Rh-Catalyzed Asymmetric Hydrogenation of γ -Phthalimido Substituted α,β -Unsaturated Carboxylic Acid Esters: An Efficient Enantioselective Synthesis of β -Aryl- γ -Amino Acids

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Instruments and Materials

All solvents were dried and degassed by standard methods and stored under nitrogen. All the commercially available catalysts and ligands were brought from Strem Chemicals and without further purification. Ligand **2a-e** were synthesized according to the procedures reported previously.^{1,2} All reactions were carried out under an atmosphere of nitrogen using the standard Schlenk techniques or in a nitrogen-filled glovebox, unless otherwise noted.

NMR spectra were recorded on BRUKER DRX 400 spectrometers. Chemical shifts are reported in parts per million (ppm) down field from TMS with the solvent resonance as the internal standard. Coupling constants (J) are reported in Hz and refer to apparent peak multiplications. High resolution mass spectra (HRMS) were recorded on Q-Tof (Micromass) or Bruker APEX II spectrometer. Enantiomeric excess (ee) were determined by HPLC analysis on an Agilent HP-1100. Optical rotations were measured on a JASCO P-1020 high sensitive polarimeter.

Preparation of (S_C, R_{Fc}, R_P)-**3**: (R_{Fc})-**2**-{(S_C)-1-*N*-methyl-*N*-[bis(4-trifluoromethylphenyl)phosphino]aminoethyl}-1-[(R_P)-(1-naphthyl)phenylphosphino]ferrocene:

To a solution of (S_C, R_{Fc}, R_P)-PPFNHMe⁴ (477 mg, 1.0 mmol) and Et₃N (0.28 mL, 2.0 mmol) in toluene (2.5 mL) was added dropwise bis[4-(trifluoromethyl)phenyl]chlorophosphine (0.374 g, 1.05 mmol) at 0°C. The mixture was warmed to room temperature, and stirred at room temperature overnight. The reaction mixture was filtered through a pad of neutral aluminium oxide and eluted with hexane-EtOAc (10:1) to afford the title compound (717 mg, 90%) as orange foam. ¹H NMR (400 MHz, CDCl₃): δ = 1.58 (3H, d, J = 6.8 Hz; CH₃CH), 2.22 (3H, d, J = 3.3 Hz; CH₃N), 3.44 (5H, s; C₅H₅), 4.29 (1H, m; C₅H), 4.44 (1H, m; C₅H), 4.55 (1H, s; C₅H), 5.00-5.06 (1H, m; CHCH₃), 6.86 (1H, m; Ar-H),

7.02-7.07 (6H, m; Ar-**H**), 7.12-7.14 (1H, m; Ar-**H**), 7.34-7.36 (5H, m; Ar-**H**), 7.39-7.42 (3H, m; Ar-**H**), 7.56-7.58 (1H, m; Ar-**H**), 7.88-7.90 (2H, m; Ar-**H**), 9.42 (1H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ = 19.26, 19.32, 31.53, 31.64, 58.21, 58.30, 58.59, 58.68, 69.48, 69.94, 70.07, 71.96, 75.40, 75.54, 97.70, 124.64, 125.27, 125.55, 126.09, 126.40, 126.85, 126.97, 127.20, 127.79, 128.38, 128.75, 129.68, 130.19, 131.95, 132.11, 134.83, 136.34, 142.19 ppm; ^{31}P NMR (162 MHz, CDCl_3): δ = 55.44, -42.40 ppm; HRMS (EI) m/z calcd for $\text{C}_{43}\text{H}_{35}\text{NF}_6\text{FeP}_2^+$: 797.1498, found: 797.1493. In a similar method, (**R**_C,**S**_{Fe},**S**_P)-**3** was also prepared.

General Procedure for the Preparation of phthalimidobut-2-enoate **1**.^{5,6,7}

To a suspension of sodium hydride (60% dispersion in mineral oil, 4 mmol) in dry THF (200 mL) was added dropwise triethyl phosphonoacetate (4 mmol) at 0°C under argon atmosphere. After 30 min, the appropriate ketone **5** (2.7 mmol) was added to the reaction mixture, which was then allowed to warm to room temperature and stirred for 24h and then cooled with a water bath. A saturated aqueous ammonium chloride solution (20 mL) was then added dropwise to the cold mixture. The aqueous phase was extracted with diethyl ether (4 x 50mL) and the combined organic phase was washed with brine (3 x 50mL), dried over sodium sulfate, and concentrated in vacuo. Flash chromatography (hexanes/ethyl acetate, 95:5) yielded ester **6** as a clear oil with 60-80% yields.

A solution of α,β -unsaturated ester **6** (1 mmol), NBS (1.2 mmol) and benzoylperoxide (0.04 mmol) in dry CCl_4 (35 mL) was refluxed under nitrogen atmosphere for 10 h. The resulting reaction mixture was cooled to room temperature and then filtered through a sintered funnel to separate succinimide formed during the reaction. The filtrate was concentrated under reduced pressure to obtain bromoester **7**.

It was then purified by column chromatography packed with silica gel to give pure pale yellow colored gum, bromoester **7** with above 90% yields.

To the suspension of potassium phthalimide (10.0 mmol) in dry DMF (10 mL) was added a solution of bromoester **7** in dry DMF (10 mL). The reaction was run at room temperature and monitored by TLC. After the reaction was complete, the reaction mixture was poured into water and extracted with ether and concentrated in vacuum to leave a white solid. The residue was purified by flash chromatography (hexanes/ethyl acetate, 5:1), recrystallized with ethyl acetate/ hexane (1/4), to give the desired compounds **1**.

Ethyl (Z)-3-phenyl-4-phthalimidobut-2-enoate (1a). ^1H NMR (400 MHz, CDCl_3): δ = 1.34 (3H, t, J = 7.2 Hz; CH_3CH_2), 4.25 (2H, q, J = 7.2 Hz; CH_2CH_3), 5.47 (2H, s; CH_2N), 6.17 (1H, s; = CH), 7.23-7.27 (3H, m; Ar- H), 7.36 (2H, m; Ar- H), 7.60-7.62 (2H, m; Ar- H), 7.68-7.71 (2H, m; Ar- H) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ = 14.3, 37.0, 60.5, 121.0, 123.2, 127.0, 128.4, 129.0, 131.7, 133.9, 138.0, 153.9, 166.1, 167.8 ppm.

Ethyl (Z)-3-(2-methoxyphenyl)-4-phthalimidobut-2-enoate (1b). ^1H NMR (400 MHz, CDCl_3): δ = 1.33 (3H, t, J = 7.2 Hz; CH_3CH_2), 3.74 (3H, s; CH_3O), 4.25 (2H, q, J = 7.2 Hz; CH_2CH_3), 5.41 (2H, d; CH_2N), 6.00 (1H, t; = CH), 6.68-6.70 (1H, m; Ar- H), 6.77-6.79 (1H, m; Ar- H), 7.06-7.08 (1H, m; Ar- H), 7.14 (1H, m; Ar- H), 7.59-7.61 (2H, m; Ar- H), 7.67-7.69 (2H, m; Ar- H) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ = 14.3, 38.5, 55.3, 60.4, 109.9, 120.3, 121.5, 122.9, 127.7, 129.9, 130.2, 131.8, 133.7, 154.2, 156.3, 166.0, 167.6 ppm.

Ethyl (Z)-3-(3-methoxyphenyl)-4-phthalimidobut-2-enoate (1c). ^1H NMR (400 MHz, CDCl_3): δ = 1.34 (3H, t, J = 7.2 Hz; CH_3CH_2), 3.75 (3H, s; CH_3O), 4.26 (2H, q, J = 7.2 Hz; CH_2CH_3), 5.46 (2H, d; CH_2N), 6.20 (1H, s; = CH), 6.76-6.78 (1H, m; Ar- H), 6.91 (1H, m; Ar- H), 6.95-6.97 (1H, m; Ar- H), 7.13-7.17 (1H, m; Ar- H), 7.61-7.63 (2H, m; Ar- H), 7.70-7.72 (2H, m; Ar- H) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ = 14.3, 37.0, 55.3, 60.5, 111.8, 115.6, 119.5, 121.0, 123.2, 129.4, 131.8, 133.9,

139.4, 153.7, 159.4, 166.1, 167.9 ppm.

