Diastereo- and Enantioselective synthesis of
 α,γ -Diaminobutyric Acid Derivatives via Cu-Catalyzed
Asymmetric Michael Reaction

Qing Li, Chang-Hua Ding, Xue-Long Hou* and Li-Xin Dai

*State Key Laboratory of Organometallic Chemistry, Shanghai Institute of Organic
Chemistry, Chinese Academy of Sciences, 354 Fenglin Road, Shanghai 200032, China*

E-mail: xlhoul@mail.sioc.ac.cn

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1. General

1-1. General methods

Unless stated otherwise, all reactions were carried out in flame-dried glassware under a dry argon atmosphere. All solvents were purified and dried according to standard methods prior to use. The ligands **L1-L5** were prepared according to literature procedures.^[1] Nitroalkenes **2a-l** were prepared using the literature procedures.^[2]

1-2. Instrumentation

¹H and ¹³C NMR spectra were recorded on a NMR instrument. Data for ¹H NMR are recorded as follows: chemical shift (δ, ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet or unresolved, br = broad singlet, coupling constant(s) in Hz, integration). Data for ¹³C NMR are reported in terms of chemical shift (δ, ppm).

2. Experimental Procedure and Characterization

2-1. General Procedure for the Catalytic Reactions.

To a flame-dried Schlenk tube, a stirring bar was added $\text{CuClO}_4 \cdot (\text{CH}_3\text{CN})_4$ (3.3 mg, 0.01 mmol) and the phosphino-oxazoline ligand (0.01 mmol) under argon atmosphere. The mixture was dried for 30 min under vacuum with stirring, then freshly distilled anhydrous THF (1 mL) was added. After being stirred for 30 min, the solution was cooled to -10°C before the glycine derivatives **1** was added (0.1 mmol), followed by Et_3N (10 μL , 1M in THF) and by the nitroalkenes **2** (0.1 mmol). The reaction mixture was stirred at -10°C , then filtered through a short plug of silica gel. After evaporation the solvent, the crude product was analyzed by ^1H NMR spectroscopy to determine the diastereomeric ratio, and then purified by chromatography on silica gel (EtOAc/petroleum mixtures).

2-2. Characterization of the Products

tert-Butyl-2-(diphenylmethyleamino)-4-nitro-3-phenylbutanoate (**6a**)

97% ee, dr = 94:6, $[\alpha]_{\text{D}}^{20} = -188.3^\circ$ ($c = 1.03$, CHCl_3); ^1H NMR (400 MHz, CDCl_3): δ 1.36 (s, 9H), 4.17 (d, $J = 4.4$ Hz, 1H), 4.27-4.32 (m, 1H), 5.09-5.12 (m, 2H), 6.63 (d, $J = 7.2$ Hz, 2H), 7.12-7.15 (m, 2H), 7.21-7.44 (m, 9H), 7.63-7.65 (m, 2H); ^{13}C NMR

(100 MHz, CDCl₃): δ 27.8, 46.8, 69.2, 76.4, 82.2, 127.3, 127.7, 128.1, 128.2, 128.3, 128.5, 128.6, 128.8, 130.7, 135.5, 137.4, 138.8, 168.9, 172.3; IR (thin film): 1720, 1550, 1381, 1370, 1240, 1152, 783, 765, 697; MS (ESI): 445.2 (M+1); HRMS (ESI): C₂₇H₂₉N₂O₄, Calc: 445.2118, Found: 445.2122; HPLC (chiralpak IC, hexane/2-propanol = 90/10, Flow rate = 0.7 mL/min): t_R (minor) 13.2 min, t_R (major) 15.6 min.

***tert*-Butyl-3-(4-bromophenyl)-2-(diphenylmethyleamino)-4-nitrobutanoate (6b)**

97% ee, dr = 95:5, $[\alpha]_D^{20} = -121.4^\circ$ (c = 1.32, CHCl₃); ¹H NMR (300 MHz, CDCl₃): δ 1.38 (s, 9H), 4.14 (d, *J* = 4.5 Hz, 1H), 4.22-4.30 (m, 1H), 5.07-5.11 (m, 2H), 6.67 (d, *J* = 6.6 Hz, 2H), 7.02 (d, *J* = 8.1 Hz, 2H), 7.25-7.48 (m, 8H), 7.62 (d, *J* = 7.2 Hz, 2H); ¹³C NMR (75 MHz, CDCl₃): δ 27.8, 46.2, 68.8, 76.1, 82.4, 121.7, 127.2, 128.2, 128.3, 128.7, 128.8, 130.0, 130.9, 131.7, 135.3, 136.5, 138.6, 168.6, 172.6; IR (thin film): 1732, 1554, 1370, 1280, 1150, 1075, 1012, 697; MS (ESI): 523.1 (M+1); HRMS (ESI): C₂₇H₂₈BrN₂O₄, Calc: 523.1225, Found: 523.1227; HPLC (chiralpak IC, hexane/2-propanol = 90/10, Flow rate = 0.7 mL/min): t_R (minor) 12.8 min, t_R (major) 14.4 min.

***tert*-Butyl-3-(4-chlorophenyl)-2-(diphenylmethyleamino)-4-nitrobutanoate (6c)**

98% ee, dr = 98:2, $[\alpha]_D^{20} = -157.1^\circ$ (c = 1.14, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 1.38 (s, 9H), 4.14 (d, *J* = 4.4 Hz, 1H), 4.26-4.28 (m, 1H), 5.07-5.09 (m, 2H), 6.69 (d, *J* = 6.8 Hz, 2H), 7.08-7.44 (m, 10H), 7.62 (d, *J* = 7.2 Hz, 2H); ¹³C NMR (75 MHz, CDCl₃): δ 27.8, 46.2, 68.9, 76.3, 82.4, 127.2, 128.2, 128.3, 128.7, 128.7, 128.8, 129.6, 130.9, 133.6, 135.4, 136.0, 138.6, 168.6, 172.6; IR (thin film): 1733, 1554, 1493, 1447, 1370, 1288, 1152, 701; MS (ESI): 479.3 (M+1); HRMS (ESI): C₂₇H₂₈N₂O₄Cl, Calc: 479.1729, Found: 479.1732; HPLC (chiralpak IC, hexane/2-propanol = 80/20, Flow rate = 0.7 mL/min): t_R (minor) 9.0 min, t_R (major) 10.0 min.

