

Supporting Information

Further Studies of Intramolecular Michael Reactions of Nitrosoalkenes for Construction of Functionalized Bridged Ring Systems

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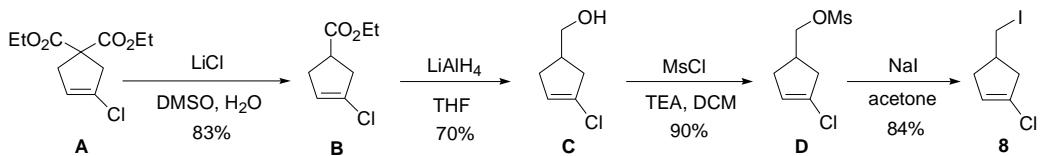
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Scheme 1. Preparation of Iodoalkene 8

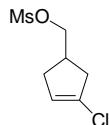


3-Chlorocyclopent-3-ene-carboxylic Acid Ethyl Ester (B). To a stirred solution of diester **A** (242 mg, 0.98 mmol) and water (0.05 mL) in DMSO (5 mL) was added LiCl (91 mg, 2.15 mmol). The reaction mixture was heated at reflux for 6 h, and then cooled to rt. Saturated aqueous NH₄Cl was added and the aqueous phase was extracted with ether. The combined organic layers were dried over Na₂SO₄. The solvent was removed under reduced pressure, and the residue was purified by flash chromatography (15% ether/pentane) to afford the ester **B** as a clear oil (143 mg, 83%). ¹H NMR (300 MHz, CDCl₃) δ 5.53-5.50 (m, 1H), 4.09 (q, *J* = 7.1 Hz, 2H), 3.22-3.11 (m, 1H), 2.86-2.78 (m, 1H), 2.72-2.65 (m, 1H), 2.63-2.58 (m, 2H), 1.19 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 175.0, 130.7, 124.9, 61.1, 41.4, 40.5, 35.2, 14.5; HRMS-EI [M]⁺ calcd for C₈H₁₁ClO₂, 174.0448; found, 174.0452.

(3-Chlorocyclopent-3-enyl)-methanol (C). To a stirred suspension of LiAlH₄ (56 mg, 1.47 mmol) in THF (5 mL) at 0 °C was added dropwise ester **B** (143 mg, 0.82 mmol) in ether (5 mL). The mixture was stirred for 1 h at rt, and then diluted with ethyl acetate. The mixture was poured into 1 M HCl solution. Saturated aqueous NH₄Cl and EtOAc were added. The organic layer was dried over Na₂SO₄ and the solvent was removed under reduced pressure. The residue was purified by flash chromatography (50% ether/pentane) to afford the alcohol **C** as a colorless oil (76 mg, 70%). ¹H NMR (300 MHz, CDCl₃) δ 5.54-5.51 (m, 1H), 3.50 (d, *J* = 6.3 Hz,

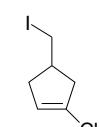
2H), 2.64-2.42 (m, 3H), 2.28-2.23 (m, 1H), 2.11-2.04 (m, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 131.5, 125.6, 66.8, 40.6, 39.2, 34.5; HRMS-EI $[\text{M}]^+$ calcd for $\text{C}_6\text{H}_9\text{ClO}$, 132.0342; found, 132.0337.

Methanesulfonic Acid 3-Chlorocyclopent-3-enyl Methyl Ester (D).



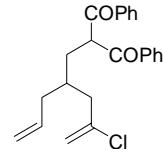
To a solution of alcohol **C** (71 mg, 0.53 mmol) in dichloromethane (5 mL) at 0 °C was added portionwise triethylamine (0.21 mL, 1.59 mmol) and mesyl chloride (0.11 mL, 1.59 mmol). The reaction mixture was stirred at 0 °C for 30 min, and then at rt for 3 h. The organic phase was diluted with dichloromethane and washed consecutively with brine, 1 M aqueous KHSO_4 , brine, 5% aqueous NaHCO_3 , brine, and dried over Na_2SO_4 . The solvent was removed under reduced pressure and the residue was purified by flash chromatography (50% ether/pentane) affording the mesylate **D** as a colorless oil (106 mg, 94%). ^1H NMR (300 MHz, CDCl_3) δ 5.55-5.52 (m, 1H), 4.08 (dd, $J = 2.2, 6.9$ Hz, 2H), 2.96 (s, 3H), 2.80-2.61 (m, 2H), 2.57-2.47 (m, 1H), 2.34-2.26 (m, 1H), 2.17-2.09 (m, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ 131.3, 125.4, 72.6, 40.4, 37.9, 36.5, 34.5; LRMS-ES $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_7\text{H}_{11}\text{ClNaO}_3\text{S}$, 233.0; found, 233.0.

1-Chloro-4-iodomethylcyclopentene (8). To a solution of mesylate **D**



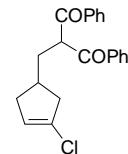
(108 mg, 0.51 mmol) in acetone (5 mL) was added sodium iodide (384 mg, 2.55 mmol) and the mixture was stirred at reflux for 12 h. The solvent was then removed under reduced pressure and the residue was purified by flash chromatography (pentane) to produce the iodide **8** as a clear oil (104 mg, 84%). ^1H NMR (300 MHz, CDCl_3) δ 5.48-5.46 (m, 1H), 3.09 (d, $J = 6.6$ Hz, 2H), 2.60-2.38 (m, 3H), 2.20-2.13 (m, 1H), 2.02-1.93 (m, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ 131.1, 125.6, 44.7, 39.9, 39.0, 13.6; HRMS-EI $[\text{M}]^+$ calcd for $\text{C}_6\text{H}_8\text{ClI}$, 241.9359; found, 241.9368.

2-(2-Allyl-4-chloropent-4-enyl)-1,3-diphenylpropane-1,3-dione



(15a). To a stirred solution of 1,3-Diphenyl-propane-1,3-dione (238 mg, 1.06 mmol) in acetonitrile (10 mL) was added Verkade's base (230 mg, 1.06 mmol), and the mixture was stirred for 30 min at rt. Iodide **14** (261 mg, 0.96 mmol) was then added and the mixture was stirred for 72 h at rt. The solvent was removed under reduced pressure, and the residue was purified by flash chromatography (13% ether/hexanes) to afford the diene **15a** as a pale yellow oil (182 mg, 51%). ¹H NMR (300 MHz, CDCl₃) δ 7.82-7.75 (m, 4H), 7.36-7.20 (m, 6H), 5.61-5.48 (m, 1H), 5.17 (t, *J* = 6.4 Hz, 1H), 4.98-4.83 (m, 4H), 2.22-2.03 (m, 2H), 2.01-1.80 (m, 5H); ¹³C NMR (75 MHz, CDCl₃) δ 196.3, 196.2, 141.5, 136.4, 136.3, 135.9, 133.9, 132.9, 129.3, 129.1, 129.0, 127.6, 117.9, 114.7, 55.8, 44.3, 38.1, 34.4, 33.2; HRMS-ES [M + H]⁺ calcd for C₂₃H₂₄ClO₂, 367.1465; found, 367.1470.

2-(3-Chlorocyclopent-3-enylmethyl)-1,3-diphenylpropane-1,3-dione (16a).

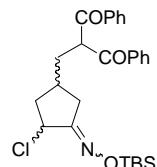
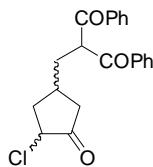


A flame dried 250 mL two-necked flask equipped with a magnetic stirring bar and a condenser was charged with diene **15a** (172 mg, 0.47 mmol) and benzene (120 mL). The solution was deaerated by bubbling argon through the mixture for 2 h. The second-generation Grubbs catalyst (40 mg, 0.047 mmol) in 2 mL of benzene was added and the argon bubbling was continued for an additional 30 min. The mixture was heated and stirred at 65 °C for 2-3 days until TLC showed the reaction was complete. The solvent was removed under reduced pressure and the residue was purified by flash chromatography (17% ether/hexanes) to afford the vinyl chloride **16a** as a yellow oil (100 mg, 63%). ¹H NMR (300 MHz, CDCl₃) δ 7.93-7.87 (m, 4H), 7.53-7.47 (m, 2H), 7.41-7.36 (m, 4H), 5.52-5.50 (m, 1H), 5.14 (t, *J* = 6.7 Hz, 1H), 2.62-

2.01 (m, 7H); ^{13}C NMR (75 MHz, CDCl_3) δ 196.0, 136.3, 136.3, 134.0, 131.5, 129.3, 129.0, 125.8, 56.3, 43.8, 38.1, 36.3, 36.1; HRMS-ES $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{21}\text{H}_{20}\text{ClO}_2$, 339.1152; found, 339.1177.

2-(3-Chloro-4-oxocyclopentylmethyl)-1,3-diphenylpropane-1,3-dione (17a). To a solution of vinyl chloride **16a** (92 mg, 0.27 mmol), acetone (2.5 mL) and glacial acetic acid (1 mL) at 0 °C was added dropwise sodium hypochlorite (0.23 mL of 10% solution, 0.27 mmol) via syringe. The reaction mixture was stirred at 0 °C for 30 min and quenched by addition of saturated aqueous NaHCO_3 solution. The mixture was then extracted with dichloromethane. The combined organic layers were washed with brine and dried over Na_2SO_4 . The solvent was removed under reduced pressure and the residue was purified by flash chromatography (50% ether/hexanes) affording the α -chloroketone **17a** as a white solid (38 mg, 40%). ^1H NMR (300 MHz, CDCl_3) δ 7.88 (d, $J = 7.7$ Hz, 4H), 7.54-7.36 (m, 6H), 5.15 (t, $J = 6.5$ Hz, 1H), 4.06 (d, $J = 5.6$ Hz, 1H), 2.70-2.53 (m, 1H), 2.33-1.78 (m, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 209.7, 195.7, 195.6, 136.0, 135.9, 134.3, 134.2, 129.5, 129.4, 129.4, 129.0, 129.0, 57.5, 56.3, 42.7, 40.2, 34.6, 33.0; HRMS-ES $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{21}\text{H}_{20}\text{ClO}_3$, 355.1101; found, 355.1096.

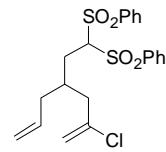
2-(3-Chloro-*tert*-butyldimethylsilyloxyiminocyclopentylmethyl)-1,3-diphenylpropane-1,3-dione (18a). To a solution of α -chloroketone **17a** (23 mg, 0.065 mmol) in dichloromethane (3 mL) were added *O*-(*tert*-butyldimethylsilyl)-hydroxylamine (10 mg, 0.065 mmol), 4 Å molecular sieves (crushed), and a catalytic amount of PPTS. The mixture was stirred at rt for 24 h and then filtered through a pad of Celite. The solvent was removed under reduced pressure and the



residue was purified by flash chromatography (25% ether/hexanes) to afford the α -chloroketoxime **18a** as a colorless oil (19 mg, 61%). ^1H NMR (300 MHz, CDCl_3 , complex mixture of diastereomers including oxime geometric isomers) δ 7.84-7.76 (m, 4H), 7.46-7.27 (m, 6H), 5.08 (q, $J = 6.5$ Hz, 1H), 4.84-4.61 (m, 1H), 2.78-2.64 (m, 1H), 2.53-2.39 (m, 1H), 2.28-1.88 (m, 4H), 1.72-1.55 (m, 1H), 0.82-0.68 (m, 9H), 0.02-0.00 (m, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 196.0, 195.8, 195.8, 195.6, 167.3, 166.1, 136.2, 136.2, 136.1, 136.0, 134.1, 134.1, 129.4, 129.4, 129.0, 129.0, 128.9, 59.1, 56.8, 56.4, 51.5, 43.0, 42.6, 35.4, 35.2, 34.6, 34.6, 34.2, 32.8, 26.3, 26.3, 18.4, 18.4, -4.8, -4.8; HRMS-ES [M + H] $^+$ calcd for $\text{C}_{27}\text{H}_{35}\text{ClNO}_3\text{Si}$, 484.2075; found, 484.2066.

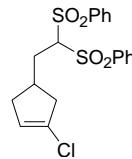
2-(2-Allyl-4-chloropent-4-enyl)-1,1-bis-benzenesulfonylmethane

(15b). To a stirred suspension of sodium hydride (60% dispersion in mineral oil, 18 mg, 0.44 mmol) in DMF (1 mL) at 0 °C was dropwise added a solution of bis-benzenesulfonyl methane (137 mg, 0.44 mmol) in DMF (1 mL). After stirring the mixture for 30 min at 0 °C, a solution of iodide **14** (100 mg, 0.37 mmol) in DMF (1 mL) was added. The resulting mixture was warmed to rt and stirred for 72 h. Saturated aqueous NH_4Cl and ethyl acetate were added. The organic layer was separated and the aqueous layer was extracted with ethyl acetate. The combined organic layer was washed with water and brine. The organic layer was dried over Na_2SO_4 and the solvent was removed under reduced pressure. The residue was purified by flash chromatography (25% ethyl acetate/hexanes) to afford the diene **15b** as a colorless oil (113 mg, 70%). ^1H NMR (300 MHz, CDCl_3) δ 7.90-7.84 (m, 4H), 7.63-7.55 (m, 2H), 7.48 (t, $J = 7.1$ Hz, 4H), 5.62-5.48 (m, 1H), 5.08 (d, $J = 18.9$ Hz, 2H), 4.99-4.92 (m, 2H), 4.54 (t, $J = 5.3$ Hz, 1H), 2.26-1.88 (m, 7H); ^{13}C NMR (75 MHz, CDCl_3) δ 140.6, 138.3, 137.6, 135.1, 135.0,

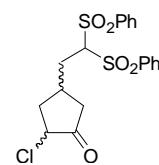


130.2, 129.9, 129.5, 129.5, 118.6, 115.3, 81.7, 43.9, 37.5, 33.5, 29.6; HRMS-ES [M + NH₄]⁺ calcd for C₂₁H₂₇ClNO₄S₂, 456.1070; found, 456.1067.

4-(2,2-Bis-benzenesulfonylethyl)-2-chlorocyclopentene (16b). A flame dried 100 mL two-necked flask equipped with a magnetic stirring bar and a condenser was charged with diene **15b** (129 mg, 0.29 mmol) and benzene (60 mL). The solution was deaerated by bubbling argon through the mixture for 2 h. The second-generation Grubbs catalyst (25 mg, 0.029 mmol) in 2 mL of benzene was added and the argon bubbling was continued for an additional 30 min. The mixture was heated and stirred at 65 °C for 2-3 days until TLC showed the reaction was complete. The solvent was removed under reduced pressure and the residue was purified by flash chromatography (25% ethyl acetate/hexanes) to afford the vinyl chloride **16b** as a pale yellow oil (61 mg, 51%). ¹H NMR (300 MHz, CDCl₃) δ 7.93-7.79 (m, 4H), 7.67-7.57 (m, 2H), 7.51 (t, *J* = 7.6 Hz, 4H), 5.47-5.44 (m, 1H), 4.25 (t, *J* = 5.6 Hz, 1H), 2.73-2.39 (m, 2H), 2.20-2.11 (m, 3H), 2.02-1.81 (m, 2H); ¹³C NMR (75 MHz, CDCl₃) δ 138.0, 137.9, 135.2, 135.1, 131.1, 130.0, 130.0, 129.6, 129.6, 129.5, 129.4, 125.3, 82.8, 43.1, 37.3, 35.7, 32.0; HRMS-ES [M + NH₄]⁺ calcd for C₁₉H₂₃ClNO₄S₂, 428.0757; found, 428.0740.