Ethyl (Z)-3-(4-methoxyphenyl)-4-phthalimidobut-2-enoate (1d). ^1H NMR (400 MHz, CDCl_3): δ =

1.33 (3H, t, J = 7.2 Hz; CH_3CH_2), 3.72 (3H, s; CH_3O), 4.27 (2H, m, CH_2CH_3), 5.47 (2H, s; CH_2N), 6.15 (1H, s; $=\text{CH}$), 6.76-6.78 (2H, m; Ar-H), 7.33-7.36 (2H, m; Ar-H), 7.61-7.63 (2H, m; Ar-H), 7.70-7.72 (2H, m; Ar-H) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ = 14.3, 36.6, 55.2, 60.3, 113.8, 119.7, 123.2, 128.4, 130.2, 131.8, 133.9, 153.2, 160.3, 166.2, 167.9 ppm.

Ethyl (Z)-3-(4-fluorophenyl)-4-phthalimidobut-2-enoate (1e). ^1H NMR (400 MHz, CDCl_3): δ = 1.34

(3H, t, J = 7.2 Hz; CH_3CH_2), 4.26 (2H, q, J = 7.2 Hz; CH_2CH_3), 5.45 (2H, s; CH_2N), 6.14 (1H, s; $=\text{CH}$), 6.91 (2H, m; Ar-H), 7.34-7.37 (2H, m; Ar-H), 7.62-7.65 (2H, m; Ar-H), 7.71-7.73 (2H, m; Ar-H) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ = 14.3, 37.0, 60.5, 115.3, 115.6, 120.6, 121.1, 123.2, 128.9, 129.0, 131.7, 134.0, 152.8, 161.9, 164.4, 165.9, 167.8 ppm.

Ethyl (Z)-3-(4-chlorophenyl)-4-phthalimidobut-2-enoate (1f). ^1H NMR (400 MHz, CDCl_3): δ = 1.34

(3H, t, J = 7.2 Hz; CH_3CH_2), 4.26 (2H, q, J = 7.2 Hz; CH_2CH_3), 5.44 (2H, s; CH_2N), 6.15 (1H, s; $=\text{CH}$), 7.21 (2H, m; Ar-H), 7.31 (2H, m; Ar-H), 7.65 (2H, m; Ar-H), 7.71-7.72 (2H, m; Ar-H) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ = 14.3, 36.8, 60.6, 121.4, 123.3, 128.4, 128.7, 131.7, 134.0, 135.1, 136.5, 152.6, 165.8, 167.8 ppm.

Ethyl (Z)-3-(4-bromophenyl)-4-phthalimidobut-2-enoate (1g). ^1H NMR (400 MHz, CDCl_3): δ = 1.34

(3H, t, J = 7.2 Hz; CH_3CH_2), 4.27 (2H, q, J = 7.2 Hz; CH_2CH_3), 5.44 (2H, s; CH_2N), 6.15 (1H, s; $=\text{CH}$), 7.24 (2H, d, J = 8.4 Hz; Ar-H), 7.37 (2H, d, J = 8.4 Hz; Ar-H), 7.63-7.65 (2H, m; Ar-H), 7.71-7.73 (2H, m; Ar-H) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ = 14.3, 36.8, 60.6, 121.5, 123.3, 123.4, 128.7, 131.6, 134.0, 137.0, 152.6, 165.8, 167.8 ppm.

Ethyl (Z)-3-(4-trifluoromethylphenyl)-4-phthalimidobut-2-enoate (1h). ^1H NMR (400 MHz,

CDCl_3): δ = 1.35 (3H, t, J = 7.2 Hz; CH_3CH_2), 4.27 (2H, q, J = 7.2 Hz; CH_2CH_3), 5.47 (2H, s; CH_2N), 6.19 (1H, s; $=\text{CH}$), 7.48-7.53 (4H, m; Ar-H), 7.63-7.66 (2H, m; Ar-H), 7.71-7.73 (2H, m; Ar-H) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ = 14.3, 36.9, 60.7, 122.5, 123.3, 125.4, 127.5, 131.6, 134.1, 141.7, 152.3, 165.6, 167.8 ppm.

Ethyl (Z)-3-[3-(cyclopentyloxy)-4-methoxyphenyl]-4-phthalimidobut-2-enoate (1i). ^1H NMR (400

MHz, CDCl_3): δ = 1.34 (3H, t, J = 7.2 Hz; CH_3CH_2), 1.56-1.59 (2H, m; CH_2CH_2), 1.78 (4H, m; CH_2CH_2), 1.91-1.93 (2H, m; CH_2CH_2), 3.75 (3H, s; CH_3O), 4.27 (2H, m; CH_2CH_3), 4.72 (1H, m; CHO), 5.45 (2H, s; CH_2N), 6.14 (1H, s; $=\text{CH}$), 6.71 (1H, d, J = 8.4 Hz; Ar-H), 6.87 (1H, d, J = 1.6 Hz; Ar-H), 6.94 (1 H, dd, J = 1.6 Hz, 8.4 Hz; Ar-H), 7.61-7.63 (2H, m; Ar-H), 7.70-7.71 (2H, m; Ar-H) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ = 14.4, 24.1, 32.8, 36.9, 55.9, 60.4, 80.4, 111.4, 113.2, 119.7, 119.8, 123.2, 130.5, 131.8, 133.9, 147.2, 150.8, 153.8, 166.3, 167.9 ppm.

Ethyl (Z)-3-(2-Naphthyl)-4-phthalimidobut-2-enoate (1j). ^1H NMR (400 MHz, CDCl_3): δ = 1.36 (3H,

t, J = 7.2 Hz; CH_3CH_2), 4.28 (2H, q, J = 7.2 Hz; CH_2CH_3), 5.59 (2H, s; CH_2N), 6.32 (1H, s; $=\text{CH}$), 7.41-7.45 (2H, m; Ar-H), 7.49-7.55 (3H, m; Ar-H), 7.64-7.66 (2H, m; Ar-H), 7.71-7.73 (2H, m; Ar-H), 7.79-7.82 (1H, m; Ar-H), 7.91 (1H, s; Ar-H) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ = 14.3, 36.9, 60.5, 121.5, 123.2, 124.5, 126.4, 126.7, 126.8, 127.5, 128.1, 128.5, 131.7, 133.0, 133.4, 133.8, 135.4, 153.7, 166.1, 167.9 ppm.

Ethyl (Z)-3-(6-methoxy-naphthalen-2-yl)-4-phthalimidobut-2-enoate (1k). ^1H NMR (400 MHz,

CDCl_3): δ = 1.35 (3H, t, J = 7.2 Hz; CH_3CH_2), 3.85 (3H, s; CH_3O), 4.28 (2H, q, J = 7.2 Hz; CH_2CH_3), 5.58 (2H, s; CH_2N), 6.30 (1H, s; $=\text{CH}$), 7.01 (1H, s; Ar-H), 7.07 (1H, d, J = 8.4 Hz; Ar-H), 7.46 (1H, d, J = 8.4 Hz; Ar-H), 7.54-7.55 (2H, m; Ar-H), 7.60-7.70 (4H, m; Ar-H), 7.84 (1H, s; Ar-H) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ = 14.3, 36.8, 55.3, 60.5, 105.5, 119.3, 120.8, 123.2, 125.0, 126.6, 126.9, 128.4, 130.0, 131.7, 133.1, 133.8,

134.8, 153.7, 158.4, 166.2, 167.9 ppm.

Ethyl (Z)-3-(2-thienyl)-4-phthalimidobut-2-enoate (11). ^1H NMR (400 MHz, CDCl_3): δ = 1.35 (3H, t, J = 7.2 Hz; CH_3CH_2), 4.28 (2H, q, J = 7.2 Hz; CH_2CH_3), 5.47 (2H, s; CH_2N), 6.43 (1H, d; =CH), 6.94 (1H, dd, J = 4.8, 4.0 Hz; Ar-H), 7.22 (1H, d, J = 4.8 Hz; Ar-H), 7.38 (1H, d, J = 4.0 Hz; Ar-H), 7.64-7.67 (2H, m; Ar-H), 7.76-7.78 (2H, m; Ar-H) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ =

14.3, 35.8, 60.5, 119.7, 123.3, 127.2, 127.6, 128.1, 131.9, 134.0, 141.3, 144.1, 166.0, 167.9 ppm.