***tert*-Butyl-2-(diphenylmethyleamino)-3-(4-fluorophenyl)-4-nitrobutanoate (6d)**

97% ee, dr = 98:2, $[\alpha]_D^{20} = -242.0^\circ$ (c = 0.43, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 1.37 (s, 9H), 4.14 (d, *J* = 4.4 Hz, 1H), 4.25-4.30 (m, 1H), 5.05-5.07 (m, 2H), 6.70 (d, *J* = 6.8 Hz, 2H), 6.91-6.96 (m, 2H), 7.11-7.14 (m, 2H), 7.29-7.46 (m, 6H), 7.63 (d, *J* = 7.2 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 27.8, 46.2, 69.1, 76.6, 82.3, 115.4 (d, *J* = 20.9 Hz), 127.3, 128.2, 128.3, 128.7, 128.8, 129.9, 130.0, 130.9, 133.2 (d, *J* = 3 Hz), 135.5, 138.7, 161.0 (d, *J* = 245.4 Hz), 168.7, 172.5; IR (thin film): 1722, 1550, 1512, 1242, 1223, 1152, 847, 700; MS (ESI): 463.1 (M+1); HRMS (ESI): C₂₇H₂₈N₂O₄F, Calc: 463.2017, Found: 463.2028; HPLC (chiralpak AD-H, hexane/2-propanol = 90/10, Flow rate = 0.7 mL/min): t_R (major) 8.5 min, t_R (minor) 11.0 min.

***tert*-Butyl-2-(diphenylmethyleamino)-3-(4-methoxyphenyl)-4-nitrobutanoate (6e)**

98% ee, dr = 98:2, $[\alpha]_D^{20} = -172.8^\circ$ (c = 1.11, CHCl₃); ¹H NMR (300 MHz, CDCl₃): δ 1.37 (s, 9H), 3.75 (s, 3H), 4.15 (d, *J* = 4.5 Hz, 1H), 4.24-4.26 (m, 1H), 5.04-5.07 (m, 2H), 6.70-6.80 (m, 4H), 7.06 (d, *J* = 8.4 Hz, 2H), 7.29-7.46 (m, 6H), 7.64 (d, *J* = 7.2 Hz, 2H); ¹³C NMR (75 MHz, CDCl₃): δ 27.8, 46.2, 55.2, 69.3, 76.6, 82.1, 113.9, 127.4, 128.1, 128.3, 128.6, 128.8, 129.2, 129.3, 130.7, 135.6, 138.8, 159.0, 168.9, 172.2; IR (thin film): 1731, 1554, 1515, 1281, 1253, 1180, 1151, 697; MS (ESI): 475.2 (M+1); HRMS (ESI): C₂₈H₃₁N₂O₅, Calc: 475.2228, Found: 475.2228; HPLC (chiralpak IC, hexane/2-propanol = 90/10, Flow rate = 0.7 mL/min): *t*_R (minor) 19.0 min, *t*_R (major) 22.2 min.

***tert*-Butyl-2-(4-methylphenyl)-3-((diphenylmethyleneamino)oxy)-4-nitrobutanoate (6f)**

97% ee, dr = 95:5, $[\alpha]_D^{20} = -150.8^\circ$ (c = 1.27, CHCl₃); ¹H NMR (300 MHz, CDCl₃): δ 1.37 (s, 9H), 2.29 (s, 3H), 4.15 (d, *J* = 4.8 Hz, 1H), 4.22-4.30 (m, 1H), 5.03-5.14 (m, 2H), 6.65 (d, *J* = 6.9 Hz, 2H), 7.03 (s, 4H), 7.36-7.48 (m, 6H), 7.63 (d, *J* = 7.2 Hz, 2H); ¹³C NMR (75 MHz, CDCl₃): δ 21.0, 27.8, 46.5, 69.2, 71.7, 82.1, 127.4, 128.1, 128.2, 128.5, 128.8, 129.2, 130.7, 134.2, 135.5, 137.3, 138.8, 168.9, 172.1; IR (thin film): 1733, 1554, 1447, 1370, 1317, 1290, 1151, 697; MS (ESI): 459.2 (M+1); HRMS (ESI): C₂₈H₃₁N₂O₄, Calc: 459.2277, Found: 459.2278; HPLC (chiralpak AD-H, hexane/2-propanol = 90/10, Flow rate = 0.7 mL/min): *t*_R (major) 7.7 min, *t*_R (minor) 8.9 min.

***tert*-Butyl-3-(3-bromophenyl)-2-(diphenylmethyleamino)-4-nitrobutanoate (6g)**

98% ee, dr = 92:8, $[\alpha]_D^{20} = -186.8^\circ$ (c = 1.04, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 1.39 (s, 9H), 4.14 (d, *J* = 4.8 Hz, 1H), 4.23-4.27 (m, 1H), 5.06-5.10 (m, 2H), 6.67 (d, *J* = 7.2 Hz, 2H), 7.09-7.46 (m, 10H), 7.61-7.64 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 27.8, 46.4, 68.8, 76.0, 82.5, 122.6, 126.5, 127.2, 128.1, 128.3, 128.7, 128.8, 130.1, 130.8, 130.8, 131.7, 135.4, 138.6, 139.8, 168.6, 172.7; IR (thin film): 1733, 1596, 1554, 1477, 1447, 1370, 1151, 696; MS (ESI): 523.1 (M+1); HRMS (ESI): C₂₇H₂₈N₂O₄Br, Calc: 523.1225, Found: 523.1227; HPLC (chiralpak IC, hexane/2-propanol = 90/10, Flow rate = 0.7 mL/min): t_R (minor) 12.2 min, t_R (major) 15.1 min.

***tert*-Butyl-2-(diphenylmethyleamino)-3-(3-methoxyphenyl)-4-nitrobutanoate (6h)**

97% ee, dr = 96:4, $[\alpha]_D^{20} = -182.9^\circ$ (c = 1.18, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 1.38 (s, 9H), 3.64 (s, 3H), 4.17 (d, *J* = 4.4 Hz, 1H), 4.24-4.29 (m, 1H), 5.06-5.15 (m, 2H), 6.66 (s, 2H), 6.72-6.78 (m, 2H), 7.12-7.47 (m, 8H), 7.63 (d, *J* = 7.2 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 27.8, 46.7, 55.0, 69.1, 76.3, 82.2, 113.5, 113.7, 120.4, 127.3, 128.1, 128.2, 128.5, 128.8, 129.6, 130.7, 135.5, 138.8, 138.9, 159.6, 168.9, 172.2; IR (thin film): 1733, 1554, 1491, 1370, 1280, 1261, 1151, 697; MS (ESI):

475.1 (M+1); HRMS (ESI): C₂₈H₃₁N₂O₅, Calc: 475.2225, Found: 475.2228; HPLC (chiralpak IC, hexane/2-propanol = 90/10, Flow rate = 0.7 mL/min): t_R (minor) 14.5 min, t_R (major) 28.0 min.

