— Supporting Information —

Chiral Boronate Derivatives via Catalytic Enantioselective Conjugate Addition of Grignard Reagents on 3-Boronyl Unsaturated Esters and Thioesters

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1. Experimental Details and Compound Characterization Data

1.1 General Information

Unless otherwise stated, all reactions were performed under an argon atmosphere using flame-dried glassware. Toluene and dichloromethane were distilled from CaH₂. THF, 2-methyl THF and Et₂O were distilled from sodium with benzophenone as an indicator. Acetone was distilled from 4 Å molecular sieves. Anhydrous *t*BuOMe was used as received from commercial source. Thin layer chromatography (TLC) was performed on Merck Silica Gel 60 F254 plates and was visualized with UV light and KMnO₄ stain. NMR spectra were recorded on Varian INOVA-300, INOVA-400 or INOVA-500 MHz instruments. The residual solvent protons (¹H) or the solvent carbons (¹³C) were used as internal standards. Boron NMR spectra are referenced to external BF₃·OEt₂. ¹H NMR data is presented as follows: chemical shift in ppm (δ) downfield from tetramethylsilane (multiplicity, coupling constant, integration). The following abbreviations are used in reporting NMR data: s, singlet; br s, broad singlet; d, doublet; t, triplet; q, quartet; sxt, sextet; spt, septet, dd, doublet of doublets; tt, triplet of triplets, m, multiplet. High-resolution mass spectra were recorded by the University of Alberta mass spectrometry services laboratory using either electron impact (EI) or electrospray ionization (ESI) techniques. Infrared spectra were obtained on a Nicolet Magna-IR with frequencies expressed in cm⁻¹. Grignard reagents were either purchased from Aldrich (MeMgBr, EtMgBr, PhMgBr) or prepared from the corresponding alkyl bromides and magnesium turnings in Et₂O following standard procedures. Grignard reagents were titrated using the method developed by Krasovskiy and Knochel.¹ Racemic 1,4-addition products were obtained by reaction of the enoates with the corresponding RMgX reagents (−20 °C, Et₂O) and

¹ Krasovskiy, A.; Knochel, P. *Synthesis* **2006**, 890-891.

CuI (100 mol%) or using *rac*-BINAP by following the catalytic conjugate addition method detailed below. The starting materials were synthesized according to the literature procedures:

(*E*)-3-methoxy-3-oxoprop-1-enylboronic acid (**S1**)² and

(*E*)-methyl 3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)acrylate (**1**)³.

1.2 Preparation of Protected 3-Boronyl Unsaturated Esters or Thioesters

1.2.1 (*E*)-Methyl 3-(6-methyl-4,8-dioxo-1,3,6,2-dioxazaborocan-2-yl)acrylate (**3**)

To a stirred solution of boronic acid **S1** (1.00 g, 7.70 mmol) in toluene (8.0 mL) and DMSO (3.0 mL) was added *N*-methyliminodiacetic acid (1.30 g, 8.50 mmol). The reaction was then heated under reflux for 16 h with azeotropic removal of water. Once the reaction was done, the reaction mixture was concentrated *in vacuo* and purified by flash column chromatography (CH₃CN/EtOAc = 1:1) to give **3** (1.59 g, 85%) as a white solid.

¹H NMR (400 MHz, CD₃CN) δ 6.91 (d, *J* = 18.0 Hz, 1H), 6.28 (d, *J* = 17.8 Hz, 1H), 4.00 (d, *J* = 17.0 Hz, 2H), 3.85 (d, *J* = 17.0 Hz, 2H), 3.70 (s, 3H), 2.80 (s, 3H).

¹³C NMR (100 MHz, CD₃CN) δ 169.1, 167.3, 133.7, 62.8, 52.2, 47.9, 38.1.

¹¹B NMR (160 MHz, CD₃CN) δ 10.0.

² Gravel, M.; Touré, B. B. *Org. Prep. Proc. Intl.* **2004**, 36, 573–579.

³ Rasset-Deloge, C.; Martinez-Fresneda, P.; Vaultier, M. *Bull. Soc. Chim. Fr.* **1992**, 129, 285-290.

IR (Microscope, cm^{-1}) 3049, 3021, 2998, 2962, 1755, 1738, 1725, 1714.

HRMS (ESI) for $\text{C}_9\text{H}_{12}\text{BNO}_6$: calcd. 241.0758; found 241.0766.

M. P. 177 – 179 °C.

1.2.2 Tetrabutylammonium (*E*)-trifluoro(3-methoxy-3-oxoprop-1-enyl)borate (**4**)

To a stirred solution of **S1** (500 mg, 3.85 mmol) in methanol (7.5 mL) was added a solution of KHF_2 (1.50 g, 19.0 mmol) in H_2O (4.0 mL). The mixture was stirred for 1 hour at room temperature. The reaction mixture was then concentrated *in vacuo* and washed with hot acetone (3×20 mL). The acetone fractions were then filtered and concentrated *in vacuo* to provide the crude product. The solid was further washed with hexanes and diethyl ether to give the trifluoroborate salt.

The trifluoroborate salt was dissolved in a mixture of dichloromethane (5.0 mL) and H_2O (5.0 mL). The solution was cooled to 0 °C, at which point a solution of tetrabutylammonium hydroxide (2.4 mL, 3.7 mmol) was added to the solution slowly. The solution was then warmed to room temperature and stirred for another hour. Once the reaction was complete, the reaction mixture was diluted with dichloromethane (20 mL) and the layers were separated. The aqueous layer was further extracted with dichloromethane (3×20 mL) and the combined organic layer was then dried over MgSO_4 , filtered and concentrated *in vacuo* to afford **4** as a yellow solid (1.22 g, 80%).

^1H NMR (400 MHz, CDCl_3) δ 7.11 (d, $J = 18.1$ Hz, 1H), 6.11 (d, $J = 18.0$ Hz, 1H), 3.62 (s, 3H), 3.13 (m, 8H), 1.55 (m, 8H), 1.37 (sxt, $J = 7.3$ Hz, 8H), 0.95 (t, $J = 7.1$ Hz, 12H)

^{13}C NMR (100 MHz, CDCl_3) δ 168.7, 125.4, 58.5, 50.8, 23.8, 19.6, 13.6. (The

boron-bound carbon was not detected due to quadrupolar relaxation)

^{11}B NMR (160 MHz, CDCl_3) δ 2.1.

IR (Microscope, cm^{-1}) 2964, 2878, 1708, 1615.

HRMS (ESI) for $\text{C}_{20}\text{H}_{41}\text{BF}_3\text{NO}_2$: calcd. 153.0341; found 153.0340.

M. P. 63 – 64 $^{\circ}\text{C}$.

1.2.3 Naphthalene-1,8-diamido (dan) derivative (**5**)

A stirred mixture of boronic acid **S1** (500 mg, 3.85 mmol), 1,8-diaminonaphthalene (615 mg, 3.89 mmol) in toluene (40 mL) was heated for 2 h under reflux with azeotropic removal of water. The reaction mixture was then concentrated *in vacuo* and recrystallized (EtOAc) to give **5** (873 mg, 90%) as a yellow solid.

^1H NMR (400 MHz, CDCl_3) δ 7.09 (dd, $J = 8.3, 7.2$ Hz, 2H), 7.02 (dd, $J = 8.3, 1.1$ Hz, 2H), 7.00, (d, $J = 18.5$ Hz, 1H), 6.43 (d, $J = 18.4$ Hz, 1H), 6.33 (dd, $J = 7.1, 1.1$ Hz, 2H), 5.80 (br s, 2H), 3.79 (s, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 166.6, 140.5, 136.4, 132.9, 127.6, 120.3, 118.3, 106.2, 51.9. (The boron-bound carbon was not detected due to quadrupolar relaxation)

^{11}B NMR (160 MHz, CDCl_3) δ 27.9.

IR (Microscope, cm^{-1}) 3404, 3387, 3374, 3055, 3014, 2946, 1711, 1653, 1626, 1598, 1513.

HRMS (ESI) for $\text{C}_{14}\text{H}_{13}\text{BN}_2\text{O}_3$: calcd. 253.1145; found 253.1145.

M. P. 160 – 165 $^{\circ}\text{C}$ (decomposed).

1.2.4 Naphthalene-1,8-diamido (dan) derivative (7)

To a stirred solution of **5** (889 mg, 3.53 mmol) in THF was added AlCl_3 (564 mg, 4.22 mmol) and TMSSMe (848 mg, 7.05 mmol) sequentially. The solution was then stirred under reflux for 3 h before being quenched by the addition of aq. phosphate buffer solution ($\text{pH} = 7$) at room temperature. The layers were separated and the aqueous layer was extracted with diethyl ether ($3 \times 50 \text{ mL}$). The combined organic layer was then dried over MgSO_4 , filtered and concentrated *in vacuo*. The crude product was purified with flash column chromatography (hexanes/ $\text{EtOAc} = 19:1$) to give **7** (766 mg, 81%) as a red solid.

^1H NMR (400 MHz, CDCl_3) δ 7.09 (dd, $J = 8.3, 7.2 \text{ Hz}$, 2H), 7.02 (dd, $J = 8.4 \text{ Hz}$, 1.1 Hz, 2H), 6.87 (d, $J = 18.1 \text{ Hz}$, 1H), 6.67 (d, $J = 18.1 \text{ Hz}$, 1H), 6.33 (dd, $J = 7.3, 1.1 \text{ Hz}$, 2H), 5.81 (br s, 2H), 2.41 (s, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 166.6, 140.5, 136.4, 132.9, 127.6, 120.3, 118.3, 106.2, 51.9. (The boron-bound carbon was not detected due to quadrupolar relaxation)

^{11}B NMR (160 MHz, CDCl_3) δ 27.5.