General procedure for asymmetric hydrogenation

In a nitrogen-filled glovebox, to a solution of $[\text{Rh}(\text{COD})_2]\text{BF}_4$ (1.0 mg, 0.0025 mmol) in anhydrous and degassed CH_2Cl_2 (1 mL) was added ligand (0.00275 mmol). After stirring the mixture for 30 min, a substrate (0.25 mmol) dissolved in CH_2Cl_2 (1 mL) was added. The reaction mixture was transferred to a Parr stainless autoclave. The autoclave was purged three times with hydrogen and the pressure was set to the desired pressure. The hydrogenation was performed at room temperature for 24 h. After carefully releasing the hydrogen, the solvent was removed. The enantiomeric excess was determined by HPLC after purification on silica gel.

Ethyl (S)-3-phenyl-4-phthalimidobutanoate (4a). ^1H NMR (400 MHz, CDCl_3): δ = 1.06 (3H, t, J =

7.2 Hz; CH_3CH_2), 2.72 (2H, m, J = 8.0 Hz; CH_2CH), 3.70-3.78 (1H, m; CHCH_2), 3.89-3.95 (4H, m; OCH_2CH_3 , CH_2N), 7.18-7.22 (1H, m; Ar-H), 7.25-7.28 (4H, m; Ar-H), 7.67-7.70 (2H, m; Ar-H), 7.79-7.81 (2H, m; Ar-H) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ = 14.0, 38.6, 40.8, 43.2, 60.5, 123.3,

127.3, 127.7, 128.6, 131.9, 134.0, 140.4, 168.1, 171.5 ppm; HRMS (ESI) m/z calcd for $\text{C}_{20}\text{H}_{19}\text{NO}_4\text{Na}^+$: 360.1212, found: 360.1223; $[\alpha]_D^{25}$ = -62.8 (c 1.00, CHCl_3) for 95% ee; HPLC (UV 254 nm, Chiralpak AD (0.46 cm x 25 cm), i -PrOH/Hexane = 3/97, 0.8 mL/min): t_1 = 47.0 min, t_2 = 56.3 min.

Ethyl 3-(2-methoxyphenyl)-4-phthalimidobutanoate (4b). ^1H NMR (400 MHz, CDCl_3): δ = 1.08 (3H, t, J = 7.2 Hz; CH_3CH_2), 2.80-2.84 (2H, m; CH_2CH), 3.76 (3H, s; CH_3O), 3.94-3.99 (5H, m; CH_2O ,

CH_2N , $-\text{CH}-$), 6.78-6.80 (1H, m; Ar-**H**), 6.83-6.86 (1H, m; Ar-**H**), 7.66-7.69 (2H, m; Ar-**H**), 7.77-7.79 (2H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ = 14.3, 36.7, 37.2, 41.9, 55.5, 60.6, 110.9, 120.7, 123.3, 128.5, 128.7, 129.2, 132.2, 134.0, 157.9, 168.4, 172.3 ppm; HRMS (ESI) m/z calcd for $\text{C}_{21}\text{H}_{21}\text{NO}_5\text{Na}^+$: 390.1317, found: 390.1336; $[\alpha]_{\text{D}}^{25}$ = -81.9 (c 0.76, CHCl_3) for 96% ee; HPLC (UV 254 nm, Chiralcel OD-H (0.46 cm x 25 cm), *i*-PrOH/Hexane = 10/90, 1.0 mL/min): t_1 = 9.6 min, t_2 = 21.4 min.

Ethyl 3-(3-methoxyphenyl)-4-phthalimidobutanoate (4c). ^1H NMR (400 MHz, CDCl_3): δ = 1.08 (3H,

t , J = 7.2 Hz; CH_3CH_2), 2.70-2.72 (2H, d, J = 7.6 Hz; CH_2CH), 3.72-3.76 (4H, m; CH_3O , $-\text{CH}-$), 3.88-3.94 (4H, m; CH_2O , CH_2N), 6.76 (1H, m; Ar-**H**), 6.81 (1H, s; Ar-**H**), 6.86-6.88 (1H, m; Ar-**H**), 7.18-7.20 (1H, m; Ar-**H**), 7.69-7.71 (2H, m; Ar-**H**), 7.80-7.82 (2H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ = 14.1, 38.6, 40.8, 43.2, 55.2, 60.5, 112.9, 113.3, 120.0, 123.3, 129.6, 131.9, 134.0, 142.1, 159.7, 168.2, 171.5 ppm; HRMS (ESI) m/z calcd for $\text{C}_{21}\text{H}_{21}\text{NO}_5\text{Na}^+$: 390.1317, found: 390.1328; $[\alpha]_{\text{D}}^{25}$ = -70.0 (c 1.00, CHCl_3) for 94% ee; HPLC (UV 254 nm, Chiralcel OD-H (0.46 cm x 25 cm), *i*-PrOH/Hexane = 3/97, 1.0 mL/min): t_1 = 22.3 min, t_2 = 35.1 min.

Ethyl 3-(4-methoxyphenyl)-4-phthalimidobutanoate (4d). ^1H NMR (400 MHz, CDCl_3): δ = 1.07 (3H,

t , J = 7.2 Hz; CH_3CH_2), 2.67-2.70 (2H, m; CH_2CH), 3.68-3.76 (1H, m; $-\text{CH}-$), 3.82 (3H, s; CH_3O), 3.85-3.88 (2H, m; CH_2N), 3.92-3.96 (2H, m; CH_2O), 6.80 (2H, d, J = 8.0 Hz; Ar-**H**), 7.18 (2H, d, J = 8.0 Hz; Ar-**H**), 7.67-7.71 (2H, m; Ar-**H**), 7.78-7.81 (2H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ = 14.1, 38.9, 40.0, 43.3, 55.2, 60.5, 114.0, 123.3, 128.7, 131.9, 132.4, 134.0, 158.7, 168.2, 171.6 ppm; HRMS (ESI) m/z calcd for $\text{C}_{21}\text{H}_{21}\text{NO}_5\text{Na}^+$: 390.1317, found: 390.1328; $[\alpha]_{\text{D}}^{25}$ = -83.9 (c 0.94, CHCl_3) for 94% ee; HPLC (UV 254 nm, Chiralcel AD (0.46 cm x 25 cm), *i*-PrOH/Hexane = 5/95, 0.8 mL/min): t_1 = 54.4 min, t_2 = 74.6 min.

Ethyl 3-(4-fluorophenyl)-4-phthalimidobutanoate (4e). ^1H NMR (400 MHz, CDCl_3): δ = 1.07 (3H, t , J = 7.2 Hz; CH_3CH_2), 2.68-2.72 (2H, m; CH_2CH), 3.72-3.76 (1H, m; $-\text{CH}-$), 3.86-3.88 (2H, m; CH_2N),

3.89-3.98 (2H, m; CH_2O), 6.94 (2H, t, $J = 8.4$ Hz; Ar-**H**), 7.22-7.28 (2H, m; Ar-**H**), 7.69-7.71 (2H, m; Ar-**H**), 7.79-7.81 (2H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, CDCl_3): $\delta = 14.0, 38.4, 38.7, 40.1, 43.1, 60.5, 115.4, 115.6, 123.3, 129.2, 129.3, 131.8, 134.0, 136.1, 160.7, 163.2, 168.1, 171.3$ ppm; HRMS (ESI) m/z calcd for $\text{C}_{20}\text{H}_{18}\text{NO}_4\text{FNa}^+$: 378.1118, found: 378.1126; $[\alpha]_{\text{D}}^{25} = -88.8$ (c 1.00, CHCl_3) for 96% ee; HPLC (UV 254 nm, Chiralpak AD (0.46 cm x 25 cm), *i*-PrOH/Hexane = 5/95, 0.8 mL/min): $t_1 = 37.3$ min, $t_2 = 50.4$ min.

