Supporting Information for

Asymmetric Nitroallylation of Arylboronic Acids with Nitroallyl Acetates Catalyzed by Chiral Rhodium Complexes and Its Application in a Concise Total Synthesis of Optically Pure (+)- γ -Lycorane

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General. All manipulations were carried out under an argon atmosphere. NMR spectra were recorded on a Bruker-300MHz spectrometer. Mass spectra were recorded on VG 7070E. Elemental analysis was recorded on Italy CARCOERBA 1106. HR-MS (ESI) spectra were recorded on Bruker BioTOF Q.

Materials. THF and dioxane were dried with sodium/ benzophenone. Diethyl ether, petroleum ether (PE) and ethyl acetate for column chromatography were distilled before use.

General procedure for the asymmetric nitroallylation of arylboronic acids with nitroallyl acetates catalyzed by chiral rhodium complexes: Under Argon atmosphere, $[\text{Rh}(\text{OH})(\text{COD})]_2^1$ (3.4 mg, 15 μmol , 5 mol%), (S)-BINAP (11.2 mg, 18 μmol , 6 mol%) and phenylboronic acid (183 mg, 1.5 mmol, 5 equiv) in 1,4-dioxane (1 mL) was stirred at 50 $^\circ\text{C}$ for 2 h. Substrate **1a**² (0.3 mmol) in 1,4-dioxane (1 mL) was added to the above mixture, and followed by addition of water (200 μL). The reaction mixture was stirred at 50 $^\circ\text{C}$ for 20 h. After evaporation of the solvent, the residue was purified by column chromatography on silica (Diethyl ether/ petroleum ether =1/20) to yield 1-nitro-6-phenylcyclohexene (**2a**) as a white solid (34 mg, 56% yield). m.p. 71.3-72.0 $^\circ\text{C}$; $[\alpha]_{\text{D}}^{20} = +92.5$ ($c = 1.07$, CHCl_3); IR (KBr): γ 3456, 3023, 2946, 2920, 2867, 1663, 1510, 1491, 1451, 1413, 1333, 1077, 938 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz): δ (ppm) 1.55-1.62 (m, 2H), 1.89-1.92 (m, 1H), 2.01-2.03 (m, 1H), 2.40 - 2.49 (m, 2H), 4.29 (brs, 1H), 7.14-7.34 (m, 5H), 7.57 (t, $J = 4.0$ Hz, 1H); ^{13}C NMR (CDCl_3 , 75 MHz): δ (ppm) 16.5, 25.0, 31.5, 39.5, 126.7, 127.1, 128.5, 136.2, 141.9, 150.9; HRMS for $\text{C}_{12}\text{H}_{13}\text{NO}_2$, calcd: 203.0940, found: 203.0928. Anal. calcd for $\text{C}_{12}\text{H}_{13}\text{NO}_2$: C, 70.93; H, 6.41; N, 6.89. Found: C, 70.84; H, 6.44; N, 6.84. Enantiomeric excess: 97%, determined

by chiral GC analysis (CP–Cyclodextrin- 2, 3, 6 -M-19, 0.25 mm×25 m, column temperature = 168 °C (isothermal), inject temperature = 240 °C, detector temperature = 260 °C, inlet pressure = 10 psi).

Crystal data of **2a**: C₁₂H₁₃NO₂, MW= 203.23, Orthorhombic, space group P2₁2₁2₁, *a*= 6.1206(5), *b*= 12.592(1), *c*= 14.007(1) Å, α = 90°, β = 90°, γ = 90°, *U*= 1079.57(12) Å³, *T*= 290(2) K, *Z*= 4, *D_c*= 1.250 mg/m³, μ = 0.085 mm⁻¹, λ = 0.71073 Å, *F*(000) 432, crystal size: 0.56 x 0.50 x 0.42 mm³, 1483 reflections collected, 1380 [R(int) = 0.0122]; Refinement method: Full-matrix least-squares on *F*²; goodness-of-fit on *F*²= 0.961, final R indices [*I*> 2σ(*I*)] R1 = 0.0327, wR2 = 0.0652.

1–Nitro–6-(4-methylphenyl)cyclohexene (2b), White solid (41 mg, 63% yield). m.p. 54.4-55.9 °C; [α]_D²⁰ = +84.3 (*c*= 0.86, CHCl₃); IR (KBr): γ 3447, 3022, 2950, 2872, 1660, 1506, 1336, 809 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz): δ (ppm) 1.57(m, 2H), 1.89(m, 1H), 2.00 (m, 1H), 2.32 (s, 3H), 2.39 (m, 2H), 4.26 (brs, 1H), 7.04 (d, *J*= 8.0 Hz, 2H), 7.14 (d, *J*= 8.0 Hz, 2H), 7.55 (t, *J*= 4.0 Hz, 1H); ¹³C NMR(CDCl₃, 75 MHz): δ (ppm) 16.4, 20.8, 24.9, 31.5, 39.0, 126.9, 129.1, 135.9, 136.2, 138.8, 151.0; HRMS for C₁₃H₁₅NO₂, calcd: 217.1097, found: 217.1095. Enantiomeric excess: 95%, determined by chiral GC analysis (CP-Cyclodextrin-2, 3, 6 – M-19, 0.25 mm×25 m, column temperature= 168 °C (isothermal), inject temperature= 240 °C, detector temperature= 260 °C, inlet pressure = 10 psi).

