Supporting Information

Fe/Cr- and Co/Cr-Mediated Catalytic Asymmetric 2-Haloallylations of Aldehydes

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General Procedures and Methods: ^1H NMR spectra were recorded on a Varian Inova 500, Varian Mercury 400, or Varian Mercury 300 spectrometer. ^{13}C NMR spectra were recorded at 100 MHz or 125 Mhz. Chemical shifts were reported in parts per million (ppm). The residual solvent peak was used as an internal reference. Fast atom bombardment (FAB) mass spectra were obtained with 3-nitrobenzyl alcohol or glycerol as the matrix. Sodium iodide was added when indicated. Chemical ionization (CI) mass spectra were obtained with ammonia as the reagent gas. Electrospray ionization experiments were performed on Micromass Inc. Platform II Atmospheric Pressure Ionization Mass Spectrometer. Infrared spectra were obtained as films on sodium chloride plates. Analytical thin layer chromatography (TLC) was performed with E. Merck precoated TLC plates, silica gel 60F-254, layer thickness 0.25 mm. Preparative thin layer chromatography (PTLC) separations were performed on E. Merck precoated TLC plates, silica gel 60F-254, layer thickness 0.25 or 0.50 mm. Flash chromatography separations were performed on E. Merck kieselgel 60 (230-400) mesh silica gel.

Reagents and solvents are commercial grade and were used as supplied, with the following exceptions. A bulk THF was purified through Al_2O_3 column (solvent purification system) and distilled over LiAlH_4 . Benzene, ether, dichloromethane were purified by solvent purification systems. CrBr_3 (99%, Cerac), $\text{CrCl}_3 \cdot 3\text{THF}$ (Aldrich), CoPc (Strem), $\text{Fe}(\text{TMHD})_3$ (Aldrich), and Mn powder (99.9%, Aldrich) were purchased from the indicated supplier and used as supplied. All reactions were conducted under nitrogen atmosphere. Reaction vessels were flame-dried or oven-dried and cooled under an inert atmosphere.

Experimental Procedures and Compound data:

2,3-dibromopropene (5a)

2,3-Dibromopropene (>95% purity by ^1H -NMR analysis) was prepared according to the procedure given in Organic Synthesis.^a

3-Bromo-2-iodopropene (5b)

To a stirred solution of NaI (0.467 mol, 1.2 eq) in anhydrous CH_3CN (150mL) at 0 °C was added TMSI (57 mL, 1.2 eq). After 15 min, H_2O (4.2 mL, 0.6 eq) and propargyl alcohol (20 mL) were added into the reaction. This was warmed to RT over 1h and stirred for 4h. The reaction mixture was quenched with aq. NaHCO_3 , and aq. NaHSO_3 . The water phase was extracted with Et_2O . The combined extracts were washed with brine, dried over Na_2SO_4 , and concentrated *in vacuo*. Distillation at 80 °C under vacuum (5 mmHg) gave 2-iodo-prop-2-en-1-ol (b.p. 50 °C/5 mmHg, 34g, 53%). To a stirred solution of NBS (44.1g, 0.252 mol) in CH_2Cl_2 (100 mL) at -25 °C was added Me_2S (20.3 mL, 0.277mmol).^b The reaction mixture was stirred for 20 min and 2-iodo-prop-2-en-1-ol (35g, 0.21 mol, 0.8 eq) in CH_2Cl_2 (20 mL) was added. After 3h at RT, the reaction was diluted with pentane, and poured into ice water. The organic phase was washed with brine, dried over Na_2SO_4 , filtered, and concentrated *in vacuo* to give the crude product. This

was distilled at 70 °C under vacuum (5 mmHg) to give 3-bromo-2-iodo-prene (**5b**) (b.p. 40 °C/5 mmHg, 30g, 58%). Data for **5b**: ¹H-NMR (500 MHz, C₆D₆): 5.78 ppm (1H, m), 5.56 (1H, m), 3.56 (2H, m). ¹³C-NMR (125 MHz, C₆D₆): 128.3 ppm, 95.0, 41.4.

Deuterated 3-bromo-2-iodopropene (1-propene-1-d, 3-bromo-2-iodo, **5d**)

To a stirred solution of propargyl alcohol (10g, 0.178 mol) in CH₂Cl₂ (100 mL) at 0 °C was added imidazole (15.7g, 0.231 mol) and TMSCl (28.1 mL, 0.231 mol). The reaction was warmed to RT and quenched with water. The aqueous phase was extracted with pentane. The extracts were washed with brine, dried over Na₂SO₄, and concentrated *in vacuo*. This was distilled at 40 °C under vacuum (5 mmHg) to give trimethyl-prop-2-ynyloxysilane (13.6 g, 60%). To a stirred solution of NaI (7.03 g, 0.0469 mol) in anhydrous CH₃CN (60 mL) at 0 °C was added TMSCl (5.74 mL, 0.0469 mmol). After 15 min, D₂O (0.427 mL, 0.6 eq) and trimethyl-prop-2-ynyloxysilane (5 g, 0.0391 mol) were added into the reaction. The reaction mixture was warmed to RT over 0.5h and stirred for 4h. The reaction mixture was quenched with aq. NaHCO₃, and aq. NaHSO₃. The water phase was extracted with Et₂O. The combined extracts were washed with brine, dried over Na₂SO₄, and concentrated *in vacuo*. Distillation at 80 °C under vacuum (5 mmHg) gave a 1:3 mixture of 2-iodo-prop-2-en-1-ol and its deuterated form (3.25g, ~45%). To a stirred suspension of NBS (4.33 g, 0.0243 mol) in CH₂Cl₂ (50 mL) at -25 °C was added Me₂S (2.14 mL, 0.0291 mmol). The reaction mixture was stirred for 20 min and 2-iodo-prop-2-en-1-ol (3 g, 0.0162 mmol) in CH₂Cl₂ (10 mL) was added. After 3h at RT, the reaction was diluted with pentane, and poured into ice water. The organic phase was washed with brine, dried over Na₂SO₄, filtered, and concentrated *in vacuo* to give the crude product. This was distilled at 70 °C under vacuum (5 mmHg) to give a 1:3 mixture of 3-bromo-2-iodo-propene (**5b**) and its deuterated form **5d** (2.0 g, ~50%). Data for **5d**: ¹H-NMR (500 MHz, C₆D₆): 5.30 (1H, s), 3.41 (2H, s).