Ethyl (S)-3-(4-chlorophenyl)-4-phthalimidobutanoate (4f). ^1H NMR (400 MHz, CDCl_3): $\delta = 1.08$

(3H, t, $J = 7.2$ Hz; CH_3CH_2), 2.68-2.72 (2H, m; CH_2CH), 3.72-3.75 (1H, m; -CH-), 3.86-3.91 (2H, m; CH_2N), 3.92 (2H, q, $J = 7.2$ Hz; CH_2O), 7.20-7.26 (4H, m; Ar-**H**), 7.69-7.71 (2H, m; Ar-**H**), 7.79-7.81 (2H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, CDCl_3): $\delta = 14.1, 38.6, 40.2, 42.9, 60.6, 123.3, 128.8, 129.1, 131.8, 133.0, 134.1, 138.9, 168.1, 171.2$ ppm; HRMS (ESI) m/z calcd for $\text{C}_{20}\text{H}_{18}\text{NO}_4\text{ClNa}^+$: 394.0822, found: 394.0836; $[\alpha]_{\text{D}}^{25} = -96.5$ (c 0.80, CHCl_3) for 95% ee; HPLC (UV 254 nm, Chiralpak AD (0.46 cm x 25 cm), *i*-PrOH/Hexane = 5/95, 0.8 mL/min): $t_1 = 42.8$ min, $t_2 = 62.1$ min. For **Ethyl (R)-3-(4-chlorophenyl)-4-phthalimidobutanoate (4f)**. $[\alpha]_{\text{D}}^{25} = 63.1$ (c 0.50, CHCl_3) for 94% ee; HPLC (UV 254 nm, Chiralpak AD-H (0.46 cm x 25 cm), *i*-PrOH/Hexane = 15/85, 1.0 mL/min): $t_1 = 17.3$ min, $t_2 = 23.9$ min.

Ethyl 3-(4-bromophenyl)-4-phthalimidobutanoate (4g). ^1H NMR (400 MHz, CDCl_3): $\delta = 1.08$ (3H, t,

$J = 7.2$ Hz; CH_3CH_2), 2.63-2.74 (2H, m; CH_2CH), 3.70-3.74 (1H, m; -CH-), 3.82-3.90 (2H, m; CH_2N), 3.92 (2H, q, $J = 7.2$ Hz; CH_2O), 7.15 (2H, d, $J = 8.0$ Hz; Ar-**H**), 7.39 (2H, d, $J = 8.0$ Hz; Ar-**H**), 7.69-7.71 (2H, m; Ar-**H**), 7.79-7.82 (2H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, CDCl_3): $\delta = 14.1, 38.5, 40.3, 42.9, 60.6, 121.2, 123.3, 129.5, 131.7, 134.1, 139.5, 168.1, 171.1$ ppm; HRMS (ESI) m/z calcd for $\text{C}_{20}\text{H}_{18}\text{NO}_4\text{BrNa}^+$: 438.0317, found: 438.0327; $[\alpha]_{\text{D}}^{25} = -106.7$ (c 0.96, CHCl_3) for 94% ee; HPLC (UV 254 nm, Chiralpak AD (0.46 cm x 25 cm), *i*-PrOH/Hexane = 5/95, 1.0 mL/min): $t_1 = 38.0$ min, $t_2 = 56.4$ min.

Ethyl 3-(4-trifluoromethylphenyl)-4-phthalimidobutanoate (4h). ^1H NMR (400 MHz, CDCl_3): δ = 1.06-1.11 (3H, m; CH_3CH_2), 2.72-2.76 (2H, m; CH_2CH), 3.81-3.87 (1H, m; $-\text{CH}-$), 3.90-3.98 (4H, m;

CH_2N , CH_2O), 7.40 (2H, d, J = 8.0 Hz; Ar-**H**), 7.54 (2H, d, J = 8.0 Hz; Ar-**H**), 7.69-7.72 (2H, m; Ar-**H**), 7.80-7.83 (2H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ = 14.0, 38.4, 40.7, 42.8, 60.7, 123.4, 125.6, 128.2, 131.7, 134.2, 144.6, 168.1, 171.1 ppm; HRMS (ESI) m/z calcd for $\text{C}_{21}\text{H}_{18}\text{NO}_4\text{F}_3\text{Na}^+$: 428.1086, found: 428.1102; $[\alpha]_{\text{D}}^{25}$ = -73.0 (c 1.10, CHCl_3)

for 93% ee; HPLC (UV 254 nm, Chiralpak AD (0.46 cm x 25 cm), *i*-PrOH/Hexane = 10/90, 1.0 mL/min): t_1 = 15.9 min, t_2 = 24.4 min.

Ethyl (S)-3-[3-(cyclopentyloxy)-4-methoxyphenyl]-4-phthalimidobutanoate (4i). ^1H NMR (400

MHz, CDCl_3): δ = 1.09 (3H, t, J = 7.2 Hz; CH_3CH_2), 1.55-1.57 (2H, m; CH_2CH_2), 1.73-1.89 (6H, m; CH_2CH_2), 2.68 (2H, d, J = 7.2 Hz; CH_2CH), 3.68 (1H, m; $-\text{CH}-$), 3.78 (3H, s; CH_3O), 3.85-3.89 (2H, m; CH_2N), 3.93-3.98 (2H, m; CH_2O), 4.70 (1H, m; $-\text{CH}-$), 6.73-6.80 (3H, m; Ar-**H**), 7.67-7.71 (2H, m; Ar-**H**), 7.78-7.80 (2H, m; Ar-**H**) ppm; ^{13}C NMR (100

MHz, CDCl_3): δ = 14.1, 24.1, 32.7, 32.9, 38.9, 40.2, 43.3, 56.0, 60.5, 80.3, 111.9, 114.7, 119.6, 123.3, 131.9, 132.7, 134.0, 147.4, 149.0, 168.1, 171.6 ppm; HRMS (ESI) m/z calcd for $\text{C}_{26}\text{H}_{29}\text{NO}_6\text{Na}^+$: 474.1893, found: 474.1916; $[\alpha]_{\text{D}}^{25}$ = -80.4 (c 0.9, CHCl_3) for 97% ee; HPLC (UV 254 nm, Chiralpak AD (0.46 cm x 25 cm), *i*-PrOH/Hexane = 10/90, 0.8 mL/min): t_1 = 22.5 min, t_2 = 30.3 min. For **Ethyl (R)-3-[3-(cyclopentyloxy)-4-methoxyphenyl]-4-phthalimidobutanoate (4i)**. $[\alpha]_{\text{D}}^{25}$ = 60.8 (c 0.5, CHCl_3) for 95% ee; HPLC (UV 254 nm, Chiralpak AD (0.46 cm x 25 cm), *i*-PrOH/Hexane = 10/90, 1.0 mL/min): t_1 = 21.5 min, t_2 = 29.8 min.

Ethyl 3-(2-Naphthyl)-4-phthalimidobutanoate (4j). ^1H NMR (400 MHz, CDCl_3): δ = 1.02-1.07 (3H,

m; CH_3CH_2), 2.80-2.82 (2H, m; CH_2CH), 3.88-3.93 (3H, m; $-\text{CH}-$, CH_2N), 3.95-4.01 (2H, m; CH_2O), 7.42-7.47 (3H, m; Ar-**H**), 7.65-7.68 (2H, m; Ar-**H**), 7.72 (1H, s; Ar-**H**), 7.77-7.81 (5H, m; Ar-**H**), 7.73 (2H, m; Ar-**H**), 7.79 (1H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ = 14.0, 38.8, 40.9, 43.1, 60.5, 123.3, 125.7, 125.8, 126.1, 126.7, 127.7, 127.8, 128.4, 131.9, 132.7, 133.4,

134.0, 137.9, 168.2, 171.5 ppm; HRMS (ESI) m/z calcd for $\text{C}_{24}\text{H}_{21}\text{NO}_4\text{Na}^+$: 410.1368, found: 410.1374.

$[\alpha]_D^{25} = -98.7$ (c 0.94, CHCl_3) for 94% ee; HPLC (UV 254 nm, Chiralcel OD-H (0.46 cm x 25 cm), *i*-PrOH/Hexane = 3/97, 1.0 mL/min): $t_1 = 27.9$ min, $t_2 = 34.9$ min.

Ethyl 3-(6-methoxy-naphthalen-2-yl)-4-phthalimidobutanoate (4k). ^1H NMR (400 MHz, CDCl_3): δ

$= 1.03$ (3H, t, $J = 7.2$ Hz; CH_3CH_2), 2.78 (2H, d, $J = 7.2$ Hz; CH_2CH), 3.88-3.94 (6H, m; CH_3O , $-\text{CH}-$, CH_2N), 3.96 (2H, m; CH_2O), 7.08-7.11 (2H, m; Ar-H), 7.40-7.42 (1H, m; Ar-H), 7.64-7.70 (5H, m; Ar-H), 7.78-7.80 (2H, m; Ar-H) ppm; ^{13}C NMR (100 MHz, CDCl_3): $\delta = 14.1, 38.8, 40.8, 43.2, 55.3, 60.5, 105.6, 118.9, 123.3, 126.2, 126.5, 127.2, 128.9, 129.3, 131.9, 133.8, 134.0, 135.6, 157.6, 168.2, 171.6$ ppm; HRMS (ESI) calcd for $\text{C}_{25}\text{H}_{23}\text{NO}_5\text{Na}^+$:

440.1474, found: 440.1485; $[\alpha]_D^{25} = -151.2$ (c 0.80, CHCl_3) for 94% ee; HPLC (UV 254 nm, Chiralcel OD-H (0.46 cm x 25 cm), *i*-PrOH/Hexane = 2/98, 1.0 mL/min): $t_1 = 53.3$ min, $t_2 = 60.7$ min.