1–Nitro–6-(4-methoxyphenyl)cyclohexene (2c), White solid (40.5 mg, 58% yield). m.p. 56.8-57.9 °C; [α]_D²⁰ = +93.4 (*c*=1.06, CHCl₃); IR (KBr): γ 3494, 2996, 2859, 2936, 2833, 1658, 1613, 1507, 1460, 1448, 1336, 1258, 1177, 1031, 823, 815 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz): δ (ppm) 1.54-1.61 (m, 2H), 1.82-1.88 (m, 1H), 1.96-2.00 (m, 1H), 2.39 -2.47 (m, 2H), 3.77 (s, 3H), 4.23 (brs, 1H), 6.81 (d, *J*= 11.5 Hz, 2H), 7.04 (d, *J*= 11.5 Hz, 2H), 7.52 (t, *J*= 4.0 Hz, 1H); ¹³C NMR (CDCl₃, 75 MHz): δ (ppm) 16.5, 25.0, 31.6, 38.7, 55.2, 113.9, 128.1, 134.0, 135.8, 151.2, 158.3; HRMS for C₁₃H₁₅NO₃, calcd: 233.1046, found: 233.1033. Enantiomeric excess: 96%, determined by chiral HPLC (Daicel Chiralcel OD; hexane: isopropanol = 99:1, flow = 1.0mL / min, *R*-isomer, *t_R*= 12.4 min, *S*-isomer, *t_R*= 14.5 min).

1–Nitro–6-(4-chlorophenyl)cyclohexene (2d), colorless oil (45 mg, 64% yield); [α]_D²⁰ = +102.2 (*c*= 1.15, CHCl₃); IR (neat): γ 2943, 2867, 1662, 1517, 1488, 1336, 1091, 1013, 819; ¹H NMR

(CDCl₃, 300 MHz): δ (ppm) 1.25-1.62 (m, 2H), 1.84-1.87 (m, 1H), 2.03 (m, 1H), 2.41-2.48 (m, 2H), 4.24 (brs, 1H), 7.06 (d, J = 11.0 Hz, 2H), 7.24 (d, J = 11.0 Hz, 2H), 7.56 (t, J = 4.0 Hz, 1H); ¹³C NMR (CDCl₃, 75 MHz): δ (ppm) 16.5, 24.9, 31.4, 39.0, 128.4, 128.7, 132.5, 136.6, 140.5, 150.5; HRMS for C₁₂H₁₂ClNO₂, calcd: 237.0551, found: 237.0569. Enantiomeric excess: 99%; determined by chiral GC analysis (CP-Cyclodextrin -2, 3, 6 -M-19, 0.25 mm×25 m, column temperature= 168 °C (iso thermal), inject temperature= 240 °C, detector temperature= 260 °C, inlet pressure = 10 psi).

1-Nitro-6-(4-fluorophenyl)cyclohexene (2e), colorless oil (40 mg, 61% yield); [α]_D²⁰ = +87.3 (c = 1.22, CHCl₃); IR (neat): γ 3069, 2945, 2868, 1662, 1602, 1509, 1336, 1222, 826, 816 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz): δ (ppm) 1.52-1.61 (m, 2H), 1.84-1.87 (m, 1H), 2.02 (m, 1H), 2.39-2.48 (m, 2H), 4.26 (brs, 1H), 6.95-7.01 (m, 2H), 7.10- 7.13 (m, 2H), 7.55 (t, J = 4.0 Hz, 1H); ¹³C NMR (CDCl₃, 75MHz): δ (ppm) 16.4, 24.9, 31.5, 38.8, 115.1, 115.4, 128.5, 128.6, 136.4, 137.7, 150.8, 160.0, 163.2; HRMS for C₁₂H₁₂FNO₂, calcd: 221.0846, found: 221.0834. Enantiomeric excess: 98%, determined by Chiral GC analysis (CP -Cyclodextrin-2, 3, 6-M-19, 0.25 mm×25 m, column temperature= 168 °C (isothermal), inject temperature = 240 °C, detector temperature= 260 °C, inlet pressure= 10 psi).