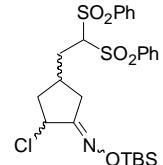


4-(2,2-Bis-benzenesulfonylethyl)-2-chlorocyclopentanone (17b). To a solution of vinyl chloride **16b** (51 mg, 0.124 mmol), acetone (2.5 mL) and glacial acetic acid (1 mL) at 0 °C was added dropwise sodium hypochlorite (0.11 mL of 10% solution, 0.124 mmol) via syringe. The reaction mixture was stirred at 0 °C for 30 min and quenched by addition of saturated aqueous NaHCO₃ solution. The mixture was then extracted with dichloromethane. The combined organic layers were washed with brine and dried over Na₂SO₄. The solvent was removed under

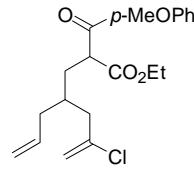


reduced pressure and the residue was purified by flash chromatography (30% ethyl acetate/hexanes) affording the α -chloroketone **17b** as a colorless oil (21 mg, 40%). ^1H NMR (300 MHz, CDCl_3 , diastereomer mixture) δ 8.03-7.83 (m, 4H), 7.72-7.62 (m, 2H), 7.56-7.51 (m, 4H), 4.30 (t, $J = 5.5$ Hz, 1H), 4.02 (d, $J = 6.9$ Hz, 1H), 2.76-2.00 (m, 7H); ^{13}C NMR (75 MHz, CDCl_3) δ 209.0, 208.5, 137.7, 137.7, 136.0, 135.9, 135.4, 135.1, 132.3, 132.2, 130.1, 130.0, 129.9, 129.7, 129.6, 129.4, 129.3, 82.3, 82.2, 57.2, 56.6, 43.8, 41.7, 41.1, 39.4, 32.5, 31.0, 30.7; HRMS-ES $[\text{M} + \text{NH}_4]^+$ calcd for $\text{C}_{19}\text{H}_{23}\text{ClNO}_5\text{S}_2$, 444.0706; found, 444.0699.

4-(2,2-Bis-benzenesulfonylethyl)-2-chlorocyclopentanone-*tert*-butyldimethylsilyloxyoxime (18b). To a solution of α -chloroketone **17b** (19.7 mg, 0.046 mmol) in dichloromethane (3 mL) were added *O*-(*tert*-butyldimethylsilyl)-hydroxylamine (15 mg, 0.092 mmol), 4 \AA molecular sieves (crushed), and a catalytic amount of PPTS. The mixture was stirred at rt for 19 h and then filtered through a pad of Celite. The solvent was removed under reduced pressure and the residue was purified by flash chromatography (25% ethyl acetate/hexanes) to afford the α -chloroketoxime **18b** as a colorless oil (15.6 mg, 61%). ^1H NMR (300 MHz, CDCl_3 , complex mixture of diastereomers including oxime geometric isomers) δ 7.92-7.77 (m, 4H), 7.62-7.54 (m, 2H), 7.44 (t, $J = 7.4$ Hz, 4H), 4.88-4.44 (m, 1H), 4.23-4.17 (m, 1H), 2.84-2.40 (m, 2H), 2.20-1.71 (m, 3H), 1.52-1.40 (m, 2H), 0.79-0.67 (m, 9H), 0.01-0.00 (m, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 166.3, 165.2, 137.9, 137.9, 137.8, 135.8, 135.2, 132.4, 132.2, 130.0, 130.0, 130.0, 129.7, 129.4, 129.2, 82.7, 58.7, 51.1, 42.4, 41.9, 34.7, 34.5, 34.2, 34.1, 32.2, 32.0, 30.9, 30.6, 30.1, 26.4, 26.3, 18.5, 18.4, 18.3, -4.8.



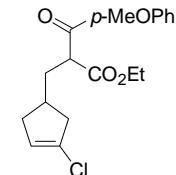
4-Allyl-6-chloro-2-(4-methoxybenzoyl)-hept-6-enoic Acid



Ethyl Ester (15c). To a stirred solution of 3-(4-methoxyphenyl)-3-oxo-propionic acid ethyl ester (57 mg, 0.26 mmol) in acetonitrile (3 mL) was

added Verkade's base (56 mg, 0.26 mmol), and the mixture was stirred for 30 min at 0 °C. Iodide **14** (70 mg, 0.26 mmol) was then added and the mixture was stirred for 7 h at 0 °C. The solvent was removed under reduced pressure, and the residue was purified by flash chromatography (20% ether/hexanes) to afford the diene **15c** as a colorless oil (65 mg, 68%). ¹H NMR (300 MHz, CDCl₃, diastereomer mixture) δ 7.99-7.95 (m, 2H), 6.92 (d, *J* = 8.4 Hz, 2H), 5.81-5.65 (m, 1H), 5.19-5.12 (m, 2H), 5.08-5.00 (m, 2H), 4.43-4.38 (m, 1H), 4.12 (q, *J* = 7.1 Hz, 2H), 3.84 (s, 3H), 2.39-1.83 (m, 7H), 1.15 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 193.9, 193.8, 170.7, 170.6, 164.5, 141.7, 141.6, 136.0, 131.6, 129.7, 118.1, 118.0, 114.8, 114.5, 62.0, 62.0, 56.1, 52.5, 52.4, 44.4, 44.2, 38.0, 37.8, 33.9, 33.7, 33.0, 32.8, 14.6; HRMS-ES [M + H]⁺ calcd for C₂₀H₂₆ClO₄, 365.1520; found, 365.1524.

2-(3-Chlorocyclopent-3-enylmethyl)-3-(4-methoxyphenyl)-3-oxopropionic Acid Ethyl Ester (16c). A flame dried 250 mL two-necked flask equipped with a magnetic stirring bar and a condenser was charged

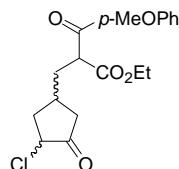
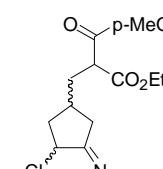


with diene **15c** (300 mg, 0.82 mmol) and benzene (200 mL). The solution was deaerated by bubbling argon through the mixture for 2 h. The second-generation Grubbs catalyst (70 mg, 0.082 mmol) in 2 mL of benzene was added and the argon bubbling was continued for an additional 30 min. The mixture was heated and stirred at 65 °C for 72 h. The solvent was removed under reduced pressure and the residue was purified by flash chromatography (30% ether/hexanes) to afford the vinyl chloride **16c** as a pale yellow oil

(216 mg, 78%). ^1H NMR (300 MHz, CDCl_3 , diastereomer mixture) δ 7.92-7.88 (m, 2H), 6.90-6.86 (m, 2H), 5.51-5.50 (m, 1H), 4.23-4.17 (m, 1H), 4.07 (q, $J = 7.1$ Hz, 2H), 3.81 (s, 3H), 2.55-1.97 (m, 7H), 1.10 (t, $J = 7.1$ Hz, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 193.2, 193.2, 170.0, 164.0, 131.2, 131.1, 129.3, 129.2, 125.5, 125.5, 114.1, 61.5, 55.6, 52.6, 52.5, 43.4, 37.7, 37.7, 35.6, 35.3, 35.3, 14.1; HRMS-ES $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{18}\text{H}_{22}\text{ClO}_4$, 337.1207; found, 337.1208.

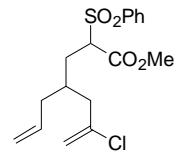
2-(3-Chloro-4-oxocyclopentylmethyl)-3-(4-methoxyphenyl)-3-oxopropionic Acid Ethyl Ester (17c). To a solution of vinyl chloride **16c** (168 mg, 0.49 mmol), acetone (2.5 mL) and glacial acetic acid (1 mL) at 0°C was added dropwise sodium hypochlorite (0.42 mL of 10% solution, 0.49 mmol) via syringe. The reaction mixture was stirred at 0°C for 30 min and quenched by addition of saturated aqueous NaHCO_3 solution. The mixture was then extracted with dichloromethane. The combined organic layers were washed with brine and dried over Na_2SO_4 . The solvent was removed under reduced pressure and the residue was purified by flash chromatography (60% ether/hexanes) affording the α -chloroketone **17c** as a colorless oil (101 mg, 57%). ^1H NMR (300 MHz, CDCl_3 , diastereomer mixture) δ 7.98-7.85 (m, 2H), 6.92-6.86 (m, 2H), 4.32-4.20 (m, 1H), 4.16-3.96 (m, 3H), 3.81 (s, 3H), 2.75-1.83 (m, 7H), 1.18-1.08 (m, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 209.9, 209.9, 193.0, 192.9, 170.1, 164.5, 131.4, 129.3, 129.2, 114.5, 114.4, 62.1, 57.5, 56.0, 52.8, 52.7, 42.5, 42.5, 40.1, 40.1, 34.4, 32.4, 32.4, 14.4; HRMS-ES $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{18}\text{H}_{22}\text{ClO}_5$, 353.1156; found, 353.1151.

2-(3-Chloro-4-*tert*-butyldimethylsilyloxyiminocyclopentylmethyl)-3-(4-methoxyphenyl)-3-oxopropionic Acid Ethyl Ester (18c). To a solution

of α -chloroketone **17c** (15.5 mg, 0.044 mmol) in dichloromethane (3 mL) were added *O*-(*tert*-butyldimethylsilyl)-hydroxylamine (6.5 mg, 0.044 mmol), 4 \AA molecular sieves (crushed), and a catalytic amount of PPTS. The mixture was stirred at rt for 5 h and then filtered through a pad of Celite. The solvent was removed under reduced pressure and the residue was purified by flash chromatography (50% ether/hexanes) to afford the α -chloroketoxime **18c** as a colorless oil (10.5 mg, 50%). ^1H NMR (300 MHz, CDCl_3 , complex mixture of diastereomers including oxime geometric isomers) δ 7.86-7.82 (m, 2H), 6.82-6.75 (m, 2H), 4.84-4.61 (m, 1H), 4.18-4.11 (m, 1H), 4.04-3.95 (m, 2H), 3.73 (s, 3H), 2.78-2.58 (m, 1H), 2.37-2.32 (m, 1H), 2.13-1.81 (m, 4H), 1.65-1.52 (m, 1H), 1.06-1.01 (m, 3H), 0.82-0.70 (m, 9H), 0.01-0.00 (m, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 193.3, 193.1, 193.1, 170.2, 170.2, 170.1, 167.5, 167.5, 166.2, 164.3, 164.3, 131.4, 129.4, 129.3, 129.3, 114.3, 61.9, 59.2, 55.9, 53.2, 53.1, 51.5, 42.9, 42.9, 35.2, 34.2, 33.9, 33.9, 32.6, 26.6, 26.3, 26.3, 26.0, 18.4, 18.4, 14.4, -4.8, -4.9; HRMS-ES [M + H] $^+$ calcd for $\text{C}_{24}\text{H}_{37}\text{ClNO}_5\text{Si}$, 482.2130; found, 482.2110.

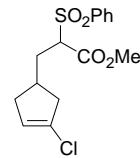
4-Allyl-2-benzenesulfonyl-6-chlorohept-6-enoic Acid Methyl Ester (15d). To a stirred suspension of sodium hydride (60% dispersion in mineral oil, 70 mg, 1.73 mmol) in DMF (8 mL) at 0 °C was dropwise added a solution of benzenesulfonyl acetic acid methyl ester (0.27 mL, 1.44 mmol) in DMF (2 mL). After stirring the mixture for 30 min at 0 °C, a solution of iodide **14** (468 mg, 1.73 mmol) in DMF (1 mL) was added. The resulting mixture was warmed to rt and stirred for 24 h. Saturated aqueous NH_4Cl and ethyl acetate were added. The organic layer was separated and the aqueous layer was extracted with ethyl acetate. The combined organic layer was washed with water and brine. The organic layer was dried



over Na_2SO_4 and the solvent was removed under reduced pressure. The residue was purified by flash chromatography (20% ethyl acetate/hexanes) to afford the diene **15d** as a colorless oil (420 mg, 77%). ^1H NMR (300 MHz, CDCl_3 , diastereomer mixture) δ 7.85 (d, $J = 7.6$ Hz, 2H), 7.68 (t, $J = 7.3$ Hz, 1H), 7.56 (t, $J = 7.6$ Hz, 2H), 5.70-5.54 (m, 1H), 5.13 (d, $J = 18.3$ Hz, 2H), 5.04-4.93 (m, 2H), 4.12-4.06 (m, 1H), 3.64 (d, $J = 6.3$ Hz, 3H), 2.38-1.82 (m, 7H); ^{13}C NMR (75 MHz, CDCl_3) δ 165.1, 164.9, 139.1, 138.9, 135.9, 135.8, 133.4, 133.2, 132.9, 128.0, 127.9, 117.0, 116.7, 113.6, 113.5, 67.6, 51.8, 51.8, 42.3, 41.7, 36.3, 34.9, 31.8, 31.4, 28.9, 28.3; HRMS-ES $[\text{M} + \text{NH}_4]^+$ calcd for $\text{C}_{17}\text{H}_{25}\text{ClNO}_4\text{S}$, 374.1193; found, 374.1188.

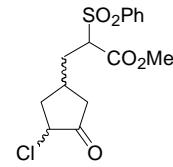
2-Benzenesulfonyl-3-(3-chlorocyclopent-3-enyl)-propionic Acid

Methyl Ester (16d). A flame dried 250 mL two-necked flask equipped with a magnetic stirring bar and a condenser was charged with diene **15d** (148 mg, 0.41 mmol) and toluene (100 mL). The solution was deaerated by bubbling argon through the mixture for 2 h. The second-generation Grubbs catalyst (35 mg, 0.041 mmol) in 2 mL of toluene was added and the argon bubbling was continued for an additional 30 min. The mixture was heated and stirred at 85 °C for 20 h. The solvent was removed under reduced pressure and the residue was purified by flash chromatography (25% ethyl acetate/hexanes) to afford the vinyl chloride **16d** as a pale yellow oil (100 mg, 74%). ^1H NMR (300 MHz, CDCl_3 , diastereomer mixture) δ 7.87 (d, $J = 7.7$ Hz, 2H), 7.70 (t, $J = 6.8$ Hz, 1H), 7.58 (t, $J = 7.6$ Hz, 2H), 5.57-5.55 (m, 1H), 4.06-3.90 (m, 1H), 3.66 (s, 3H), 2.62-1.91 (m, 7H); ^{13}C NMR (75 MHz, CDCl_3) δ 166.4, 136.8, 136.8, 134.4, 131.1, 130.7, 129.3, 129.3, 129.1, 125.3, 124.9, 69.7, 69.6, 53.1, 43.2, 42.5, 37.6, 36.8, 35.1, 32.7; HRMS-ES $[\text{M} + \text{NH}_4]^+$ calcd for $\text{C}_{15}\text{H}_{21}\text{ClNO}_4\text{S}$, 346.0880; found, 346.0889.



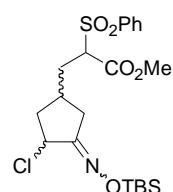
2-Benzenesulfonyl-3-(3-chloro-4-oxocyclopentyl)-propionic Acid Methyl Ester (17d).

