 | 0.044 | -1.988 | 0.859 | -3.773 |
| H53 | 0.075 | -2.344 | -0.745 | -3.051 |
| H54 | 0.055 | -3.509 | 0.018 | -4.174 |
| H55 | 0.083 | -0.876 | 2.540 | 1.291 |
| H56 | 0.045 | -2.492 | 2.055 | 1.914 |
| H57 | 0.060 | -2.183 | 3.754 | 1.464 |
| H58 | 0.052 | -6.311 | -1.671 | 1.085 |
| H59 | 0.064 | -4.934 | -1.665 | 2.184 |
| H60 | 0.055 | -6.168 | -3.937 | 1.255 |
| H61 | 0.064 | -4.925 | -3.810 | 0.013 |
| H62 | 0.064 | -4.367 | -3.707 | 3.042 |
| H63 | 0.054 | -4.383 | -5.207 | 2.117 |
| H64 | 0.093 | -2.629 | -3.091 | 0.859 |
| H65 | 0.052 | -2.335 | -4.829 | 1.095 |
| P66 | 1.833 | -0.437 | -2.558 | 2.588 |

| | | | | |
|---|---|---|---|---|
| N67 | -0.350 | 0.984 | -3.552 | 2.924 |
| C68 | -0.063 | -1.716 | -4.718 | 3.728 |
| N69 | -0.330 | -0.423 | -1.399 | 3.921 |
| O70 | -0.746 | -0.538 | -1.782 | 1.226 |
| C71 | -0.065 | 1.266 | -4.667 | 1.994 |
| C72 | -0.054 | -1.178 | -0.138 | 3.815 |
| C73 | -0.061 | -0.312 | -2.000 | 5.236 |
| C74 | -0.025 | 2.145 | -2.876 | 3.479 |
| C75 | -0.128 | -1.427 | -2.010 | 6.123 |
| C76 | 0.003 | 0.906 | -2.541 | 5.625 |
| C77 | -0.021 | 0.979 | -3.271 | 6.855 |
| C78 | -0.045 | -0.143 | -3.307 | 7.710 |
| C79 | -0.066 | -1.336 | -2.631 | 7.335 |
| C80 | -0.128 | 3.327 | -2.754 | 2.693 |
| C81 | -0.016 | 2.105 | -2.388 | 4.778 |
| C82 | -0.013 | 3.237 | -1.668 | 5.285 |
| C83 | -0.043 | 4.402 | -1.558 | 4.497 |
| C84 | -0.070 | 4.429 | -2.129 | 3.196 |
| C85 | -0.092 | 5.488 | -0.263 | 6.230 |
| C86 | -0.091 | 5.532 | -0.857 | 4.998 |
| C87 | -0.087 | 1.061 | -4.707 | 9.273 |
| Si88 | 1.407 | -0.110 | -1.055 | -0.270 |
| Cl89 | -0.513 | 0.808 | 0.706 | 0.882 |
| Cl90 | -0.596 | 1.735 | -2.514 | -0.239 |
| Cl91 | -0.545 | -1.285 | -2.673 | -1.417 |
| O92 | -0.341 | 0.619 | -0.369 | -1.715 |
| H93 | 0.057 | -2.714 | -5.086 | 4.007 |
| H94 | 0.040 | -1.150 | -5.565 | 3.308 |
| H95 | 0.060 | -1.216 | -4.367 | 4.643 |
| H96 | 0.086 | 2.108 | -4.453 | 1.308 |
| H97 | 0.051 | 1.514 | -5.554 | 2.591 |
| H98 | 0.045 | 0.374 | -4.882 | 1.389 |
| H99 | 0.059 | -0.964 | 0.491 | 4.690 |
| H100 | 0.077 | -0.830 | 0.394 | 2.912 |
| H101 | 0.024 | -2.268 | -0.281 | 3.756 |
| H102 | 0.113 | -2.359 | -1.511 | 5.831 |
| H103 | 0.112 | -2.186 | -2.631 | 8.029 |
| H104 | 0.147 | 3.321 | -3.154 | 1.661 |
| H105 | 0.107 | 5.343 | -2.041 | 2.595 |
| C106 | 0.421 | 0.986 | -0.998 | -2.776 |
| C107 | -0.172 | 0.736 | -0.345 | -4.084 |
| C108 | -0.064 | 0.571 | 1.037 | -4.220 |
| C109 | -0.030 | 0.633 | -1.163 | -5.216 |

| | | | | |
|---|---|---|---|---|
| C110 | -0.115 | 0.368 | -0.605 | -6.460 |
| C111 | -0.064 | 0.209 | 0.771 | -6.590 |
| C112 | -0.136 | 0.311 | 1.589 | -5.469 |
| C113 | -0.323 | 3.157 | -1.023 | -2.778 |
| C114 | -0.098 | 3.219 | -1.024 | 6.553 |
| C115 | -0.095 | 2.144 | -3.994 | 7.235 |
| C116 | -0.090 | -0.077 | -4.028 | 8.932 |
| C117 | 0.398 | 3.734 | 0.263 | -2.918 |
| H118 | 0.118 | 2.310 | -1.086 | 7.165 |
| C119 | -0.090 | 4.313 | -0.341 | 7.011 |
| O120 | -0.194 | 3.276 | -1.876 | -3.878 |
| H121 | 0.122 | 3.015 | -3.976 | 6.568 |
| C122 | -0.087 | 2.181 | -4.694 | 8.411 |
| H123 | 0.111 | -0.953 | -4.034 | 9.591 |
| H124 | 0.111 | 1.116 | -5.267 | 10.213 |
| H125 | 0.108 | 4.290 | 0.152 | 7.989 |
| H126 | 0.110 | 3.080 | -5.252 | 8.695 |
| H127 | 0.108 | 6.356 | 0.279 | 6.621 |
| H128 | 0.106 | 6.434 | -0.799 | 4.379 |
| H129 | 0.116 | 1.010 | -2.109 | -2.734 |
| H130 | 0.118 | 0.651 | 1.697 | -3.343 |
| H131 | 0.118 | 0.772 | -2.248 | -5.119 |
| H132 | 0.112 | 0.290 | -1.251 | -7.342 |
| H133 | 0.106 | 0.005 | 1.210 | -7.572 |
| H134 | 0.117 | 0.187 | 2.677 | -5.562 |
| C135 | 0.039 | 4.260 | -2.872 | -3.712 |
| O136 | -0.331 | 3.857 | 0.898 | -4.043 |
| Si137 | 0.718 | 4.539 | 2.464 | -4.337 |
| C141 | -0.270 | 4.308 | 2.568 | -6.205 |
| C142 | -0.304 | 3.606 | 3.863 | -3.476 |
| C143 | -0.295 | 6.364 | 2.504 | -3.858 |
| O144 | -0.252 | 4.121 | 1.136 | -1.966 |
| C145 | 0.136 | 4.930 | 0.697 | -0.829 |
| H146 | 0.170 | 3.231 | -1.494 | -1.769 |
| H156 | 0.066 | 4.785 | 1.728 | -6.718 |
| H157 | 0.062 | 3.246 | 2.564 | -6.473 |
| H158 | 0.062 | 4.750 | 3.493 | -6.591 |
| H159 | 0.065 | 4.303 | 4.523 | -2.953 |
| H160 | 0.063 | 3.041 | 4.472 | -4.196 |
| H161 | 0.069 | 2.881 | 3.494 | -2.734 |
| H162 | 0.062 | 6.878 | 1.581 | -4.139 |
| H163 | 0.067 | 6.488 | 2.643 | -2.773 |
| H164 | 0.068 | 6.869 | 3.334 | -4.361 |

| | | | | |
|---|---|---|---|---|
| C165 | -0.167 | 6.071 | -0.217 | -1.282 |
| C166 | -0.216 | 4.074 | 0.011 | 0.223 |
| C167 | -0.137 | 5.502 | 2.016 | -0.297 |
| H168 | 0.058 | 6.689 | 0.254 | -2.061 |
| H169 | 0.064 | 6.728 | -0.451 | -0.434 |
| H170 | 0.057 | 5.700 | -1.173 | -1.682 |
| H171 | 0.102 | 3.235 | 0.646 | 0.555 |
| H172 | 0.061 | 4.673 | -0.237 | 1.110 |
| H173 | 0.091 | 3.633 | -0.932 | -0.152 |
| H174 | 0.058 | 6.086 | 2.543 | -1.072 |
| H175 | 0.059 | 6.167 | 1.825 | 0.555 |
| H176 | 0.060 | 4.709 | 2.695 | 0.046 |
| H177 | 0.049 | 4.182 | -3.447 | -4.638 |
| H178 | 0.034 | 4.057 | -3.516 | -2.848 |
| H179 | 0.014 | 5.266 | -2.445 | -3.616 |




*Z*-(+sc)$_{anti}$

| Atom | Partial Charge | X Coordinate | Y Coordinate | Z Coordinate |
|---|---|---|---|---|
| C1 | -0.085 | -3.757 | 8.147 | -2.807 |
| C2 | -0.086 | -2.468 | 7.967 | -2.256 |
| C3 | -0.096 | -1.916 | 6.717 | -2.172 |
| C4 | -0.020 | -2.629 | 5.576 | -2.632 |
| C5 | -0.040 | -3.925 | 5.758 | -3.159 |
| C6 | -0.089 | -4.472 | 7.066 | -3.249 |
| C7 | -0.072 | -4.679 | 4.631 | -3.585 |
| C8 | -0.106 | -4.185 | 3.369 | -3.423 |
| C9 | -0.051 | -2.890 | 3.173 | -2.862 |
| C10 | -0.011 | -2.083 | 4.253 | -2.547 |
| H11 | 0.111 | -4.175 | 9.157 | -2.872 |
| H12 | 0.110 | -1.915 | 8.842 | -1.896 |
| H13 | 0.120 | -0.913 | 6.577 | -1.750 |
| H14 | 0.112 | -5.474 | 7.193 | -3.674 |
| H15 | 0.114 | -5.668 | 4.793 | -4.031 |
| H16 | 0.118 | -4.776 | 2.498 | -3.733 |
| N17 | -0.403 | -2.504 | 1.786 | -2.634 |

| | | | | |
|---|---|---|---|---|
| C18 | -0.033 | -0.687 | 4.057 | -2.110 |
| C19 | -0.046 | -0.383 | 3.336 | -0.964 |
| C20 | -0.019 | 0.381 | 4.569 | -2.917 |
| C21 | -0.127 | 0.968 | 3.198 | -0.534 |
| C22 | -0.083 | 1.983 | 3.740 | -1.264 |
| C23 | -0.042 | 1.715 | 4.408 | -2.487 |
| C24 | -0.089 | 1.194 | 5.670 | -4.924 |
| N25 | -0.343 | -1.449 | 2.732 | -0.180 |
| C26 | -0.089 | 2.527 | 5.504 | -4.486 |
| C27 | -0.099 | 0.150 | 5.212 | -4.166 |
| P28 | 1.842 | -2.168 | 1.320 | -0.970 |
| H29 | 0.150 | 1.190 | 2.602 | 0.375 |
| O30 | -0.753 | -1.229 | 0.080 | -0.876 |
| H31 | 0.124 | 3.025 | 3.627 | -0.916 |
| N32 | -0.418 | -3.726 | 1.027 | -0.209 |
| C33 | -0.102 | 2.784 | 4.881 | -3.295 |
| H34 | 0.110 | 1.005 | 6.169 | -5.881 |
| H35 | 0.108 | 3.346 | 5.874 | -5.112 |
| H36 | 0.121 | -0.884 | 5.342 | -4.510 |
| H37 | 0.114 | 3.816 | 4.711 | -2.950 |
| C38 | -0.070 | -4.400 | -0.256 | -0.563 |
| C39 | -0.074 | -4.657 | 2.173 | -0.120 |
| C40 | -0.045 | -1.852 | 1.107 | -3.767 |
| C41 | -0.083 | -1.168 | 2.588 | 1.265 |
| C42 | -0.119 | -5.395 | -0.655 | 0.528 |
| H43 | 0.051 | -4.924 | -0.176 | -1.539 |
| H44 | 0.105 | -3.615 | -1.046 | -0.680 |
| H45 | 0.049 | -4.925 | 2.597 | -1.102 |
| H46 | 0.057 | -4.204 | 2.957 | 0.501 |
| H47 | 0.064 | -5.588 | 1.835 | 0.368 |
| C48 | -0.094 | -5.923 | -2.070 | 0.319 |
| C49 | -0.111 | -5.256 | -3.086 | 1.237 |
| C50 | -0.054 | -3.794 | -3.351 | 0.856 |
| N51 | -0.473 | -2.942 | -3.477 | 2.071 |
| H52 | 0.040 | -0.783 | 1.367 | -3.881 |
| H53 | 0.070 | -1.927 | 0.019 | -3.613 |
| H54 | 0.054 | -2.373 | 1.369 | -4.697 |
| H55 | 0.081 | -0.175 | 2.135 | 1.467 |
| H56 | 0.054 | -1.942 | 1.955 | 1.734 |
| H57 | 0.059 | -1.199 | 3.583 | 1.728 |
| H58 | 0.055 | -6.228 | 0.083 | 0.542 |
| H59 | 0.063 | -4.921 | -0.566 | 1.534 |
| H60 | 0.055 | -7.013 | -2.083 | 0.514 |

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|---|---|---|---|---|
| H61 | 0.063 | -5.810 | -2.381 | -0.739 |
| H62 | 0.058 | -5.343 | -2.740 | 2.289 |
| H63 | 0.055 | -5.825 | -4.037 | 1.200 |
| H64 | 0.094 | -3.391 | -2.528 | 0.216 |
| H65 | 0.063 | -3.715 | -4.271 | 0.239 |
| P66 | 1.837 | -1.478 | -2.522 | 2.276 |
| N67 | -0.338 | -0.239 | -3.507 | 3.056 |
| C68 | -0.040 | -3.063 | -4.794 | 2.730 |
| N69 | -0.329 | -1.875 | -1.351 | 3.556 |
| O70 | -0.746 | -1.137 | -1.698 | 0.975 |
| C71 | -0.059 | 0.233 | -4.761 | 2.426 |
| C72 | -0.061 | -2.780 | -0.230 | 3.245 |
| C73 | -0.054 | -2.014 | -1.927 | 4.883 |
| C74 | -0.042 | 0.779 | -2.744 | 3.760 |
| C75 | -0.131 | -3.301 | -2.043 | 5.484 |
| C76 | 0.004 | -0.876 | -2.332 | 5.568 |
| C77 | -0.017 | -1.021 | -2.984 | 6.836 |
| C78 | -0.045 | -2.302 | -3.104 | 7.414 |
| C79 | -0.064 | -3.437 | -2.599 | 6.723 |
| C80 | -0.118 | 2.091 | -2.645 | 3.215 |
| C81 | -0.010 | 0.463 | -2.155 | 4.975 |
| C82 | -0.017 | 1.445 | -1.338 | 5.625 |
| C83 | -0.040 | 2.739 | -1.238 | 5.072 |
| C84 | -0.064 | 3.049 | -1.924 | 3.866 |
| C85 | -0.093 | 3.409 | 0.258 | 6.853 |
| C86 | -0.090 | 3.719 | -0.436 | 5.716 |
| C87 | -0.088 | -1.366 | -4.270 | 9.316 |
| Si88 | 1.396 | -0.291 | -1.315 | -0.486 |
| Cl89 | -0.517 | 1.150 | 0.018 | 0.696 |
| Cl90 | -0.589 | 0.872 | -3.298 | -0.054 |
| Cl91 | -0.547 | -1.815 | -2.508 | -1.738 |
| O92 | -0.351 | 0.704 | -1.058 | -1.917 |
| H93 | 0.051 | -4.129 | -5.014 | 2.887 |
| H94 | 0.037 | -2.633 | -5.611 | 2.130 |
| H95 | 0.054 | -2.570 | -4.771 | 3.711 |
| H96 | 0.079 | 1.276 | -4.683 | 2.064 |
| H97 | 0.049 | 0.177 | -5.554 | 3.184 |
| H98 | 0.058 | -0.401 | -5.029 | 1.571 |
| H99 | 0.059 | -3.009 | 0.340 | 4.156 |
| H100 | 0.055 | -2.266 | 0.452 | 2.545 |
| H101 | 0.041 | -3.740 | -0.544 | 2.789 |
| H102 | 0.112 | -4.189 | -1.685 | 4.948 |
| H103 | 0.111 | -4.424 | -2.680 | 7.194 |

| | | | | |
|---|---|---|---|---|
| H104 | 0.143 | 2.302 | -3.152 | 2.254 |
| H105 | 0.105 | 4.062 | -1.847 | 3.455 |
| C106 | 0.387 | 1.170 | -1.954 | -2.727 |
| C107 | -0.168 | 1.001 | -1.740 | -4.192 |
| C108 | -0.071 | 0.773 | -0.489 | -4.766 |
| C109 | -0.062 | 1.036 | -2.875 | -5.014 |
| C110 | -0.109 | 0.845 | -2.754 | -6.384 |
| C111 | -0.074 | 0.631 | -1.501 | -6.952 |
| C112 | -0.121 | 0.598 | -0.371 | -6.141 |
| C113 | -0.246 | 3.258 | -1.913 | -2.705 |
| C114 | -0.097 | 1.150 | -0.594 | 6.801 |
| C115 | -0.097 | 0.087 | -3.549 | 7.526 |
| C116 | -0.089 | -2.453 | -3.745 | 8.672 |
| C117 | 0.404 | 3.776 | -0.585 | -2.738 |
| H118 | 0.118 | 0.141 | -0.659 | 7.226 |
| C119 | -0.093 | 2.106 | 0.183 | 7.396 |
| O120 | -0.196 | 3.636 | -2.808 | -1.713 |
| H121 | 0.121 | 1.085 | -3.471 | 7.075 |
| C122 | -0.087 | -0.083 | -4.176 | 8.731 |
| H123 | 0.110 | -3.455 | -3.817 | 9.113 |
| H124 | 0.110 | -1.477 | -4.768 | 10.285 |
| H125 | 0.108 | 1.872 | 0.754 | 8.301 |
| H126 | 0.109 | 0.775 | -4.611 | 9.255 |
| H127 | 0.107 | 4.161 | 0.878 | 7.353 |
| H128 | 0.106 | 4.725 | -0.382 | 5.283 |
| H129 | 0.122 | 1.173 | -3.011 | -2.378 |
| H130 | 0.121 | 0.701 | 0.409 | -4.131 |
| H131 | 0.118 | 1.200 | -3.866 | -4.570 |
| H132 | 0.114 | 0.859 | -3.647 | -7.018 |
| H133 | 0.110 | 0.481 | -1.406 | -8.033 |
| H134 | 0.108 | 0.421 | 0.615 | -6.583 |
| C135 | 0.000 | 3.737 | -2.348 | -0.386 |
| O136 | -0.348 | 3.786 | 0.232 | -1.733 |
| Si137 | 0.742 | 5.081 | 1.213 | -1.144 |
| C141 | -0.313 | 5.574 | 2.603 | -2.314 |
| C142 | -0.293 | 4.337 | 1.851 | 0.460 |
| C143 | -0.316 | 6.522 | 0.025 | -0.874 |
| O144 | -0.250 | 4.109 | 0.167 | -3.807 |
| C145 | 0.127 | 4.931 | -0.379 | -4.882 |
| H146 | 0.127 | 3.389 | -2.501 | -3.639 |
| H156 | 0.070 | 5.854 | 2.224 | -3.308 |
| H157 | 0.069 | 4.766 | 3.336 | -2.458 |
| H158 | 0.068 | 6.438 | 3.141 | -1.909 |

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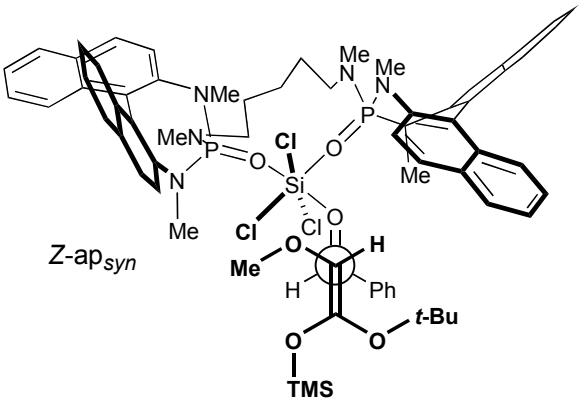
|---|---|---|---|---|
| H159 | 0.061 | 3.981 | 2.884 | 0.344 |
| H160 | 0.105 | 3.471 | 1.237 | 0.753 |
| H161 | 0.065 | 5.063 | 1.827 | 1.277 |
| H162 | 0.062 | 6.979 | -0.270 | -1.829 |
| H163 | 0.076 | 6.175 | -0.891 | -0.372 |
| H164 | 0.071 | 7.297 | 0.480 | -0.252 |
| C165 | -0.172 | 4.117 | -1.265 | -5.818 |
| C166 | -0.177 | 6.133 | -1.143 | -4.321 |
| C167 | -0.136 | 5.402 | 0.883 | -5.614 |
| H168 | 0.072 | 3.226 | -0.745 | -6.205 |
| H169 | 0.067 | 4.726 | -1.573 | -6.678 |
| H170 | 0.062 | 3.763 | -2.180 | -5.312 |
| H171 | 0.067 | 6.645 | -0.582 | -3.518 |
| H172 | 0.070 | 6.868 | -1.335 | -5.113 |
| H173 | 0.062 | 5.840 | -2.117 | -3.903 |
| H174 | 0.062 | 5.908 | 1.583 | -4.925 |
| H175 | 0.059 | 4.565 | 1.422 | -6.076 |
| H176 | 0.062 | 6.110 | 0.623 | -6.411 |
| H177 | 0.059 | 3.671 | -3.273 | 0.195 |
| H178 | 0.088 | 2.927 | -1.664 | -0.082 |
| H179 | 0.013 | 4.718 | -1.867 | -0.233 |