1-Nitro-6-(4-*tert*-butylphenyl)cyclohexene (2f), white solid (52 mg, 67% yield). mp: 82.0-83.5 °C; [α]_D²⁰ = + 98.2 (c = 0.70, CHCl₃); IR (KBr): γ 2958, 2865, 1659, 1513, 1342, 1330, 1075, 1018, 823 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz): δ (ppm) 1.28 (s, 9H), 1.57-1.86 (m, 2H), 1.87-2.02 (m, 2H), 2.40-2.48 (m, 2H), 4.25 (brs, 1H), 7.04 (d, J = 8.3 Hz, 2H), 7.28 (d, J = 8.3 Hz, 2H), 7.53 (t, J = 4.0 Hz, 1H); ¹³C NMR (CDCl₃, 75 MHz): δ (ppm) 16.6, 24.9, 31.2, 31.4, 34.3, 39.0, 125.3, 126.7, 135.8, 138.7, 149.3, 149.3, 151.1; HRMS for C₁₆H₂₁NO₂, calcd: 259.1567, found: 259.1554. Enantiomeric excess: 90%, determined by chiral GC analysis (CP-Cyclodextrin-2, 3, 6-M-19, 0.25 mm×25 m, column temperature= 168 °C (isothermal), inject temperature= 240 °C, detector temperature = 260 °C, inlet pressure = 10 psi).

1-Nitro-6-(4-trifluoromethylphenyl)cyclohexene (2g), colorless oil (42 mg, 52% yield); [α]_D²⁰ = +107.2 (c = 0.83, CHCl₃); IR (neat): γ 2943, 2871, 1664, 1618, 1519, 1325, 1163, 1112, 1066, 1017; ¹H NMR (CDCl₃, 300 MHz): δ (ppm) 1.52-1.63 (m, 2H), 1.87-1.90 (m, 1H), 2.08 (m, 1H), 2.44-2.52 (m, 2H), 4.32 (brs, 1H), 7.25 (d, J = 8.2 Hz, 2H), 7.55 (d, J = 8.2 Hz, 2H), 7.62 (t, J = 4.0

Hz, 1H); ^{13}C NMR (CDCl_3 , 75 MHz): δ (ppm) 16.5, 24.9, 31.4, 39.5, 122.2, 125.5, 127.4, 128.4, 128.9, 129.3, 129.4, 137.0, 146.1, 150.2; HRMS for $\text{C}_{13}\text{H}_{12}\text{F}_3\text{NO}_2$, calcd: 271.0814, found: 271.0808. Enantiomeric excess: 94%, determined by chiral GC analysis (CP- Cyclodextrin -2, 3, 6-M-19, 0.25 mm $\times$ 25 m, column temperature = 168 °C (isothermal), inject temperature = 240 °C, detector temperature = 260 °C, inlet pressure = 10 psi).

1-Nitro-6-(3-methoxyphenyl)cyclohexene (2h), white solid (41 mg, 58% yield). m.p.: 81.7-83.6 °C; $[\alpha]_{\text{D}}^{20} = +108.5$ ($c = 1.29$, CHCl_3); IR (KBr): γ 2942, 2866, 2835, 1662, 1600, 1583, 1515, 1487, 1330, 1285, 1263, 1042, 781 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz): δ (ppm) 1.57-1.62 (m, 2H), 1.88-1.93 (m, 1H), 1.98-2.17 (m, 1H), 2.40-2.48 (m, 2H), 3.78 (s, 3H), 4.24 (brs, 1H), 6.68 (s, 1H), 6.72-6.78 (m, 2H), 7.19 (m, 1H), 7.55 (d, $J = 8.2$ Hz, 2H), 7.62 (t, $J = 4.0$ Hz, 1H); ^{13}C NMR (CDCl_3 , 75 MHz): δ (ppm) 16.6, 25.0, 31.4, 39.5, 55.1, 111.6, 113.4, 119.5, 129.5, 136.2, 143.6, 150.8, 159.7; HRMS for $\text{C}_{13}\text{H}_{15}\text{NO}_3$, calcd: 233.1046, found: 233.1055. Enantiomeric excess: 96%, determined by chiral HPLC analysis (Daicel Chiralcel OD; hexane: isopropanol = 99:1, flow = 1.0 mL / min, *R*-isomer, $t_{\text{R}} = 22.5$ min, *S*-isomer, $t_{\text{R}} = 25.3$ min).