IR (Microscope, cm^{-1}) 3404, 3387, 3374, 3055, 3014, 2946, 1711, 1653, 1626, 1598, 1513.

HRMS (ESI) for $\text{C}_{14}\text{H}_{13}\text{BN}_2\text{OS}$: calcd. 268.0842; found 268.0842.

M. P. 173 – 176 $^\circ\text{C}$ (decomposed).

1.3 Catalytic Asymmetric Conjugate Addition

1.3.1 General procedure (Table 2)

(*R*)-Tol-BINAP (25.5 mg, 0.0375 mmol) and CuI (4.8 mg, 0.025 mmol) were dissolved in dichloromethane (1.0 mL) and stirred at room temperature for 30 minutes until the solution turned yellow. Substrate **5** (126 mg, 0.500 mmol) or **7** (134 mg, 0.500 mmol) and dichloromethane (7.0 mL) were then sequentially added into the reaction mixture, which was stirred for 5 minutes before being cooled to $-78\text{ }^{\circ}\text{C}$. Then, the Grignard reagent (1.25 mmol) was added into the reaction mixture dropwise over a span of 2 minutes. After stirring for the period specified in Table 2, the reaction mixture was quenched with MeOH (1.0 mL) and 1 M NH_4Cl (4.0 mL) and was allowed to warm to room temperature. The layers were separated, and the aqueous layer was further extracted with diethyl ether ($3 \times 10\text{ mL}$). The combined organic layer was dried over MgSO_4 , filtered, and concentrated *in vacuo*. The crude product was then purified by flash column chromatography to give the pure product.

1.3.2 Naphthalene-1,8-diamido (dan) derivative **6a** (Table 2, entry 1)

The title compound was prepared using the general procedure for the catalytic asymmetric conjugate addition. Flash column chromatography (hexanes/EtOAc = 9:1) yielded **6a** (92% yield) as a colourless oil.

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.09 (dd, $J = 8.2, 7.3\text{ Hz}$, 2H), 7.00 (dd, $J = 8.3, 0.9\text{ Hz}$, 2H), 6.30 (dd, $J = 7.3, 1.0\text{ Hz}$, 2H), 5.77 (br s, 2H), 3.66 (s, 3H), 2.44 (m, 2H),

1.49 (m, 2H), 1.36 (m, 1H), 0.97 (t, $J = 7.3$ Hz, 3H)

^{13}C NMR (100 MHz, CDCl_3) δ 174.4, 141.0, 136.3, 127.5, 119.7, 117.5, 105.7, 51.7, 36.1, 24.6, 13.4. (The boron-bound carbon was not detected due to quadrupolar relaxation)

IR (Microscope, cm^{-1}) 3394, 3053, 2955, 2929, 2872, 1722, 1629, 1601, 1510.

HRMS (ESI) for $\text{C}_{16}\text{H}_{19}\text{BN}_2\text{O}_2$: calcd. 283.1615; found 283.1617.

$[\alpha]_{\text{D}}^{20}$: -5.3 ($c = 1.62$, chloroform) for 95% ee.

HPLC (Chiralcel OD): 10:90 *i*-PrOH/Hexanes, 0.5 mL/minute, $\lambda = 250$ nm, $T_{\text{major}} = 53.7$ min, $T_{\text{minor}} = 38.0$ min, ee = 95%.

1.3.3 Naphthalene-1,8-diamido (dan) derivative **6c** (Table 2, entry 3)

The title compound was prepared using the general procedure for the catalytic asymmetric conjugate addition. Flash column chromatography (hexanes/EtOAc = 9:1) yielded **6c** (94% yield) as a colourless oil.

^1H NMR (400 MHz, CDCl_3) δ 7.08 (dd, $J = 8.4, 7.3$ Hz, 2H), 6.99 (dd, $J = 8.3, 0.9$ Hz, 2H), 6.30 (dd, $J = 7.3, 1.0$ Hz, 2H), 5.74 (br s, 2H), 3.65 (s, 3H), 2.42, (m, 2H), 1.42 (m, 3H), 1.31 (m, 4H), 0.88 (t, $J = 7.0$ Hz, 3H)

^{13}C NMR (100 MHz, CDCl_3) δ 174.4, 141.0, 136.3, 127.5, 119.7, 117.5, 105.7, 51.6, 36.4, 31.5, 31.1, 22.9, 14.0. (The boron-bound carbon was not detected due to quadrupolar relaxation)

IR (Microscope, cm^{-1}) 3395, 3054, 2954, 2925, 2870, 2856, 1723, 1629, 1602, 1511.

HRMS (ESI) for C₁₈H₂₃BN₂O₂: calcd. 310.1853; found 310.1858.

[α]_D²⁰: +5.0 (c = 4.01, chloroform) for 95% ee

HPLC (Chiralcel OD): 20:80 *i*-PrOH/Hexane, 0.5 mL/minute, λ = 250 nm, T_{major} = 32.0 min, T_{minor} = 21.8 min, ee = 95%.

1.3.4 Naphthalene-1,8-diamido (dan) derivative **6d** (Table 2, entry 4)

The title compound was prepared using the general procedure for the catalytic asymmetric conjugate addition. Flash column chromatography (hexanes/EtOAc = 9:1) yielded **6d** (81% yield) as a colourless oil.

¹H NMR (400 MHz, CDCl₃) δ 7.09 (dd, *J* = 8.2, 7.4 Hz, 2H), 7.00 (dd, *J* = 8.4, 1.0 Hz, 2H), 6.31 (dd, *J* = 7.3, 1.0 Hz, 2H), 5.76 (br s, 2H), 3.66 (s, 3H), 2.43 (m, 2H), 1.27-1.43 (m, 9H), 0.87 (t, *J* = 6.8 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 174.4, 141.1, 136.3, 127.5, 119.7, 117.5, 105.7, 51.6, 36.4, 32.1, 31.8, 28.6, 22.5, 14.1. (The boron-bound carbon was not detected due to quadrupolar relaxation)

IR (Microscope, cm⁻¹) 3395, 3054, 2953, 2924, 2854, 1724, 1629, 1602, 1510.

HRMS (ESI) for C₁₉H₂₅BN₂O₂: calcd. 325.2085; found 325.2085.

[α]_D²⁰: +5.9 (c = 1.50, chloroform) for 95% ee

HPLC (Chiralcel OD): 20:80 *i*-PrOH/Hexane, 0.5 mL/minute, λ = 210 nm, T_{major} = 30.6 min, T_{minor} = 21.3 min, ee = 95%.

1.3.5 Naphthalene-1,8-diamido (dan) derivative 6e (Table 2, entry 5)

The title compound was prepared using the general procedure for the catalytic asymmetric conjugate addition. Flash column chromatography (hexanes/EtOAc = 9:1) yielded **6e** (87% yield) as a colourless oil.

¹H NMR (400 MHz, CDCl₃) δ 7.08 (dd, *J* = 8.3, 7.3 Hz, 2H), 7.00 (dd, *J* = 8.4, 0.9 Hz, 2H), 6.30 (dd, *J* = 7.3, 1.0 Hz, 2H), 5.83-5.74 (m, 3H), 5.06-4.94 (m, 2H), 3.66 (s, 3H), 2.44 (m, 2H), 2.12 (m, 2H), 1.41-1.62 (m, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 174.2, 141.0, 138.3, 136.3, 127.5, 119.7, 117.6, 115.0, 105.7, 51.7, 36.3, 33.0, 30.9. (The boron-bound carbon was not detected due to quadrupolar relaxation)

IR (Microscope, cm⁻¹) 3394, 3055, 2950, 2918, 2850, 1723, 1629, 1602, 1510.

HRMS (ESI) for C₁₈H₂₁BN₂O₂: calcd. 309.1772; found 309.1765.

[α]_D²⁰: +1.6 (c = 2.44, chloroform) for 98% ee

HPLC (Chiralcel OD): 20:80 *i*-PrOH/Hexanes, 0.5 mL/minutes, λ = 250 nm, T_{major} = 37.1 min, T_{minor} = 23.1 min, ee = 98%.

1.3.6 Naphthalene-1,8-diamido (dan) derivative 6f (Table 2, entry 6)

The title compound was prepared using the general procedure for the catalytic asymmetric conjugate addition. Flash column chromatography (hexanes/EtOAc = 9:1) yielded **6f** (88% yield) as a colourless oil.

¹H NMR (400 MHz, CDCl₃) δ 7.08 (dd, *J* = 8.2, 7.3 Hz, 2H), 6.99 (dd, *J* = 8.3, 0.9 Hz, 2H), 6.29 (dd, *J* = 7.3, 1.0 Hz, 2H), 5.80-5.72 (m, 3H), 5.02-4.92 (m, 2H), 3.65 (s, 3H), 2.42 (m, 2H), 2.05 (m, 2H), 1.44 (m, 5H).

¹³C NMR (100 MHz, CDCl₃) δ 174.3, 141.0, 138.5, 136.3, 127.5, 119.7, 117.5, 114.8, 105.7, 51.7, 36.4, 33.9, 31.2, 28.2. (The boron-bound carbon was not detected due to quadrupolar relaxation)

IR (Microscope, cm⁻¹) 3394, 3055, 2975, 2924, 2853, 1723, 1629, 1602, 1501.

HRMS (ESI) for C₁₉H₂₃BN₂O₂: calcd. 322.1853; found 322.1856.

[α]_D²⁰: +4.2 (c = 3.71, chloroform) for 96% ee.

HPLC (Chiralcel OD): 20:80 *i*-PrOH/Hexanes, 0.5 mL/minute, λ = 250 nm, T_{major} = 30.8 min, T_{minor} = 23.3 min, ee = 96%.

