Supporting information

Total Synthesis of (±)-Chamobtusin A

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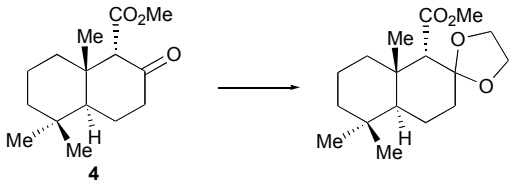
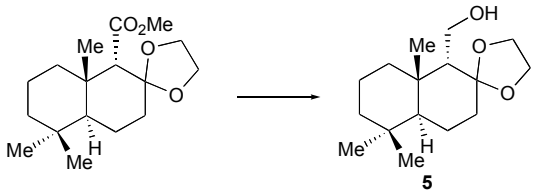
General Methods. Reported melting points are uncorrected. Unless otherwise stated ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ or CD_3OD, on a 400 MHz instrument. Proton chemical shifts are given in δ (ppm) relative to internal $CHCl_3$ (7.26 ppm) or CD_3OH (4.78 ppm). Carbon chemical shifts are given relative to $CDCl_3$ (77.05 ppm) or CD_3OD (49.3 ppm). Analytical TLC was carried out with precoated silica gel $60F_{254}$ plates (Merck). Flash column chromatography was performed on Silica gel 60N (spherical, neutral, 40-50 mm, Kanto Chemical Co., Inc.).



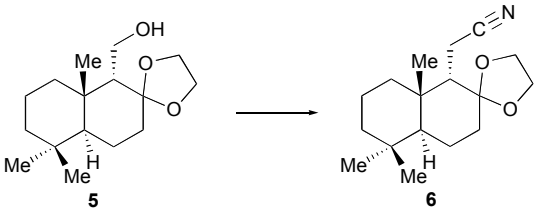
Methyl (1´*S,4a´*S**,8a´*S**)-5´,5´,8a´-Trimethyloctahydro-1´*H*-spiro[[1,3]dioxolane-2,2´-naphthalene]-1´-carboxylate.** Ethylene glycol (1.1 mL, 19.6 mmol) and trimethylsilyl chloride (2.1 mL, 15.7 mmol) were added to a solution of **4** (990 mg, 3.92 mmol)) in dry CH_2Cl_2 (8.0 mL) at room temperature. The mixture was heated at reflux for 23 h. After cooling, the reaction was quenched with saturated aqueous $NaHCO_3$ (8.0 mL) at 0 °C, and the resulting mixture was extracted with Et_2O (3 x 8.0 mL). The combined extracts were washed with brine, then dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel flash column chromatography (hexane/EtOAc 10:1) to give the title compound (1.14 g, 98%) as white crystals. mp 78-79 °C (from hexane); IR (KBr) 1735 cm^{-1}; ^{1}H NMR (400MHz, $CDCl_3$) δ 0.66 (3H, s), 0.82 (3H, s), 0.99-1.02 (1H, m), 1.14 (3H, s), 1.02-1.09 (1H, m), 1.34-1.41 (3H, m), 1.47 (1H, td, *J* = 13.6, 4.0 Hz), 1.57-1.73 (3H, m), 2.13 (1H, dd, *J* = 12.5, 2.38 Hz), 2.25 (1H, td, *J* = 13.8, 5.78 Hz), 2.44 (1H, d, *J* = 1.78

Hz), 3.66 (3H, s), 3.86-3.99 (4H, m); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 18.5 (CH_2), 19.7 (CH_2), 21.4 (CH_3), 21.9 (CH_3), 33.1 (C), 33.4 (CH_2), 33.5 (CH_3), 38.2 (C), 38.5 (CH_2), 41.8 (CH_2), 45.3 (CH), 51.1 (CH), 62.6 (CH_3), 63.9 (CH_2), 64.5 (CH_2), 109.1 (C) 172.6 (C); HRMS (ESI) calcd for $C_{17}H_{29}O_4$ $[M+H]^+$ 297.2066, found 297.2063. Anal. Calcd for $C_{17}H_{28}O_4$: C, 68.89; H, 9.52. Found: C, 68.92; H, 9.54.



5

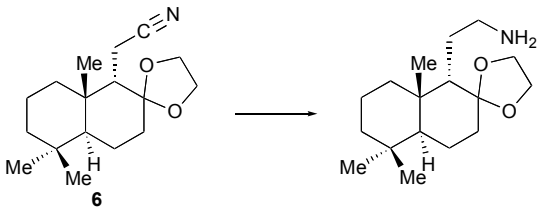
2-((1´*R,4a´*S**,8a´*S**)-5´,5´,8a´-Trimethyloctahydro-1´*H*-spiro[[1,3]dioxolane-2,2´-naphthalene]-1´-yl)methanol (5).** A solution of the above ester (9.20 g, 31.0 mmol) in dry Et_2O (30 mL) was added to a stirred suspension of lithium aluminium hydride (3.53 g, 93.0 mmol) in dry Et_2O (280 mL) at 0 °C under argon, and stirring was continued for 19 h at room temperature. The reaction was quenched with successive addition of water (3.5 mL), 4M aqueous sodium hydroxide (3.5 mL), and water (7.0 mL). The suspension was filtrated through a Celite pad and the filtrate was concentrated under reduced pressure. The residue was purified by silica gel flash column chromatography (hexane/AcOEt 2:1) to give **5** (8.20 g, 99%) as white crystals. mp 77-79 °C (from hexane); IR (KBr) 3330 cm^{-1}; ^{1}H NMR (400MHz, $CDCl_3$) δ 0.81 (3H, s), 0.85 (3H, s), 0.94 (1H, dd, *J* = 12.3, 3.11 Hz), 1.08-1.16 (1H, m), 1.16 (3H, s), 1.22-1.27 (1H, m), 1.37-1.77 (9H, m), 3.29 (1H, dd, *J* = 10.0, 2.41 Hz), 3.76-4.01 (6H, m); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 18.7 (CH_2), 20.4 (CH_2), 22.1 (CH_3), 22.6 (CH_3), 33.1 (CH_2), 33.4 (C), 33.5 (CH_3), 37.3 (CH_2), 38.5 (C), 42.4 (CH_2), 48.2 (CH), 57.2 (CH), 61.2 (CH_2), 63.5 (CH_2), 64.2 (CH_2), 113.1 (C); HRMS (ESI) calcd for $C_{16}H_{29}O_3$ $[M+H]^+$ 269.2117, found 269.2113. Anal. Calcd for $C_{16}H_{28}O_3$: C, 71.60; H, 10.52. Found: C, 71.50; H, 10.49.



5 6

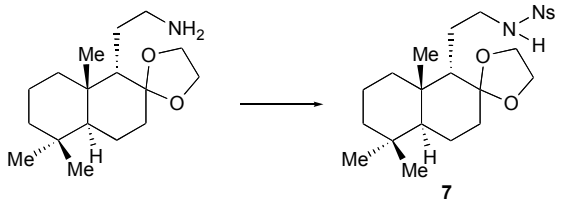
2-((1´*S,4a´*S**,8a´*S**)-5´,5´,8a´-Trimethyloctahydro-1´*H*-spiro[[1,3]dioxolane-2,2´-**

naphthalene]-1´-yl)acetonitrile (6). *p*-Toluenesulfonyl chloride (12.3 g, 61.2 mmol) was added to a solution of **5** (8.20 g, 30.6 mmol) in pyridine (31.0 mL) at 0 °C under argon. After stirring for 2 h at 0 °C, the reaction was quenched with a saturated aqueous solution of copper (II) sulfate penta hydrate (30 mL), and the resulting mixture was extracted with Et_2O (3 x 50 mL). The combined extracts were washed with brine, then dried over $MgSO_4$ and concentrated under reduced pressure to afford a yellow oil, which was used in the next step without further purification. KCN (4.98 g, 76.5 mmol) and 18-crown-6 (5.65 g, 30.6 mmol) were added to a solution of the above residue in MeCN (61.0 mL) at room temperature. The mixture was heated at reflux for 4 h. After cooling, the reaction was diluted with a saturated aqueous solution of $NaHCO_3$ (30 mL) at 0 °C, and the resulting mixture was extracted with Et_2O (3 x 60 mL). The combined extracts were washed with brine, then dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel flash column chromatography (hexane/EtOAc 4:1) to give **6** (6.44 g, 76% from **5**) as a yellow oil. IR (neat) 2242 cm^{-1}; ^1H NMR (400MHz, $CDCl_3$) δ 0.83 (3H, s), 0.89 (3H, s), 0.89-1.18 (1H, m), 1.20 (3H, s), 1.38-1.56 (8H, m), 1.68-1.71 (3H, m), 1.37-1.58 (8H, m), 1.68-1.71 (3H, m), 2.64 (1H, dd, J = 17.3, 7.3 Hz), 2.65 (1H, dd, J = 17.3, 3.69 Hz), 3.87-4.03 (4H, m); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 15.1 (CH_2), 18.5 (CH_2), 19.8 (CH_2), 21.8 (CH_3), 22.3 (CH_3), 33.1 (C), 33.2 (CH_2), 33.5 (CH_3), 37.8 (CH_2), 38.6 (C), 42.2 (CH_2), 46.3 (CH), 52.9 (CH), 63.6 (CH_2), 64.9 (CH_2), 109.9 (C), 121.5 (C); HRMS (ESI) calcd for $C_{17}H_{28}NO_2$ $[M+H]^+$ 278.2120, found 278.2125. Anal. Calcd for $C_{17}H_{27}NO_2$: C, 73.61; H, 9.81; N, 5.05. Found: C, 73.46; H, 9.72; N, 5.16.



2-((1´*S,4a´*S**,8a´*S**)-5´,5´,8a´-Trimethyloctahydro-1´*H*-spiro[[1,3]dioxolane-2,2´-naphthalene]-1´-yl)ethanamine.** A solution of **6** (6.44 g, 23.2 mmol) in dry Et_2O (10 mL) was added to a stirred suspension of lithium aluminium hydride (2.64 g, 69.6 mmol) in dry Et_2O (220 mL) at 0 °C under argon, and stirring was continued for 2 h at 0 °C. The reaction was quenched with successive addition of water (2.5 mL), 4M aqueous sodium hydroxide (2.5 mL), and water (5.0 mL). The suspension was filtrated through a Celite pad and the

filtrate was concentrated under reduced pressure. The residue was purified by silica gel flash column chromatography ($CHCl_3$/MeOH/NH_4OH 100:9:1) to give the title compound (6.26 g, 96%) as a colorless oil. IR (neat) 3372 cm^{-1}; ^1H NMR (400MHz, $CDCl_3$) δ 0.80 (3H, s), 1.02-1.06 (3H, m), 1.13 (3H, s), 1.35-1.38 (4H, m), 1.38-1.62 (9H, m), 2.59-2.64 (2H, m), 3.78-4.11 (4H, m); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 18.5 (CH_2), 20.2 (CH_2), 21.6 (CH_3), 22.8 (CH_3), 32.7 (C), 32.9 (CH_2), 33.3 (CH_2), 33.4 (CH_3), 36.4 (CH_2), 38.8 (C), 42.3 (CH_2), 44.7 (CH_2), 46.3 (CH), 52.9 (CH), 63.3 (CH_2), 64.5 (CH_2). 112.3 (C); HRMS (ESI) calcd for $C_{17}H_{32}NO_2$ $[M+H]^+$ 282.2433, found 282.2435. Anal. Calcd for $C_{17}H_{31}NO_2$: C, 72.55; H, 11.10; N, 4.98. Found: C, 72.33; H, 10.84; N, 5.02.



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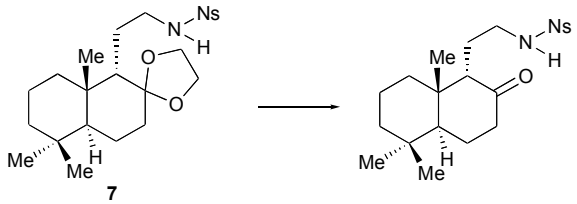
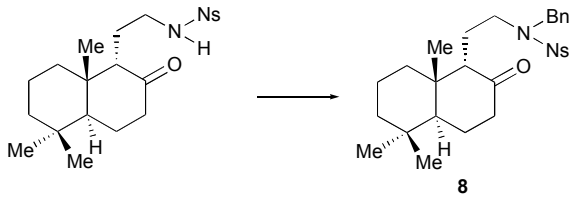
2-Nitro-*N*-(2-((1´*S,4a´*S**,8a´*S**)-5´,5´,8a´-trimethyloctahydro-1´*H*-spiro[[1,3]-dioxolane-2,2´-naphthalene]-1´-yl)ethyl)benzenesulfonamide (7).** Triethylamine (1.66 mL, 11.9 mmol) and *o*-NsCl (791mg, 3.57 mmol) were added to a stirred solution of the above amine in dry CH_2Cl_2 (24 mL) at 0 °C under argon. After 5 min, the reaction was quenched with a saturated aqueous solution of $NaHCO_3$ (30 mL) at 0 °C, and the resulting mixture was extracted with Et_2O (3 x 40 mL). The combined extracts were washed with brine, then dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel flash column chromatography (hexane/EtOAc 2:1) to give **7** (1.11 g, 100%) as an amorphous solid. IR (KBr) 3334, 1541 cm^{-1}; ^1H NMR (400MHz, $CDCl_3$) δ 0.79 (3H, s), 0.83 (3H, s), 0.88-0.90 (1H, m), 0.98-1.09 (2H, m), 1.10 (3H, s), 1.14-1.15 (1H, m), 1.22-1.48 (7H, m), 1.59-1.67 (3H, m), 2.96-3.04 (1H, m), 3.10-3.16 (1H, m), 3.83-3.98 (4H, m), 5.74 (1H, t, *J* = 4.75 Hz), 7.71-7.74 (2H, m), 7.84-7.86 (1H, m), 8.10-8.13 (1H, m); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 18.4 (CH_2), 20.0 (CH_2), 21.5 (CH_3), 22.6 (CH_3), 27.0 (CH_2), 32.90 (C), 32.91 (CH_3), 33.4 (CH_2), 36.0 (CH_2), 38.9 (C), 42.3 (CH_2), 45.5 (CH_2), 46.4 (CH), 53.3 (CH), 63.2 (CH_2), 64.8 (CH_2), 111.8 (C), 125.3 (CH), 131.1 (CH), 132.6 (CH), 133.3 (CH), 133.9 (C), 148.1 (C); HRMS (ESI) calcd for $C_{23}H_{35}N_2O_6S$ $[M+H]^+$ 467.2216, found 467.2200.



7

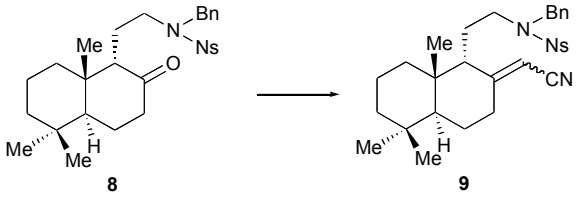
2-Nitro-*N*-(2-((1´*S,4a´*S**,8a´*S**)-5´,5´,8a´-trimethyl-2-oxodecahydronaphtalene-1-yl)ethyl)benzenesulfonamide .** An aqueous 5% HCl solution (0.98 mL, 1.35 mmol) was added to a stirred solution of **7** (126 mg, 0.27 mmol) in dry THF (2.7 mL) at 0 °C under argon. After stirring for 8 h at 0 °C, the reaction was quenched with saturated aqueous $NaHCO_3$ (3.0 mL) at 0 °C, and the resulting mixture was extracted with Et_2O (3 x 10 mL). The combined extracts were washed with brine, then dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel flash column chromatography (hexane/EtOAc 3:1) to give the title compound (101 mg, 89%) as white crystals. mp 135-136 °C; IR (KBr) 3300, 1687, 1541 cm^{-1}; ^1H NMR (400MHz, $CDCl_3$) δ 0.85 (3H, s), 0.90 (3H, s), 0.95 (3H, s), 1.01-1.05 (1H, m), 1.14-1.22 (1H, m), 1.41-1.63 (6H, m), 1.70-1.80 (2H, m), 1.90-2.01 (2H, m), 2.31-2.37 (2H, m), 2.81-2.88 (1H, m), 3.00-3.08 (1H, m), 5.56 (1H, t, *J* = 6.21 Hz), 7.70-7.74 (2H, m), 7.84-7.86 (1H, m), 8.07-8.10 (1H, m); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 18.5 (CH_2), 22.0 (CH_3), 22.1 (CH_3), 23.1 (CH_2), 27.8 (CH_2), 33.3 (C), 33.4 (CH_3), 36.3 (CH_2), 38.4 (CH_2), 39.7 (C), 42.0 (CH_2), 42.2 (CH_2), 44.8 (CH), 61.9 (CH), 125.4 (CH), 131.0 (CH), 132.8 (CH), 133.6 (CH), 133.7 (C), 140.1 (C), 215.2 (C); HRMS (ESI) calcd for $C_{21}H_{31}N_2O_5S$ $[M+H]^+$ 423.1954, found 423.1977. Anal. Calcd for $C_{21}H_{30}N_2O_5S$: C, 59.69; H, 7.16; N, 6.63. Found: C, 59.67 H, 7.04; N, 6.67.



8

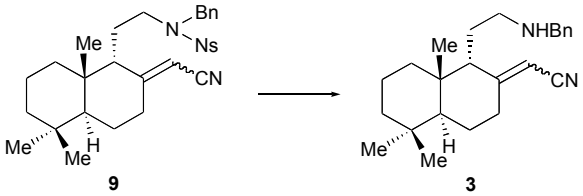
***N*-Benzyl-2-nitro-*N*-(2-((1´*S**,4a´*S**,8a´*S**)-5´,5´,8a´-trimethyl-2-oxodecahydro-naphthalene-1-yl)ethyl)benzenesulfonamide (8).** Benzyl alcohol (72.0 μL, 0.69 mmol) and triphenyl phosphine (157 mg, 0.60 mmol) were added to a stirred solution of the above ketone (195 mg, 0.46 mmol) in dry toluene (4.6 mL) at room temperature under argon. And then, diethyl azodicarboxyrate (40 % in toluene, 0.27 mL, 0.60 mmol) was added to the above

mixture at 0 °C. After stirring for 10 min at room temperature, the mixture was directly purified by silica gel flash column chromatography (hexane/EtOAc 2:1) to give **8** (236 mg, 100%) as white crystals. mp 137-138 °C; IR (KBr) 1702, 1543 cm^{-1}; ^1H NMR (400MHz, $CDCl_3$) δ 0.79 (3H, s), 0.80 (3H, s), 0.90 (3H, s), 1.05-1.30 (3H, m), 1.37-1.51 (7H, m), 1.70-1.85 (2H, m), 2.09-2.14 (2H, m), 2.93-3.06 (2H, m), 4.45 (1H, d, *J* = 14.9 Hz), 4.51 (1H, d, *J* = 14.9 Hz), 7.29-7.32 (5H, m), 7.63-7.70 (3H, m), 7.91-7.94 (1H, m); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 18.5 (CH_2), 21.7 (CH_3), 22.0 (CH_3), 23.1 (CH_2), 26.7 (CH_2), 33.2 (C), 33.3 (CH_3), 36.1 (CH_2), 38.0 (CH_2), 39.8 (C), 42.1 (CH_2), 44.6 (CH), 47.0 (CH_2), 53.1 (CH_2), 62.3 (CH), 124.3 (CH), 128.3 (CH), 128.7 (CH, 2 carbons), 128.8 (CH, 2 carbons), 130.1 (CH), 131.7 (CH), 133.1 (C), 133.5 (CH), 135.9 (C), 148.2 (C), 214.8 (C); HRMS (ESI) calcd for $C_{28}H_{37}N_2O_5S$ $[M+H]^+$ 513.2423, found 513.2437. Anal. Calcd for $C_{28}H_{36}N_2O_5S$: C, 65.60; H, 7.08; N, 5.46. Found: C, 65.63 H, 7.00; N, 5.49.

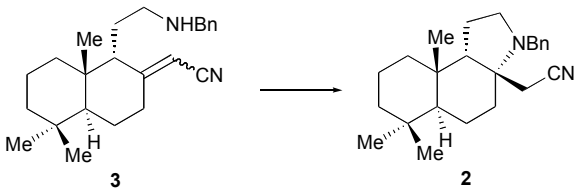


***N*-Benzyl-*N*-(2-((1*R**,4a*S**,8a*S**)-2-(cyanomethylene)-5´,5´,8a´-trimethyl-2-oxodeca-hydronaphthalene-1-yl)ethyl)-2-nitrobenzenesulfonamide (9).** *n*-BuLi (1.6 M solution in hexane, 12.0 mL, 19.2 mmol) was added dropwise to a stirred solution of diethyl cyanomethylphosphonate (3.89 mL, 23.0 mmol) in dry THF (60 mL) at 0 °C under argon, and stirring was continued for 1 h at room temperature. A solution of **8** (3.93 g, 7.67 mmol) in dry THF (20 mL) was added dropwise to the above mixture at 0 °C. After stirring for 24 h at room temperature, the reaction was quenched with water (30 mL) at 0 °C, and the resulting mixture was extracted with Et_2O (3 x 100 mL). The combined extracts were washed with brine, then dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel flash column chromatography (hexane/EtOAc 4:1) to give **9** (as an E/Z mixture in the ratio of 1:1) (3.78 g, 92%) as a yellow oil. IR (neat) 2214, 1545 cm^{-1}; ^1H NMR (400MHz, $CDCl_3$) δ 0.74-0.85 (9H, m), 0.85-0.95 (1H, m), 1.05-1.58 (10H, m), 1.65-1.67 (3H, m), 2.00-2.11 (1H, m), 2.14-2.20 (0.5H, m), 2.65-2.75 (0.5H, m), 2.80-3.14 (2H, m), 4.40 (1H, t, *J* = 14.1 Hz), 4.55 (1H, d, *J* = 14.6 Hz), 4.86 (0.5H, d, *J* = 1.46 Hz), 5.07 (0.5H, d, *J* = 1.84 Hz), 7.27-7.34 (5H, m), 7.64-7.71 (3H, m), 7.92-8.00 (1H, m); ^{13}C NMR

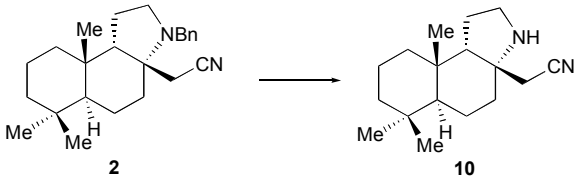
(100.6 MHz, $CDCl_3$) δ 18.7 (CH_2, 2 carbons), 21.2 (CH_3), 21.9 (CH_3, 3 carbons), 23.2 (CH_2), 23.6 (CH_2), 26.2 (CH_2), 27.4 (CH_2), 29.3 (CH_2), 32.2 (CH_2), 33.17 (C, 2 carbons), 33.23 (CH_3), 33.27 (CH_3), 36.08 (CH_2), 36.12 (CH_2), 39.20 (C), 39.29 (C), 42.1 (CH_2, 2 carbons), 45.2 (CH), 45.3 (CH), 47.0 (CH_2), 47.4 (CH_2), 52.5 (CH_2), 53.1 (CH), 53.3 (CH_2), 56.2 (CH), 94.1 (CH), 94.2 (CH), 116.5 (C), 117.0 (C), 124.2 (CH), 124.3 (CH), 128.2 (CH), 128.3 (CH), 128.66 (CH, 2 carbons), 128.79 (CH, 2 carbons), 128.83 (CH, 2 carbons), 128.86 (CH, 2 carbons), 130.7 (CH), 130.9 (CH), 131.7 (CH), 131.8 (CH), 133.0 (C), 133.1 (C), 133.5 (CH), 133.7 (CH), 135.6 (C), 136.0 (C), 148.1 (C), 148.2 (C), 168.9 (C), 169.5 (C); HRMS (ESI) calcd for $C_{30}H_{38}N_3O_4S$ $[M+H]^+$ 536.2583, found 536.2562.



2-((1*R,4a*S**,8a*S**)-1-(2-(Benzylamino)ethyl)-5,5,8,1-trimethyloctahydronaphthalene-2(1*H*)-ylidene)acetonitrile (3).** Thiophenol (0.31 mL, 2.96 mmol) and 5 M aqueous KOH (0.30 mL, 1.48 mmol) were added dropwise to a stirred solution of **9** (396 mg, 0.739 mmol) in MeCN (4.0 mL) at 0 °C under argon. After stirring for 4 h at room temperature, the mixture was directly purified by silica gel flash column chromatography ($CHCl_3$/MeOH 20:1) to give **3** (236 mg, 91%) as a yellow oil. IR (neat) 3313, 2214 cm^{-1}; ^1H NMR (major diastereomer, 400MHz, $CDCl_3$) δ 0.81 (3H, s), 0.88 (3H, s), 0.95 (3H, s), 1.15-1.19 (2H, m), 1.38-1.75 (9H, m), 1.90-2.00 (1H, m), 2.26-2.30 (2H, m), 3.68 (1H, d, J = 13.0 Hz), 3.75 (1H, d, *J* = 12.9 Hz), 5.12 (1H, s), 7.22-7.37 (5H, m); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 18.8 (CH_2), 21.5 (CH_3), 22.0 (CH_3), 23.9 (CH_2), 28.1 (CH_2), 32.5 (CH_2), 33.3 (C), 33.4 (CH_3), 36.4 (CH_2), 39.5 (C), 42.3 (CH_2), 45.4 (CH), 47.9 (CH_2), 53.5 (CH), 54.3 (CH_2), 94.0 (CH), 117.3 (C), 126.9 (CH), 128.2 (CH, 2 carbons), 128.4 (CH, 2 carbons), 140.3 (C), 170.1 (C); HRMS (ESI) calcd for $C_{24}H_{35}N_2$ $[M+H]^+$ 351.2800, found 351.2794.