1-Nitro-6-(3,4-methylenedioxy-phenyl)cyclohexene (2i), colorless oil (48 mg, 65% yield); $[\alpha]_{\text{D}}^{20} = +98.2$ ($c = 1.40$, CHCl_3); IR (neat): γ 2942, 2897, 1661, 1607, 1513, 1486, 1439, 1333, 1249, 1231, 1037, 934, 809 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz): δ (ppm) 1.55-1.62 (m, 2H), 1.81-1.87 (m, 1H), 1.95-2.00 (m, 1H), 2.38-2.47 (m, 2H), 4.19 (brs, 1H), 5.92 (s, 2H), 6.57-6.84 (m, 3H), 7.52 (t, $J = 4.0$ Hz, 1H); ^{13}C NMR (CDCl_3 , 75 MHz): δ (ppm) 16.4, 24.9, 31.5, 39.1, 100.9, 107.7, 108.1, 120.0, 135.8, 136.1, 146.3, 147.774, 150.9; HRMS for $\text{C}_{13}\text{H}_{13}\text{NO}_4$, calcd: 247.0839, found: 247.0852. Enantiomeric excess: 98%, determined by chiral HPLC analysis (Daicel Chiralcel OD; hexane: isopropanol = 99:1, flow = 1.0 mL / min, *R*-isomer, $t_{\text{R}} = 15.3$ min, *S*-isomer, $t_{\text{R}} = 21.5$ min).

Methyl 1,2-cis-2,3-cis-2-nitro-3-benzo[1,3]dioxol-5-ylcyclohexylacetate (8). Colorless oil (138 mg, 72% yield); IR (KBr): 2939, 2864, 1733, 1543, 1504, 1440; ^1H NMR (CDCl_3 , 300 MHz): δ (ppm) 1.48-1.78 (m, 4H), 2.03 (m, 1H), 2.28 (m, 2H), 2.39- 2.45 (m, 2H), 2.45-2.99 (m, 1H), 3.68 (s, 3H), 4.98 (t, $J = 4.0$ Hz, 1H), 5.91 (s, 2H), 6.63 -6.71 (m, 2H), 6.74 (d, $J = 8.1$ Hz, 1H); ^{13}C NMR (CDCl_3 , 75MHz): δ (ppm) 24.1, 24.9, 25.3, 36.7, 36.9, 45.5, 51.8, 91.1, 101.0, 107.6, 108.3, 120.4, 133.7, 146.7, 147.8, 171.8; HRMS for $\text{C}_{16}\text{H}_{19}\text{NO}_6$, calcd: 321.1207, found: 321.1206.

3a, 7a-cis-7, 7a-cis-7-benzo [1, 3] dioxol-5-yl-octahydroindol-2-one (9): To a solution of **8** (96mg, 0.3mmol) in absolute ethanol (1 mL) in autoclave was added Raney-nickel (5 mol%) previously rinsed with absolute ethanol. Hydrogen was introduced into the autoclave until the pressure reached 80 atm. The autoclave was warmed up to 55 °C and was maintained at the temperature for 24 h. After the autoclave was cooled down to room temperature, the mixture was filtered and the filtrate was concentrated. Recrystallization from CHCl₃ / hexane gave **9** as a white solid (74 mg, 95% yield); [α]_D²⁰ = 112.3 (*c* = 1.30, CHCl₃); m.p.: 185.9-187.2 °C; IR (KBr): γ 3252, 2927, 2915, 2853, 1681, 1502, 1488, 1242, 1230, 1039; ¹H NMR (CDCl₃, 300 MHz): δ (ppm) 1.25-1.33 (m, 2H), 1.68-1.75 (m, 3H), 1.82(m, 1H), 1.96 (d, *J*=16.0 Hz, 1H), 2.47 (m, 1H), 2.52 (m, 1H), 2.78-2.82 (m, 1H), 3.84 (t, *J* = 4.2 Hz, 1H), 5.33 (brs, 1H), 5.95 (s, 2H), 6.63 (d, *J*= 1.5 Hz, 1H), 6.66 (dd, *J*= 1.5 Hz, 7.9 Hz, 1H), 6.75 (d, *J*= 7.9 Hz, 1H); ¹³C NMR (CDCl₃, 75 MHz): δ (ppm) 24.2, 27.5, 35.1, 40.2, 43.7, 58.8, 101.0, 107.5, 108.5, 119.7, 136.2, 146.4, 148.0, 177.9; HRMS for C₁₅H₁₇NO₃, calcd: 259.1203, found: 259.1203. Enantiomeric excess: 98%, determined by chiral HPLC (Daicel Chiralcel OJ-H; hexane: isopropanol = 70:30, flow = 1.0 mL / min, *R*- isomer, *t*_R = 8.1 min, *S*-isomer, *t*_R = 13.3 min).