Ethyl 3-(2-thienyl)-4-phthalimidobutanoate (4l). ^1H NMR (400 MHz, CDCl_3): $\delta = 1.12$ (3H, t, $J = 7.2$

Hz; CH_3CH_2), 2.75 (2H, d, $J = 7.2$ Hz; CH_2CH), 3.88-4.07 (5H, m; CH_2O , $-\text{CH}-$, CH_2N), 6.90 (2H, d, $J = 4.0$ Hz; Ar-H), 7.14 (1H, m; Ar-H), 7.70-7.72 (2H, m; Ar-H), 7.82 (2H, s; Ar-H) ppm; ^{13}C NMR (100 MHz, CDCl_3): $\delta = 14.1, 36.3, 39.6, 43.7, 60.7, 123.4, 124.2, 124.9, 126.8, 131.9, 134.1, 143.5, 168.1, 171.1$ ppm; HRMS (ESI) m/z calcd for $\text{C}_{18}\text{H}_{17}\text{NO}_4\text{SNa}^+$: 366.0776, found: 366.0784; $[\alpha]_D^{25} =$

-62.8 (c 0.2, CHCl_3) for 97% ee; HPLC (UV 254 nm, Chiralpak AD (0.46 cm x 25 cm), *i*-PrOH/Hexane = 5/95, 1.0 mL/min): $t_1 = 28.3$ min, $t_2 = 34.0$ min.

General Procedure for Hydrolysis of Ethyl 3-(4-chlorophenyl)-4-phthalimidobutanoate to Form (*R*)-Baclofen.⁸

A mixture of (*R*)-**4f** (0.5 mmol, 188 mg), 6 M HCl (10 mL) was heated under reflux for 24 h. The solution was cooled and the precipitated phthalic acid was filtered off. The filtrate was evaporated to dryness and the result solid was resuspended in water (10 mL). Filtered, and the filtrate was evaporated to dryness under reduced pressure. The solid was dried under vacuum to give 118 mg (94% yield) of (*R*)-baclofen. $[\alpha]_D^{25} = -1.7$ (c 0.6, H₂O); ¹H NMR (400 MHz, D₂O): $\delta = 2.73$ -2.83 (2H, m; CH₂CH), 3.24-3.40 (3H, m; CH₂N, -CH-), 7.34-7.43 (4H, m; Ar-H) ppm; ¹³C NMR (100 MHz, D₂O): $\delta = 38.4$, 39.5, 43.6, 129.3, 129.4, 133.4, 137.0, 175.5 ppm.

Synthesis of (*R*)-Rolipram.^{9,10,11}

To a solution of **4i** (209 mg, 0.47 mmol) in THF (10 mL) was added hydrazine hydrate (0.5 mL) and the solution was stirred for 24 h. After complete consumption of the starting material, the reaction mixture was extracted with dichloromethane and concentrated in vacuum, the residue was dissolved in a mixture of Et₃N (0.5 mL) and toluene (2 mL) and the resultant solution heated at 110 °C for 20 h. After cooling to RT, the solvents were evaporated under reduced pressure. The residue was purified by flash chromatography (silica gel, EtOAc) to yield colorless crystals 91 mg (71%); $[\alpha]_D^{25} = -28.2$ (c 1.0, MeOH); ¹H NMR (400 MHz, CDCl₃): $\delta = 1.60$ -1.63 (2H, m; CH₂CH₂), 1.83-1.92 (6 H, m; -C-CH₂-CH₂), 2.45 (1 H, dd, *J* = 8.8, 16.9 Hz; CH₂CO), 2.69 (1H, dd, *J* = 8.8, 16.9 Hz; CH₂CO), 3.37-3.41 (1H, m; CH₂N), 3.63-3.65 (1H, m; -CH-), 3.74-3.79 (1H, m; CH₂N), 3.83 (3H, s; CH₃O), 4.75-4.78 (1H, m; CHO), 6.21 (1H, br; N-H), 6.76-6.84 (3H, m; Ar-H) ppm; ¹³C NMR (100 MHz, CDCl₃): $\delta = 24.1$, 32.8, 38.2, 40.0, 49.9, 56.2, 80.6, 112.2, 113.9, 118.8, 134.6, 147.9, 149.2, 177.9 ppm.

References:

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Current Data Parameters

NAME d1
EXPNO 742
PROCNO 1

F2 - Acquisition Parameters

Date_ 20080519
Time 15.14
INSTRUM drx400
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 64
DW 62.400 usec
DE 6.00 usec
TE 0.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

***** CHANNEL f1 *****

NUC1 1H
P1 8.20 usec
PL1 5.00 dB
SF01 400.1332521 MHz

F2 - Processing parameters

SI 32768
SF 400.1300216 MHz
WDW GM
SSB 0
LB -0.50 Hz
GB 0.1
PC 1.40

1D NMR plot parameters

CX 20.00 cm
CY 10.00 cm
F1P 12.000 ppm
F1 4801.56 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.65000 ppm/cm
HZCM 260.08450 Hz/cm

Chemical structure of (R_C, S_{Fc}, S_P) -3 is shown. The ^{13}C NMR spectrum (ppm) is displayed below the structure, with chemical shifts labeled for each peak.

Chemical shifts (ppm): 142.24, 136.30, 135.54, 134.84, 132.12, 131.95, 129.69, 128.76, 128.37, 128.83, 127.79, 127.20, 126.97, 126.86, 126.40, 125.08, 125.52, 125.27, 124.67, 97.75, 97.63, 97.45, 97.33, 77.36, 77.04, 76.72, 75.56, 75.41, 72.00, 71.96, 70.06, 69.93, 69.48, 58.68, 58.59, 58.30, 58.21, 31.73, 31.52, 19.39, 19.34, 0.02.

Current Data Parameters

NAME duan
EXPNO 103
PROCNO 1

F2 - Acquisition Parameters

Date_ 20070129
Time 17.40
INSTRUM drx400
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 45.3
DW 62.400 usec
DE 6.00 usec
TE 0.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====

NUC1 1H
P1 8.20 usec
PL1 5.00 dB
SFO1 400.1332521 MHz

F2 - Processing parameters

SI 32768
SF 400.1300086 MHz
WDW GM
SSB 0
LB -0.50 Hz
GB 0.1
PC 1.40

1D NMR plot parameters

CX 20.00 cm
CY 8.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCH 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

Current Data Parameters
 NAME duan
 EXPNO 105
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20070129
 Time 18.05
 INSTRUM drx400
 PROBHD 5 mm PABBO BB-
 PULPROG zg
 TD 32768
 SOLVENT CDCl₃
 NS 8
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.244532 Hz
 AQ 2.0447731 sec
 RG 40.3
 DW 62.400 usec
 DE 6.00 usec
 TE 0.0 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWPK 0.01500000 sec

===== CHANNEL f1 =====

NUC1 1H
 P1 8.20 usec
 PL1 5.00 dB
 SFO1 400.1332521 MHz

F2 - Processing parameters

SI 32768
 SF 400.1300108 MHz
 WDW GM
 SSB 0
 LB -0.50 Hz
 GB 0.1
 PC 1.40

1D NMR plot parameters

CX 20.00 cm
 CY 8.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P -1.000 ppm
 F2 -400.13 Hz
 PPMCM 0.55000 ppm/cm
 HZCM 220.07150 Hz/cm

Current Data Parameters

NAME d1
EXPNO 376
PROCNO 1

F2 - Acquisition Parameters

Date_ 20070413
Time 11.34
INSTRUM drx400
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 64
DM 62.400 usec
DE 5.00 usec
TE 295.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 8.20 usec
PL1 5.00 dB
SFO1 400.1332521 MHz

F2 - Processing parameters

SI 32768
SF 400.1300142 MHz
WDW GM
SSB 0
LB -0.50 Hz
GB 0.1
PC 1.40

1D NMR plot parameters

CX 20.00 cm
CY 8.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

Current Data Parameters

NAME d1
EXPNO 400
PROCNO 1

F2 - Acquisition Parameters

Date_ 20070419
Time 12.49
INSTRUM drx400
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SMH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 71.8
DM 62.400 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRR 0.01500000 sec