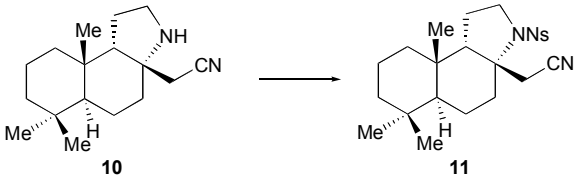


2-((3a*S,5a*S**,9a*S**)-3-Benzyl-6,6,9a-trimethylperhydro-1*H*-benzo[*e*]indol-3a-yl)acetonitrile (2).** *N,N*-Diisopropylethylamine (0.36 mL, 2.18 mmol) was added to a stirred solution of **3** (153 mg, 0.436 mmol) in EtOH (4.4 mL) at room temperature under argon. The mixture was heated at reflux for 24 h. After cooling, the reaction was quenched with saturated aqueous NH_4Cl (5.0 mL) at 0 °C, and the resulting mixture was extracted with Et_2O (3 x 10 mL). The combined extracts were washed with brine, then dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel flash column chromatography (hexane/EtOAc 5:1) to give **2** (123 mg, 80%) as white crystals. mp 154-156 °C; IR (KBr) 2243 cm^{-1}; ^1H NMR (400MHz, $CDCl_3$) δ 0.83 (3H,s), 0.90 (3H, s), 1.10-1.20 (3H, m), 1.23 (3H, s), 1.23-1.29 (2H, m), 1.50-1.70 (3H, m), 1.78-1.81 (2H, m), 2.14 (1H, t, *J* = 10.2 Hz), 2.25-2.31 (1H, m), 2.65 (1H, d, *J* = 17.4 Hz), 2.92-2.97 (1H, m), 2.99 (1H, d, *J* = 17.4 Hz), 3.14 (1H, d, *J* = 13.2 Hz), 3.91 (1H, d, *J* = 13.2 Hz), 7.12-7.73 (5H, m); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 18.2 (CH_2), 19.5 (CH_2), 21.6 (CH_3), 22.7 (CH_3), 23.7 (CH_2), 26.6 (CH_2), 28.3 (CH_2), 32.9 (C), 33.4 (CH_3), 36.3 (C), 37.3 (CH_2), 42.2 (CH_2), 46.5 (CH), 48.9 (CH_2), 52.0 (CH_2), 55.6 (CH), 62.3 (C), 118.6 (C), 126.9 (CH), 128.3 (CH, 2 carbons), 128.4 (CH, 2 carbons), 139.6 (C); HRMS (ESI) calcd for $C_{24}H_{35}N_2$ $[M+H]^+$ 351.2800, found 351.2812. Anal. Calcd for $C_{24}H_{34}N_2$: C, 82.23; H, 9.78; N, 7.99. Found: C, 82.08 H, 9.86; N, 7.98.



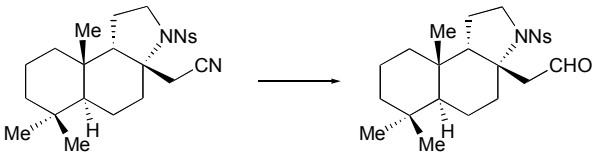
2-((3a*S,5a*S**,9a*S**)-6,6,9a-Trimethylperhydro-1*H*-benzo[*e*]indol-3a-yl)acetonitrile (10).** Celium(IV) ammonium nitrate (64.0 mg, 0.116 mmol) was added to a stirred solution of **2** (13.5 mg, 38.5 μmol) in ($MeCN/CH_2Cl_2/H_2O$ 4:1:1, 0.5 mL) at 0 °C under argon. After stirring for 3.5 h at room temperature, the reaction was quenched with saturated aqueous

$NaHCO_3$ (1.0 mL) at 0 °C, and the resulting mixture was extracted with $CHCl_3$ (3 x 10 mL). The combined extracts were washed with brine, then dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel flash column chromatography ($CHCl_3$/MeOH 20:1) to give **10** (8.5 mg, 85%) as white crystals. mp 87-89 °C; IR (KBr) 3435, 2240 cm^{-1}; ^1H NMR (400MHz, $CDCl_3$) δ 0.81 (3H, s), 0.88 (3H, s), 1.11 (3H, s), 1.18-1.30 (5H, m), 1.38-1.42 (2H, m), 1.74 (1H, t, *J* = 10.1 Hz), 1.89-1.92 (2H, m), 1.98 (1H, br s), 2.69 (1H, d, *J* = 16.7 Hz), 2.70 (1H, d, *J* = 16.7 Hz), 2.97-3.02 (2H, m); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 18.3 (CH_2), 19.9 (CH_2), 21.7 (CH_3), 22.7 (CH_3), 27.8 (CH_2), 29.5 (CH_2), 32.9 (C), 33.4 (CH_3), 34.9 (CH_2), 36.0 (C) 38.2 (CH_2), 42.2 (CH, 2 carbons), 46.5 (CH), 57.0 (CH), 60.4 (C), 118.5 (C); HRMS (ESI) calcd for $C_{17}H_{29}N_2$ $[M+H]^+$ 261.2331, found 261.2333. Anal. Calcd for $C_{17}H_{28}N_2$: C, 78.41; H, 10.84; N, 10.76. Found: C, 78.23 H, 10.63; N, 10.63.

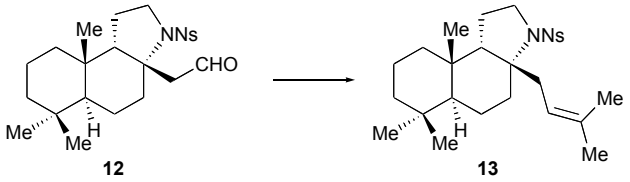


2-((3a*S,5a*S**,9a*S**)-6,6,9a-Trimethyl-3-(2-nitrobenzenesulfonyl)perhydro-1*H*-benzo[*e*]indol-3a-yl)acetonitrile (11).** Triethylamine (0.28 mL, 2.00 mmol) and *o*-NsCl (221 mg, 0.988 mmol) were added to a stirred solution of **10** (104 mg, 0.399 mmol) in CH_2Cl_2 (4.0 mL) at 0 °C under argon. After stirring for 8 h at room temperature, the reaction was quenched with saturated aqueous $NaHCO_3$ (4.0 mL) at 0 °C, and the resulting mixture was extracted with Et_2O (3 x 15 mL). The combined extracts were washed with brine, then dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel flash column chromatography (hexane/EtOAc 2:1) to give **11** (147 mg, 83%) as white crystals. mp 221-224 °C; IR (KBr) 2246, 1545 cm^{-1}; ^1H NMR (400MHz, $CDCl_3$) δ 0.80 (3H, s), 0.88 (3H, s), 1.11-1.23 (3H, m), 1.21 (3H, s), 1.30-1.33 (2H, m), 1.41-1.47 (2H, m), 1.56-1.63 (2H, m), 1.94-1.98 (2H, m), 2.01-2.15 (1H, m), 2.26 (1H, dd, *J* = 12.98, 6.35 Hz), 2.37 (1H, d, *J* = 13.9 Hz), 3.05 (1H, d, *J* = 17.8 Hz), 3.13 (1H, td, *J* = 10.7, 6.89 Hz), 3.74 (1H, d, *J* = 17.9 Hz), 3.79 (1H, t, *J* = 9.27 Hz), 7.56-7.59 (1H, m), 7.67-7.72 (2H, m), 8.11-8.13 (1H, m); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 18.2 (CH_2), 20.0 (CH_2), 21.6 (CH_3), 22.4 (CH_3), 25.2 (CH_2), 27.4 (CH_2), 32.9 (C), 33.3 (CH_3), 36.4 (CH_2), 38.1 (CH_2), 41.9 (CH_2), 46.8

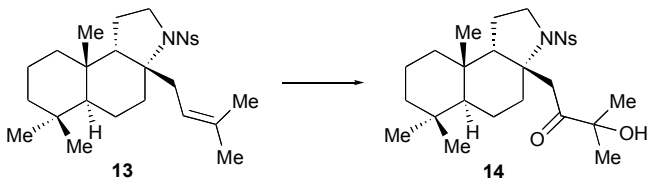
(CH), 47.6 (CH_2), 57.2 (CH), 69.1 (C), 118.3 (C), 124.0 (CH), 130.3 (CH), 131.8 (CH), 133.0 (C), 133.8 (CH), 148.6 (C); HRMS (ESI) calcd for $C_{23}H_{33}N_3O_4S$ $[M+H]^+$ 446.2114, found 446.2118. Anal. Calcd for $C_{23}H_{32}N_3O_4S$: C, 62.00; H, 7.01; N, 9.42. Found: C, 61.93; H, 7.04; N, 9.45.



2-((3a*S,5a*S**,9a*S**)-6,6,9a-Trimethyl-3-(2-nitrobenzenesulfonyl)perhydro-1*H*-benzo[*e*]indol-3a-yl)acetoaldehyde (12).** DIBAL-H (1.01 M in toluene, 0.65 mL, 0.66 mmol) was added dropwise to a stirred solution of **11** (147 mg, 0.33 mmol) in CH_2Cl_2 (3.3 mL) at –78 °C under argon. After stirring for 30 min at –78 °C, 1M HCl (1.0 mL) was added to the mixture. After warming to room temperature very slowly, the reaction was quenched with saturated aqueous $NaHCO_3$ (4.0 mL) at 0 °C, and the resulting mixture was extracted with Et_2O (3 x 15 mL). The combined extracts were washed with brine, then dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel flash column chromatography (hexane/EtOAc 2:1) to give **12** (125 mg, 85%) as white crystals. mp 201-203 °C; IR (KBr) 1720, 1543 cm^{-1}; ^1H NMR (400MHz, $CDCl_3$) δ 0.77 (3H, s), 0.86 (3H, s), 1.08 (3H, s), 1.13-1.28 (5H, m), 1.38-1.45 (2H, m), 1.52-1.68 (2H, m), 1.66 (1H, td, *J* = 13.1, 4.22 Hz), 1.90 (1H, dd, *J* = 12.1, 6.06), 1.94-2.07 (1H, m), 2.16 (1H, dd, *J* = 13.1, 6.14), 2.21-2.25 (1H, br d, *J* = 13.7 Hz), 3.06 (1H, dd, *J* = 18.2, 3.13 Hz), 3.27 (1H, td, *J* = 10.11, 6.6 Hz), 3.54 (1H, d, *J* = 18.2 Hz), 3.78 (1H, d, *J* = 9.08 Hz), 9.74 (1H, d, *J* = 2.96 Hz); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 18.3 (CH_2), 20.0 (CH_2), 21.7 (CH_3), 23.1 (CH_3), 25.3 (CH_2), 32.9 (C), 33.3 (CH_3), 36.9 (C), 37.7 (CH_2), 38.2 (CH_2), 42.0 (CH_2), 46.9 (CH), 48.3 (CH_2), 48.8 (CH_2), 56.5 (CH), 68.8 (C), 123.9 (CH), 130.1 (CH), 131.5 (CH), 133.6 (CH), 134.1 (CH), 148.3 (C), 201.8 (CH); HRMS (ESI) calcd for $C_{23}H_{33}N_2O_5S$ $[M+H]^+$ 449.2110, found 449.2102. Anal. Calcd for $C_{23}H_{32}N_2O_5S$: C, 61.58; H, 7.19; N, 6.24. Found: C, 61.43; H, 7.23; N, 6.32.

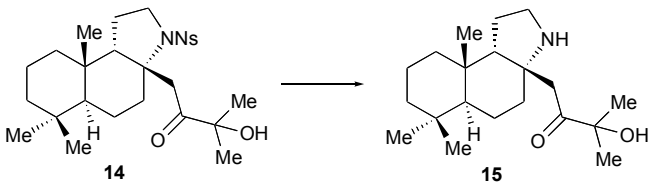


(3a*S,5a*S**,9a*S**)-6,6,9a-Trimethyl-3a-(3-methylbut-2-enyl)-3-(2-nitrobenzenesulfonyl)-perhydro-1*H*-benzo[*e*]indole) (13).** *n*-BuLi (1.6 M solution in hexane, 33.0 μL, 53.0 μmol) was added dropwise to a stirred suspension of isopropyltriphenylphosphonium iodide (24.0 mg, 54.4 μmol) in dry THF (300 μL) at 0 °C under argon, and stirring was continued for 30 min at the same temperature. A solution of **12** (6.10 mg, 13.6 μmol) in dry THF (300 μL) was added dropwise to the above mixture at 0 °C. After stirring for 15 h at room temperature, the reaction was quenched with saturated aqueous NH_4Cl (1.0 mL) at 0 °C, and the resulting mixture was extracted with Et_2O (3 x 10 mL). The combined extracts were washed with brine, then dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel flash column chromatography (hexane/EtOAc 4;1) to give **13** (4.50 mg, 70%) as yellow crystals. mp 141-143 °C; IR (KBr) 1543 cm^{-1}; ^1H NMR (400MHz, $CDCl_3$) δ 0.78 (3H, s), 0.87 (3H, s), 1.00 (3H, s), 1.10-1.41 (7H, m), 1.48 (6H, s), 1.48-1.70 (2H, m), 1.80-1.95 (4H, m), 2.32 (1H, dt, *J* = 13.7, 3.06 Hz), 2.65-2.85 (2H, m), 3.23-3.28 (1H, m), 3.81-3.85 (1H, m), 4.79-4.81 (1H, br s), 7.48-7.50 (1H, m), 7.50-7.61 (2H, m), 7.98-8.10 (1H, m); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 18.4 (CH_2), 18.4 (CH_3), 20.4 (CH_2), 21.8 (CH_3), 22.1 (CH_3), 25.1 (CH_2), 25.8 (CH_3), 32.9 (CH_3), 33.4 (C), 34.5 (CH_2), 36.6 (C), 38.4 (CH_2), 38.5 (CH_2), 42.2 (CH_2), 47.1 (CH), 48.5 (CH_2), 55.0 (CH), 71.3 (C), 120.0 (CH), 123.4 (CH), 130.7 (CH), 131.0 (CH), 132.8 (CH), 132.8 (CH), 135.1 (C), 148.4 (C); HRMS (ESI) calcd for $C_{26}H_{39}N_2O_4S$ $[M+H]^+$ 475.2631, found 475.2652. Anal. Calcd for $C_{26}H_{38}N_2O_4S$: C, 65.79; H, 8.07; N, 5.90. Found: C, 65.69 H, 8.03; N, 5.95.



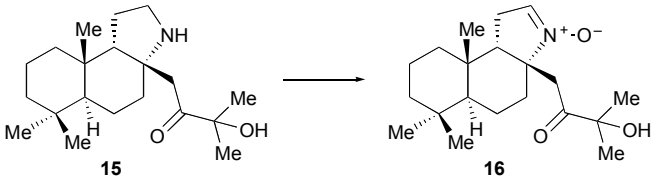
3-Hydroxy-3-methyl-1-((3aS*,5aS*,9aS*,9bS*)-6,6,9a-trimethyl-3-(2-nitrobenzene-sulfonyl)perhydro-1*H*-benzo[*e*]indol-3a-yl)butan-2-one (14). *N*-Methylmorpholine *N*-oxide (78.0 mg, 0.632 mmol) and osmium(IV) tetroxide (0.20 mL, 31.6 μmol) was added to a

stirred solution of **13** (150 mg, 0.316 mmol) in (MeCN/H_2O 4:1, 3.2 mL) at room temperature under argon. After stirring for 17 h at room temperature, the reaction was quenched with saturated aqueous $Na_2S_2O_3$ (4.0 mL) at 0 °C, and the resulting mixture was extracted with Et_2O (3 x 10 mL). The combined extracts were washed with brine, then dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was roughly purified by silica gel frash column chromatography (hexane/EtOAc 4:1) to afford a yellow oil, which was used in the next step without further purification. *N*-Methylmorpholine *N*-oxide (57.0 mg, 0.474 mmol) and MS4A were added to a stirred solution of the above residue in MeCN (2.7 mL) at room temperature under argon. After stirring for 15 min, a solution of TPAP (5.70 mg, 15.8 μmol) in MeCN (0.5 mL) was added dropwise to the above mixture. After stirring for 1 h, the solvent was removed under reduced pressure. The residue was diluted with $CHCl_3$ and through a Celite pad and the filtrate was concentrated under reduced pressure. The residue was purified by silica gel flash column chromatography (hexane/AcOEt 2:1) to give **14** (115 mg, 72% from **13**) as a yellow oil. IR (neat) 3493, 1713, 1545cm^{-1}; ^{1}H NMR (400MHz, $CDCl_3$) δ 0.77 (3H, s), 0.85 (3H, s), 1.02 (3H, s), 1.13-1.28 (5H, m), 1.34 (6H, s), 1.34-1.41 (2H, m), 1.55-1.77 (2H, m), 1.78 (1H, td, *J* = 13.6, 4.1 Hz), 1.87-2.03 (2H, m), 2.17-2.20 (1H, m), 2.67 (1H, dd, *J* = 12.9, 6.35 Hz), 3.40-3.47 (3H, m), 3.73-3.81 (1H, m) 7.51-7.53 (1H, m), 7.61-7.64 (2H, m), 7.85-7.87 (1H, m); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 18.3 (CH_2), 20.1 (CH_2), 21.7 (CH_2), 23.4 (CH_2), 25.7 (CH_2), 27.1 (CH_3, 2 carbons), 32.9 (C), 33.3 (CH_3), 36.5 (C), 38.2 (CH_2), 38.7 (CH_2), 41.3 (CH_2), 42.0 (CH_2), 47.1 (CH), 48.7 (CH_2), 54.5 (CH), 68.2 (C), 123.9 (CH), 130.1 (CH), 131.4 (CH), 133.2 (CH), 134.7 (C), 148.3 (C), 213.1 (C); HRMS (ESI) calcd for $C_{26}H_{39}N_2O_6S$ $[M+H]^+$ 507.2529, found 507.2503.

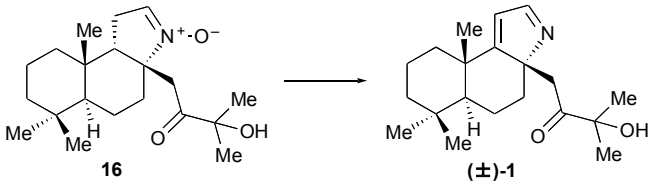


3-Hydroxy-3-methyl-1-((3aS*,5aS*,9aS*,9bS*)-6,6,9a-trimethylperhydro-1*H*-benzo[*e*]indol-3a-yl)butan-2-one (15). Thiophenol (56.0 μL, 0.109 mmol) and 5 M aqueous KOH (87.0 μL, 0.436 mmol) were added dropwise to a stirred solution of **14** (55.2 mg, 0.109 mmol) in MeCN (1.1 mL) at 0 °C under argon. After stirring for 17 h at room temperature, the mixture was directly purified by silica gel flash column chromatography

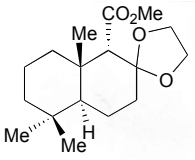
($CHCl_3$/MeOH/NH_4OH 100:9:1) to give **15** (25.6 mg, 73%) as a yellow oil. IR (neat) 3314, 1702 cm^{-1}; ^1H NMR (400MHz, $CDCl_3$) δ 0.81 (3H, s), 0.88 (3H, s), 1.12 (3H, s), 1.14-1.18 (2H, m), 1.23 (3H, s), 1.26 (3H, s), 1.28-1.42 (6H, m), 1.50-1.60 (1H, m), 1.60-1.76 (1H, m), 1.76-1.90 (3H, m), 1.98-2.05 (1H, m), 2.71 (1H, d, *J* = 11.5 Hz), 2.76-2.83 (1H, m), 3.01-3.06 (1H, m) 3.22 (1H, d, *J* = 11.5 Hz); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 18.4 (CH_2), 20.0 (CH_2), 21.8 (CH_3), 23.0 (CH_3), 26.5 (CH_3), 26.8 (CH_3), 27.8 (CH_2), 33.0 (C), 33.5 (CH_3), 36.1 (C), 37.0 (CH_2), 38.7 (CH_2), 42.2 (CH_2), 43.1 (CH_2), 46.6 (CH_2), 47.4 (CH), 59.2 (CH), 62.5 (C), 76.2 (C), 216.6 (C); HRMS (ESI) calcd for $C_{20}H_{36}NO_2[M+H]^+$ 322.2746, found 322.2726.

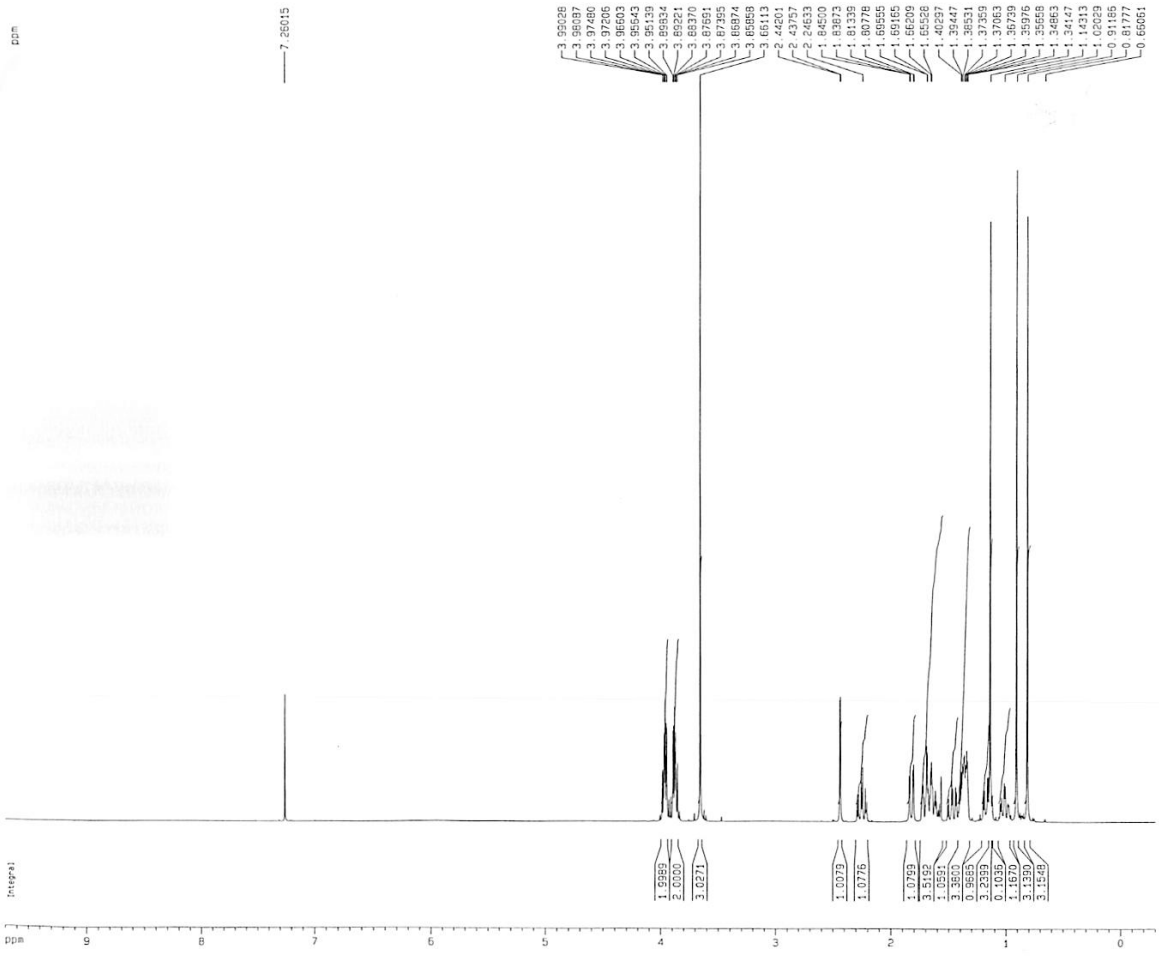


(3aS*,5aS*,9aS*,9bS*)-3a-(3-Hydroxy-3-methyl-2-oxobutyl)-6,6,9a-trimethyl-3a,4,5,5a,6,7,8,9,9a,9b-decahydro-1*H*-benzo[*e*]indole 3-oxide (16). $Na_2WO_4 \cdot 2H_2O$ (14.4 mg, 43.6 μmol) was added to a stirred solution of **15** (28.0 mg, 87.1 μmol) in MeOH (870 μL) at room temperature under argon. After cooling at 0 °C, 30% H_2O_2 (30.0 μL, 0.261 mmol) was added dropwise to the mixture. The resulting mixture was gradually warmed to room temperature over 30 min. After stirring for an additional 1 h at room temperature, the reaction was extracted with $CHCl_3$ (3 x 10 mL). The combined extracts were washed with brine, then dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was roughly purified by silica gel frash column chromatography ($CHCl_3$/MeOH 40:1) to afford **16** (25.2 mg, 86%) as a yellow oil. IR (neat) 3348, 1714 cm^{-1}; ^1H NMR (400MHz, $CDCl_3$) δ 0.84 (3H, s), 0.93 (3H, s), 1.13 (3H, s), 1.13-1.25 (2H, m), 1.27 (3H, s), 1.27-1.35 (2H, m), 1.37 (3H, s), 1.43-1.55 (2H, m), 1.63-1.70 (4H, m), 2.50-2.60 (4H, m), 3.07 (1H, d, *J* = 14.4 Hz), 3.24 (1H, d, *J* = 14.5 Hz), 5.04 (1H, s), 6.81 (1H, t, *J* = 2.34 Hz); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 18.0 (CH_2), 18.2 (CH_2), 21.9 (CH_3), 23.0 (CH_3), 26.3 (CH_3), 26.5 (CH_3), 28.7 (CH_2), 32.9 (C), 33.4 (CH_3), 35.8 (C), 35.9 (CH_2), 36.9 (CH_2), 38.7 (CH_2), 42.0 (CH_2), 48.4 (CH), 53..6 (CH), 53.6 (C), 78.5 (C), 133.4 (CH), 214.2 (C); HRMS (ESI) calcd for $C_{20}H_{34}NO_3[M+H]^+$ 336.2539, found 336.2545.