3-Bromo-2-chloropropene (**5c**)

To a stirred solution of 2,3-dichloropropene (10g, 0.090 mol) in DMF (200 mL) was added NaBr (23g, 2.5 eq). The reaction was stirred at 90 °C overnight and quenched with water. The water phase was extracted with pentane (two times). The combined extracts were washed with brine, dried over Na₂SO₄, and concentrated *in vacuo* at 5-10 °C. This was distilled at 120 °C to give 3-bromo-2-chloro-propene (b.p. 110 °C, 8 g, 57%). Data for **5c**: ¹H-NMR (500 MHz, C₆D₆): 4.86 ppm (1H, s), 4.80 (1H, s), 3.36 (2H, m). ¹³C-NMR (125 MHz, C₆D₆): 138.0 ppm, 116.4, 34.5.

3-Iodo-2-methylpropene (**2c**)

To a stirred solution of 3-chloro-2-methylpropene (10g, 0.110 mol) in DMF (200 mL) was added NaI (41 g, 0.275 mol). The reaction was stirred at 90 °C overnight and quenched with water. The aqueous phase was extracted with pentane (two times). The combined extracts were washed with water, brine, dried over Na₂SO₄, and concentrated *in vacuo* at 5 °C. The residue was distilled at 90 °C to give 3-iodo-2-methylpropene (9.9 g, 50%). Data for **2c**: ¹H-NMR (400 MHz, C₆D₆): 4.81 ppm (1H, s), 4.56 (1H, s), 3.39 (2H, m).

N-[2-(4-*tert*-Butyl-4,5-dihydro-oxazol-2-yl)-phenyl]-*C*-phenyl-methanesulfonamide (**1d**)

To a stirred solution of 2-aminobenzonitrile (2.0g, 0.017 mol) in PhCl (40 mL) was added (*S*)-*tert*-leucinol (2.2g, 0.019 mol) and ZnCl₂ (2.6g, 0.019 mol). The reaction mixture was heated at 130 °C for 36h and cooled to RT. The reaction was quenched with water and ethylenediamine and stirred for 30 min. The water phase was extracted with EtOAc (thrice). The combined extracts were washed with brine, dried over Na₂SO₄, and concentrated *in vacuo*. Purification by silica gel chromatography (10:1, hexanes:EtOAc) gave 2-(4-*tert*-butyl-4,5-dihydro-oxazol-2-yl)-phenylamine (3.2g, 88%). This was recrystallized from hexanes to give fine needles (2.0g). To a stirred solution of 2-(4-*tert*-butyl-4,5-dihydro-oxazol-2-yl)-phenylamine (2.0g, 9.17 mmol) in CH₂Cl₂ (20 mL) at 0 °C was added Et₃N (1.9 mL, 14.6 mmol) and α-toluenesulfonyl chloride (2.5g, 14.6 mmol). The reaction mixture was stirred for 1h and quenched with aq. NaHCO₃. The aqueous phase was extracted with EtOAc. The combined extracts were washed with brine, dried over Na₂SO₄, and concentrated *in vacuo*. Purification by silica gel chromatography (8:1:1, hexanes:EtOAc:CH₂Cl₂) gave *N*-[2-(4-*tert*-butyl-4,5-dihydro-oxazol-2-yl)-phenyl]-*C*-phenyl-methanesulfonamide (**1d**, 2.73g, 80%). This was recrystallized from EtOAc to afford fine needles (2.3g). Data for **1d**: [α]_D + 10.3 ° (*c* 1.0, CHCl₃, at 25 °C), ¹H-NMR (500 MHz, CDCl₃) 12.2 ppm (1H, brs), 7.79 (1H, dd, *J* = 7.5, 1.5 Hz), 7.69 (1H, d, *J* = 9.0 Hz), 7.41 (1H, m), 7.25 (3H, m), 7.15 (2H, m), 7.07 (1H, t, *J* = 7.5 Hz), 4.38 (2H, ABq, *J* = 14.5 Hz), 4.24 (1H, dd, *J* = 10.0, 8.5 Hz), 4.10 (1H, t, *J* = 8.5 Hz), 3.87 (1H, dd, *J* = 10.0, 8.5 Hz), 0.79 (9H, s). ¹³C-NMR (125 MHz, CDCl₃): 163.2 ppm, 140.1, 132.8, 180.9, 129.7, 128.8, 128.7, 122.3, 116.9, 113.1, 75.7, 67.9, 58.1, 33.8, 26.0.

(*S*)-*N*-[2-(4-*tert*-Butyl-4,5-dihydro-oxazol-2-yl)-4-methyl-phenyl]-*C*-phenyl-methanesulfonamide (1e**)**

2-Iodo-4-methylphenylamine was synthesized according to the literature procedure.⁶ To a stirred solution of 2-iodo-4-methylphenylamine (7.4g, 0.0319 mol) in DMF (40 mL) was added CuCN (4.29 g, 0.0479 mol). The reaction was heated at 130 °C for 3h and cooled to rt. The reaction was quenched with aq. NaCN solution and stirred for 1h. The aqueous phase was extracted with EtOAc. The combined extracts were washed with brine, dried over Na₂SO₄, and concentrated *in vacuo*. Purification by silica gel chromatography (10:1, hexanes:EtOAc) gave 2-amino-5-methylbenzonitrile (3.39g, 80%). To a stirred solution of 2-amino-5-methylbenzonitrile (2.25g, 0.017 mol) in PhCl (40 mL) was added (*S*)-*tert*-leucinol (2.2g, 0.019 mol) and ZnCl₂ (2.6g, 0.019 mol). The reaction mixture was heated at 130 °C for 36h and cooled to RT. The reaction was quenched with water and ethylenediamine and stirred for 30 min. The water phase was extracted with EtOAc (thrice). The combined extracts were washed with brine, dried over Na₂SO₄, and concentrated *in vacuo*. Purification by silica gel chromatography (10:1, hexanes:EtOAc) gave 2-(4-*tert*-butyl-4,5-dihydro-oxazol-2-yl)-4-methylphenylamine (3.2g, 88%). To a stirred solution of 2-(4-*tert*-butyl-4,5-dihydro-oxazol-2-yl)-4-methylphenylamine (2.12g, 9.17 mmol) in CH₂Cl₂ (20 mL) at 0 °C was added Et₃N (1.9 mL, 14.6 mmol) and α-toluenesulfonyl chloride (2.5g, 14.6 mmol). The reaction mixture was stirred for 1h and quenched with aq. NaHCO₃. The aqueous phase was extracted with EtOAc. The combined extracts were washed with brine, dried over Na₂SO₄, and concentrated *in vacuo*. Purification by silica gel chromatography (8:1:1,