*Z*-ap$_{syn}$

| Atom | Partial Charge | X Coordinate | Y Coordinate | Z Coordinate |
|---|---|---|---|---|
| C1 | -0.085 | -0.537 | 9.439 | -2.389 |
| C2 | -0.086 | 0.651 | 8.680 | -2.494 |
| C3 | -0.097 | 0.606 | 7.312 | -2.466 |
| C4 | -0.022 | -0.633 | 6.629 | -2.324 |
| C5 | -0.040 | -1.813 | 7.392 | -2.191 |
| C6 | -0.090 | -1.744 | 8.810 | -2.241 |
| C7 | -0.074 | -3.060 | 6.737 | -1.996 |
| C8 | -0.101 | -3.113 | 5.379 | -1.863 |

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|---|---|---|---|---|
| C9 | -0.061 | -1.923 | 4.604 | -1.965 |
| C10 | -0.012 | -0.712 | 5.199 | -2.277 |
| H11 | 0.112 | -0.478 | 10.532 | -2.424 |
| H12 | 0.111 | 1.609 | 9.201 | -2.599 |
| H13 | 0.120 | 1.526 | 6.721 | -2.553 |
| H14 | 0.112 | -2.671 | 9.388 | -2.154 |
| H15 | 0.114 | -3.973 | 7.342 | -1.938 |
| H16 | 0.118 | -4.072 | 4.874 | -1.685 |
| N17 | -0.392 | -2.070 | 3.178 | -1.696 |
| C18 | -0.032 | 0.483 | 4.374 | -2.538 |
| C19 | -0.039 | 0.991 | 3.512 | -1.576 |
| C20 | -0.018 | 1.092 | 4.414 | -3.835 |
| C21 | -0.120 | 2.139 | 2.714 | -1.852 |
| C22 | -0.085 | 2.736 | 2.765 | -3.078 |
| C23 | -0.048 | 2.231 | 3.623 | -4.092 |
| C24 | -0.088 | 1.183 | 5.237 | -6.119 |
| N25 | -0.364 | 0.360 | 3.435 | -0.268 |
| C26 | -0.087 | 2.345 | 4.469 | -6.358 |
| C27 | -0.099 | 0.572 | 5.210 | -4.894 |
| P28 | 1.835 | -1.216 | 2.627 | -0.253 |
| H29 | 0.146 | 2.523 | 2.041 | -1.063 |
| O30 | -0.751 | -1.124 | 1.076 | -0.183 |
| H31 | 0.114 | 3.596 | 2.116 | -3.315 |
| N32 | -0.410 | -2.134 | 3.229 | 1.126 |
| C33 | -0.101 | 2.855 | 3.675 | -5.368 |
| H34 | 0.112 | 0.778 | 5.858 | -6.926 |
| H35 | 0.110 | 2.826 | 4.517 | -7.341 |
| H36 | 0.124 | -0.327 | 5.814 | -4.711 |
| H37 | 0.108 | 3.750 | 3.065 | -5.543 |
| C38 | -0.054 | -3.524 | 2.696 | 1.202 |
| C39 | -0.068 | -2.036 | 4.663 | 1.468 |
| C40 | -0.071 | -2.176 | 2.301 | -2.877 |
| C41 | -0.068 | 1.270 | 3.143 | 0.861 |
| C42 | -0.122 | -4.036 | 2.706 | 2.642 |
| H43 | 0.052 | -4.211 | 3.278 | 0.550 |
| H44 | 0.079 | -3.506 | 1.653 | 0.809 |
| H45 | 0.047 | -2.376 | 5.327 | 0.657 |
| H46 | 0.059 | -0.996 | 4.903 | 1.725 |
| H47 | 0.061 | -2.668 | 4.852 | 2.353 |
| C48 | -0.093 | -5.344 | 1.932 | 2.775 |
| C49 | -0.117 | -5.145 | 0.518 | 3.304 |
| C50 | -0.039 | -4.492 | -0.406 | 2.271 |
| N51 | -0.473 | -3.746 | -1.507 | 2.939 |

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|---|---|---|---|---|
| H52 | 0.039 | -1.214 | 2.179 | -3.412 |
| H53 | 0.092 | -2.525 | 1.307 | -2.548 |
| H54 | 0.057 | -2.911 | 2.722 | -3.575 |
| H55 | 0.083 | 1.962 | 2.303 | 0.650 |
| H56 | 0.053 | 0.677 | 2.885 | 1.749 |
| H57 | 0.054 | 1.861 | 4.043 | 1.074 |
| H58 | 0.058 | -4.164 | 3.761 | 2.970 |
| H59 | 0.069 | -3.268 | 2.283 | 3.331 |
| H60 | 0.057 | -6.015 | 2.475 | 3.470 |
| H61 | 0.058 | -5.884 | 1.904 | 1.808 |
| H62 | 0.060 | -4.545 | 0.558 | 4.240 |
| H63 | 0.058 | -6.125 | 0.099 | 3.609 |
| H64 | 0.072 | -3.787 | 0.162 | 1.621 |
| H65 | 0.059 | -5.262 | -0.821 | 1.587 |
| P66 | 1.842 | -2.001 | -1.649 | 2.708 |
| N67 | -0.325 | -1.530 | -3.321 | 3.028 |
| C68 | -0.033 | -4.590 | -2.678 | 3.246 |
| N69 | -0.337 | -1.275 | -0.777 | 4.064 |
| O70 | -0.746 | -1.603 | -0.986 | 1.345 |
| C71 | -0.072 | -1.970 | -4.357 | 2.068 |
| C72 | -0.062 | -1.072 | 0.680 | 3.994 |
| C73 | -0.051 | -1.589 | -1.341 | 5.363 |
| C74 | -0.036 | -0.193 | -3.502 | 3.576 |
| C75 | -0.125 | -2.498 | -0.685 | 6.241 |
| C76 | -0.001 | -0.969 | -2.525 | 5.739 |
| C77 | -0.018 | -1.371 | -3.163 | 6.956 |
| C78 | -0.045 | -2.295 | -2.518 | 7.805 |
| C79 | -0.066 | -2.827 | -1.253 | 7.438 |
| C80 | -0.126 | 0.820 | -4.099 | 2.771 |
| C81 | -0.008 | 0.074 | -3.124 | 4.885 |
| C82 | -0.015 | 1.411 | -3.251 | 5.386 |
| C83 | -0.040 | 2.404 | -3.845 | 4.579 |
| C84 | -0.066 | 2.077 | -4.281 | 3.267 |
| C85 | -0.093 | 4.058 | -3.506 | 6.314 |
| C86 | -0.089 | 3.731 | -3.975 | 5.071 |
| C87 | -0.089 | -2.206 | -4.379 | 9.351 |
| Si88 | 1.389 | -0.759 | -0.615 | -0.112 |
| Cl89 | -0.520 | 1.072 | -0.068 | 1.147 |
| Cl90 | -0.597 | -0.276 | -2.923 | -0.180 |
| Cl91 | -0.481 | -2.680 | -0.938 | -1.281 |
| O92 | -0.356 | 0.145 | -0.423 | -1.611 |
| H93 | 0.049 | -5.488 | -2.337 | 3.782 |
| H94 | 0.033 | -4.918 | -3.212 | 2.340 |

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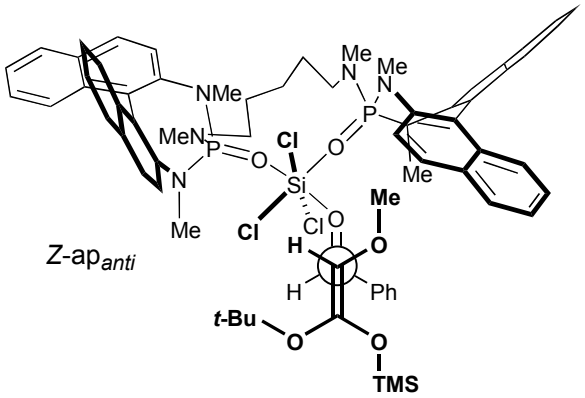
|---|---|---|---|---|
| H95 | 0.056 | -4.048 | -3.377 | 3.896 |
| H96 | 0.088 | -1.175 | -4.655 | 1.357 |
| H97 | 0.052 | -2.275 | -5.241 | 2.644 |
| H98 | 0.048 | -2.831 | -3.993 | 1.492 |
| H99 | 0.059 | -0.550 | 1.024 | 4.897 |
| H100 | 0.077 | -0.431 | 0.888 | 3.121 |
| H101 | 0.030 | -2.014 | 1.254 | 3.904 |
| H102 | 0.110 | -2.934 | 0.280 | 5.951 |
| H103 | 0.110 | -3.518 | -0.749 | 8.124 |
| H104 | 0.147 | 0.573 | -4.383 | 1.731 |
| H105 | 0.106 | 2.857 | -4.744 | 2.651 |
| C106 | 0.395 | 0.569 | -1.267 | -2.485 |
| C107 | -0.170 | 0.273 | -0.969 | -3.917 |
| C108 | -0.071 | 0.148 | 0.329 | -4.413 |
| C109 | -0.071 | 0.063 | -2.052 | -4.779 |
| C110 | -0.105 | -0.259 | -1.836 | -6.113 |
| C111 | -0.072 | -0.364 | -0.538 | -6.605 |
| C112 | -0.118 | -0.158 | 0.542 | -5.753 |
| C113 | -0.256 | 2.665 | -1.029 | -2.521 |
| C114 | -0.098 | 1.788 | -2.762 | 6.668 |
| C115 | -0.095 | -0.888 | -4.449 | 7.328 |
| C116 | -0.090 | -2.695 | -3.146 | 9.015 |
| C117 | 0.375 | 3.124 | -2.130 | -3.307 |
| H118 | 0.118 | 1.027 | -2.282 | 7.296 |
| C119 | -0.092 | 3.075 | -2.885 | 7.117 |
| O120 | -0.194 | 3.148 | -0.820 | -1.243 |
| H121 | 0.121 | -0.177 | -4.956 | 6.664 |
| C122 | -0.089 | -1.298 | -5.039 | 8.493 |
| H123 | 0.109 | -3.404 | -2.626 | 9.671 |
| H124 | 0.109 | -2.514 | -4.867 | 10.282 |
| H125 | 0.107 | 3.357 | -2.503 | 8.104 |
| H126 | 0.109 | -0.924 | -6.030 | 8.772 |
| H127 | 0.107 | 5.080 | -3.601 | 6.696 |
| H128 | 0.106 | 4.485 | -4.452 | 4.435 |
| H129 | 0.109 | 0.649 | -2.338 | -2.198 |
| H130 | 0.121 | 0.259 | 1.196 | -3.740 |
| H131 | 0.118 | 0.157 | -3.087 | -4.405 |
| H132 | 0.111 | -0.432 | -2.690 | -6.777 |
| H133 | 0.109 | -0.615 | -0.368 | -7.657 |
| H134 | 0.111 | -0.249 | 1.566 | -6.133 |
| C135 | 0.005 | 3.101 | -1.880 | -0.317 |
| O136 | -0.335 | 3.222 | -3.345 | -2.882 |
| Si137 | 0.734 | 3.467 | -4.814 | -3.773 |

| | | | | |
|---|---|---|---|---|
| C141 | -0.272 | 3.665 | -6.035 | -2.356 |
| C142 | -0.292 | 1.931 | -5.188 | -4.798 |
| C143 | -0.300 | 5.025 | -4.724 | -4.835 |
| O144 | -0.255 | 3.264 | -2.185 | -4.650 |
| C145 | 0.136 | 4.132 | -1.235 | -5.339 |
| H146 | 0.125 | 2.733 | -0.026 | -2.987 |
| H156 | 0.063 | 4.535 | -5.800 | -1.736 |
| H157 | 0.068 | 2.784 | -6.041 | -1.706 |
| H158 | 0.066 | 3.797 | -7.049 | -2.749 |
| H159 | 0.065 | 1.087 | -4.545 | -4.507 |
| H160 | 0.062 | 2.113 | -5.029 | -5.866 |
| H161 | 0.069 | 1.616 | -6.228 | -4.663 |
| H162 | 0.064 | 5.748 | -4.003 | -4.427 |
| H163 | 0.067 | 4.792 | -4.393 | -5.859 |
| H164 | 0.071 | 5.514 | -5.699 | -4.895 |
| C165 | -0.173 | 5.517 | -1.221 | -4.685 |
| C166 | -0.187 | 3.534 | 0.166 | -5.383 |
| C167 | -0.142 | 4.219 | -1.824 | -6.751 |
| H168 | 0.070 | 5.925 | -2.244 | -4.589 |
| H169 | 0.068 | 6.221 | -0.635 | -5.289 |
| H170 | 0.059 | 5.491 | -0.777 | -3.681 |
| H171 | 0.076 | 2.486 | 0.157 | -5.722 |
| H172 | 0.066 | 4.104 | 0.800 | -6.076 |
| H173 | 0.066 | 3.550 | 0.668 | -4.392 |
| H174 | 0.060 | 3.248 | -1.797 | -7.263 |
| H175 | 0.067 | 4.556 | -2.878 | -6.728 |
| H176 | 0.062 | 4.935 | -1.257 | -7.360 |
| H177 | 0.073 | 3.297 | -1.365 | 0.630 |
| H178 | 0.015 | 3.889 | -2.614 | -0.525 |
| H179 | 0.074 | 2.122 | -2.377 | -0.270 |



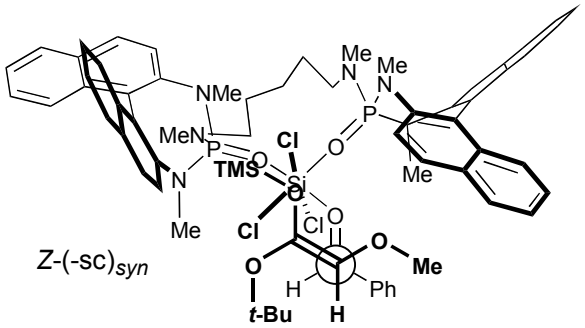

*Z*-ap$_{anti}$

| Atom | Partial Charge | X Coordinate | Y Coordinate | Z Coordinate |
|---|---|---|---|---|
| C1 | -0.085 | -2.225 | 9.300 | -2.587 |
| C2 | -0.086 | -0.941 | 8.742 | -2.774 |
| C3 | -0.095 | -0.754 | 7.388 | -2.698 |
| C4 | -0.021 | -1.845 | 6.518 | -2.425 |
| C5 | -0.040 | -3.121 | 7.083 | -2.215 |
| C6 | -0.090 | -3.294 | 8.490 | -2.314 |
| C7 | -0.074 | -4.222 | 6.243 | -1.895 |
| C8 | -0.103 | -4.038 | 4.901 | -1.719 |
| C9 | -0.059 | -2.746 | 4.327 | -1.897 |
| C10 | -0.011 | -1.679 | 5.098 | -2.327 |
| H11 | 0.111 | -2.353 | 10.386 | -2.661 |
| H12 | 0.110 | -0.095 | 9.407 | -2.981 |
| H13 | 0.120 | 0.242 | 6.952 | -2.851 |
| H14 | 0.112 | -4.295 | 8.912 | -2.163 |
| H15 | 0.114 | -5.216 | 6.691 | -1.778 |
| H16 | 0.119 | -4.882 | 4.254 | -1.449 |
| N17 | -0.400 | -2.631 | 2.911 | -1.575 |
| C18 | -0.034 | -0.385 | 4.476 | -2.665 |
| C19 | -0.042 | 0.347 | 3.763 | -1.726 |
| C20 | -0.016 | 0.099 | 4.561 | -4.012 |
| C21 | -0.134 | 1.608 | 3.201 | -2.073 |
| C22 | -0.061 | 2.098 | 3.323 | -3.340 |
| C23 | -0.044 | 1.350 | 3.997 | -4.340 |
| C24 | -0.085 | -0.170 | 5.235 | -6.330 |
| N25 | -0.345 | -0.152 | 3.604 | -0.370 |
| C26 | -0.091 | 1.096 | 4.691 | -6.645 |
| C27 | -0.104 | -0.656 | 5.172 | -5.052 |
| P28 | 1.839 | -1.563 | 2.540 | -0.229 |
| H29 | 0.133 | 2.173 | 2.636 | -1.309 |
| O30 | -0.748 | -1.099 | 1.053 | -0.170 |
| H31 | 0.117 | 3.058 | 2.850 | -3.603 |
| N32 | -0.413 | -2.432 | 2.991 | 1.234 |
| C33 | -0.085 | 1.840 | 4.082 | -5.671 |
| H34 | 0.110 | -0.758 | 5.710 | -7.123 |
| H35 | 0.110 | 1.469 | 4.761 | -7.672 |
| H36 | 0.121 | -1.637 | 5.603 | -4.812 |
| H37 | 0.110 | 2.820 | 3.649 | -5.902 |
| C38 | -0.072 | -3.672 | 2.199 | 1.483 |
| C39 | -0.073 | -2.593 | 4.434 | 1.516 |
| C40 | -0.061 | -2.839 | 1.965 | -2.686 |
| C41 | -0.067 | 0.885 | 3.407 | 0.667 |

| | | | | |
|---|---|---|---|---|
| C42 | -0.119 | -4.059 | 2.250 | 2.960 |
| H43 | 0.053 | -4.514 | 2.573 | 0.860 |
| H44 | 0.099 | -3.477 | 1.143 | 1.166 |
| H45 | 0.049 | -3.111 | 4.980 | 0.711 |
| H46 | 0.058 | -1.605 | 4.882 | 1.682 |
| H47 | 0.063 | -3.188 | 4.547 | 2.440 |
| C48 | -0.094 | -5.190 | 1.278 | 3.278 |
| C49 | -0.112 | -4.696 | -0.005 | 3.932 |
| C50 | -0.050 | -3.946 | -0.914 | 2.951 |
| N51 | -0.474 | -2.825 | -1.614 | 3.638 |
| H52 | 0.040 | -1.960 | 1.876 | -3.352 |
| H53 | 0.087 | -3.064 | 0.973 | -2.259 |
| H54 | 0.056 | -3.696 | 2.294 | -3.288 |
| H55 | 0.073 | 1.569 | 2.566 | 0.436 |
| H56 | 0.049 | 0.396 | 3.217 | 1.633 |
| H57 | 0.058 | 1.481 | 4.326 | 0.751 |
| H58 | 0.056 | -4.345 | 3.294 | 3.220 |
| H59 | 0.064 | -3.172 | 2.031 | 3.601 |
| H60 | 0.055 | -5.907 | 1.770 | 3.964 |
| H61 | 0.063 | -5.774 | 1.041 | 2.365 |
| H62 | 0.057 | -4.061 | 0.253 | 4.807 |
| H63 | 0.056 | -5.560 | -0.553 | 4.357 |
| H64 | 0.090 | -3.541 | -0.332 | 2.087 |
| H65 | 0.063 | -4.641 | -1.656 | 2.504 |
| P66 | 1.845 | -1.168 | -1.464 | 3.065 |
| N67 | -0.340 | -0.309 | -2.979 | 3.355 |
| C68 | -0.037 | -3.259 | -2.815 | 4.380 |
| N69 | -0.331 | -0.375 | -0.364 | 4.212 |
| O70 | -0.742 | -1.121 | -0.800 | 1.641 |
| C71 | -0.062 | -0.770 | -4.226 | 2.707 |
| C72 | -0.053 | -0.657 | 1.080 | 4.133 |
| C73 | -0.052 | -0.190 | -0.910 | 5.545 |
| C74 | -0.039 | 1.133 | -2.844 | 3.486 |
| C75 | -0.129 | -0.984 | -0.441 | 6.631 |
| C76 | -0.000 | 0.790 | -1.872 | 5.750 |
| C77 | -0.017 | 0.904 | -2.486 | 7.040 |
| C78 | -0.045 | 0.100 | -2.024 | 8.103 |
| C79 | -0.063 | -0.829 | -0.972 | 7.879 |
| C80 | -0.132 | 1.979 | -3.306 | 2.439 |
| C81 | -0.003 | 1.668 | -2.298 | 4.644 |
| C82 | -0.014 | 3.086 | -2.105 | 4.727 |
| C83 | -0.036 | 3.914 | -2.560 | 3.679 |
| C84 | -0.076 | 3.333 | -3.180 | 2.540 |