***tert*-Butyl-2-(diphenylmethyleamino)-3-(furan-2-yl)-4-nitrobutanoate (6i)**

95% ee, dr = 94:6, $[\alpha]_D^{20} = -148.8^\circ$ (c = 1.27, CHCl₃); ¹H NMR (300 MHz, CDCl₃): δ 1.42 (s, 9H), 4.33 (d, *J* = 4.2 Hz, 1H), 4.42-4.44 (m, 1H), 5.05 (d, *J* = 6.6 Hz, 2H), 6.12 (d, *J* = 3.3 Hz, 1H), 6.25 (dd, *J* = 1.5, 3.3 Hz, 1H), 6.84 (d, *J* = 6.6 Hz, 2H), 7.25-7.44 (m, 7H), 7.59 (d, *J* = 7.2 Hz, 2H); ¹³C NMR (75 MHz, CDCl₃): δ 27.8, 40.8, 67.1, 75.0, 82.4, 107.5, 110.4, 127.5, 128.1, 128.3, 128.7, 128.8, 130.7, 135.5, 138.8, 142.0, 150.9, 168.7, 172.5; IR (thin film): 1734, 1625, 1557, 1447, 1371, 1290, 1151, 697; MS (ESI): 435.2 (M+1); HRMS (ESI): C₂₅H₂₇N₂O₅, Calc: 435.1913, Found: 435.1915; HPLC (chiralpak AD-H, hexane/2-propanol = 90/10, Flow rate = 0.7 mL/min): t_R (minor) 7.5 min, t_R (major) 10.0 min.

***tert*-Butyl-3-(2-bromophenyl)-2-(diphenylmethyleamino)-4-nitrobutanoate (6j)**

93% ee, dr = 90:10, $[\alpha]_D^{20} = -93.4^\circ$ (c = 1.05, CHCl₃); ¹H NMR (300 MHz, CDCl₃): δ 1.42 (s, 9H), 4.30 (d, *J* = 3.3 Hz, 1H), 4.83-4.89 (m, 1H), 5.13-5.19 (m, 1H), 5.26-5.34 (m, 1H), 6.48 (d, *J* = 6.6 Hz, 2H), 7.07-7.62 (m, 12H); ¹³C NMR (75 MHz, CDCl₃): δ 27.9, 45.1, 66.5, 75.0, 82.3, 125.3, 127.1, 127.4, 128.1, 128.2, 128.6, 128.7, 129.0, 129.1, 130.7, 133.4, 135.3, 136.3, 138.6, 168.7, 172.7; IR (thin film): 1733,

1554, 1446, 1370, 1292, 1152, 1025, 698; MS (ESI): 523.1 (M+1); HRMS (ESI): C₂₇H₂₈N₂O₄Br, Calc: 523.1225, Found: 523.1227; HPLC (chiralpak IC, hexane/2-propanol = 90/10, Flow rate = 0.7 mL/min): t_R (minor) 9.8 min, t_R (major) 18.3 min.

***tert*-Butyl-2-(diphenylmethyleneamino)-4-nitro-3-(2-(trifluoromethyl)phenyl)butanoate (6k)**

86% ee, dr = 82:18, ¹H NMR (400 MHz, CDCl₃): δ 1.42 (s, 9H), 4.20 (d, *J* = 2.8 Hz, 1H), 4.79-4.82 (m, 1H), 5.20-5.25 (m, 1H), 5.35-5.41 (m, 1H), 6.45 (d, *J* = 7.2 Hz, 2H), 7.18-7.44 (m, 10H), 7.63-7.65 (d, *J* = 7.2 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 27.8, 41.7, 67.8, 75.8, 82.4, 125.1, 126.5 (q, *J* = 5.6 Hz), 127.0, 127.6, 128.1, 128.2, 128.6, 128.8, 128.9, 129.3, 130.8, 131.6, 135.4, 136.5, 138.7, 168.6, 173.0; MS (ESI): 513.4 (M+1); HRMS (ESI): C₂₈H₂₈F₃N₂O₄, Calc: 513.2011, Found: 513.1996; HPLC (chiralpak IC, hexane/2-propanol = 80/20, Flow rate = 0.5 mL/min): t_R (minor) 8.9 min, t_R (major) 15.2 min.

***tert*-Butyl-3-(2-chlorophenyl)-2-(diphenylmethyleneamino)-4-nitrobutanoate (6l)**

93% ee, dr = 90:10, [α]_D²⁰ = -141.2° (c = 1.23, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 1.40 (s, 9H), 4.29 (d, *J* = 4.4 Hz, 1H), 4.87-4.90 (m, 1H), 5.13-5.18 (m, 1H), 5.24-5.30 (m, 1H), 6.53 (d, *J* = 6.8 Hz, 2H), 7.14-7.44 (m, 10H), 7.60-7.62 (m, 2H);

^{13}C NMR (100 MHz, CDCl_3): δ 27.9, 42.8, 66.6, 75.0, 82.3, 126.8, 127.2, 128.1, 128.2, 128.6, 128.6, 128.7, 128.8, 130.0, 130.7, 134.5, 134.8, 135.4, 138.7, 168.8, 172.7; MS (ESI): 479.3 ($\text{M}+1$); HRMS (ESI): $\text{C}_{27}\text{H}_{28}\text{ClN}_2\text{O}_4$, Calc: 479.1738, Found: 479.1732; HPLC (chiralpak IC, hexane/2-propanol = 90/10, Flow rate = 0.7 mL/min), t_R (minor) 9.4 min, t_R (major) 16.5 min.

3. Synthesis of the new ligand (L5)

Synthesis of 1-[di 1-pyrrolyl phosphino]-2-[(*S*)- 4'-isopropyl-2,5-oxazolinyl] ferrocene (L5)

Compound **5** (298 mg, 1.0 mmol) was dissolved in freshly distilled Et₂O (10 mL) under argon atmosphere and cooled to -78 °C. At this temperature, TMEDA (0.2 mL, 1.3 mmol) and *s*-BuLi [0.9 mL, 1.2 mmol, 1.3M solution in cyclohexane/hexane (98/2)] were added, and the solution was stirred for 1 hour. Ar₂PCl (1.3 mmol) was then added, and the resulting mixture was continually stirred and allowed to warm to room temperature over 30 min. The reaction mixture was diluted with ether (20 mL), washed with saturated aqueous NaHCO₃, and dried over Na₂SO₄. The solvent was removed under reduced pressure, and the resulting residue was purified by chromatography (silica gel, ethyl acetate/petroleum 1:10) to give ligand **L5** (57% yield). $[\alpha]_D^{20} = -125.8^\circ$ ($c = 0.96$, CHCl_3); ^1H NMR (400 MHz, CDCl_3): δ 0.86 (d, $J = 6.4$ Hz, 3H), 0.95 (d, $J = 6.8$ Hz, 3H), 1.67-1.71 (m, 1H), 3.81-3.86 (m, 2H), 4.02 (s, 1H), 4.21-4.26 (m, 6H), 4.45-4.46 (m, 1H), 4.92 (s, 1H), 6.19-6.20 (m, 2H), 6.30-6.31