hexanes:EtOAc:CH₂Cl₂) gave (*S*)-*N*-[2-(4-*tert*-Butyl-4,5-dihydro-oxazol-2-yl)-4-methyl-phenyl]-*C*-phenyl-methanesulfonamide (**1e**, 2.83g, 80%). Data for **1e**: [α]_D + 14.0 ° (*c* 1.0, CHCl₃, at 25 °C), ¹H-NMR (500 MHz, CDCl₃) 12.1 ppm (1H, brs), 7.62-7.15 (8H, m), 4.34 (2H, ABq, *J* = 13.5 Hz), 4.26 (1H, t-like), 4.10 (1H, t-like), 3.86 (1H, dd, *J* = 10.0, 8.5 Hz), 2.33 (3H, s), 0.79 (9H, s). ¹³C-NMR (125 MHz, CDCl₃): 163.0 ppm, 133.6, 132.0, 130.9, 130.0, 129.1, 128.8, 128.7, 117.3, 75.7, 67.9, 57.9, 33.8, 26.0, 20.8.

Representative procedures for 2-bromo- and 2-iodoallylations of aldehydes

(*R*)-2-Bromo-7-(*tert*-butyl-diphenyl-silanyloxy)-hept-1-en-4-ol (**7b**)

2-Bromoallylation with 10 mol% of the Ligand-Cr Complex (Procedure A). The ligand (**1d**) (66 mg, 0.171 mmol), CrBr₃ (46 mg, 0.155 mmol), iron tris(2,2,6,6-tetramethyl-3,5-heptanedionate) [Fe(TMHD)₃] (20.7 mg, 0.031 mmol), Mn (511mg, 9.3 mmol) were placed in a round-bottomed flask. THF (6 mL) was added. To a suspension of the reaction mixture was added NEt₃ (47 μL, 1.42 mmol). The reaction mixture was stirred vigorously for 1.5h (the color of solution turned into green). At RT, 2,6-lutidine (410 μL, 3.41 mmol) and 2,3-dibromopropene (**5a**) (410 μL, 3.5 mmol) was added. After 5 min at RT, the reaction was cooled to 0 °C and stirred for 10 min. 4-(*tert*-Butyldiphenylsilanyloxy)-butyraldehyde (**6b**) (505 mg, 1.55 mmol) in THF (0.5 mL) and TMSCl (1.7 mL, 4.65 mmol) were added. The reaction mixture was stirred for 20h at 0 °C and diluted with Et₂O. The precipitates were filtered through a SiO₂ pad (eluted with Et₂O). The filtrate was evaporated *in vacuo* to give the TMS protected homoallylic alcohol. This was dissolved in THF(10 mL) and 70% AcOH (20 mL) was added. The reaction mixture was stirred for 1h and quenched with NaHCO₃. The aqueous phase was extracted with Et₂O (thrice) and the combined extracts were dried over Na₂SO₄. All volatiles were evaporated *in vacuo* to give the crude homoallylic alcohol. Purification by silica gel chromatography (15:1 to 10:1, hexanes: EtOAc) afforded 2-bromo-7-(*tert*-butyldiphenyl-silanyloxy)-hept-1-en-4-ol (**7b**, 520 mg, 75 %). Data for **7b**: ¹H-NMR (500 MHz, CDCl₃): 7.69-7.43 ppm (10H, m), 5.70 (1H, s), 5.53 (1H, s), 4.00 (1H, m), 3.72 (2H, m), 2.55 (2H, m), 1.72 (3H, m), 1.56 (1H, m), 1.06 (9H, s). ¹³C-NMR (125 MHz, CDCl₃): 135.6 ppm, 135.5, 133.6, 130.8, 129.6, 127.7, 119.4, 68.9, 64.0, 49.3, 33.3, 28.6, 26.8, 19.1. The *ee* of **7b** was determined to be 92% by HPLC analysis (Daicel Chiralcel OD-H (0.46 cm φ x 25 cm; hexanes/^tBuOH=100/1 with flow rate 1.0 mL/min and a UV detector at 245 nm) (*S*)-enantiomer: *t*_R=12.3 min, (*R*)-enantiomer: *t*_R=15.2 min) as well as ¹H-NMR analysis of its (+)-Mosher ester.

Determination of the absolute configuration of 2-bromo-7-(*tert*-butyldiphenylsilanyloxy)-hept-1-en-4-ol

Enantiomerically pure (*R*)-2-bromo-7-(*tert*-butyldiphenylsilanyloxy)-hept-1-en-4-ol was synthesized from the optically pure epoxide as illustrated below. The absolute configuration of the compound synthesized by the catalytic asymmetric 2-bromoallylation with the ligand **1d** was established by the comparison of the retention time of HPLC.

(*R*)-2-Bromo-7-(*tert*-butyldimethylsilyloxy)-hept-1-en-4-ol (7c)

Data for **7c**: $^1\text{H-NMR}$ (500 MHz, CDCl_3): 5.69 ppm (1H, s), 5.51 (1H, s), 3.96 (1H, m), 3.68 (2H, m), 2.59 (1H, m), 2.51 (1H, m), 1.68 (3H, m), 1.52 (1H, m), 0.90 (9H, s), 0.06 (6H, s). $^{13}\text{C-NMR}$ (125 MHz, CDCl_3): 131.2 ppm, 119.4, 69.1, 63.6, 49.5, 34.0, 29.2, 26.2, 18.6. The *ee* of **7c** was determined to be 91% by $^1\text{H-NMR}$ analysis of its (+)-Mosher ester.