| | | | | |
|---|---|---|---|---|
| C85 | -0.090 | 5.875 | -1.738 | 4.835 |
| C86 | -0.092 | 5.321 | -2.376 | 3.759 |
| C87 | -0.089 | 1.075 | -3.666 | 9.589 |
| Si88 | 1.408 | -0.891 | -0.654 | -0.062 |
| Cl89 | -0.532 | 1.349 | -0.273 | 0.325 |
| Cl90 | -0.617 | -0.797 | -3.007 | -0.017 |
| Cl91 | -0.520 | -3.144 | -0.832 | -0.432 |
| O92 | -0.340 | -0.548 | -0.717 | -1.778 |
| H93 | 0.051 | -4.102 | -2.542 | 5.030 |
| H94 | 0.036 | -3.592 | -3.627 | 3.715 |
| H95 | 0.053 | -2.442 | -3.183 | 5.014 |
| H96 | 0.074 | -0.090 | -4.563 | 1.902 |
| H97 | 0.049 | -0.820 | -5.006 | 3.479 |
| H98 | 0.061 | -1.768 | -4.086 | 2.267 |
| H99 | 0.059 | -0.106 | 1.614 | 4.919 |
| H100 | 0.056 | -0.302 | 1.444 | 3.158 |
| H101 | 0.037 | -1.731 | 1.333 | 4.235 |
| H102 | 0.112 | -1.727 | 0.350 | 6.462 |
| H103 | 0.111 | -1.429 | -0.606 | 8.720 |
| H104 | 0.141 | 1.514 | -3.753 | 1.535 |
| H105 | 0.108 | 3.980 | -3.531 | 1.719 |
| C106 | 0.419 | -0.236 | -1.655 | -2.602 |
| C107 | -0.152 | -0.572 | -1.428 | -4.034 |
| C108 | -0.041 | -0.819 | -0.155 | -4.555 |
| C109 | -0.077 | -0.676 | -2.540 | -4.878 |
| C110 | -0.115 | -1.019 | -2.378 | -6.214 |
| C111 | -0.066 | -1.262 | -1.107 | -6.726 |
| C112 | -0.116 | -1.160 | 0.002 | -5.894 |
| C113 | -0.319 | 1.883 | -1.654 | -2.633 |
| C114 | -0.096 | 3.693 | -1.434 | 5.825 |
| C115 | -0.098 | 1.784 | -3.578 | 7.282 |
| C116 | -0.088 | 0.211 | -2.625 | 9.385 |
| C117 | 0.398 | 2.350 | -2.773 | -3.383 |
| H118 | 0.119 | 3.053 | -1.064 | 6.636 |
| C119 | -0.091 | 5.049 | -1.255 | 5.875 |
| O120 | -0.184 | 2.157 | -0.406 | -3.190 |
| H121 | 0.120 | 2.401 | -3.950 | 6.454 |
| C122 | -0.087 | 1.864 | -4.151 | 8.522 |
| H123 | 0.110 | -0.411 | -2.243 | 10.204 |
| H124 | 0.110 | 1.163 | -4.134 | 10.576 |
| H125 | 0.108 | 5.508 | -0.736 | 6.724 |
| H126 | 0.109 | 2.542 | -4.993 | 8.700 |
| H127 | 0.108 | 6.959 | -1.592 | 4.901 |

| | | | | |
|---|---|---|---|---|
| H128 | 0.105 | 5.950 | -2.748 | 2.943 |
| H129 | 0.097 | -0.227 | -2.707 | -2.246 |
| H130 | 0.120 | -0.759 | 0.732 | -3.905 |
| H131 | 0.111 | -0.468 | -3.556 | -4.502 |
| H132 | 0.108 | -1.091 | -3.256 | -6.866 |
| H133 | 0.107 | -1.533 | -0.982 | -7.780 |
| H134 | 0.108 | -1.350 | 1.005 | -6.292 |
| C135 | 0.008 | 3.299 | 0.207 | -2.625 |
| O136 | -0.328 | 2.565 | -2.752 | -4.660 |
| Si137 | 0.719 | 2.796 | -4.086 | -5.748 |
| C141 | -0.273 | 2.679 | -3.190 | -7.400 |
| C142 | -0.294 | 4.496 | -4.855 | -5.492 |
| C143 | -0.295 | 1.402 | -5.348 | -5.573 |
| O144 | -0.250 | 2.516 | -4.055 | -3.033 |
| C145 | 0.136 | 2.915 | -4.517 | -1.708 |
| H146 | 0.159 | 2.007 | -1.715 | -1.525 |
| H156 | 0.065 | 2.897 | -3.879 | -8.223 |
| H157 | 0.064 | 1.674 | -2.783 | -7.559 |
| H158 | 0.065 | 3.389 | -2.360 | -7.462 |
| H159 | 0.063 | 5.291 | -4.110 | -5.591 |
| H160 | 0.066 | 4.590 | -5.317 | -4.498 |
| H161 | 0.069 | 4.676 | -5.634 | -6.240 |
| H162 | 0.065 | 0.467 | -4.856 | -5.262 |
| H163 | 0.064 | 1.640 | -6.116 | -4.830 |
| H164 | 0.068 | 1.214 | -5.850 | -6.527 |
| C165 | -0.190 | 1.672 | -4.796 | -0.867 |
| C166 | -0.182 | 3.855 | -3.549 | -0.993 |
| C167 | -0.142 | 3.649 | -5.826 | -2.031 |
| H168 | 0.076 | 0.771 | -4.962 | -1.477 |
| H169 | 0.059 | 1.812 | -5.690 | -0.244 |
| H170 | 0.095 | 1.437 | -3.959 | -0.181 |
| H171 | 0.078 | 3.395 | -2.555 | -0.850 |
| H172 | 0.057 | 4.797 | -3.412 | -1.538 |
| H173 | 0.072 | 4.101 | -3.926 | 0.016 |
| H174 | 0.063 | 4.428 | -5.684 | -2.802 |
| H175 | 0.059 | 2.958 | -6.594 | -2.407 |
| H176 | 0.061 | 4.135 | -6.225 | -1.131 |
| H177 | 0.048 | 3.361 | 1.162 | -3.166 |
| H178 | 0.015 | 4.208 | -0.380 | -2.794 |
| H179 | 0.051 | 3.169 | 0.389 | -1.548 |




Z-(-sc)$_{syn}$

| Atom | Partial Charge | X Coordinate | Y Coordinate | Z Coordinate |
|---|---|---|---|---|
| C1 | -0.086 | -0.216 | 9.108 | -3.039 |
| C2 | -0.087 | 0.903 | 8.406 | -2.538 |
| C3 | -0.096 | 0.889 | 7.039 | -2.464 |
| C4 | -0.020 | -0.251 | 6.299 | -2.884 |
| C5 | -0.042 | -1.374 | 7.007 | -3.361 |
| C6 | -0.089 | -1.331 | 8.425 | -3.443 |
| C7 | -0.071 | -2.545 | 6.299 | -3.744 |
| C8 | -0.111 | -2.615 | 4.945 | -3.581 |
| C9 | -0.046 | -1.498 | 4.225 | -3.067 |
| C10 | -0.011 | -0.300 | 4.867 | -2.804 |
| H11 | 0.111 | -0.180 | 10.202 | -3.098 |
| H12 | 0.110 | 1.783 | 8.969 | -2.209 |
| H13 | 0.120 | 1.761 | 6.493 | -2.081 |
| H14 | 0.111 | -2.207 | 8.959 | -3.831 |
| H15 | 0.113 | -3.393 | 6.859 | -4.155 |
| H16 | 0.117 | -3.528 | 4.402 | -3.856 |
| N17 | -0.413 | -1.722 | 2.810 | -2.812 |
| C18 | -0.034 | 0.913 | 4.114 | -2.430 |
| C19 | -0.042 | 0.964 | 3.328 | -1.288 |
| C20 | -0.016 | 2.061 | 4.170 | -3.289 |
| C21 | -0.139 | 2.171 | 2.670 | -0.918 |
| C22 | -0.068 | 3.292 | 2.797 | -1.684 |
| C23 | -0.040 | 3.255 | 3.529 | -2.899 |
| C24 | -0.088 | 3.144 | 4.876 | -5.345 |
| N25 | -0.338 | -0.213 | 3.204 | -0.441 |
| C26 | -0.091 | 4.346 | 4.256 | -4.934 |
| C27 | -0.100 | 2.033 | 4.836 | -4.547 |
| P28 | 1.856 | -1.506 | 2.243 | -1.166 |
| H29 | 0.153 | 2.197 | 2.014 | -0.017 |
| O30 | -0.758 | -1.207 | 0.717 | -1.097 |
| H31 | 0.119 | 4.224 | 2.300 | -1.363 |
| N32 | -0.425 | -2.994 | 2.628 | -0.308 |
| C33 | -0.090 | 4.401 | 3.595 | -3.738 |

| | | | | |
|---|---|---|---|---|
| H34 | 0.109 | 3.113 | 5.391 | -6.312 |
| H35 | 0.109 | 5.225 | 4.310 | -5.585 |
| H36 | 0.120 | 1.104 | 5.325 | -4.867 |
| H37 | 0.106 | 5.323 | 3.102 | -3.410 |
| C38 | -0.067 | -4.159 | 1.735 | -0.571 |
| C39 | -0.072 | -3.363 | 4.057 | -0.208 |
| C40 | -0.035 | -1.644 | 1.906 | -3.974 |
| C41 | -0.084 | 0.055 | 2.937 | 0.990 |
| C42 | -0.119 | -5.141 | 1.779 | 0.600 |
| H43 | 0.050 | -4.679 | 2.026 | -1.510 |
| H44 | 0.101 | -3.783 | 0.691 | -0.720 |
| H45 | 0.049 | -3.509 | 4.540 | -1.188 |
| H46 | 0.057 | -2.582 | 4.593 | 0.347 |
| H47 | 0.063 | -4.309 | 4.139 | 0.356 |
| C48 | -0.094 | -6.247 | 0.741 | 0.446 |
| C49 | -0.111 | -6.026 | -0.489 | 1.315 |
| C50 | -0.051 | -4.861 | -1.356 | 0.824 |
| N51 | -0.475 | -4.066 | -1.873 | 1.974 |
| H52 | 0.043 | -0.608 | 1.633 | -4.246 |
| H53 | 0.066 | -2.196 | 0.984 | -3.735 |
| H54 | 0.052 | -2.114 | 2.386 | -4.843 |
| H55 | 0.082 | 0.826 | 2.155 | 1.145 |
| H56 | 0.053 | -0.872 | 2.611 | 1.495 |
| H57 | 0.057 | 0.401 | 3.868 | 1.456 |
| H58 | 0.057 | -5.566 | 2.805 | 0.675 |
| H59 | 0.063 | -4.599 | 1.625 | 1.564 |
| H60 | 0.055 | -7.216 | 1.200 | 0.725 |
| H61 | 0.063 | -6.361 | 0.441 | -0.616 |
| H62 | 0.058 | -5.874 | -0.167 | 2.368 |
| H63 | 0.056 | -6.955 | -1.093 | 1.332 |
| H64 | 0.093 | -4.186 | -0.778 | 0.146 |
| H65 | 0.061 | -5.240 | -2.204 | 0.214 |
| P66 | 1.840 | -2.320 | -1.666 | 2.056 |
| N67 | -0.334 | -1.586 | -3.132 | 2.717 |
| C68 | -0.040 | -4.704 | -3.016 | 2.658 |
| N69 | -0.330 | -2.060 | -0.478 | 3.352 |
| O70 | -0.745 | -1.763 | -1.042 | 0.723 |
| C71 | -0.059 | -1.783 | -4.421 | 2.019 |
| C72 | -0.060 | -2.419 | 0.932 | 3.110 |
| C73 | -0.053 | -2.301 | -0.978 | 4.694 |
| C74 | -0.037 | -0.274 | -2.922 | 3.306 |
| C75 | -0.131 | -3.442 | -0.546 | 5.430 |
| C76 | 0.001 | -1.386 | -1.856 | 5.260 |

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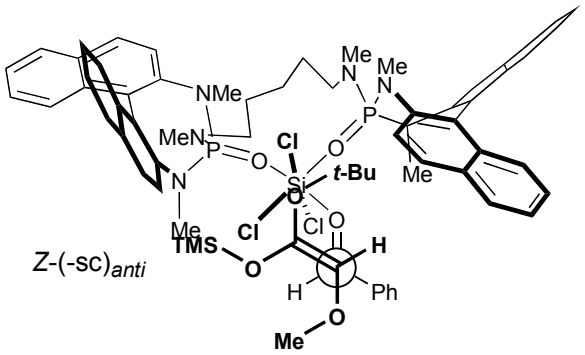
|---|---|---|---|---|
| C77 | -0.017 | -1.659 | -2.412 | 6.551 |
| C78 | -0.045 | -2.799 | -1.986 | 7.265 |
| C79 | -0.063 | -3.674 | -1.026 | 6.687 |
| C80 | -0.123 | 0.890 | -3.413 | 2.648 |
| C81 | -0.012 | -0.174 | -2.261 | 4.523 |
| C82 | -0.016 | 1.124 | -1.920 | 5.024 |
| C83 | -0.037 | 2.270 | -2.333 | 4.313 |
| C84 | -0.076 | 2.127 | -3.133 | 3.148 |
| C85 | -0.091 | 3.698 | -1.161 | 5.876 |
| C86 | -0.103 | 3.558 | -1.922 | 4.748 |
| C87 | -0.088 | -2.251 | -3.483 | 9.086 |
| Si88 | 1.402 | -0.886 | -0.940 | -0.762 |
| Cl89 | -0.514 | 0.970 | -0.307 | 0.403 |
| Cl90 | -0.624 | -0.500 | -3.250 | -0.398 |
| Cl91 | -0.512 | -2.810 | -1.411 | -1.909 |
| O92 | -0.323 | 0.022 | -1.031 | -2.266 |
| H93 | 0.052 | -5.751 | -2.761 | 2.876 |
| H94 | 0.035 | -4.697 | -3.933 | 2.049 |
| H95 | 0.053 | -4.194 | -3.214 | 3.610 |
| H96 | 0.076 | -0.839 | -4.833 | 1.616 |
| H97 | 0.048 | -2.200 | -5.132 | 2.745 |
| H98 | 0.056 | -2.482 | -4.307 | 1.179 |
| H99 | 0.059 | -2.312 | 1.517 | 4.034 |
| H100 | 0.056 | -1.716 | 1.351 | 2.367 |
| H101 | 0.041 | -3.451 | 1.071 | 2.733 |
| H102 | 0.112 | -4.139 | 0.175 | 4.985 |
| H103 | 0.111 | -4.545 | -0.690 | 7.262 |
| H104 | 0.150 | 0.786 | -4.001 | 1.709 |
| H105 | 0.111 | 3.025 | -3.500 | 2.632 |
| C106 | 0.403 | 0.456 | -2.044 | -2.921 |
| C107 | -0.148 | 0.329 | -2.078 | -4.401 |
| C108 | -0.044 | 0.004 | -0.962 | -5.171 |
| C109 | -0.083 | 0.534 | -3.314 | -5.032 |
| C110 | -0.108 | 0.381 | -3.432 | -6.405 |
| C111 | -0.066 | 0.037 | -2.316 | -7.167 |
| C112 | -0.114 | -0.143 | -1.085 | -6.549 |
| C113 | -0.329 | 2.607 | -2.036 | -2.935 |
| C114 | -0.094 | 1.303 | -1.130 | 6.194 |
| C115 | -0.098 | -0.829 | -3.410 | 7.134 |
| C116 | -0.088 | -3.075 | -2.531 | 8.547 |
| C117 | 0.426 | 3.130 | -2.174 | -1.620 |
| H118 | 0.118 | 0.415 | -0.816 | 6.758 |
| C119 | -0.094 | 2.556 | -0.769 | 6.611 |

| | | | | |
|---|---|---|---|---|
| O120 | -0.177 | 2.852 | -0.828 | -3.580 |
| H121 | 0.121 | 0.053 | -3.753 | 6.578 |
| C122 | -0.087 | -1.120 | -3.930 | 8.366 |
| H123 | 0.110 | -3.958 | -2.179 | 9.093 |
| H124 | 0.110 | -2.460 | -3.908 | 10.073 |
| H125 | 0.108 | 2.686 | -0.165 | 7.515 |
| H126 | 0.109 | -0.478 | -4.700 | 8.808 |
| H127 | 0.106 | 4.689 | -0.844 | 6.218 |
| H128 | 0.111 | 4.429 | -2.203 | 4.134 |
| H129 | 0.110 | 0.474 | -3.032 | -2.404 |
| H130 | 0.120 | -0.131 | 0.020 | -4.689 |
| H131 | 0.109 | 0.835 | -4.188 | -4.429 |
| H132 | 0.109 | 0.531 | -4.401 | -6.892 |
| H133 | 0.108 | -0.088 | -2.412 | -8.251 |
| H134 | 0.106 | -0.404 | -0.205 | -7.148 |
| C135 | 0.016 | 4.117 | -0.743 | -4.198 |
| O136 | -0.337 | 3.531 | -1.162 | -0.920 |
| Si137 | 0.744 | 4.874 | -0.897 | 0.120 |
| C141 | -0.299 | 5.229 | -2.167 | 1.462 |
| C142 | -0.308 | 6.346 | -0.798 | -1.070 |
| C143 | -0.288 | 4.420 | 0.793 | 0.815 |
| O144 | -0.228 | 3.199 | -3.220 | -0.794 |
| C145 | 0.131 | 3.101 | -4.621 | -1.174 |
| H146 | 0.121 | 2.681 | -2.937 | -3.579 |
| H156 | 0.074 | 4.716 | -1.935 | 2.407 |
| H157 | 0.065 | 4.932 | -3.179 | 1.163 |
| H158 | 0.063 | 6.305 | -2.189 | 1.670 |
| H159 | 0.058 | 6.938 | -1.717 | -1.058 |
| H160 | 0.066 | 6.011 | -0.626 | -2.104 |
| H161 | 0.066 | 7.007 | 0.029 | -0.794 |
| H162 | 0.064 | 4.814 | 0.936 | 1.824 |
| H163 | 0.094 | 3.322 | 0.910 | 0.850 |
| H164 | 0.060 | 4.806 | 1.596 | 0.169 |
| C165 | -0.149 | 4.455 | -5.059 | -1.737 |
| C166 | -0.202 | 1.982 | -4.969 | -2.150 |
| C167 | -0.143 | 2.816 | -5.296 | 0.175 |
| H168 | 0.057 | 5.280 | -4.807 | -1.056 |
| H169 | 0.065 | 4.472 | -6.146 | -1.888 |
| H170 | 0.046 | 4.664 | -4.586 | -2.705 |
| H171 | 0.106 | 1.006 | -4.582 | -1.799 |
| H172 | 0.068 | 1.892 | -6.059 | -2.244 |
| H173 | 0.059 | 2.146 | -4.568 | -3.170 |
| H174 | 0.078 | 1.884 | -4.906 | 0.633 |

| | | | | |
|---|---|---|---|---|
| H175 | 0.052 | 3.629 | -5.129 | 0.896 |
| H176 | 0.059 | 2.702 | -6.380 | 0.049 |
| H177 | 0.052 | 4.080 | 0.225 | -4.703 |
| H178 | 0.022 | 4.282 | -1.544 | -4.928 |
| H179 | 0.022 | 4.938 | -0.749 | -3.461 |




*Z*-(-sc)$_{anti}$

| Atom | Partial Charge | X Coordinate | Y Coordinate | Z Coordinate |
|---|---|---|---|---|
| C1 | -0.085 | -4.922 | 7.840 | -3.003 |
| C2 | -0.085 | -3.616 | 7.822 | -2.463 |
| C3 | -0.097 | -2.916 | 6.649 | -2.375 |
| C4 | -0.019 | -3.490 | 5.425 | -2.819 |
| C5 | -0.041 | -4.803 | 5.445 | -3.334 |
| C6 | -0.089 | -5.504 | 6.677 | -3.430 |
| C7 | -0.070 | -5.419 | 4.232 | -3.744 |
| C8 | -0.107 | -4.773 | 3.040 | -3.580 |
| C9 | -0.050 | -3.459 | 3.007 | -3.032 |
| C10 | -0.013 | -2.787 | 4.179 | -2.728 |
| H11 | 0.112 | -5.459 | 8.791 | -3.072 |
| H12 | 0.111 | -3.169 | 8.761 | -2.117 |
| H13 | 0.120 | -1.900 | 6.635 | -1.961 |
| H14 | 0.112 | -6.519 | 6.677 | -3.846 |
| H15 | 0.114 | -6.425 | 4.268 | -4.180 |
| H16 | 0.118 | -5.257 | 2.101 | -3.880 |
| N17 | -0.401 | -2.905 | 1.678 | -2.803 |
| C18 | -0.026 | -1.377 | 4.157 | -2.292 |
| C19 | -0.053 | -0.992 | 3.494 | -1.136 |
| C20 | -0.020 | -0.375 | 4.777 | -3.108 |
| C21 | -0.130 | 0.369 | 3.501 | -0.716 |
| C22 | -0.080 | 1.322 | 4.122 | -1.470 |
| C23 | -0.046 | 0.972 | 4.766 | -2.686 |
| C24 | -0.090 | 0.297 | 5.937 | -5.133 |
| N25 | -0.335 | -1.989 | 2.778 | -0.352 |