1.3.7 Naphthalene-1,8-diamido (dan) derivative **6g** (Table 2, entry 7)

The title compound was prepared using the general procedure for the catalytic asymmetric conjugate addition. Flash column chromatography (hexanes/EtOAc = 9:1) yielded **6g** (88% yield) as a colourless oil.

¹H NMR (400 MHz, CDCl₃) δ 7.31-7.26 (m, 2H), 7.22-7.16 (m, 2H), 7.10 (dd, *J* = 8.3, 7.2 Hz, 2H), 7.02 (dd, *J* = 8.3, 0.9 Hz, 2H), 6.29 (dd, *J* = 7.3, 1.0 Hz, 2H), 5.70 (br s, 2H), 3.67 (s, 3H), 2.64(m, 2H), 2.44 (m, 2H), 1.71 (m, 2H), 1.48 (m, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 174.3, 142.2, 140.9, 136.3, 128.4, 128.3, 127.5, 125.8, 119.7, 117.5, 105.8, 51.7, 36.4, 36.0, 31.1, 30.6. (The boron-bound carbon was not detected due to quadrupolar relaxation)

IR (Microscope, cm^{-1}) 3399, 3056, 3026, 2927, 2855, 1723, 1629, 1602, 1510.

HRMS (ESI) for $\text{C}_{23}\text{H}_{25}\text{BN}_2\text{O}_2$: calcd. 372.2009; found 372.2004.

$[\alpha]_{\text{D}}^{20}$: +3.3 ($c = 3.57$, chloroform) for 98.5% ee.

HPLC (Chiralcel OD): 20:80 *i*-PrOH/Hexanes, 0.5 mL/minute, $\lambda = 230$ nm, $T_{\text{major}} = 46.7$ min, $T_{\text{minor}} = 30.1$ min, ee = 98.5%.

1.3.8 Naphthalene-1,8-diamido (dan) derivative **6h** (Table 2, entry 8)

The title compound was prepared using the general procedure for the catalytic asymmetric conjugate addition. Flash column chromatography (hexanes/EtOAc = 9:1) yielded **6h** (89% yield) as a colourless oil.

^1H NMR (400 MHz, CDCl_3) δ 7.08 (dd, $J = 8.2, 7.3$ Hz, 2H), 6.99 (dd, $J = 8.4, 0.9$ Hz, 2H), 6.29 (dd, $J = 7.3, 1.0$ Hz, 2H), 5.74 (s, 2H), 3.64 (s, 3H), 2.52 (dd, $J = 16.0, 5.4$ Hz, 1H), 2.39 (dd, $J = 16.0, 9.9$ Hz, 1H), 1.78 (spt, $J = 7.3$ Hz, 1H), 1.22 (m, 1H), 0.97 (dd, $J = 6.7, 4.6$ Hz, 6H).

^{13}C NMR (100 MHz, CDCl_3) δ 174.6, 141.0, 136.3, 127.5, 119.7, 117.5, 105.7, 51.7, 33.9, 29.6, 22.3, 21.8.

IR (Microscope, cm^{-1}) 3396, 3054, 2955, 2869, 1723, 1629, 1601, 1509.

HRMS (ESI) for $\text{C}_{17}\text{H}_{21}\text{BN}_2\text{O}_2$: calcd. 297.1772; found 297.1774.

$[\alpha]_{\text{D}}^{20}$: +5.8 ($c = 1.58$, chloroform) for 91% ee.

HPLC (Chiralcel OD): 20:80 *i*-PrOH/Hexanes, 0.5 mL/minute, λ = 210 nm, T_{major} = 33.1 min, T_{minor} = 20.0 min, ee = 91%.

1.3.9 Naphthalene-1,8-diamido (dan) derivative **6i** (Table 2, entry 9)

The title compound was prepared using the general procedure for the catalytic asymmetric conjugate addition. Flash column chromatography (hexanes/EtOAc = 9:1) yielded **6i** (81% yield) as a colourless oil.

^1H NMR (400 MHz, CDCl_3) δ 7.08 (dd, J = 8.1, 7.2 Hz, 2H), 6.99 (dd, J = 8.2, 0.7 Hz, 2H), 6.29 (dd, J = 7.3, 0.9 Hz, 2H), 5.73 (br s, 2H), 3.64 (s, 3H), 2.53 (dd, J = 16.1, 5.3 Hz, 1H), 2.40 (dd, J = 16.1, 9.7 Hz, 1H), 1.58-1.83 (m, 6H), 1.32-1.44 (m, 1H), 0.82-1.30 (m, 8H).

^{13}C NMR (100 MHz, CDCl_3) δ 174.6, 141.0, 136.3, 127.5, 119.7, 117.4, 105.7, 51.7, 39.6, 33.6, 32.9, 32.4, 26.6. (The boron-bound carbon was not detected due to quadrupolar relaxation)

IR (Microscope, cm^{-1}) 3394, 3053, 2923, 2850, 1723, 1629, 1601, 1510.

HRMS (ESI) for $\text{C}_{20}\text{H}_{25}\text{BN}_2\text{O}_2$: calcd. 336.2009; found 336.2011.

$[\alpha]_{\text{D}}^{20}$: -1.8 (c = 1.98, chloroform) for 91% ee.

HPLC (Chiralcel OD): 20:80 *i*-PrOH/Hexanes, 0.5 mL/minute, λ = 210 nm, T_{major} = 34.0 min, T_{minor} = 20.0 min, ee = 91%.

1.3.10 Naphthalene-1,8-diamido (dan) derivative 6j (Table 2, entry 10)

The title compound was prepared using the general procedure for the catalytic asymmetric conjugate addition. Flash column chromatography (hexanes/EtOAc = 9:1) yielded **6j** (31% yield) as a yellow oil.

¹H NMR (400 MHz, CDCl₃) δ 7.40-7.31 (m, 2H), 7.27-7.17 (m, 3H), 7.08 (dd, J = 8.3, 7.3 Hz, 2H), 7.00 (dd, J = 8.3, 1.0 Hz, 2H), 6.25 (dd, J = 7.2, 1.1 Hz, 2H), 5.68 (br s, 2H), 3.68 (s, 3H), 3.00-2.74 (m, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 174.0, 142.7, 140.8, 136.3, 129.0, 127.9, 127.5, 125.7, 119.6, 117.8, 106.0, 51.9, 36.7. (The boron-bound carbon was not detected due to quadrupolar relaxation)

IR (Microscope, cm⁻¹) 3395, 3056, 2950, 1725, 1628, 1601, 1559, 1507.

HRMS (ESI) for C₂₀H₁₉BN₂O₂: calcd. 330.1536; found 330.1545.

$[\alpha]_D^{20}$: +0.7 (c = 1.35, chloroform) for 89% ee.

HPLC (Chiralcel OD): 10:90 *i*-PrOH/Hexanes, 0.5 mL/minute, λ = 250 nm, T_{major} = 51.2 min, T_{minor} = 58.7 min, ee = 89%.

1.3.11 Naphthalene-1,8-diamido (dan) derivative 8a (Table 2, entry 11)

The title compound was prepared using the general procedure for the catalytic asymmetric conjugate addition. Flash column chromatography (hexanes/EtOAc = 19:1) yielded **8a** (82% yield) as a yellow solid.

¹H NMR (400 MHz, CDCl₃) δ 7.08 (dd, *J* = 8.3, 7.3 Hz, 2H), 6.99 (dd, *J* = 8.3, 1.0 Hz, 2H), 6.30 (dd, *J* = 7.2, 1.0 Hz, 2H), 5.70 (br s, 2H), 2.71 (dd, *J* = 15.4, 7.5 Hz, 1H), 2.61 (dd, *J* = 15.4, 7.1 Hz, 1H), 2.30 (s, 3H), 1.61 (sxt, *J* = 7.5 Hz, 1H), 1.08 (d, *J* = 7.4 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 200.3, 141.0, 136.3, 127.6, 119.7, 117.6, 105.8, 47.8, 16.2, 11.8. (The boron-bound carbon was not detected due to quadrupolar relaxation)

IR (Microscope, cm⁻¹) 3398, 3053, 2952, 2927, 2872, 1676, 1629, 1602, 1509.

HRMS (ESI) for C₁₅H₁₇BN₂OS: calcd. 284.1155; found 284.1160.

[α]_D²⁰: +19.7 (c = 0.37, chloroform) for 98% ee.

HPLC (Chiralcel OD): 10:90 *i*-PrOH/Hexanes, 0.5 mL/minute, λ = 230 nm, T_{major} = 39.7 min, T_{minor} = 36.8 min, ee = 98%.

1.3.12 Naphthalene-1,8-diamido (dan) derivative **8b** (Table 2, entry 12)

The title compound was prepared using the general procedure for the catalytic asymmetric conjugate addition. Flash column chromatography (hexanes/EtOAc = 19:1) yielded **8b** (80% yield) as a yellow oil.

¹H NMR (400 MHz, CDCl₃) δ 7.37-7.32 (m, 2H), 7.27-7.18 (m, 3H), 7.07 (dd, *J* = 8.3, 7.2 Hz, 2H), 7.01 (dd, *J* = 8.4, 1.0 Hz, 2H), 6.25 (dd, *J* = 7.1, 1.1 Hz, 2H), 5.64 (br s, 2H), 3.15-3.00 (m, 3H), 2.31 (s, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 200.0, 142.3, 140.8, 136.3, 129.0, 127.9, 127.5, 126.1, 119.6, 117.8, 106.0, 46.2, 11.9. (The boron-bound carbon was not detected due to quadrupolar relaxation)

IR (Microscope, cm^{-1}) 3404, 3054, 3027, 2978, 2928, 1678, 1629, 1601, 1508.