(*R*)-2-Bromo-7,7-bisethysulfanyl-hept-1-en-4-ol (7d)

Data for **7d**: $^1\text{H-NMR}$ (500 MHz, CDCl_3): 5.70 ppm (1H, s), 5.54 (1H, s), 3.96 (1H, m), 3.82 (1H, t, $J = 6.5$ Hz), 2.7-2.5 (4H, m), 2.05 (1H, m), 1.92 (1H, m), 1.8-1.6 (4H, m), 1.26 (6H, t, $J = 6.5$ Hz). $^{13}\text{C-NMR}$ (125 MHz, CDCl_3): 130.7 ppm, 120.1, 68.9, 51.4, 49.7, 34.3, 32.4, 24.5, 24.4, 14.7. The *ee* of **7d** was determined to be 90% by $^1\text{H-NMR}$ analysis of its (+)-Mosher ester.

(*S*)-3-Bromo-1-cyclohexylbut-3-en-1-ol (7e)

Data for **7e**: $^1\text{H-NMR}$ (500 MHz, CDCl_3): 5.70 ppm (1H, s), 5.54 (1H, s), 3.70 (1H, m), 2.62 (1H, m), 2.49 (1H, m), 1.8-1.4 (11H, m). $^{13}\text{C-NMR}$ (125 MHz, CDCl_3): 131.8 ppm, 119.8, 73.1, 46.7, 43.1, 29.3, 28.1, 26.7, 26.5, 26.3. The *ee* of **7e** was determined to be 89% by $^1\text{H-NMR}$ analysis of its (+)-Mosher ester.

(*S*)-2-Bromonona-1,5-dien-4-ol (7f)

Data for **7f**: $^1\text{H-NMR}$ (500 MHz, CDCl_3): 5.75 ppm (1H, m), 5.68 (1H, d, $J = 1.0$ Hz), 5.53 (1H, d, $J = 1.0$ Hz), 5.50 (1H, m), 4.41 (1H, m), 2.61 (2H, m), 2.02 (2H, m), 1.41 (2H, m), 0.89 (3H, t-like). $^{13}\text{C-NMR}$ (125 MHz, CDCl_3): 133.2 ppm, 131.2, 130.4, 120.0, 70.5, 49.6, 34.5, 22.4, 13.9. The *ee* of **7f** was determined to be 87% by $^1\text{H-NMR}$ analysis of its (+)-Mosher ester.

2-Bromoallylation with 5 mol% of the ligand-Cr complex. The ligand (**1d**) (660 mg, 1.71 mmol), CrBr₃ (460 mg, 1.55 mmol), iron tris(2,2,6,6-tetramethyl-3,5-heptanedionate) [Fe(TMHD)₃] (207 mg, 0.31 mmol), Mn (5.11 g, 93.0 mmol) were placed in a round-bottomed flask. THF (60 mL) was added. To a suspension of the reaction mixture was added NEt₃ (470 μ L, 14.2 mmol). The reaction mixture was stirred vigorously for 1.5h. At RT 2,6-lutidine (10 mL, 85.9 mmol) and 2,3-dibromopropene (7.9 mL, 75.8 mmol) were added. After 5 min at RT, the reaction was cooled to 0 °C and stirred for 15 min. 4-(*tert*-Butyldiphenylsilyloxy)-butyraldehyde (9.9 g, 30.3 mmol) in THF (15 mL) and TMSCl (11.1 mL, 90.9 mmol) were added. The reaction mixture was stirred for 36h at 0 °C and diluted with Et₂O. The precipitates were filtered through SiO₂ plug (eluted with Et₂O). The filtrate was evaporated *in vacuo* to give the TMS protected homoallylic alcohol. This was dissolved in THF (45mL) and 70% AcOH (90 mL) was added. The reaction mixture was stirred for 1.5h and quenched with NaHCO₃. The aqueous phase was extracted with Et₂O (thrice) and the combined extracts were dried over Na₂SO₄. All volatiles were evaporated *in vacuo* to give the crude homoallylic alcohol. Purification by silica gel chromatography (15:1 to 10:1, hexanes: EtOAc) afforded 2-bromo-7-(*tert*-butyldiphenylsilyloxy)-hept-1-en-4-ol (**7b**, 9.5 g, 70 %). The *ee* of **7b** was determined to be 92% by ¹H-NMR analysis of its (+)-Mosher ester.

(*R*)-7-(*tert*-Butyldiphenylsilyloxy)-2-iodohept-1-en-4-ol (7j**)**

2-Iodoallylation with 10 mol% of the ligand-Cr complex. The ligand **1d** (236 mg, 0.611 mmol), CrBr₃ (160 mg, 0.548 mmol), [Fe(TMHD)₃] (66.9 mg, 0.110 mmol), Mn (904 mg, 16.5 mmol) were placed in a flask. THF (21 mL) was added. To a stirred suspension of the reaction mixture was added NEt₃ (170 μ L, 1.21 mmol). The reaction mixture was stirred vigorously for 1.5h. At RT, 2,6-lutidine (1.4 mL, 3.50 mmol) and 3-bromo-2-iodopropene (**5b**) (1.4 mL, 3.50 mmol) were added. After 5 min at RT, the reaction was cooled to 0 °C and stirred for 15 min. 4-(*tert*-Butyldiphenylsilyloxy)-butyraldehyde (**6b**) (1.76 g, 5.48 mmol) in THF (2.6 mL) and TMSCl (2.0 mL, 16.3 mmol) were added. The reaction mixture was stirred for 14h at 0 °C and diluted with Et₂O. The precipitates were filtered through a SiO₂ pad (eluted with Et₂O). The filtrate was evaporated *in vacuo* to give the TMS protected homoallylic alcohol. This was dissolved in THF (10 mL) and 70% AcOH (20 mL) was added. The reaction mixture was stirred for 1h and quenched with NaHCO₃. The aqueous phase was extracted with Et₂O (thrice) and the combined extracts were dried over Na₂SO₄. All volatiles were evaporated *in vacuo* to give the crude homoallylic alcohol. Purification by silica gel chromatography (15:1 to 10:1, hexanes: EtOAc) afforded 7-(*tert*-butyldiphenylsilyloxy)-2-iodohept-1-en-4-ol (**7j**, 1.54 g, 57 %). Data for **7j**: ¹H-NMR (500 MHz, C₆D₆): 7.77 ppm (4H, m), 7.24 (6H, m), 5.72 (1H, s), 5.56 (1H, s), 3.78 (1H, m), 3.60 (2H, m), 2.21 (2H, m), 1.63 (1H, m), 1.53 (2H, m), 1.37 (1H, m), 1.17 (9H, s). ¹³C-NMR (100 MHz, CDCl₃): 135.6 ppm, 135.5, 129.6, 128.3, 127.7, 69.7, 64.0, 52.8, 33.0, 28.7, 26.8, 19.2. The *ee* of **7j** was determined to be 93% by HPLC analysis (Daicel Chiralcel OD-H (0.46 cm ϕ x 25 cm; hexanes/ⁱBuOH=100/1 with flow rate=1.0 mL/min and a UV detector at 245 nm; (*S*)-enantiomer: *t*_R=21.0 min and (*R*)-enantiomer: *t*_R = 26.7 min) as well as ¹H-NMR analysis of its (+)-Mosher ester.