| | | | | |
|---|---|---|---|---|
| C26 | -0.090 | 1.640 | 5.943 | -4.693 |
| C27 | -0.100 | -0.684 | 5.374 | -4.362 |
| P28 | 1.830 | -2.480 | 1.274 | -1.145 |
| H29 | 0.153 | 0.651 | 2.953 | 0.205 |
| O30 | -0.763 | -1.343 | 0.209 | -1.057 |
| H31 | 0.120 | 2.382 | 4.076 | -1.165 |
| N32 | -0.415 | -3.957 | 0.690 | -0.387 |
| C33 | -0.092 | 1.971 | 5.371 | -3.495 |
| H34 | 0.112 | 0.050 | 6.395 | -6.097 |
| H35 | 0.112 | 2.407 | 6.412 | -5.320 |
| H36 | 0.123 | -1.728 | 5.381 | -4.702 |
| H37 | 0.110 | 3.009 | 5.369 | -3.141 |
| C38 | -0.070 | -4.379 | -0.685 | -0.785 |
| C39 | -0.073 | -5.080 | 1.647 | -0.292 |
| C40 | -0.052 | -2.212 | 1.064 | -3.952 |
| C41 | -0.083 | -1.725 | 2.689 | 1.102 |
| C42 | -0.118 | -5.301 | -1.292 | 0.271 |
| H43 | 0.052 | -4.892 | -0.673 | -1.771 |
| H44 | 0.101 | -3.463 | -1.320 | -0.905 |
| H45 | 0.050 | -5.409 | 2.036 | -1.270 |
| H46 | 0.057 | -4.782 | 2.486 | 0.350 |
| H47 | 0.064 | -5.940 | 1.136 | 0.177 |
| C48 | -0.094 | -5.607 | -2.755 | -0.032 |
| C49 | -0.111 | -4.815 | -3.717 | 0.841 |
| C50 | -0.045 | -3.314 | -3.718 | 0.522 |
| N51 | -0.474 | -2.507 | -3.672 | 1.775 |
| H52 | 0.047 | -1.279 | 1.587 | -4.222 |
| H53 | 0.070 | -1.978 | 0.016 | -3.707 |
| H54 | 0.054 | -2.882 | 1.084 | -4.821 |
| H55 | 0.083 | -0.663 | 2.468 | 1.335 |
| H56 | 0.052 | -2.346 | 1.894 | 1.552 |
| H57 | 0.060 | -1.993 | 3.650 | 1.559 |
| H58 | 0.057 | -6.237 | -0.692 | 0.321 |
| H59 | 0.063 | -4.845 | -1.192 | 1.285 |
| H60 | 0.056 | -6.687 | -2.941 | 0.131 |
| H61 | 0.063 | -5.431 | -2.980 | -1.104 |
| H62 | 0.061 | -5.002 | -3.473 | 1.908 |
| H63 | 0.056 | -5.219 | -4.741 | 0.707 |
| H64 | 0.093 | -3.039 | -2.846 | -0.123 |
| H65 | 0.058 | -3.046 | -4.621 | -0.066 |
| P66 | 1.834 | -1.179 | -2.537 | 1.980 |
| N67 | -0.333 | 0.227 | -3.337 | 2.689 |
| C68 | -0.042 | -2.466 | -4.963 | 2.494 |

| | | | | |
|---|---|---|---|---|
| N69 | -0.326 | -1.693 | -1.430 | 3.268 |
| O70 | -0.751 | -0.964 | -1.721 | 0.655 |
| C71 | -0.079 | 0.831 | -4.483 | 1.978 |
| C72 | -0.061 | -2.781 | -0.472 | 2.999 |
| C73 | -0.057 | -1.640 | -1.990 | 4.608 |
| C74 | -0.049 | 1.163 | -2.402 | 3.296 |
| C75 | -0.132 | -2.845 | -2.296 | 5.305 |
| C76 | 0.005 | -0.405 | -2.190 | 5.211 |
| C77 | -0.018 | -0.351 | -2.819 | 6.497 |
| C78 | -0.045 | -1.551 | -3.130 | 7.170 |
| C79 | -0.062 | -2.799 | -2.836 | 6.558 |
| C80 | -0.129 | 2.386 | -2.093 | 2.638 |
| C81 | -0.007 | 0.842 | -1.821 | 4.514 |
| C82 | -0.016 | 1.707 | -0.812 | 5.049 |
| C83 | -0.034 | 2.892 | -0.476 | 4.361 |
| C84 | -0.078 | 3.221 | -1.143 | 3.150 |
| C85 | -0.090 | 3.412 | 1.204 | 6.025 |
| C86 | -0.093 | 3.740 | 0.542 | 4.873 |
| C87 | -0.087 | -0.302 | -4.070 | 9.017 |
| Si88 | 1.398 | -0.174 | -1.004 | -0.694 |
| Cl89 | -0.513 | 0.849 | 0.466 | 0.732 |
| Cl90 | -0.573 | 1.410 | -2.694 | -0.565 |
| Cl91 | -0.541 | -1.381 | -2.327 | -2.139 |
| O92 | -0.330 | 0.831 | -0.327 | -1.974 |
| H93 | 0.053 | -3.496 | -5.319 | 2.645 |
| H94 | 0.036 | -1.918 | -5.742 | 1.940 |
| H95 | 0.053 | -2.001 | -4.827 | 3.479 |
| H96 | 0.092 | 1.758 | -4.214 | 1.428 |
| H97 | 0.054 | 1.076 | -5.249 | 2.725 |
| H98 | 0.048 | 0.116 | -4.903 | 1.257 |
| H99 | 0.061 | -3.099 | 0.022 | 3.929 |
| H100 | 0.056 | -2.399 | 0.314 | 2.322 |
| H101 | 0.041 | -3.674 | -0.934 | 2.535 |
| H102 | 0.112 | -3.816 | -2.098 | 4.833 |
| H103 | 0.112 | -3.724 | -3.063 | 7.102 |
| H104 | 0.154 | 2.634 | -2.614 | 1.686 |
| H105 | 0.123 | 4.136 | -0.854 | 2.595 |
| C106 | 0.425 | 1.262 | -0.881 | -3.041 |
| C107 | -0.177 | 1.200 | -0.081 | -4.292 |
| C108 | -0.069 | 1.235 | 1.316 | -4.297 |
| C109 | -0.050 | 1.079 | -0.772 | -5.503 |
| C110 | -0.106 | 0.974 | -0.072 | -6.699 |
| C111 | -0.063 | 1.000 | 1.319 | -6.697 |

| | | | | |
|---|---|---|---|---|
| C112 | -0.124 | 1.134 | 2.011 | -5.497 |
| C113 | -0.335 | 3.505 | -0.909 | -3.033 |
| C114 | -0.097 | 1.390 | -0.100 | 6.239 |
| C115 | -0.098 | 0.882 | -3.171 | 7.113 |
| C116 | -0.088 | -1.505 | -3.752 | 8.447 |
| C117 | 0.416 | 4.159 | -1.273 | -1.827 |
| H118 | 0.118 | 0.466 | -0.352 | 6.775 |
| C119 | -0.092 | 2.221 | 0.880 | 6.713 |
| O120 | -0.204 | 3.861 | -1.606 | -4.185 |
| H121 | 0.121 | 1.818 | -2.941 | 6.589 |
| C122 | -0.086 | 0.903 | -3.782 | 8.338 |
| H123 | 0.111 | -2.447 | -3.974 | 8.961 |
| H124 | 0.111 | -0.261 | -4.552 | 10.000 |
| H125 | 0.108 | 1.969 | 1.423 | 7.630 |
| H126 | 0.110 | 1.856 | -4.052 | 8.806 |
| H127 | 0.107 | 4.064 | 1.988 | 6.425 |
| H128 | 0.104 | 4.656 | 0.790 | 4.324 |
| H129 | 0.121 | 1.298 | -1.991 | -3.105 |
| H130 | 0.119 | 1.346 | 1.870 | -3.353 |
| H131 | 0.121 | 1.101 | -1.874 | -5.493 |
| H132 | 0.113 | 0.878 | -0.618 | -7.643 |
| H133 | 0.109 | 0.922 | 1.870 | -7.640 |
| H134 | 0.117 | 1.160 | 3.108 | -5.491 |
| C135 | 0.034 | 3.314 | -2.895 | -4.343 |
| O136 | -0.342 | 4.599 | -2.469 | -1.596 |
| Si137 | 0.727 | 5.450 | -3.220 | -0.298 |
| C141 | -0.292 | 6.177 | -2.135 | 1.062 |
| C142 | -0.298 | 4.234 | -4.456 | 0.429 |
| C143 | -0.275 | 6.839 | -4.090 | -1.237 |
| O144 | -0.238 | 4.368 | -0.582 | -0.694 |
| C145 | 0.139 | 4.649 | 0.848 | -0.677 |
| H146 | 0.133 | 3.524 | 0.164 | -3.297 |
| H156 | 0.059 | 6.823 | -1.350 | 0.655 |
| H157 | 0.067 | 5.394 | -1.646 | 1.663 |
| H158 | 0.065 | 6.782 | -2.750 | 1.738 |
| H159 | 0.083 | 3.318 | -3.941 | 0.771 |
| H160 | 0.067 | 3.932 | -5.204 | -0.311 |
| H161 | 0.059 | 4.671 | -4.981 | 1.283 |
| H162 | 0.062 | 7.329 | -4.826 | -0.592 |
| H163 | 0.057 | 7.600 | -3.383 | -1.577 |
| H164 | 0.064 | 6.456 | -4.618 | -2.116 |
| C165 | -0.209 | 3.477 | 1.678 | -1.183 |
| C166 | -0.158 | 5.911 | 1.145 | -1.491 |

| | | | | |
|---|---|---|---|---|
| C167 | -0.148 | 4.899 | 1.125 | 0.813 |
| H168 | 0.110 | 2.535 | 1.395 | -0.679 |
| H169 | 0.059 | 3.625 | 2.757 | -1.003 |
| H170 | 0.058 | 3.326 | 1.553 | -2.269 |
| H171 | 0.058 | 6.760 | 0.525 | -1.168 |
| H172 | 0.060 | 6.203 | 2.195 | -1.368 |
| H173 | 0.056 | 5.758 | 0.966 | -2.565 |
| H174 | 0.050 | 5.914 | 0.827 | 1.114 |
| H175 | 0.079 | 4.198 | 0.572 | 1.469 |
| H176 | 0.057 | 4.787 | 2.194 | 1.033 |
| H177 | 0.044 | 3.739 | -3.218 | -5.297 |
| H178 | 0.021 | 2.212 | -2.855 | -4.414 |
| H179 | 0.043 | 3.601 | -3.590 | -3.546 |

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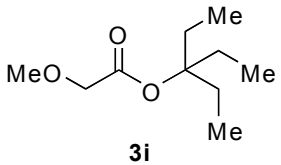
01/24/2007, chung, SED, WJC-XVIII-95 aft
er distillation

exp1  s2pul

      SAMPLE            DEC. & VT
date   Jan 24 2007  dfrq       499.432
solvent      CDCl3  dn               H1
file /export/home/~ dpwr             25
user1d/data/ui500n~ dof               0
b/chung/WJC-XVIII-~ dm              nnn
95-distillation.fi~ dmm               c
                  d  dmf             200
   ACQUISITION       dseq
sfrq       499.432  dres            1.0
tn               H1  homo              n
at            4.096        DEC2
np            65536  dfrq2             0
sw           8000.0  dn2
fb             4000  dpwr2             1
bs                4  dof2              0
tpwr             62  dm2               n
pw              5.0  dmm2              c
d1           10.000  dmf2            200
tof               0  dseq2
nt               16  dres2           1.0
ct               16  homo2             n
alock             n      PROCESSING
gain      not used  lb             0.30
      FLAGS         wtfile
il                n  proc             ft
in                n  fn         not used
dp                y  math              f
hs               nn
     DISPLAY        werr
sp           -249.9  wexp
wp           5493.7  wbs
vs              151  wnt
sc                0
wc              250
hzmm          21.97
is           321.89
rfl          5135.5
rfp          3625.9
th                4
ins           2.000
nm    ph
```

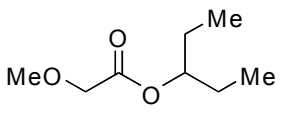



**3i**

```
02/12/2007, chung, SED, WJC-XIX-13 after
 distillation

exp1  s2pul

      SAMPLE            DEC. & VT
date   Feb 12 2007  dfrq       499.432
solvent      CDCl3  dn               H1
file /export/home/~ dpwr             25
user1d/data/ui500n~ dof               0
b/chung/WJC-XIX-13~ dm              nnn
 -distillation.fid  dmm               c
   ACQUISITION      dmf             200
sfrq       499.432  dseq
tn               H1  dres            1.0
at            4.096  homo              n
np            65536        DEC2
sw           8000.0  dfrq2             0
fb             4000  dn2
bs                4  dpwr2             1
tpwr             62  dof2              0
pw              5.0  dm2               n
d1           10.000  dmm2              c
tof               0  dmf2            200
nt               16  dseq2
ct               16  dres2           1.0
alock             n  homo2             n
gain      not used     PROCESSING
      FLAGS          lb             0.30
il                n  wtfile
in                n  proc              ft
dp                y  fn         not used
hs               nn  math              f
     DISPLAY
sp           -249.9  werr
wp           5493.7  wexp
vs              151  wbs
sc                0  wnt
wc              250
hzmm          21.97
is           221.04
rfl          5135.5
rfp          3625.9
th                2
ins           2.000
nm    ph
```

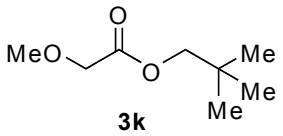



**3j**

```
01/24/2007, chung, SED, WJC-XVIII-97 aft
er distillation

exp1  s2pul

      SAMPLE            DEC. & VT
date   Jan 24 2007  dfrq       499.432
solvent      CDCl3  dn               H1
file /export/home/~ dpwr             25
user1d/data/ui500n~ dof               0
b/chung/WJC-XVIII-~ dm              nnn
97-distillation.fi~ dmm               c
                  d  dmf             200
   ACQUISITION       dseq
sfrq       499.432  dres            1.0
tn               H1  homo              n
at            4.096        DEC2
np            65536  dfrq2             0
sw           8000.0  dn2
fb             4000  dpwr2             1
bs                4  dof2              0
tpwr             62  dm2               n
pw              5.0  dmm2              c
d1           10.000  dmf2            200
tof               0  dseq2
nt               16  dres2           1.0
ct               16  homo2             n
alock             n      PROCESSING
gain      not used  lb             0.30
      FLAGS         wtfile
il                n  proc             ft
in                n  fn         not used
dp                y  math              f
hs               nn
     DISPLAY        werr
sp           -249.9  wexp
wp           5493.7  wbs
vs              151  wnt
sc                0
wc              250
hzmm          21.97
is           297.53
rfl          5135.5
rfp          3625.9
th                2
ins           2.000
nm    ph
```

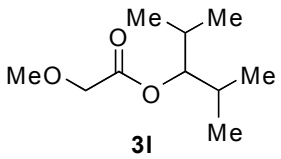



**3k**

```
01/24/2007, chung, SED, WJC-XVIII-37 aft
er distillation

exp1  s2pul

      SAMPLE            DEC. & VT
date   Jan 24 2007  dfrq       499.432
solvent      CDCl3  dn               H1
file /export/home/~ dpwr             25
user1d/data/ui500n~ dof               0
b/chung/WJC-XVIII-~ dm              nnn
37-distillation.fi~ dmm               c
                  d  dmf             200
   ACQUISITION       dseq
sfrq       499.432  dres            1.0
tn               H1  homo              n
at            4.096        DEC2
np            65536  dfrq2             0
sw           8000.0  dn2
fb             4000  dpwr2             1
bs                4  dof2              0
tpwr             62  dm2               n
pw              5.0  dmm2              c
d1           10.000  dmf2            200
tof               0  dseq2
nt               16  dres2           1.0
ct               16  homo2             n
alock             n      PROCESSING
gain      not used  lb             0.30
      FLAGS         wtfile
il                n  proc             ft
in                n  fn         not used
dp                y  math              f
hs               nn
     DISPLAY        werr
sp           -249.9  wexp
wp           5493.7  wbs
vs              151  wnt
sc                0
wc              250
hzmm          21.97
is           164.39
rfl          5135.3
rfp          3625.9
th                2
ins           2.000
nm    ph
```

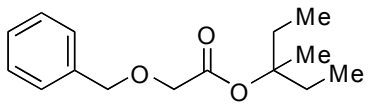



**3l**

```
11/13/2006, chung, SED, WJC-XVIII-31 aft
er distillation

exp1  s2pul

      SAMPLE            DEC. & VT
date   Nov 13 2006  dfrq       499.432
solvent      CDCl3  dn               H1
file /export/home/~ dpwr             25
user1d/data/ui500n~ dof               0
b/chung/WJC-XVIII-~ dm              nnn
31-distillation.fi~ dmm               c
                  d  dmf             200
   ACQUISITION       dseq
sfrq       499.432  dres            1.0
tn               H1  homo              n
at            4.096        DEC2
np            65536  dfrq2             0
sw           8000.0  dn2
fb             4000  dpwr2             1
bs                4  dof2              0
tpwr             62  dm2               n
pw              5.0  dmm2              c
d1           10.000  dmf2            200
tof               0  dseq2
nt               16  dres2           1.0
ct               16  homo2             n
alock             n      PROCESSING
gain      not used  lb             0.30
      FLAGS         wtfile
il                n  proc             ft
in                n  fn         not used
dp                y  math              f
hs               nn
     DISPLAY        werr
sp          -249.9  wexp
wp          5493.7  wbs
vs             151  wnt
sc               0
wc             250
hzmm         21.97
is          239.68
rfl         5134.0
rfp         3625.9
th               4
ins          2.000
nm    ph
```

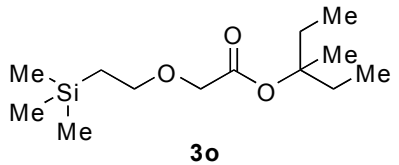



**3n**

```
08/10/2005, chung, SED, WJC-XII-52 after
 filtration through silica gel

exp1  std1h

      SAMPLE            DEC. & VT
date   Aug 10 2005  dn               H1
solvent      CDCl3  dof               0
file /export/home/~ dm              nnn
user1d/data/ui500n~ dmm               c
b/chung/WJC-XII-52~ dmf             200
   -filtration.fid  dpwr             20
   ACQUISITION          PROCESSING
sfrq       399.951  lb             0.30
tn              H1  wtfile
at           4.096  proc             ft
np           65536  fn        not used
sw          8000.0  math              f
fb            4400
bs              16  werr
tpwr            63  wexp
pw             7.3  wbs
d1               0  wnt
tof         -425.7       DISPLAY
nt             100  sp          -200.0
ct              20  wp          4399.4
alock            n  vs             151
gain      not used  sc               0
      FLAGS         wc             250
il               n  hzmm         17.60
in               n  is         1520.00
dp               y  rfl          4903.2
hs              nn  rfp          2903.6
                    th               20
                    ins           2.000
                    nm    ph
```

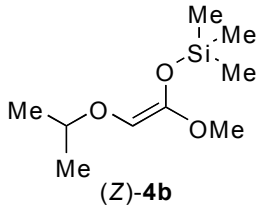



**3o**

```
08/25/2006, chung, SED, WJC-XVII-18 afte
r distillation

exp1  s2pul

      SAMPLE            DEC. & VT
date   Aug 25 2006  dfrq       499.438
solvent      CDCl3  dn               H1
file /export/home/~ dpwr             25
user1d/data/ui500n~ dof               0
b/chung/WJC-XVII-1~ dm              nnn
8-distillation.fid  dmm               c
   ACQUISITION      dmf             200
sfrq       499.438  dseq
tn              H1  dres            1.0
at           4.096  homo              n
np           65536        DEC2
sw          8000.0  dfrq2    undefined
fb            4400  dn2      undefined
bs               4  dpwr2    undefined
tpwr            62  dof2     undefined
pw             5.0  dm2      undefined
d1          10.000  dmm2     undefined
tof              0  dmf2     undefined
nt              16  dseq2    undefined
ct              16  dres2    undefined
alock            n  homo2    undefined
gain      not used      PROCESSING
      FLAGS          lb            0.30
il               n  wtfile
in               n  proc            ft
dp               y  fn        not used
hs              nn  math              f
     DISPLAY
sp          -249.7  werr
wp          5493.7  wexp
vs             151  wbs
sc               0  wnt
wc             250
hzmm         21.98
is          685.49
rfl         5137.1
rfp         3625.9
th              20
ins          3.000
nm    ph
```

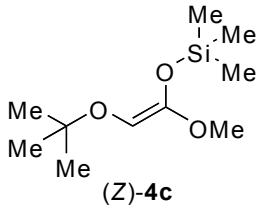



(*Z*)-**4b**

```
08/19/2006, chung, SED, WJC-XVII-17 afte
r distillation

exp1  s2pul

      SAMPLE            DEC. & VT
date   Aug 19 2006  dfrq       499.438
solvent      CDCl3  dn               H1
file /export/home/~ dpwr             25
user1d/data/ui500n~ dof               0
b/chung/WJC-XVII-1~ dm              nnn
7-distillation-2.f~ dmm               c
                 id  dmf             200
   ACQUISITION       dseq
sfrq       499.438  dres            1.0
tn               H1  homo              n
at            4.096        DEC2
np            65536  dfrq2    undefined
sw           8000.0  dn2      undefined
fb             4400  dpwr2    undefined
bs                4  dof2     undefined
tpwr             62  dm2      undefined
pw              5.0  dmm2     undefined
d1           10.000  dmf2     undefined
tof               0  dseq2    undefined
nt               16  dres2    undefined
ct               16  homo2    undefined
alock             n      PROCESSING
gain      not used  lb             0.30
      FLAGS         wtfile
il                n  proc             ft
in                n  fn         not used
dp                y  math              f
hs               nn
     DISPLAY        werr
sp           -249.7  wexp
wp           5493.7  wbs
vs              151  wnt
sc                0
wc              250
hzmm          21.98
is           725.08
rfl          5135.2
rfp          3625.9
th               18
ins           3.000
nm    ph
```

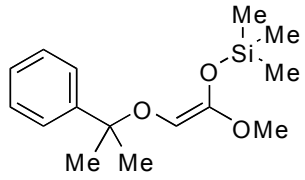



(*Z*)-**4c**

09/06/2007, chung, SED, WJC-XV-67 after distillation

exp1 s2pul

| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Sep 6 2007 | dfrq | 500.070 |
| solvent | CDCl3 | dn | H1 |
| file | exp | dpwr | 18 |
| ACQUISITION | | dof | 0 |
| sfrq | 500.070 | dm | nnn |
| tn | H1 | dmm | c |
| at | 4.096 | dmf | 200 |
| np | 65536 | dseq | |
| sw | 8000.0 | dres | 1.0 |
| fb | 4000 | homo | n |
| bs | 16 | DEC2 | |
| tpwr | 55 | dfrq2 | 0 |
| pw | 7.7 | dn2 | |
| d1 | 10.000 | dpwr2 | 1 |
| tof | 0 | dof2 | 0 |
| nt | 16 | dm2 | n |
| ct | 16 | dmm2 | c |
| alock | n | dmf2 | 200 |
| gain | not used | dseq2 | |
| FLAGS | | dres2 | 1.0 |
| il | n | homo2 | n |
| in | n | PROCESSING | |
| dp | y | lb | 0.30 |
| hs | nn | wtfile | |
| DISPLAY | | proc | ft |
| sp | -250.1 | fn | not used |
| wp | 5500.7 | math | f |
| vs | 21 | | |
| sc | 0 | werr | |
| wc | 250 | wexp | |
| hzmm | 22.00 | wbs | |
| is | 365.64 | wnt | |
| rfl | 5141.1 | | |
| rfp | 3630.5 | | |
| th | 7 | | |
| ins | 300.000 | | |
| ai | ph | | |

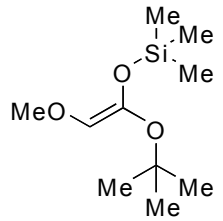



(*Z*)-**4d**