(±)-Chamobtusin A (1). Benzoyl chloride (1.71 μL, 14.7 μmol) was added to a stirred, refluxing solution of **16** (4.50 mg, 13.4 μmol) in pyridine (600 μL) under argon. After stirring for 1h at reflux, the solvent was removed under reduced pressure. The residue was purified by silica gel flash column chromatography ($CHCl_3$/MeOH 20:1) to give (±)-chamobtusin A (**1**) (2.30 mg, 54%) as a yellow solid. IR (neat) 3334, 2929, 1710, 1604, 1518, 1464, 1374, 1236 cm^{-1}; ^1H NMR (400MHz, $CDOD_3$) δ 0.61-0.70 (2H, m), 0.83 (3H, s), 0.92 (3H, s), 1.10 (3H, s), 1.15 (3H, s), 1.16 (3H, s), 1.39-1.79 (9H, m), 2.54-2.58 (1H, m), 3.32 (1H, d, *J* = 17.8 Hz), 3.47 (1H, d, *J* = 17.8 Hz), 6.03 (1H, s), 7.90 (1H, s); ^{13}C NMR (100.6 MHz, $CDOD_3$) δ 17.6 (CH_3), 19.8 (CH_2), 20.6 (CH_2), 22.4 (CH_3), 27.1 (CH_3, 2 carbons), 34.5 (CH_3), 35.2 (C), 39.1 (CH_2), 41.8 (C), 42.4 (CH_2), 42.8 (CH_2), 43.5 (CH_2), 59.9 (CH), 78.2 (C), 81.1 (C), 118.4 (CH), 166.0 (CH), 184.8 (C), 213.9 (C); HRMS (ESI) calcd for $C_{20}H_{32}NO_2[M+H]^+$ 318.2433, found 318.2447.





Current Data Parameters
NAME suzuki
EXPNO 44
PROCNO 1

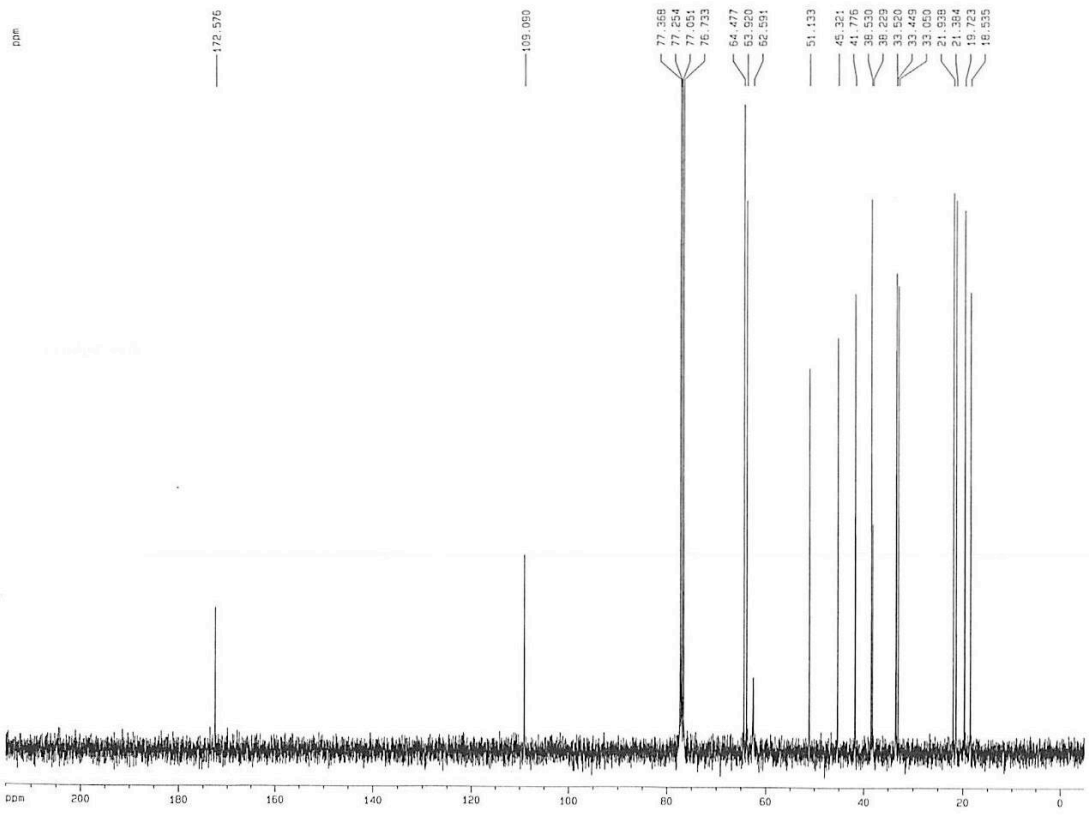
F2 - Acquisition Parameters
Date_ 20091204
Time 23.29
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 181
DW 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.0300065 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 25.00 cm
F1P 9.700 ppm
F1 3880.29 Hz
F2P -0.300 ppm
F2 -120.01 Hz
PPMCM 0.31250 ppm/cm
HZCM 125.00938 Hz/cm

DPX400 carbon



Current Data Parameters
NAME suzuki
EXPNO 45
PROCNO 1

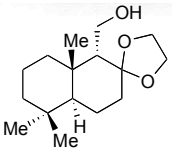
F2 - Acquisition Parameters
Date_ 20091204
Time 23.41
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 372
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 4096
DW 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

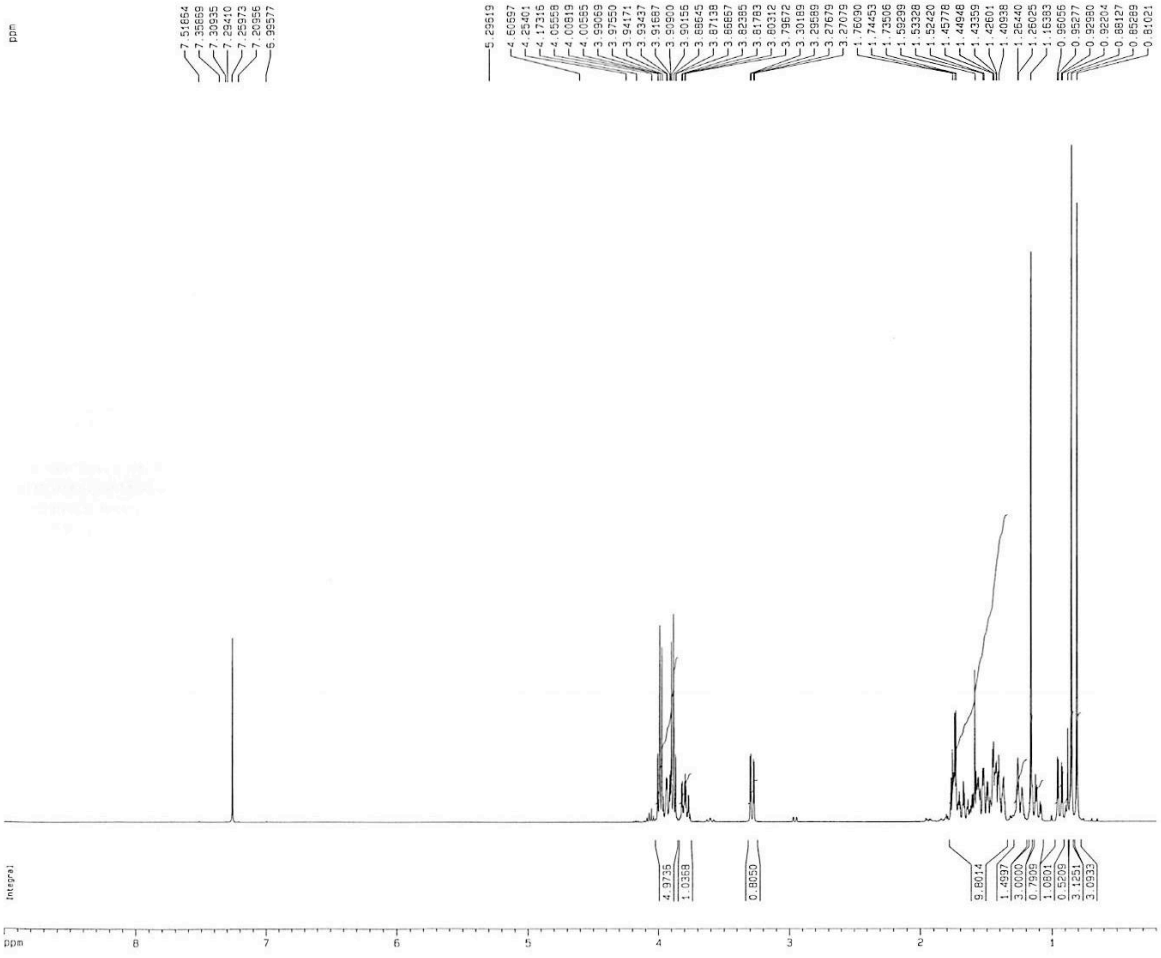
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

F2 - Processing parameters
SI 32768
SF 100.5876190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 30.00 cm
CY 18.00 cm
F1P 215.000 ppm
F1 21626.34 Hz
F2P -5.000 ppm
F2 -502.94 Hz
PPMCM 7.33333 ppm/cm
HZCM 737.64252 Hz/cm





Current Data Parameters
NAME suzuki
EXPNO 95
PROCNO 1

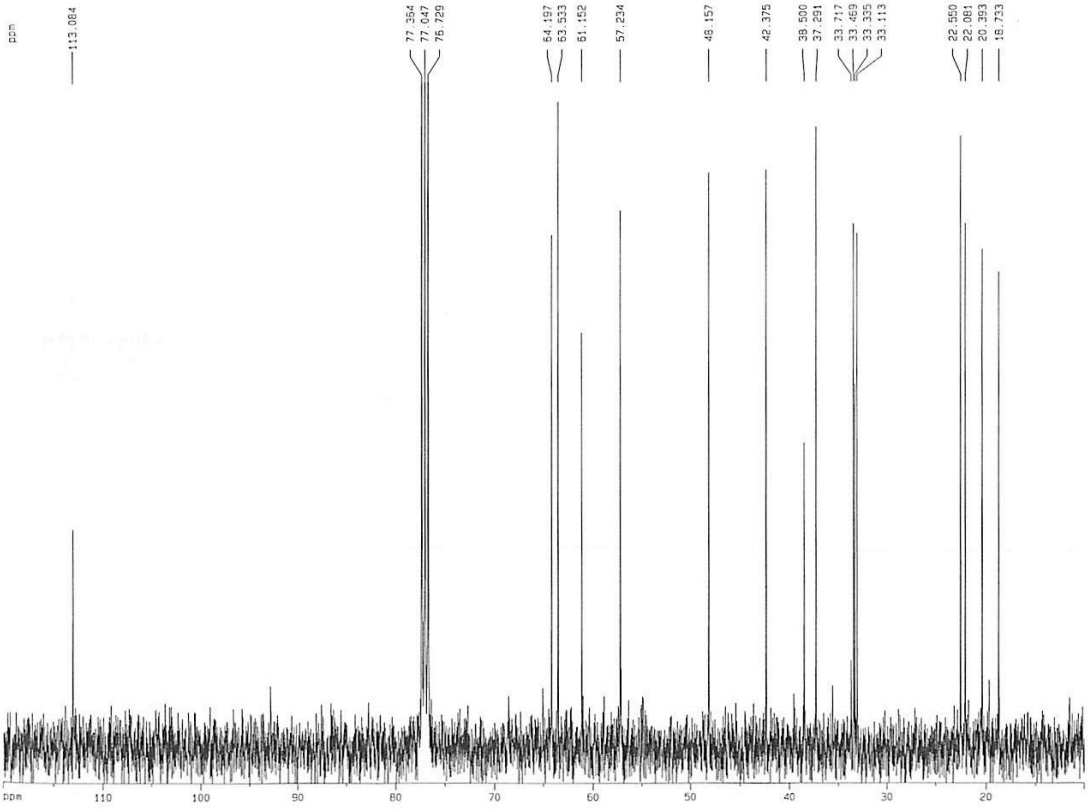
F2 - Acquisition Parameters
Date_ 20100109
Time 3.24
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 228.1
DW 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.0300067 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 19.00 cm
F1P 9.000 ppm
F1 3600.27 Hz
F2P 0.200 ppm
F2 80.01 Hz
PPMCM 0.27500 ppm/cm
HZCM 110.00825 Hz/cm

DPX400 carbon



Current Data Parameters
NAME suzuki
EXPNO 67
PROCNO 1

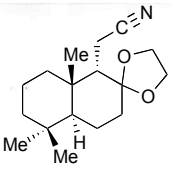
F2 - Acquisition Parameters
Date_ 20091219
Time 19.04
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 226
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 8192
DW 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

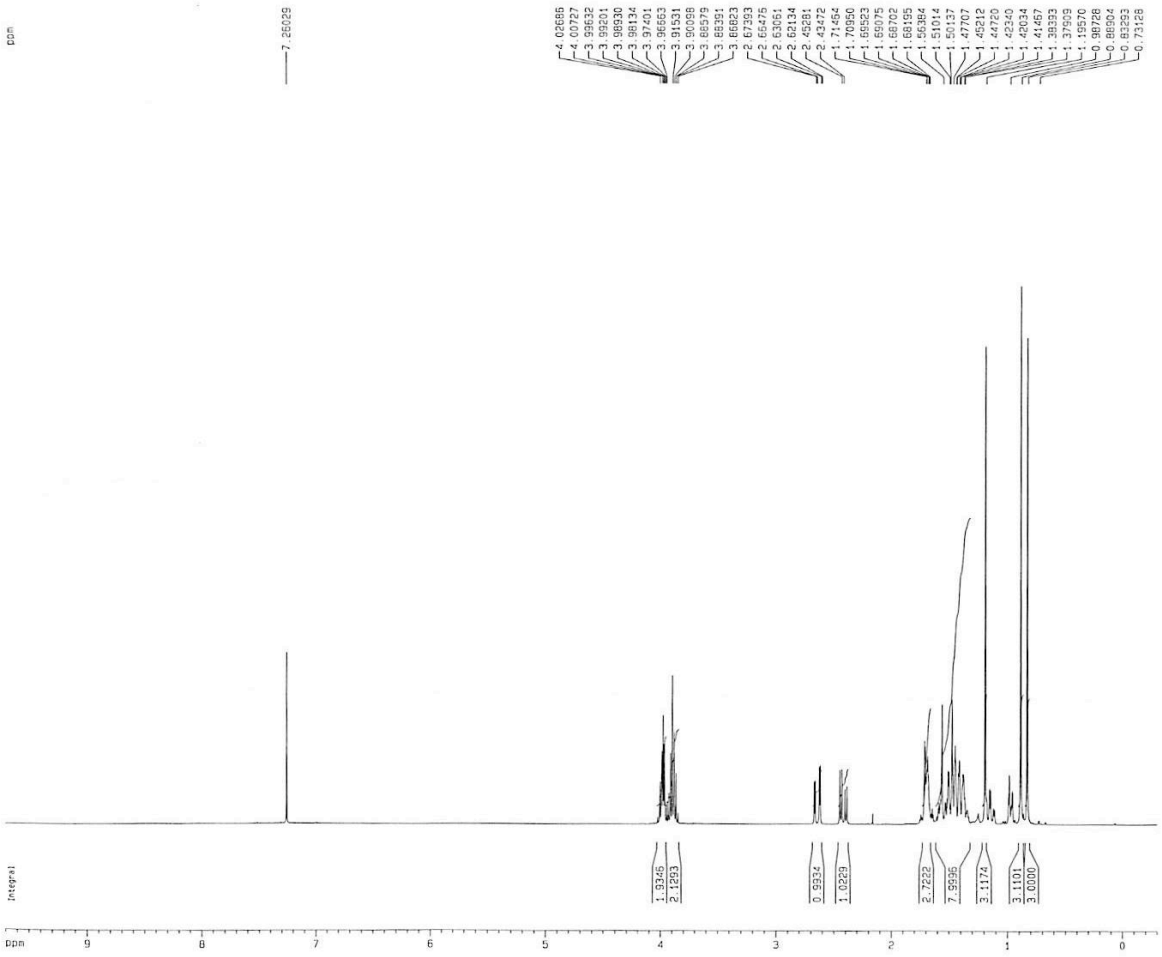
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

F2 - Processing parameters
SI 32768
SF 100.5876198 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

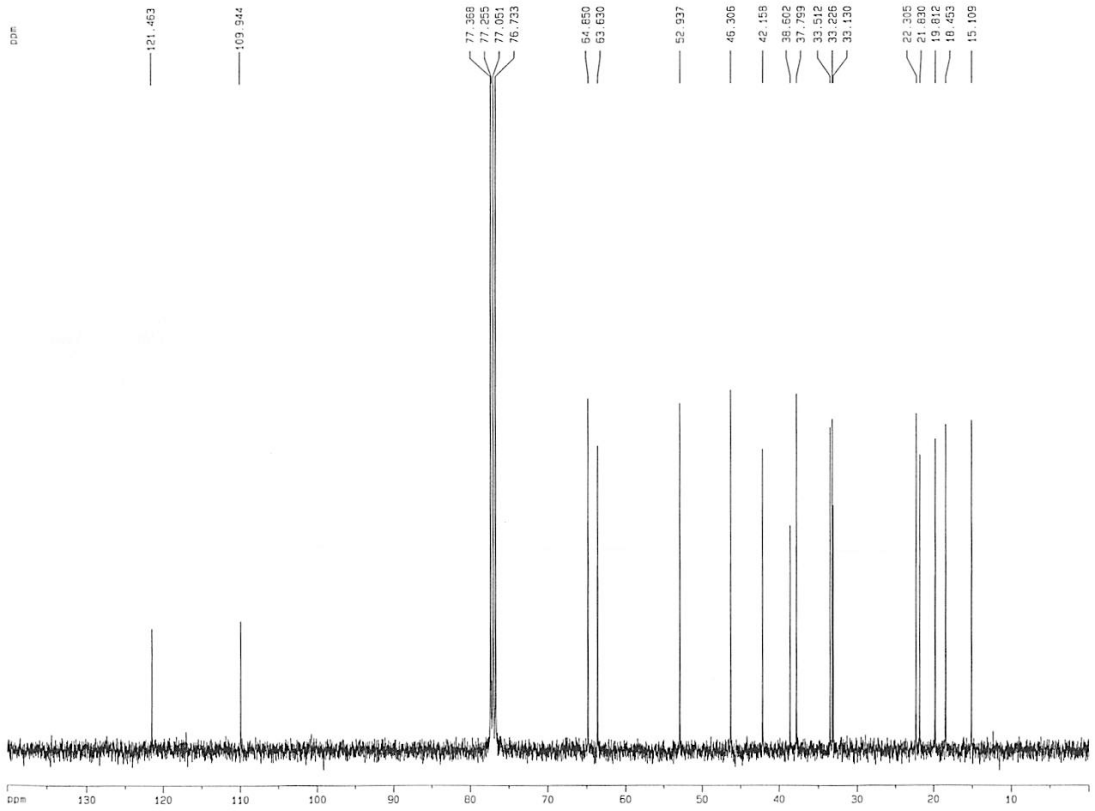
1D NMR plot parameters
CX 30.00 cm
CY 18.00 cm
F1P 120.000 ppm
F1 12070.51 Hz
F2P 10.000 ppm
F2 1005.68 Hz
PPMCM 3.66667 ppm/cm
HZCM 368.82126 Hz/cm



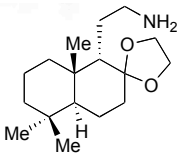


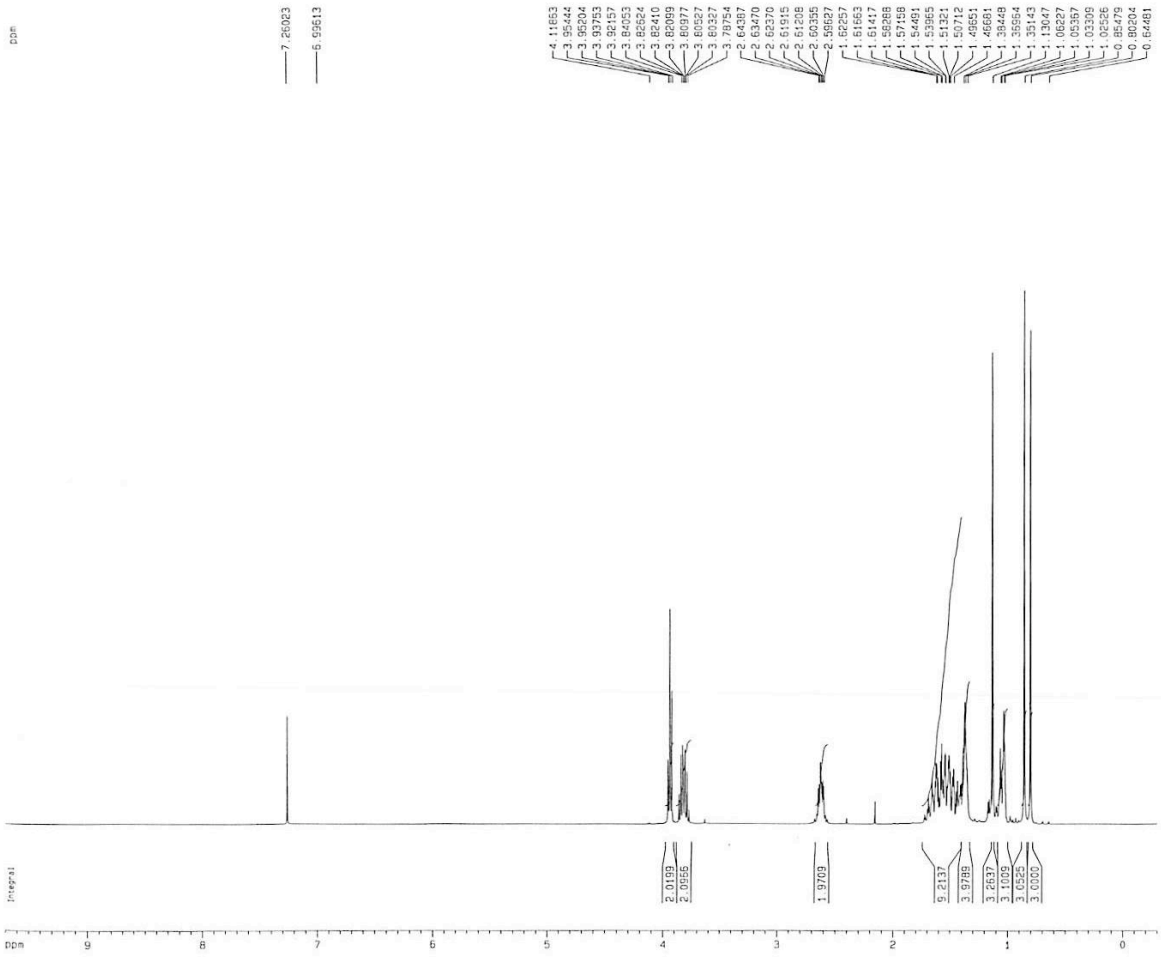

```
Current Data Parameters
NAME                 suzuki
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_               20091114
Time                   20.23
INSTRUM                spect
PROBHD   5 mm QNP 1H/13
PULPROG                zg30
TD                    65536
SOLVENT                CDCl3
NS                        32
DS                         2
SWH                8278.146 Hz
FIDRES            0.126314 Hz
AQ                3.9584243 sec
RG                       256
DW                  60.400 usec
DE                   6.00 usec
TE                   300.2 K
D1                1.00000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                    11.50 usec
PL1                   -4.00 dB
SFO1            400.0324703 MHz

F2 - Processing parameters
SI                        32768
SF                400.0300065 MHz
WDW                       EM
SSB                        0
LB                     0.30 Hz
GB                         0
PC                      1.00

1D NMR plot parameters
CX                     32.00 cm
CY                     15.00 cm
F1P                    9.700 ppm
F1                   3880.29 Hz
F2P                   -0.300 ppm
F2                   -120.01 Hz
PPMCM                0.31250 ppm/cm
HZCM               125.00938 Hz/cm
```