To a solution of vinyl chloride **16d** (64 mg, 0.19 mmol), acetone (2.5 mL) and glacial acetic acid (1 mL) at 0 °C was added dropwise sodium hypochlorite (0.17 mL of 10% solution, 0.19 mmol) via syringe. The reaction mixture was stirred at 0 °C for 30 min and quenched by addition of saturated aqueous NaHCO₃ solution. The mixture was then extracted with dichloromethane. The combined organic layers were washed with brine and dried over Na₂SO₄. The solvent was removed under reduced pressure and the residue was purified by flash chromatography (25% ethyl acetate/hexanes) affording the α-chloroketone **17d** as a colorless oil (50 mg, 75%). ¹H NMR (300 MHz, CDCl₃, diastereomer mixture) δ 7.80 (d, *J* = 7.4 Hz, 2H), 7.65 (t, *J* = 7.4 Hz, 1H), 7.53 (t, *J* = 7.6 Hz, 2H), 4.05 (d, *J* = 5.9 Hz, 1H), 3.97-3.85 (m, 1H), 3.59 (s, 3H), 2.59-1.76 (m, 7H); ¹³C NMR (75 MHz, CDCl₃) δ 208.8, 208.5, 166.5, 166.5, 137.0, 136.9, 135.0, 129.7, 129.6, 69.8, 69.7, 57.3, 57.0, 53.7, 53.6, 53.5, 42.0, 41.7, 39.9, 39.6, 32.1, 32.0, 32.0, 31.9; HRMS-ES [M + NH₄]⁺ calcd for C₁₅H₂₁ClNO₅S, 362.0829; found, 362.0828.



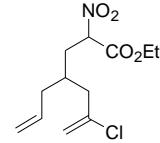
2-Benzenesulfonyl-3-(3-chloro-4-*tert*-butyldimethylsilyloxyiminocyclopentyl)-propionic Acid Methyl Ester (18d).

To a solution of α-chloroketone **17d** (30 mg, 0.087 mmol) in dichloromethane (3 mL) were added *O*-(*tert*-butyldimethylsilyl)-hydroxylamine (27 mg, 0.174 mmol), 4 Å molecular sieves (crushed), and a catalytic amount of PPTS. The mixture was stirred at rt for 24 h and then filtered through a pad of Celite. The solvent was removed under reduced pressure and the residue was purified by flash chromatography (20% ethyl acetate/hexanes) to afford the α-chloroketoxime **18d** as a

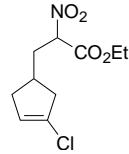


colorless oil (28 mg, 68%). ^1H NMR (300 MHz, CDCl_3 , complex mixture of diastereomers including oxime geometric isomers) δ 7.72 (d, $J = 7.7$ Hz, 2H), 7.56 (t, $J = 7.4$ Hz, 1H), 7.44 (t, $J = 7.6$ Hz, 2H), 4.83-4.61 (m, 1H), 3.98-3.77 (m, 1H), 3.53 (s, 3H), 2.73-2.51 (m, 1H), 2.39-2.19 (m, 1H), 2.09-1.81 (m, 4H), 1.59-1.51 (m, 1H), 0.84-0.67 (m, 9H), 0.01-0.00 (m, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 165.4, 165.4, 165.3, 165.2, 164.9, 135.7, 135.6, 133.5, 128.4, 128.3, 128.3, 128.2, 128.2, 68.8, 68.8, 57.6, 57.3, 52.3, 52.2, 41.4, 41.1, 33.5, 32.2, 32.1, 30.9, 30.7, 30.6, 30.2, 25.0, 24.9, 24.9, 17.1, 17.0, 17.0, -6.2; HRMS-ES $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{21}\text{H}_{33}\text{ClNO}_5\text{SSI}$, 474.1537; found, 474.1553.

4-Allyl-6-chloro-2-nitrohept-6-enoic Acid Ethyl Ester (15e). To a stirred solution of tetrabutyl ammonium hydroxide (1.71 g, 2.64 mmol) in dichloromethane (2.5 mL) was slowly added nitroacetic acid ethyl ester (0.3 mL, 2.64 mmol), and the mixture was stirred at rt for 10 min. A solution of iodide **14** (783 mg, 2.90 mmol) in dichloromethane (0.5 mL) was then added in one portion and the mixture was stirred at rt for 72 h. The organic layer was separated and the aqueous layer was extracted with dichloromethane. The combined organic layer was washed with brine and dried over MgSO_4 . The solvent was removed under reduced pressure, and the residue was purified by flash chromatography (7% ethyl acetate/hexanes) to afford the diene **15e** as an oil (217 mg, 30%). ^1H NMR (300 MHz, CDCl_3 , diastereomer mixture) δ 5.72-5.60 (m, 1H), 5.23-4.93 (m, 5H), 4.22 (q, $J = 7.1$ Hz, 2H), 2.39-1.95 (m, 6H), 1.91-1.81 (m, 1H), 1.24 (t, $J = 7.1$ Hz, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 164.9, 164.9, 140.5, 140.4, 134.8, 134.6, 118.8, 118.6, 115.4, 115.2, 86.6, 86.5, 63.5, 43.7, 43.5, 37.6, 36.8, 33.8, 33.7, 32.2, 32.0, 14.2; HRMS-ES $[\text{M}+\text{NH}_4]^+$ calcd for $\text{C}_{12}\text{H}_{22}\text{ClN}_2\text{O}_4$, 293.1268; found, 293.1281.

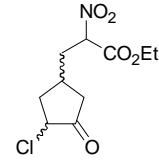


3-(3-Chlorocyclopent-3-enyl)-2-nitropropionic Acid Ethyl Ester



(16e). A flame dried 100 mL two-necked flask equipped with a magnetic stirring bar and a condenser was charged with diene **15e** (70 mg, 0.25 mmol) and toluene (60 mL). The solution was deaerated by bubbling argon through the mixture for 2 h. The second-generation Grubbs catalyst (44 mg, 0.05 mmol) in 2 mL of toluene was added and the argon bubbling was continued for an additional 30 min. The mixture was heated and stirred at 95 °C for 19 h. The solvent was removed under reduced pressure and the residue was purified by flash chromatography (9% ethyl acetate/hexanes) to afford the vinyl chloride **16e** as a pale yellow oil (37 mg, 59%). ¹H NMR (300 MHz, CDCl₃, diastereomer mixture) δ 5.62-5.53 (m, 1H), 5.12-5.01 (m, 1H), 4.22 (q, *J* = 7.1 Hz, 2H), 2.54-1.97 (m, 7H), 1.24 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 164.8, 164.8, 131.6, 131.2, 125.8, 125.4, 87.5, 87.3, 63.7, 63.6, 43.6, 43.1, 37.9, 37.4, 36.8, 36.7, 34.4, 34.4, 14.4.

3-(3-Chloro-4-oxocyclopentyl)-2-nitropropionic Acid Ethyl Ester

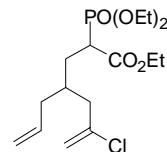
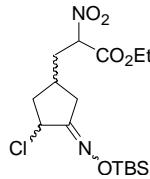


(17e). To a solution of vinyl chloride **16e** (32 mg, 0.13 mmol), acetone (2.5 mL) and glacial acetic acid (1 mL) at 0 °C was added dropwise sodium hypochlorite (0.12 mL of 10% solution, 0.13 mmol) via syringe. The reaction mixture was stirred at 0 °C for 20 min and quenched by addition of saturated aqueous NaHCO₃ solution. The mixture was then extracted with dichloromethane. The combined organic layers were washed with brine and dried over Na₂SO₄. The solvent was removed under reduced pressure and the residue was purified by flash chromatography (25% ethyl acetate/hexanes) affording the α-chloroketone **17e** as a colorless oil (14 mg, 41%). ¹H NMR (300 MHz, CDCl₃, diastereomer mixture) δ 5.12-5.06 (m, 1H), 4.36-4.19 (m, 2H),

4.08 (d, $J = 5.5$ Hz, 1H), 2.74-1.82 (m, 7H), 1.25 (t, $J = 7.1$ Hz, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 208.4, 208.3, 164.3, 87.0, 86.7, 63.9, 63.8, 57.1, 56.9, 41.8, 41.6, 39.7, 39.5, 35.6, 35.5, 31.0, 14.5, 14.3; HRMS-ES [M + NH₄]⁺ calcd for C₁₀H₁₈ClN₂O₅, 281.0904; found, 281.0893.

3-(3-Chloro-4-*tert*-butyldimethylsilyloxyiminocyclopentyl)-2-nitropropionic Acid Ethyl Ester (18e). To a solution of α -chloroketone **17e** (12.0 mg, 0.046 mmol) in dichloromethane (2 mL) were added *O*-(*tert*-butyldimethylsilyl)-hydroxylamine (8.2 mg, 0.055 mmol), 4 \AA molecular sieves (crushed), and a catalytic amount of PPTS. The mixture was stirred at rt for 22 h and then filtered through a pad of Celite. The solvent was removed under reduced pressure and the residue was purified by flash chromatography (15% ethyl acetate/hexanes) to afford the α -chloroketoxime **18e** as a colorless oil (12.4 mg, 70%). ^1H NMR (300 MHz, CDCl_3 , complex mixture of diastereomers including oxime geometric isomers) δ 5.05-4.94 (m, 1H), 4.85-4.61 (m, 1H), 4.23-4.11 (m, 2H), 2.82-2.53 (m, 1H), 2.36-2.23 (m, 2H), 2.15-2.00 (m, 2H), 1.96-1.85 (m, 1H), 1.66-1.56 (m, 1H), 1.15 (t, $J = 7.1$ Hz, 3H), 0.82-0.66 (m, 9H), 0.01-0.00 (m, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 166.2, 164.5, 87.4, 87.1, 63.7, 58.8, 58.6, 51.1, 42.5, 42.4, 42.0, 35.5, 35.4, 35.2, 34.7, 32.9, 32.4, 32.3, 32.2, 32.1, 26.6, 26.3, 26.3, 18.5, 14.3, -4.8; HRMS-ES [M + H]⁺ calcd for C₁₆H₃₀ClN₂O₅Si, 393.1613; found, 393.1614.

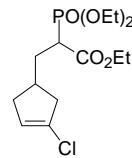
4-Allyl-6-chloro-2-(diethoxyphosphoryl)-hept-6-enoic Acid Ethyl Ester (15f). To a stirred suspension of sodium hydride (60% dispersion in mineral oil, 50 mg, 1.25 mmol) in DMF (2 mL) at 0 °C was dropwise added a solution of diethoxyphosphoryl acetic acid ethyl ester (0.26 mL, 1.25



mmol) in DMF (2 mL). After stirring for 1 h at rt, reaction mixture was re-cooled to 0 °C and a solution of iodide **14** (405 mg, 1.5 mmol) in DMF (1 mL) was added. The resulting mixture was then heated at 55 °C for 13 h. Saturated aqueous NH₄Cl and ethyl acetate were added. The organic layer was separated and the aqueous layer was extracted with ethyl acetate. The combined organic layer was washed with water and brine. The organic layer was dried over MgSO₄ and the solvent was removed under reduced pressure. The residue was purified by flash chromatography (50% ethyl acetate/hexanes) to afford the diene **15f** as a colorless oil (236 mg, 52%). ¹H NMR (300 MHz, CDCl₃, diastereomer mixture) δ 5.68-5.53 (m, 1H), 5.07 (d, *J* = 16.0 Hz, 2H), 4.97-4.82 (m, 2H), 4.35-3.80 (m, 6H), 3.04-2.90 (m, 1H), 2.29-1.56 (m, 7H), 1.23-1.11 (m, 9H); ¹³C NMR (75 MHz, CDCl₃) δ 169.4, 169.4, 169.2, 169.1, 141.2, 141.1, 135.6, 135.0, 118.0, 117.7, 114.6, 114.4, 63.1, 63.0, 62.9, 61.8, 61.7, 44.7, 44.6, 43.8, 43.1, 42.9, 42.9, 37.7, 36.1, 34.2, 34.0, 33.8, 33.6, 30.7, 30.6, 30.2, 30.1, 16.7, 16.6, 14.4; HRMS-ES [M+H]⁺ calcd for C₁₆H₂₉ClO₅P, 367.1441; found, 367.1431.

3-(3-Chlorocyclopent-3-enyl)-2-(diethoxyphosphoryl)-propionic Acid Ethyl Ester (**16f**).

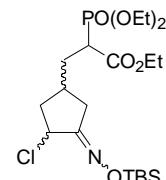
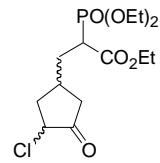
A flame dried 250 mL two-necked flask equipped with a magnetic stirring bar and a condenser was charged with diene **15f** (197 mg, 0.54 mmol) and toluene (120 mL). The solution was deaerated by bubbling argon through the mixture for 2 h. The second-generation Grubbs catalyst (46 mg, 0.054 mmol) in 2 mL of toluene was added and the argon bubbling was continued for an additional 30 min. The mixture was heated and stirred at 85 °C for 20 h. The solvent was removed under reduced pressure and the residue was purified by flash chromatography (50% ethyl acetate/hexanes) to afford the vinyl chloride **16f** as a yellow oil (100 mg,



55%). ^1H NMR (300 MHz, CDCl_3 , diastereomer mixture) δ 5.64-5.55 (m, 1H), 4.23-4.07 (m, 6H), 2.99-2.87 (m, 1H), 2.62-1.86 (m, 7H), 1.33-1.22 (m, 9H); ^{13}C NMR (75 MHz, CDCl_3) δ 169.0, 169.0, 131.1, 130.7, 125.4, 125.0, 62.8, 62.7, 62.7, 62.6, 61.4, 45.3, 45.1, 44.8, 43.8, 43.5, 43.4, 43.3, 43.2, 42.5, 37.6, 36.7, 36.2, 36.0, 33.1, 33.0, 33.0, 32.9, 16.3, 16.3, 16.2, 16.2, 14.0; HRMS-ES $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{14}\text{H}_{25}\text{ClO}_5\text{P}$, 339.1128; found, 339.1128.

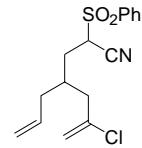
3-(3-Chloro-4-oxocyclopentyl)-2-(diethoxyphosphoryl)-propionic Acid Ethyl Ester (17f). To a solution of vinyl chloride **16f** (70 mg, 0.21 mmol), acetone (3.5 mL) and glacial acetic acid (1.4 mL) at 0 °C was added dropwise sodium hypochlorite (0.19 mL of 10% solution, 0.21 mmol) via syringe. The reaction mixture was stirred at 0 °C for 30 min and quenched by addition of saturated aqueous NaHCO_3 solution. The mixture was then extracted with dichloromethane. The combined organic layers were washed with brine and dried over Na_2SO_4 . The solvent was removed under reduced pressure and the residue was purified by flash chromatography (75% ethyl acetate/hexanes) affording the α -chloroketone **17f** as a colorless oil (31 mg, 43%). ^1H NMR (300 MHz, CDCl_3 , diastereomer mixture) δ 4.60-4.24 (m, 7H), 3.23-3.15 (m, 1H), 2.88-2.02 (m, 7H), 1.58-1.44 (m, 9H); ^{13}C NMR (75 MHz, CDCl_3) δ 209.3, 209.0, 208.9, 208.6, 168.8, 168.8, 168.7, 168.7, 63.0, 62.9, 62.8, 61.7, 57.2, 56.9, 45.0, 43.2, 41.9, 41.5, 39.4, 39.0, 32.8, 32.6, 32.6, 32.4, 32.0, 16.4, 16.3, 16.3, 16.2, 14.0; HRMS-ES $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{14}\text{H}_{25}\text{ClO}_6\text{P}$, 355.1077; found, 355.1072.