===== CHANNEL f1 =====

NUC1 1H
P1 8.20 usec
PL1 5.00 dB
SFO1 400.1332521 MHz

F2 - Processing parameters

SI 32768
SF 400.1300133 MHz
WDW GM
SSB 0
LB -0.50 Hz
GB 0.1
PC 1.40

1D NMR plot parameters

CX 20.00 cm
CY 8.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

¹³C NMR DJ-5 IN CDCl₃ 07/04/19

Current Data Parameters

NAME dj
EXPNO 401
PROCNO 1

F2 - Acquisition Parameters

Date_ 20070419
Time 12.57
INSTRUM drx400
PROBHD 5 mm PABBO BB-
PULPROG zgpgc
TD 65536
SOLVENT CDCl₃
NS 102
DS 16
SMH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 16384
DW 21.600 usec
DE 6.00 usec
TE 296.2 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec

***** CHANNEL f1 *****

NUC1 ¹³C
P1 8.20 usec
PL1 1.50 dB
SF01 100.6234215 MHz

***** CHANNEL f2 *****

CPDPRG2 waltz16
NUC2 ¹H
PCPD2 80.00 usec
PL2 5.00 dB
PL12 23.00 dB
SF02 400.1320007 MHz

F2 - Processing parameters

SF 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 4.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
CY 6.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPHMC 11.50000 ppm/cm
HZCM 1157.04688 Hz/cm

Current Data Parameters

NAME dj
EXPNO 359
PROCNO 1

F2 - Acquisition Parameters

Date_ 20070412
Time 9.54
INSTRUM drx400
PROBHD 5 mm PA880 BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 128
DW 62.400 usec
DE 6.00 usec
TE 293.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCNPK 0.01500000 sec

----- CHANNEL f1 -----

NUC1 1H
P1 8.20 usec
PL1 5.00 dB
SFO1 400.1332521 MHz

F2 - Processing parameters

SI 32768
SF 400.1300162 MHz
WDW GM
SSB 0
LB -0.50 Hz
GB 0.1
PC 1.40

1D NMR plot parameters

CX 20.00 cm
CY 10.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCH 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

13C NMR DJ-4 IN CDCL3 07/04/19

Current Data Parameters

NAME d1
EXPNO 399
PROCNO 1

F2 - Acquisition Parameters

Date_ 20070419
Time 12.44
INSTRUM drx400
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 90
DS 16
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 16384
DM 21.600 usec
DE 6.00 usec
TE 296.2 K
D1 1.00000000 sec
d11 0.03000000 sec
WDWREST 0.00000000 sec
MCWRK 0.01500000 sec

***** CHANNEL f1 *****

NUC1 13C
P1 8.20 usec
PL1 1.50 dB
SFO1 100.6234215 MHz

***** CHANNEL f2 *****

CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 5.00 dB
PL12 23.00 dB
SFO2 400.1320007 MHz

F2 - Processing parameters

S1 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 4.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
CY 6.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPNOM 11.50000 ppm/cm
HZCM 1157.04688 Hz/cm

```

1D NMR plot parameters
CX          20.00 cm
CY          10.00 cm
F1P         10.000 ppm
F1          4001.30 Hz
F2P         -1.000 ppm
F2          -400.13 Hz
PPMCM       0.55000 ppm/cm
HZCM        220.07150 Hz/cm

```

¹³C NMR DJ-1 IN CDCL₃ 08/05/16

```

=====
Injection Date : 4/27/2007 11:02:29 PM
Sample Name : sample                      Location : Vial 1
Acq. Operator : dj
Acq. Instrument : Instrument 2
Acq. Method : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed : 4/28/2007 12:12:15 AM by dj
              (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed : 5/15/2007 8:36:38 PM by bayer
              (modified after loading)

```

Area Percent Report

```

=====
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Sample Amount  :      1.80400e-1 [mg/ml] (not used in calc.)

```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	46.989	BB	2.3733	6927.28271	41.79746	49.9369
2	56.259	BB	2.7509	6944.79639	34.92500	50.0631

Totals : 1.38721e4 76.72246

Results obtained with enhanced integrator!

Summed Peaks Report

Signal 1: VWD1 A, Wavelength=254 nm

```

=====
Injection Date : 4/27/2007 8:37:36 PM
Sample Name : sample                      Location : Vial 1
Acq. Operator : dj
Acq. Instrument : Instrument 2
Acq. Method : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed : 4/27/2007 8:23:26 PM by dj
              (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed : 5/15/2007 10:28:01 PM by bayer
              (modified after loading)

```

Area Percent Report

```

=====
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Sample Amount  :      1.80400e-1 [mg/ml] (not used in calc.)

```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	47.017	MM	2.6687	2905.57812	18.14620	97.5534
2	56.550	MM	2.6206	72.86999	4.63451e-1	2.4466

Totals : 2978.44811 18.60965

Results obtained with enhanced integrator!

Summed Peaks Report

Signal 1: VWD1 A, Wavelength=254 nm


```

=====
Injection Date   : 4/26/2007 9:25:09 AM
Sample Name     : sample                      Location : Vial 1
Acq. Operator   : d1
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 4/26/2007 9:33:21 AM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 9:20:05 PM by bayer
                  (modified after loading)

```

Area Percent Report

```

Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.80400e-1 [ug/ml] (not used in calc.)

```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	*s	Height [mAU]	Area %
1	9.646	VB	0.3185	2822.03467		135.59676	49.3745
2	21.437	BB	0.7589	2893.53101		58.96656	50.6255

Totals : 5715.56567 194.56332

Results obtained with enhanced integrator!

Summed Peaks Report

Signal 1: VWD1 A, Wavelength=254 nm

```

=====
Injection Date   : 4/26/2007 8:48:29 AM
Sample Name     : sample                      Location : Vial 1
Acq. Operator   : d1
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 4/26/2007 9:21:29 AM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 10:54:34 PM by bayer
                  (modified after loading)

```

Area Percent Report

```

Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.80400e-1 [ug/ml] (not used in calc.)

```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	*s	Height [mAU]	Area %
1	9.747	VB	0.3091	99.15491		4.89565	2.0538
2	21.207	BB	0.7670	4728.65869		93.14376	97.9462

Totals : 4827.81360 98.03941

Results obtained with enhanced integrator!

Summed Peaks Report

Signal 1: VWD1 A, Wavelength=254 nm

```

=====
Injection Date : 4/25/2007 4:32:35 PM
Sample Name : sample Location : Vial 1
Acq. Operator : dj
Acq. Instrument : Instrument 2
Acq. Method : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed : 4/25/2007 4:16:29 PM by dj
              (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed : 5/15/2007 9:17:54 PM by bayer
              (modified after loading)
=====

```

Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.80400e-1 [mg/ml] (not used in calc.)
=====

```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	22.288	PB	0.8357	1050.99048	18.71658	49.5314
2	35.134	PB	1.3970	1070.87659	10.96646	50.4686

Totals : 2121.86707 29.68304

Results obtained with enhanced integrator!

Summed Peaks Report

Signal 1: VWD1 A, Wavelength=254 nm

```

=====
Injection Date : 4/25/2007 2:48:13 PM
Sample Name : sample Location : Vial 1
Acq. Operator : dj
Acq. Instrument : Instrument 2
Acq. Method : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed : 4/25/2007 1:18:49 PM by wdy
              (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed : 5/15/2007 10:56:54 PM by bayer
              (modified after loading)
=====

```

Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.80400e-1 [mg/ml] (not used in calc.)
=====

```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	23.116	BB	0.7508	60.53914	1.17844	2.8994
2	35.189	BB	1.4614	2027.42590	20.78243	97.1006

Totals : 2087.96505 21.96087

Results obtained with enhanced integrator!