HRMS (ESI) for $\text{C}_{20}\text{H}_{19}\text{BN}_2\text{OS}$: calcd. 346.1311; found 346.1314.

$[\alpha]_{\text{D}}^{20}$: -16.3 ($c = 0.92$, chloroform) for 91% ee.

HPLC (Chiralcel OD): 20:80 *i*-PrOH/Hexanes, 0.5 mL/minute, $\lambda = 210$ nm, $T_{\text{major}} = 23.5$ min, $T_{\text{minor}} = 27.1$ min, ee = 91%.

1.3.13 Naphthalene-1,8-diamido (dan) derivative **8c** (Table 2, entry 13)

The title compound was prepared using the general procedure for the catalytic asymmetric conjugate addition. Flash column chromatography (hexanes/EtOAc = 19:1) yielded **8c** (62% yield) as a yellow oil.

^1H NMR (300 MHz, CDCl_3) δ 7.17-7.05 (m, 6H), 7.00 (dd, $J = 8.4, 1.2$ Hz, 2H), 6.25 (dd, $J = 7.3, 1.0$ Hz, 2H), 5.65 (br s, 2H), 3.12-2.95 (m, 3H), 2.35 (s, 3H), 2.31 (s, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 200.0, 140.8, 139.1, 136.3, 135.6, 129.7, 127.8, 127.5, 119.6, 117.7, 106.0, 46.3, 21.0, 11.9. (The boron-bound carbon was not detected due to quadrupolar relaxation)

^{11}B NMR (160 MHz, CDCl_3) δ 31.2.

IR (Microscope, cm^{-1}) 3403, 3052, 2926, 1678, 1628, 1600, 1506.

HRMS (ESI) for C₂₀H₁₉BN₂OS: calcd. 360.1468; found 360.1470.

[α]_D²⁰: -12.7 (c = 2.25, chloroform) for 82% ee.

HPLC (Chiralcel OD): 20:80 *i*-PrOH/Hexanes, 0.5 mL/minute, λ = 230 nm, T_{major} = 20.5 min, T_{minor} = 23.3 min, ee = 81%.

1.3.14 Naphthalene-1,8-diamido (dan) derivative 8d (Table 2, entry 14)

The title compound was prepared using the general procedure for the catalytic asymmetric conjugate addition. Flash column chromatography (hexanes/EtOAc = 19:1) yielded **8d** (46% yield) as a yellow oil.

¹H NMR (300 MHz, CDCl₃) δ 7.25-7.10 (m, 4H), 7.07 (dd, *J* = 8.3, 7.2 Hz, 2H), 7.01 (dd, *J* = 8.3, 1.2 Hz, 2H), 6.23 (dd, *J* = 7.2, 1.2 Hz, 2H), 5.61 (br s, 2H), 3.24-3.05 (m, 3H), 2.34 (s, 3H), 2.32 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 200.2, 140.8, 140.7, 136.2, 135.9, 127.5, 126.7, 126.6, 126.0, 119.6, 117.8, 106.0, 45.7, 20.3, 11.9. (The boron-bound carbon was not detected due to quadrupolar relaxation)

¹¹B NMR (160 MHz, CDCl₃) δ 31.3.

IR (Microscope, cm⁻¹) 3412, 3054, 2928, 1679, 1629, 1600, 1504.

HRMS (ESI) for C₂₀H₁₉BN₂OS: calcd. 360.1468; found 360.1474.

HPLC (Chiralcel OD): 20:80 *i*-PrOH/Hexanes, 0.5 mL/minute, λ = 230 nm, T = 17.5 min and 21.9 min, ee = 0%.

1.3.15 Naphthalene-1,8-diamido (dan) derivative **8e** (Table 2, entry 15)

The title compound was prepared using the general procedure for the catalytic asymmetric conjugate addition. Flash column chromatography (hexanes/EtOAc = 19:1) yielded **8e** (80% yield) as a yellow oil.

¹H NMR (300 MHz, CDCl₃) δ 7.20-7.00 (m, 8H), 6.26 (dd, *J* = 7.3, 1.0 Hz, 2H), 5.60 (br s, 2H), 3.14-2.95 (m, 3H), 2.30 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 199.6, 161.3 (d, *J* = 245.0 Hz), 140.6, 137.8 (d, *J* = 3.3 Hz), 136.3, 129.4 (d, *J* = 7.9 Hz), 127.5, 119.6, 118.0, 115.8 (d, *J* = 21.2 Hz), 106.1, 46.3, 11.9. (The boron-bound carbon was not detected due to quadrupolar relaxation)

¹¹B NMR (160 MHz, CDCl₃) δ 31.3.

¹⁹F NMR (376 MHz, CDCl₃) δ -117.3.

IR (Microscope, cm⁻¹) 3419, 3399, 3054, 2929, 1679, 1629, 1602, 1507.

HRMS (ESI) for C₂₀H₁₉BN₂OS: calcd. 364.1217; found 364.1227.

[α]_D²⁰: -16.2 (c = 2.62, chloroform) for 95% ee.

HPLC (Chiralcel OD): 20:80 *i*-PrOH/Hexanes, 0.5 mL/minute, λ = 230 nm, T_{major} = 25.4 min, T_{minor} = 30.3 min, ee = 95%.

1.3.16 Naphthalene-1,8-diamido (dan) derivative **8f** (Table 2, entry 16)

The title compound was prepared using the general procedure for the catalytic asymmetric conjugate addition. Flash column chromatography (hexanes/EtOAc = 19:1) yielded **8f** (65% yield) as a yellow oil.

¹H NMR (300 MHz, CDCl₃) δ 7.30 (m, 2H), 7.21 (m, 2H), 7.07 (dd, *J* = 8.3, 7.2 Hz, 2H), 7.00 (dd, *J* = 8.3, 0.9 Hz, 2H), 6.26 (dd, *J* = 7.2, 0.9 Hz, 2H), 5.58 (br s, 2H), 3.12-2.90 (m, 3H), 2.30 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 199.5, 140.7, 140.6, 136.2, 131.9, 129.3, 129.1, 127.5, 119.6, 118.0, 106.1, 46.0, 11.9. (The boron-bound carbon was not detected due to quadrupolar relaxation)

¹¹B NMR (160 MHz, CDCl₃) δ 31.2.

IR (Microscope, cm⁻¹) 3417, 3055, 2928, 1678, 1629, 1602, 1511.

HRMS (ESI) for C₂₀H₁₉BN₂OS: calcd. 380.0921; found 380.0929.

[α]_D²⁰: -1.8 (c = 1.42, chloroform) for 91% ee.

HPLC (Chiralcel OD): 20:80 *i*-PrOH/Hexanes, 0.5 mL/minute, λ = 366 nm, T_{major} = 27.7 min, T_{minor} = 34.7 min, ee = 91%.*

* The racemic sample prepared from *rac*-BINAP did not react completely and the product synthesized could not be separated from the starting material since they show up on the same TLC spot. Thus, a starting material peak was observed in the HPLC spectrum for the racemic sample.

1.3.17 Naphthalene-1,8-diamido (dan) derivative **8g** (Table 2, entry 17)

The title compound was prepared using the general procedure for the catalytic asymmetric conjugate addition. Flash column chromatography (hexanes/EtOAc = 19:1) yielded **8g** (50% yield) as a yellow oil.

¹H NMR (300 MHz, CDCl₃) δ 7.60 (d, *J* = 8.3 Hz, 2H), 7.31 (d, *J* = 8.3 Hz, 2H), 7.08 (m, 2H), 7.03 (m, 2H), 6.27 (d, *J* = 7.2, 2H), 5.59 (br s, 2H), 3.20-3.08 (m, 3H), 2.31 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 199.3, 146.5, 140.4, 136.2, 128.2, 127.5, 125.9 (q, *J* = 3.7 Hz), 124.2 (q, *J* = 272 Hz), 119.6, 118.1, 106.2, 45.6, 11.9. (The boron-bound carbon was not detected due to quadrupolar relaxation)

¹¹B NMR (160 MHz, CDCl₃) δ 31.0.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.7.

IR (Microscope, cm⁻¹) 3398, 3054, 2929, 1677, 1629, 1602, 1508.

HRMS (ESI) for C₂₀H₁₉BN₂OS: calcd. 414.1185; found 414.1186.

[α]_D²⁰: -1.9 (c = 1.37, chloroform) for 92.5% ee.

HPLC (Chiralcel OD): 20:80 *i*-PrOH/Hexanes, 0.5 mL/minute, λ = 230 nm, T_{major} = 28.1 min, T_{minor} = 37.7 min, ee = 92.5%.*

* The racemic sample prepared from *rac*-BINAP did not react completely and the product synthesized could not be separated from the starting material since they show up on the same TLC spot. Thus, a starting material peak was observed in the HPLC spectrum for the racemic sample.

1.3.18 Naphthalene-1,8-diamido (dan) derivative **8h** (Table 2, entry 18)

The title compound was prepared using the general procedure for the catalytic asymmetric conjugate addition. Flash column chromatography (hexanes/EtOAc = 19:1) yielded **8h** (54% yield) as a colourless oil.