(m, 2H), 6.80-6.82 (m, 2H), 6.96-6.98 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ 18.3, 18.5, 32.5, 70.32, 71.1, 71.5, 72.4, 72.8, 73.1, 111.0, 111.1, 123.8, 123.9, 124.0, 124.2, 163.8; ^{31}P NMR (162 MHz, CDCl_3): δ 62.9; MS (MALDI): 393.6 (M- $\text{C}_4\text{H}_4\text{N}$); HRMS (ESI): $\text{C}_{20}\text{H}_{22}\text{N}_2\text{O}^{54}\text{Fe}^{+1}$, Calc: 391.0872, Found: 391.0860.

4. Scale-up reaction of glycine imine **1c** and nitroalkene **2a**

$\text{CuClO}_4 \cdot (\text{CH}_3\text{CN})_4$ (330 mg, 1 mmol) and the phosphino-oxazoline ligand (750 mg, 1 mmol) was added to a 100 mL three-necked bottle under argon atmosphere. The mixture was dried for 30 min under vacuum with stirring, then freshly distilled anhydrous THF (30 mL) was added. After being stirred for 30 min, the glycine derivatives **1c** was added (2.95 g, 10 mmol) at room temperature. After glycine **1c** was dissolved, Et_3N (138 μL , 1 mmol) was added dropwise at -10°C . Then the solution of nitroalkene **2a** (1.49 g, 10 mmol) in 15 mL THF was added dropwise in 30 min. The reaction mixture was stirred at -10°C for 8 h until the reaction completed (monitored by TLC), then reaction mixture was filtered through a short plug of silica gel. After evaporation the solvent, the crude product was analyzed by ^1H NMR spectroscopy to determine the diastereomeric ratio, and then purified by chromatography on silica gel (EtOAc/petroleum mixtures), the desired product (**6a**, 4.42 g, > 99% yield, dr = 94:6, 97% ee) was obtained.

5. Transformation of the product

tert-Butyl-2-(diphenylmethylenamino)-4-nitro-3-phenylbutanoate (**7a**)

3ca (22.2 mg, 0.05 mmol) was dissolved into THF (0.5 mL), and 1N HCl (0.5 mL) was added at 0 °C. After the mixture was stirred at 0 °C for 1h, THF was removed under reduced pressure. The resulting aqueous solution was washed with ether (3 × 10 mL) and neutralized with NaHCO₃. The mixture was then extracted with CH₂Cl₂ (3 × 10 mL). The organic layers were combined and dried over anhydrous Na₂SO₄. After filtration and concentration under reduced pressure, the desired product (**7a**, 13.3 mg, 95% yield, dr > 99:1, 98% ee) was obtained. $[\alpha]_{\text{D}}^{20} = 8.6^{\circ}$ (c = 1.15, CHCl₃), ¹H NMR (400 MHz, CDCl₃): δ 1.22 (s, 9H), 3.54 (d, *J* = 8.8 Hz, 1H), 3.66-3.71 (m, 1H), 4.69 (dd, *J* = 7.2, 12.8 Hz, 1H), 5.04 (dd, *J* = 5.2, 12.8 Hz, 1H), 7.22-7.34 (m, 5H); ¹³C NMR (100 MHz, CDCl₃): δ 27.5, 48.2, 58.2, 77.9, 81.8, 128.0, 128.3, 128.7, 136.6, 172.9; MS (ESI): 303.1 (M+Na⁺); HRMS (ESI): C₁₄H₂₀N₂NaO₄, Calc: 303.1321, Found: 303.1315; HPLC (chiralpak Phenomenex Lux 5μ Amylose -2, hexane/2-propanol = 70/30, Flow rate = 0.7 mL / min): t_R (minor) 10.9 min, t_R (major) 12.0min.

tert-Butyl-2,4-bis(tert-butoxycarbonylamino)-3-phenylbutanoate (**8**)

To a suspension of **7a** (28.0 mg, 0.10 mmol) in MeOH (1 mL) was added Pd/C (5 mg) at room temperature. The reaction mixture was stirred under H₂ atmosphere (50 atm) for 24 h at room temperature. Then, the mixture was diluted with ethyl acetate and filtered through a celite pad, and the filtrate was concentrated under reduced pressure. The residue was dissolved in CH₂Cl₂ (2 mL) and Boc₂O (54.5 mg, 0.25 mmol) was added. Then, the mixture was stirred at room temperature. The reaction mixture was evaporated under reduced pressure and purified by silica gel column chromatography

(hexane/ethyl acetate = 5/1) to afford **8** (38.4 mg, 84% yield), $[\alpha]_D^{20} = -16.9^\circ$ ($c = 0.67$, CHCl_3), ^1H NMR (300 MHz, CDCl_3): δ 1.21 (s, 9H), 1.39 (s, 9H), 1.44 (s, 9H), 3.08 (br, 1H), 3.30 (br, 1H), 3.74 (br, 1H), 4.46 (t, $J = 8.4$ Hz, 1H), 4.69 (br, 1H), 5.21 (br, 1H), 7.18-7.30 (m, 5H); ^{13}C NMR (75 MHz, CDCl_3): δ 27.5, 28.0, 28.2, 42.4, 49.2, 56.3, 79.2, 79.9, 82.0, 127.5, 128.4, 128.8, 138.0, 155.5, 155.7, 170.4; MS (ESI): 473.4 ($\text{M} + \text{Na}^+$); HRMS (ESI): $\text{C}_{24}\text{H}_{38}\text{N}_2\text{NaO}_6$, Calc: 473.2632, Found: 473.2622; HPLC (chiralpak Phenomenex Lux 5μ Cellulose-1, hexane/2-propanol = 90/10, Flow rate = 0.7 mL / min): t_R (minor) 5.4 min, t_R (major) 5.7 min.