DPX400 carbon




```
Current Data Parameters
NAME                 suzuki
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_               20091114
Time                   20.43
INSTRUM                spect
PROBHD   5 mm QNP 1H/13
PULPROG              zgpg30
TD                    65536
SOLVENT                CDCl3
NS                       440
DS                         4
SWH               26178.010 Hz
FIDRES            0.399445 Hz
AQ                1.2517875 sec
RG                      8192
DW                  19.100 usec
DE                   6.00 usec
TE                   300.2 K
D1                2.00000000 sec
d11               0.03000000 sec
DELTA             1.89999998 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                    10.00 usec
PL1                   -3.50 dB
SFO1            100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                   -4.00 dB
PL12                  17.00 dB
PL13                  17.00 dB
SFO2            400.0316001 MHz

F2 - Processing parameters
SI                        32768
SF               100.5876198 MHz
WDW                       EM
SSB                        0
LB                     1.00 Hz
GB                         0
PC                      1.40

1D NMR plot parameters
CX                     30.00 cm
CY                     10.00 cm
F1P                  140.000 ppm
F1                 14082.27 Hz
F2P                    0.000 ppm
F2                     0.00 Hz
PPMCM              4.66667 ppm/cm
HZCM             469.40887 Hz/cm
```





Current Data Parameters
NAME suzuki
EXPNO 9
PROCNO 1

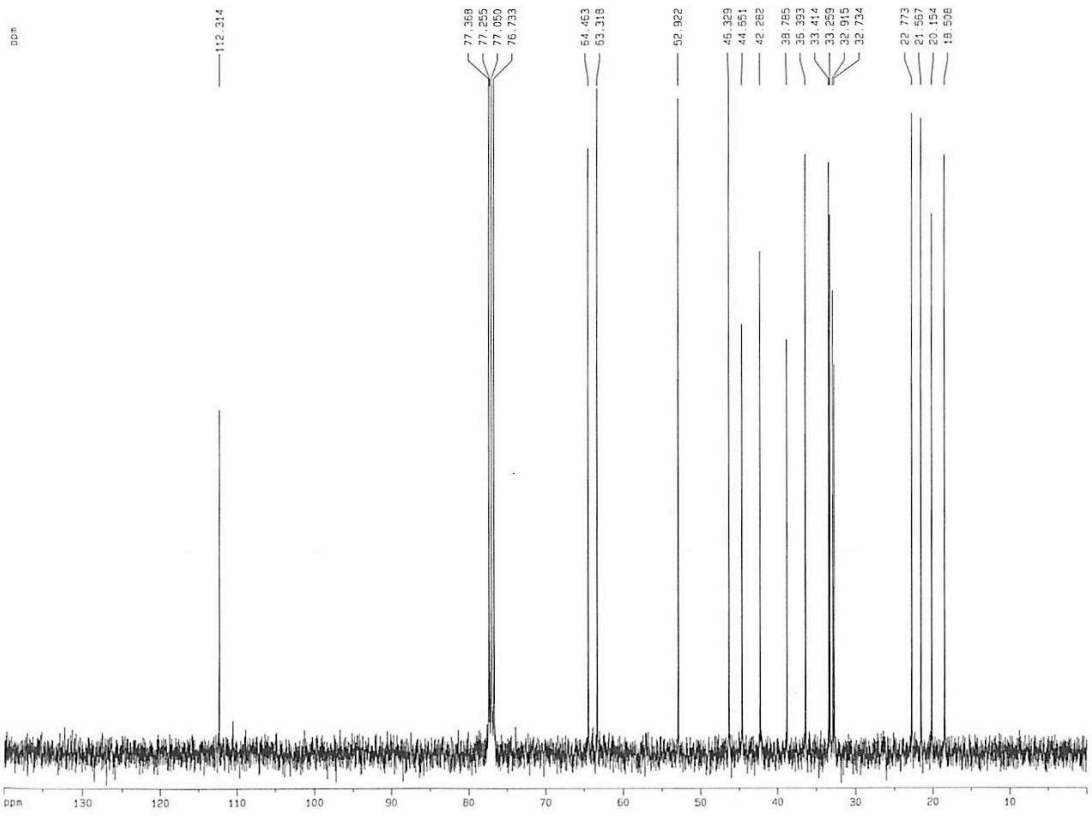
F2 - Acquisition Parameters
Date_ 20091114
Time 22.28
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 143.7
DW 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.0300065 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 15.00 cm
F1P 9.700 ppm
F1 3880.29 Hz
F2P -0.300 ppm
F2 -120.01 Hz
PPMCM 0.31250 ppm/cm
HZCM 125.00938 Hz/cm

DPX400 carbon



Current Data Parameters
NAME suzuki
EXPNO 10
PROCNO 1

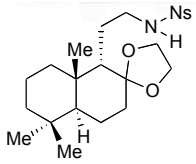
F2 - Acquisition Parameters
Date_ 20091114
Time 22.39
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 192
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 2560.3
DW 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

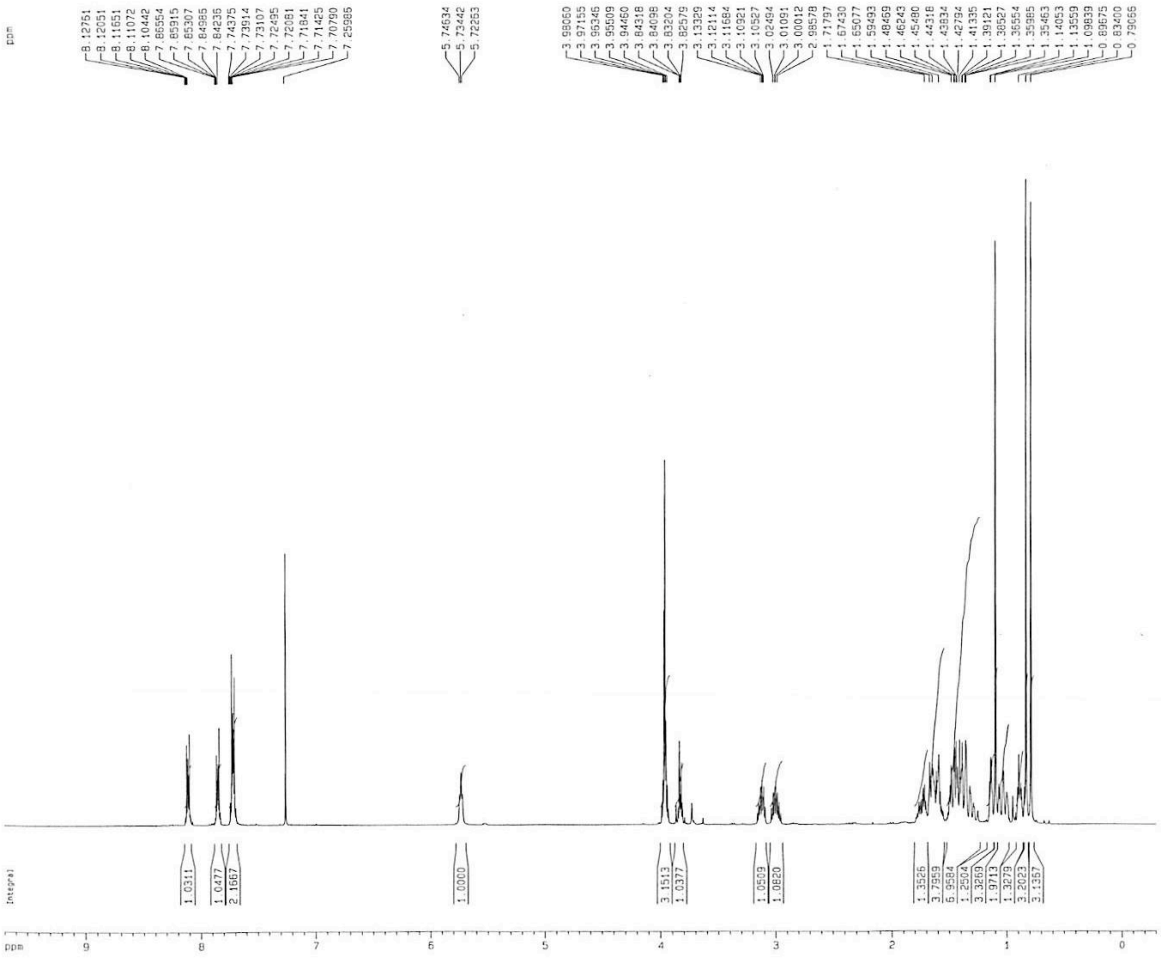
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

F2 - Processing parameters
SI 32768
SF 100.5876206 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 30.00 cm
CY 19.00 cm
F1P 140.000 ppm
F1 14082.27 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 4.66667 ppm/cm
HZCM 469.40891 Hz/cm





Current Data Parameters
NAME suzuki
EXPNO 12
PROCNO 1

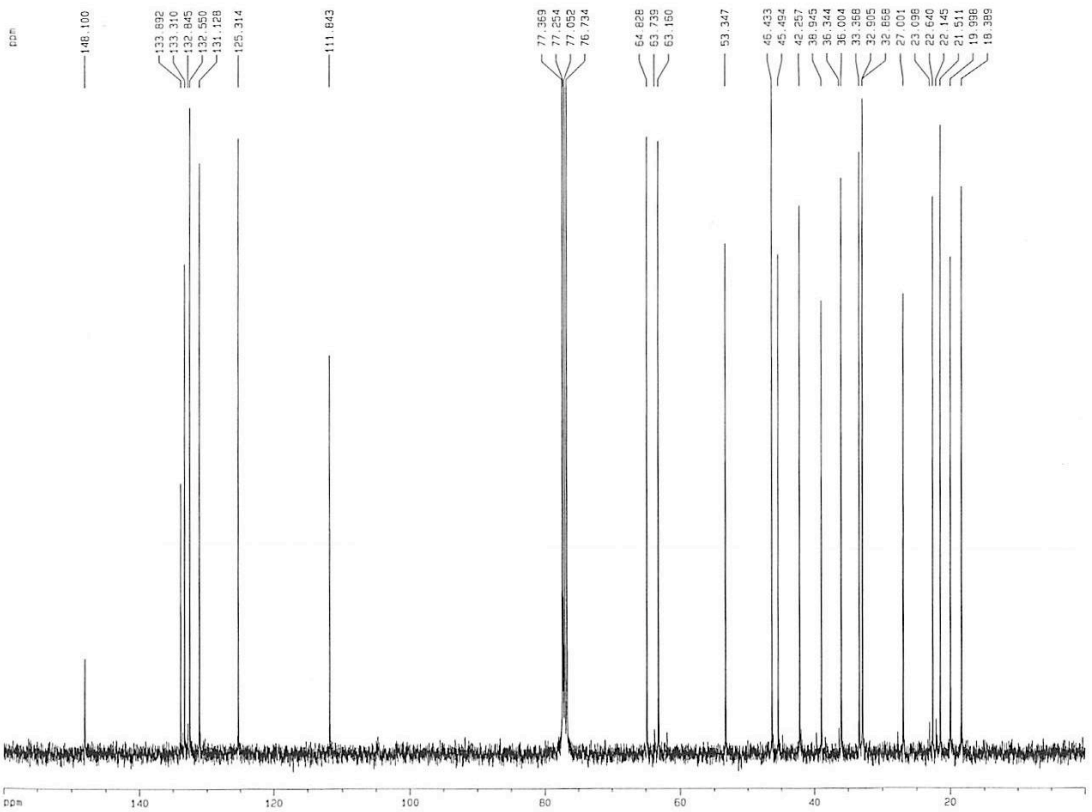
F2 - Acquisition Parameters
Date_ 20091114
Time 23.11
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 161.3
DW 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.0300067 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 18.00 cm
F1P 9.700 ppm
F1 3880.29 Hz
F2P -0.300 ppm
F2 -120.01 Hz
PPMCM 0.31250 ppm/cm
HZCM 125.00938 Hz/cm

DPX400 carbon



Current Data Parameters
NAME suzuki
EXPNO 13
PROCNO 1

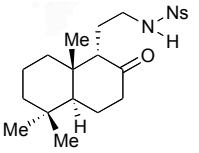
F2 - Acquisition Parameters
Date_ 20091114
Time 23.23
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1230
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 3251
DW 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

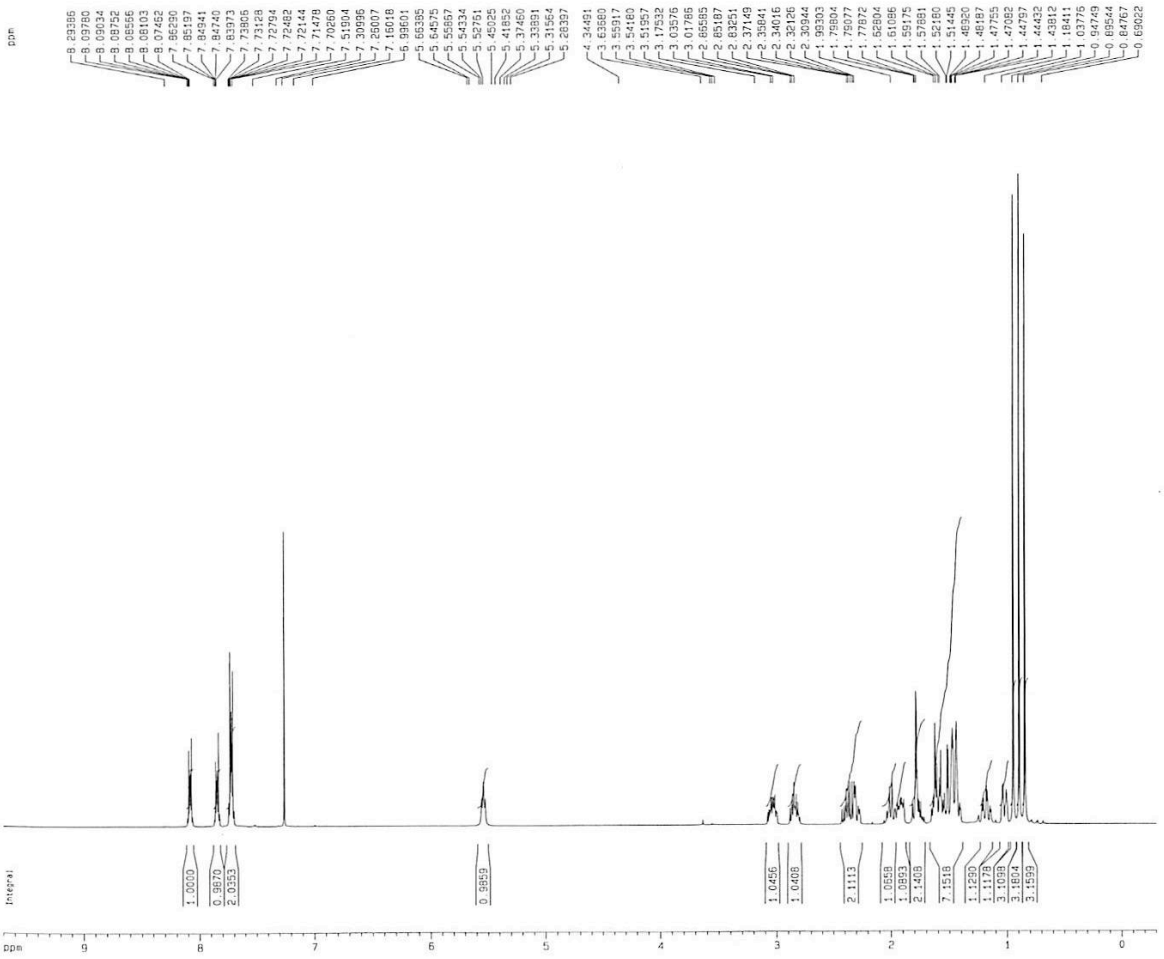
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

F2 - Processing parameters
SI 32768
SF 100.5876206 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 30.00 cm
CY 19.00 cm
F1P 160.000 ppm
F1 16094.02 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 5.33333 ppm/cm
HZCM 536.46735 Hz/cm





Current Data Parameters
NAME suzuki
EXPNO 24
PROCNO 1

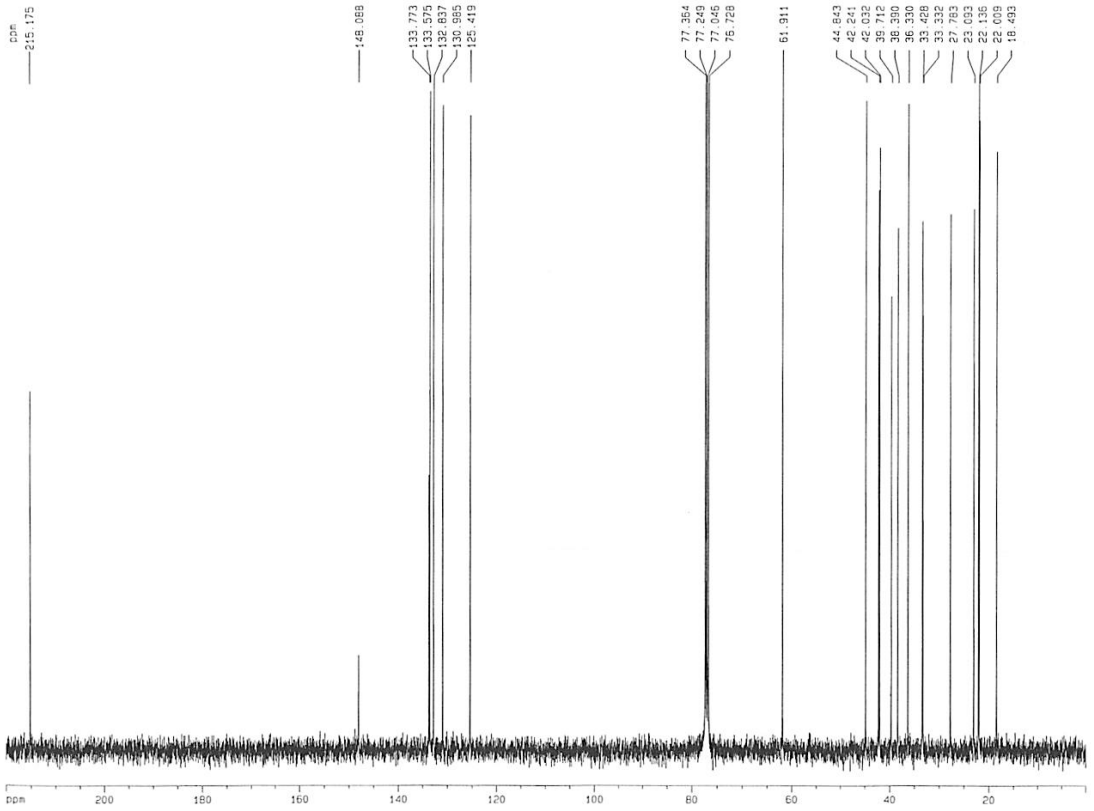
F2 - Acquisition Parameters
Date_ 20091115
Time 4.09
INSTRUM spect
PROBHD 5 mm GNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 203.2
DW 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.0300065 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 18.00 cm
F1P 9.700 ppm
F1 3880.29 Hz
F2P -0.300 ppm
F2 -120.01 Hz
PPMCM 0.31250 ppm/cm
HZCM 125.00938 Hz/cm

DPX400 carbon



Current Data Parameters
NAME suzuki
EXPNO 25
PROCNO 1

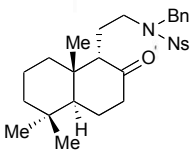
F2 - Acquisition Parameters
Date_ 20091115
Time 4.17
INSTRUM spect
PROBHD 5 mm GNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1029
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 8192
DW 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

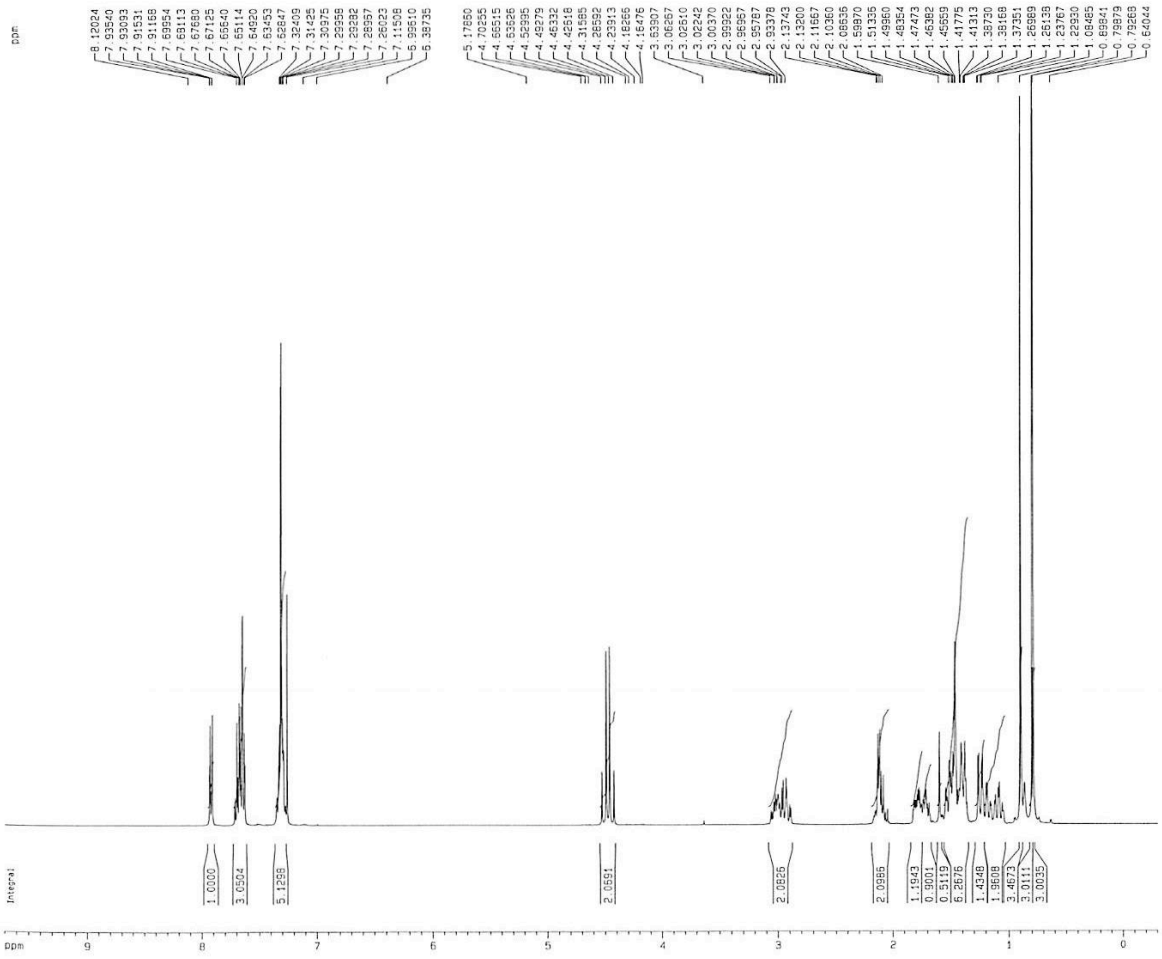
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

F2 - Processing parameters
SI 32768
SF 100.5876214 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