¹H NMR (400 MHz, CDCl₃) δ 7.07 (dd, *J* = 8.2, 7.3 Hz, 2H), 7.00 (dd, *J* = 8.2, 0.9 Hz, 2H), 6.29 (dd, *J* = 7.2, 1.0 Hz, 2H), 5.75 (s, 2H), 4.89 (m, 1H), 4.72 (s, 1H), 2.91 (dd, *J* = 15.7, 7.4 Hz, 1H), 2.81 (dd, *J* = 15.7, 8.1 Hz, 1H), 2.40 (t, *J* = 7.7 Hz, 1H), 2.30 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 200.0, 146.6, 140.9, 136.3, 127.6, 117.8, 109.7, 106.0, 44.3, 23.7, 11.8. (The boron-bound carbon was not detected due to quadrupolar relaxation)

IR (Microscope, cm⁻¹) 3401, 3054, 2969, 2929, 1681, 1629, 1602, 1505.

HRMS (ESI) for C₁₇H₁₉BN₂OS: calcd. 310.1311; found 310.1315.

HPLC (Chiralcel OD): 20:80 *i*-PrOH/Hexanes, 0.5 mL/minute, λ = 230 nm, T = 70.4 and 76.3 min, ee = 0%.

1.4 Transformations to Other Protected Boronates

1.4.1 Methyl 3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanoate (**2a**)

To a stirred mixture of **6a** (127 mg, 0.500 mmol) in THF was added 2 M H₂SO₄ (1.0 mL, 2.0 mmol) and pinacol (177 mg, 1.50 mmol) sequentially. The reaction was stirred for 24 h at room temperature before being quenched by the addition of water (10 mL). The mixture was then extracted by diethyl ether (3 x 10 mL), dried over MgSO₄, filtered, and concentrated *in vacuo*. The crude product was then purified by flash column chromatography (hexane/EtOAc = 19:1) to give pure **2a** (98.0 mg, 90%) as a colourless oil.

¹H NMR (400 MHz, CDCl₃) δ 3.61 (s, 3H), 2.39 (d, *J* = 6.2 Hz, 1H), 2.37 (d, *J* = 4.2 Hz, 1H), 1.23-1.55 (m, 3H), 1.21 (d, *J* = 3.2 Hz, 12 H), 0.89 (t, *J* = 7.3 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 174.4, 83.1, 51.4, 35.3, 24.8, 23.6, 13.2. (The boron-bound carbon was not detected due to quadrupolar relaxation)

¹¹B NMR (160 MHz, CDCl₃) δ 33.8.

IR (Microscope, cm⁻¹) 2978, 2961, 2934, 2876, 1739.

HRMS (ESI) for C₁₂H₂₃BO₄: calcd. 242.1690; found 242.1697.

[α]_D²⁰: +14.1° (c = 1.63, chloroform).

1.4.2 Potassium trifluoro(1-methoxy-1-oxopentan-3-yl)borate (**10a**)

To a stirred solution of **2a** (1.00 g, 4.10 mmol) in methanol (8.0 mL) was added a solution of KHF_2 (1.60 g, 20.5 mmol) in H_2O (5.0 mL). The mixture was stirred for 1 hour at room temperature. The reaction mixture was then concentrated *in vacuo*, washed with hot acetone (3×20 mL). The acetone fractions were then filtered and concentrated *in vacuo* to give the crude product. The solid was further washed with hexane and diethyl ether to give trifluoroborate salt **10a** (871 mg, 95%) as a white solid.

^1H NMR (400 MHz, acetone- d_6) δ 4.90 (s, 3H), 3.57 (dd, $J = 14.0, 6.9$ Hz, 1H), 3.40 (dd, $J = 13.9, 8.2$ Hz, 1H), 2.76 (m, 1H), 2.51 (m, 1H), 2.20 (t, $J = 7.3$ Hz, 3H), 2.04 (m, 1H).

^{13}C NMR (100 MHz, acetone- d_6) δ 177.7, 50.9, 36.8, 24.7, 13.8. (The boron-bound carbon was not detected due to quadrupolar relaxation)

^{11}B NMR (160 MHz, acetone- d_6) δ 5.3.

IR (Microscope, cm^{-1}) 2955, 2937, 2917, 2856, 1727.

HRMS (ESI) for $\text{C}_6\text{H}_{11}\text{BF}_3\text{KO}_2$: calcd. 183.0811; found 183.0808.

$[\alpha]_{\text{D}}^{20}$: -7.2° ($c = 0.47$, chloroform).

M. P. 113 – 115 $^\circ\text{C}$.

1.5 Determination of Absolute Stereochemistry

1.5.1 (*S*)-Methyl 3-hydroxypentanoate (**9a**)

The title alcohol was prepared independently using the reported procedure (92%).⁴

The characterization data for this compound matched that of a previous report.⁵

The $[\alpha]_D^{20}$ of this compound was measured to be +28.9 ($c = 0.91$, chloroform), opposite to the reported for the (*R*)-isomer (−37.2, $c = 1.00$, chloroform). Thus, this proves that the compounds synthesized using our procedure from (*R*)-Tol-BINAP as the ligand have *S*-configurations on the stereogenic centre.

1.6 Enantiomeric Excess Measurement of the Oxidized Product

1.6.1 (*S*)-Methyl 3-(phenylcarbamoyloxy)pentanoate (**S2**)

To a stirred solution of alcohol **9a** (40 mg, 0.36 mmol) in dichloromethane (1.0 mL) at 0 °C was added pyridine (40 μ L, 0.54 mmol) followed by phenylisocyanate (57 μ L, 0.71 mmol). The mixture was stirred overnight while being allowed to reach room temperature. It was quenched with water and the water layer was extracted dichloromethane (3 $\times$ 5.0 mL). The combined organic phase was dried over anhydrous MgSO₄, filtered and concentrated *in vacuo*. The crude product was purified

⁴ Lee, J.-E.; Yun, J. *Angew. Chem. Int. Ed.* **2008**, 47, 145-147.

⁵ Guan, Y.; Wu, J.; Sun, L.; Dai, W.-M. *J. Org. Chem.* **2007**, 72, 4953-4960

with flash column chromatography to yield **S2** (28 mg, 37%) as a colourless oil.

¹H NMR (400 MHz, CDCl₃) δ 7.42-7.35 (m, 2H), 7.35-7.25 (m, 2H), 7.06 (tt, *J* = 7.3, 1.2 Hz, 1H), 6.24 (br s, 1H), 5.16 (m, 1H), 3.69 (s, 3H), 2.64 (m, 2H), 1.72 (m, 2H), 0.97 (t, *J* = 7.5 Hz, 3H)

¹³C NMR (100 MHz, CDCl₃) δ 170.9, 152.9, 137.8, 129.0, 123.2, 118.6, 51.8, 38.8, 27.2, 22.1, 9.4.

IR (Microscope, cm⁻¹) 3324, 3060, 2978, 2935, 2881, 1733, 1599, 1542, 1501

HRMS (ESI) for C₁₃H₁₇NO₄: calcd. 251.1158; found 251.1157.

[α]_D²⁰: −0.9 (c = 1.16, chloroform) for 96% ee.

HPLC (Chiralcel OD): 10:90 *i*-PrOH/Hexanes, 0.5 mL/minute, λ = 250 nm, T_{major} = 17.2 min, T_{minor} = 22.3 min, ee = 96%.

2. NMR Spectra for New Compounds

2.1 ^1H - and ^{13}C - NMR of 3 in CDCl_3 at 25°C

JLN-1-183B
 399.953 MHz ^1H 1D in CD_3CN
 date: Nov 6 2009 sweep width: 5206Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.2 Hz/pt hr/mm:21.7
 spectrometer:d601 file:/mnt/d600/home14/hallnmr/nmrdata/DATA_FROM_NMRSERVICE/Jack/2009.11/2009.11.06.s4_JLN-1-183B_H1_1D.fid
 File: 2009.11.06.s4_JLN-1-183B_H1_1D
 Pulse Sequence: s2pul

Jack Lee, JLN-1-183B
 100.579 MHz ^{13}C [^1H] 1D in CD_3CN
 date: Nov 6 2009 sweep width: 27211Hz acq.time: 2.0s relax.time: 0.1s # scans: 640 dig.res.: 0.4 Hz/pt hr/mm:113.4
 spectrometer:d601 file:/mnt/d600/home14/hallnmr/nmrdata/DATA_FROM_NMRSERVICE/Jack/2009.11/2009.11.06.s4_JLN-1-183B_C13_1D.fid
 File: 2009.11.06.s4_JLN-1-183B_C13_1D
 Pulse Sequence: s2pul

2.2 ^1H - and ^{13}C - NMR of 4 in CDCl_3 at 25 °C

Jack Lee, JLN-2-127B
 399.953 MHz ^1H 1D in CDCl_3
 date: Nov 6 2009 sweep width: 5206Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.2 Hz/pt hs/mm:21.7
 spectrometer:d601 file:/mnt/d600/home14/hallnmr/nmrdata/DATA_FROM_NMRSERVICE/Jack/2009.11/2009.11.06.s4_JLN-2-127B_H1_1D.fid
 File: 2009.11.06.s4_JLN-2-127B_H1_1D

Pulse Sequence: s2pul

Jack Lee, JLN-3-127B
 100.579 MHz ^{13}C 1D in CDCl_3
 date: Nov 6 2009 sweep width: 27211Hz acq.time: 2.0s relax.time: 0.1s # scans: 512 dig.res.: 0.4 Hz/pt hs/mm:113.4
 spectrometer:d601 file:/mnt/d600/home14/hallnmr/nmrdata/DATA_FROM_NMRSERVICE/Jack/2009.11/2009.11.06.s4_JLN-3-127_C13_1D.fid
 File: 2009.11.06.s4_JLN-3-127_C13_1D

Pulse Sequence: s2pul

2.3 ^1H - and ^{13}C - NMR of 5 in CDCl_3 at 25 $^\circ\text{C}$

JLN-2-162B

File: /mnt/d600/home14/hallnmr/nmrdata/Jack/JLN-2/JLN-2-162B.fid
 Date: Nov 6 2009 sweep width: 3601Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt hz/mm:15.0
 spectrometer: d601