6. Determination of absolute configuration of **7b**, and the ORTEP diagram of X-ray diffraction structure of **7b**

The procedure of **6b** transformed to **7b** was the same to that of **7a**.

tert-Butyl 2-amino-3-(4-bromophenyl)-4-nitrobutanoate (**7b**)

96%ee, $[\alpha]_D^{20} = 8.9^\circ$ ($c = 1.15$, CHCl_3), ^1H NMR (400 MHz, CDCl_3): δ 1.25 (s, 9H), 3.50 (d, $J = 8.4$ Hz, 1H), 3.63-3.69 (m, 1H), 4.65 (dd, $J = 10, 13.2$ Hz, 1H), 5.02 (dd, $J = 4.8, 12.8$ Hz, 1H), 7.12 (d, $J = 8.4$ Hz, 2H), 7.45 (d, $J = 8.0$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ 27.6, 47.6, 57.9, 77.6, 82.1, 122.1, 130.1, 131.8, 135.7, 172.6; MS (ESI): 302.9 ($\text{M} - \text{C}_4\text{H}_9$); HRMS (ESI): $\text{C}_{10}\text{H}_{12}\text{BrN}_2\text{O}_4$, Calc: 302.9981, Found: 302.9975; HPLC (chiralpak Phenomenex Lux 5μ Amylose -2, hexane/2-propanol = 80/20, Flow rate = 0.7 mL / min): t_R (minor) 13.2 min, t_R (major) 16.2 min.

the ORTEP diagram of X-ray diffraction structure of **7b**:

7. References

- [1] (a) Sammakia, T.; Stangeland, E. L. *J. Org. Chem.* **1997**, 62, 6104. (b) Supporting information, Longmire, J. M.; Wang, B.; Zhang, X. *J. Am. Chem. Soc.* **2002**, 124, 13400. (c) Supporting information, Yan, X.-X.; Peng, Q.; Zhang, Y.; Zhang, K.; Hong, W.; Hou, X.-L.; Wu, Y.-D. *Angew. Chem. Int. Ed.* **2006**, 45, 1979.
- [2] A. F. McAnda, K. D. Roberts, A. J. Smallridge, A. Ten, M. A. Trehwella, *J. Chem. Soc. Perkin Trans. 1* **1998**, 501-504.

Std proton;blank line

USER: -- DATE: Nov 11 2008

F1: 400.037	F2: 100.598	SW1: 6410		OF1: 2394.4		PTS1d: 13132 , 16384	
EX: s2pul	PW: 6.9 us	PD: 1.0 sec		NA: 8	LB: 0.0	Nuts - \$lq-6-61-H.fid

lq-6-52-C

USER: -- DATE: Nov 11 2008

F1: 100.599	F2: 400.037	SW1: 24510		OF1: 10556.0		PTS1d: 31875 , 32768	
EX: s2pul	PW: 4.1 us	PD: 1.0 sec		NA: 68	LB: 2.0	Nuts - \$lq-6-61-c.fid	

HPLC REPORT

Data File: LQ-6-54+-...che

Sample Name:

Date: 2009-06-29

Time: 13:35

Operator:

No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1		10.285	50804.5	770936.9	2.2927
2	2		11.618	42163.2	822632.2	2.4464
3	3		13.018	787188.3	15932907.4	47.3827
4	4		15.352	634005.8	16099542.7	47.8782
Total				1514161.7	33626019.3	100.0000

HPLC REPORT

Data File: LQ-6-61. che

Sample Name:

Date: 2008-12-29

Time: 10:08

Operator:

No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1		10.348	1787.6	24382.1	0.2359
2	2		11.750	20972.0	417562.9	4.0393
3	3		13.210	7143.6	147226.0	1.4242
4	4		15.627	371620.0	9748212.7	94.3006
Total				401523.2	10337383.7	100.0000

STANDARD 1H OBSERVE:blank line

USER: -- DATE: Nov 20 2008

F1: 300.029	F2: 75.449	SW1: 4803		OF1: 1797.6		PTS1d: 9592 , 16384	
EX: s2pul	PW: 4.2 us	PD: 1.0 sec		NA: 16	LB: 1.0	Nuts - \$lq-6-62-H.fid

wwq-14-92				USER: -- DATE: Nov 20 2008			
F1: 75.450	F2: 300.028	SW1: 18868		OF1: 8274.4		PTS1d: 30188 , 32768	
EX: s2pul	PW: 4.6 us	PD: 1.0 sec		NA: 40	LB: 2.0	Nuts - \$lq-6-62-C.fid	

HPLC REPORT

Data File: LQ-6-62+- .che

Sample Name:

Date: 2008-11-21

Time: 12:06

Operator:

No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1		12.852	373245.5	7259153.4	49.3251
2	2		14.552	251770.5	7457797.4	50.6749
Total				625016.0	14716950.7	100.0000

HPLC REPORT

Data File: LQ-6-62. che

Sample Name:

Date: 2008-11-21

Time: 12:35

Operator:

No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1		12.785	11539.8	222605.7	1.4374
2	2		14.418	559610.5	15263916.9	98.5626
Total				571150.3	15486522.6	100.0000

;blank line

USER: -- DATE: Nov 16 2009

F1: 400.037	F2: 100.598	SW1: 6410		OF1: 2397.6		PTS1d: 19231 , 32768	
EX: s2pul	PW: 4.4 us	PD: 1.0 sec		NA: 8	LB: 0.0	Nuts - \$lq-6-85-H.fid

cjp-8-56-Cmix

USER: -- DATE: Dec 15 2008

F1: 75.450	F2: 300.028	SW1: 18868		OF1: 8276.1		PTS1d: 30188 , 32768	
EX: s2pul	PW: 4.6 us	PD: 1.0 sec		NA: 40	LB: 2.0	Nuts - \$lq-6-85-C.fid	

HPLC REPORT

Data File: 6-85rac. che

Sample Name:

Date: 2008-12-17

Time: 09:40

Operator:

No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1		9.060	691886.2	10084778.7	50.7923
2	2		10.093	592879.8	9770171.3	49.2077
Total				1284766.1	19854950.0	100.0000

HPLC REPORT

Data File: 6-85.che

Sample Name:

Date: 2008-12-17

Time: 09:58

Operator:

No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1		9.043	31100.0	355514.0	1.1091
2	2		10.043	1853402.9	31698110.4	98.8909
Total				1884502.8	32053624.4	100.0000

fxp-7-28-1hh

USER: -- DATE: Dec 17 2008

F1: 400.037	F2: 100.598	SW1: 6410		OF1: 2396.0		PTS1d: 19231 , 32768	
EX: s2pul	PW: 4.4 us	PD: 1.0 sec		NA: 12	LB: 0.0	Nuts - \$lq-6-88-H.fid

2005147-x0784

USER: -- DATE: Dec 17 2008

F1: 100.599	F2: 400.037	SW1: 24510		OF1: 10558.0		PTS1d: 31875 , 32768	
EX: s2pul	PW: 7.3 us	PD: 1.0 sec		NA: 60	LB: 2.0	Nuts - \$lq-6-88-C.fid	

AD-H, 0.7ml/min, 90/10, 214nm

=====

Injection Date : 12/30/08 3:05:51 PM
Sample Name : 1q-6-88-rac Location : Vial 1
Acq. Operator :
Method : C:\HPCHEM\1\METHODS\OPEN.M
Last changed : 12/30/08 2:41:12 PM
(modified after loading)

=====

=====

Area Percent Report

=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	8.483	MM	0.2714	6560.56445	402.88379	49.7858
2	10.960	VB	0.3362	6617.02246	289.86191	50.2142

Totals : 1.31776e4 692.74570

Results obtained with enhanced integrator!