3-(3-Chloro-4-*tert*-butyldimethylsilyloxyiminocyclopentyl)-2-(diethoxyphosphoryl)-propionic Acid Ethyl Ester (18f). To a solution of α -chloroketone **17f** (25.0 mg, 0.07 mmol) in dichloromethane (3 mL) were



added *O*-(*tert*-butyldimethylsilyl)-hydroxylamine (24.0 mg, 0.14 mmol), 4Å molecular sieves (crushed), and a catalytic amount of PPTS. The mixture was stirred at rt for 21 h and then filtered through a pad of Celite. The solvent was removed under reduced pressure and the residue was purified by flash chromatography (75% ethyl acetate/hexanes) to afford the α -chloroketoxime **18f** as a colorless oil (25.5 mg, 75%). ^1H NMR (300 MHz, CDCl_3 , complex mixture of diastereomers including oxime geometric isomers) δ 5.10-4.58 (m, 1H), 4.31-4.09 (m, 6H), 3.04-2.79 (m, 2H), 2.52-2.49 (m, 1H), 2.27-2.12 (m, 2H), 2.06-1.84 (m, 2H), 1.74-1.63 (m, 1H), 1.35-1.24 (m, 9H), 0.97-0.84 (m, 9H), 0.17-0.13 (m, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 169.1, 168.9, 167.1, 166.7, 63.0, 62.8, 62.7, 61.6, 58.9, 58.6, 45.6, 43.8, 42.3, 42.0, 34.3, 34.1, 32.1, 31.8, 26.0, 25.9, 25.9, 18.1, 18.0, 16.4, 16.3, 14.1, -5.2, -5.2; HRMS-ES [M + H] $^+$ calcd for $\text{C}_{20}\text{H}_{40}\text{ClNO}_6\text{PSi}$, 484.2051; found, 484.2039.

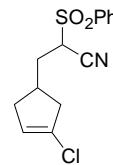
4-Allyl-2-benzenesulfonyl-6-chlorohept-6-enenitrile (15g). To a stirred suspension of sodium hydride (60% dispersion in mineral oil, 43 mg, 1.07 mmol) in THF (2 mL) at 50 °C was dropwise added a solution of benzenesulfonyl acetonitrile (157 mg, 0.85 mmol) in THF (2 mL). After stirring the mixture for 15 min at 50 °C, a solution of iodide **14** (252 mg, 0.93 mmol) in THF (1 mL) was added in one portion. The resulting mixture was then heated at reflux for 19 h. Saturated aqueous NH_4Cl and ethyl acetate were added. The organic layer was separated and the aqueous layer was extracted with ethyl acetate. The combined organic layer was washed with water and brine. The organic layer was dried over Na_2SO_4 and the solvent was removed under reduced pressure. The residue was purified by flash chromatography (20% ethyl acetate/hexanes) to afford the diene **15g** as a colorless oil (214 mg, 78%). ^1H



NMR (300 MHz, CDCl₃, diastereomer mixture) δ 7.83 (d, *J* = 7.5 Hz, 2H), 7.61 (t, *J* = 7.4 Hz, 1H), 7.48 (t, *J* = 7.7 Hz, 2H), 5.61-5.46 (m, 1H), 5.08-4.84 (m, 4H), 3.97-3.91 (m, 1H), 2.32-1.73 (m, 7H); ¹³C NMR (75 MHz, CDCl₃) δ 140.2, 140.1, 135.8, 134.7, 134.3, 130.0, 130.0, 130.0, 119.2, 118.9, 115.8, 115.5, 114.5, 114.2, 56.1, 43.8, 43.3, 38.1, 36.6, 33.2, 33.0, 30.4, 30.0; HRMS-ES [M + NH₄]⁺ calcd for C₁₆H₂₂ClN₂O₂S, 341.1091; found, 341.1089.

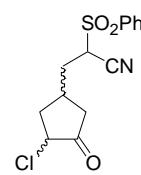
2-Benzenesulfonyl-3-(3-chlorocyclopent-3-enyl)-propionitrile (16g).

A flame dried 250 mL two-necked flask equipped with a magnetic stirring bar and a condenser was charged with diene **15g** (214 mg, 0.66 mmol) and toluene (150 mL). The solution was deaerated by bubbling argon through the mixture for 2 h. The second-generation Grubbs catalyst (112 mg, 0.132 mmol) in 2 mL of toluene was added and the argon bubbling was continued for an additional 30 min. The mixture was heated and stirred at 95 °C for 19 h. The solvent was removed under reduced pressure and the residue was purified by flash chromatography (20% ethyl acetate/hexanes) to afford the vinyl chloride **16g** as a pale yellow oil (74 mg, 38%). ¹H NMR (300 MHz, CDCl₃, diastereomer mixture) δ 7.95 (d, *J* = 7.7 Hz, 2H), 7.72 (t, *J* = 7.4 Hz, 1H), 7.59 (t, *J* = 7.7 Hz, 2H), 5.64-5.52 (m, 1H), 3.90-3.79 (m, 1H), 2.74-2.49 (m, 3H), 2.28-1.89 (m, 4H); ¹³C NMR (75 MHz, CDCl₃) δ 135.8, 135.8, 135.7, 131.6, 130.9, 130.1, 130.1, 130.0, 125.7, 125.0, 114.3, 56.8, 56.6, 43.7, 42.8, 38.1, 37.1, 35.2, 35.1, 32.9, 32.9; HRMS-ES [M + NH₄]⁺ calcd for C₁₄H₁₈ClN₂O₂S, 313.0778; found, 313.0776.



2-Benzenesulfonyl-3-(3-chloro-4-oxocyclopentyl)-propionitrile

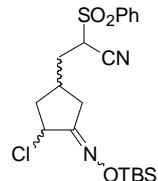
(17g). To a solution of vinyl chloride **16g** (27.8 mg, 0.094 mmol), acetone (1.8 mL) and glacial acetic acid (0.7 mL) at 0 °C was added dropwise sodium



hypochlorite (0.086 mL of 10% solution, 0.094 mmol) via syringe. The reaction mixture was stirred at 0 °C for 30 min and quenched by addition of saturated aqueous NaHCO₃ solution. The mixture was then extracted with dichloromethane. The combined organic layers were washed with brine and dried over MgSO₄. The solvent was removed under reduced pressure and the residue was purified by flash chromatography (25% ethyl acetate/hexanes) affording the α -chloroketone **17g** as a colorless oil (16.4 mg, 56%). ¹H NMR (300 MHz, CDCl₃, diastereomer mixture) δ 7.96 (d, *J* = 7.2 Hz, 2H), 7.74 (t, *J* = 6.8 Hz, 1H), 7.61 (t, *J* = 7.6 Hz, 2H), 4.10 (d, *J* = 5.3 Hz, 1H), 3.94-3.81 (m, 1H), 2.80-2.62 (m, 2H), 2.40-2.20 (m, 2H), 2.17-1.81 (m, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 208.0, 207.7, 136.0, 135.5, 135.5, 130.2, 130.1, 128.3, 114.1, 58.5, 57.1, 56.7, 56.6, 56.2, 55.9, 42.0, 41.9, 41.4, 41.3, 39.9, 39.8, 39.4, 39.2, 32.5, 32.4, 32.0, 31.9, 31.9, 31.1, 31.0; HRMS-ES [M + NH₄]⁺ calcd for C₁₄H₁₈ClN₂O₃S, 329.0727; found, 329.0728.

2-Benzenesulfonyl-3-(3-chloro-4-*tert*-butyldimethylsilyloxyiminocyclopentyl)-propionitrile (18g). To a solution of α -chloroketone **17g** (34.0 mg, 0.11 mmol) in dichloromethane (3 mL) were added *O*-(*tert*-butyldimethylsilyl)-hydroxylamine (34.0 mg, 0.22 mmol), 4 Å molecular sieves (crushed), and a catalytic amount of PPTS. The mixture was stirred at rt for 21 h and then filtered through a pad of Celite. The solvent was removed under reduced pressure and the residue was purified by flash chromatography (20% ethyl acetate/hexanes) to afford the α -chloroketoxime **18g** as a colorless oil (20.0 mg, 42%).

¹H NMR (300 MHz, CDCl₃, complex mixture of diastereomers including oxime geometric isomers) δ 7.86 (d, *J* = 7.6 Hz, 2H), 7.64 (t, *J* = 7.4 Hz, 1H), 7.51 (t, *J* = 7.6 Hz, 2H), 4.95-4.50 (m, 1H), 3.82-3.62 (m, 1H), 2.88-2.38 (m, 2H), 2.24-1.82 (m, 4H), 1.71-



1.57 (m, 1H), 0.77-0.69 (m, 9H), 0.01-0.00 (m, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 166.1, 165.7, 164.9, 164.6, 135.9, 135.6, 130.1, 130.1, 130.1, 114.2, 58.7, 58.4, 56.9, 56.9, 56.6, 42.8, 42.2, 34.6, 33.8, 33.4, 33.4, 32.5, 31.9, 31.8, 26.4, 26.3, 26.3, 18.5, 18.4, -4.8, -4.8; HRMS-ES $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{20}\text{H}_{30}\text{ClN}_2\text{O}_3\text{SSi}$, 441.1435; found, 441.1452.

Figure 1: Ortep Diagram of X-ray Diffraction Structure of 22b.

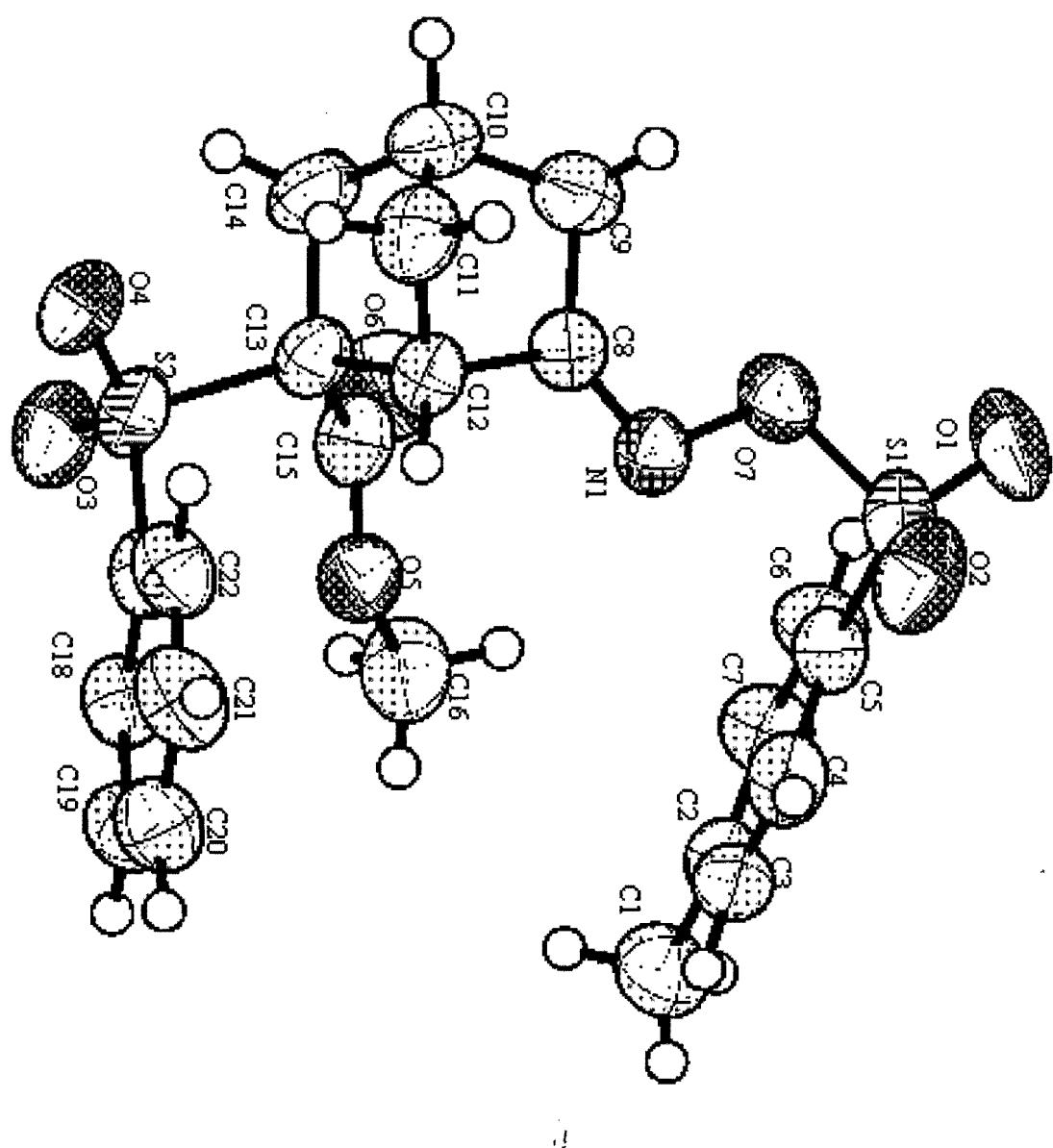


Figure 2: Ortep Diagram of X-ray Diffraction Structure of 39.