Determination of the absolute configuration of 7-(*tert*-butyldiphenylsilyloxy)-2-iodohept-1-en-4-ol

The absolute configuration of the 2-iodohomoallylic alcohol obtained by the catalytic asymmetric reaction with the ligand **3** was established through the HPLC comparison of its (*R*)-Mosher ester with the authentic sample. According to the procedure illustrated below, the authentic 2-iodohomoallylic alcohol was prepared from the optically active alcohol and converted to both (*R*)- and (*S*)-Mosher esters.

Co/Cr Mediated 2-Iodoallylation. The ligand **1d** (50.2 mg, 0.134 mmol), CrBr₃ (35.8 mg, 0.123 mmol), CoPc (14.0 mg, 0.0245 mmol), Mn (202 mg, 3.6 mmol) were placed in a flask. THF (5 mL) was added. To a stirred suspension of the reaction mixture was added NEt₃ (206 μ L, 1.19 M in THF). The reaction mixture was stirred vigorously for 1.5h. At RT, 2,6-lutidine (0.313 mL, 2.70 mmol) and 3-bromo-2-iodopropene (**5b**) (754 mg, 3.10 mmol) were added. After 1 min at RT, the reaction was cooled to 0 °C and stirred for 10 min. 4-(*tert*-Butyldiphenylsilyloxy)-butyraldehyde (**6b**) (400 mg, 1.22 mmol) in THF (1.5 mL) and TMSCl (0.45 mL, 3.68 mmol) were added. The reaction mixture was stirred for 12h at 0 °C and diluted with Et₂O. The precipitates were filtered through a SiO₂ pad (eluted with Et₂O). The filtrate was evaporated *in vacuo* to give the TMS protected homoallylic alcohol. This was dissolved in THF (1.5 mL) and 70% AcOH (3 mL) was added. The reaction mixture was stirred for 1h and quenched with NaHCO₃. The aqueous phase was extracted with Et₂O (thrice) and the combined extracts were dried over Na₂SO₄. All volatiles were evaporated *in vacuo* to give the crude homoallylic alcohol. Purification by silica gel chromatography (15:1 to 10:1, hexanes: EtOAc) afforded 7-(*tert*-butyldiphenylsilyloxy)-2-iodohept-1-en-4-ol (**7j**, 381.9 mg, 63 %). The *ee* of **7j** was determined to be 93% by HPLC analysis (Daicel Chiralcel OD-H (0.46 cm ϕ x 25 cm; hexanes/^tBuOH=100/1 with flow rate=1.0 mL/min and a UV detector at 245 nm; (*S*)-enantiomer: t_R =21.0 min and (*R*)-enantiomer: t_R = 26.7 min) as well as ¹H-NMR analysis of its (+)-Mosher ester.

Representative procedure for the allylation of aldehyde with the ligand-CrCl₃ complex

(*R*)-7-(*tert*-Butyldiphenylsilyloxy)-2-chlorohept-1-en-4-ol (**7l**)

Procedure B. The ligand **1d** (17.9 mg, 0.0478 mmol), $\text{CrCl}_3 \cdot 3\text{THF}$ (13.5 mg, 0.043 mmol), Mn (70 mg, 1.27 mmol) were placed in a round-bottomed flask. THF (2 mL) was added. To a stirred suspension of the reaction mixture was added NEt_3 (78 μL , 1.19 M in THF). The reaction mixture was stirred vigorously for 1.5h (the solution color became green). At RT, 2,6-lutidine (110 μL , 0.944 mmol) and 3-bromo-2-chloropropene (170 mg, 1.1 mmol) was added. After 10 min at RT, the reaction was cooled to 0 °C and stirred for 10 min. 4-(*tert*-Butyldiphenylsilyloxy)-butyraldehyde (140 mg, 0.43 mmol) in THF (0.3 mL) and TMSCl (160 μL , 1.31 mmol) were added. The reaction mixture was stirred for 16h at 0 °C and diluted with Et_2O . The precipitates were filtered through a SiO_2 plug (eluted with Et_2O). The filtrate was evaporated *in vacuo* to give the TMS protected homoallylic alcohol. This was dissolved in THF (0.5 mL) and 70% AcOH (1 mL) was added. The reaction mixture was stirred for 1h and quenched with NaHCO_3 . The aqueous phase was extracted with Et_2O (thrice) and the combined extracts were dried over Na_2SO_4 . All volatiles were evaporated *in vacuo* to give the crude homoallylic alcohol. Purification by silica gel chromatography (15:1 to 10:1, hexanes: EtOAc) afforded 7-(*tert*-butyldiphenylsilyloxy)-2-chlorohept-1-en-4-ol (**7I**, 155 mg, 90%). Data for **7I**: ^1H -NMR (500 MHz, CDCl_3): 7.6-7.4 ppm (10H, m), 5.27 (1H, dd, $J = 1.5$ Hz), 5.24 (1H, dd, $J = 1.5$ Hz), 3.99 (1H, m), 3.70 (2H, m), 2.47 (2H, m), 1.7-1.5 (4H, m), 1.05 (9H, s). ^{13}C -NMR (100 MHz, CDCl_3): 135.6 ppm, 135.5, 129.7, 127.7, 114.9, 68.4, 64.0, 47.2, 33.4, 28.6, 26.8. The *ee* of **7I** was determined to be 92% by ^1H -NMR analysis of its (+)-Mosher ester.

(*R*)-Dec-1-en-4-ol (**4a**) was synthesized *via* procedure B.