Jack Lee, JLN-3-162B
 100.579 MHz C13[H1] 1D in CDCl3
 Date: Nov 6 2009 sweep width: 27211Hz acq.time: 2.0s relax.time: 0.1s # scans: 680 dig.res.: 0.4 Hz/pt hz/mm:113.4
 spectrometer: d601 File: /mnt/d600/home14/hallnmr/nmrdata/DATE_FROM_NMRSERVICE/Jack/2009.11/2009.11.06.s4_JLN-2-162B_C13_1D.fid
 File: 2009.11.06.s4_JLN-2-162B_C13_1D

Pulse Sequence: s2pul

2.4 ^1H - and ^{13}C - NMR of 7 in CDCl_3 at 25 $^\circ\text{C}$

Jack Lee, JLN-3-138B
 399.953 MHz ^1H 1D in CDCl_3
 date: Nov 12 2009 sweep width: 5205Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.2 Hz/pt hz/mm:21.7
 spectrometer:d601 file:/mnt/d600/home14/hallnmc/nmrdata/DATA_FROM_NMRSERVICE/Jack/2009.11/2009.11.12.s4_JLN-3-138B_H1_1D.fid
 *File: 2009.11.12.s4_JLN-3-138B_H1_1D
 Pulse Sequence: s2pul

Jack Lee, JLN-3-138B
 100.579 MHz ^{13}C 1D in CDCl_3
 date: Nov 12 2009 sweep width: 27211Hz acq.time: 2.0s relax.time: 0.1s # scans: 52 dig.res.: 0.4 Hz/pt hz/mm:113.4
 spectrometer:d601 file:/mnt/d600/home14/hallnmc/nmrdata/DATA_FROM_NMRSERVICE/Jack/2009.11/2009.11.12.s4_JLN-3-138B_C13_1D.fid
 *File: 2009.11.12.s4_JLN-3-138B_C13_1D
 Pulse Sequence: s2pul

2.5 ^1H - and ^{13}C - NMR of 6a in CDCl_3 at 25 $^\circ\text{C}$

ACA-Et-1H

Pulse sequence: zgpg30, F2 - Acquisition width: 4799Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt hz/mm:20.0
spectrometer: d501 file: /mnt/d600/home14/hallnmr/nmrdata/Jack/JLN-2/ACA-Et-1H.fid

ACA-Et-13C

Pulse sequence: zgpg30, F2 - Acquisition width: 26991Hz acq.time: 2.5s relax.time: 0.1s # scans: 360 dig.res.: 0.2 Hz/pt hz/mm:112.5
spectrometer: d501 file: /mnt/d600/home14/hallnmr/nmrdata/Jack/JLN-2/ACA-Et-13C.fid

2.6 ^1H - and ^{13}C - NMR of 6c in CDCl_3 at 25 °C

JLH-3-30B-1H

File: /mnt/d600/home14/hallnmr/nmrdata/Jack/JLH-3/JLH-3-30B-1H.fid
 Date: 20080820 14:00:00
 Spectrometer: d601
 Acquisition: 400 MHz
 Pulse program: zgpg30
 F2: 400.146 MHz
 F1: 101.625 MHz
 Aq: 5.00 s
 Relax: 0.1 s
 Scans: 16
 Dig: 0.1 Hz/pt
 Hz/mm: 20.0

JLH-3-30B-13C

File: /mnt/d600/home14/hallnmr/nmrdata/Jack/JLH-3/JLH-3-30B-13C.fid
 Date: 20080820 14:00:00
 Spectrometer: d601
 Acquisition: 125 MHz
 Pulse program: zgpg30
 F2: 125.761 MHz
 F1: 101.625 MHz
 Aq: 2.50 s
 Relax: 0.1 s
 Scans: 400
 Dig: 0.2 Hz/pt
 Hz/mm: 112.5

2.7 ^1H - and ^{13}C - NMR of 6d in CDCl_3 at 25 $^\circ\text{C}$

JLN-2-199B-1H2

Date: 2008-09-08 14:00:00 File: /mnt/d600/home14/hallnmr/nmrdata/Jack/JLN-2/JLN-2-199B-purew.fid
 spectrometer: d601

JLN-2-199B-13C

Date: 2008-09-08 14:00:00 File: /mnt/d600/home14/hallnmr/nmrdata/Jack/JLN-2/JLN-2-199B-13C.fid
 spectrometer: d601

2.8 ^1H - and ^{13}C - NMR of 6e in CDCl_3 at 25 °C

JLN-2-206B-1H

File: /mnt/d600/home14/hallnuz/nmrdata/Jack/JLN-2/JLN-2-206B-1H.fid
 Date: 20200820 14:00:00
 Spectrometer: d601
 Pulse sequence: zgpg30
 F2: 479.925 MHz
 F1: 101.625 MHz
 Aq: 5.00 s
 Relax: 0.10 s
 Scans: 16
 Dig. res.: 0.1 Hz/pt
 Hz/mm: 20.0

JLN-2-206B-13C

File: /mnt/d600/home14/hallnuz/nmrdata/Jack/JLN-2/JLN-2-206B-13C.fid
 Date: 20200820 14:00:00
 Spectrometer: d601
 Pulse sequence: zgpg30
 F2: 101.625 MHz
 F1: 269.135 MHz
 Aq: 2.50 s
 Relax: 0.10 s
 Scans: 204
 Dig. res.: 0.2 Hz/pt
 Hz/mm: 112.5

JLH-3-17B

```

=====
data: sequence: 2002, sweep width: 4799Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt hz/mm: 20.0
spectrometer: d601 file: /mnt/d600/home14/hallnmr/nmrdata/Jack/JLH-3/JLH-3-17B-1H.fid
=====

```

JLH-3-17B-13C

```

Date_# 20080220 10:00:00 1000 MHz spectrometer: d601
file: /mnt/d600/home14/hallnmr/nmrdata/Jack/JLH-3/JLH-3-17B-13C.fid

```

2.10 ^1H - and ^{13}C - NMR of 6g in CDCl_3 at 25 °C

JLN-3-22B-1H

Date Acquired: 20080220 Exp width: 4795Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt hz/mm:20.0
spectrometer: d601 file: /mnt/d600/home14/hallnmr/nmrdata/Jack/JLN-3/JLN-3-22B-1H.fid

JLN-3-22B-13C

Date Acquired: 20080220 Exp width: 26951Hz acq.time: 2.5s relax.time: 0.1s # scans: 384 dig.res.: 0.2 Hz/pt hz/mm:112.5
spectrometer: d601 file: /mnt/d600/home14/hallnmr/nmrdata/Jack/JLN-3/JLN-3-22B-13C.fid

2.11 ^1H - and ^{13}C - NMR of 6h in CDCl_3 at 25 $^\circ\text{C}$

JLN-2-203B-1H

data acquisition: 400 MHz, pulse width: 4799 Hz, acq. time: 5.0 s, relax. time: 0.1 s, # scans: 16, dig. res.: 0.1 Hz/pt, hz/mm: 20.0
spectrometer: d601 file: /mnt/d600/home14/hallnmr/nmrdata/Jack/JLN-2/JLN-2-203B-1H.fid

JLN-2-203B-13C

data acquisition: 400 MHz, pulse width: 2699 Hz, acq. time: 2.5 s, relax. time: 0.1 s, # scans: 340, dig. res.: 0.2 Hz/pt, hz/mm: 112.5
spectrometer: d601 file: /mnt/d600/home14/hallnmr/nmrdata/Jack/JLN-2/JLN-2-203B-13C.fid

2.12 ^1H - and ^{13}C - NMR of 6i in CDCl_3 at 25 $^\circ\text{C}$

399.794 MHz ^1H 1D in cdcl_3 (ref. to CDCl_3 @ 7.26 ppm), temp 26.5 C -> actual temp = 27.0 C, autotxmb probe
 date: Aug 11 2009 sweep width: 4799Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt hz/mm:20.0
 spectrometer:d601 file:/mnt/d600/home14/hallnmr/nmrdata/Jack/JLN-3/JLN-3-27B-1H.fid
 pulse sequence: zgpg30

JLN-3-27B-13C

date: Aug 11 2009 sweep width: 26991Hz acq.time: 2.5s relax.time: 0.1s # scans: 428 dig.res.: 0.2 Hz/pt hz/mm:112.5
 spectrometer:d601 file:/mnt/d600/home14/hallnmr/nmrdata/Jack/JLN-3/JLN-3-27B-13C.fid

2.13 ^1H - and ^{13}C - NMR of 6j in CDCl_3 at 25 $^\circ\text{C}$

JLN-2-185B

Pulse Sequence: zgpg30 sweep width: 3601Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt hz/mm:15.0
spectrometer: d601 file: /mnt/d600/home14/hallnmc/nmrdata/Jack/JLN-2/JLN-2-185B-2.fid

Jack Lee, JLN-2-185B
100.579 MHz ^{13}C [M1] 1D in CDCl_3
date: Nov 6 2009 sweep width: 27211Hz acq.time: 2.0s relax.time: 0.1s # scans: 11440 dig.res.: 0.4 Hz/pt hz/mm:113.4
spectrometer: d601 file: /mnt/d600/home14/hallnmc/nmrdata/DATA_FROM_NMRSERVICE/Jack/2009.11/2009.11.06.s4_JLN-2-185B_C13_1D.fid
File: 2009.11.06.s4_JLN-2-185B_C13_1D