Preparation and characterization of compound 10: To a solution of **9** (52 mg, 0.20 mmol) in anhydrous Cl(CH₂)₂Cl (5 mL) was added, sequentially, paraformaldehyde (23.1 mg, 0.77 mmol) and CF₃CO₂H (0.19 mL, 2.5 mmol) at room temperature. The reaction mixture was stirred at room temperature for 24 h, and then quenched with saturated aqueous NaHCO₃ (10 mL) followed by addition of CH₂Cl₂ (40 mL). The organic layer was separated, and the aqueous phases were washed by brine, and dried over NaSO₄. After removal of the solvent under reduced pressure, the residue was purified by column chromatography on silica gel (AcOEt / PET = 3/1) to give **10** (47 mg, 88% yield) as a white solid. m.p.: 144.0-147.7 °C; IR (KBr): γ 2927, 2854, 1676, 1503, 1483, 1440, 1418; ¹H NMR (CDCl₃, 300 MHz): δ (ppm) 1.12 -1.32 (m, 3H), 1.68 (m, 3H), 2.04 (d, *J*= 16.0 Hz, 1H), 2.39 (m, 1H), 2.51 (dd, *J* = 16.0 Hz, 6.8 Hz, 1H), 2.69 (m, 1H), 3.73 (t, *J* = 4.5 Hz, 1H), 3.76 (d, *J*= 17.3 Hz, 1H), 4.49 (d, *J*= 17.3 Hz, 1H), 5.89 (m, 2H), 6.57 (s, 1H), 6.59 (s, 1H); ¹³C NMR (CDCl₃, 75 MHz): δ (ppm) 23.5, 27.8, 30.2, 32.9, 39.7, 40.2, 42.6, 55.6, 100.9, 106.6, 108.4, 123.2, 131.5, 146.5, 146.6, 175.6; HRMS for C₁₆H₁₇NO₃, calcd: 271.1203, found: 271.1193.

(+)- γ -Lycorane: A mixture of **10** (40 mg, 0.15 mmol) and LiAlH₄ (17.1 mg, 0.45 mmol) in THF (5

mL) was stirred at reflux for 18 h. NaSO₄ was added, and the precipitate was removed by filtration. The filtrate was concentrated, and the residue was purified by flash column chromatography (Et₂O) to give (+)- γ -Lycorane as a colorless oil (37 mg, 98% yield); $[\alpha]^{20}_D = +17.3$ ($c = 0.98$, EtOH), [lit.³ $[\alpha]^{20}_D +17.1$ ($c = 0.25$, EtOH)]; IR (KBr): γ 2928, 2848, 1505, 1483, 1375, 1318, 1245, 1226, 1038; ¹HNMR (CDCl₃, 300 MHz): δ (ppm) 1.25-1.35 (m, 5H), 1.42 (m, 3H), 1.69-2.20 (m, 3H), 2.36 (t, $J = 4.5$ Hz, 1H), 2.70 (m, 1H), 3.18 (d, $J = 14.4$ Hz, 1H), 3.23 (m, 1H), 3.99 (d, $J = 14.4$ Hz, 1H), 5.87 (m, 2H), 6.49 (s, 1H), 6.61 (s, 1H); ¹³C NMR (CDCl₃, 75 MHz): δ (ppm) 25.2, 29.2, 30.3, 31.7, 37.3, 39.4, 53.7, 57.1, 62.8, 100.6, 106.2, 108.3, 127.3, 133.1, 145.6, 146.0; HRMS for C₁₆H₂₀NO₂, $[M+H]^+$ calcd: 258.1489, found: 258.1496. The spectral data are in agreement with those reported in the literature.⁴

Determination of the absolute configuration of compound **2a**:

To a solution of the nitro olefin **2a** (40.6 mg, 0.2 mmol) in ethanol (10 mL) at 0 °C was added NaBH₄ (15.2 mg, 0.4 mmol) over 20 min. After stirring for 2 h at r.t., the ethanol was removed and 2N HCl (5 mL) was added. The product was extracted with CH₂Cl₂ (3 × 10 mL) and combined organic layers were dried (NaSO₄), and the CH₂Cl₂ was removed to give the crude nitro **2a'** (40 mg, 98% yield).⁵ To a solution of **2a'** in methanol (0.2 mL) was added sodium methoxide (13.2 mg, 0.24 mmol) in methanol (0.2 mL) at 0 °C and the mixture was stirred for 0.5 h. To the nitronate solution was added a mixture of concentrated sulfuric acid (40 μ L) and methanol (0.2 mL). During the addition, the mixture was kept at -40 °C and after additional stirring at -20 °C for 1 h, it was poured into dichloromethane (50 mL). The organic layer was washed with water (20 mL) and was extracted with dichloromethane (20 mL). The combined organic layers were dried over NaSO₄, and the CH₂Cl₂ was removed. Dichloromethane (20 mL) and hydrochloric acid (2M, 20 mL) were added, and the mixture was stirred at r.t for 2 h. The aqueous layer was extracted with dichloromethane, and the combined organic layers were dried over NaSO₄. After the CH₂Cl₂ was removed, the residue was purified by column chromatography on silica gel to give 2-phenylcyclohexanone (**(R)-2a''**): $[\alpha]^{20}_D = +94$ ($c = 0.75$, benzene).⁶ The reported specific rotation of (**2S**)-**2a''** is $[\alpha]^{20}_D -114$ ($c = 0.60$, benzene).⁷

References

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Current Data Parameters
NAME 1367-d1-535
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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1D NMR plot parameters
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OL-Zn-3a

Current Data Parameters
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PROCNO 1