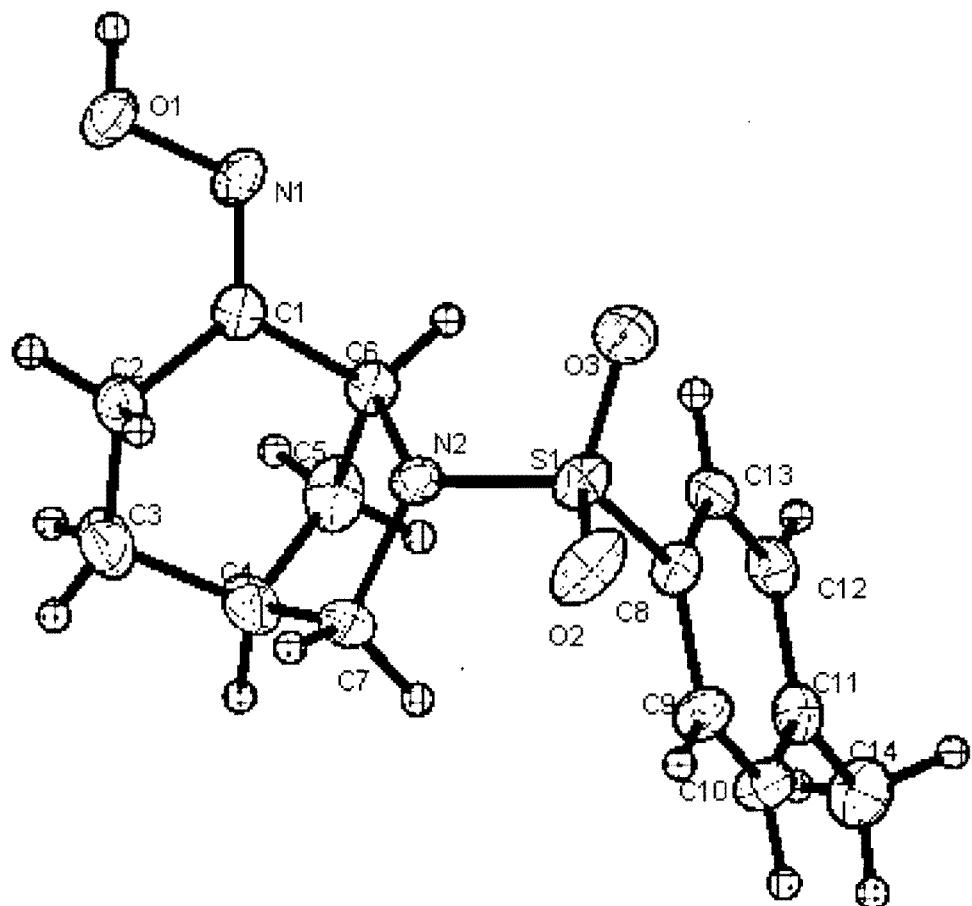
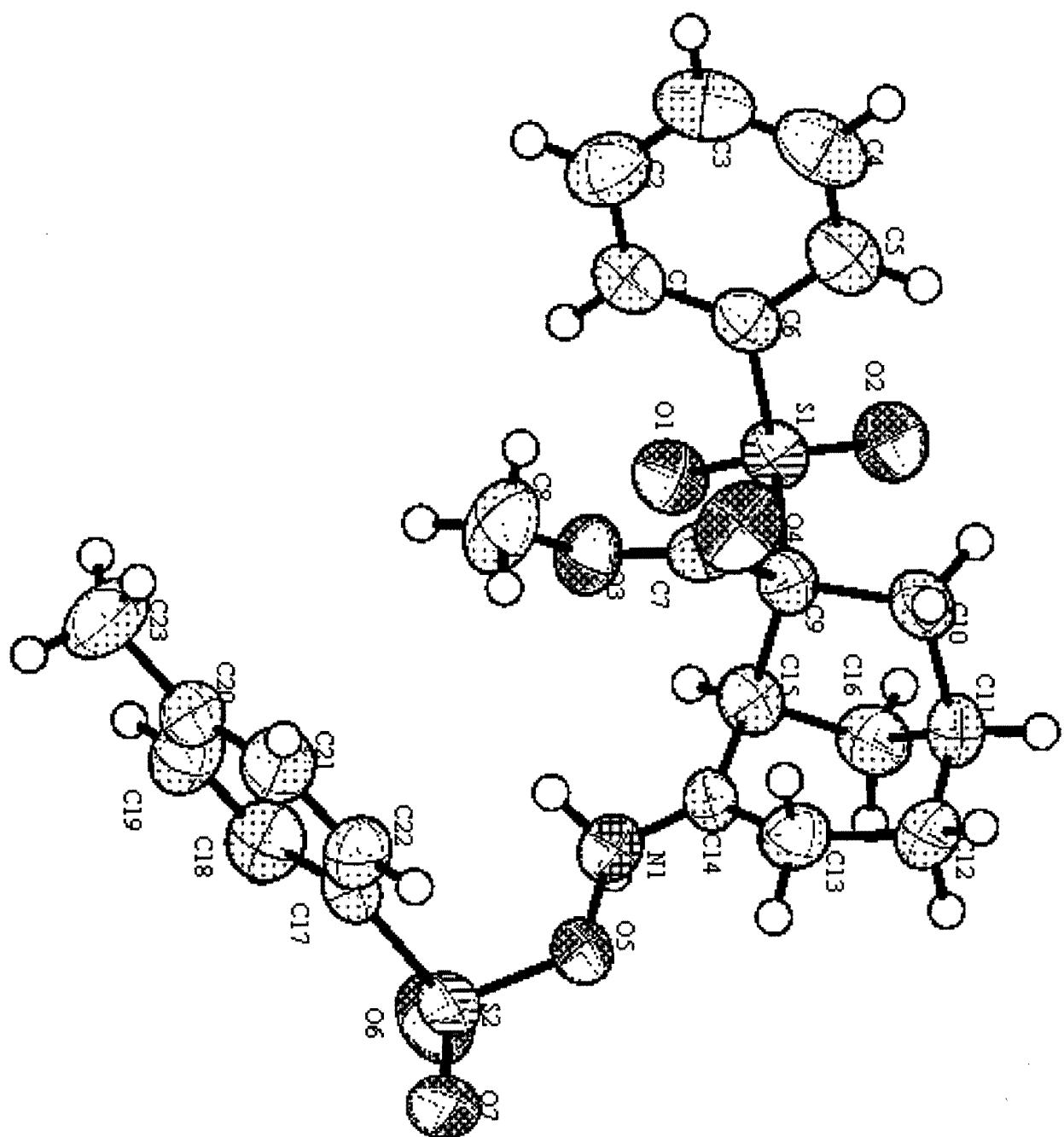
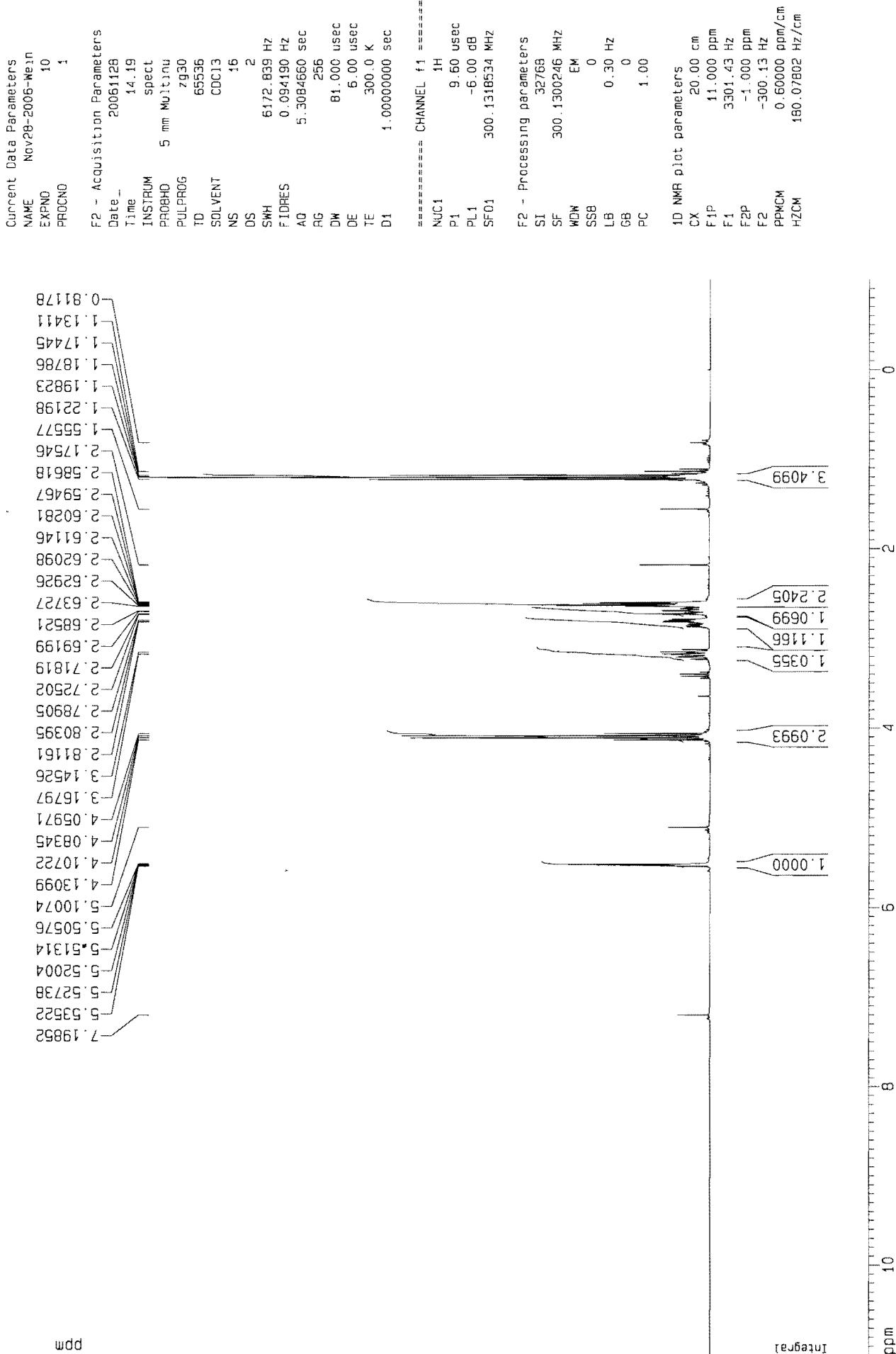
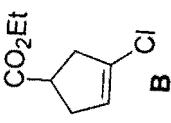
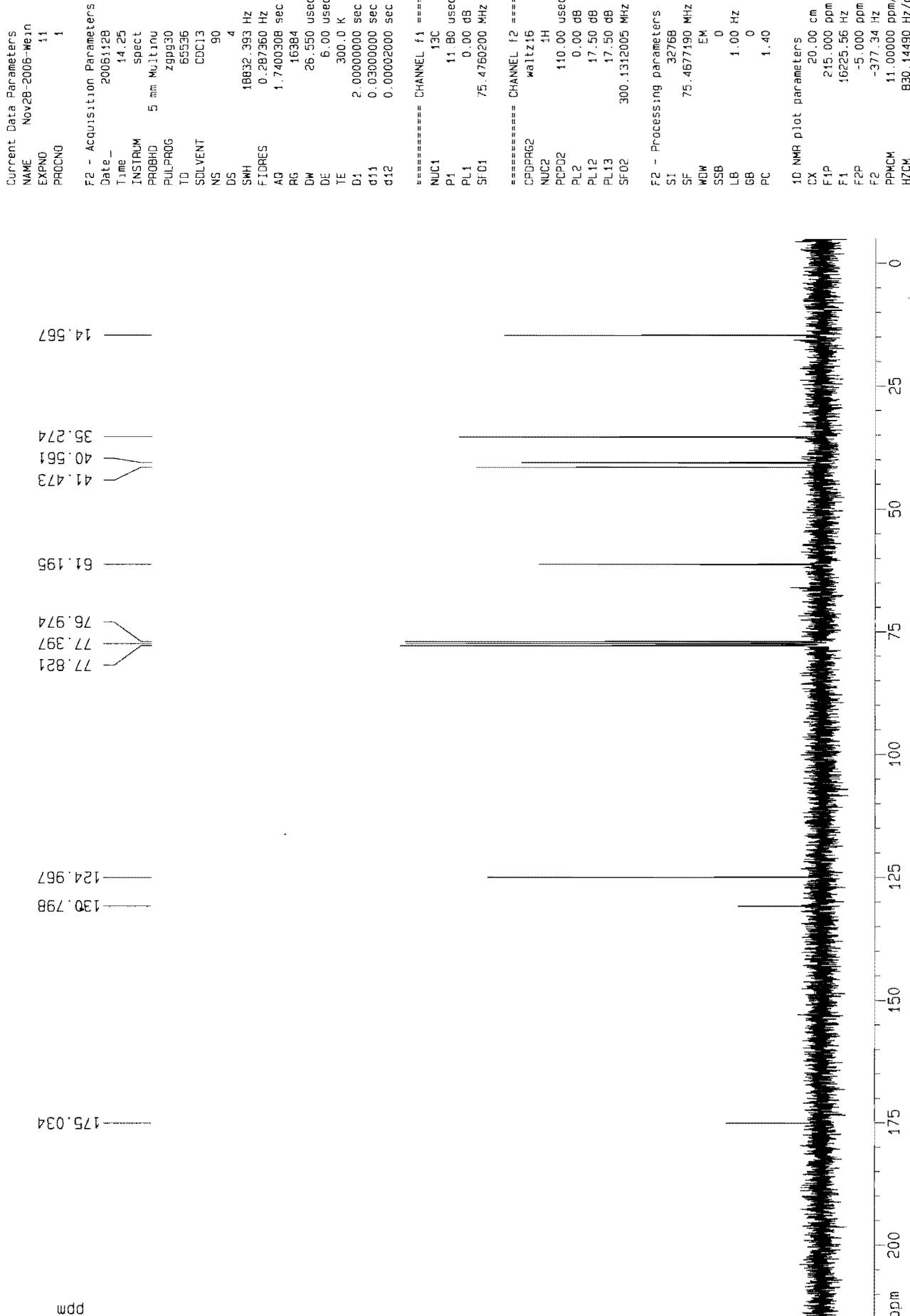
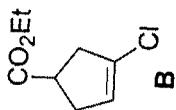
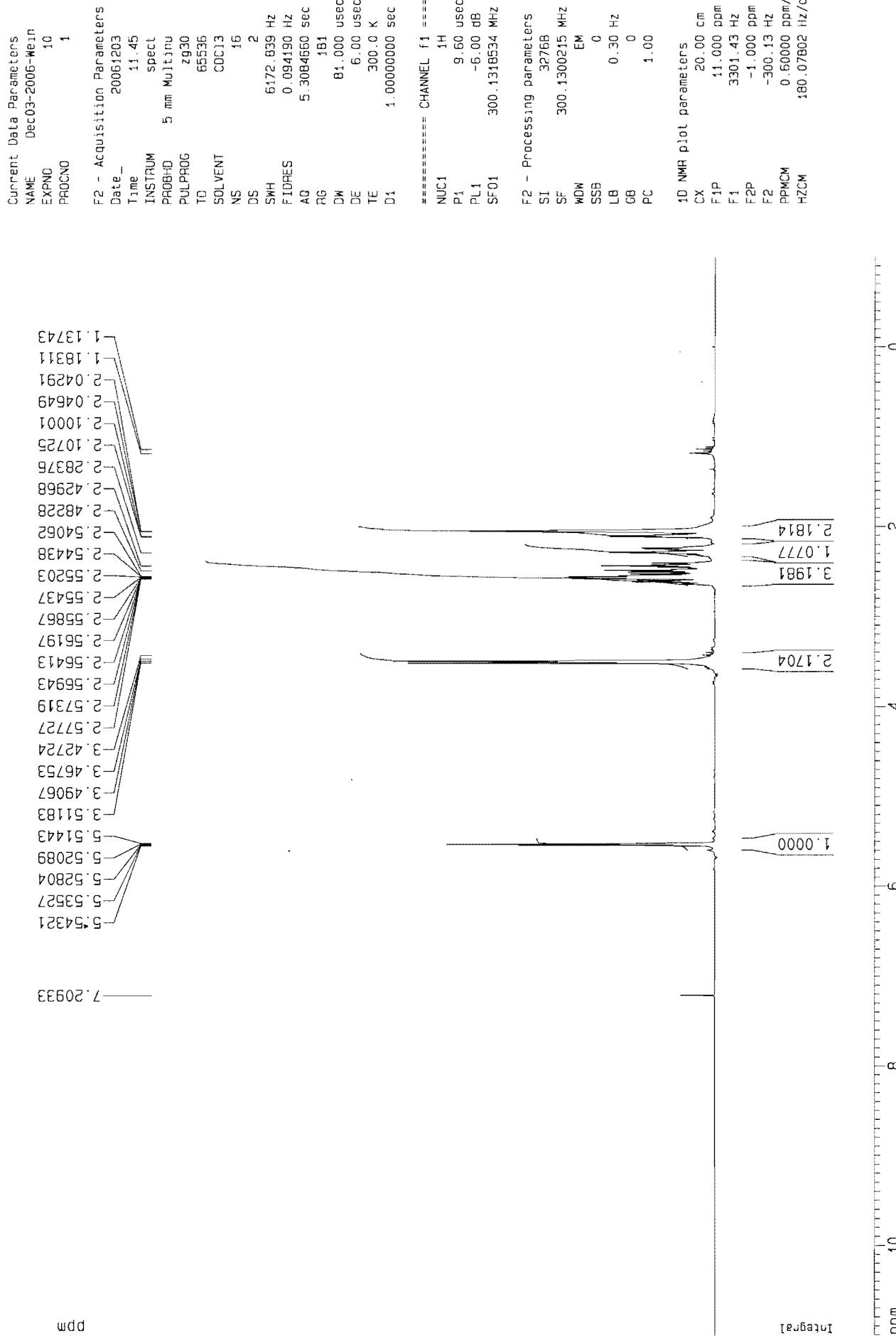
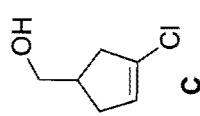


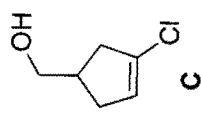
Figure 3: Ortep Diagram of X-ray Diffraction Structure of 33b.