=====

*** End of Report ***

=====

AD-H, 0.7ml/min, 90/10, 214nm

Injection Date : 12/30/08 2:25:56 PM
Sample Name : 1q-6-88
Acq. Operator :
Method : C:\HPCHEM\1\METHODS\OPEN.M
Last changed : 12/30/08 2:02:12 PM
(modified after loading)

Location : Vial 1

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	8.518	VB	0.2365	7716.37207		481.30634	98.5269
2	10.990	VV	0.3207	115.37175		4.85910	1.4731

Totals : 7831.74382 486.16544

Results obtained with enhanced integrator!

*** End of Report ***

STANDARD 1H OBSERVE:blank line

USER: -- DATE: Nov 20 2008

F1: 300.029	F2: 75.449	SW1: 4803		OF1: 1800.2		PTS1d: 9592 , 16384	
EX: s2pul	PW: 4.2 us	PD: 1.0 sec		NA: 16	LB: 0.0	Nuts - \$lq-6-63-H.fid

wwq-14-92				USER: -- DATE: Nov 20 2008			
F1: 75.450	F2: 300.028	SW1: 18868		OF1: 8275.6		PTS1d: 30188 , 32768	
EX: s2pul	PW: 4.6 us	PD: 1.0 sec		NA: 58	LB: 2.0	Nuts - \$lq-6-63-C.fid	

HPLC REPORT

Data File: LQ-6-63+-.che

Sample Name:

Date: 2008-11-21

Time: 13:13

Operator:

No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1		18.818	114310.6	3452383.9	49.9614
2	2		21.952	93010.1	3457714.6	50.0386
Total				207320.6	6910098.4	100.0000

HPLC REPORT

Data File: LQ-6-63. che

Sample Name:

Date: 2008-11-21

Time: 14:01

Operator:

No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1		19.018	1003.7	34059.4	0.9454
2	2		22.218	94035.6	3568683.1	99.0546
Total				95039.4	3602742.6	100.0000

STANDARD 1H OBSERVE:blank line

USER: -- DATE: Nov 20 2008

F1: 300.029	F2: 75.449	SW1: 4803		OF1: 1796.4		PTS1d: 9592 , 16384	
EX: s2pul	PW: 4.2 us	PD: 1.0 sec		NA: 18	LB: 1.0	Nuts - \$lq-6-64-H.fid

wwq-14-92

USER: -- DATE: Nov 20 2008

F1: 75.450	F2: 300.028	SW1: 18868		OF1: 8275.4		PTS1d: 30188 , 32768	
EX: s2pul	PW: 4.6 us	PD: 1.0 sec		NA: 114	LB: 2.0	Nuts - \$lq-6-64-C.fid	

1q-6-64-rac

AD-H, 0.7 ml/min, 90/10, 214nm

Injection Date : 12/25/08 4:29:54 PM
Sample Name : 1q-6-64-rac Location : Vial 1
Acq. Operator :
Method : C:\HPCHEM\1\METHODS\OPEN.M
Last changed : 12/25/08 4:38:32 PM
(modified after loading)

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.605	VV	0.2283	7023.80029	451.04880	51.0488
2	8.791	VB	0.2507	6735.18115	399.20950	48.9512

Totals : 1.37590e4 850.25830

Results obtained with enhanced integrator!

*** End of Report ***

AD-H, 0.7 ml/min, 90/10, 214nm

Injection Date : 12/25/08 4:45:02 PM
Sample Name : 1q-6-64
Acq. Operator :
Method : C:\HPCHEM\1\METHODS\OPEN.M
Last changed : 12/25/08 4:38:32 PM
(modified after loading)

Location : Vial 1

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	7.662	VV	0.1971	2.98961e4	2112.95337	98.3103
2	8.870	VV	0.3171	513.84552	22.52590	1.6897

Totals : 3.04099e4 2135.47927

Results obtained with enhanced integrator!

*** End of Report ***

;blank line

USER: -- DATE: Nov 16 2009

F1: 400.037	F2: 100.598	SW1: 6410	OF1: 2398.9	PTS1d: 19231 , 32768
EX: s2pul	PW: 4.4 us	PD: 1.0 sec	NA: 8	LB: 0.0

Nuts - \$lq-6-90-H.fid

XWH-galac-Thr

USER: -- DATE: Dec 18 2008

F1: 100.599	F2: 400.037	SW1: 24510		OF1: 10554.5		PTS1d: 31875 , 32768	
EX: s2pul	PW: 7.3 us	PD: 1.0 sec		NA: 32	LB: 2.0	Nuts - \$lq-6-90-C.fid	

HPLC REPORT

Data File: LQ-6-90+-.che

Sample Name:

Date: 2008-12-29

Time: 10:35

Operator:

No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1		9.945	184444.3	2888886.4	5.3181
2	2		10.793	160929.7	3125917.4	5.7544
3	3		12.193	1252396.4	24275646.8	44.6886
4	4		15.210	812888.0	24031333.5	44.2389
Total				2410658.5	54321784.2	100.0000

HPLC REPORT

Data File: LQ-6-90.che

Sample Name:

Date: 2008-12-29

Time: 11:05

Operator:

No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1		9.922	7087.0	126269.9	0.6876
2	2		10.760	108571.4	2026454.6	11.0343
3	3		12.187	11512.0	190941.4	1.0397
4	4		15.127	579853.3	16021446.3	87.2385
Total				707023.7	18365112.2	100.0000

Std proton;blank line

USER: -- DATE: Jan 4 2009

F1: 400.037	F2: 100.598	SW1: 6410		OF1: 2395.2		PTS1d: 19231 , 32768	
EX: s2pul	PW: 4.4 us	PD: 1.0 sec		NA: 4	LB: 0.0	Nuts - \$lq-7-3-H.fid