```
12/30/2006, chung, SED, WJC-XVIII-86 aft
er distillation

exp1  s2pul

      SAMPLE            DEC. & VT
date   Dec 30 2006  dfrq       500.075
solvent      CDCl3  dn               H1
file /export/home/~ dpwr             18
user1d/data/ui500n~ dof               0
b/chung/WJC-XVIII-~ dm              nnn
86-distillation.fi~ dmm               c
                  d  dmf             200
   ACQUISITION       dseq
sfrq       500.075  dres            1.0
tn               H1 homo              n
at            4.096        DEC2
np            65536  dfrq2             0
sw           8000.0  dn2
fb        not used   dpwr2             1
bs               16  dof2              0
tpwr             55  dm2               n
pw              7.7  dmm2              c
d1           10.000  dmf2            200
tof               0  dseq2
nt               16  dres2           1.0
ct               16  homo2             n
alock             n      PROCESSING
gain      not used   lb            0.30
      FLAGS          wtfile
il                n  proc             ft
in                n  fn         not used
dp                y  math              f
hs               nn
     DISPLAY         werr
sp           -250.1  wexp
wp           5500.7  wbs
vs               31  wnt
sc                0
wc              250
hzmm          22.00
is           436.75
rfl          5133.3
rfp          3630.5
th               19
ins           3.000
ai    ph
```

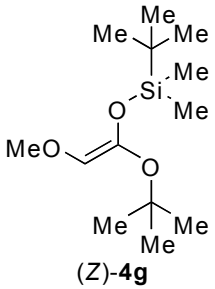



(*Z*)-**4f**

```
12/29/2006, chung, SED, WJC-XVIII-81 aft
er distillation

exp1  s2pul

      SAMPLE            DEC. & VT
date   Dec 29 2006  dfrq       500.075
solvent      CDCl3  dn               H1
file /export/home/~ dpwr             18
user1d/data/ui500n~ dof               0
b/chung/WJC-XVIII-~ dm              nnn
81-distillation.fi~ dmm               c
                  d  dmf             200
   ACQUISITION       dseq
sfrq       500.075  dres            1.0
tn               H1 homo              n
at            4.096        DEC2
np            65536  dfrq2             0
sw           8000.0  dn2
fb        not used   dpwr2             1
bs               16  dof2              0
tpwr             55  dm2               n
pw              7.7  dmm2              c
d1           10.000  dmf2            200
tof               0  dseq2
nt               16  dres2           1.0
ct               16  homo2             n
alock             n      PROCESSING
gain      not used   lb            0.30
      FLAGS          wtfile
il                n  proc             ft
in                n  fn         not used
dp                y  math              f
hs               nn
     DISPLAY         werr
sp           -250.1  wexp
wp           5500.7  wbs
vs               25  wnt
sc                0
wc              250
hzmm          22.00
is           687.76
rfl          5133.3
rfp          3630.5
th                7
ins           3.000
ai    ph
```

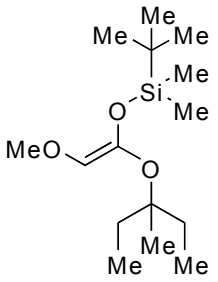



(*Z*)-**4g**

```
09/06/2007, chung, SED, WJC-XV-68 after
distillation

exp2  s2pul

      SAMPLE            DEC. & VT
date   Sep  6 2007  dfrq       500.070
solvent      CDCl3  dn              H1
file           exp  dpwr            18
   ACQUISITION      dof              0
sfrq       500.070  dm             nnn
tn              H1  dmm              c
at           4.096  dmf            200
np           65536  dseq
sw          8000.0  dres           1.0
fb            4000  homo             n
bs              16        DEC2
tpwr            55  dfrq2            0
pw             7.7  dn2
d1          10.000  dpwr2            1
tof              0  dof2             0
nt              16  dm2              n
ct              16  dmm2             c
alock            n  dmf2           200
gain      not used  dseq2
      FLAGS         dres2          1.0
il               n  homo2            n
in               n     PROCESSING
dp               y  lb           0.30
hs              nn  wtfile
     DISPLAY        proc             ft
sp          -250.1  fn        not used
wp          5500.7  math             f
vs              26
sc               0  werr
wc             250  wexp
hzmm         22.00  wbs
is          408.42  wnt
rfl         5141.1
rfp         3630.5
th               7
ins        300.000
ai    ph
```

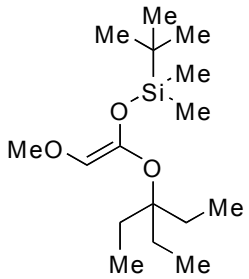



(*Z*)-**4h**

```
01/29/2007, chung, SED, WJC-XIX-10 after
 distillation

exp1  s2pul

      SAMPLE            DEC. & VT
date   Jan 29 2007  dfrq       500.075
solvent      CDCl3  dn               H1
file /export/home/~ dpwr             18
user1d/data/ui500n~ dof               0
b/chung/WJC-XIX-10~ dm              nnn
 -distillation.fid  dmm               c
   ACQUISITION      dmf             200
sfrq       500.075  dseq
tn               H1  dres            1.0
at            4.096  homo              n
np            65536        DEC2
sw           8000.0  dfrq2             0
fb        not used  dn2
bs               16  dpwr2             1
tpwr             55  dof2              0
pw              7.7  dm2               n
d1           10.000  dmm2              c
tof               0  dmf2            200
nt               16  dseq2
ct               16  dres2           1.0
alock             n  homo2             n
gain      not used      PROCESSING
      FLAGS          lb             0.30
il                n  wtfile
in                n  proc              ft
dp                y  fn         not used
hs               nn  math              f
     DISPLAY
sp           -250.1  werr
wp           5500.7  wexp
vs               17  wbs
sc                0  wnt
wc              250
hzmm          22.00
is           367.87
rfl          5132.3
rfp          3630.5
th               13
ins           6.000
ai    ph
```

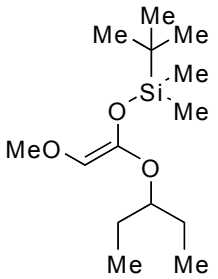



(*Z*)-**4i**

```
02/13/2007, chung, SED, WJC-XIX-22 after
 distillation

exp1  s2pul

      SAMPLE            DEC. & VT
date   Feb 13 2007  dfrq       500.075
solvent      CDCl3  dn               H1
file /export/home/~ dpwr             18
user1d/data/ui500n~ dof               0
b/chung/WJC-XIX-22~ dm              nnn
 -distillation.fid  dmm               c
   ACQUISITION      dmf             200
sfrq       500.075  dseq
tn               H1  dres            1.0
at            4.096  homo              n
np            65536        DEC2
sw           8000.0  dfrq2             0
fb             4000  dn2
bs               16  dpwr2             1
tpwr             55  dof2              0
pw              7.7  dm2               n
d1           10.000  dmm2              c
tof               0  dmf2            200
nt               16  dseq2
ct               16  dres2           1.0
alock             n  homo2             n
gain      not used     PROCESSING
      FLAGS          lb             0.30
il                n  wtfile
in                n  proc              ft
dp                y  fn         not used
hs               nn  math              f
     DISPLAY
sp           -250.1  werr
wp           5500.7  wexp
vs               16  wbs
sc                0  wnt
wc              250
hzmm          22.00
is           369.76
rfl          5132.8
rfp          3630.5
th               24
ins           3.000
ai    ph
```

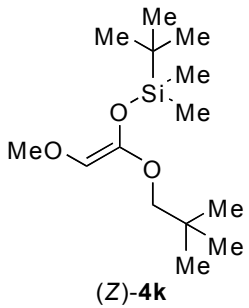



(*Z*)-**4j**

```
01/29/2007, chung, SED, WJC-XIX-11 after
 distillation

exp1  s2pul

      SAMPLE            DEC. & VT
date   Jan 29 2007  dfrq       500.075
solvent      CDCl3  dn               H1
file /export/home/~ dpwr             18
user1d/data/ui500n~ dof               0
b/chung/WJC-XIX-11~ dm              nnn
 -distillation.fid  dmm               c
   ACQUISITION      dmf             200
sfrq       500.075  dseq
tn               H1  dres            1.0
at            4.096  homo              n
np            65536        DEC2
sw           8000.0  dfrq2             0
fb        not used  dn2
bs               16  dpwr2             1
tpwr             55  dof2              0
pw              7.7  dm2               n
d1           10.000  dmm2              c
tof               0  dmf2            200
nt               16  dseq2
ct               16  dres2           1.0
alock             n  homo2             n
gain      not used      PROCESSING
      FLAGS          lb             0.30
il                n  wtfile
in                n  proc              ft
dp                y  fn         not used
hs               nn  math              f
     DISPLAY
sp           -250.1  werr
wp           5500.7  wexp
vs               15  wbs
sc                0  wnt
wc              250
hzmm          22.00
is           333.07
rfl          5132.3
rfp          3630.5
th               18
ins           3.000
ai    ph
```

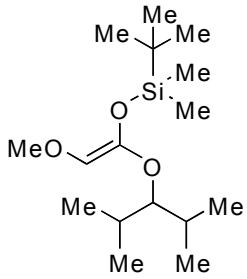



(*Z*)-**4k**

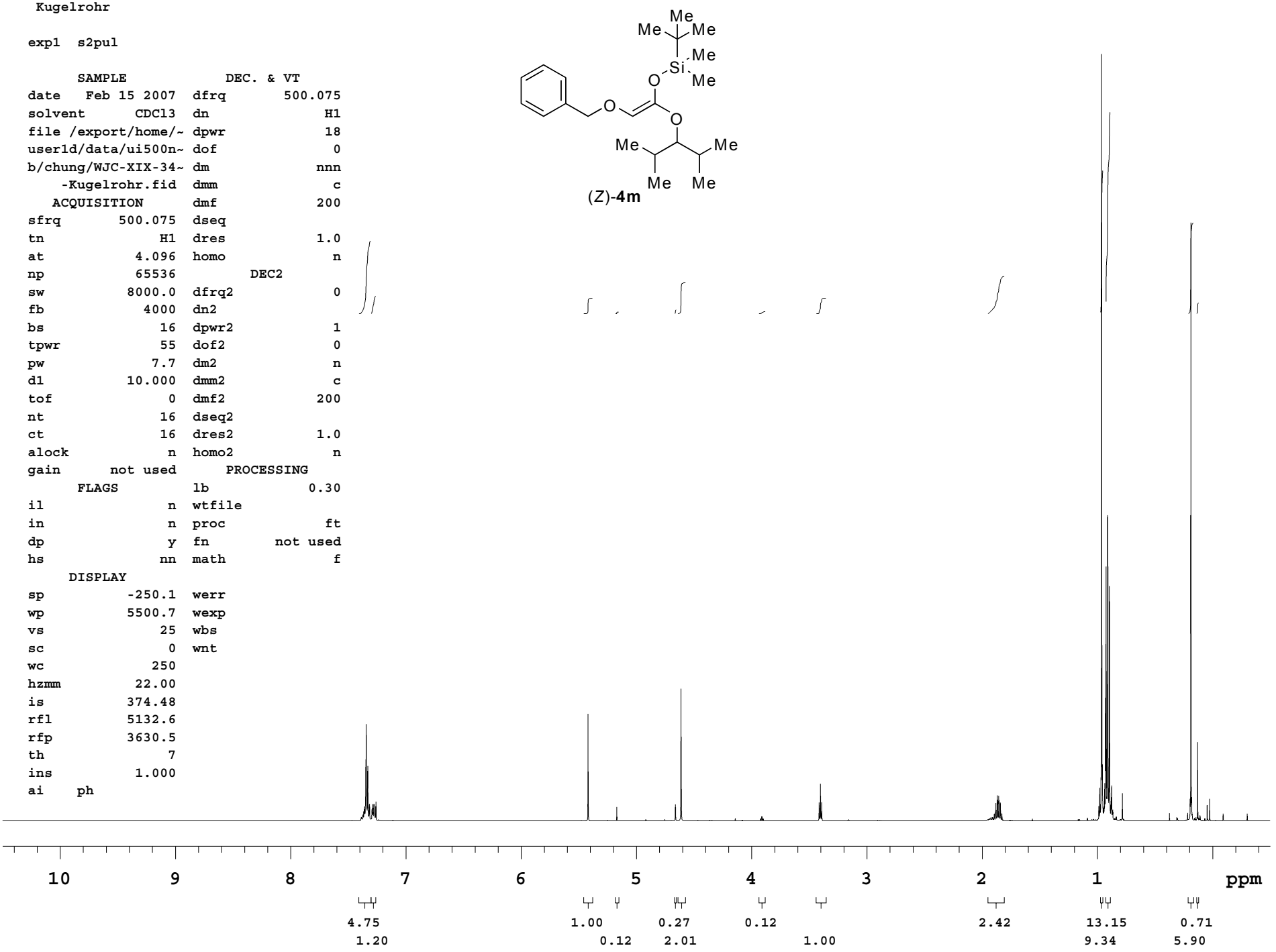
```
02/04/2007, chung, SED, WJC-XIX-16 after
 distillation

exp1  s2pul

      SAMPLE            DEC. & VT
date   Feb  4 2007  dfrq       500.075
solvent      CDCl3  dn              H1
file /export/home/~ dpwr            18
user1d/data/ui500n~ dof              0
b/chung/WJC-XIX-16~ dm             nnn
 -distillation.fid  dmm              c
   ACQUISITION      dmf            200
sfrq       500.075  dseq
tn              H1  dres           1.0
at           4.096  homo             n
np           65536        DEC2
sw          8000.0  dfrq2            0
fb        not used  dn2
bs              16  dpwr2            1
tpwr            55  dof2             0
pw             7.7  dm2              n
d1          10.000  dmm2             c
tof              0  dmf2           200
nt              16  dseq2
ct              16  dres2          1.0
alock            n  homo2            n
gain      not used      PROCESSING
      FLAGS          lb            0.30
il               n  wtfile
in               n  proc             ft
dp               y  fn         not used
hs              nn  math              f
     DISPLAY
sp          -250.1  werr
wp          5500.7  wexp
vs              34  wbs
sc               0  wnt
wc             250
hzmm         22.00
is          454.00
rfl         5132.8
rfp         3630.5
th               4
ins          3.000
ai    ph
```



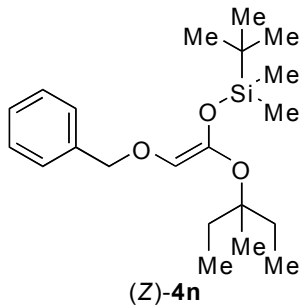

(*Z*)-**4l**


02/15/2007, chung, SED, WJC-XIX-34 after Kugelrohr
exp1 s2pul
SAMPLE
date Feb 15 2007
solvent CDCl3
file /export/home/~
user1d/data/ui500n~
b/chung/WJC-XIX-34~
-Kugelrohr.fid
ACQUISITION
sfrq 500.075
tn H1
at 4.096
np 65536
sw 8000.0
fb 4000
bs 16
tpwr 55
pw 7.7
d1 10.000
tof 0
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.1
wp 5500.7
vs 25
sc 0
wc 250
hzmm 22.00
is 374.48
rfl 5132.6
rfp 3630.5
th 7
ins 1.000
ai ph
DEC. & VT
dfrq 500.075
dn H1
dpwr 18
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
PROCESSING
lb 0.30
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
Me
Me
Me
Me
Si
Me
O
O
O
Me
Me
Me
Me
(Z)-4m
10
9
8
7
6
5
4
3
2
1
ppm
4.75
1.20
1.00
0.12
0.27
2.01
0.12
1.00
2.42
13.15
9.34
0.71
5.90

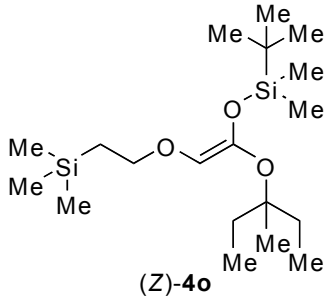
```
07/12/2005, chung, SED, WJC-XII-28 after
 Kugelrohr

exp1  std1h

      SAMPLE            DEC. & VT
date   Jul 12 2005  dn               H1
solvent      CDCl3  dof              0
file /export/home/~ dm             nnn
user1d/data/ui500n~ dmm              c
b/chung/WJC-XII-29~ dmf            200
    -Kugelrohr.fid  dpwr            20
   ACQUISITION          PROCESSING
sfrq       399.951  lb           0.30
tn              H1  wtfile
at           4.096  proc           ft
np           65536  fn       not used
sw          8000.0  math           f
fb            4400
bs              16  werr
tpwr            63  wexp
pw             7.3  wbs
d1               0  wnt
tof         -425.7       DISPLAY
nt             100  sp         -200.0
ct              19  wp         4399.4
alock            n  vs            151
gain      not used  sc              0
      FLAGS         wc            250
il               n  hzmm         17.60
in               n  is          548.50
dp               y  rfl         4903.2
hs              nn  rfp         2903.6
                    th              20
                    ins          2.000
                    nm    ph
```




(*Z*)-**4n**

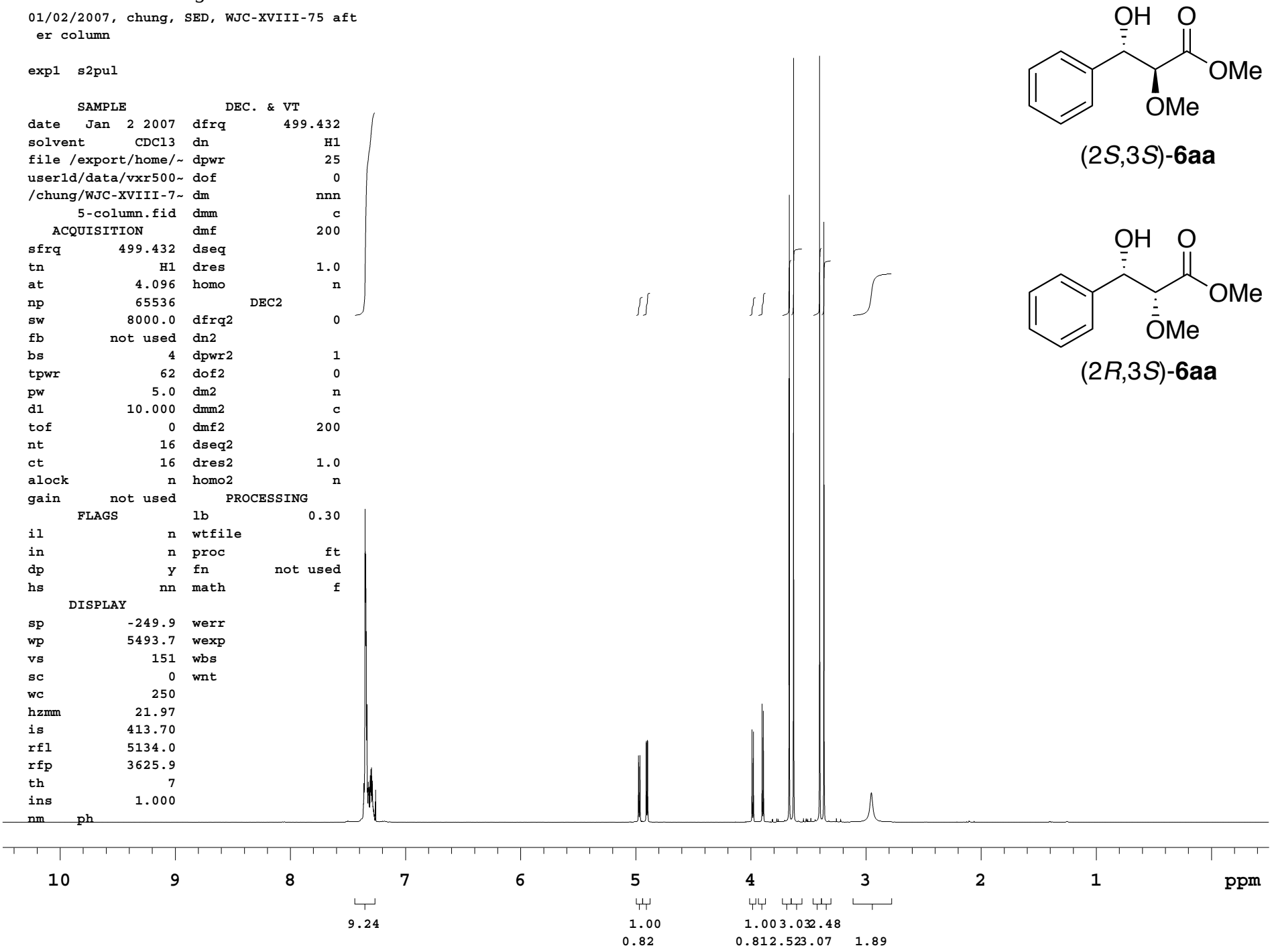



(*Z*)-**4o**