Summed Peaks Report

Signal 1: VWD1 A, Wavelength=254 nm

```

=====
Injection Date   : 4/27/2007 6:07:53 PM
Sample Name     : sample                      Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZCHC.M
Last changed    : 4/27/2007 1:43:53 PM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 9:16:16 PM by bayer
                  (modified after loading)
=====

```

Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.80400e-1 [mg/ml] (not used in calc.)
=====

```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	*s	Height [mAU]	Area %
1	54.352	MM	3.0580	1317.85986		7.18251	49.9632
2	74.641	MM	4.1931	1319.80042		5.24587	50.0368

Totals : 2637.66028 12.42838

Results obtained with enhanced integrator!

Summed Peaks Report

Signal 1: VWD1 A, Wavelength=254 nm

Data File C:\HPCHEM\2\DATA\DJ\07042705.D

Sample Name: sample

```

=====
Injection Date   : 4/27/2007 4:34:30 PM
Sample Name     : sample                      Location : Vial 1
Acq. Operator   : dj
Acq. Method     : C:\HPCHEM\2\METHODS\DZCHC.M
Last changed    : 4/27/2007 1:43:53 PM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\DZCHC.M
Last changed    : 5/16/2007 8:04:18 PM by hjd
                  (modified after loading)
=====

```

Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
=====

```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	*s	Height [mAU]	Area %
1	53.937	BB	2.5940	4128.97363		22.51291	96.8695
2	74.485	BB	2.5963	133.43394		6.02974e-1	3.1305

Totals : 4262.40758 23.11589

Results obtained with enhanced integrator!

*** End of Report ***

```

=====
Injection Date   : 4/27/2007 9:58:30 AM
Sample Name     : sample                      Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZCH.M
Last changed    : 4/27/2007 11:01:31 AM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 8:39:24 PM by bayer
                  (modified after loading)
=====

```

Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.80400e-1 [mg/ml] (not used in calc.)
=====

```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	37.291	MM	2.0963	332.47919	2.64341	49.7977
2	50.419	MM	2.8279	335.18005	1.97547	50.2023

Totals : 667.65924 4.61887

Results obtained with enhanced integrator!

Summed Peaks Report

Signal 1: VWD1 A, Wavelength=254 nm

```

=====
Injection Date   : 4/27/2007 8:48:25 AM
Sample Name     : sample                      Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZCH.M
Last changed    : 4/27/2007 9:42:51 AM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 10:32:57 PM by bayer
                  (modified after loading)
=====

```

Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.80400e-1 [mg/ml] (not used in calc.)
=====

```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	37.394	MM	2.1008	1402.50891	11.12665	97.9899
2	50.727	MM	2.1828	28.77086	2.19679e-1	2.0101

Totals : 1431.27977 11.34633

Results obtained with enhanced integrator!

Summed Peaks Report

Signal 1: VWD1 A, Wavelength=254 nm

```

=====
Injection Date   : 4/27/2007 1:49:23 PM
Sample Name     : sample                      Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 4/27/2007 1:43:53 PM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 8:41:28 PM by bayer
                  (modified after loading)
=====

```

Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.80400e-1 [mg/ml] (not used in calc.)
=====

```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	42.843	MM	2.3475	258.86438	1.83790	50.3366	
2	62.108	MM	3.4173	255.40279	1.24564	49.6634	

Totals : 514.26717 3.08354

Results obtained with enhanced integrator!

Summed Peaks Report

Signal 1: VWD1 A, Wavelength=254 nm

```

=====
Injection Date   : 4/27/2007 11:06:53 AM
Sample Name     : sample                      Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 4/27/2007 11:01:31 AM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 10:32:57 PM by bayer
                  (modified after loading)
=====

```

Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.80400e-1 [mg/ml] (not used in calc.)
=====

```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	42.437	BB	2.1624	2436.85229	16.50326	97.7808	
2	61.934	BB	1.8104	55.30472	3.60449e-1	2.2192	

Totals : 2492.15702 16.86371

Results obtained with enhanced integrator!

Summed Peaks Report

Signal 1: VWD1 A, Wavelength=254 nm

```

=====
Injection Date   : 5/16/2007 8:50:19 AM
Sample Name     : samp                      Location : Vial 1
Acq. Operator   : dj
Acq. Method     : C:\HPCHEM\2\METHODS\DZCH.M
Last changed    : 5/16/2007 9:59:59 AM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\DZCH.M
Last changed    : 5/16/2007 7:54:31 PM by hjd
                  (modified after loading)
=====

```

Area Percent Report

```

Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000

```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	37.992	MM	2.2956	925.44507	6.71894	50.0096
2	56.423	MM	3.3648	925.08972	4.58220	49.9904

Totals : 1850.53479 11.30114

Results obtained with enhanced integrator!

*** End of Report ***

```

=====
Injection Date   : 5/16/2007 10:02:56 AM
Sample Name     : samp                      Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZCH.M
Last changed    : 5/16/2007 9:59:59 AM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/31/2007 9:35:46 AM by wyh
                  (modified after loading)
=====

```

Area Percent Report

```

Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.80400e-1 [mg/ml] (not used in calc.)

```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	37.754	BB	1.9298	2961.13745	20.89705	97.0880
2	56.126	MM	2.5572	88.81391	5.78845e-1	2.9120

Totals : 3049.95136 21.47590

Results obtained with enhanced integrator!

Summed Peaks Report

Signal 1: VWD1 A, Wavelength=254 nm

```

=====
Injection Date   : 4/29/2007 10:21:53 AM
Sample Name     : sample                      Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZCH.M
Last changed    : 4/29/2007 9:02:04 AM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 9:59:27 PM by bayer
                  (modified after loading)
=====

```

Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.80400e-1 [mg/ml] (not used in calc.)
=====

```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	15.870	PB	0.8070	1515.77112	27.15453	49.5519
2	24.361	PB	1.2815	1543.18640	16.93499	50.4481

Totals : 3058.95752 44.08952

Results obtained with enhanced integrator!

Summed Peaks Report

Signal 1: VWD1 A, Wavelength=254 nm

```

=====
Injection Date   : 4/29/2007 9:41:42 AM
Sample Name     : sample                      Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZCH.M
Last changed    : 4/29/2007 9:02:04 AM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 11:16:56 PM by bayer
                  (modified after loading)
=====

```

Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.80400e-1 [mg/ml] (not used in calc.)
=====

```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	16.416	MM	0.9525	2171.16895	37.99152	96.6633
2	24.986	MM	1.3222	74.94664	9.44756e-1	3.3367

Totals : 2246.11559 38.93627

Results obtained with enhanced integrator!

Summed Peaks Report

Signal 1: VWD1 A, Wavelength=254 nm

```

=====
Injection Date : 5/1/2007 3:47:27 PM
Sample Name    : sample                      Location : Vial 1
Acq. Operator  : dj
Acq. Instrument : Instrument 2
Acq. Method    : C:\HPCHEM\2\METHODS\DZCH.M
Last changed   : 5/1/2007 4:23:34 PM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed   : 5/15/2007 10:02:56 PM by bayer
                  (modified after loading)
=====

```

Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.80400e-1 [mg/ml] (not used in calc.)
=====

```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	22.456	VB	1.0568	1312.91663	17.68629	50.1832
2	30.250	BB	1.3612	1303.33203	13.13865	49.8168

Totals : 2616.24866 30.82494

Results obtained with enhanced integrator!

Summed Peaks Report

Signal 1: VWD1 A, Wavelength=254 nm

```

=====
Injection Date : 5/1/2007 2:55:29 PM
Sample Name    : sample                      Location : Vial 1
Acq. Operator  : dj
Acq. Instrument : Instrument 2
Acq. Method    : C:\HPCHEM\2\METHODS\DZCH.M
Last changed   : 5/1/2007 2:42:33 PM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed   : 5/15/2007 11:11:58 PM by bayer
                  (modified after loading)
=====

```

Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.80400e-1 [mg/ml] (not used in calc.)
=====

```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	22.841	MM	1.2616	972.34998	12.84551	98.6141
2	30.884	MM	1.2443	13.66558	1.83038e-1	1.3859

Totals : 986.01555 13.02855

Results obtained with enhanced integrator!

Summed Peaks Report

Signal 1: VWD1 A, Wavelength=254 nm


```

=====
Injection Date   : 4/25/2007 6:29:02 PM
Sample Name     : sample                      Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 4/25/2007 7:13:25 PM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 9:45:34 PM by bayer
                  (modified after loading)
=====

```

Area Percent Report

```

=====
Sorted By      : Signal
Multiplier    : 1.0000
Dilution      : 1.0000
Sample Amount  : 1.80400e-1 [mg/ml] (not used in calc.)
=====

```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	27.876	BB	0.9404	991.31299	16.08426	50.2068	
2	34.912	BB	1.4725	983.14655	9.32990	49.7932	

Totals : 1974.45953 25.41416

Results obtained with enhanced integrator!