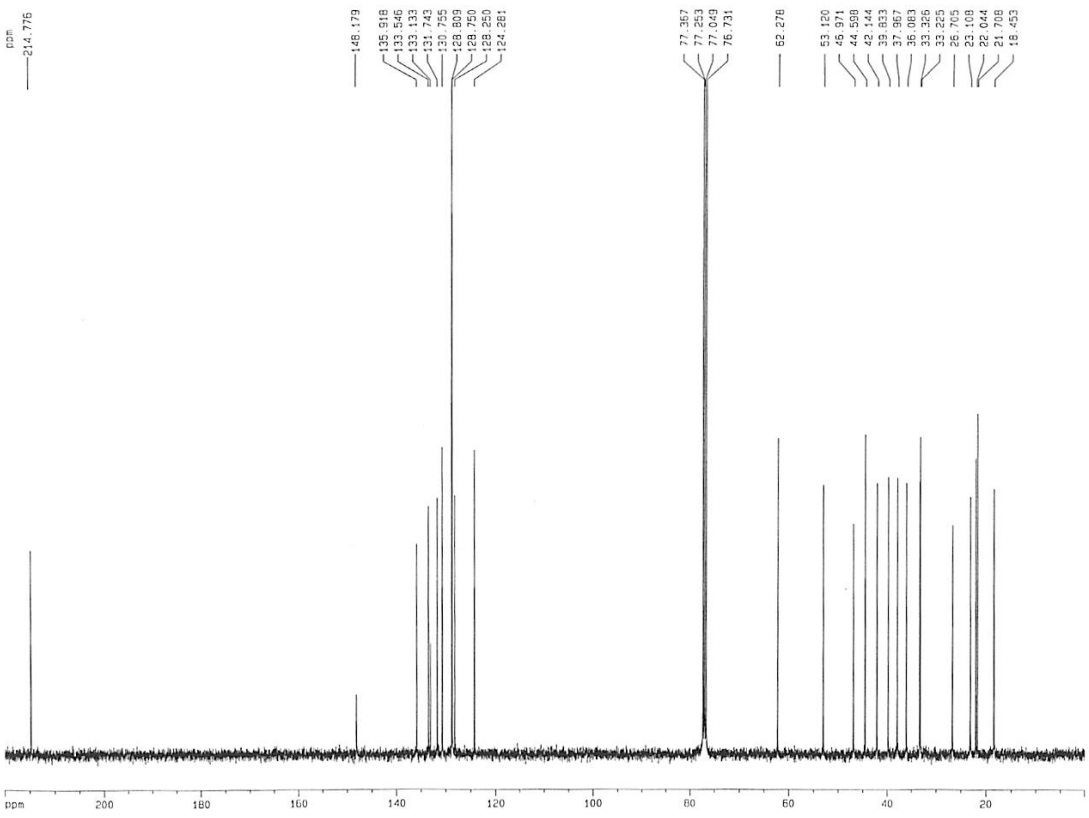
1D NMR plot parameters
CX 30.00 cm
CY 19.00 cm
F1P 220.000 ppm
F1 22129.28 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 7.33333 ppm/cm
HZCM 737.64258 Hz/cm



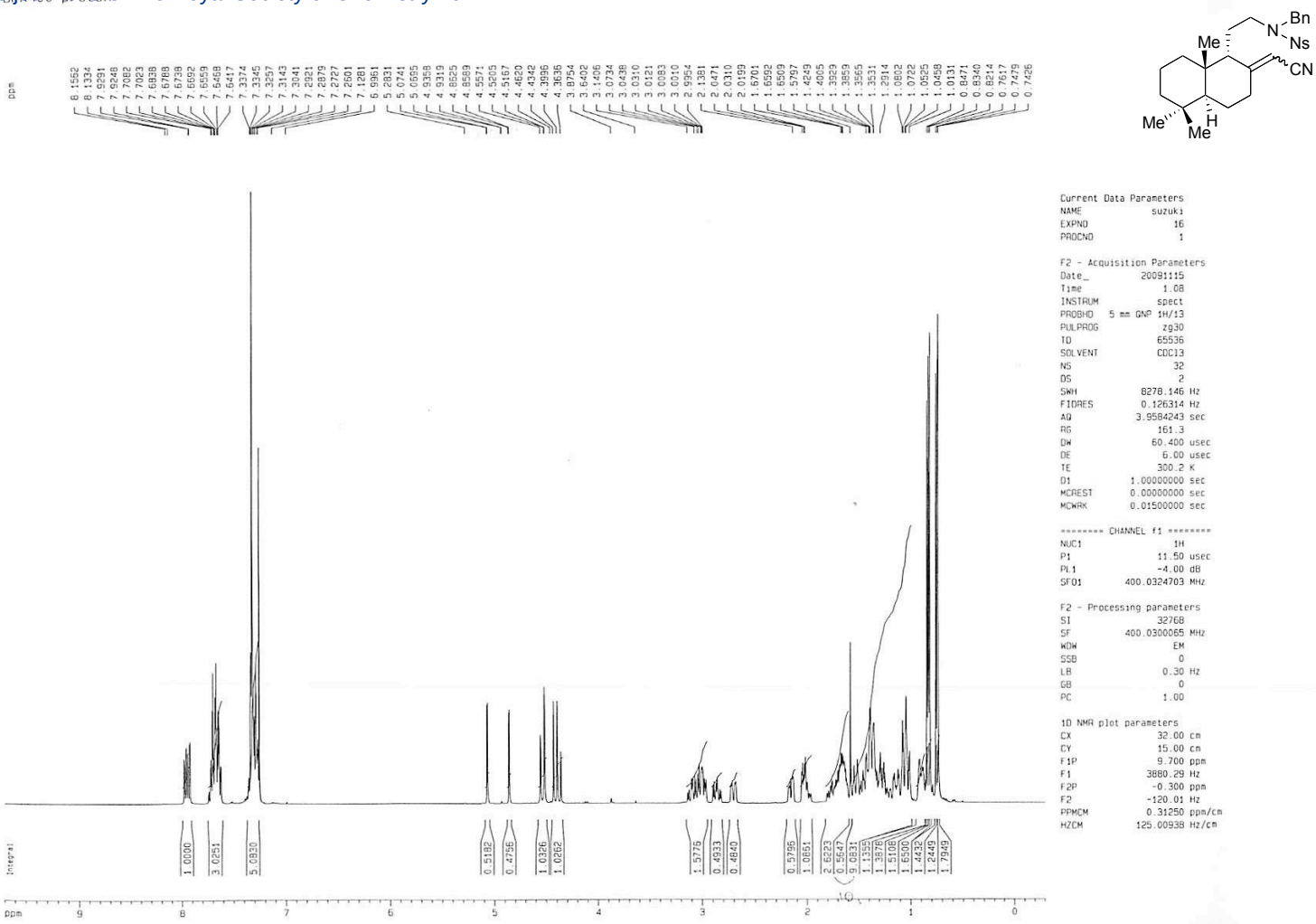


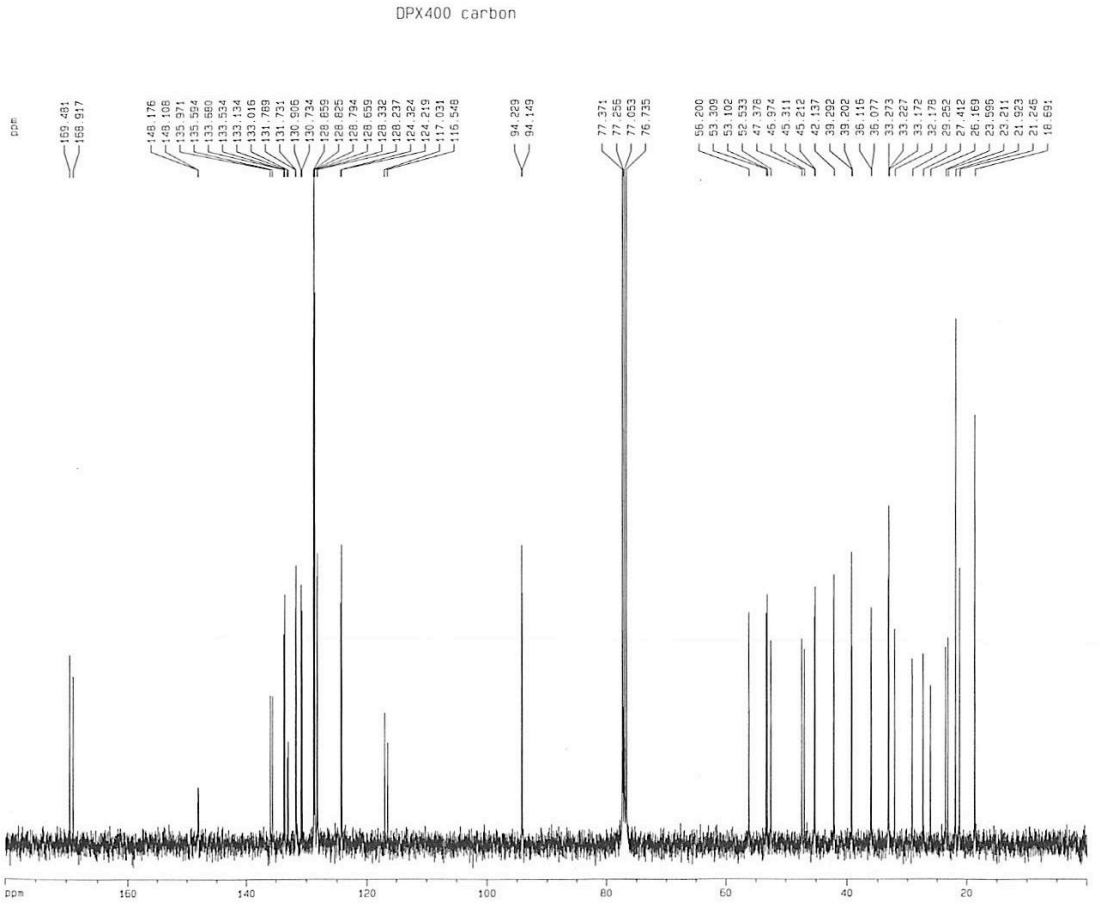
```
Current Data Parameters
NAME                 suzuki
EXPNO                    20
PROCNO                    1

F2 - Acquisition Parameters
Date_              20091115
Time                   2.33
INSTRUM               spect
PROBHD   5 mm QNP 1H/13
PULPROG               zg30
TD                   65536
SOLVENT               CDCl3
NS                       32
DS                        2
SWH                8278.146 Hz
FIDRES            0.126314 Hz
AQ                3.9584243 sec
RG                      181
DW                   60.400 usec
DE                    6.00 usec
TE                   300.2 K
D1                1.00000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                    11.50 usec
PL1                   -4.00 dB
SFO1             400.0324703 MHz

F2 - Processing parameters
SI                       32768
SF                400.0300065 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                       32.00 cm
CY                       21.00 cm
F1P                       9.700 ppm
F1                     3880.29 Hz
F2P                      -0.300 ppm
F2                    -120.01 Hz
PPMCM                 0.31250 ppm/cm
HZCM                125.00938 Hz/cm
```

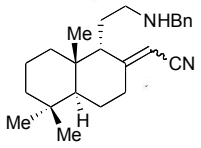
DPX400 carbon

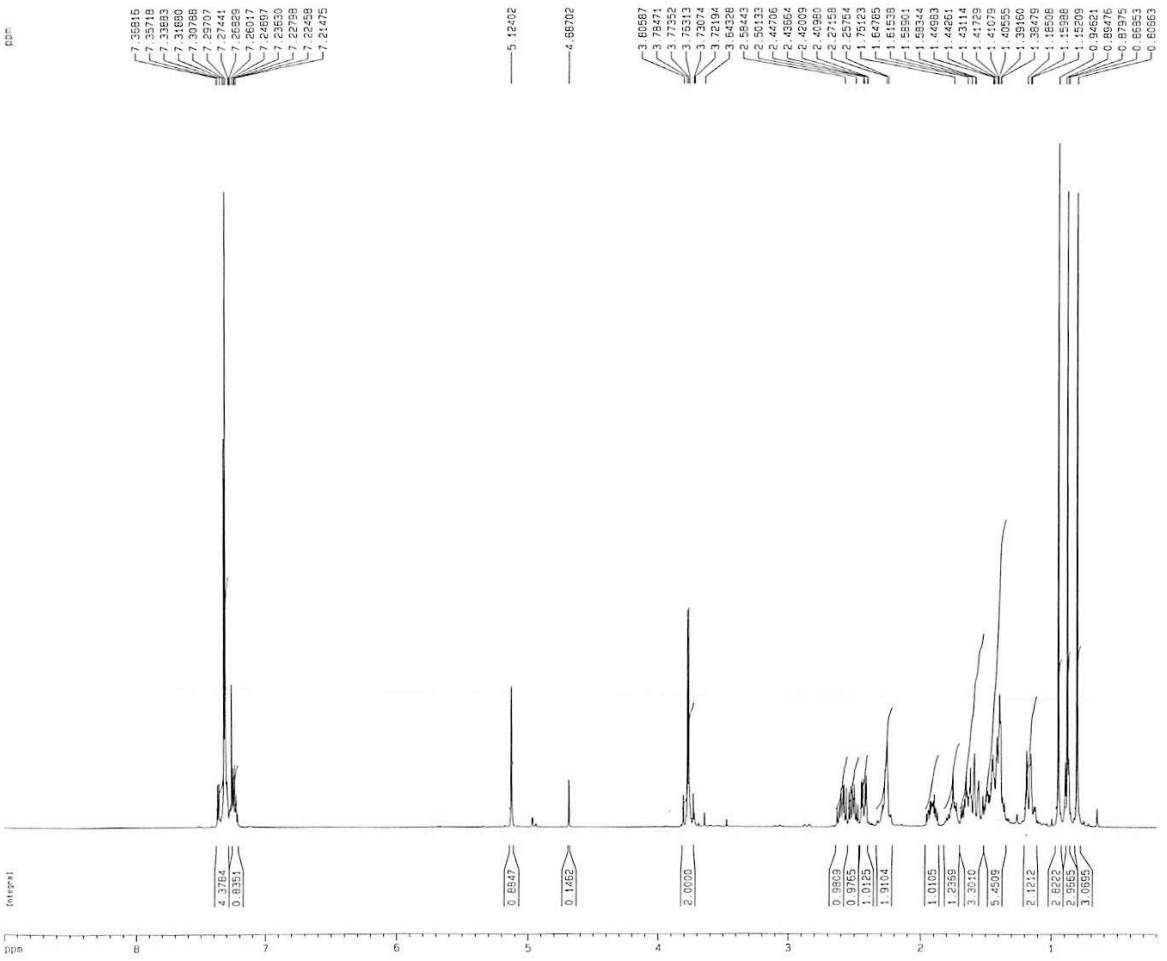


```
Current Data Parameters
NAME                 suzuki
EXPNO                    21
PROCNO                    1

F2 - Acquisition Parameters
Date_              20091115
Time                   2.42
INSTRUM               spect
PROBHD   5 mm QNP 1H/13
PULPROG             zgpg30
TD                   65536
SOLVENT               CDCl3
NS                      882
DS                        4
SWH               26178.010 Hz
FIDRES            0.399445 Hz
AQ                1.2517875 sec
RG                     8192
DW                   19.100 usec
DE                    6.00 usec
TE                   300.2 K
D1                2.00000000 sec
d11               0.03000000 sec
DELTA             1.89999998 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                    10.00 usec
PL1                   -3.50 dB
SFO1            100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
PCPD2                100.00 usec
PL2                   -4.00 dB
PL12                  17.00 dB
PL13                  17.00 dB
SFO2             400.0316001 MHz

F2 - Processing parameters
SI                       32768
SF               100.5876214 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40

1D NMR plot parameters
CX                       30.00 cm
CY                       19.00 cm
F1P                     220.000 ppm
F1                   22129.28 Hz
F2P                      0.000 ppm
F2                       0.00 Hz
PPMCM                 7.33333 ppm/cm
HZCM                737.64258 Hz/cm
```


Bn
N
Ns
Me
CN
Me
H
Me
Current Data Parameters
NAME suzuki
EXPNO 16
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091115
Time 1.08
INSTRUM spect
PROBHD 5 mm GNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 161.3
DW 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz
F2 - Processing parameters
SI 32768
SF 400.0300065 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 32.00 cm
CY 15.00 cm
F1P 9.700 ppm
F1 3880.29 Hz
F2P -0.300 ppm
F2 -120.01 Hz
PPMCM 0.31250 ppm/cm
HZCM 125.00938 Hz/cm

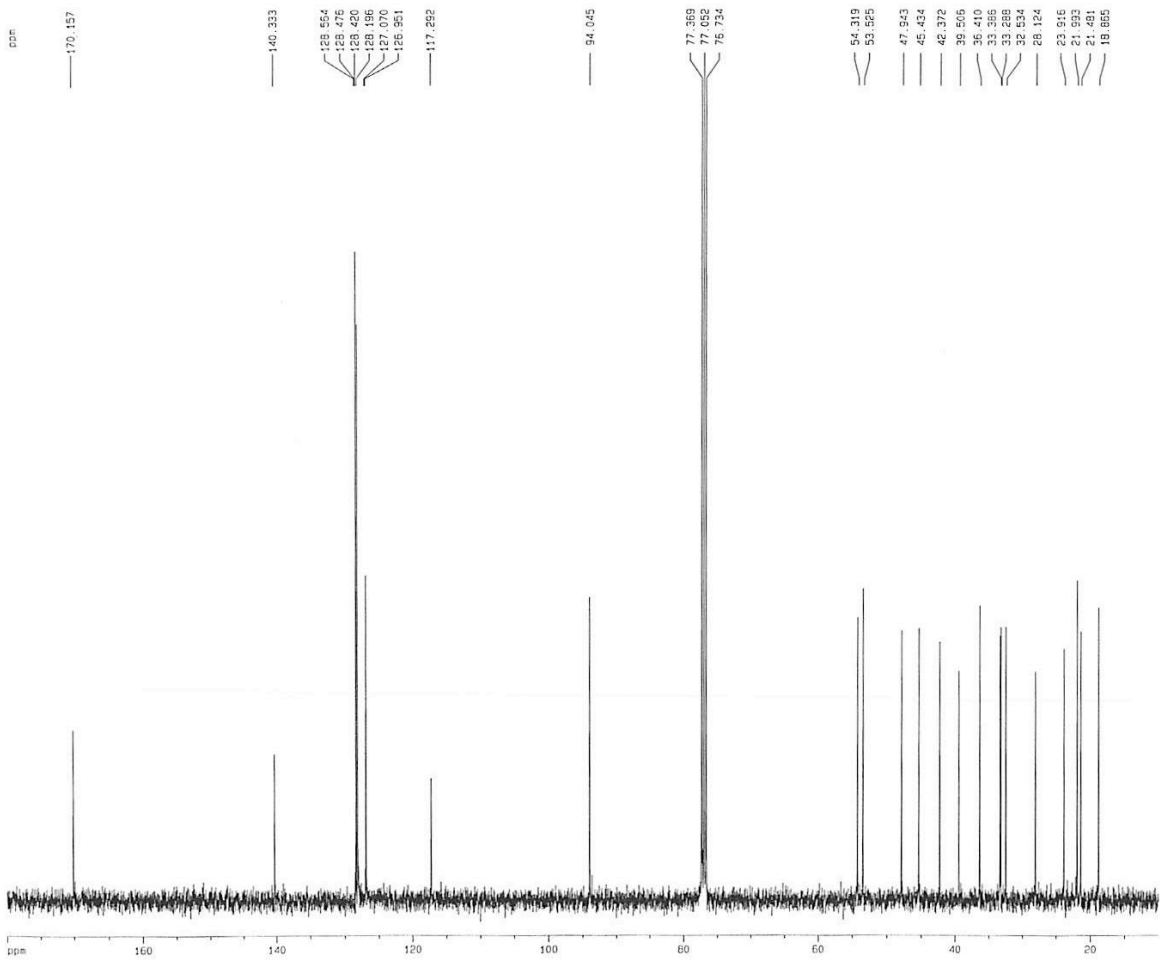

DPX400 carbon
Current Data Parameters
NAME suzuki
EXPNO 17
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091115
Time 1.30
INSTRUM spect
PROBHD 5 mm GNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 392
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 8192
DW 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5986886 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz
F2 - Processing parameters
SI 32768
SF 100.5876222 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 30.00 cm
CY 19.00 cm
F1P 180.000 ppm
F1 18105.77 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 6.00000 ppm/cm
HZCM 603.52570 Hz/cm



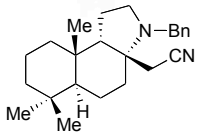


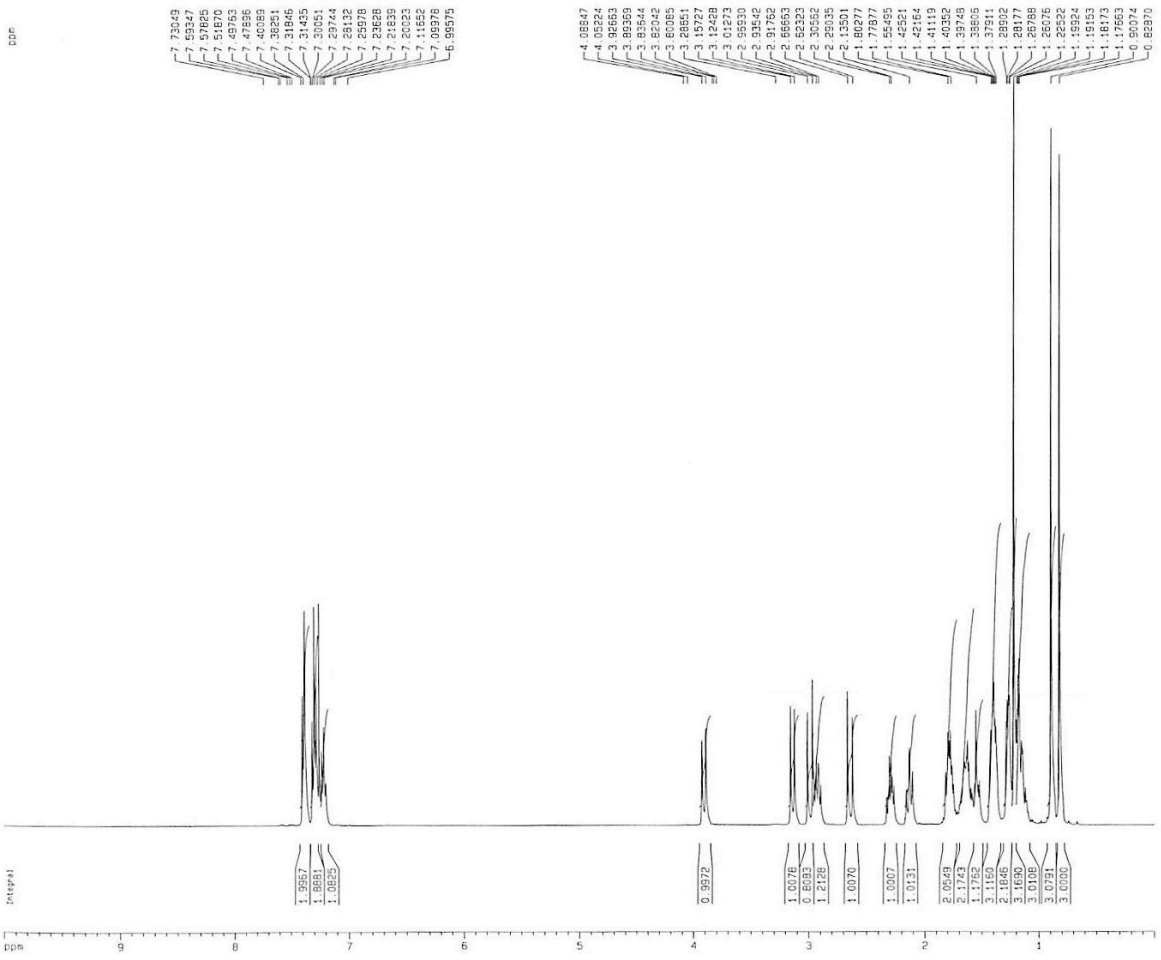
```
Current Data Parameters
NAME                 suzuki
EXPNO                    62
PROCNO                    1

F2 - Acquisition Parameters
Date_              20091219
Time                  18.09
INSTRUM               spect
PROBHD   5 mm GNP 1H/13
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       32
DS                        2
SWH               8278.146 Hz
FIDRES            0.126314 Hz
AQ                3.9584243 sec
RG                      181
DW                 60.400 usec
DE                  6.00 usec
TE                  300.2 K
D1                1.00000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                  11.50 usec
PL1                 -4.00 dB
SFO1          400.0324703 MHz

F2 - Processing parameters
SI                    32768
SF              400.0300065 MHz
WDW                      EM
SSB                       0
LB                   0.30 Hz
GB                        0
PC                     1.00

1D NMR plot parameters
CX                   32.00 cm
CY                   19.00 cm
F1P                   9.000 ppm
F1                 3600.27 Hz
F2P                   0.200 ppm
F2                   80.01 Hz
PPMCM             0.27500 ppm/cm
HZCM            110.00825 Hz/cm
```