```
08/10/2005, chung, SED, WJC-XII-53 after
 Kugelrohr

exp1  s2pul

      SAMPLE            DEC. & VT
date   Aug 10 2005  dfrq       500.075
solvent      CDCl3  dn               H1
file /export/home/~ dpwr             18
user1d/data/ui500n~ dof               0
b/chung/WJC-XII-53~ dm              nnn
    -Kugelrohr.fid  dmm               c
   ACQUISITION      dmf             200
sfrq       500.075  dseq
tn              H1  dres            1.0
at           4.096  homo              n
np           65536        DEC2
sw          8000.0  dfrq2             0
fb            4000  dn2
bs              16  dpwr2             1
tpwr            55  dof2              0
pw             7.6  dm2               n
d1               0  dmm2              c
tof              0  dmf2            200
nt             100  dseq2
ct              29  dres2           1.0
alock            n  homo2             n
gain      not used      PROCESSING
      FLAGS          lb             0.30
il               n  wtfile
in               n  proc              ft
dp               y  fn         not used
hs              nn  math               f
     DISPLAY
sp          -250.1  werr
wp          5500.7  wexp
vs              43  wbs
sc               0  wnt
wc             250
hzmm         22.00
is          588.85
rfl         5132.8
rfp         3630.5
th               7
ins          6.000
ai    ph
```







(2*S*,3*S*)-**6aa**

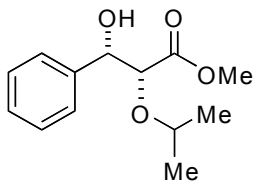



(2*R*,3*S*)-**6aa**

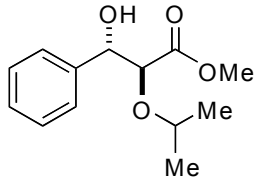
12/18/2006, chung, SED, WJC-XVIII-69 after column

exp1 s2pul

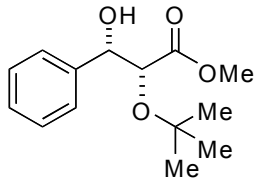
| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Dec 18 2006 | dfrq | 500.075 |
| solvent | CDCl3 | dn | H1 |
| file | /export/home/~ | dpwr | 18 |
| user1d/data/ui500n~ | | dof | 0 |
| b/chung/WJC-XVIII-~ | | dm | nnn |
| 69-column.fid | | dmm | c |
| ACQUISITION | | dmf | 200 |
| sfrq | 500.075 | dseq | |
| tn | H1 | dres | 1.0 |
| at | 4.096 | homo | n |
| np | 65536 | DEC2 | |
| sw | 8000.0 | dfrq2 | 0 |
| fb | not used | dn2 | |
| bs | 16 | dpwr2 | 1 |
| tpwr | 55 | dof2 | 0 |
| pw | 7.7 | dm2 | n |
| d1 | 10.000 | dmm2 | c |
| tof | 0 | dmf2 | 200 |
| nt | 16 | dseq2 | |
| ct | 16 | dres2 | 1.0 |
| alock | n | homo2 | n |
| gain | not used | PROCESSING | |
| FLAGS | | lb | 0.30 |
| il | n | wtfile | |
| in | n | proc | ft |
| dp | y | fn | not used |
| hs | nn | math | f |
| DISPLAY | | | |
| sp | -250.1 | werr | |
| wp | 5500.7 | wexp | |
| vs | 60 | wbs | |
| sc | 0 | wnt | |
| wc | 250 | | |
| hzmm | 22.00 | | |
| is | 1096.00 | | |
| rfl | 5133.5 | | |
| rfp | 3630.5 | | |
| th | 7 | | |
| ins | 1.000 | | |
| ai | ph | | |




(2*R*,3*S*)-**6ba**




(2*S*,3*S*)-**6ba**




(2*R*,3*S*)-**6ca**

```
12/18/2006, chung, SED, WJC-XVIII-65 aft
er column

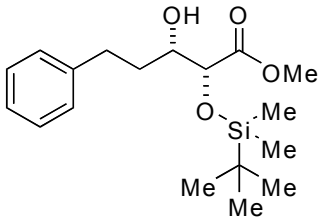
exp1  s2pul

      SAMPLE            DEC. & VT
date   Dec 18 2006  dfrq       500.075
solvent      CDCl3  dn               H1
file /export/home/~ dpwr             18
user1d/data/ui500n~ dof               0
b/chung/WJC-XVIII-~ dm              nnn
     65-column.fid  dmm               c
   ACQUISITION      dmf             200
sfrq       500.075  dseq
tn              H1  dres            1.0
at           4.096  homo              n
np           65536        DEC2
sw          8000.0  dfrq2             0
fb        not used  dn2
bs              16  dpwr2             1
tpwr            55  dof2              0
pw             7.7  dm2               n
d1          10.000  dmm2              c
tof              0  dmf2            200
nt              16  dseq2
ct              16  dres2           1.0
alock            n  homo2             n
gain      not used      PROCESSING
      FLAGS          lb            0.30
il               n  wtfile
in               n  proc              ft
dp               y  fn         not used
hs              nn  math              f
     DISPLAY
sp          -250.1  werr
wp          5500.7  wexp
vs              15  wbs
sc               0  wnt
wc             250
hzmm         22.00
is          382.87
rfl         5133.5
rfp         3630.5
th               7
ins          1.000
ai    ph
```

```
07/02/2007, chung, SED, WJC-XIX-26 after
 column

exp1  s2pul

      SAMPLE            DEC. & VT
date   Jul  2 2007  dfrq       500.070
solvent      CDCl3  dn               H1
file /export/home/~ dpwr             18
user1d/data/ui500n~ dof               0
b/chung/WJC-XIX-26~ dm              nnn
       -column.fid  dmm               c
   ACQUISITION      dmf             200
sfrq       500.070  dseq
tn               H1  dres            1.0
at            4.096  homo              n
np            65536        DEC2
sw           8000.0  dfrq2             0
fb             4000  dn2
bs               16  dpwr2             1
tpwr             55  dof2              0
pw              7.7  dm2               n
d1           10.000  dmm2              c
tof               0  dmf2            200
nt               16  dseq2
ct               16  dres2           1.0
alock             n  homo2             n
gain      not used     PROCESSING
      FLAGS          lb             0.30
il                n  wtfile
in                n  proc              ft
dp                y  fn         not used
hs               nn  math              f
     DISPLAY
sp           -250.1  werr
wp           5500.7  wexp
vs               23  wbs
sc                0  wnt
wc              250
hzmm          22.00
is           318.69
rfl         5140.6
rfp         3630.5
th                7
ins           1.000
ai    ph
```

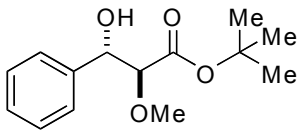



(2*R*,3*S*)-**6ej**

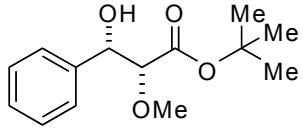
```
01/21/2007, chung, SED, WJC-XVIII-91 aft
er column

exp1  s2pul

      SAMPLE            DEC. & VT
date   Jan 21 2007  dfrq       500.075
solvent      CDCl3  dn               H1
file /export/home/~ dpwr             18
user1d/data/ui500n~ dof               0
b/chung/WJC-XVIII-~ dm              nnn
     91-column.fid  dmm               c
   ACQUISITION      dmf             200
sfrq       500.075  dseq
tn               H1  dres            1.0
at            4.096  homo              n
np            65536        DEC2
sw           8000.0  dfrq2             0
fb             4000  dn2
bs               16  dpwr2             1
tpwr             55  dof2              0
pw              7.7  dm2               n
d1           10.000  dmm2              c
tof               0  dmf2            200
nt               16  dseq2
ct               16  dres2           1.0
alock             n  homo2             n
gain      not used     PROCESSING
      FLAGS          lb             0.30
il                n  wtfile
in                n  proc              ft
dp                y  fn         not used
hs               nn  math              f
     DISPLAY
sp           -250.1  werr
wp           5500.7  wexp
vs               11  wbs
sc                0  wnt
wc              250
hzmm          22.00
is           255.08
rfl         5132.8
rfp         3630.5
th                1
ins           1.000
ai    ph
```




(2*S*,3*S*)-**6fa**

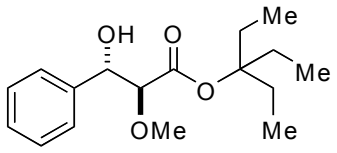



(2*R*,3*S*)-**6fa**

```
07/01/2007, chung, SED, WJC-XIX-19 after
 column

exp1  s2pul

      SAMPLE            DEC. & VT
date   Jul  1 2007  dfrq       500.070
solvent      CDCl3  dn               H1
file /export/home/~ dpwr             18
user1d/data/ui500n~ dof               0
b/chung/WJC-XIX-19~ dm              nnn
       -column.fid  dmm               c
   ACQUISITION      dmf             200
sfrq       500.070  dseq
tn               H1  dres            1.0
at             4.096  homo             n
np             65536        DEC2
sw            8000.0  dfrq2            0
fb              4000  dn2
bs                16  dpwr2            1
tpwr              55  dof2             0
pw               7.7  dm2              n
d1            10.000  dmm2             c
tof                0  dmf2           200
nt                16  dseq2
ct                16  dres2          1.0
alock              n  homo2            n
gain      not used     PROCESSING
      FLAGS          lb             0.30
il                 n  wtfile
in                 n  proc             ft
dp                 y  fn         not used
hs                nn  math              f
     DISPLAY
sp            -250.1  werr
wp            5500.7  wexp
vs                39  wbs
sc                 0  wnt
wc               250
hzmm          22.00
is            278.22
rfl           5140.9
rfp           3630.5
th                 7
ins           1.000
ai    ph
```

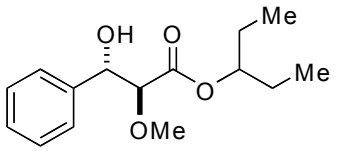



(2*S*,3*S*)-**6ia**

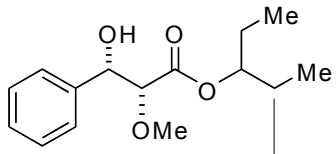
```
07/01/2007, chung, SED, WJC-XIX-29 after
 column

exp1  s2pul

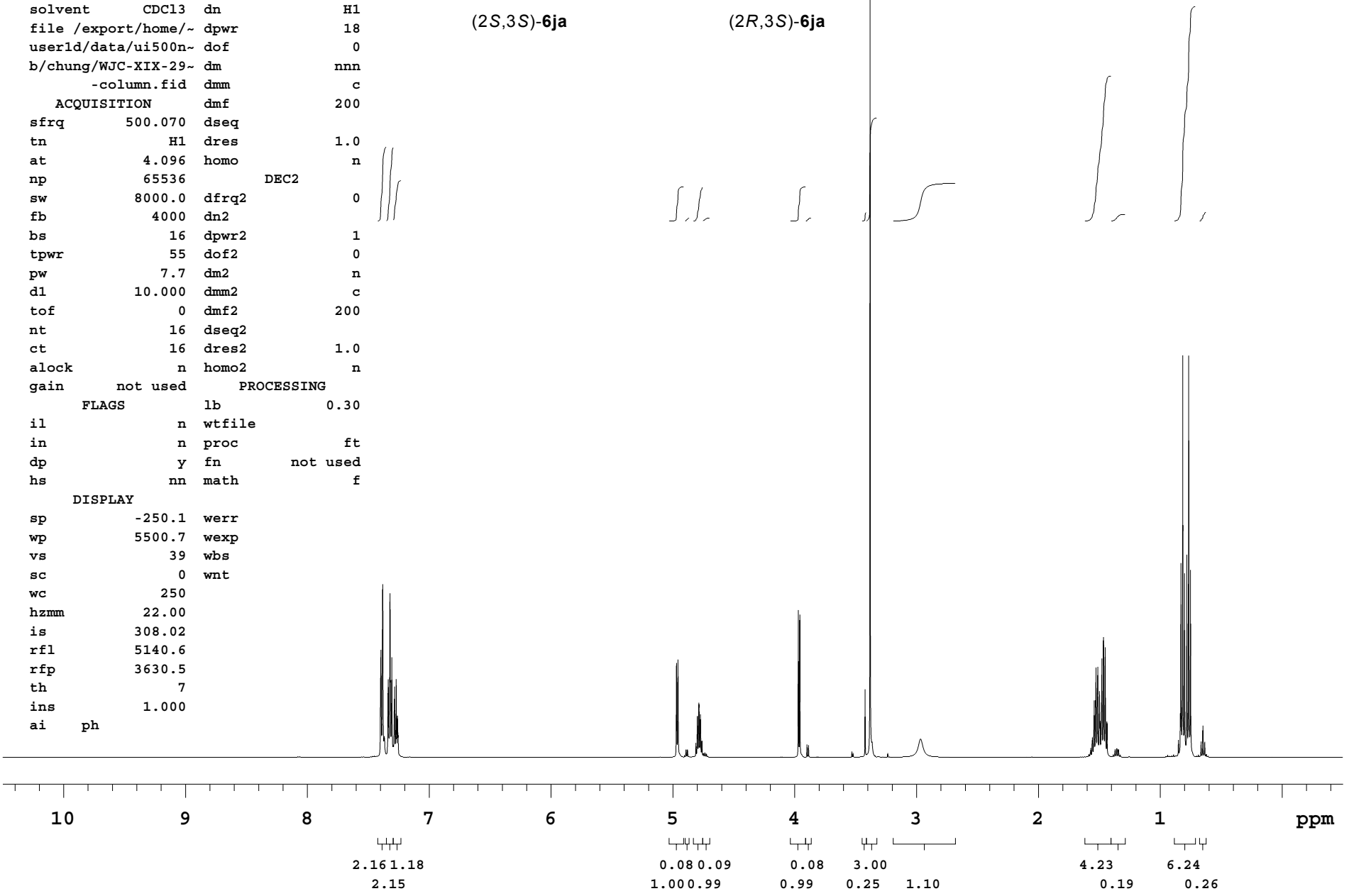
      SAMPLE            DEC. & VT
date   Jul  1 2007  dfrq       500.070
solvent      CDCl3  dn              H1
file /export/home/~ dpwr            18
user1d/data/ui500n~ dof              0
b/chung/WJC-XIX-29~ dm             nnn
       -column.fid  dmm              c
   ACQUISITION      dmf            200
sfrq       500.070  dseq
tn              H1  dres           1.0
at           4.096  homo             n
np           65536        DEC2
sw          8000.0  dfrq2            0
fb            4000  dn2
bs              16  dpwr2            1
tpwr            55  dof2             0
pw             7.7  dm2              n
d1          10.000  dmm2             c
tof              0  dmf2           200
nt              16  dseq2
ct              16  dres2          1.0
alock            n  homo2            n
gain      not used     PROCESSING
      FLAGS          lb            0.30
il               n  wtfile
in               n  proc             ft
dp               y  fn        not used
hs              nn  math             f
     DISPLAY
sp          -250.1  werr
wp          5500.7  wexp
vs              39  wbs
sc               0  wnt
wc             250
hzmm         22.00
is          308.02
rfl         5140.6
rfp         3630.5
th               7
ins          1.000
ai    ph
```




(2*S*,3*S*)-**6ja**



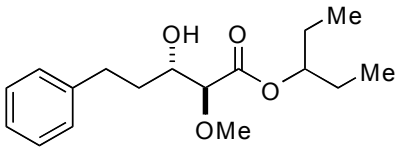

(2*R*,3*S*)-**6ja**



```
04/15/2007, chung, SED, WJC-XIX-41 after
 column

exp1  s2pul

      SAMPLE            DEC. & VT
date   Apr 15 2007  dfrq       500.070
solvent      CDCl3  dn               H1
file /export/home/~ dpwr             18
user1d/data/ui500n~ dof               0
b/chung/WJC-XIX-41~ dm              nnn
       -column.fid  dmm               c
   ACQUISITION      dmf             200
sfrq       500.070  dseq
tn               H1  dres            1.0
at            4.096  homo              n
np            65536        DEC2
sw           8000.0  dfrq2             0
fb             4000  dn2
bs               16  dpwr2             1
tpwr             55  dof2              0
pw              7.7  dm2               n
d1           10.000  dmm2              c
tof               0  dmf2            200
nt               16  dseq2
ct               16  dres2          1.0
alock             n  homo2            n
gain      not used     PROCESSING
      FLAGS          lb            0.30
il                n  wtfile
in                n  proc             ft
dp                y  fn         not used
hs               nn  math              f
     DISPLAY
sp           -250.1  werr
wp           5500.7  wexp
vs               26  wbs
sc                0  wnt
wc              250
hzmm          22.00
is           309.33
rfl         5141.1
rfp         3630.5
th               28
ins           1.000
ai    ph
```

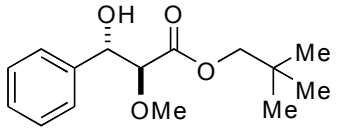



(2*S*,3*S*)-**6jj**

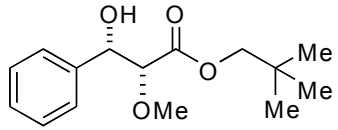
```
07/04/2007, chung, SED, WJC-XIX-17 after
 column

exp1  s2pul

      SAMPLE            DEC. & VT
date   Jul  4 2007  dfrq       500.070
solvent      CDCl3  dn              H1
file /export/home/~ dpwr            18
user1d/data/ui500n~ dof              0
b/chung/WJC-XIX-17~ dm             nnn
       -column.fid  dmm              c
   ACQUISITION      dmf            200
sfrq       500.070  dseq
tn              H1  dres           1.0
at           4.096  homo             n
np           65536        DEC2
sw          8000.0  dfrq2            0
fb            4000  dn2
bs              16  dpwr2            1
tpwr            55  dof2             0
pw             7.7  dm2              n
d1          10.000  dmm2             c
tof              0  dmf2           200
nt              16  dseq2
ct              16  dres2          1.0
alock            n  homo2            n
gain      not used     PROCESSING
      FLAGS          lb            0.30
il               n  wtfile
in               n  proc             ft
dp               y  fn         not used
hs              nn  math              f
     DISPLAY
sp          -250.1  werr
wp          5500.7  wexp
vs              18  wbs
sc               0  wnt
wc             250
hzmm         22.00
is          224.64
rfl         5140.6
rfp         3630.5
th              44
ins          1.000
ai    ph
```




(2*S*,3*S*)-**6ka**

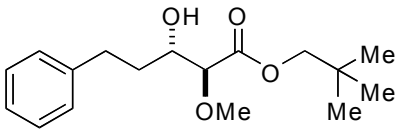



(2*R*,3*S*)-**6ka**

```
06/09/2007, chung, SED, WJC-XIX-24 after
 column

exp1  s2pul

      SAMPLE            DEC. & VT
date   Jun  9 2007  dfrq       500.070
solvent      CDCl3  dn              H1
file /export/home/~ dpwr            18
user1d/data/ui500n~ dof              0
b/chung/WJC-XIX-24~ dm             nnn
       -column.fid  dmm              c
   ACQUISITION      dmf            200
sfrq       500.070  dseq
tn              H1  dres           1.0
at           4.096  homo             n
np           65536        DEC2
sw          8000.0  dfrq2            0
fb            4000  dn2
bs              16  dpwr2            1
tpwr            55  dof2             0
pw             7.7  dm2              n
d1          10.000  dmm2             c
tof              0  dmf2           200
nt              16  dseq2
ct              16  dres2          1.0
alock            n  homo2            n
gain      not used     PROCESSING
      FLAGS          lb            0.30
il               n  wtfile
in               n  proc             ft
dp               y  fn         not used
hs              nn  math              f
     DISPLAY
sp          -250.1  werr
wp          5500.7  wexp
vs              16  wbs
sc               0  wnt
wc             250
hzmm         22.00
is          304.91
rfl         5141.1
rfp         3630.5
th               7
ins          3.000
ai    ph
```

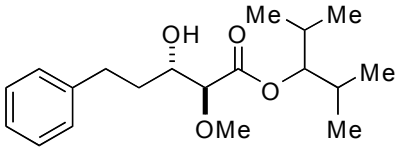



(2*S*,3*S*)-**6kj**

```
06/09/2007, chung, SED, WJC-XIX-39 after
 column

exp1  s2pul

      SAMPLE            DEC. & VT
date   Jun  9 2007  dfrq       500.070
solvent      CDCl3  dn              H1
file /export/home/~ dpwr            18
user1d/data/ui500n~ dof              0
b/chung/WJC-XIX-39~ dm             nnn
       -column.fid  dmm              c
   ACQUISITION      dmf            200
sfrq       500.070  dseq
tn              H1  dres           1.0
at           4.096  homo             n
np           65536        DEC2
sw          8000.0  dfrq2            0
fb            4000  dn2
bs              16  dpwr2            1
tpwr            55  dof2             0
pw             7.7  dm2              n
d1          10.000  dmm2             c
tof              0  dmf2           200
nt              16  dseq2
ct              16  dres2          1.0
alock            n  homo2            n
gain      not used     PROCESSING
      FLAGS          lb            0.30
il               n  wtfile
in               n  proc             ft
dp               y  fn           131072
hs              nn  math              f
     DISPLAY
sp          -250.1  werr
wp          5500.7  wexp
vs              43  wbs
sc               0  wnt
wc             250
hzmm         22.00
is          249.05
rfl         5141.1
rfp         3630.5
th              12
ins          1.000
ai    ph
```

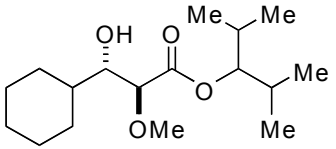



(2*S*,3*S*)-**6lj**