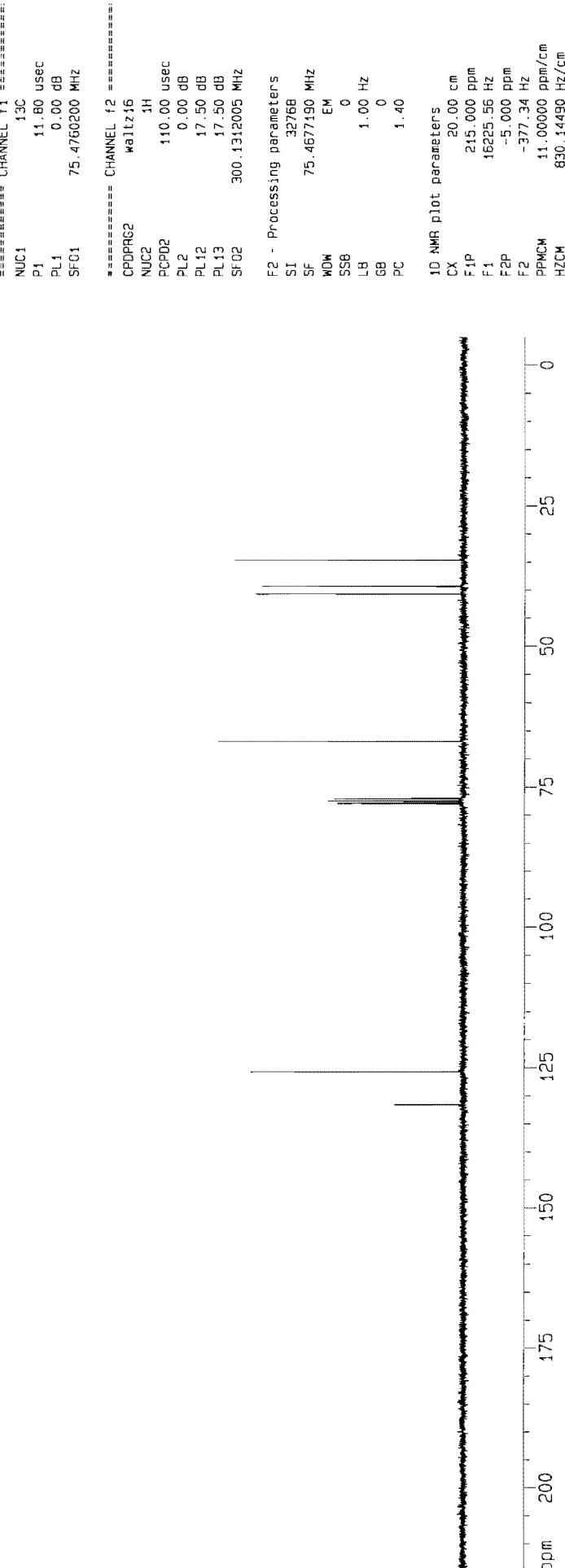
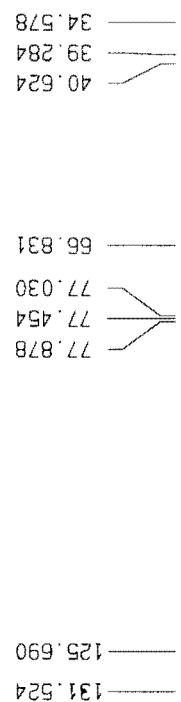
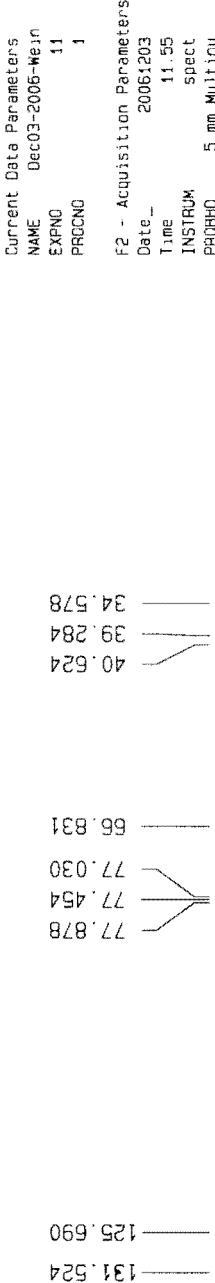


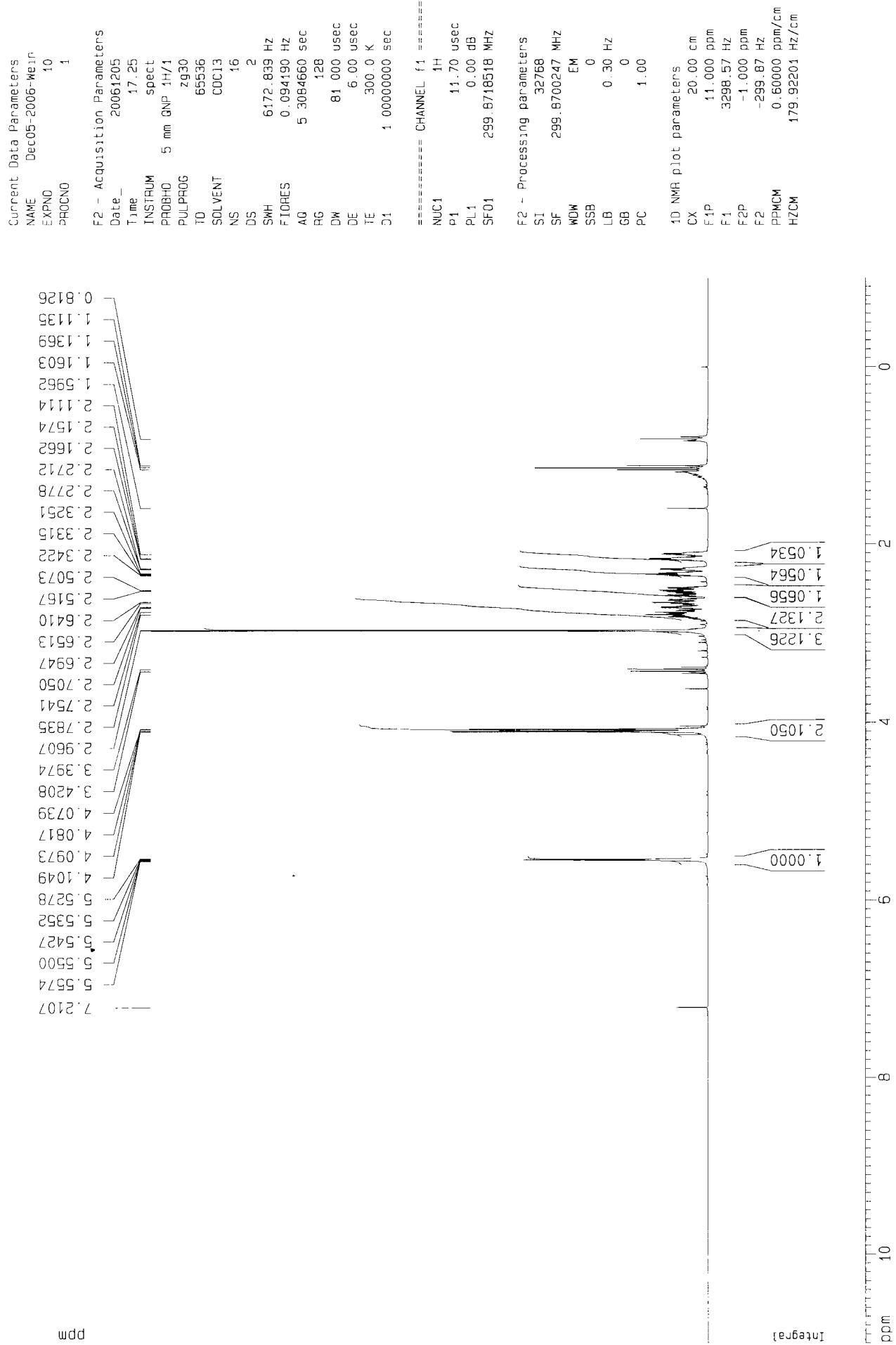
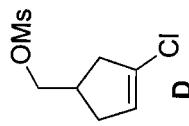


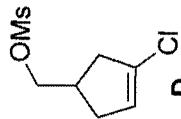




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Current Data Parameters
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EXPNO 11
PROCNO 1

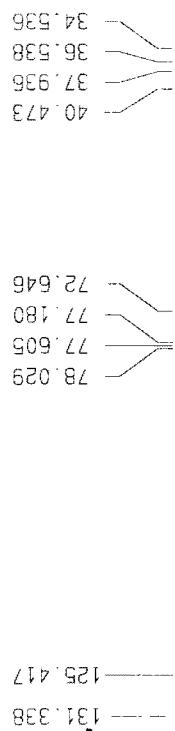
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NS 129
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SWH 18796.992 Hz
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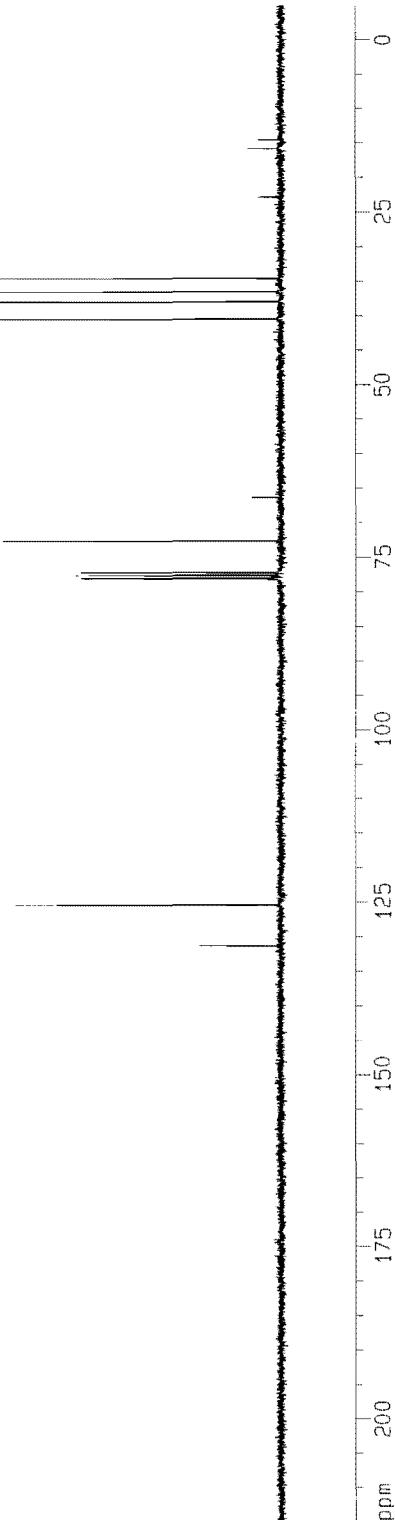
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F2 - Processing parameters
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1D NMR plot parameters
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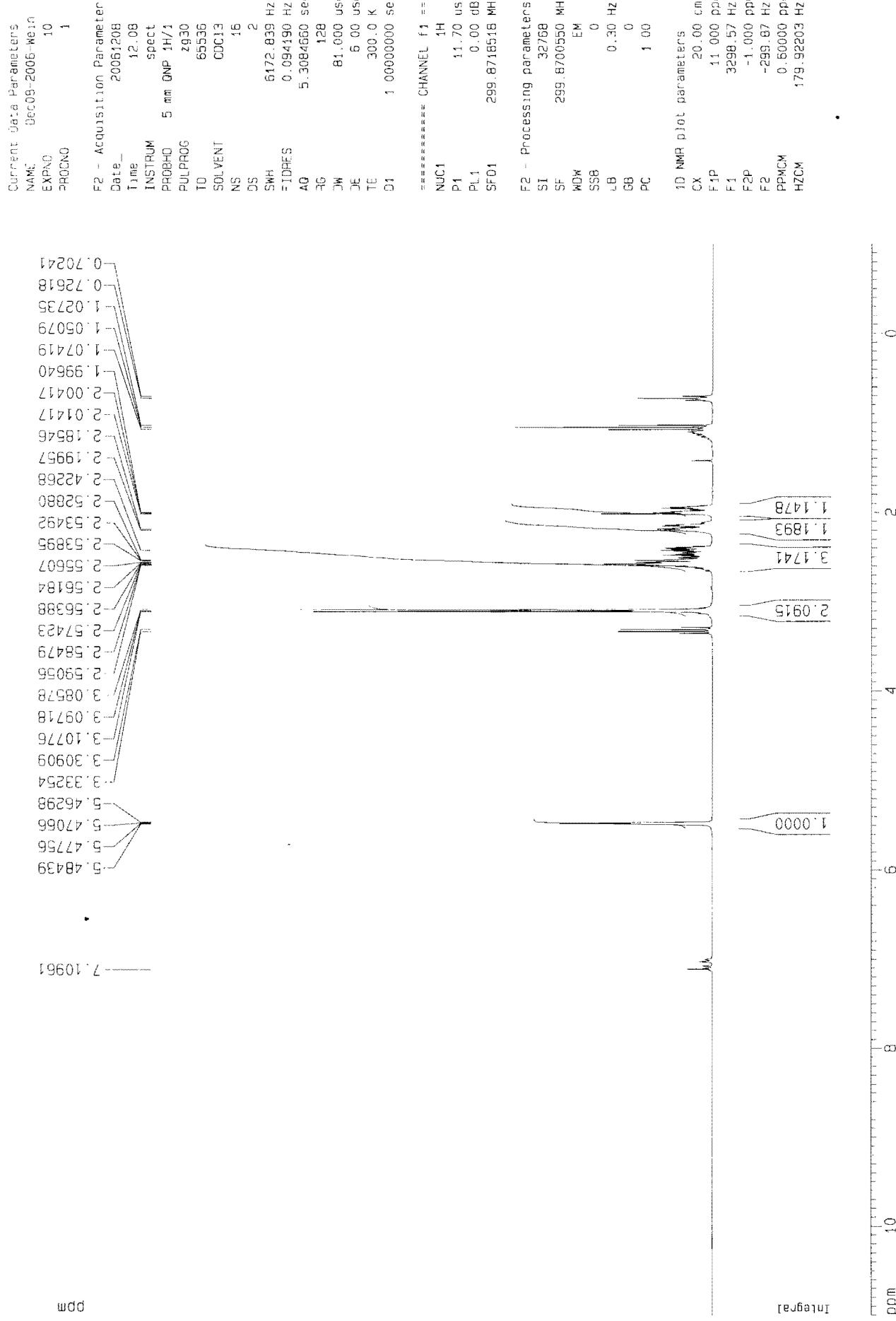