DPX400 carbon



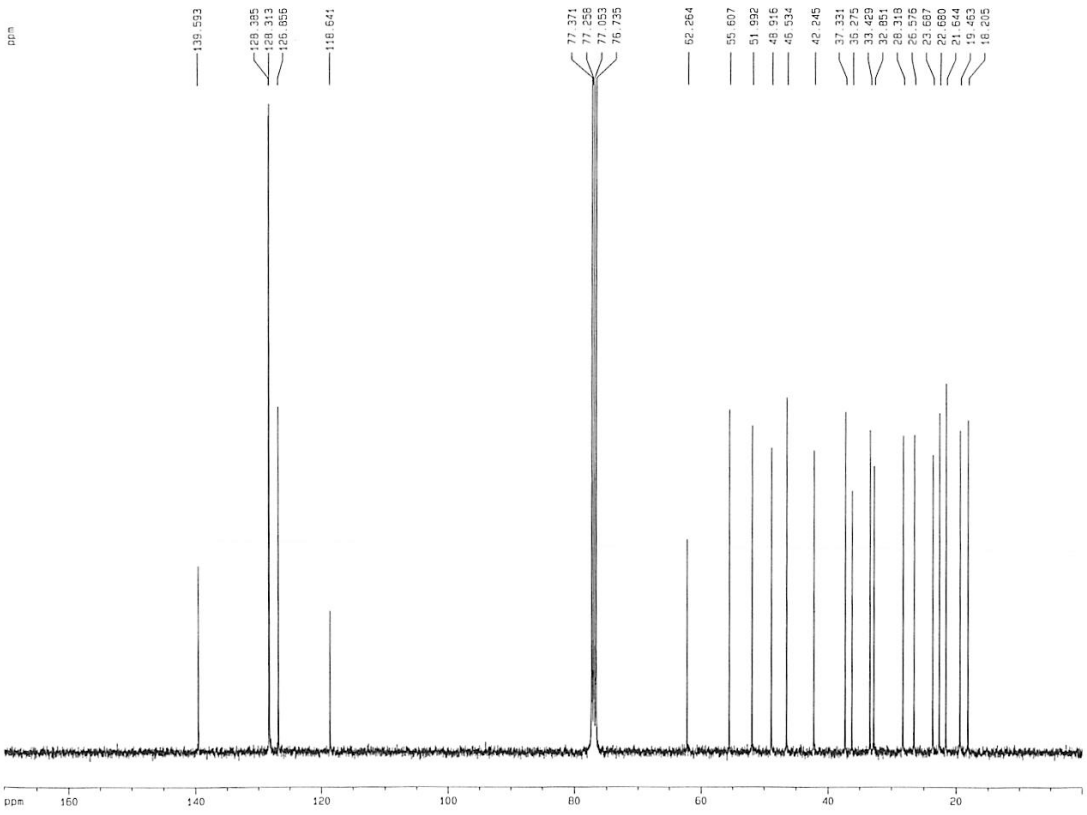
```
Current Data Parameters
NAME                 suzuki
EXPNO                    63
PROCNO                    1

F2 - Acquisition Parameters
Date_              20091219
Time                  18.22
INSTRUM               spect
PROBHD   5 mm GNP 1H/13
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      265
DS                        4
SWH              26178.010 Hz
FIDRES            0.399445 Hz
AQ                1.2517875 sec
RG                     8192
DW                 19.100 usec
DE                  6.00 usec
TE                  300.2 K
D1                2.00000000 sec
d11               0.03000000 sec
DELTA             1.89999998 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                  10.00 usec
PL1                  -3.50 dB
SFO1          100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2              100.00 usec
PL2                  -4.00 dB
PL12                 17.00 dB
PL13                 17.00 dB
SFO2          400.0316001 MHz

F2 - Processing parameters
SI                    32768
SF              100.5876214 MHz
WDW                      EM
SSB                       0
LB                   1.00 Hz
GB                        0
PC                     1.40

1D NMR plot parameters
CX                   32.00 cm
CY                   18.00 cm
F1P                 180.000 ppm
F1                18105.77 Hz
F2P                  10.000 ppm
F2                 1005.88 Hz
PPMCM             5.31250 ppm/cm
HZCM            534.37177 Hz/cm
```



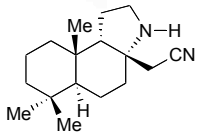


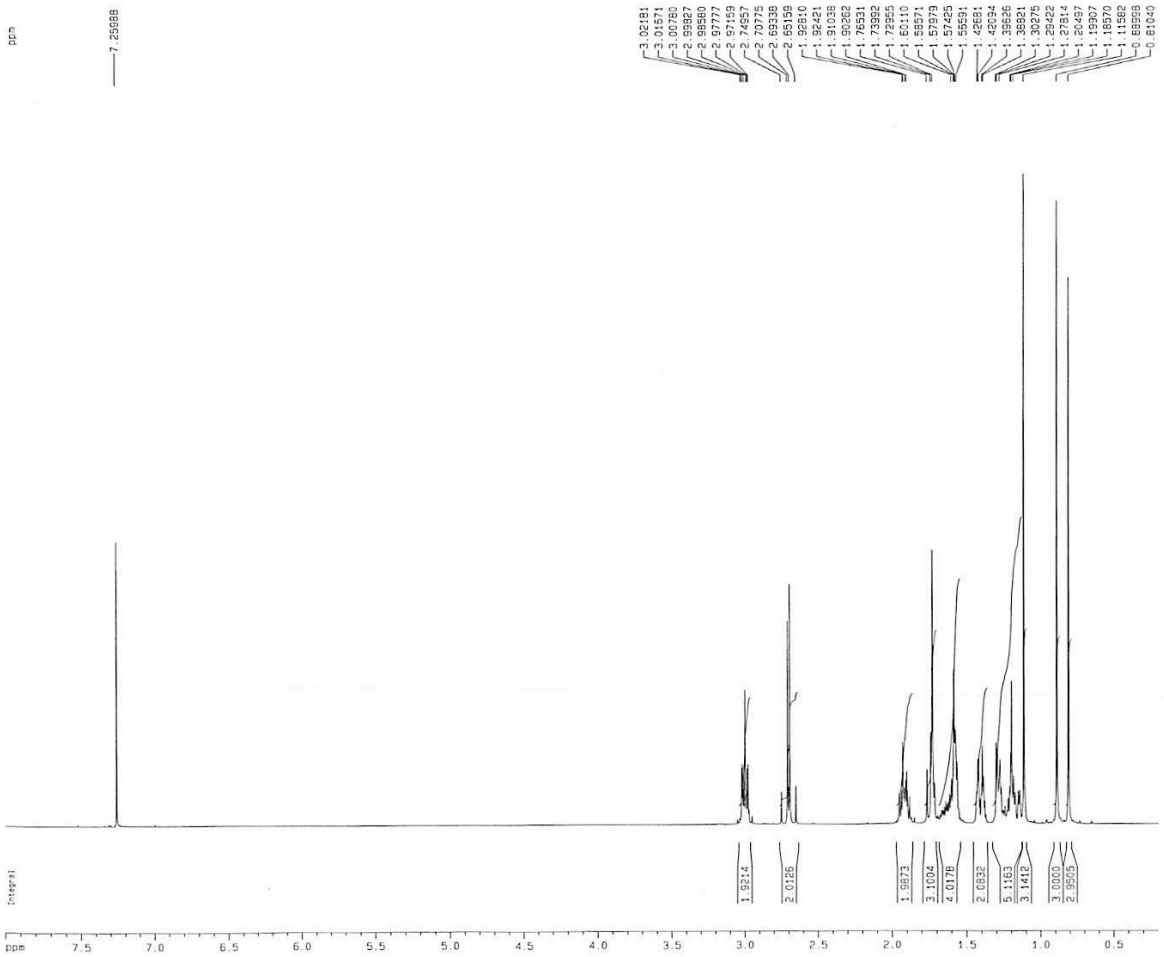
```
Current Data Parameters
NAME                 suzuki
EXPNO                    16
PROCNO                    1

F2 - Acquisition Parameters
Date_              20081205
Time                  22.13
INSTRUM               spect
PROBHD   5 mm QNP 1H/13
PULPROG               zg30
TD                   65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH               8278.146 Hz
FIDRES            0.126314 Hz
AQ                3.9584243 sec
RG                    161.3
DW                   60.400 usec
DE                    6.00 usec
TE                   300.2 K
D1                1.00000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                    11.50 usec
PL1                   -4.00 dB
SFO1            400.0324703 MHz

F2 - Processing parameters
SI                       32768
SF                400.0300070 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                       32.00 cm
CY                       21.00 cm
F1P                     10.000 ppm
F1                    4000.30 Hz
F2P                      0.000 ppm
F2                       0.00 Hz
PPMCM                 0.31250 ppm/cm
HZCM                125.00938 Hz/cm
```

DPX400 carbon



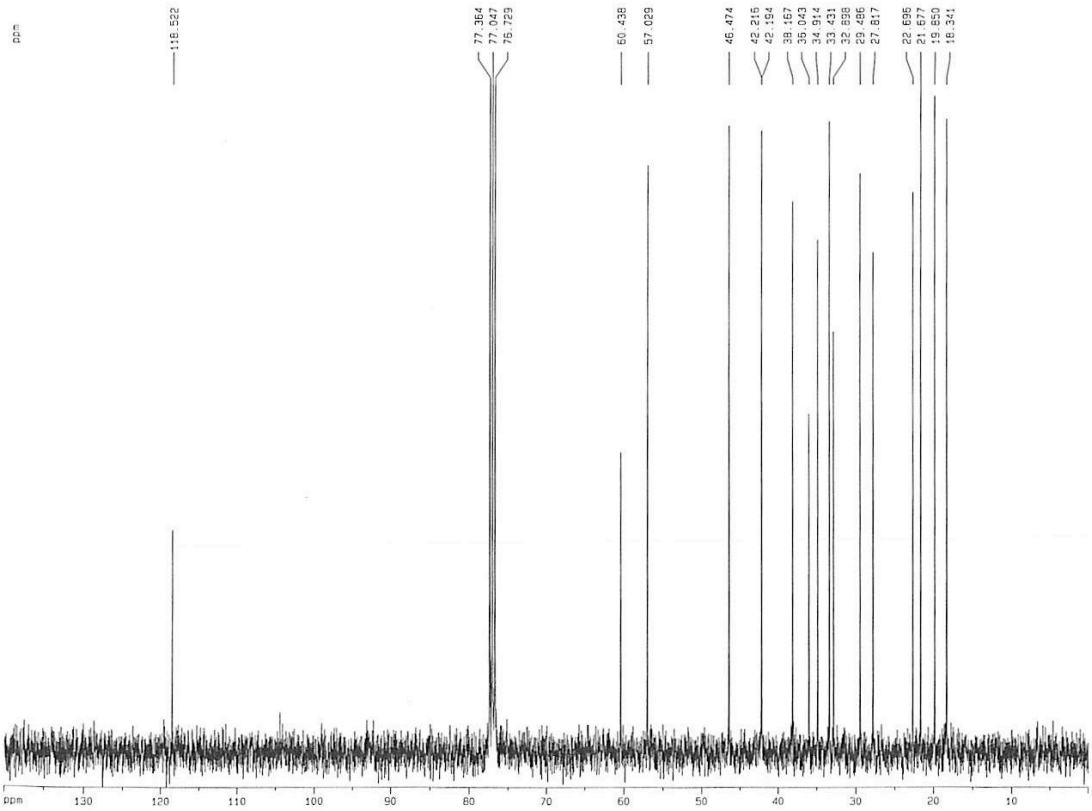
```
Current Data Parameters
NAME                 suzuki
EXPNO                    17
PROCNO                    1

F2 - Acquisition Parameters
Date_              20081205
Time                  22.34
INSTRUM               spect
PROBHD   5 mm QNP 1H/13
PULPROG              zgpg30
TD                   65536
SOLVENT               CDCl3
NS                     2366
DS                        4
SWH              26178.010 Hz
FIDRES            0.399445 Hz
AQ                1.2517875 sec
RG                     8192
DW                   19.100 usec
DE                    6.00 usec
TE                   300.2 K
D1                2.00000000 sec
d11               0.03000000 sec
DELTA             1.89999998 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                    10.00 usec
PL1                   -3.50 dB
SFO1            100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                100.00 usec
PL2                   -4.00 dB
PL12                  17.00 dB
PL13                  17.00 dB
SFO2            400.0316001 MHz

F2 - Processing parameters
SI                       32768
SF                100.5876206 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40

1D NMR plot parameters
CX                       30.00 cm
CY                       19.00 cm
F1P                     170.000 ppm
F1                   17099.90 Hz
F2P                      0.000 ppm
F2                       0.00 Hz
PPMCM                 5.66667 ppm/cm
HZCM                569.99652 Hz/cm
```



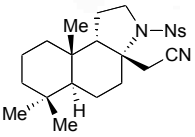


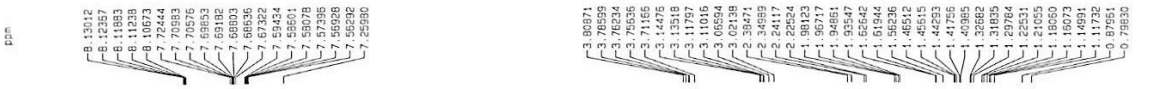

```
Current Data Parameters
NAME                 suzuki
EXPNO                   11B
PROCNO                    1

F2 - Acquisition Parameters
Date_               20101006
Time                   17.55
INSTRUM                spect
PROBHD   5 mm GNP 1H/13
PULPROG                zg30
TD                    65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                8278.146 Hz
FIDRES             0.126314 Hz
AQ                 3.9584243 sec
RG                     456.1
DW                    60.400 usec
DE                     6.00 usec
TE                    300.2 K
D1                 1.00000000 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                     11.50 usec
PL1                    -4.00 dB
SFO1           400.0324703 MHz

F2 - Processing parameters
SI                        32768
SF                400.0300067 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00

1D NMR plot parameters
CX                     32.00 cm
CY                     18.00 cm
F1P                    8.000 ppm
F1                   3200.24 Hz
F2P                    0.200 ppm
F2                     80.01 Hz
PPMCM                0.24375 ppm/cm
HZCM                97.50731 Hz/cm
```

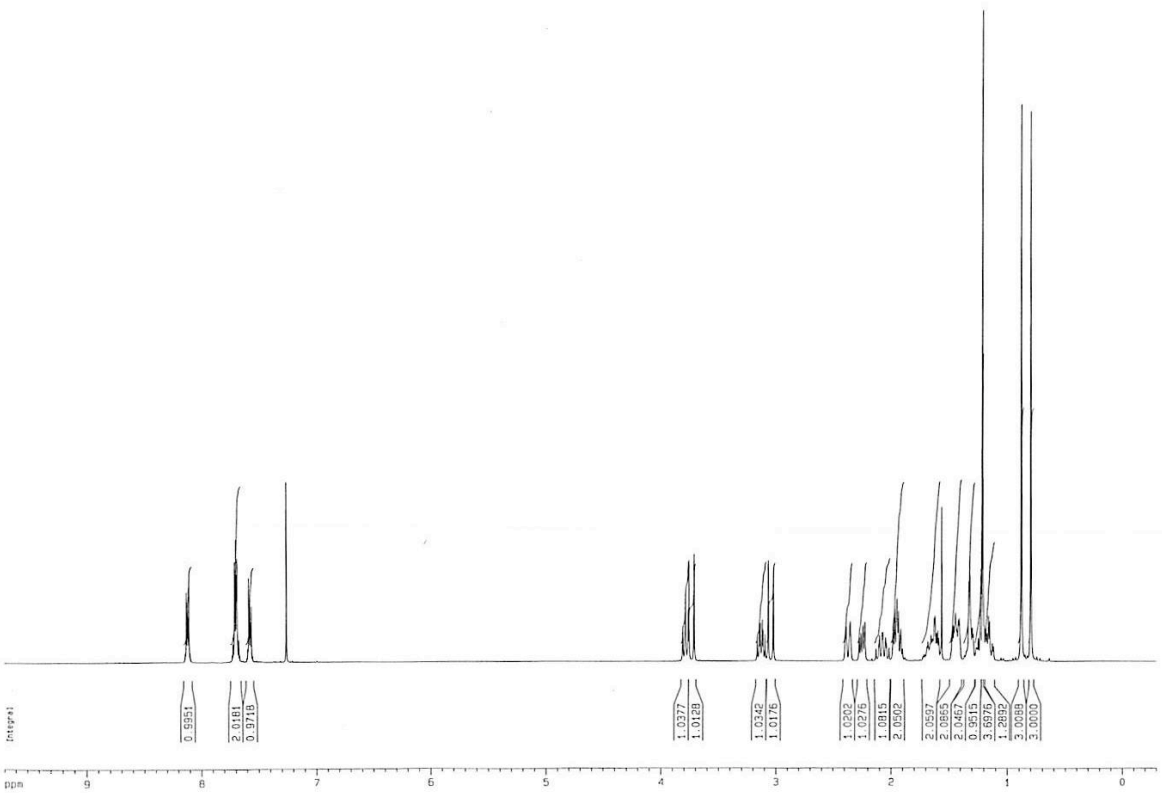
DPX400 carbon




```
Current Data Parameters
NAME                 suzuki
EXPNO                     6
PROCNO                    1

F2 - Acquisition Parameters
Date_               20091114
Time                   21.45
INSTRUM                spect
PROBHD   5 mm GNP 1H/13
PULPROG                zgpg30
TD                    65536
SOLVENT                CDCl3
NS                       193
DS                         4
SWH               26178.010 Hz
FIDRES             0.399445 Hz
AQ                 1.2517875 sec
RG                    5792.6
DW                    19.100 usec
DE                     6.00 usec
TE                    300.2 K
D1                 2.00000000 sec
d11                0.03000000 sec
DELTA              1.89999998 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                     10.00 usec
PL1                    -3.50 dB
SFO1           100.5966886 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                 100.00 usec
PL2                    -4.00 dB
PL12                   17.00 dB
PL13                   17.00 dB
SFO2           400.0316001 MHz

F2 - Processing parameters
SI                        32768
SF                100.5876214 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40

1D NMR plot parameters
CX                     30.00 cm
CY                     19.00 cm
F1P                  140.000 ppm
F1                 14082.27 Hz
F2P                    0.000 ppm
F2                     0.00 Hz
PPMCM                4.66667 ppm/cm
HZCM               469.40891 Hz/cm
```

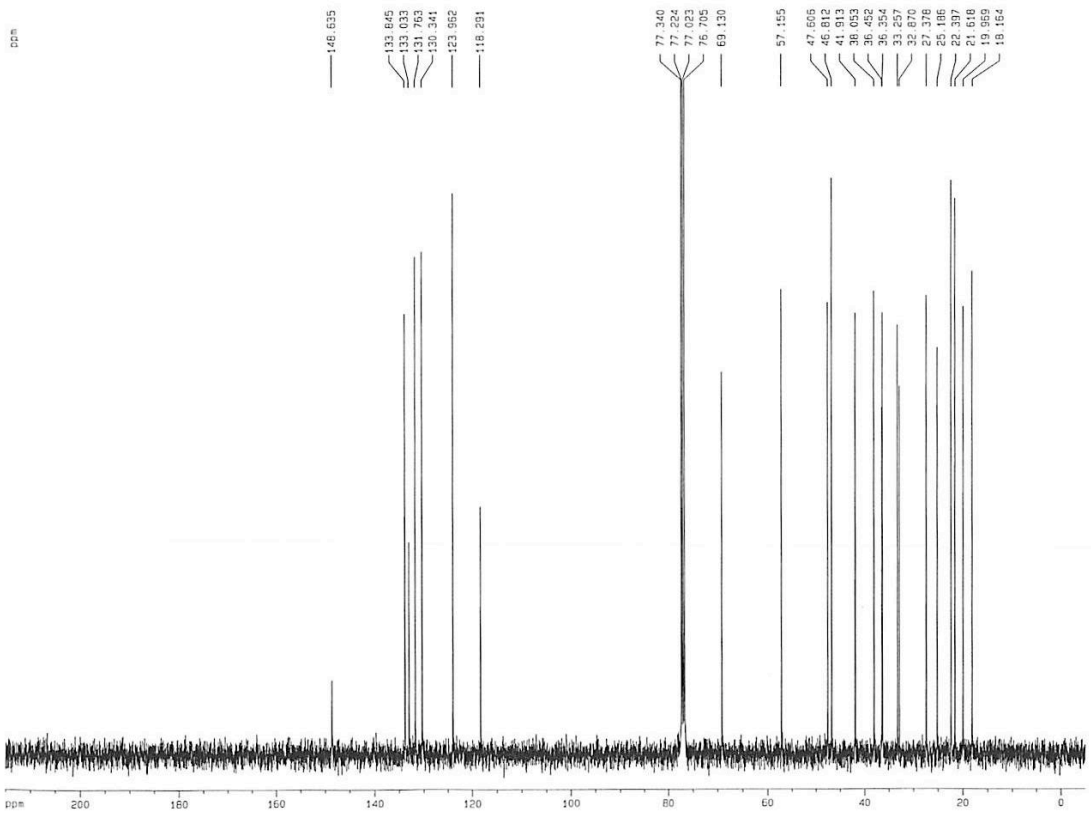




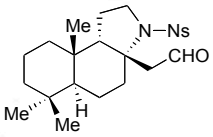


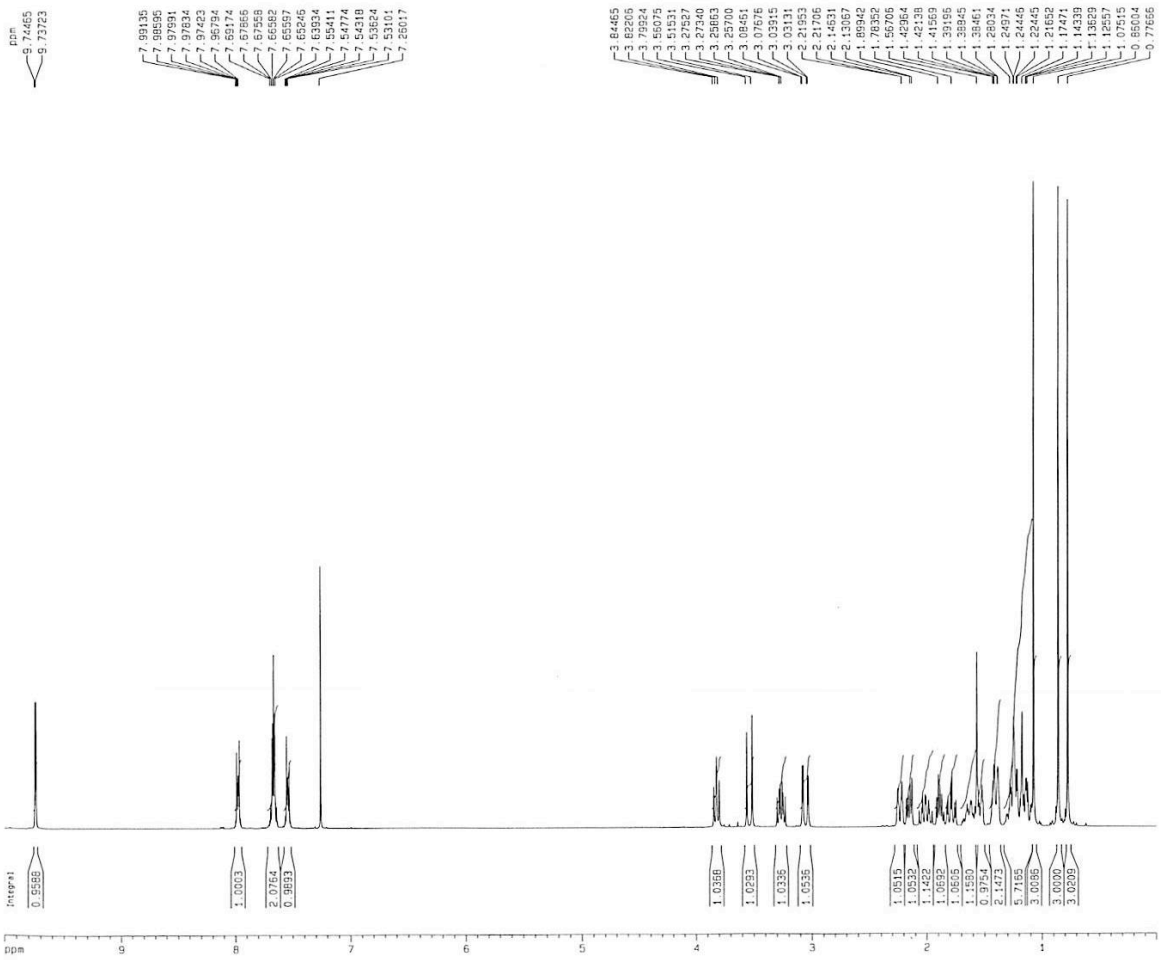
```
Current Data Parameters
NAME                 suzuki
EXPNO                    36
PROCNO                    1

F2 - Acquisition Parameters
Date_              20091203
Time                  17.53
INSTRUM               spect
PROBHD   5 mm QNP 1H/13
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       32
DS                        2
SWH               8278.146 Hz
FIDRES           0.126314 Hz
AQ               3.9584243 sec
RG                    228.1
DW                 60.400 usec
DE                  6.00 usec
TE                  300.2 K
D1              1.00000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                  11.50 usec
PL1                 -4.00 dB
SFO1          400.0324703 MHz

F2 - Processing parameters
SI                    32768
SF               400.0300067 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00

1D NMR plot parameters
CX                    32.00 cm
CY                    18.00 cm
F1P                   9.700 ppm
F1                  3880.29 Hz
F2P                  -0.300 ppm
F2                  -120.01 Hz
PPMCM              0.31250 ppm/cm
HZCM             125.00938 Hz/cm
```