Pulse Sequence: s2pul

2.14 ^1H - and ^{13}C - NMR of 8a in CDCl_3 at 25 $^\circ\text{C}$

JLN-3-140B-1H

Pulse Sequence: zgpg30
Sweep width: 3601Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt hz/mm:15.0
spectrometer:d601 file:/mnt/d600/home14/hallnmr/nmrdata/Jack/JLN-3/JLN-3-140B-1H.fid

Jack Lee, JLN-3-140B-20C
100.579 MHz C13[H1] 1D in CDCl3
date: Nov 17 2009 sweep width: 27211Hz acq.time: 2.0s relax.time: 0.1s # scans: 256 dig.res.: 0.4 Hz/pt hz/mm:113.4
spectrometer:d601 file:/mnt/d600/home14/hallnmr/nmrdata/DATA_FROM_NMRSERVICE/Jack/2009.11/2009.11.17.s4_JLN-3-140B-20C_C13_1D.fid
File: 2009.11.17.s4_JLN-3-140B-20C_C13_1D

2.15 ^1H - and ^{13}C - NMR of 8b in CDCl_3 at 25 $^\circ\text{C}$

Jack Lee, JLN-3-139B
 399.953 MHz ^1H 1D in CDCl_3
 date: Nov 12 2009 sweep width: 5206Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.2 Hz/pt hz/mm:21.7
 spectrometer: d601 file: /mnt/d600/home14/hallnmr/nmrdata/DATA_FROM_NMRSERVICE/Jack/2009.11/2009.11.12.s4_JLN-3-139B_H1_1D.fid
 File: 2009.11.12.s4_JLN-3-139B_H1_1D
 Pulse Sequence: s2pul

Jack Lee, JLN-3-139B
 100.579 MHz ^{13}C [M1] 1D in CDCl_3
 date: Nov 12 2009 sweep width: 27211Hz acq.time: 2.0s relax.time: 0.1s # scans: 1040 dig.res.: 0.4 Hz/pt hz/mm:113.4
 spectrometer: d601 file: /mnt/d600/home14/hallnmr/nmrdata/DATA_FROM_NMRSERVICE/Jack/2009.11/2009.11.12.s4_JLN-3-139B_C13_1D.fid
 File: 2009.11.12.s4_JLN-3-139B_C13_1D
 Pulse Sequence: s2pul

2.16 ^1H - and ^{13}C - NMR of 8c in CDCl_3 at 25 $^\circ\text{C}$

Jack Lee, JLN-3-199B
 399.953 MHz ^1H 1D in CDCl_3
 date: Feb 11 2010 sweep width: 5206Hz acq.time: 5.0s relax.time: 0.1s # scans: 32 dig.res.: 0.2 Hz/pt hz/mm:21.7
 spectrometer:dg601 file:/mm4/4600/home14/hallnmr/nmrdata/DATA_FROM_NMRSERVICE/Jack/2010.02/2010.02.11.s4_JLN-3-199B_H1_1D.fid
 File: 2010.02.11.s4_JLN-3-199B_H1_1D

Pulse Sequence: s2pul

Jack Lee, JLN-3-199B
 100.579 MHz ^{13}C [H1] 1D in CDCl_3
 date: Feb 11 2010 sweep width: 27211Hz acq.time: 2.0s relax.time: 0.4s # scans: 1056 dig.res.: 0.4 Hz/pt hz/mm:113.4
 spectrometer:dg601 file:/mm4/4600/home14/hallnmr/nmrdata/DATA_FROM_NMRSERVICE/Jack/2010.02/2010.02.11.s4_JLN-3-199B_C13_1D.fid
 File: 2010.02.11.s4_JLN-3-199B_C13_1D

Pulse Sequence: s2pul

2.17 ^1H - and ^{13}C - NMR of 8d in CDCl_3 at 25 $^\circ\text{C}$

JLH-3-1908-1H
299.971 MHz ^1H 1D in cdcl_3 (ref. to CDCl_3 @ 7.26 ppm), temp 27.5 C -> actual temp = 27.0 C, id300 probe
Date: 08/08/2010 10:00:00 file: /mnt/d600/homel4/hallner/nmrdata/Jack/JLH-3/JLH-3-1908
Spectrometer: u500
Pulse Sequence: s2pu1

JLH-3-1908-13C
125.690 MHz ^{13}C 1D in cdcl_3 (ref. to CDCl_3 @ 77.66 ppm), temp 27.3 C -> actual temp = 27.0 C, cold dual probe
Date: 08/08/2010 10:00:00 file: /mnt/d600/homel4/hallner/nmrdata/Jack/JLH-3/JLH-3-1908
Spectrometer: u500
Pulse Sequence: s2pu1

2.18 ^1H - and ^{13}C - NMR of 8e in CDCl_3 at 25 $^\circ\text{C}$

JLH-3-1768-1
299.371 MHz ^1H 1D in cdcl_3 (ref. to CDCl_3 @ 7.26 ppm), temp 27.5 C -> actual temp = 27.0 C, id300 probe
date: Feb 24 2010 sweep width: 3601Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt hz/mm:15.0
spectrometer:1300 file:/mnt/d600/home14/hallnmr/nmrdata/Jack/JLH-3/JLH-3-1768-1.fid

Pulse Sequence: s2pu1

Jack Lee, JLH-3-1768
100.578 MHz ^{13}C 1D in CDCl_3

expt1 Carbon

SAMPLE		SPECIAL	
date	Feb 11 2010	temp	not used
solvent	CDCl_3	gain	60
file	/mnt/d600/home14/hallnmr/nmrdata/Jack/JLH-3/JLH-3-1768-1.fid	spin	not used
id3	grommr/nmrdata= hst	0.008	
/HALL/Jack/2010.02- pu90		9.300	
/2010.02-11.4.4 JLH-	alpha	10.000	
-3-1768.C13.10.fid			
ACQUISITION		il	n
sw	27210.8	in	n
at	1.932	dp	y
np	108460	hs	nn
fb	not used		
bs	4	lb	2.00
ss	2	fn	not used
dl	0.100		
nt	1e+06	sp	-2058.7
ct	1556	wp	27210.5
TRANSMITTER		rf1	8008.8
tn	C13	rffp	7749.7
tfreq	100.578	rp	-79.2
tof	2048.7	lp	-87.2
tpwr	55		
pw	3.100	vc	180
DECOUPLER		sc	0
dn	H1	vs	4267
dof	0	th	5
dm	yyy	al	cdc ph
dmm	w		
dpr	41		
dms	10101

2.19 ^1H - and ^{13}C - NMR of 8f in CDCl_3 at 25 $^\circ\text{C}$

Jack Lee, JLN-3-1888
499.815 MHz ^1H 1D in cdcl_3 (ref. to CDCl_3 @ 7.26 ppm), temp 26.1 C -> actual temp = 27.0 C, autokdb probe
date: Feb 16 2010 sweep width: 5010Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt hz/mm:25.0
spectrometer:1400 file:/mnt/d600/home14/hallnir/nmrdata/DATA_FROM_NMRSERVICE/Jack/2010.02/2010.02.16.u5_JLN-3-1888_H1_1D.fid
Pulse Sequence: s2pul

Jack Lee, JLN-3-1888
125.690 MHz ^{13}C 1D in cdcl_3 (ref. to CDCl_3 @ 77.06 ppm), temp 26.1 C -> actual temp = 27.0 C, autokdb probe
date: Feb 16 2010 sweep width: 33784Hz acq.time: 2.5s relax.time: 0.1s # scans: 4284 dig.res.: 0.3 Hz/pt hz/mm:140.6
spectrometer:d601 file:/mnt/d600/home14/hallnir/nmrdata/DATA_FROM_NMRSERVICE/Jack/2010.02/2010.02.16.u5_JLN-3-1888_C13_1D.fid
Pulse Sequence: s2pul

2.20 ^1H - and ^{13}C - NMR of 8g in CDCl_3 at 25 $^\circ\text{C}$

JLN-4-17B-1H
499.815 MHz ^1H 1D in cdcl_3 (ref. to CDCl_3 @ 7.26 ppm), temp 26.1 C -> actual temp = 27.0 C, autokdb probe
date: Feb 20 2010 sweep width: 6010Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt hz/mm:25.0
spectrometer:d601 file:/mnt/d600/home14/hallnuz/nmrdata/Jack/JLN-4/JLN-4-17B-1H.fid
Pulse Sequence: s2pul

Jack Lee, JLN-4-17B
100.579 MHz ^{13}C 1D in CDCl_3
date: Feb 23 2010 sweep width: 27211Hz acq.time: 2.0s relax.time: 0.1s # scans: 30788 dig.res.: 0.2 Hz/pt hz/mm:113.4
spectrometer:d601 file:/mnt/d600/home14/hallnuz/nmrdata/Jack/2010.02/2010.02.24.s4_JLN-4-17B-1H.fid
Pulse Sequence: s2pul

2.21 ^1H - and ^{13}C - NMR of 8h in CDCl_3 at 25 $^\circ\text{C}$

JLN-3-141B-2

File Name: 2009.11.16.m4
Sweep width: 3601Hz acq.time: 5.0s relax.time: 0.1s # scans: 12 dig.res.: 0.1 Hz/pt hz/mm: 15.0
spectrometer: d601 file: /mnt/d600/home14/hallnmr/nmrdata/Jack/JLN-3/JLN-3-141B-2.fid

Jack_Lee, JLN-3-141B
100.690 MHz C13[M1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Nov 16 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 1000 dig.res.: 0.2 Hz/pt hz/mm: 113.2
spectrometer: d601 file: /mnt/d600/home14/hallnmr/nmrdata/DATA_FROM_NMRSERVICE/Jack_Lee/2009.11.16.m4 JLN-3-141B_12.53_C13_1D.fid
pulse sequence: s2pul