```
10/10/2005, chung, SED, WJC-XIII-66 afte
r column

exp1  s2pul

      SAMPLE            DEC. & VT
date   Oct 10 2005  dfrq       500.075
solvent      CDCl3  dn               H1
file /export/home/~ dpwr             18
user1d/data/ui500n~ dof               0
b/chung/WJC-XIII-6~ dm              nnn
      6-column.fid  dmm               c
   ACQUISITION      dmf             200
sfrq       500.075  dseq
tn               H1  dres            1.0
at            4.096  homo              n
np            65536  temp           25.0
sw           8000.0        DEC2
fb             4000  dfrq2             0
bs               16  dn2
tpwr             55  dpwr2             1
pw              7.6  dof2              0
d1                0  dm2               n
tof               0  dmm2              c
nt              100  dmf2            200
ct               33  dseq2
alock             n  dres2           1.0
gain      not used  homo2             n
      FLAGS             PROCESSING
il                n  lb             0.30
in                n  wtfile
dp                y  proc             ft
hs               nn  fn         not used
     DISPLAY        math              f
sp          -250.1
wp          5500.7  werr
vs              28  wexp
sc               0  wbs
wc             250  wnt
hzmm         22.00
is          387.46
rfl         5132.8
rfp         3630.5
th                7
ins           3.000
ai    ph
```

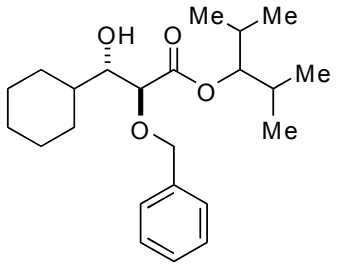



(2*S*,3*S*)-**6lo**

```
10/06/2005, chung, SED, WJC-XIII-64 afte
r column

exp1  s2pul

      SAMPLE            DEC. & VT
date   Oct  6 2005  dfrq       500.075
solvent      CDCl3  dn               H1
file /export/home/~ dpwr             18
user1d/data/ui500n~ dof               0
b/chung/WJC-XIII-6~ dm              nnn
      4-column.fid  dmm               c
   ACQUISITION      dmf             200
sfrq       500.075  dseq
tn               H1  dres            1.0
at            4.096  homo              n
np            65536        DEC2
sw           8000.0  dfrq2             0
fb             4000  dn2
bs               16  dpwr2             1
tpwr             55  dof2              0
pw              7.6  dm2               n
d1                0  dmm2              c
tof               0  dmf2            200
nt              100  dseq2
ct               34  dres2          1.0
alock             n  homo2            n
gain      not used     PROCESSING
      FLAGS          lb             0.30
il                n  wtfile
in                n  proc             ft
dp                y  fn         not used
hs               nn  math              f
     DISPLAY
sp           -250.1  werr
wp           5500.7  wexp
vs               83  wbs
sc                0  wnt
wc              250
hzmm          22.00
is           343.45
rfl          5133.1
rfp          3630.5
th                7
ins           1.000
ai    ph
```

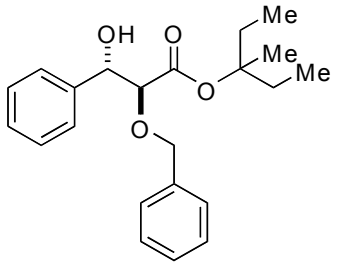



(2*S*,3*S*)-**6mo**

```
07/19/2005, chung, SED, WJC-XII-37 after
 column

exp1  s2pul

      SAMPLE            DEC. & VT
date   Jul 19 2005  dfrq       500.075
solvent      CDCl3  dn               H1
file /export/home/~ dpwr             18
user1d/data/ui500n~ dof               0
b/chung/WJC-XII-37~ dm              nnn
       -column.fid  dmm               c
   ACQUISITION      dmf             200
sfrq       500.075  dseq
tn              H1  dres            1.0
at           4.096  homo              n
np           65536        DEC2
sw          8000.0  dfrq2             0
fb            4000  dn2
bs              16  dpwr2             1
tpwr            55  dof2              0
pw             7.6  dm2               n
d1               0  dmm2              c
tof              0  dmf2            200
nt             100  dseq2
ct              27  dres2          1.0
alock            n  homo2             n
gain      not used     PROCESSING
      FLAGS          lb            0.30
il               n  wtfile
in               n  proc              ft
dp               y  fn          not used
hs              nn  math              f
     DISPLAY
sp          -250.1  werr
wp          5500.7  wexp
vs              51  wbs
sc               0  wnt
wc             250
hzmm         22.00
is          510.41
rfl         5132.8
rfp         3630.5
th               7
ins          1.000
ai    ph
```

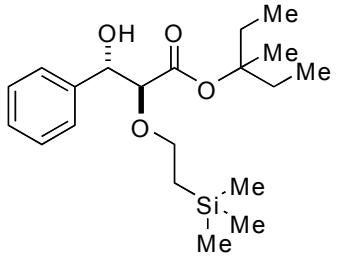



(2*S*,3*S*)-**6na**

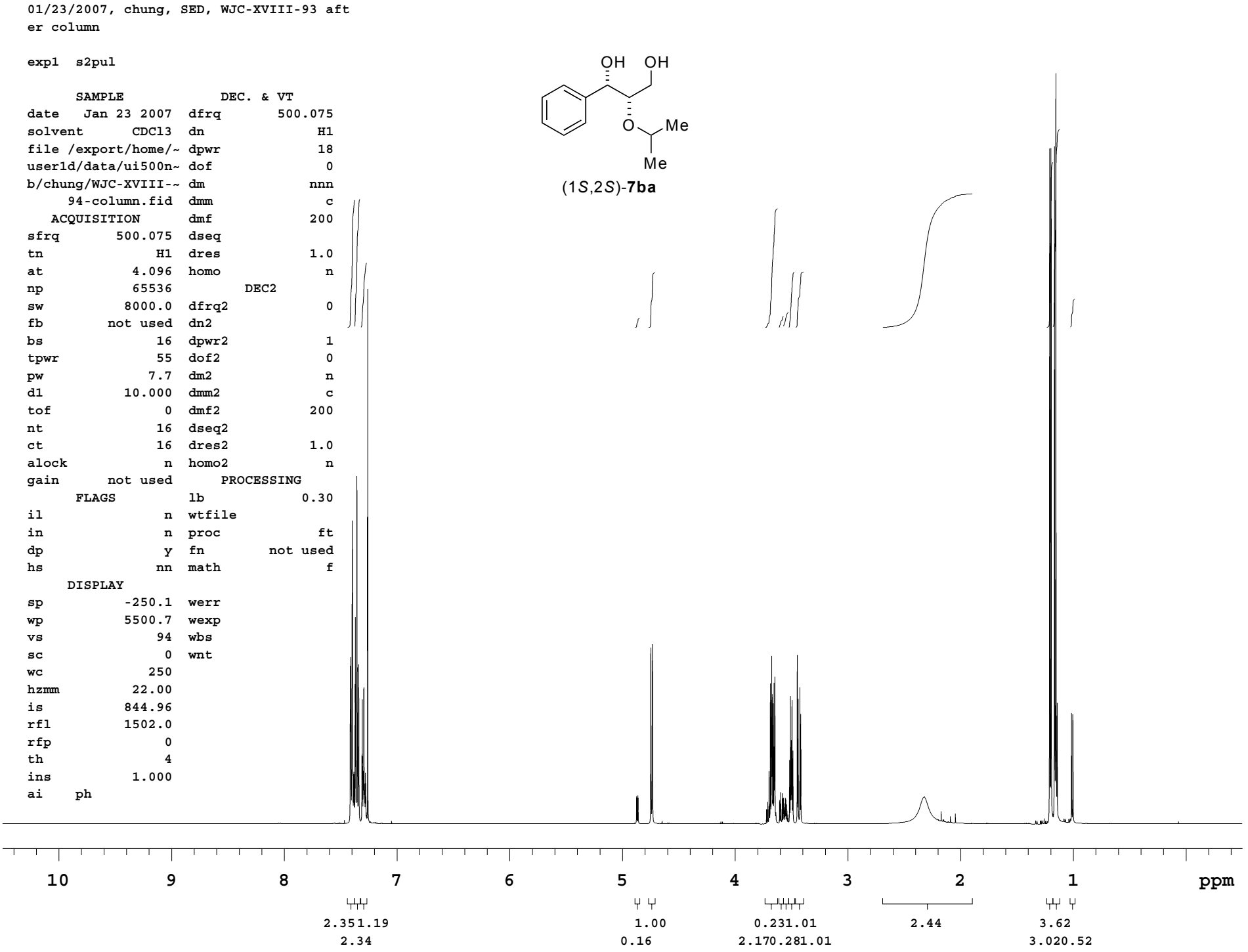
```
08/16/2005, chung, SED, WJC-XII-60 after
 column

exp1  s2pul

      SAMPLE            DEC. & VT
date   Aug 16 2005  dfrq       500.075
solvent      CDCl3  dn               H1
file /export/home/~ dpwr             18
user1d/data/ui500n~ dof               0
b/chung/WJC-XII-60~ dm              nnn
       -column.fid  dmm               c
   ACQUISITION      dmf             200
sfrq       500.075  dseq
tn               H1  dres           1.0
at            4.096  homo             n
np            65536        DEC2
sw           8000.0  dfrq2            0
fb             4000  dn2
bs               16  dpwr2            1
tpwr             55  dof2             0
pw              7.6  dm2              n
d1                0  dmm2             c
tof               0  dmf2           200
nt              100  dseq2
ct               33  dres2          1.0
alock             n  homo2            n
gain      not used     PROCESSING
      FLAGS          lb            0.30
il                n  wtfile
in                n  proc             ft
dp                y  fn        not used
hs               nn  math              f
     DISPLAY
sp           -250.1  werr
wp           5500.7  wexp
vs               17  wbs
sc                0  wnt
wc              250
hzmm          22.00
is           277.58
rfl          5132.3
rfp          3630.5
th                7
ins           1.000
ai    ph
```



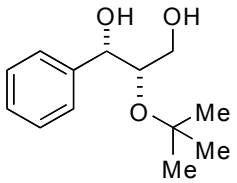

(2*S*,3*S*)-**6oa**


01/23/2007, chung, SED, WJC-XVIII-93 after column
exp1 s2pul
SAMPLE
date Jan 23 2007
solvent CDCl3
file /export/home/~
user1d/data/ui500n~
b/chung/WJC-XVIII-~
94-column.fid
ACQUISITION
sfrq 500.075
tn H1
at 4.096
np 65536
sw 8000.0
fb not used
bs 16
tpwr 55
pw 7.7
d1 10.000
tof 0
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.1
wp 5500.7
vs 94
sc 0
wc 250
hzmm 22.00
is 844.96
rfl 1502.0
rfp 0
th 4
ins 1.000
ai ph
DEC. & VT
dfrq 500.075
dn H1
dpwr 18
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
PROCESSING
lb 0.30
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
OH
OH
O
Me
Me
(1S,2S)-7ba
10
9
8
7
6
5
4
3
2
1
ppm
2.351.19
2.34
1.00
0.16
0.231.01
2.170.281.01
2.44
3.62
3.020.52

```
01/23/2007, chung, SED, WJC-XVIII-93 aft
er column

exp1  s2pul

      SAMPLE            DEC. & VT
date   Jan 23 2007  dfrq       500.075
solvent      CDCl3  dn               H1
file /export/home/~ dpwr             18
user1d/data/ui500n~ dof               0
b/chung/WJC-XVIII-~ dm              nnn
     93-column.fid  dmm               c
   ACQUISITION      dmf             200
sfrq       500.075  dseq
tn               H1  dres            1.0
at             4.096  homo              n
np             65536        DEC2
sw            8000.0  dfrq2             0
fb         not used  dn2
bs               16  dpwr2             1
tpwr             55  dof2              0
pw              7.7  dm2               n
d1           10.000  dmm2              c
tof               0  dmf2            200
nt               16  dseq2
ct               16  dres2           1.0
alock             n  homo2             n
gain       not used     PROCESSING
      FLAGS          lb             0.30
il                n  wtfile
in                n  proc              ft
dp                y  fn         not used
hs               nn  math              f
     DISPLAY
sp           -250.1  werr
wp           5500.7  wexp
vs               27  wbs
sc                0  wnt
wc              250
hzmm          22.00
is           513.75
rfl          5132.6
rfp          3630.5
th                4
ins           1.000
ai    ph
```

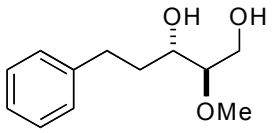



(1*S*,2*S*)-**7ca**

```
06/25/2007, chung, SED, WJC-XX-87 after
column

exp1  s2pul

      SAMPLE            DEC. & VT
date   Jun 25 2007  dfrq       500.070
solvent      CDCl3  dn               H1
file /export/home/~ dpwr             18
user1d/data/ui500n~ dof               0
b/chung/WJC-XX-87-~ dm              nnn
        column.fid  dmm               c
   ACQUISITION      dmf             200
sfrq       500.070  dseq
tn               H1  dres            1.0
at            4.096  homo              n
np            65536        DEC2
sw           8000.0  dfrq2             0
fb             4000  dn2
bs               16  dpwr2             1
tpwr             55  dof2              0
pw              7.7  dm2               n
d1            4.000  dmm2              c
tof               0  dmf2            200
nt               16  dseq2
ct               16  dres2          1.0
alock             n  homo2             n
gain      not used     PROCESSING
      FLAGS          lb             0.30
il                n  wtfile
in                n  proc              ft
dp                y  fn         not used
hs               nn  math              f
     DISPLAY
sp           -250.1  werr
wp           5500.7  wexp
vs               42  wbs
sc                0  wnt
wc              250
hzmm          22.00
is           625.49
rfl          5140.6
rfp          3630.5
th                7
ins           1.000
ai    ph
```

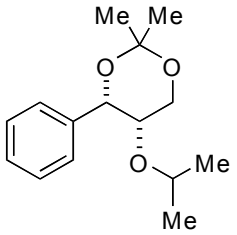



(2*R*,3*S*)-**7kj**

```
07/22/2007, chung, SED, WJC-XIX-9 after
column

exp1  s2pul

      SAMPLE            DEC. & VT
date   Jul 22 2007  dfrq       500.070
solvent      CDCl3  dn              H1
file /export/home/~ dpwr            18
user1d/data/ui500n~ dof              0
b/chung/WJC-XIX-9-~ dm             nnn
        column.fid  dmm              c
   ACQUISITION      dmf            200
sfrq       500.070  dseq
tn              H1  dres           1.0
at           4.096  homo             n
np           65536        DEC2
sw          8000.0  dfrq2            0
fb            4000  dn2
bs              16  dpwr2            1
tpwr            55  dof2             0
pw             7.7  dm2              n
d1          10.000  dmm2             c
tof              0  dmf2           200
nt              16  dseq2
ct              16  dres2          1.0
alock            n  homo2            n
gain      not used      PROCESSING
      FLAGS         lb            0.30
il               n  wtfile
in               n  proc             ft
dp               y  fn         not used
hs              nn  math              f
     DISPLAY
sp          -250.1  werr
wp          5500.7  wexp
vs              60  wbs
sc               0  wnt
wc             250
hzmm         22.00
is          358.15
rfl         5140.9
rfp         3630.5
th               7
ins          1.000
ai    ph
```

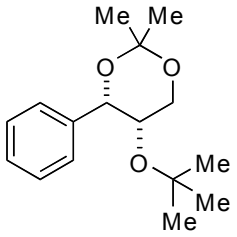



(4*S*,5*S*)-**8ba**

```
07/19/2007, chung, SED, WJC-XIX-8 after
column

exp1  s2pul

      SAMPLE            DEC. & VT
date   Jul 19 2007  dfrq       500.070
solvent      CDCl3  dn               H1
file /export/home/~ dpwr             18
user1d/data/ui500n~ dof               0
b/chung/WJC-XIX-8-~ dm              nnn
        column.fid  dmm               c
   ACQUISITION      dmf             200
sfrq       500.070  dseq
tn               H1  dres            1.0
at            4.096  homo              n
np            65536        DEC2
sw           8000.0  dfrq2             0
fb             4000  dn2
bs               16  dpwr2             1
tpwr             55  dof2              0
pw              7.7  dm2               n
d1           10.000  dmm2              c
tof               0  dmf2            200
nt               16  dseq2
ct               16  dres2           1.0
alock             n  homo2             n
gain      not used     PROCESSING
      FLAGS          lb             0.30
il                n  wtfile
in                n  proc              ft
dp                y  fn         not used
hs               nn  math              f
     DISPLAY
sp           -250.1  werr
wp           5500.7  wexp
vs               15  wbs
sc                0  wnt
wc              250
hzmm          22.00
is           201.63
rfl          5140.6
rfp          3630.5
th               44
ins           1.000
ai    ph
```

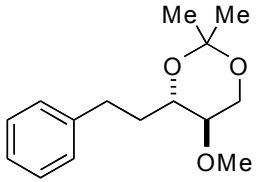



(4*S*,5*S*)-**8ca**

```
08/30/2007, chung, SED, WJC-XX-96 after
column

exp1  s2pul

      SAMPLE            DEC. & VT
date   Aug 30 2007  dfrq       500.070
solvent      CDCl3  dn              H1
file /export/home/~ dpwr            18
user1d/data/ui500n~ dof              0
b/chung/WJC-XX-96-~ dm             nnn
        column.fid  dmm              c
   ACQUISITION      dmf            200
sfrq       500.070  dseq
tn              H1  dres           1.0
at           4.096  homo             n
np           65536        DEC2
sw          8000.0  dfrq2            0
fb            4000  dn2
bs              16  dpwr2            1
tpwr            55  dof2             0
pw             7.7  dm2              n
d1          10.000  dmm2             c
tof              0  dmf2           200
nt              16  dseq2
ct              16  dres2          1.0
alock            n  homo2            n
gain      not used     PROCESSING
      FLAGS         lb            0.30
il               n  wtfile
in               n  proc             ft
dp               y  fn         not used
hs              nn  math              f
     DISPLAY
sp          -250.1  werr
wp          5500.7  wexp
vs              38  wbs
sc               0  wnt
wc             250
hzmm         22.00
is          303.11
rfl         5140.6
rfp         3630.5
th              50
ins          1.000
ai    ph
```

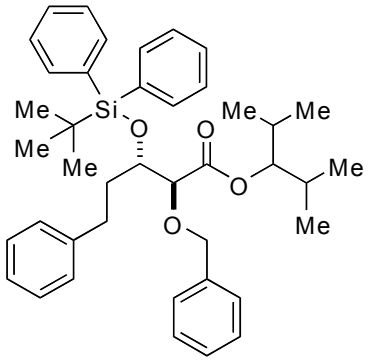



(4*S*,5*R*)-**8kj**

```
03/27/2007, chung, SED, WJC-XIX-70 after
 column

exp1  s2pul

      SAMPLE            DEC. & VT
date   Mar 27 2007  dfrq       500.070
solvent      CDCl3  dn              H1
file /export/home/~ dpwr            18
user1d/data/ui500n~ dof              0
b/chung/WJC-XIX-70~ dm             nnn
       -column.fid  dmm              c
   ACQUISITION      dmf            200
sfrq       500.070  dseq
tn              H1  dres           1.0
at           4.096  homo             n
np           65536        DEC2
sw          8000.0  dfrq2            0
fb            4000  dn2
bs              16  dpwr2            1
tpwr            55  dof2             0
pw             7.7  dm2              n
d1          10.000  dmm2             c
tof              0  dmf2           200
nt              16  dseq2
ct              16  dres2          1.0
alock            n  homo2            n
gain      not used     PROCESSING
      FLAGS          lb            0.30
il               n  wtfile
in               n  proc             ft
dp               y  fn         not used
hs              nn  math              f
     DISPLAY
sp          -250.1  werr
wp          5500.7  wexp
vs              37  wbs
sc               0  wnt
wc             250
hzmm         22.00
is          492.05
rfl         5140.9
rfp         3630.5
th              32
ins          1.000
ai    ph
```

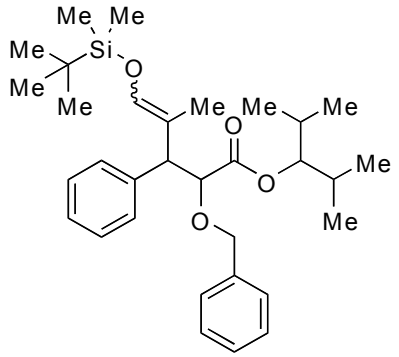



(2*S*,3*S*)-**12**

```
02/28/2007, chung, SED, WJC-XIX-47 after
 column A

exp1  s2pul

      SAMPLE            DEC. & VT
date   Feb 28 2007  dfrq       500.075
solvent      CDCl3  dn              H1
file /export/home/~ dpwr            18
user1d/data/ui500n~ dof              0
b/chung/WJC-XIX-47~ dm            nnn
      -columnA.fid  dmm             c
   ACQUISITION      dmf           200
sfrq       500.075  dseq
tn              H1  dres          1.0
at           4.096  homo            n
np           65536        DEC2
sw          8000.0  dfrq2            0
fb            4000  dn2
bs              16  dpwr2            1
tpwr            55  dof2             0
pw             7.7  dm2              n
d1          10.000  dmm2             c
tof              0  dmf2           200
nt              16  dseq2
ct              16  dres2         1.0
alock            n  homo2           n
gain      not used     PROCESSING
      FLAGS         lb           0.30
il               n  wtfile
in               n  proc            ft
dp               y  fn        not used
hs              nn  math             f
     DISPLAY
sp          -250.1  werr
wp          5500.7  wexp
vs              37  wbs
sc               0  wnt
wc             250
hzmm         22.00
is          831.74
rfl         5132.8
rfp         3630.5
th               7
ins          1.000
ai    ph
```

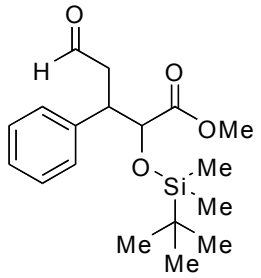


**15**