2004161-1023

USER: -- DATE: Jan 4 2009

F1: 100.599	F2: 400.037	SW1: 24510		OF1: 10555.2		PTS1d: 31875 , 32768	
EX: s2pul	PW: 7.3 us	PD: 1.0 sec		NA: 48	LB: 2.0	Nuts - \$lq-7-3-C.fid	

HPLC REPORT

Data File: LQ-7-3+-.che

Sample name:

Date: 2009-01-07

Time : 08:34

Operator:

No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1		14.618	1049749.6	27734831.9	50.0517
2	2		28.718	503146.6	27677516.7	49.9483
Total				1552896.2	55412348.7	100.0000

HPLC REPORT

Data File: 7-3. che

Sample name:

Date: 2009-01-07

Time : 09:13

Operator:

No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1		14.485	35335.0	900071.3	1.5832
2	2		27.985	1013089.4	55951933.4	98.4168
Total				1048424.4	56852004.7	100.0000

STANDARD 1H OBSERVE:blank line

USER: -- DATE: Nov 20 2008

F1: 300.029	F2: 75.449	SW1: 4803		OF1: 1798.8		PTS1d: 9592 , 16384	
EX: s2pul		PW: 4.2 us	PD: 1.0 sec	NA: 14	LB: 0.0	Nuts - \$lq-6-65-H.fid

wwq-14-92

USER: -- DATE: Nov 20 2008

F1: 75.450	F2: 300.028	SW1: 18868		OF1: 8275.0		PTS1d: 30188 , 32768	
EX: s2pul	PW: 4.6 us	PD: 1.0 sec		NA: 76	LB: 2.0	Nuts - \$lq-6-65-C.fid	

AD-H, 0.7 ml/min, 90/10, 214nm

=====
Injection Date : 12/25/08 5:01:47 PM
Sample Name : 1q-6-65-rac Location : Vial 1
Acq. Operator :
Method : C:\HPCHEM\1\METHODS\OPEN.M
Last changed : 12/25/08 5:02:23 PM
(modified after loading)
=====

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

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	7.555	MM	0.2457	1749.96326	118.68791	51.4404
2	10.175	VB	0.2831	1651.96313	86.85220	48.5596

Totals : 3401.92639 205.54012

Results obtained with enhanced integrator!

*** End of Report ***

AD-H, 0.7 ml/min, 90/10, 214nm

Injection Date : 12/25/08 5:41:58 PM
Sample Name : 1q-6-65 Location : Vial 1
Acq. Operator :
Method : C:\HPCHEM\1\METHODS\OPEN.M
Last changed : 12/25/08 5:52:20 PM
(modified after loading)

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.485	VV	0.2348	701.65350	42.84170	2.2733
2	10.009	VB	0.2490	3.01640e4	1738.68140	97.7267

Totals : 3.08656e4 1781.52310

Results obtained with enhanced integrator!

*** End of Report ***

cjp-8-56-Cmix

USER: -- DATE: Dec 15 2008

F1: 75.450	F2: 300.028	SW1: 18868		OF1: 8275.6		PTS1d: 30188 , 32768	
EX: s2pul	PW: 4.6 us	PD: 1.0 sec		NA: 56	LB: 2.0	Nuts - \$lq-6-84-C.fid	

HPLC REPORT

Data File: LQ-6-84+-.che

Sample Name:

Date: 2009-06-29

Time: 14:33

Operator:

No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1		9.787	615758.0	9381505.8	44.5883
2	2		12.685	49824.9	1097011.2	5.2139
3	3		14.185	41330.6	1080486.4	5.1353
4	4		18.318	297640.8	9481276.4	45.0625
Total				1004554.3	21040279.8	100.0000

HPLC REPORT

Data File: LQ-8-23.che

Sample Name:

Date: 2009-06-29

Time: 15:00

Operator:

No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1		9.818	84978.4	1294705.4	3.4085
2	2		12.685	3286.6	71870.7	0.1892
3	3		14.185	67123.2	1765422.8	4.6477
4	4		18.252	1074078.9	34853114.3	91.7547
Total				1229467.1	37985113.2	100.0000

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USER: -- DATE: Nov 18 2009

F1: 400.037

F2: 100.598

SW1: 6410

OF1: 2398.3

PTS1d: 19231 , 32768

EX: s2pul

PW: 4.4 us

PD: 1.0 sec

NA: 8

LB: 1.0

Nuts - \$lq-9-36-H.fid

cyf12-mecn-c

USER: -- DATE: Nov 18 2009

F1: 100.599	F2: 400.037	SW1: 26596		OF1: 10060.8		PTS1d: 34587 , 65536	
EX: s2pul	PW: 7.3 us	PD: 1.0 sec		NA: 64	LB: 2.0	Nuts - \$lq-9-36-c-4.fid	

==== Shimadzu LCsolution Analysis Report =====

C:\LabSolutions\Data\Project1\lq-6-89-r.lcd

Acquired by : Admin
 Sample Name : lq-6-89-r
 Sample ID : ic,80/20,0.5,214
 Injection Volume : 1 uL
 Data File Name : lq-6-89-r.lcd
 Report File Name : Default.lcr
 Data Acquired : 2009-11-19 16:19:29
 Data Processed : 2009-11-19 16:36:45

<Chromatogram>

PeakTable

Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.886	3344299	285359	31.312	41.309
2	9.828	2015629	150700	18.872	21.816
3	12.527	1979254	101449	18.532	14.686
4	15.053	3341248	153277	31.284	22.189
Total		10680430	690784	100.000	100.000

==== Shimadzu LCsolution Analysis Report =====

C:\LabSolutions\Data\Project1\lq\lq-9-39.lcd

Acquired by : Admin
 Sample Name : lq-9-36
 Method : ic,80/20,0.5,214
 Injection Volume : 1 uL
 Data File Name : lq-9-39.lcd
 Report File Name : Default.lcr
 Data Acquired : 2009-11-19 17:02:49
 Data Processed : 2009-11-19 17:20:25

<Chromatogram>

PeakTable

Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.905	514378	46370	6.099	11.044
2	9.870	70075	5213	0.831	1.242
3	12.650	874549	46435	10.369	11.059
4	15.227	6975381	321860	82.702	76.656
Total		8434383	419878	100.000	100.000

;blank line

USER: -- DATE: Jul 2 2009

F1: 400.037	F2: 100.598	SW1: 7022		OF1: 2697.3		PTS1d: 20365 , 32768	
EX: s2pul	PW: 4.4 us	PD: 1.0 sec		NA: 12	LB: 0.0	Nuts - \$lq-8-28-H.fid

wt-6-53-7-c13;blank line

USER: -- DATE: Jul 2 2009

F1: 100.602	F2: 400.037	SW1: 29762		OF1: 13324.6		PTS1d: 38705 , 65536	
EX: s2pul	PW: 7.3 us	PD: 1.0 sec		NA: 40	LB: 2.0	Nuts - \$lq-8-28-C.fid	