2.22 ^1H - and ^{13}C - NMR of 2a in CDCl_3 at 25 $^\circ\text{C}$

JLH-3-64B-1H

Pulse Sequence: zgpg30 sweep width: 3601Hz acq.time: 5.0s relax.time: 0.1s # scans: 8 dig.res.: 0.1 Hz/pt hz/mm:15.0
spectrometer:d601 file:/mnt/d600/home14/hallnmr/nmrdata/Jack/JLH-3/JLH-3-64B-1H.fid

Jack Lee, JLH-3-64B
100.579 MHz ^{13}C [H1] 1D in CDCl_3
date: Nov 9 2009 sweep width: 27211Hz acq.time: 2.0s relax.time: 0.1s # scans: 2424 dig.res.: 0.4 Hz/pt hz/mm:113.4
spectrometer:d601 file:/mnt/d600/home14/hallnmr/nmrdata/DATA_FROM_NMRSERVICE/Jack/2009.11/2009.11.09.s4_JLH-3-64B_13_1D.fid
File: 2009.11.09.s4_JLH-3-64B_13_1D

2.23 ^1H - and ^{13}C - NMR of 10a in CDCl_3 at 25 °C

Jack Lee, JLN-3-13B
 399.951 MHz ^1H 1D in CDCl_3 recorded on s400
 date: Nov 9 2009 sweep width: 6001Hz acq.time: 3.0s relax.time: 2.0s # scans: 16 dig.res.: 0.1 Hz/pt hz/mm:25.0
 pps: 1000000000 file: /mnt/d600/home14/hallnmr/nmrdata/DATA_FROM_NMRSERVICE/Jack/2009.11/2009.11.09.s4_JLN-3-13B_H1_1D.fid
 Pulse Sequence: zgpg30

Jack Lee, JLN-3-13B
 125 MHz ^{13}C 1D in D_2O (ref. to 1% ext. acetone @ 31.07 ppm), temp = 27.2 C -> actual temp = 27.0 C, sw probe
 date: Nov 9 2009 sweep width: 26144Hz acq.time: 2.5s relax.time: 0.1s # scans: 772 dig.res.: 0.2 Hz/pt hz/mm:106.9
 pps: 1000000000 file: /mnt/d600/home14/hallnmr/nmrdata/DATA_FROM_NMRSERVICE/Jack/2009.11/2009.11.09.s4_JLN-3-13B_C13_1D.fid
 Pulse Sequence: zgpg30

JLK-2-16B-1H

```

Date_Rep: 20080222 10:00:00 10:00:00 10:00:00 10:00:00 10:00:00 10:00:00 10:00:00 10:00:00 10:00:00
Spectrometer: d601  file: /mnt/d600/home14/hallinmr/nmrdata/Jack/JLN-2/JLN-2-16C.fid  dig.res.: 0.1 Hz/pt  hz/mm: 15.0

```

JLH-2-16B-13C

```
File Name: /mnt/ds60/home14/hallnmr/nmrdata/Jack/JLH-2/JLH-2-16B-13C.fid  
Spectrometer: ds60
```

3. Chromatograms for Enantiomeric Excess Measurement

3.1 Racemic (top) and optically enriched (bottom) 6a

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	37.647	BB	1.1557	8283.98828	101.34283	52.0144
2	56.605	BB	1.4143	7642.34229	64.68838	47.9856

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	37.997	MM	1.6168	738.94153	7.61754	1.9557
2	53.666	BB	1.6385	3.70449e4	264.74158	98.0443

3.2 Racemic (top) and optically enriched (bottom) 6c

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.714	BB	0.8094	1.93233e4	355.91275	48.5495
2	31.914	BB	1.1397	2.04779e4	251.02179	51.4505

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.849	BB	0.5667	347.61389	7.31149	2.4824
2	32.001	BB	1.0938	1.36555e4	175.87646	97.5176

3.3 Racemic (top) and optically enriched (bottom) 6d

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.084	BB	0.7582	6.82921e4	1293.07446	49.2293
2	30.393	BB	1.1397	7.04303e4	887.59790	50.7707

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.284	PB	0.6565	638.73499	12.74961	2.5613
2	30.613	BB	1.0576	2.42994e4	336.84665	97.4387

3.4 Racemic (top) and optically enriched (bottom) 6e

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	23.095	BB	0.7293	3827.79321	76.82402	50.6398
2	37.402	BB	1.0992	3731.07593	45.19379	49.3602

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	23.147	MM	0.7298	114.98170	2.62578	1.0554
2	37.129	BB	1.1021	1.07797e4	121.14185	98.9446

3.5 Racemic (top) and optically enriched (bottom) 6f

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	23.371	BB	0.7240	2225.78101	44.92277	45.0562
2	30.987	BB	0.9836	2714.23071	39.79907	54.9438

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	23.337	BB	0.5788	224.65274	4.64008	2.1538
2	30.781	BB	1.0391	1.02060e4	138.92261	97.8462

3.6 Racemic (top) and optically enriched (bottom) 6g

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	29.957	BB	1.0866	2.56397e4	345.98486	50.2981
2	46.899	VB	1.7893	2.53358e4	207.08858	49.7019

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	30.070	MM	0.6421	166.02592	3.10270	0.7437
2	46.666	BB	1.6920	2.21577e4	185.98195	99.2563

3.7 Racemic (top) and optically enriched (bottom) 6h

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.874	BB	0.7025	5.66745e4	1201.02454	48.8554
2	33.054	BB	1.1739	5.93300e4	712.16003	51.1446

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.965	BB	0.5699	1123.88794	28.51271	4.5149
2	33.149	BB	1.1135	2.37691e4	310.35181	95.4851

3.8 Racemic (top) and optically enriched (bottom) 6i

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.875	BB	0.7002	3.40113e4	715.98181	49.6012
2	34.180	BB	1.2884	3.45582e4	384.55212	50.3988

=====
Area Percent Report
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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.981	PB	0.6331	2788.42285	65.70131	4.3481
2	34.005	BB	1.3186	6.13416e4	642.07251	95.6519

3.9 Racemic (top) and optically enriched (bottom) 6j

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	51.446	BB	1.5105	1.83219e4	147.16376	50.4297
2	58.196	BB	1.6215	1.80097e4	132.51761	49.5703

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	51.231	BB	1.5642	2.89322e4	219.16153	94.8157
2	58.689	BB	1.3103	1581.94666	14.21555	5.1843

3.10 Racemic (top) and optically enriched (bottom) 8a

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Area Percent Report
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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	38.046	BV	1.0781	7100.67334	96.76659	49.0303
2	40.540	VB	1.1959	7381.54834	90.09537	50.9697

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Area Percent Report
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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	36.819	MM	0.9888	45.87392	7.73260e-1	0.7971
2	39.680	BB	1.1646	5709.34766	72.03846	99.2029

3.11 Racemic (top) and optically enriched (bottom) 8b

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	23.222	PB	0.6850	648.40863	13.15651	52.0059
2	26.589	BB	0.7473	598.39075	10.85199	47.9941

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	23.487	BB	0.7562	4337.98389	84.88074	95.6607
2	27.101	BB	0.5896	196.77542	3.93347	4.3393

3.12 Racemic (top) and optically enriched (bottom) 8c

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.432	BB	0.6676	2656.01514	58.95552	50.6887
2	23.253	BB	0.7475	2583.83838	51.31194	49.3113

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.462	BB	0.6563	2200.04907	48.58555	90.9437
2	23.314	BB	0.5748	219.08279	5.10378	9.0563

3.13 Racemic (top) and optically enriched (bottom) 8d

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.677	VB	0.5782	5984.54590	149.75688	50.5092
2	22.224	VB	0.7324	5863.89160	116.66917	49.4908

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.482	BB	0.5376	1530.99463	41.38914	50.9446
2	21.926	PB	0.6641	1474.22083	31.84567	49.0554

3.14 Racemic (top) and optically enriched (bottom) **8e**

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Area Percent Report
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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	26.292	PB	0.8565	3949.23560	68.30573	49.8803
2	31.768	BV	1.0005	3968.19629	58.56582	50.1197

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Area Percent Report
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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	25.382	BB	0.8295	5012.73633	89.77046	97.8659
2	30.325	MM	0.7299	109.31055	2.49607	2.1341

3.15 Racemic (top) and optically enriched (bottom) 8f

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	26.292	PB	0.8565	3949.23560	68.30573	51.0339
2	34.420	VB	1.0311	3789.22339	53.58016	48.9661

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.656	BB	0.8642	1500.22412	25.21399	95.6650
2	34.729	MM	0.9086	67.98183	1.24695	4.3350

3.16 Racemic (top) and optically enriched (bottom) 8g

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.608	BB	0.9609	3190.98853	48.92805	52.6285
2	36.687	PB	1.2198	2872.24609	34.55402	47.3715

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	28.103	BB	1.0685	1.56750e4	211.54683	96.2063
2	37.737	BB	0.9458	618.11810	7.82331	3.7937

3.17 Racemic (top) and optically enriched (bottom) 8h

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Area Percent Report
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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	70.517	MM	2.3301	957.71155	6.85020	50.7269
2	77.070	MM	2.5817	930.26257	6.00542	49.2731

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Area Percent Report
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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	70.418	BB	2.0588	2.10567e4	147.73604	48.0149
2	76.368	BB	2.3910	2.27978e4	132.12747	51.9851

3.18 Racemic (top) and optically enriched (bottom) S2

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.564	BB	0.5761	8840.79102	229.13657	50.0316
2	22.938	BB	0.8471	8829.63672	156.75803	49.9684

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.163	BB	0.3925	119.28202	4.03972	1.8353
2	22.348	BB	0.6704	6380.18994	138.26604	98.1647