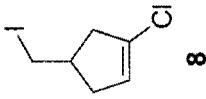
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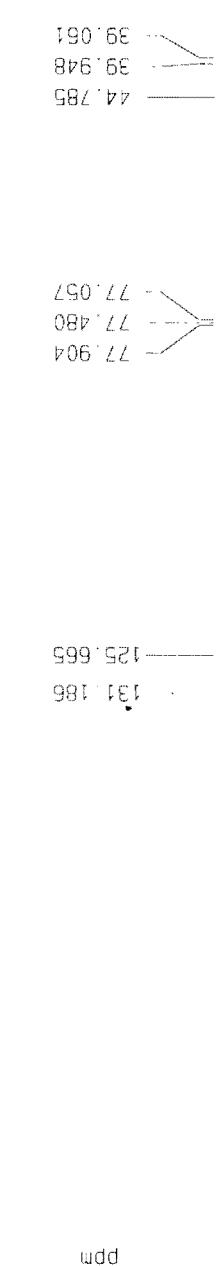


8





8



Current Data Parameters
NAME Dec09-2006Wein
EXPNO 11
PROCNO 1

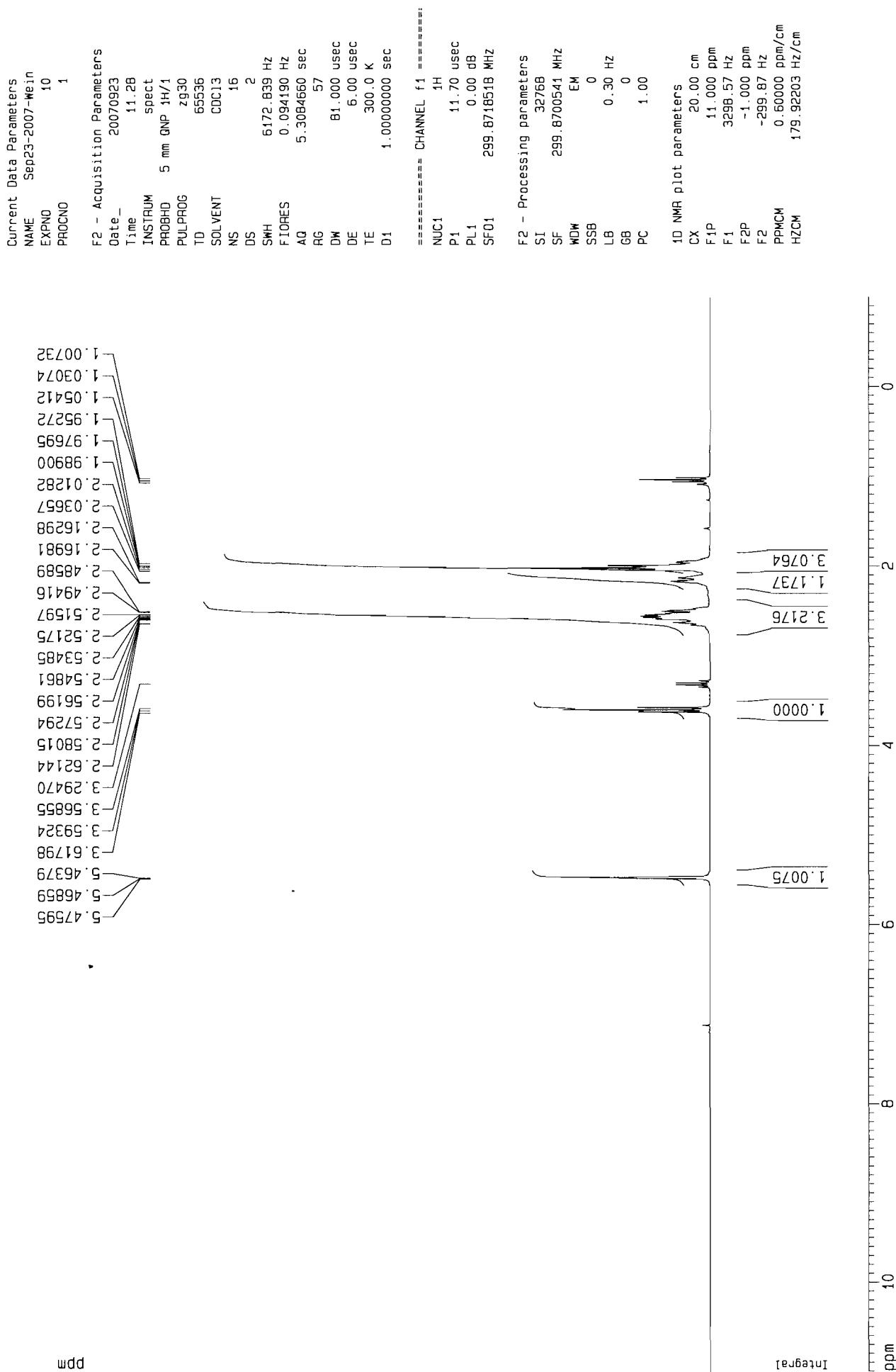
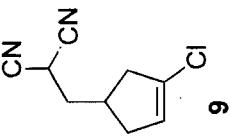
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FIDRES 0.288619 Hz
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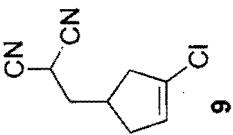
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F2 - Processing parameters
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Current Data Parameters
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EXPNO 11
PROCNO 1

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FIDRES 0.288819 Hz
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RG 1824.6
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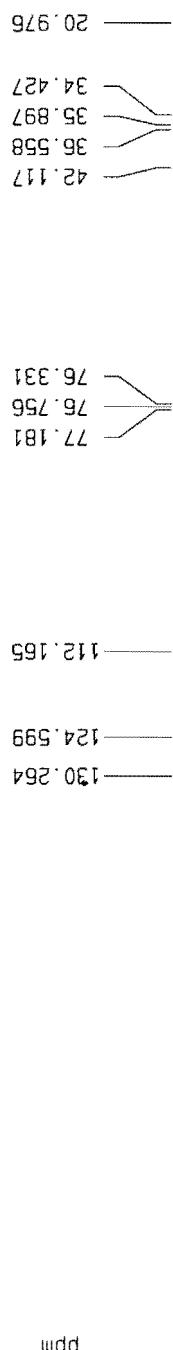
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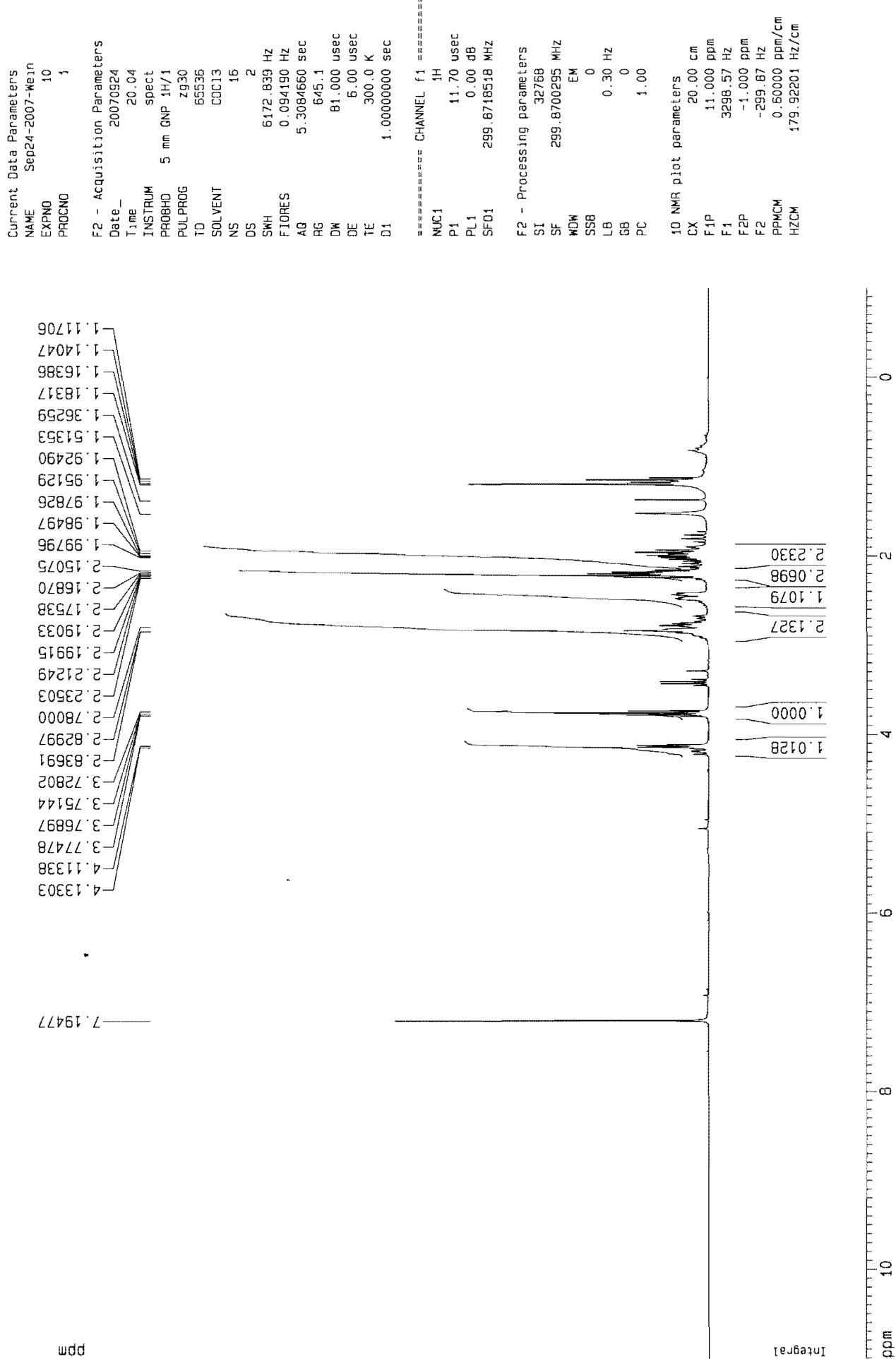
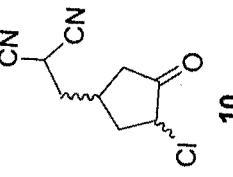
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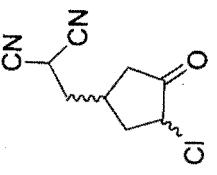
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1D NMR plot parameters

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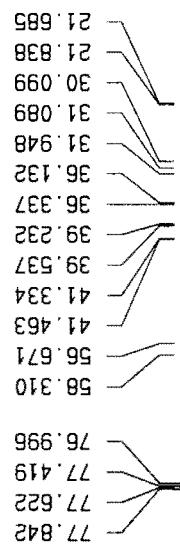
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 DE 6.00 usec
 TE 300.0 K
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 d11 0.0300000 sec
 d12 0.00002000 sec

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 PL1 0.00 dB
 SF01 75.4760200 MHz

===== CHANNEL f2 =====
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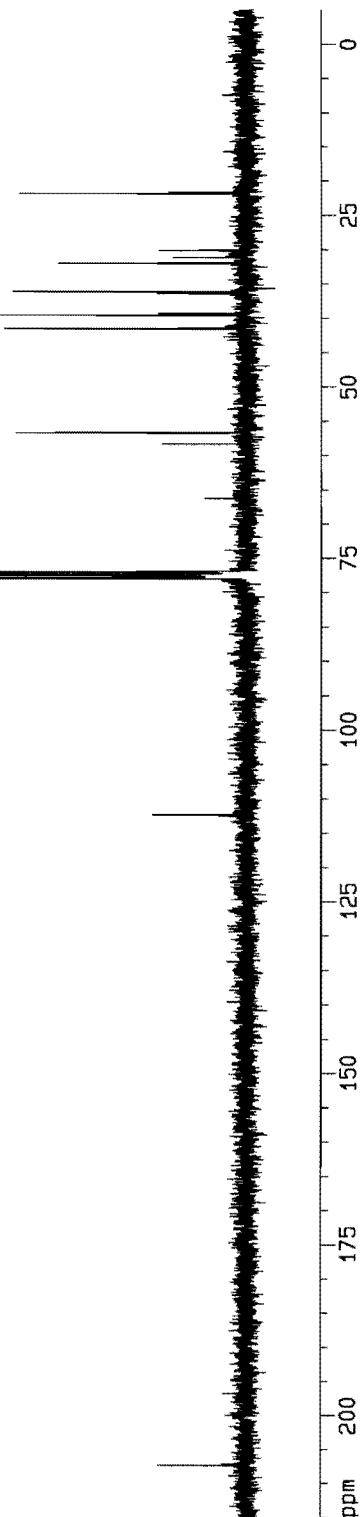
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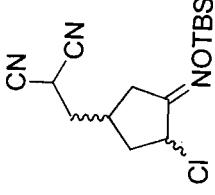
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112.342

207.327
PPM





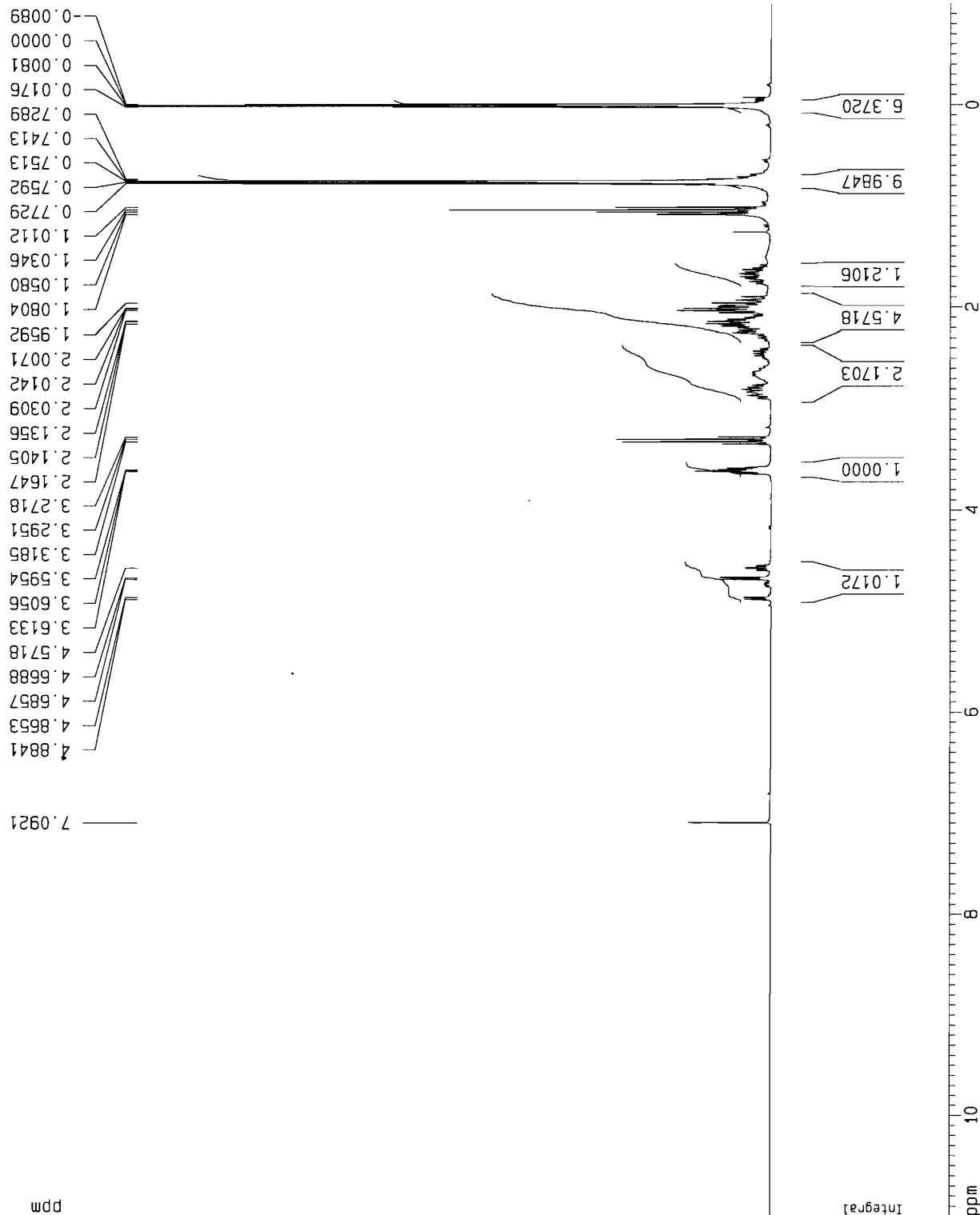
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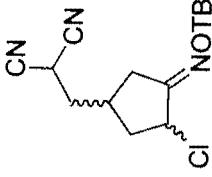
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EXPNO 10
PROCNO 1