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P2 80.00 usec
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PL13 17.71 dB
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F2 - Processing parameters
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1D NMR plot parameters
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Current Data Parameters
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PROCNO 1

F2 - Acquisition Parameters
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***** CHANNEL f1 *****
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F2 - Processing parameters
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WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 20.00 cm
FIP 10.000 ppm
F1 300.1300128 MHz
F2 -150.05 Hz
P1 0.52900 ppm/cm
H1ZCM 157.56825 Hz/cm

Current Data Parameters
NAME 1367-01-569
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050330
Time 12:00
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 256
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.445188 sec
RG 8192
CW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.00000000 sec
D12 0.00000000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 6.50 usec
PL1 -6.00 dB
SFO1 75.4775998 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
P2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312088 MHz

F2 - Processing parameters
SI 32768
SF 75.4678787 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
FIP 200.005 ppm
F1 150.98191 Hz
F2 -0.005 ppm
F3 -0.38 Hz
P1 10.00050 ppm/cm
H1ZCM 754.71933 Hz/cm

Current Data Parameters
NAME 1367-xyj-116
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050426
Time 15.12
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7326011 sec
RG 64
DM 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320082 MHz

F2 - Processing parameters
SI 32768
SF 300.1300122 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 20.00 cm
FIP 10.000 ppm
FI 3001.30 Hz
F2P -0.500 ppm
F2 150.06 Hz
PRINCH 0.52500 ppm/cm
HZCH 157.56825 Hz/cm

xyj-KP4-1

Current Data Parameters
NAME 1367-xyj-116
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050426
Time 15.48
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 276
DS 4
SWH 22675.736 Hz
FIDRES 0.346804 Hz
AQ 1.4451168 sec
RG 8192
DM 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00000000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.477598 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
P2 60.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677536 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
FIP 200.005 ppm
FI 15093.51 Hz
F2P -0.36 ppm
F2 75.46 Hz
PRINCH 10.00050 ppm/cm
HZCH 754.71527 Hz/cm

xyj-Kp5

Current Data Parameters
NAME 1367-xyj-111
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050419
Time 18.00
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 256
DM 83.400 usec
DE 6.00 usec
TE 300.0 K
U1 1.00000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320882 MHz

F2 - Processing parameters
SI 32768
SF 300.1300122 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCH 0.52500 ppm/cm
HZCM 157.56825 Hz/cm

Current Data Parameters
NAME 1367-xyj-111
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050419
Time 18.05
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 120
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4491188 sec
RG 8192
DM 23.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.4775998 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677551 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
F1P 220.005 ppm
F1 15603.29 Hz
F2P -0.005 ppm
F2 -0.38 Hz
PPMCH 11.00050 ppm/cm
HZCM 830.18311 Hz/cm

```

Current Data Parameters
NAME      1367-91111
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_     20050420
Time      11:48
INSTRUM    PROSPIN
PROBHD     5 mm QNP 1H/1
PULPROG    zgpg30
TD         1024
SOLVENT    DMSO
NS         1000
DS         4
SWH         2189.314 MHz
F2RES      2.140968 Hz
AQ         0.234341 sec
RG          100
IN          228.800 usec
NUC1        1   6.000000 usec
TE         300.2
DE         0.0500000 sec
AQ1         1.0504760 sec
NUC2        13  0.000000 usec
DE2        0.0000000 sec
IN2         0.0000000 sec
MCN1        1
MCN2        1
===== CHANNEL f1 =====
F1         1      7.00 usec
P1          2    -2.00 usec
PC          300.130000 MHz

F1 - Acquisition parameters
SI         1
TD         512
F2F1       218.913 MHz
F2RES      4.268813 Hz
AQ          0.234341 sec
PROBHD     States-T2
=====

F2 - Processing parameters
SI         1367
SF          102.0124 MHz
WDW         HANN
SSB         0
GB          0
PC          1.00

F1 - Processing parameters
SI         1367
SF          102.0124 MHz
WDW         States-T2
SSB         0
GB          0
PC          1.00

2D NMR data parameters
CX2        15.00 usec
FX2         15.00 usec
F2F1       18.00000 MHz
F2RES      23.40 Hz
AQ          0.548 usec
F1F2       7.00000 MHz
F2F1F2     239.20 Hz
AQ1         0.234341 sec
DE1         0.0500000 usec
F2CN2      145.86765 Hz/cps
F1CN2      0.865700 Hz/cps
F1CN2CN2   145.86765 Hz/cps

```

Current Data Parameters
NAME 1367-xyj-128
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050522
Time 18.13
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 512
OW 83.400 usec
OE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SF01 300.132082 MHz