Allylation of heptanal with 4 mol% of the ligand- CrCl_3 . The ligand **1d** (149 mg, 0.400 mmol), $\text{CrCl}_3 \cdot 3\text{THF}$ (134 mg, 0.36 mmol), Mn (596 mg, 10.8 mmol) were placed in a round-bottomed flask. THF (30 mL) was added. To a stirred suspension of the reaction mixture was added NEt_3 (112 μL , 0.8 mmol). The reaction mixture was stirred vigorously for 1.5h. At RT, 2,6-lutidine (2 mL, 17.2 mmol) and allyl bromide (1.9 mL, 22.5 mmol) was added. After 15 min at RT, the reaction was cooled to 0 °C and stirred for 10 min. Heptanal (1.05 g, 9 mmol) and TMSCl (2.7 mL, 22.1 mmol) were added. The reaction mixture was stirred for 36h at 0 °C and diluted with Et_2O . The precipitates were filtered through SiO_2 plug (eluted with Et_2O). The filtrate was evaporated *in vacuo* to give the TMS protected homoallylic alcohol. This was dissolved in THF (5 mL) and 70% AcOH (10 mL) was added. The reaction mixture was stirred for 2h and quenched with NaHCO_3 . The aqueous phase was extracted with Et_2O (thrice) and the combined extracts were dried over Na_2SO_4 . All volatiles were evaporated *in vacuo* to give the crude homoallylic alcohol. Purification by silica gel chromatography (9:1, hexanes: EtOAc) afforded dec-1-en-4-ol (**4a**, 1.1 g, 85%). $[\alpha]_D + 9.5^\circ$ (c 0.5, CHCl_3 , at 25 °C)^c. ^1H -NMR (500 MHz, CDCl_3): 5.83 ppm (1H, m), 5.15 (2H, m), 3.64 (1H, m), 2.35 (1H, m), 1.7-1.2 (10H, m), 0.88 (3H, t-like). ^{13}C -NMR (125 MHz, CDCl_3): 134.9 ppm, 118.1, 70.7, 41.9, 36.8, 31.8, 29.3, 25.6, 22.6, 14.1. The *ee* of **4a** was determined to be 93% by ^1H -NMR analysis of its (+)-Mosher ester.

(*R*)-7-(*tert*-Butyldiphenylsilyloxy)-hept-1-en-4-ol (**4b**) was synthesized *via* procedure B.

Data for **4b**: $^1\text{H-NMR}$ (500 MHz, CDCl_3): 7.6-7.4 ppm (10H, m), 5.70 (1H, m), 4.99 (1H, m), 3.52 (3H, m), 2.11 (1H, m), 2.04 (1H, m), 1.6-1.4 (4H, m), 0.91 (9H, s). $^{13}\text{C-NMR}$ (125 MHz, CDCl_3): 135.6 ppm, 135.0, 133.7, 129.6, 127.6, 117.8, 70.5, 64.1, 41.9, 33.6, 28.7, 26.8, 19.2. The *ee* of **4b** was determined to be 93% by $^1\text{H-NMR}$ analysis of its (+)-Mosher ester.

(R)-7-(tert-Butyldiphenylsilyloxy)-3,3-dimethyl-hept-1-en-4-ol (4c) was synthesized *via* procedure B.

Data for **4c**: $^1\text{H-NMR}$ (400 MHz, C_6D_6): 7.56 ppm (4H, m), 7.3-7.2 (6H, m), 5.72 (1H, dd, $J = 14.0, 8.8$ Hz, m), 4.95 (2H, m), 3.61 (2H, m), 3.18 (1H, d, $J = 8.8$ Hz), 1.72 (2H, m), 1.62 (1H, m), 1.50 (1H, m), 1.12 (1H, m), 1.07 (9H, s), 0.96 (3H, s), 0.95 (3H, s). $^{13}\text{C-NMR}$ (125 MHz, CDCl_3): 145.5 ppm, 135.6, 135.5, 129.6, 127.6, 113.0, 78.0, 64.0, 41.6, 29.8, 28.0, 26.8, 23.0, 22.3, 19.2. The *ee* of **4c** was determined to be 94% by $^1\text{H-NMR}$ analysis of its (+)-Mosher ester.

(R)-7-(tert-Butyldiphenylsilyloxy)-2-methyl-hept-1-en-4-ol (4d) was synthesized *via* procedure B.

Data for **4d**: $^1\text{H-NMR}$ (500 MHz, CDCl_3): 7.6-7.4 ppm (10H, m), 4.88 (1H, s), 4.79 (1H, s), 3.76 (1H, m), 3.70 (2H, m), 2.2-2.1 (2H, m), 1.75 (3H, s), 1.7-1.5 (4H, m), 1.07 (9H, s). $^{13}\text{C-NMR}$ (125 MHz, CDCl_3): 135.7 ppm, 135.6, 133.7, 129.6, 127.6, 113.3, 68.5, 64.1, 46.1, 33.7, 28.8, 26.8, 22.4, 19.1. The *ee* of **4d** was determined to be 91% by $^1\text{H-NMR}$ analysis of its (+)-Mosher ester.

References

- (a) *Org. Synthesis*, Coll. Vol. I, 2nd Ed., **1941**, 208.
- (b) Corey, E. J.; Kim, C. M.; Takeda, M. *Tetrahedron Lett.* **1972**, 13, 4339.
- (c) Adimurthy, S.; Ramachandraiah, G.; Ghosh, P. K.; Bedekar, A. V. *Tetrahedron Lett.* **2003**, 44, 5099.
- (d) Hara, S.; Dojo, H.; Takinami, S.; Suzuki, A. *Tetrahedron Lett.* **1983**, 24, 731.
- (e) Iseki, K.; Mizuno, S.; Kuroaki, Y. *Tetrahedron* **1999**, 55, 977.