Summed Peaks Report

Signal 1: VWD1 A, Wavelength=254 nm

```

=====
Injection Date   : 4/25/2007 5:21:00 PM
Sample Name     : sample                      Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 4/25/2007 4:16:29 PM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 11:03:38 PM by bayer
                  (modified after loading)
=====

```

Area Percent Report

```

=====
Sorted By      : Signal
Multiplier    : 1.0000
Dilution      : 1.0000
Sample Amount  : 1.80400e-1 [mg/ml] (not used in calc.)
=====

```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	27.334	MM	1.1399	9867.00684	144.26749	96.9323	
2	35.516	MM	1.7268	312.26526	3.01387	3.0677	

Totals : 1.01793e4 147.28136

Results obtained with enhanced integrator!

Summed Peaks Report

Signal 1: VWD1 A, Wavelength=254 nm

```

=====
Injection Date   : 4/25/2007 8:42:28 PM
Sample Name     : sample                      Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 4/25/2007 9:53:22 PM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 9:47:11 PM by bayer
                  (modified after loading)
=====

```

Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Sample Amount  : 1.80400e-1 [mg/ml] (not used in calc.)
=====

```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	53.260	MM	2.0304	1300.49597	10.67517	49.8907
2	60.738	MM	3.3159	1306.19397	6.56522	50.1093

Totals : 2606.68994 17.24039

Results obtained with enhanced integrator!

Summed Peaks Report

Signal 1: VWD1 A, Wavelength=254 nm

```

=====
Injection Date   : 4/25/2007 10:06:38 PM
Sample Name     : sample                      Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 4/25/2007 9:53:22 PM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 11:07:06 PM by bayer
                  (modified after loading)
=====

```

Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Sample Amount  : 1.80400e-1 [mg/ml] (not used in calc.)
=====

```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	51.631	MM	2.4550	9128.91602	61.97523	96.9640
2	61.930	MM	2.6937	285.82910	1.76848	3.0360

Totals : 9414.74512 63.74371

Results obtained with enhanced integrator!

Summed Peaks Report

Signal 1: VWD1 A, Wavelength=254 nm

```

=====
Injection Date   : 5/15/2007 10:26:58 PM
Sample Name     : samp                      Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 5/15/2007 11:20:20 PM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 11:34:08 PM by bayer
                  (modified after loading)
=====

```

Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.80400e-1 [mg/ml] (not used in calc.)
=====

```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	28.281	BB	1.4209	3817.57275		39.02330	50.0238
2	33.991	BB	1.6710	3813.93945		32.94393	49.9762

Totals : 7631.51221 71.96723

Results obtained with enhanced integrator!

Summed Peaks Report

Signal 1: VWD1 A, Wavelength=254 nm

```

=====
Injection Date   : 4/16/2007 7:40:20 PM
Sample Name     : dj                      Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 4/16/2007 8:27:38 PM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 11:29:20 PM by bayer
                  (modified after loading)
=====

```

Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.80400e-1 [mg/ml] (not used in calc.)
=====

```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	28.180	MM	1.5850	2118.77832		22.27925	98.5638
2	34.044	MM	1.6199	30.87293		3.17643e-1	1.4362

Totals : 2149.65125 22.59690

Results obtained with enhanced integrator!

Summed Peaks Report

Signal 1: VWD1 A, Wavelength=254 nm

Data File C:\HPCHEM\2\DATA\DJ\08051306.D

Sample Name: sa-1

=====

Injection Date : 13-May-08, 23:30:50
Sample Name : sa-1 Location : Vial 1
Acq. Operator : dj
Acq. Method : DZC-P.M
Analysis Method : C:\HPCHEM\2\METHODS\DZC-P.M
Last changed : 5/14/2008 12:18:41 PM by wdy
(modified after loading)

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height *s	Area %
1	17.336	MM	0.4226	295.22525	11.64255	50.1656
2	23.855	MM	0.5737	293.27643	8.51974	49.8344

Totals : 588.50168 20.16229

Results obtained with enhanced integrator!

*** End of Report ***

Data File C:\HPCHEM\2\DATA\DJ\08051204.D

Sample Name: rol

=====

Injection Date : 5/12/2008 11:22:14 PM
Sample Name : rol Location : Vial 1
Acq. Operator : dj
Acq. Method : C:\HPCHEM\2\METHODS\DZC-P.M
Last changed : 5/12/2008 11:10:39 PM by dj
(modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\DZC-P.M
Last changed : 5/13/2008 2:47:19 PM by dj
(modified after loading)

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height *s	Area %
1	17.271	MM	0.3544	10.10352	4.75128e-1	2.7944
2	23.674	MM	0.5637	351.45914	10.39186	97.2056

Totals : 361.56266 10.86699

Results obtained with enhanced integrator!

*** End of Report ***

Data File C:\HPCHEM\2\DATA\DJ\08051305.D

Sample Name: sa-1

```

=====
Injection Date : 5/13/2008 10:58:53 PM
Sample Name   : sa-1                      Location : Vial 1
Acq. Operator : dj
Acq. Method   : C:\HPCHEM\2\METHODS\DJZC-P.M
Last changed  : 5/13/2008 11:26:24 PM by dj
               (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\DJZC-P.M
Last changed   : 5/14/2008 12:15:27 PM by wdy
               (modified after loading)
=====

```

Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
=====

```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height *s	Area [mAU]	Area %
1	17.362	MM	0.4031	1.90961	7.89472e-2	0.2220	
2	23.811	MM	0.5717	858.40936	25.02334	99.7780	

Totals : 860.31897 25.10229

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 2 5/14/2008 12:15:33 PM wdy

Page 1 of 1

Data File C:\HPCHEM\2\DATA\DJ\08051304.D

Sample Name: r-rol-recr-1

```

=====
Injection Date : 5/13/2008 10:03:28 PM
Sample Name   : r-rol-recr-1             Location : Vial 1
Acq. Operator : dj
Acq. Method   : C:\HPCHEM\2\METHODS\DJZC-P.M
Last changed  : 5/13/2008 10:03:22 PM by dj
               (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\DJZC-P.M
Last changed   : 5/14/2008 12:23:55 PM by wdy
               (modified after loading)
=====

```

Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
=====

```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height *s	Area [mAU]	Area %
1	21.491	MM	0.5460	515.27545	15.72989	49.8571	
2	29.825	MM	0.7602	518.23016	11.36244	50.1429	

Totals : 1033.50562 27.09233

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 2 5/14/2008 12:25:25 PM wdy

Page 1 of 1

Data File C:\HPCHEM\2\DATA\DJ\08051203.D

Sample Name: rol

=====

Injection Date : 5/12/2008 10:34:54 PM
 Sample Name : rol Location : Vial 1
 Acq. Operator : dj
 Acq. Method : C:\HPCHEM\2\METHODS\VDZC-P.M
 Last changed : 5/12/2008 10:32:03 PM by dj
 (modified after loading)
 Analysis Method : C:\HPCHEM\2\METHODS\VDZC-P.M
 Last changed : 5/13/2008 2:49:49 PM by dj
 (modified after loading)

Area Percent Report

Sorted By : Signal
 Multiplier : 1.0000
 Dilution : 1.0000

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %s	Height [mAU]	Area %
1	21.295	MM	0.4679	13.03781	4.64367e-1	2.4927	
2	29.494	MM	0.7500	509.99200	11.33391	97.5073	

Totals : 523.02981 11.79828

Results obtained with enhanced integrator!

*** End of Report ***

Data File C:\HPCHEM\2\DATA\DJ\08051303.D

Sample Name: r-rol-recr-1

=====

Injection Date : 5/13/2008 9:23:23 PM
 Sample Name : r-rol-recr-1 Location : Vial 1
 Acq. Operator : dj
 Acq. Method : C:\HPCHEM\2\METHODS\VDZC-P.M
 Last changed : 5/9/2008 11:09:11 PM by AD-H
 Analysis Method : C:\HPCHEM\2\METHODS\VDZC-P.M
 Last changed : 5/14/2008 12:23:00 PM by wdy
 (modified after loading)

Area Percent Report

Sorted By : Signal
 Multiplier : 1.0000
 Dilution : 1.0000

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %s	Height [mAU]	Area %
1	21.534	MM	0.5247	3.28951	1.04483e-1	0.4621	
2	29.831	MM	0.7664	708.49658	15.40775	99.5379	

Totals : 711.78609 15.51224

Results obtained with enhanced integrator!

*** End of Report ***