DPX400 carbon



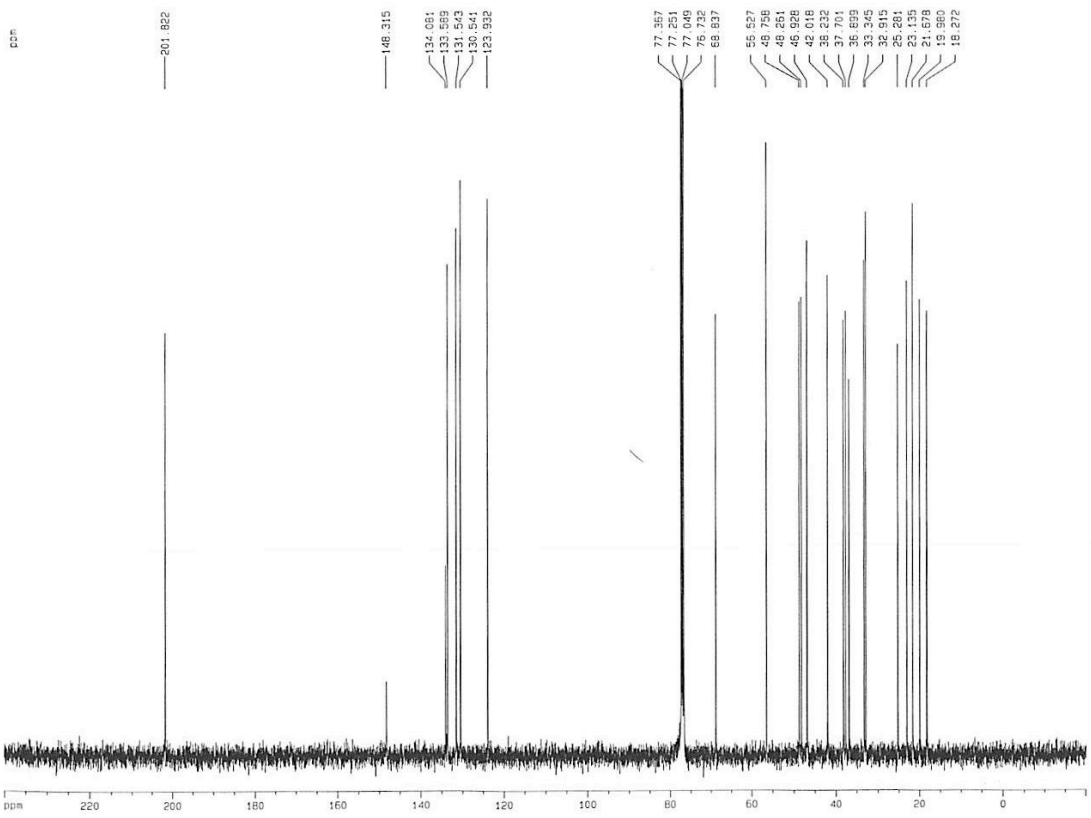
```
Current Data Parameters
NAME                 suzuki
EXPNO                    37
PROCNO                    1

F2 - Acquisition Parameters
Date_              20091203
Time                  18.13
INSTRUM               spect
PROBHD   5 mm QNP 1H/13
PULPROG              zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      620
DS                        4
SWH              26178.010 Hz
FIDRES           0.399445 Hz
AQ               1.2517875 sec
RG                   7298.2
DW                 19.100 usec
DE                  6.00 usec
TE                  300.2 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                  10.00 usec
PL1                 -3.50 dB
SFO1          100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2              100.00 usec
PL2                 -4.00 dB
PL12                17.00 dB
PL13                17.00 dB
SFO2          400.0316001 MHz

F2 - Processing parameters
SI                    32768
SF               100.5876240 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40

1D NMR plot parameters
CX                    30.00 cm
CY                    16.00 cm
F1P                 215.000 ppm
F1                21626.34 Hz
F2P                  -5.000 ppm
F2                 -502.94 Hz
PPMCM              7.33333 ppm/cm
HZCM             737.64258 Hz/cm
```



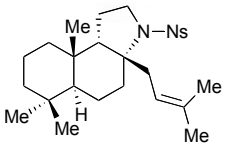


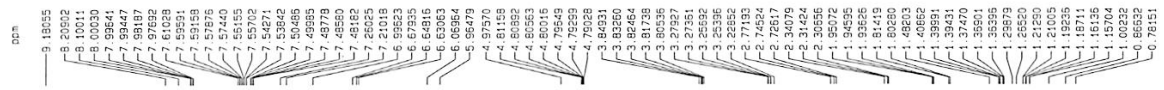
```
Current Data Parameters
NAME                 suzuki
EXPNO                    40
PROCNO                    1

F2 - Acquisition Parameters
Date_              20091204
Time                  19.57
INSTRUM               spect
PROBHD   5 mm QNP 1H/13
PULPROG               zg30
TD                   65536
SOLVENT               CDCl3
NS                       32
DS                        2
SWH               8278.146 Hz
FIDRES            0.126314 Hz
AQ                3.9584243 sec
RG                      256
DW                 60.400 usec
DE                  6.00 usec
TE                  300.2 K
D1                1.00000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                   11.50 usec
PL1                  -4.00 dB
SFO1          400.0324703 MHz

F2 - Processing parameters
SI                       32768
SF                400.0300065 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00

1D NMR plot parameters
CX                    32.00 cm
CY                    18.00 cm
F1P                 10.000 ppm
F1                4000.30 Hz
F2P                 0.000 ppm
F2                  0.00 Hz
PPMCM             0.31250 ppm/cm
HZCM            125.00938 Hz/cm
```

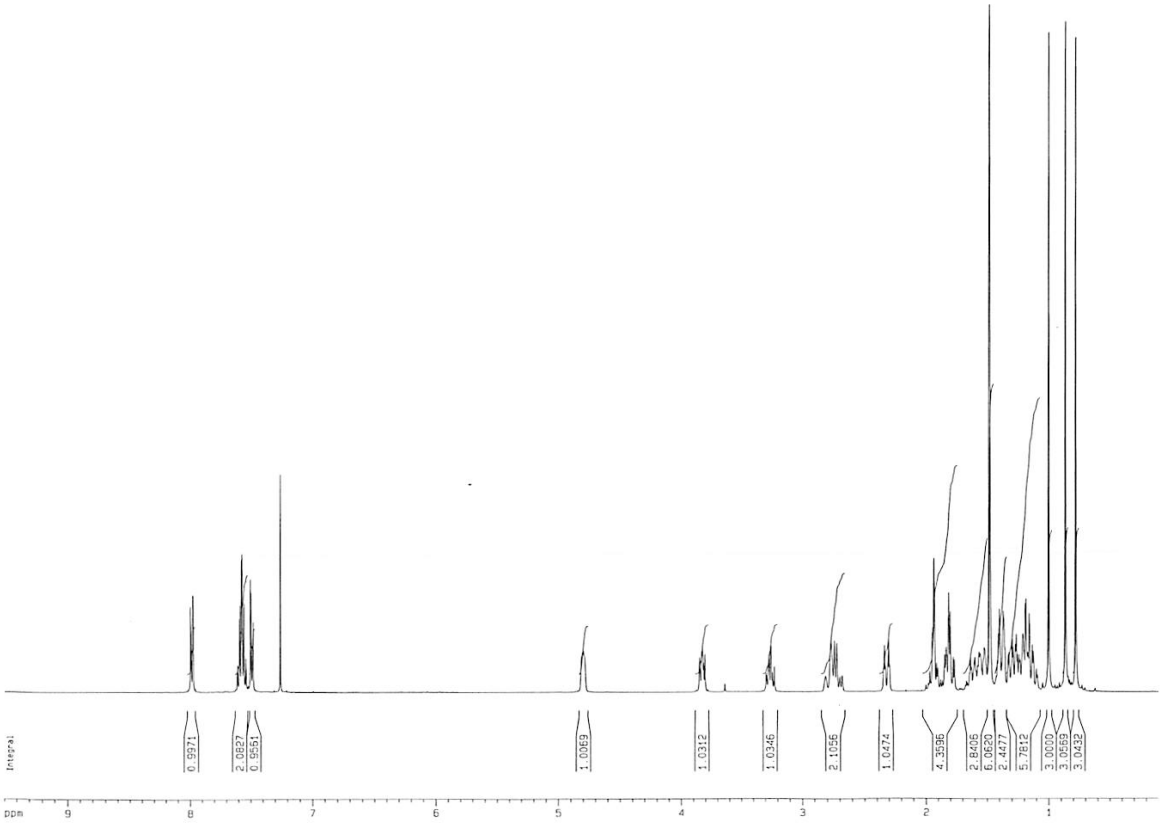
DPX400 carbon



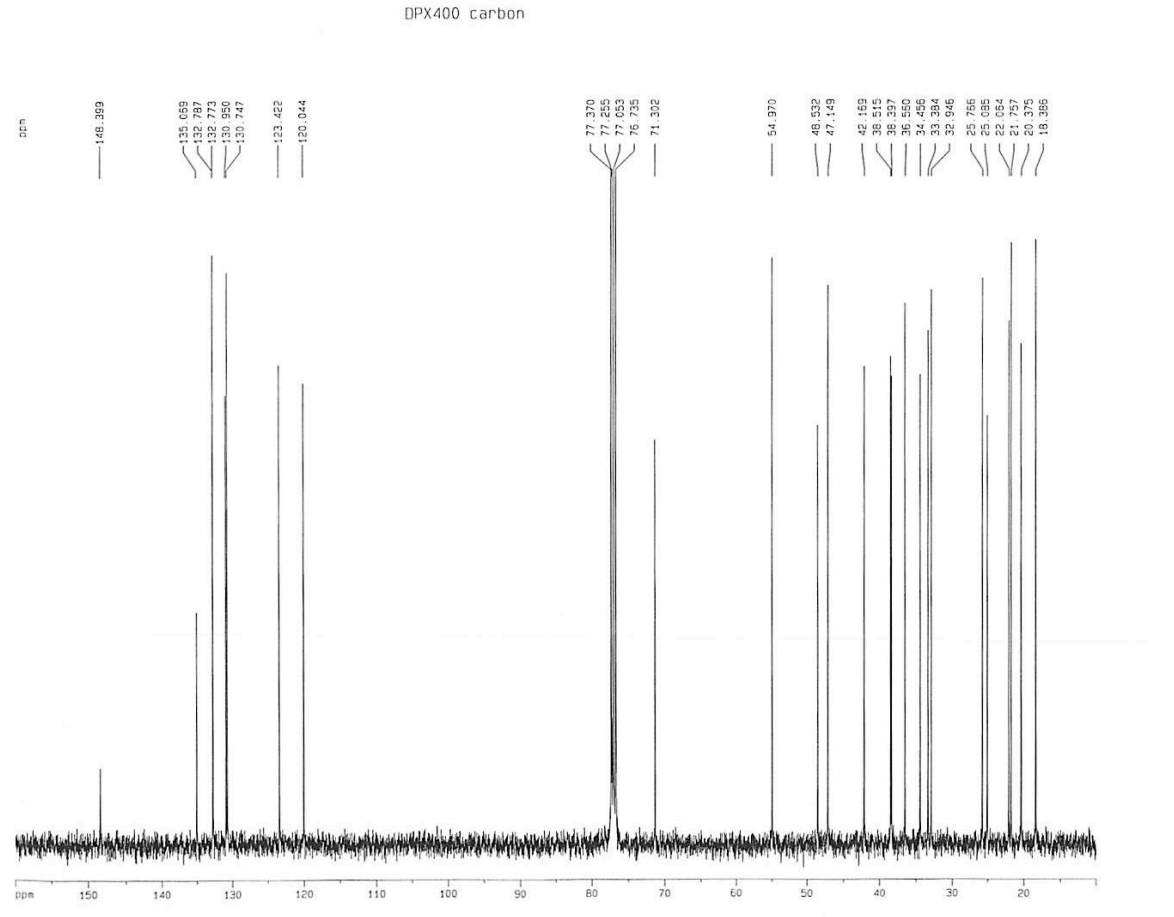
```
Current Data Parameters
NAME                 suzuki
EXPNO                    41
PROCNO                    1

F2 - Acquisition Parameters
Date_              20091204
Time                  20.10
INSTRUM               spect
PROBHD   5 mm QNP 1H/13
PULPROG             zgpg30
TD                   65536
SOLVENT               CDCl3
NS                     1683
DS                        4
SWH              26178.010 Hz
FIDRES            0.399445 Hz
AQ                1.2517875 sec
RG                    16384
DW                 19.100 usec
DE                  6.00 usec
TE                  300.2 K
D1                2.00000000 sec
d11               0.03000000 sec
DELTA             1.89999998 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                   10.00 usec
PL1                  -3.50 dB
SFO1          100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
PCPD2              100.00 usec
PL2                  -4.00 dB
PL12                 17.00 dB
PL13                 17.00 dB
SFO2          400.0316001 MHz

F2 - Processing parameters
SI                       32768
SF               100.5876206 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40

1D NMR plot parameters
CX                    30.00 cm
CY                    17.00 cm
F1P                 240.159 ppm
F1                24157.07 Hz
F2P                -20.091 ppm
F2                -2020.94 Hz
PPMCM              8.67503 ppm/cm
HZCM            872.60028 Hz/cm
```



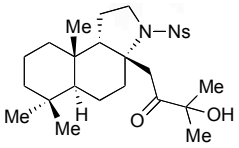


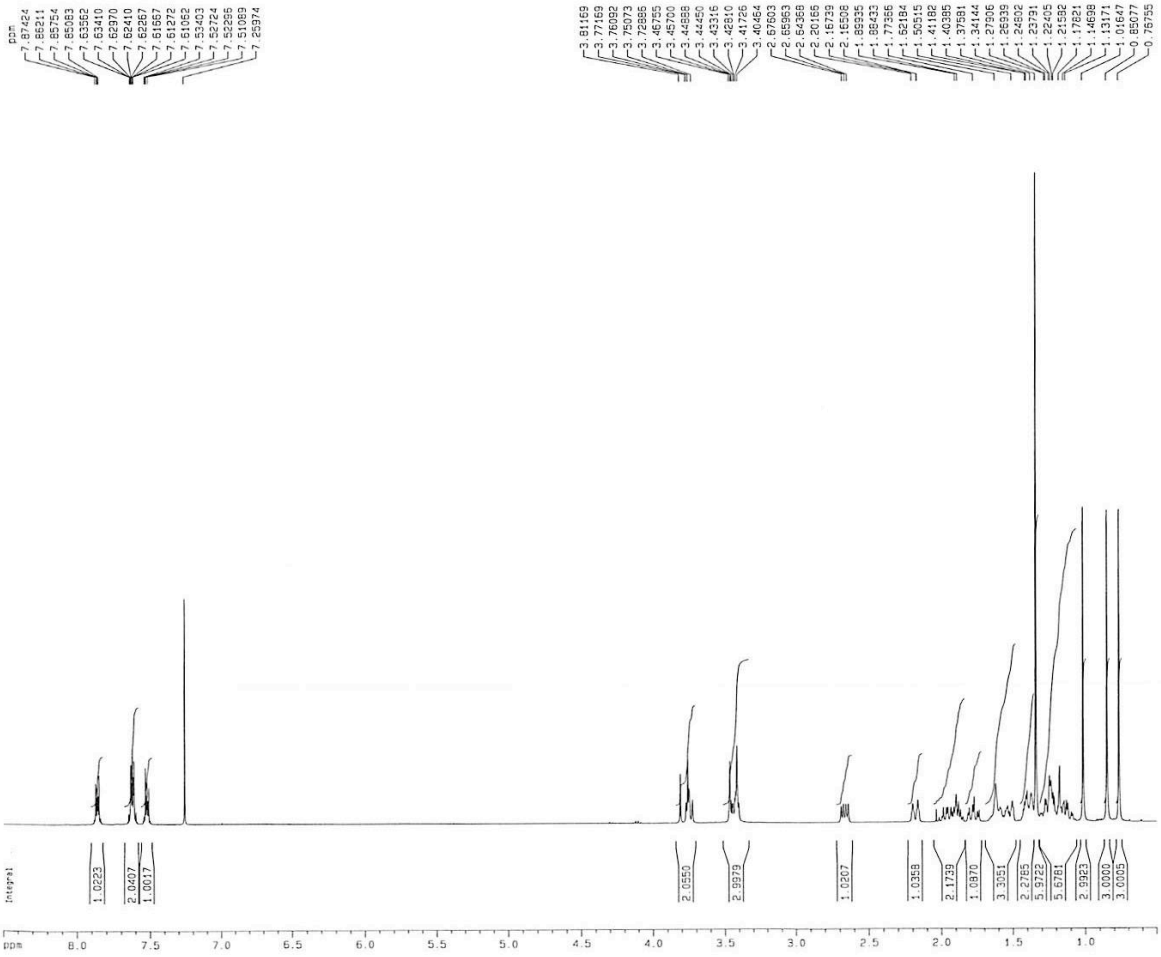


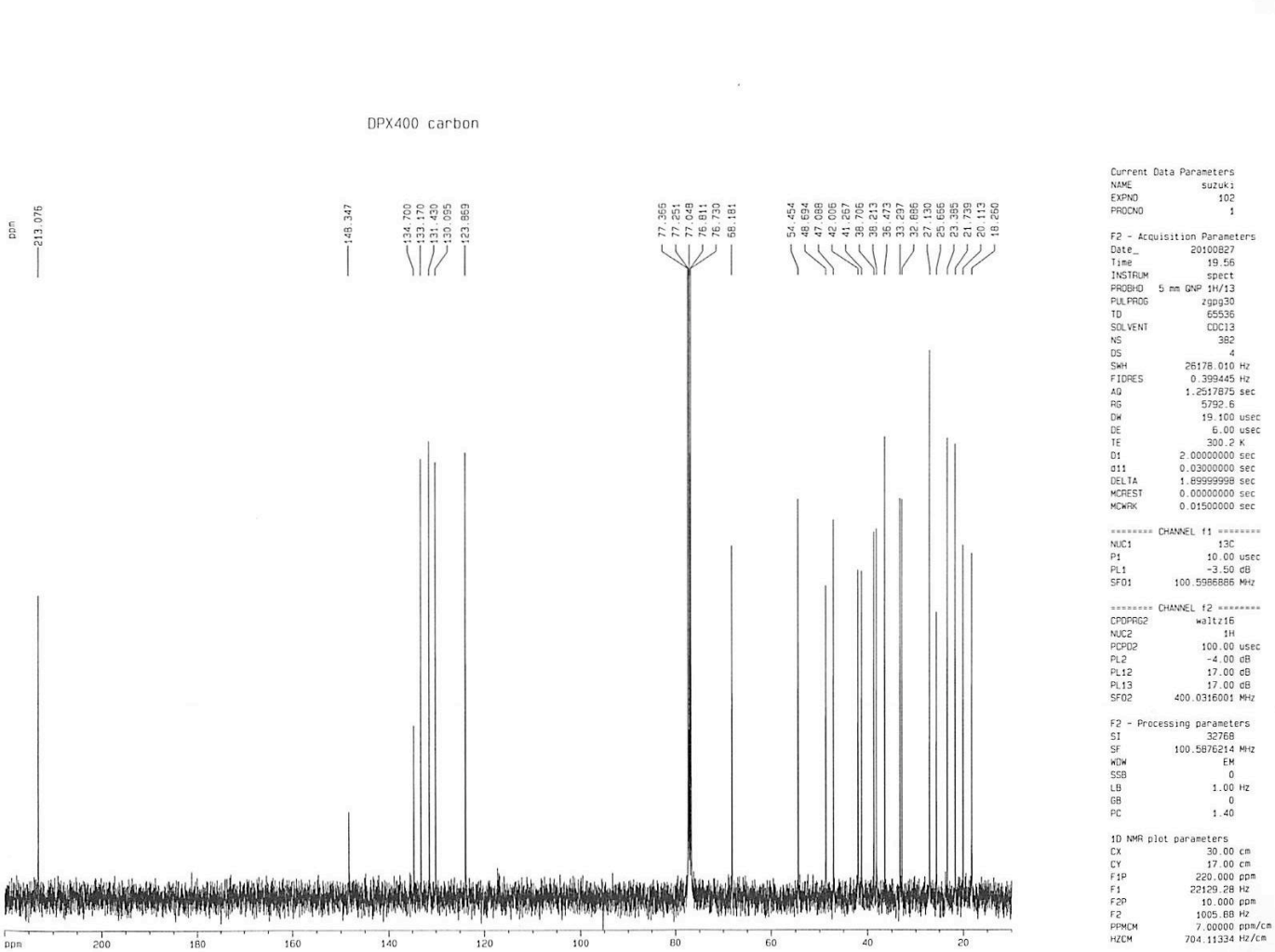
```
Current Data Parameters
NAME                 suzuki
EXPNO                   48
PROCNO                   1

F2 - Acquisition Parameters
Date_               20091209
Time                   19.52
INSTRUM                spect
PROBHD   5 mm QNP 1H/13
PULPROG                zg30
TD                    65536
SOLVENT                CDCl3
NS                        32
DS                         2
SWH                8278.146 Hz
FIDRES            0.126314 Hz
AQ                3.9584243 sec
RG                     161.3
DW                  60.400 usec
DE                   6.00 usec
TE                   300.2 K
D1                1.00000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                     11.50 usec
PL1                    -4.00 dB
SFO1             400.0324703 MHz

F2 - Processing parameters
SI                        32768
SF                400.0300065 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00

1D NMR plot parameters
CX                     32.00 cm
CY                     19.00 cm
F1P                    9.500 ppm
F1                   3800.28 Hz
F2P                    0.100 ppm
F2                     40.00 Hz
PPMCM              0.29375 ppm/cm
HZCM             117.50881 Hz/cm
```

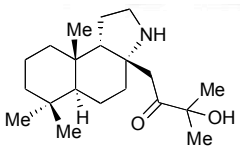


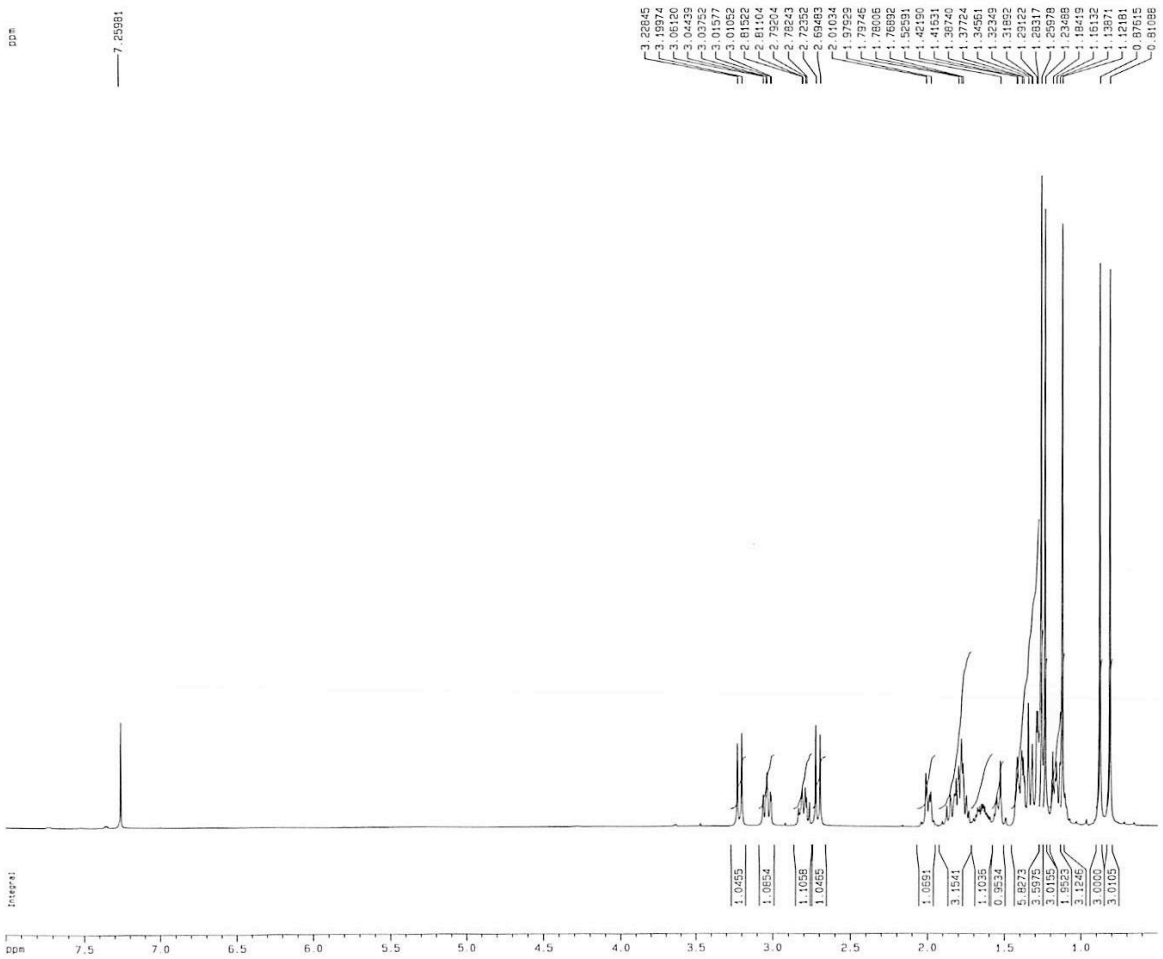

```
Current Data Parameters
NAME                 suzuki
EXPNO                   49
PROCNO                   1

F2 - Acquisition Parameters
Date_               20091209
Time                   20.01
INSTRUM                spect
PROBHD   5 mm QNP 1H/13
PULPROG              zgpg30
TD                    65536
SOLVENT                CDCl3
NS                       770
DS                         4
SWH               26178.010 Hz
FIDRES            0.399445 Hz
AQ                1.2517875 sec
RG                      8192
DW                  19.100 usec
DE                   6.00 usec
TE                   300.2 K
D1                2.00000000 sec
d11               0.03000000 sec
DELTA             1.89999998 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                     10.00 usec
PL1                    -3.50 dB
SFO1            100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                 100.00 usec
PL2                    -4.00 dB
PL12                   17.00 dB
PL13                   17.00 dB
SFO2            400.0316001 MHz

F2 - Processing parameters
SI                        32768
SF                100.5876206 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40

1D NMR plot parameters
CX                     30.00 cm
CY                     17.00 cm
F1P                   160.000 ppm
F1                  16094.02 Hz
F2P                   10.000 ppm
F2                   1005.88 Hz
PPMCM              5.00000 ppm/cm
HZCM             502.93811 Hz/cm
```