STANDARD PROTON PARAMETERS

exp1 szpul

SAMPLE May 24 2004 DEC. 8 VT
 date May 24 2004 dfrq 439.750
 solvent CDC13 dn H1
 file /export/home/~ dpwr 34
 1500/vmr/sys/data/~ dof 0
 kishi/mlin.fid/ml~ dm nmh
 n443p.fid dm c
 ACQUISITION 439.750 dmf 14300
 ln H1 dseq 1.0
 at 2.048 H1 dres
 sv 32768 temp 23.0
 fb 8000 0 PROCESSING 0.10
 bs 4000 1b wfile
 ss 2 2 proc not used
 tpwr 54 fn ft
 pw 6.0 math f
 dl 0.200 wefr
 tof 0 wefr
 nt 64 wexp
 ct 64 wbs
 alock n wnt
 gain not used wft

DISPLAY -250.2
 sp 6436.6
 wp 309
 vs 0
 sc 250
 wc 25.99
 hzmm 262.83
 ls 1501.1
 rft 0
 rfp 7
 lh 1.000
 nm

STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul
 Solvent: CDCl3
 Temp: 23.0 C / 296.1 K
 INOVA-500 "inovas500"

Relax. delay 0.200 sec
 Pulse 74.0 degrees
 Acq. time 2.048 sec
 Width 8000.0 Hz
 16 repetitions
 OBSERVE H1: 499.7479209 MHz
 DATA PROCESSING
 Line broadening 0.1 Hz
 FT size 32768
 Total time 0 min, 40 sec

STANDARD PROTON PARAMETERS

Pulse Sequence: s2pu1

Solvent: CDCl3

Temp: 23.0 C / 296.1 K

INOVA-500 "Inova500"

Relax. delay 0.200 sec

Pulse 74.0 degrees

Acq. time 2.048 sec

Width 8000.0 Hz

16 repetitions

OBSERVE H1 499.7479209 MHz

DATA PROCESSING

Line broadening 0.1 Hz

FT size 32768

Total time 0 min, 40 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pu1
Solvent: CDCl3
Temp: 23.0 C / 296.1 K
Mercury-400BB "mercury400b"
Pulse: 77.1 degrees
Acq time: 2.731 sec
Width: 5998.8 Hz
260 repetitions
OBSERVE: H1 399.8366315 MHz
DATA PROCESSING
line broadening 0.1 Hz
FT size: 32768
Total time: 13 min, 19 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Temp: 23.0 C / 296.1 K

Mercury-40088 "mercury400b"

Pulse 77.1 degrees

Acq. time 2.731 sec

Width 5998.8 Hz

16 repetitions

OBSERVE H1: 399.8366882 MHz

DATA PROCESSING

Line broadening 0.1 Hz

FT size 32768

Total time 0 min, 58 sec

STANDARD PROTON PARAMETERS

Pulse Sequence: szpu1

Solvent: CDCl3

Temp: 22.0 C / 295.1 K

INNOVA-500 "11novas500b"

Pulse 72.6 degrees

Acq. time 2.048 sec

Width 8000.0 Hz

100 repetitions

ORSERVE H1, 500.2085660 MHz

DATA PROCESSING

Line broadening 0.1 Hz

FI size 32768

Total time 3 min, 29 sec

STANDARD PROTON PARAMETERS

Pulse Sequence: szpul

Solvent: CDCl₃

Temp: 23.0 C / 296.1 K

INOVA-500 "inovas500"

Relax. delay: 0.200 sec

Pulse: 74.0 degrees

Acq. time: 2.048 sec

Width: 800.0 Hz

15 repetitions

Observed: 93.7479204 MHz

DATA PROCESSING

Line broadening: 0.1 Hz

FT size: 32768

Total time: 0 min, 40 sec

STANDARD PROTON PARAMETERS

Pulse Sequence: szpu1

Solvent: CDCl3

Temp: 23.0 C / 296.1 K

INOVA-500 "1HNOVA500"

Relax. delay 0.200 sec

Pulse 74.0 degrees

Acq. time 2.048 sec

Width 8000.0 Hz

16 repetitions

OBSERVE H1, 499.7479209 MHz

DATA PROCESSING

Line broadening 0.1 Hz

FT size 32768

Total time 0 min, 40 sec

STANDARD PROTON PARAMETERS

Pulse Sequence: szpul

Solvent: CDCl₃

Temp: 23.0 C / 296.1 K

INOVA-500 "inova500"

Relax. delay 0.200 sec

Pulse: 74.0 degrees

Acq. time 2.048 sec

Width 800.0 Hz

16 Transitions

OBSERVE H1003 493.7479204 MHz

DATA PROCESSING

Line broadening 0.1 Hz

FT size 32768

Total time 0 min, 40 sec

in C₆D₆

SAMPLE DEC. & VT
 date Jun 7 2004 dfreq 499.750
 solvent CDC13 dn H1
 file /export/home/~ 34
 1500/vmrsys/data/~ dof 0
 kishi/mln.fid/ml~ dm nnn
 nd60p.fid damm c
 ACQUISITION
 sfrq 499.750 dseq 14300
 tn H1 dres 1.0
 at 2.048 homo n
 mp 32768 temp 23.0
 sw 8000.0 PROCESSING 0.10
 fb 4000 lb wlfie
 bs 8 wlfie
 ss 2 proc ft
 tpwr 54 fn not used
 pw 6.0 math f
 dl 0.200 werr
 tof 0 wexp
 nt 128 wbs
 ct 128 wnt
 alock not used
 gain not used
 flags
 il n
 in n
 dp y
 hs mn
 DISPLAY
 sp -250.2
 wp 5497.1
 vs 308
 vc 0
 wc 250
 hzmm 21.39
 ls 262.83
 rfi 1501.1
 rfp 0
 th 0
 tms 1.000
 mm
 ph

STANDARD PROTON PARAMETERS

expt szpu1

SAMPLE
 date Jun 9 2004 DEC. 8 VT
 solvent CDC13 dfrq 499.750
 file /export/home/~/dpwr H1
 1500/vmrtsys/data/~/dof 34
 kishi/min.ftid/m1- dm 0
 n6bmsherp.ftid dm mm
 ACQUISITION 14300
 sfrq 499.750 dseq
 tn H1 dres 1.0
 al 2.048 homo n
 np 32768 temp 23.0
 sw 8000.0 PROCESSING 0.10
 fd 4000 lb ffile
 us 8 wfile ft
 ss 2 ploc not used f
 lpwr 54 tn math
 pw 6.0 weft
 dl 0.200 weft
 tof 0 weft
 nt 128 wexp
 ct 128 wds
 alock n wnt
 gain not used wft
 flags
 l1 n
 ln n
 dp y
 hs mn
 DISPLAY
 sp -250.2
 wp 5497.1
 vs 678
 sc 0
 wc 250
 hzmm 21.99
 ls 196.75
 ffl 1501.1
 rfp 0
 th 7
 ins 1.000
 nm ph