F2 - Processing parameters
SI 32768
SF 300.1300126 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
FIP 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PFCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm

xyj-KP6-2

Current Data Parameters
NAME 1367-xyj-128
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050523
Time 9.20
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 624
DS 4
SWH 22675.736 Hz
FIDRES 0.346094 Hz
AQ 1.4491188 sec
RG 8152
OW 22.050 usec
OE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SF01 75.477598 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SF02 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677551 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
FIP 200.005 ppm
F1 15693.51 Hz
F2P -5.005 ppm
F2 -377.72 Hz
PFCM 10.25050 ppm/cm
HZCM 773.56828 Hz/cm

Current Data Parameters
NAME 1367-d1-605
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050522
Time 15.09
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 512
OW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320682 MHz

F2 - Processing parameters
SI 32768
SF 300.1300125 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
FIP 10.000 ppm
F1 3001.30 Hz
F2 -150.00 ppm
F2 150.00 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm

DL-P

Current Data Parameters
NAME 1367-d1-605
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050522
Time 15.14
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 292
DS 4
SWH 22575.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
OW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00000000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.4775598 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677518 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
FIP 200.000 ppm
F1 15053.93 Hz
F2 -5.000 ppm
F2 -377.72 Hz
PPMCM 10.25050 ppm/cm
HZCM 773.58221 Hz/cm

11.898

GC Spectroscopy

Peak No.	RT(min)	Height	Area	% Area
1	8.448	53.385	1383.000	0.4067
2	11.565	23644.205	169898.766	49.9658
3	11.898	24106.625	168748.172	49.6274
Total		47804.215	340029.938	100.0000

GC Spectroscopy

Peak No.	RT(min)	Height	Area	% Area
1	11.598	976.781	5968.438	2.0723
2	11.898	44995.953	282048.281	97.9277
Total		45972.734	288016.719	100.0000

Peak No.	RT(min)	Height	Area	% Area
1	2.198	1688.608	35677.199	1.8732
2	21.665	172.576	10506.529	0.5516
3	24.198	265.953	16151.837	0.8480
4	25.665	53381.750	926318.875	48.6359
5	26.532	63053.223	915943.625	48.0912
Total		118562.110	1904598.065	100.0000

Peak No.	RT(min)	Height	Area	% Area
1	2.007	262.556	1217.450	0.2541
2	3.248	221.652	1912.620	0.3992
3	3.348	266.087	1242.450	0.2593
4	3.523	2807.348	6518.280	1.3604
5	4.157	1671.118	4672.275	0.9751
6	5.582	2452.647	7556.600	1.5770
7	6.815	593.727	2271.700	0.4741
8	16.865	93.558	1294.416	0.2701
9	17.365	1250.140	10707.479	2.2346
10	17.832	200.696	3011.700	0.6285
11	25.198	618.730	11814.473	2.4657
12	26.198	30722.973	426942.094	89.1019
Total		41161.230	479161.537	100.0000

Peak No.	RT(min)	Height	Area	% Area
1	4.765	3365.444	15588.560	1.8827
2	13.632	427.254	18123.023	2.1888
3	15.265	263.422	15125.823	1.8268
4	16.532	47785.301	392320.563	47.3816
5	17.232	47484.051	386844.594	46.7202
Total		99325.472	828002.563	100.0000

Peak No.	RT(min)	Height	Area	% Area
1	17.765	938.836	9384.417	1.0360
2	18.632	92089.922	896404.500	98.9640
Total		93028.758	905788.917	100.0000

Peak No.	RT(min)	Height	Area	% Area
1	34.848	4252.729	72584.242	50.1869
2	35.698	4299.750	72043.555	49.8131
Total		8552.479	144627.797	100.0000

Peak No.	RT(min)	Height	Area	% Area
1	22.932	11783.612	144855.188	11.8763
2	25.732	532.000	41827.398	3.4293
3	34.865	2161.471	55576.078	4.5565
4	35.932	45424.727	977439.313	80.1378
Total		59901.810	1219697.977	100.0000

GC Spectroscopy

Peak No.	RT(min)	Height	Area	% Area
1	3.082	1479.429	2192.100	1.0952
2	5.665	182.700	1460.251	0.7296
3	10.898	85.461	1277.200	0.6381
4	11.865	174.849	3494.204	1.7458
5	12.365	304.049	6376.963	3.1861
6	12.632	311.156	5923.470	2.9596
7	13.398	580.463	12176.129	6.0836
8	13.932	184.677	2518.174	1.2582
9	14.532	204.918	8630.977	4.3123
10	15.065	198.131	5402.146	2.6991
11	15.632	10154.358	75915.336	37.9299
12	16.432	10053.680	74779.664	37.3624
Total		23913.870	200146.612	100.0000

GC Spectroscopy

Peak No.	RT(min)	Height	Area	% Area
1	10.598	993.935	38361.500	17.5286
2	13.432	68.405	1457.452	0.6660
3	13.798	83.671	1782.269	0.8144
4	14.265	564.646	5726.372	2.6166
5	15.065	23493.316	167245.344	76.4198
6	15.965	118.054	2977.100	1.3603
7	18.332	38.378	1300.900	0.5944
Total		25360.406	218850.936	100.0000