Me
N–Ns
Me
H
Me
O
Me
OH
Me


Current Data Parameters
NAME suzuki
EXPNO 101
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100827
Time 19.41
INSTRUM spect
PROBHD 5 mm GNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 203.2
DW 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz
F2 - Processing parameters
SI 32768
SF 400.0300067 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 32.00 cm
CY 18.00 cm
F1P 8.500 ppm
F1 3400.25 Hz
F2P 0.500 ppm
F2 200.01 Hz
PPMCM 0.25000 ppm/cm
HZCM 100.00750 Hz/cm

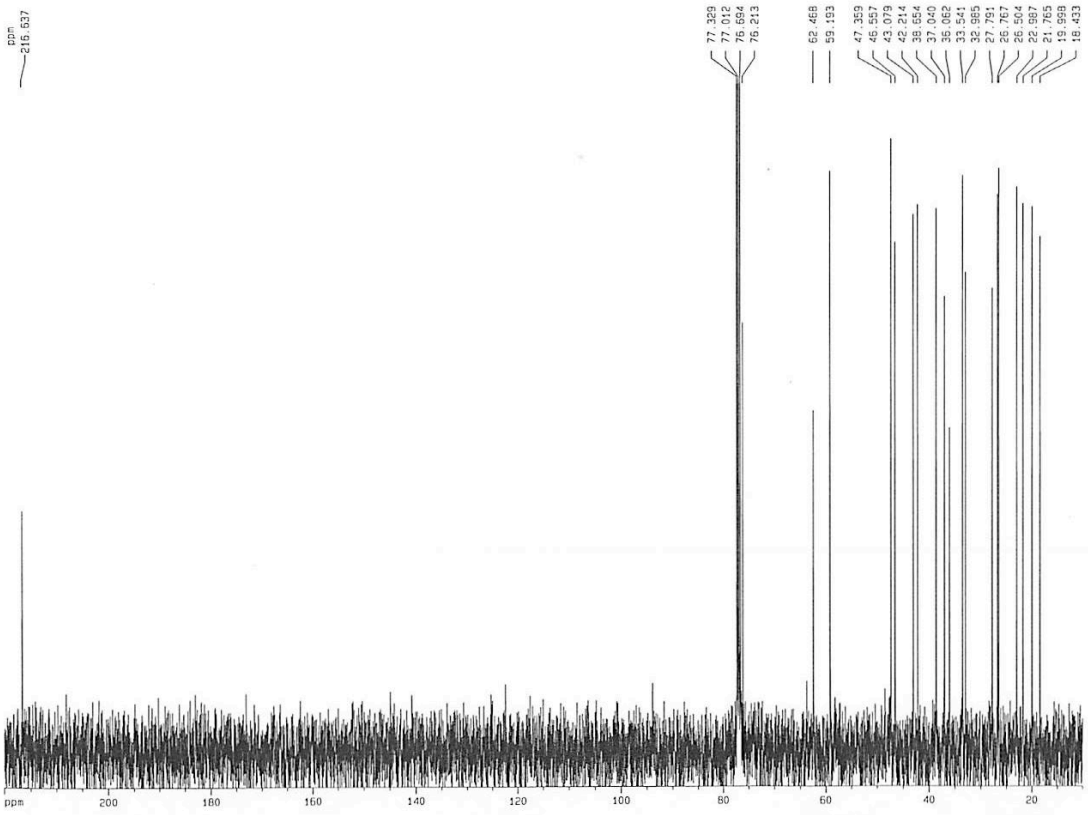

DPX400 carbon
Current Data Parameters
NAME suzuki
EXPNO 102
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100827
Time 19.56
INSTRUM spect
PROBHD 5 mm GNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 382
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 5792.6
DW 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5986886 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz
F2 - Processing parameters
SI 32768
SF 100.5876214 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 30.00 cm
CY 17.00 cm
F1P 220.000 ppm
F1 22129.28 Hz
F2P 10.000 ppm
F2 1005.88 Hz
PPMCM 7.00000 ppm/cm
HZCM 704.11334 Hz/cm



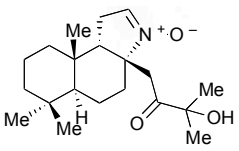


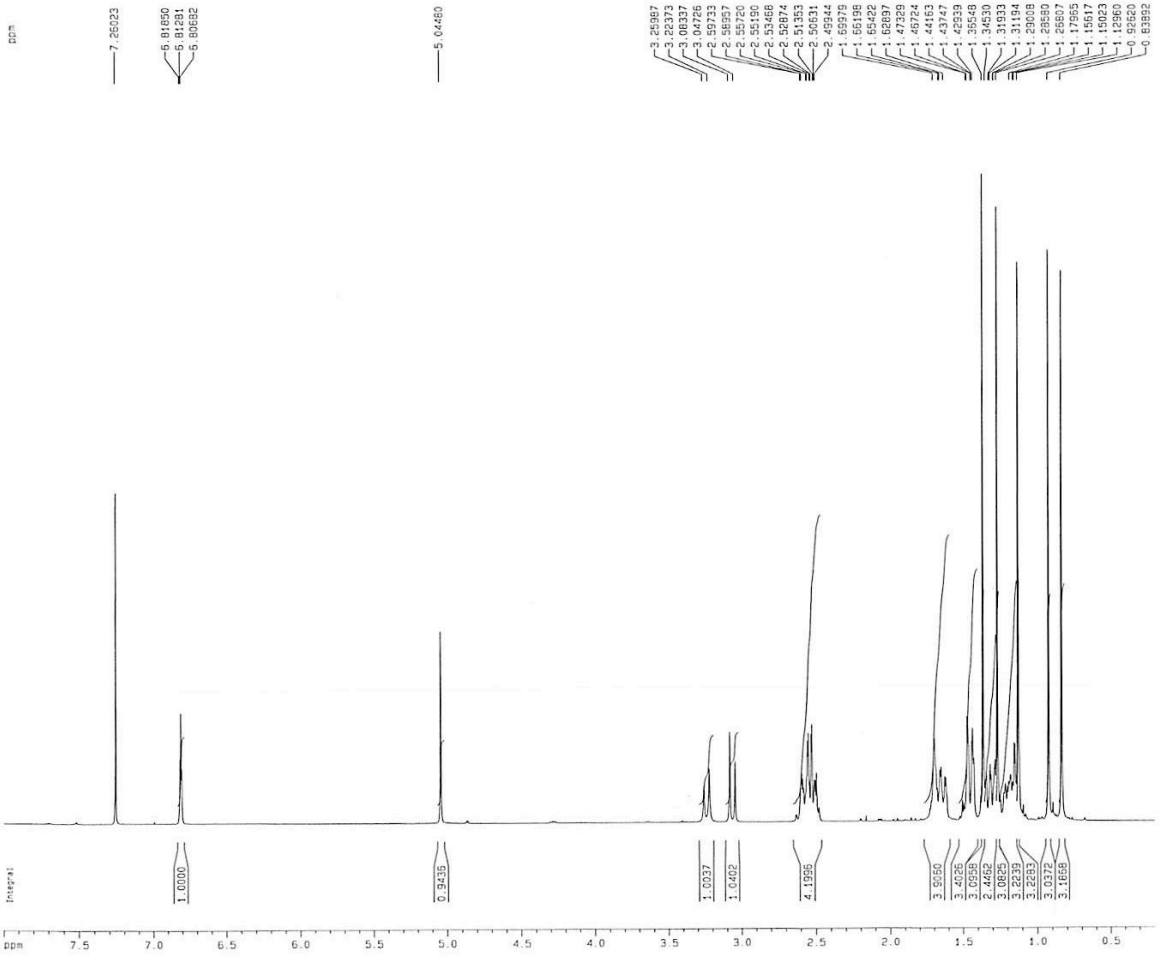

```
Current Data Parameters
NAME                 suzuki
EXPNO                   105
PROCNO                    1

F2 - Acquisition Parameters
Date_               20100827
Time                   20.38
INSTRUM                spect
PROBHD   5 mm QNP 1H/13
PULPROG                zg30
TD                    65536
SOLVENT                CDCl3
NS                        32
DS                         2
SWH                 8278.146 Hz
FIDRES              0.126314 Hz
AQ                  3.9584243 sec
RG                      203.2
DW                     60.400 usec
DE                      6.00 usec
TE                     300.2 K
D1                  1.00000000 sec
MCREST              0.00000000 sec
MCWRK               0.01500000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                      11.50 usec
PL1                     -4.00 dB
SFO1               400.0324703 MHz

F2 - Processing parameters
SI                       32768
SF                 400.0300062 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00

1D NMR plot parameters
CX                      32.00 cm
CY                      18.00 cm
F1P                     8.000 ppm
F1                    3200.24 Hz
F2P                     0.500 ppm
F2                     200.02 Hz
PPMCM                0.23437 ppm/cm
HZCM                93.75703 Hz/cm
```

DPX400 carbon



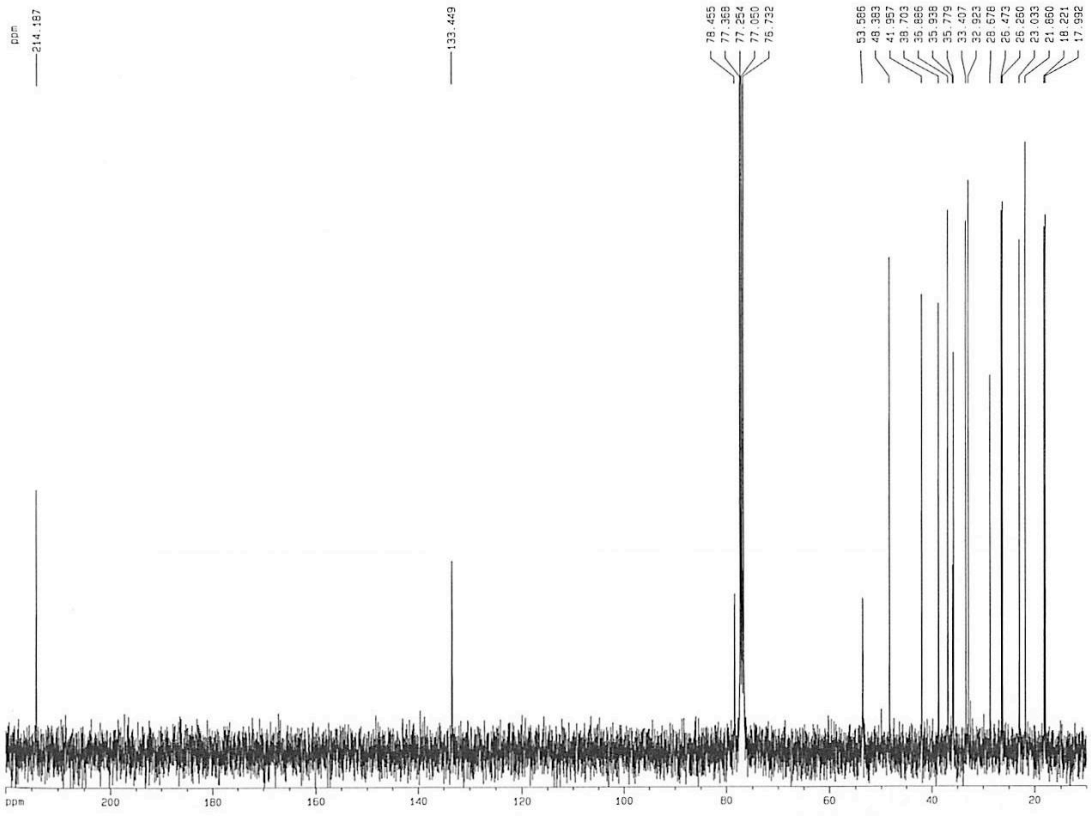

```
Current Data Parameters
NAME                 suzuki
EXPNO                   106
PROCNO                    1

F2 - Acquisition Parameters
Date_               20100827
Time                   20.46
INSTRUM                spect
PROBHD   5 mm QNP 1H/13
PULPROG              zgpg30
TD                    65536
SOLVENT                CDCl3
NS                       147
DS                         4
SWH                26178.010 Hz
FIDRES              0.399445 Hz
AQ                  1.2517875 sec
RG                      3251
DW                     19.100 usec
DE                      6.00 usec
TE                     300.2 K
D1                  2.00000000 sec
d11                 0.03000000 sec
DELTA               1.89999998 sec
MCREST              0.00000000 sec
MCWRK               0.01500000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                      10.00 usec
PL1                     -3.50 dB
SFO1               100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  100.00 usec
PL2                     -4.00 dB
PL12                    17.00 dB
PL13                    17.00 dB
SFO2               400.0316001 MHz

F2 - Processing parameters
SI                       32768
SF                 100.5876240 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40

1D NMR plot parameters
CX                      30.00 cm
CY                      17.00 cm
F1P                   220.000 ppm
F1                  22129.28 Hz
F2P                    10.000 ppm
F2                   1005.88 Hz
PPMCM                7.00000 ppm/cm
HZCM                704.11334 Hz/cm
```



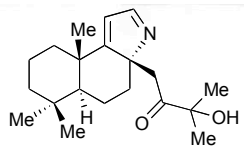


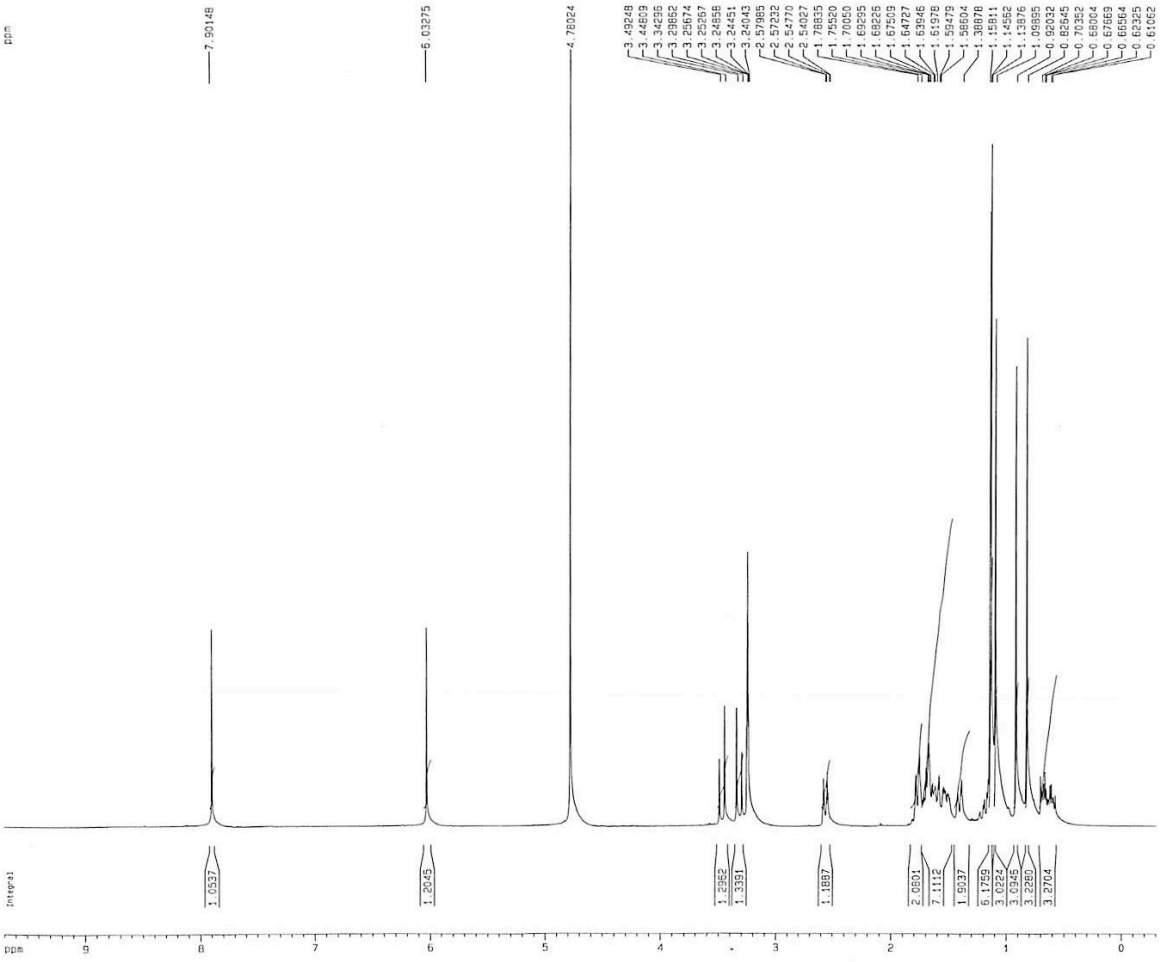

```
Current Data Parameters
NAME                 suzuki
EXPNO                   111
PROCNO                    1

F2 - Acquisition Parameters
Date_               20100912
Time                    1.49
INSTRUM                spect
PROBHD   5 mm QNP 1H/13
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       32
DS                        2
SWH                8278.146 Hz
FIDRES            0.126314 Hz
AQ                3.9584243 sec
RG                     287.4
DW                  60.400 usec
DE                   6.00 usec
TE                   300.2 K
D1                1.00000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                   11.50 usec
PL1                  -4.00 dB
SFO1           400.0324703 MHz

F2 - Processing parameters
SI                       32768
SF                400.0300065 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                       32.00 cm
CY                       18.00 cm
F1P                      6.000 ppm
F1                    3200.24 Hz
F2P                      0.200 ppm
F2                      80.01 Hz
PPMCM                 0.24375 ppm/cm
HZCM                 97.50731 Hz/cm
```

DPX400 carbon



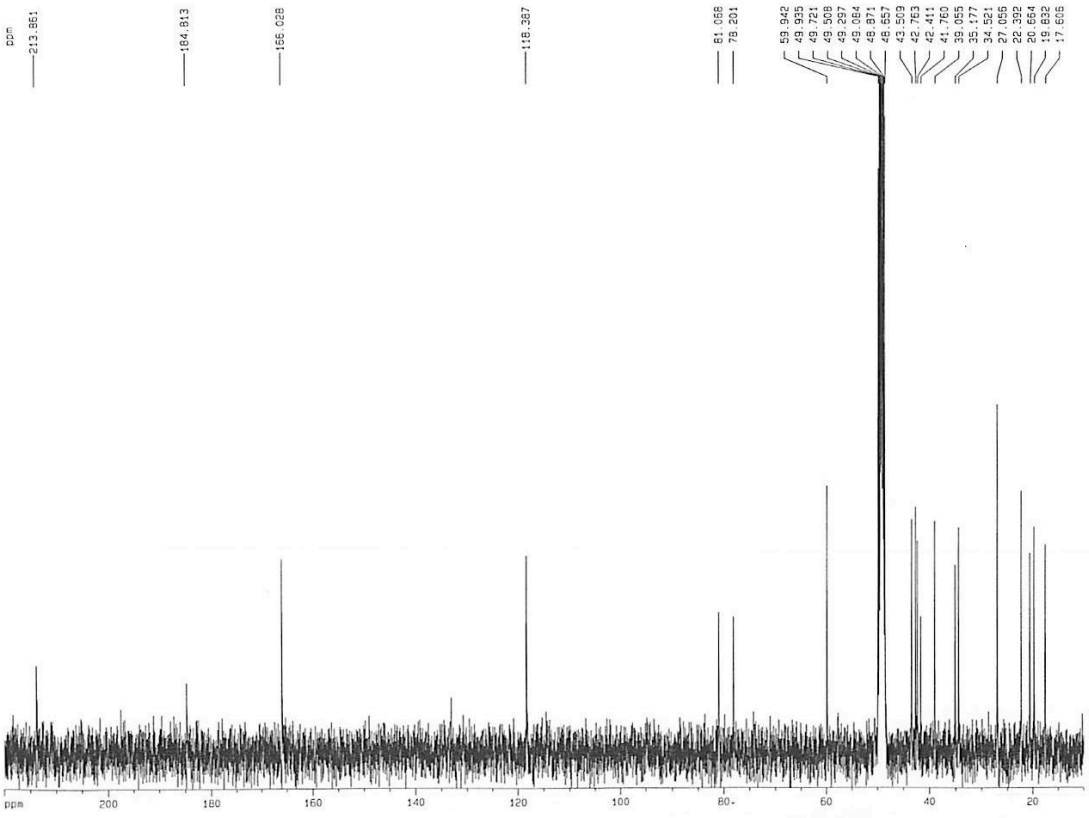

```
Current Data Parameters
NAME                 suzuki
EXPNO                   112
PROCNO                    1

F2 - Acquisition Parameters
Date_               20100912
Time                    1.55
INSTRUM                spect
PROBHD   5 mm QNP 1H/13
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      806
DS                        4
SWH               26178.010 Hz
FIDRES            0.399445 Hz
AQ                1.2517875 sec
RG                     6502
DW                  19.100 usec
DE                   6.00 usec
TE                   300.2 K
D1                2.00000000 sec
d11               0.03000000 sec
DELTA             1.89999998 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                   10.00 usec
PL1                  -3.50 dB
SFO1           100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
PCPD2                100.00 usec
PL2                  -4.00 dB
PL12                 17.00 dB
PL13                 17.00 dB
SFO2           400.0316001 MHz

F2 - Processing parameters
SI                       32768
SF                100.5876206 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40

1D NMR plot parameters
CX                       30.00 cm
CY                       17.00 cm
F1P                    220.000 ppm
F1                   22129.28 Hz
F2P                     10.000 ppm
F2                    1005.88 Hz
PPMCM                 7.00000 ppm/cm
HZCM                704.11334 Hz/cm
```






```
Current Data Parameters
NAME                 suzuki
EXPNO                   115
PROCNO                    1

F2 - Acquisition Parameters
Date_               20100916
Time                   20.15
INSTRUM                spect
PROBHD   5 mm GNP 1H/13
PULPROG                zg30
TD                    65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                8278.146 Hz
FIDRES             0.126314 Hz
AQ                 3.9584243 sec
RG                       256
DW                  60.400 usec
DE                   6.00 usec
TE                   300.2 K
D1                1.00000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                   11.50 usec
PL1                  -4.00 dB
SFO1            400.0324703 MHz

F2 - Processing parameters
SI                       32768
SF                400.0300295 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                       32.00 cm
CY                       21.00 cm
F1P                       9.700 ppm
F1                     3880.29 Hz
F2P                      -0.300 ppm
F2                     -120.01 Hz
PPMCM                 0.31250 ppm/cm
HZCM               125.00938 Hz/cm
```

DPX400 carbon




```
Current Data Parameters
NAME                 suzuki
EXPNO                   116
PROCNO                    1

F2 - Acquisition Parameters
Date_               20100916
Time                   20.18
INSTRUM                spect
PROBHD   5 mm GNP 1H/13
PULPROG              zgpg30
TD                    65536
SOLVENT                CDCl3
NS                       272
DS                         4
SWH               26178.010 Hz
FIDRES             0.399445 Hz
AQ                 1.2517875 sec
RG                    2896.3
DW                  19.100 usec
DE                   6.00 usec
TE                   300.2 K
D1                2.00000000 sec
d11               0.03000000 sec
DELTA             1.89999998 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                   10.00 usec
PL1                  -3.50 dB
SFO1            100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
PCPD2               100.00 usec
PL2                  -4.00 dB
PL12                 17.00 dB
PL13                 17.00 dB
SFO2            400.0316001 MHz

F2 - Processing parameters
SI                       32768
SF                100.5874520 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40

1D NMR plot parameters
CX                       30.00 cm
CY                       10.00 cm
F1P                     220.000 ppm
F1                   22129.24 Hz
F2P                     10.000 ppm
F2                    1005.87 Hz
PPMCM                 7.00000 ppm/cm
HZCM               704.11218 Hz/cm
```