```
12/07/2006, chung, SED, WJC-XVIII-59 aft
er column A

exp1  s2pul

      SAMPLE            DEC. & VT
date   Dec  7 2006  dfrq       500.075
solvent      CDCl3  dn              H1
file /export/home/~ dpwr            18
user1d/data/ui500n~ dof              0
b/chung/WJC-XVIII-~ dm             nnn
    59-columnA.fid  dmm              c
   ACQUISITION      dmf            200
sfrq       500.075  dseq
tn              H1  dres           1.0
at           4.096  homo             n
np           65536        DEC2
sw          8000.0  dfrq2            0
fb        not used  dn2
bs              16  dpwr2            1
tpwr            55  dof2             0
pw             7.7  dm2              n
d1          10.000  dmm2             c
tof              0  dmf2           200
nt              16  dseq2
ct              16  dres2          1.0
alock            n  homo2            n
gain      not used      PROCESSING
      FLAGS          lb            0.30
il               n  wtfile
in               n  proc             ft
dp               y  fn         not used
hs              nn  math             f
     DISPLAY
sp          -250.1  werr
wp          5500.7  wexp
vs              43  wbs
sc               0  wnt
wc             250
hzmm         22.00
is          686.76
rfl         5133.5
rfp         3630.5
th               4
ins          1.000
ai    ph
```

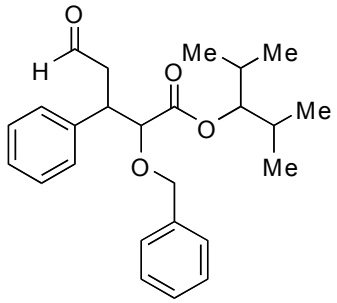



**16ep**

```
04/17/2007, chung, SED, WJC-XIX-35 after
 column A

exp1  s2pul

      SAMPLE            DEC. & VT
date   Apr 17 2007  dfrq       500.070
solvent      CDCl3  dn              H1
file /export/home/~ dpwr            18
user1d/data/ui500n~ dof              0
b/chung/WJC-XIX-35~ dm             nnn
    -columnA-2.fid  dmm              c
   ACQUISITION      dmf            200
sfrq       500.070  dseq
tn              H1  dres           1.0
at           4.096  homo             n
np           65536        DEC2
sw          8000.0  dfrq2            0
fb            4000  dn2
bs              16  dpwr2            1
tpwr            55  dof2             0
pw             7.7  dm2              n
d1          10.000  dmm2             c
tof              0  dmf2           200
nt              16  dseq2
ct              16  dres2          1.0
alock            n  homo2            n
gain      not used     PROCESSING
      FLAGS          lb            0.30
il               n  wtfile
in               n  proc             ft
dp               y  fn         not used
hs              nn  math             f
     DISPLAY
sp          -250.1  werr
wp          5500.7  wexp
vs             117  wbs
sc               0  wnt
wc             250
hzmm         22.00
is          459.49
rfl         5140.9
rfp         3630.5
th               7
ins          1.000
ai    ph
```

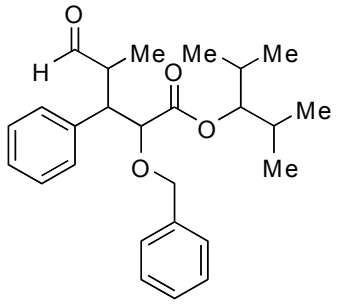



**16mp**

```
02/28/2007, chung, SED, WJC-XIX-47 after
 column C

exp1  s2pul

      SAMPLE            DEC. & VT
date   Feb 28 2007  dfrq       500.075
solvent      CDCl3  dn              H1
file /export/home/~ dpwr            18
user1d/data/ui500n~ dof              0
b/chung/WJC-XIX-47~ dm             nnn
      -columnC.fid  dmm              c
   ACQUISITION      dmf            200
sfrq       500.075  dseq
tn              H1  dres           1.0
at           4.096  homo             n
np           65536        DEC2
sw          8000.0  dfrq2            0
fb            4000  dn2
bs              16  dpwr2            1
tpwr            55  dof2             0
pw             7.7  dm2              n
d1          10.000  dmm2             c
tof              0  dmf2           200
nt              16  dseq2
ct              16  dres2          1.0
alock            n  homo2            n
gain      not used     PROCESSING
      FLAGS         lb            0.30
il               n  wtfile
in               n  proc             ft
dp               y  fn         not used
hs              nn  math             f
     DISPLAY
sp          -250.1  werr
wp          5500.7  wexp
vs             127  wbs
sc               0  wnt
wc             250
hzmm         22.00
is          284.96
rfl         5132.8
rfp         3630.5
th               7
ins          1.000
ai    ph
```

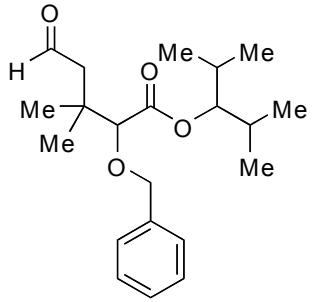



**16mq**

```
07/22/2007, chung, SED, WJC-XIX-37 after
 column A

exp1  s2pul

      SAMPLE            DEC. & VT
date   Jul 22 2007  dfrq       500.070
solvent      CDCl3  dn              H1
file /export/home/~ dpwr            18
user1d/data/ui500n~ dof              0
b/chung/WJC-XIX-37~ dm             nnn
    -columnA-1.fid  dmm              c
   ACQUISITION      dmf            200
sfrq       500.070  dseq
tn              H1  dres           1.0
at           4.096  homo             n
np           65536        DEC2
sw          8000.0  dfrq2            0
fb            4000  dn2
bs              16  dpwr2            1
tpwr            55  dof2             0
pw             7.7  dm2              n
d1          10.000  dmm2             c
tof              0  dmf2           200
nt              16  dseq2
ct              16  dres2          1.0
alock            n  homo2            n
gain      not used     PROCESSING
      FLAGS         lb            0.30
il               n  wtfile
in               n  proc             ft
dp               y  fn         not used
hs              nn  math             f
     DISPLAY
sp          -250.1  werr
wp          5500.7  wexp
vs              62  wbs
sc               0  wnt
wc             250
hzmm         22.00
is          182.85
rfl         5140.6
rfp         3630.5
th               7
ins          1.000
ai    ph
```

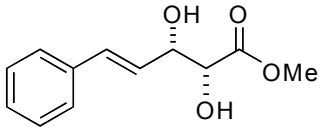



**16mr**

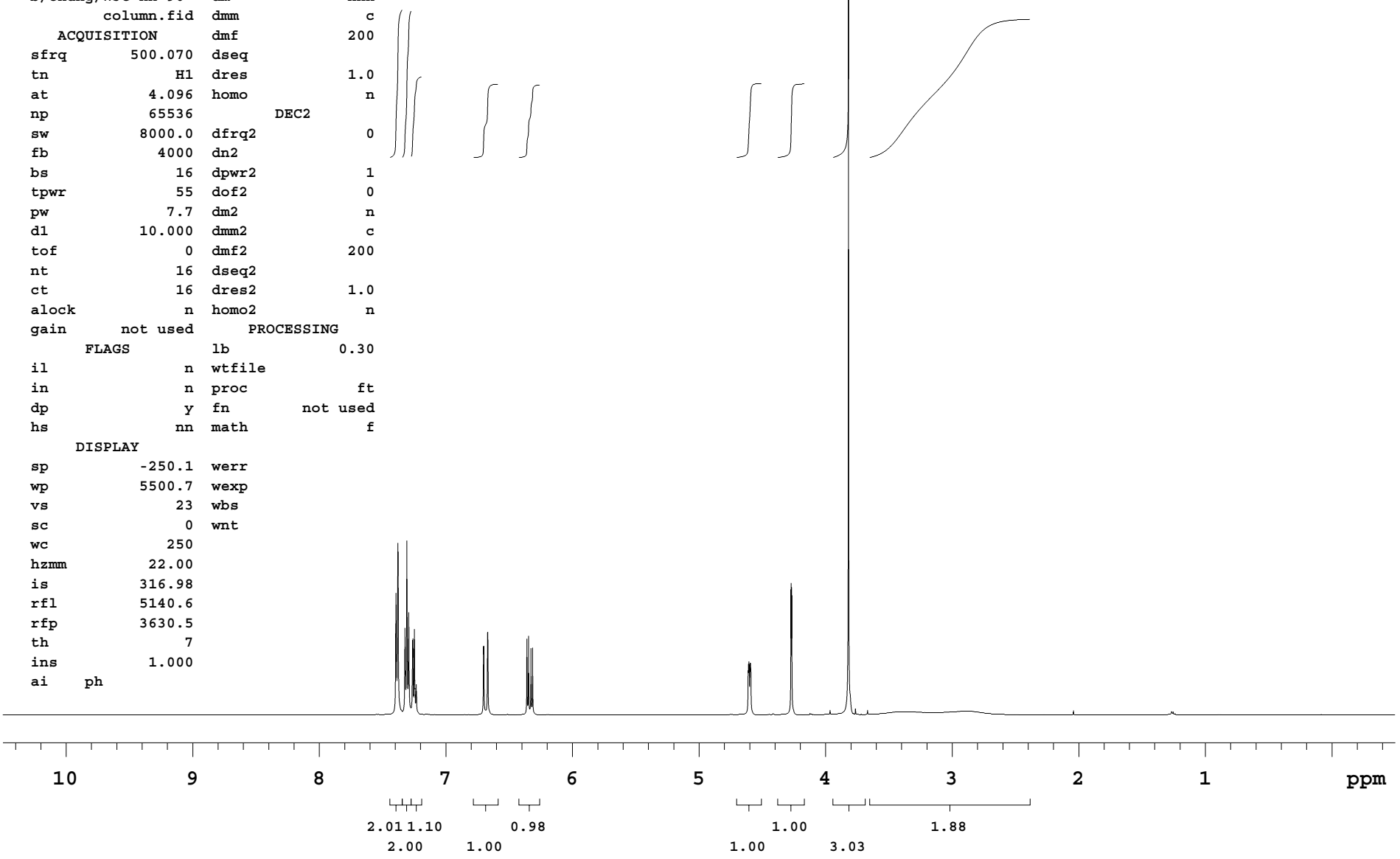
```
06/26/2007, chung, SED, WJC-XX-90 after
column

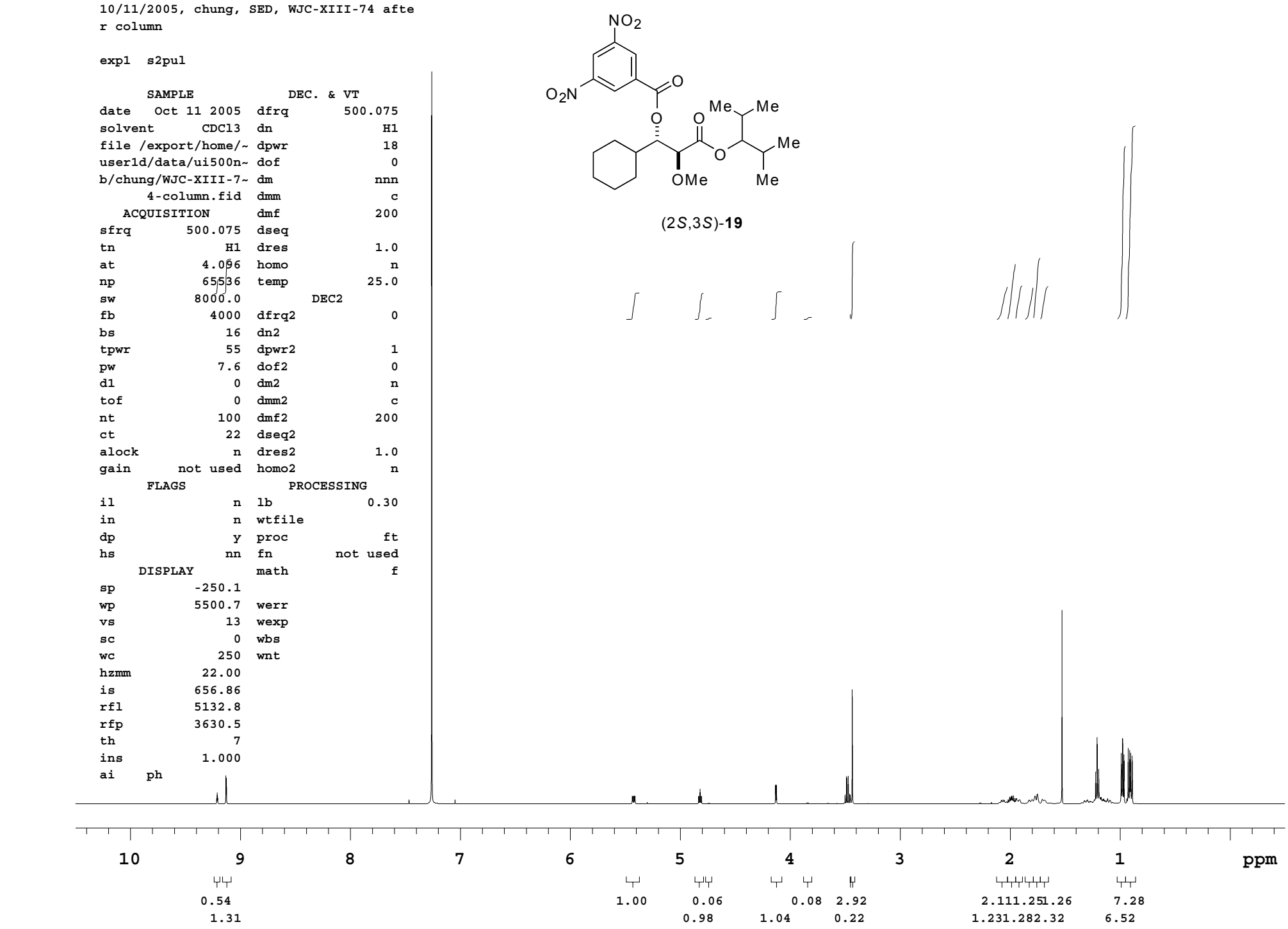
exp1  s2pul

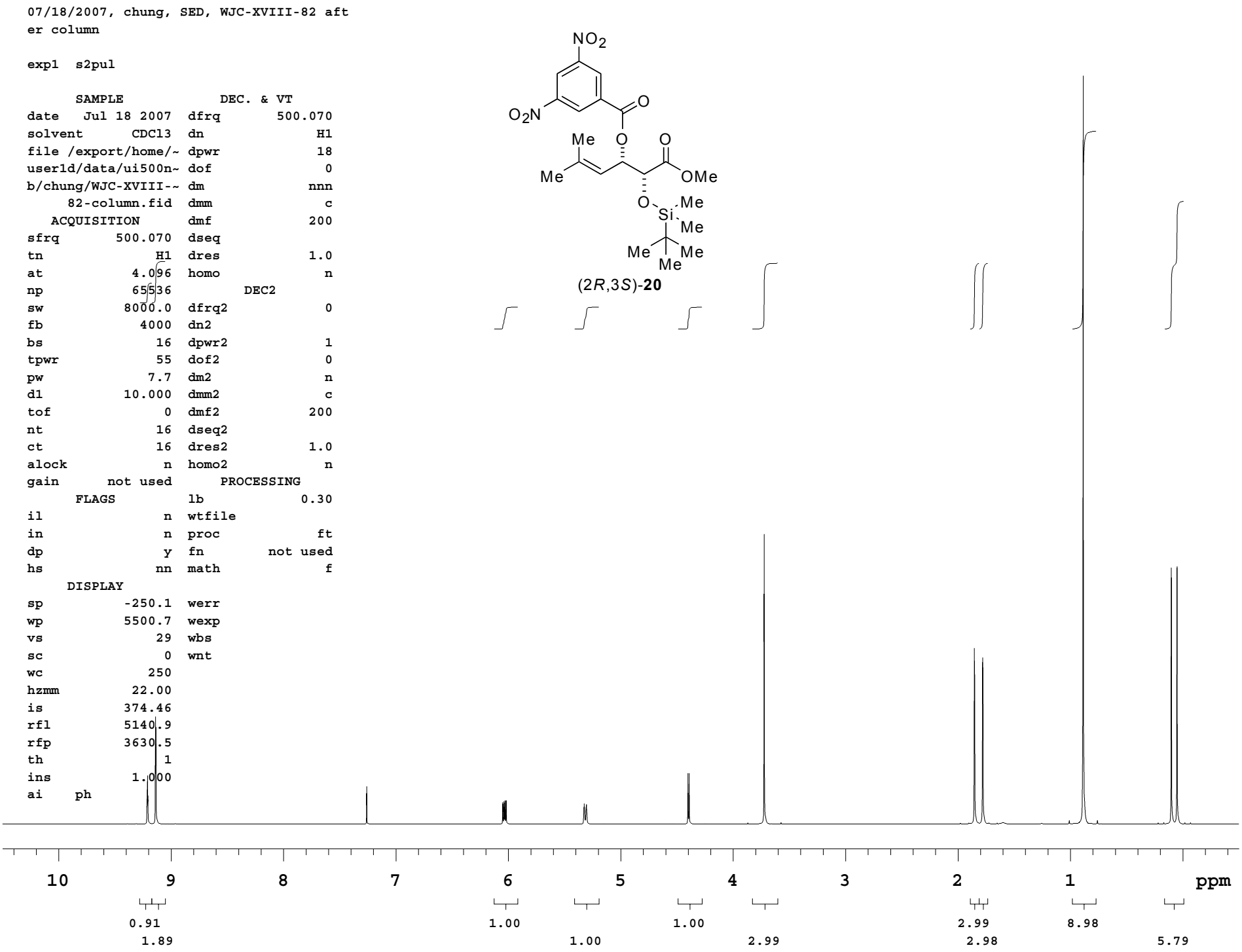
      SAMPLE            DEC. & VT
date   Jun 26 2007  dfrq       500.070
solvent      CDCl3  dn               H1
file /export/home/~ dpwr             18
user1d/data/ui500n~ dof               0
b/chung/WJC-XX-90-~ dm              nnn
        column.fid  dmm               c
   ACQUISITION      dmf             200
sfrq       500.070  dseq
tn               H1  dres            1.0
at            4.096  homo              n
np            65536        DEC2
sw           8000.0  dfrq2             0
fb             4000  dn2
bs               16  dpwr2             1
tpwr             55  dof2              0
pw              7.7  dm2               n
d1           10.000  dmm2              c
tof               0  dmf2            200
nt               16  dseq2
ct               16  dres2          1.0
alock             n  homo2            n
gain      not used     PROCESSING
      FLAGS          lb            0.30
il                n  wtfile
in                n  proc             ft
dp                y  fn         not used
hs               nn  math              f
     DISPLAY
sp           -250.1  werr
wp           5500.7  wexp
vs               23  wbs
sc                0  wnt
wc              250
hzmm          22.00
is           316.98
rfl         5140.6
rfp         3630.5
th                7
ins           1.000
ai    ph
```




(2*R*,3*S*,4*E*)-**17**




10/11/2005, chung, SED, WJC-XIII-74 afte
r column
exp1 s2pul
SAMPLE
date Oct 11 2005
solvent CDCl3
file /export/home/~
user1d/data/ui500n~
b/chung/WJC-XIII-7~
4-column.fid
ACQUISITION
sfrq 500.075
tn H1
at 4.096
np 65536
sw 8000.0
fb 4000
bs 16
tpwr 55
pw 7.6
d1 0
tof 0
nt 100
ct 22
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.1
wp 5500.7
vs 13
sc 0
wc 250
hzmm 22.00
is 656.86
rfl 5132.8
rfp 3630.5
th 7
ins 1.000
ai ph
DEC. & VT
dfrq 500.075
dn H1
dpwr 18
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
PROCESSING
lb 0.30
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
NO2
O2N
O
O
O
O
O
Me
Me
Me
Me
OMe
(2S,3S)-19
10
9
8
7
6
5
4
3
2
1
ppm
0.54
1.31
1.00
0.06
0.98
0.08
1.04
2.92
0.22
2.11
1.23
1.25
1.28
1.26
2.32
7.28
6.52


07/18/2007, chung, SED, WJC-XVIII-82 after column
exp1 s2pul
SAMPLE
date Jul 18 2007
solvent CDCl3
file /export/home/~
user1d/data/ui500n~
b/chung/WJC-XVIII-~
82-column.fid
ACQUISITION
sfrq 500.070
tn H1
at 4.096
np 65536
sw 8000.0
fb 4000
bs 16
tpwr 55
pw 7.7
d1 10.000
tof 0
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.1
wp 5500.7
vs 29
sc 0
wc 250
hzmm 22.00
is 374.46
rfl 5140.9
rfp 3630.5
th 1
ins 1.000
ai ph
DEC. & VT
dfrq 500.070
dn H1
dpwr 18
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
PROCESSING
lb 0.30
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
NO2
O2N
Me
OMe
Si
(2R,3S)-20
10
9
8
7
6
5
4
3
2
1
ppm
0.91
1.89
1.00
1.00
1.00
2.99
2.99
2.98
8.98
5.79