21.25

STANDARD PROTON PARAMETERS

exp3 s2pu1

SAMPLE 9 2004 DEC. & VI
date Jun 2004 dfrq 499.750
solvent CDCl3 dn 11
file /export/home/~dpr 34
1500/vnmr/sy/data/~dof 0
kishi/mln.fid/mln~dm nmn
n46sp.fid dm c
ACQUISITION 14300
sfrq 499.750 dseq
in at H1 dres 1.0
np 2.048 homo n
sw 32768 temp 23.0
fb 8000.0 PROCESSING
bs 4000 lb 0.10
ss 8 wfile
ss 2 proc ft
tpwr 54 fn not used f
pw 6.0 math
di 0.200
tof 0 weff
nt 128 wepp
ct 44 wbs
aLOCK n wnt
gain not used wft
FLAGS
l1 n
in n
dp y
hs nm
DISPLAY
sp -250.2
wp 5497.1
vs 309
sc 0
wc 250
hzmm 21.39
is 262.83
rfi 1501.1
th 0
ins 7
nm 1.000
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STANDARD PROTON PARAMETERS

expt s2put

SAMPLE DEC. & VT 499.750
date Jun 9 2004 dfrq HI
solvent CDCl3 dfrq HI
file /export/home/~ dfrq HI
1500/vmrtsys/data/~ dfrq HI
Kishi/m1n.ftid/m1n~ dfrq HI
n6smosherp.ftid dfrq HI
ACQUISITION dfrq HI
sfrq 499.750 dfrq HI
in at 2.048 HI
np 32768 HI
sw 8000.0 temp 23.0
fb 4000 lb
bs 8 wfile
ss 2 fproc
tpwr 54 fn
pw 6.0 math
d1 0.200 werr
nt 0 wexp
ct 128 wds
a lock n wlt
gain not used wft
flags
i1 n
in n
dp y
hs nm
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sp -250.2
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vc 151
wc 250
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is 262.83
rftl 1501.1
rtp 0
lms 1.000
nm ph

STANDARD PROTON PARAMETERS

expi szpu1

SAMPLE DEC. & VT
 date Jun 15 2004 dfrq 499.750
 solvent CDCl3 dn H1
 file /export/home/~ upwr 34
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 kishi/mln.fid/ml~ dm nm
 n466p.fid dmf c
 ACQUISITION 14300
 sfrq 499.750 dseq
 tn H1 dres 1.0
 at 2.048 homo n
 np 32768 temp 23.0
 sw 8000.0 PROCESSING
 fb 4000 lb wftlle 0.10
 bs 8 wftlle
 ss 2 pproc ft
 ipwr 54 fn not used
 pw 6.0 math f
 dl 0.200 weft
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STANDARD PROTON PARAMETERS

exp3 s2put1

SAMPLE date Jun 21, 2004 DEC. & VT 499.750
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 file /export/home/~ dpvr 34
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 kishi/m1n.fid/m1~ dm nm
 n46wosherp.fid dnm nm
 ACQUISITION 499.750 dnt 14300
 sfrq 499.750 dseq 1.0
 nu 32.0 dcs 1.0
 an 2.048 hpc 1.0
 nd 32768 temp 23.0
 sw 8000.0 lb temp PROCESSING 0.10
 fb 4000 1b wfile ft
 bs 2 wfile ft
 ss 2 proc not used f
 tpwr 54 fn
 pw 6.0 math
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STANDARD PROTON PARAMETERS

expl s2put1

SAMPLE DEC. & VT
date Jun 15 2004 dfrq 499.750
solvent CDC13 dn H1
file /export/home/~ dpvr 34
1500/vnmr/sy/data/~ dof 0
kishi/mln.fid/ml~ dm nm
n467p.fid dnm C
ACQUISITION 14300
sfrq 499.750 dseq
tn H1 dres 1.0
at 2.048 homo n
np 32768 temp 23.0
sw 8000.0 PROCESSING 0.10
fb 4000 lb wfile
bs 8 wfile
ss 2 proc ft
tpvr 54 fn not used
pw 6.0 math
d1 0.200 weff
tof 0 weff
nt 128 wexp
ct 128 wbs
atock n wnt
gain not used wft
flags
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f1631 n
f1632 n
f1633 n
f1634 n
f1635 n
f1636 n
f1637 n
f1638 n
f1639 n
f1640 n
f1

STANDARD PROTON PARAMETERS

exp3 s2pul

SAMPLE DEC. & VT
 date Jun 21 2004 dfrq 499.750
 solvent CDCl3 dn H1
 file /export/home/~ dpwr 34
 1500/vnmr-sys/data/~ dof 0
 kishi/min.fid/m1~ nmh
 n467mosherp.fid c 14300
 ACQUISITION
 sfrq 499.750 dmf
 tn H1 dseq 1.0
 at 2.048 homo n
 np 32.768 temp 23.0
 sw 8000.0 lb
 fb 4000.0 wlfle 0.10
 bs 8 2 proc ft
 ss 54 fn not used
 tpwr 6.0 math f
 pw 0.200 werr
 d1 0 wexp
 nt 128 wbs
 ct 80 wnt
 alock n
 gain not used wft
 FLAGS
 l1 n
 in n
 dp y
 hs mn
 DISPLAY
 sp -250.2
 wp 5497.1
 vs 167
 sc 0
 wc 250
 hzmm 21.99
 is 254.04
 rfl 1501.1
 rfp 0
 th 7
 ns 1.000
 nm ph

STANDARD PROTON PARAMETERS

Pulse Sequence: s2pu1

Solvent: CDCl3

Temp.: 23.0 C / 296.1 K

INOVA-500 "inovas500"

Relax. delay 0.200 sec

Pulse 74.0 degrees

Acq. time 2.048 sec

Width 8000.0 Hz

72 repetitions

OBSERVE H1, 499.7479495 MHz

DATA PROCESSING

Line broadening 0.1 Hz

FT size 32768

Total time 4 min, 53 sec

C_6D_6
500 MHz

STANDARD PROTON PARAMETERS

Pulse Sequence: s2pu1
 Solvent: CDCl3
 Temp: 23.0 C / 296.1 K
 INOVA-500 "INOVAS500"

Relax. delay 0.200 sec
 Pulse 74.0 degrees
 Acq. time 2.048 sec
 Width 8000.0 Hz
 72 repetitions
 OBSERVE H1, 499.7479500 MHz
 DATA PROCESSING
 Line broadening 0.1 Hz
 FT size 32768
 Total time 4 min, 53 sec