HPLC REPORT

Data File: LQ-8-28+-..che

Sample Name:

Date: 2009-07-03

Time: 13:30

Operator:

No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1		9.318	507466.1	7298072.8	47.4843
2	2		10.218	19188.7	341714.5	2.2233
3	3		11.252	15635.4	313413.0	2.0392
4	4		16.352	268073.2	7416244.2	48.2532
Total				810363.4	15369444.5	100.0000

HPLC REPORT

Data File: LQ-8-28. che

Sample Name:

Date: 2009-07-03

Time: 13:52

Operator:

No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1		9.385	81954.5	1167800.9	2.9557
2	2		10.318	74693.1	1107858.9	2.8040
3	3		11.352	162216.5	3202255.0	8.1048
4	4		16.452	1190072.3	34032684.0	86.1356
Total				1508936.5	39510598.7	100.0000

;blank line

USER: -- DATE: Jul 2 2009

F1: 400.037	F2: 100.599	SW1: 7022		OF1: 2694.5		PTS1d: 20365 , 32768	
EX: s2pul	PW: 4.4 us	PD: 1.0 sec	NA: 6	LB: 0.0	Nuts - \$lq-8-29-H.fid

L5

ly-8-42-recry-C

F1: 100.602	F2: 400.037	SW1: 29762	OF1: 13322.8	PTS1d: 38705 , 65536	USER: -- DATE: Jul 2 2009
EX: s2pul	PW: 7.3 us	PD: 1.0 sec	NA: 76	LB: 2.0	Nuts - \$lq-8-29-C.fid

L5

62.928

pxg-020-03029-2-P				USER: -- DATE: Jul 2 2009			
F1: 161.937	F2: 400.037	SW1: 65789		OF1: 83.6		PTS1d: 65794 , 131072	
EX: s2pul	PW: 5.0 us	PD: 1.0 sec		NA: 12	LB: 0.0	Nuts - \$lq-8-29-P.fid

dxw-14-23c

USER: -- DATE: Nov 4 2009

F1: 100.601

F2: 400.037

SW1: 32895

OF1: 12066.3

PTS1d: 42779 , 65536

EX: s2pul

PW: 7.3 us

PD: 1.0 sec

NA: 48

LB: 0.0

Nuts - \$lq-9-22-C.fid

Software Version : 6.3.1.0504 Date : 2009-11-10 16:25:26
Sample Name : LQ-9-23-2+- PA-2 73 214 0.7 Data Acquisition Time : 2009-11-10 15:36:05
Instrument Name : NCI901 Channel : A
Rack/Vial : 0/0 Operator : manager
Sample Amount : 1.000000 Dilution Factor : 1.000000
Cycle : 1

Result File :
Sequence File : D:\2009011010-.seq

HPLC REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	BL
1		8.868	152316.94	7450.53	4.387	BV
2		9.573	148395.03	8569.97	4.274	VV
3		10.325	1568862.45	83979.43	45.187	VV
4		11.457	1602356.08	74161.10	46.152	*VB

3471930.51 174161.02 1e+02

HPLC REPORT

Data File: LQ-9-33.che

Sample Name:

Date: 2009-11-17

Time: 09:34

Operator:

No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1		10.885	682.8	13033.9	1.0053
2	2		12.018	57131.9	1283507.7	98.9947
Total				57814.7	1296541.6	100.0000

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USER: -- DATE: Dec 9 2009

F1: 300.029

F2: 300.028

SW1: 5051

OF1: 2118.9

PTS1d: 10067 , 16384

EX: s2pul

PW: 4.2 us

PD: 1.0 sec

NA: 10

LB: 1.0

Nuts - \$lq-9-50-2-H.fid

txy-19-5a-c-300

USER: -- DATE: Dec 9 2009

F1: 75.450	F2: 300.028	SW1: 20000		OF1: 8069.9		PTS1d: 36000 , 65536	
EX: s2pul	PW: 4.6 us	PD: 1.0 sec		NA: 96	LB: 2.0	Nuts - \$lq-9-50-2-c24.fid	

HPLC REPORT

Data File: LQ-9-49-2+- PC-1 91 214 0.7..che

Sample Name:

Date: 2009-12-14

Time: 09:38

Operator:

No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1		5.372	173348.0	1509507.4	42.4784
2	2		5.718	164234.5	1558231.4	43.8496
3	3		8.518	18786.1	242914.3	6.8358
4	4		9.318	18420.1	242931.9	6.8362
Total				374788.6	3553585.1	100.0000

HPLC REPORT

Data File: LQ-9-50-2....che

Sample Name:

Date: 2009-12-14

Time: 09:56

Operator:

No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1		5.352	4486.4	38642.8	0.7894
2	2		5.685	480947.5	4789476.3	97.8357
3	3		8.440	5043.7	63806.4	1.3034
4	4		9.253	208.5	3501.9	0.0715
Total				490686.1	4895427.4	100.0000

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USER: -- DATE: Dec 2 2009

F1: 400.037

F2: 100.598

SW1: 5656

OF1: 2026.4

PTS1d: 15837 , 16384

EX: s2pul

PW: 4.4 us

PD: 1.0 sec

NA: 8

LB: 0.0

Nuts - \$lq-7-49-1-H.fid

ly-9-69-re-2C400

USER: -- DATE: Dec 2 2009

F1: 100.599	F2: 400.037	SW1: 25510		OF1: 10557.0		PTS1d: 33175 , 65536	
EX: s2pul	PW: 7.3 us	PD: 1.0 sec		NA: 24	LB: 0.0	Nuts - \$lq-7-49-1-C.fid	

HPLC REPORT

Data File: LQ-7-71-2+- PA-2 82 214 0.7. che

Sample Name:

Date: 2009-12-03

Time: 09:52

Operator:

No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1		7.968	79952.4	981371.2	9.1179
2	2		9.847	62228.9	974156.0	9.0509
3	3		13.285	180804.1	4422764.0	41.0918
4	4		16.285	144758.4	4384841.8	40.7395
Total				467743.7	10763132.9	100.0000

HPLC REPORT

Data File: LQ-7-49-1..che

Sample Name:

Date: 2009-12-03

Time: 10:31

Operator:

No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1		13.218	2913.4	67360.4	2.1305
2	2		16.185	104476.0	3094352.7	97.8695
Total				107389.5	3161713.1	100.0000