GC Spectroscopy

Peak No.	RT(min)	Height	Area	% Area
1	11.132	1484.923	8422.314	50.7121
2	11.365	1587.015	8185.781	49.2879
Total		3071.938	16608.095	100.0000

GC Spectroscopy

Peak No.	RT(min)	Height	Area	% Area
1	11.593	344.618	1831.899	0.6493
2	11.938	44855.289	280286.281	99.3507
Total		45199.907	282118.180	100.0000

ulas

Project Name: Defaults
Reported by User: System

ulas

Project Name: Defaults
Reported by User: System

SAMPLE INFORMATION			
Sample Name:	dl-3b-0	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	3/28/05 5:53:42 PM
Vial:	1	Acq. Method:	100A
Injection #:	4	Date Processed:	3/28/05 6:12:43 PM
Injection Volume:	20.00 ul	Channel Name:	2487Channel 1
Run Time:	180.00 Minutes	Sample Set Name:

RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1 4.001	17523	0.93	1119	1.79
2 12.255	939309	48.78	33260	53.07
3 14.506	930073	48.29	28291	45.14

SAMPLE INFORMATION			
Sample Name	dl-3b-1	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	3/28/05 6:13:43 PM
Vial:	1	Acq. Method:	100A
Injection #:	5	Date Processed:	3/28/05 6:33:10 PM
Injection Volume:	20.00 ul	Channel Name:	2487Channel 1
Run Time:	180.00 Minutes	Sample Set Name:

RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1 3.999	7329	0.15	1432	1.04
2 4.318	11521	0.24	1536	1.11
3 12.401	100021	2.06	3844	2.79
4 14.670	4678486	97.62	131155	95.06

ulas

Project Name: Defaults
Reported by User: System

SAMPLE INFORMATION

Sample Name: dl-3c-0
Sample Type: Unknown
Vial: 1
Injection #: 1
Injection Volume: 20.00 ul
Run Time: 180.00 Minutes
Acquired By: System
Date Acquired: 3/28/05 4:15:08 PM
Acq. Method: 100A
Date Processed: 3/28/05 4:48:43 PM
Channel Name: 2487Channel 1
Sample Set Name:

	RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1	10.696	61478	2.15	2616	4.80
2	22.595	1400030	48.96	27431	50.29
3	25.308	1398273	46.89	24501	44.92

ulas

Project Name: Defaults
Reported by User: System

SAMPLE INFORMATION

Sample Name: dl-3c
Sample Type: Unknown
Vial: 1
Injection #: 2
Injection Volume: 20.00 ul
Run Time: 180.00 Minutes
Acquired By: System
Date Acquired: 3/28/05 4:49:31 PM
Acq. Method: 100A
Date Processed: 3/28/05 5:18:18 PM
Channel Name: 2487Channel 1
Sample Set Name:

	RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1	4.025	7784	0.28	1004	1.93
2	4.312	29202	1.04	1982	3.81
3	11.683	38660	1.37	1583	3.04
4	21.905	42601	1.51	967	1.86
5	24.398	2701142	95.81	46456	89.35

SAMPLE INFORMATION			
Sample Name:	dl-k-0	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	3/31/05 12:52:33 PM
Vial:	1	Acq. Method:	100A
Injection #:	2	Date Processed:	3/31/05 2:34:45 PM
Injection Volume:	20.00 ul	Channel Name:	2487Channel 1
Run Time:	180.00 Minutes	Sample Set Name:

RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1 3.803	248349	0.12	19501	0.54
2 3.974	55605	0.03	8106	0.22
3 4.379	103844	0.05	11704	0.32
4 4.647	345391	0.17	18743	0.52
5 4.931	103887	0.05	11942	0.33
6 6.258	118503	0.06	5155	0.14
7 15.314	101073644	49.70	2173078	60.25
8 21.591	101320312	49.82	1358813	37.87

SAMPLE INFORMATION			
Sample Name:	dl-k-3	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	4/4/05 10:39:39 AM
Vial:	1	Acq. Method:	100A
Injection #:	3	Date Processed:	4/4/05 11:09:38 AM
Injection Volume:	20.00 ul	Channel Name:	2487Channel 1
Run Time:	180.00 Minutes	Sample Set Name:

RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1 3.183	39209	0.08	3803	0.48
2 3.226	50710	0.10	4453	0.56
3 3.484	39320	0.08	4762	0.60
4 3.664	254228	0.52	31962	4.00
5 4.391	290002	0.59	13126	1.64
6 5.145	85784	0.17	4493	0.56
7 5.903	34022	0.07	4160	0.52
8 6.058	85731	0.17	6972	0.87
9 14.788	426787	0.87	12059	1.51
10 21.351	47917977	97.35	712716	89.26