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11

Current Data Parameters

NAME Oct03-2007-Me.in
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PROCND 1

F2 - Acquisition Parameters

Date 2007/10/03
Time 18.49
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg99J30
TD 65536
SOLVENT CDCl₃
NS 70
DS 4
SWH 18796.992 Hz
FIDRES 0.288819 Hz
AQ 1.7433076 sec
RG 2048
DW 600 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec
D11 0.0300000 sec
D12 0.0000200 sec

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

NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SF01 75.4106357 MHz

===== CHANNEL f2 =====

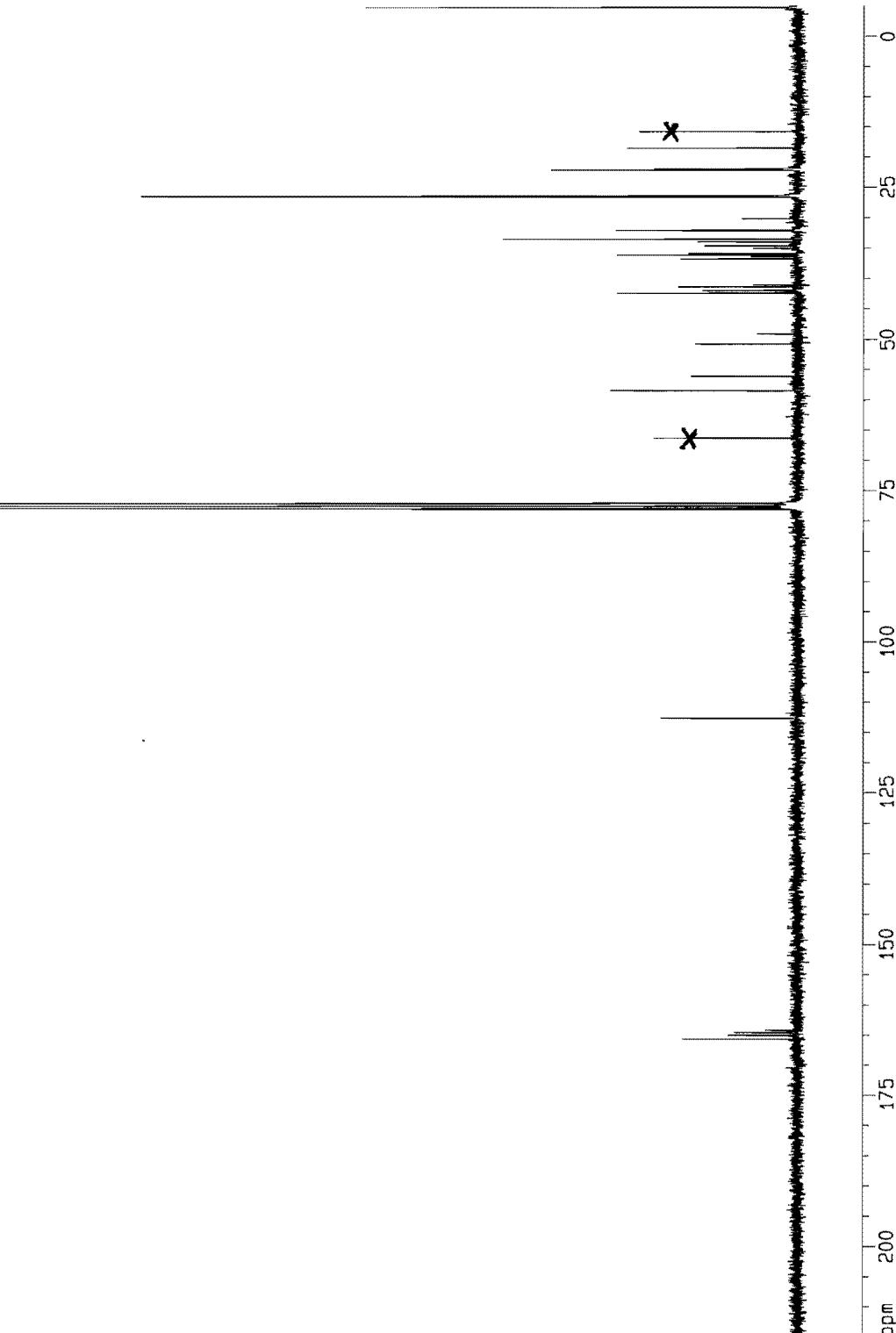
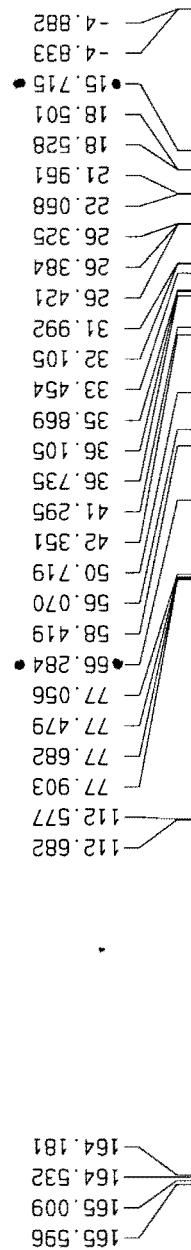
CPDPG2 WALTZ16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SF02 299.8711995 MHz

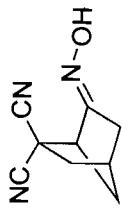
F2 - Processing parameters

SI 32768
SF 75.4023410 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
F1P 215.000 ppm
F1 16.11.50 Hz
F2P -5.000 ppm
F2 -377.01 Hz
PPMCM 11.00000 ppm/cm
H2CM 829.42578 Hz/cm





13

Current Data Parameters

NAME Sep27-2007-Me in
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters

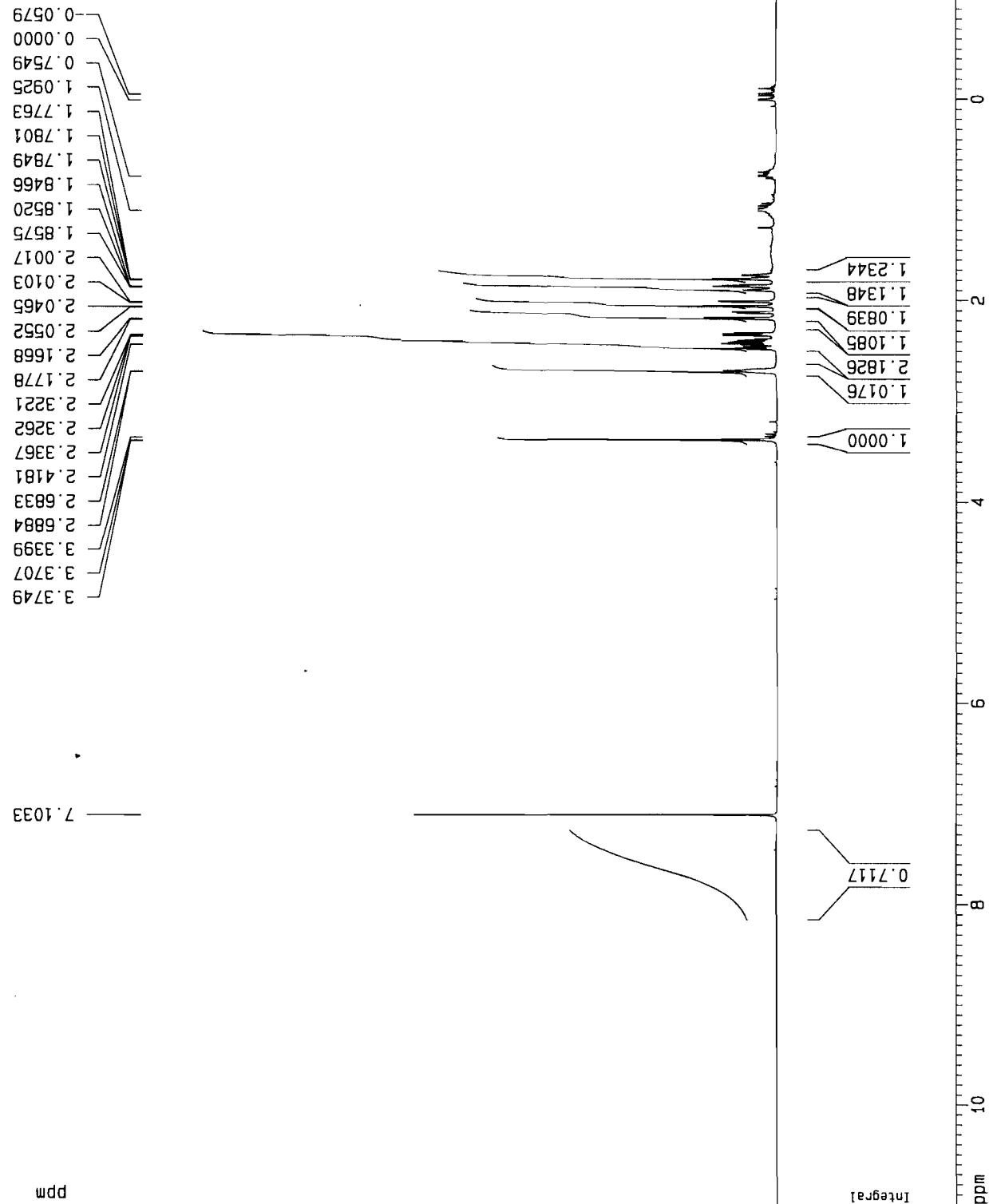
Date_ 20070927
Time 23.34
INSTRUM Spect
PROBHD 5 mm Multinu
PULPROG zg30
TD 65536
SOLVENT DDC13
NS 52
DS 0
SWH 6172.839 Hz
FTDRES 0.094190 Hz
AQ 5.304660 sec
RG 574.7
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec

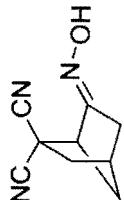
===== CHANNEL f1 =====

NUC1 1H
P1 9.60 usec
PL1 -6.00 dB
SF01 300.1338534 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

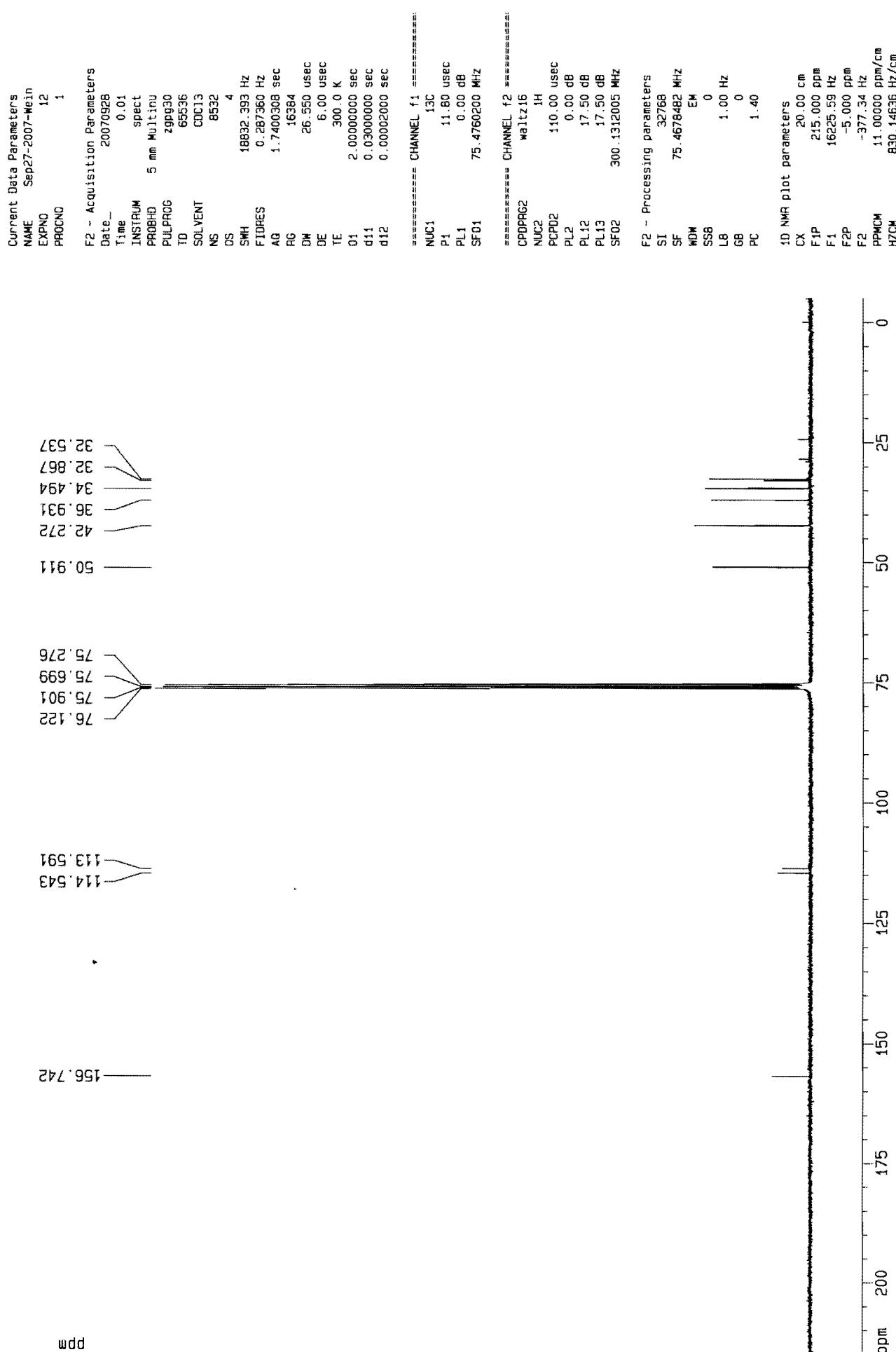
1D NMR pilot parameters

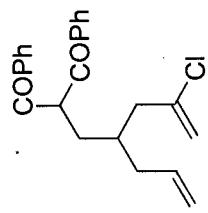
CX 20.00 cm
F1P 11.000 ppm
F1 3301.43 Hz
F2P -1.000 ppm
F2 -300.13 Hz
PPMCM 0.60000 ppm/cm
HZCM 180.07803 Hz/cm



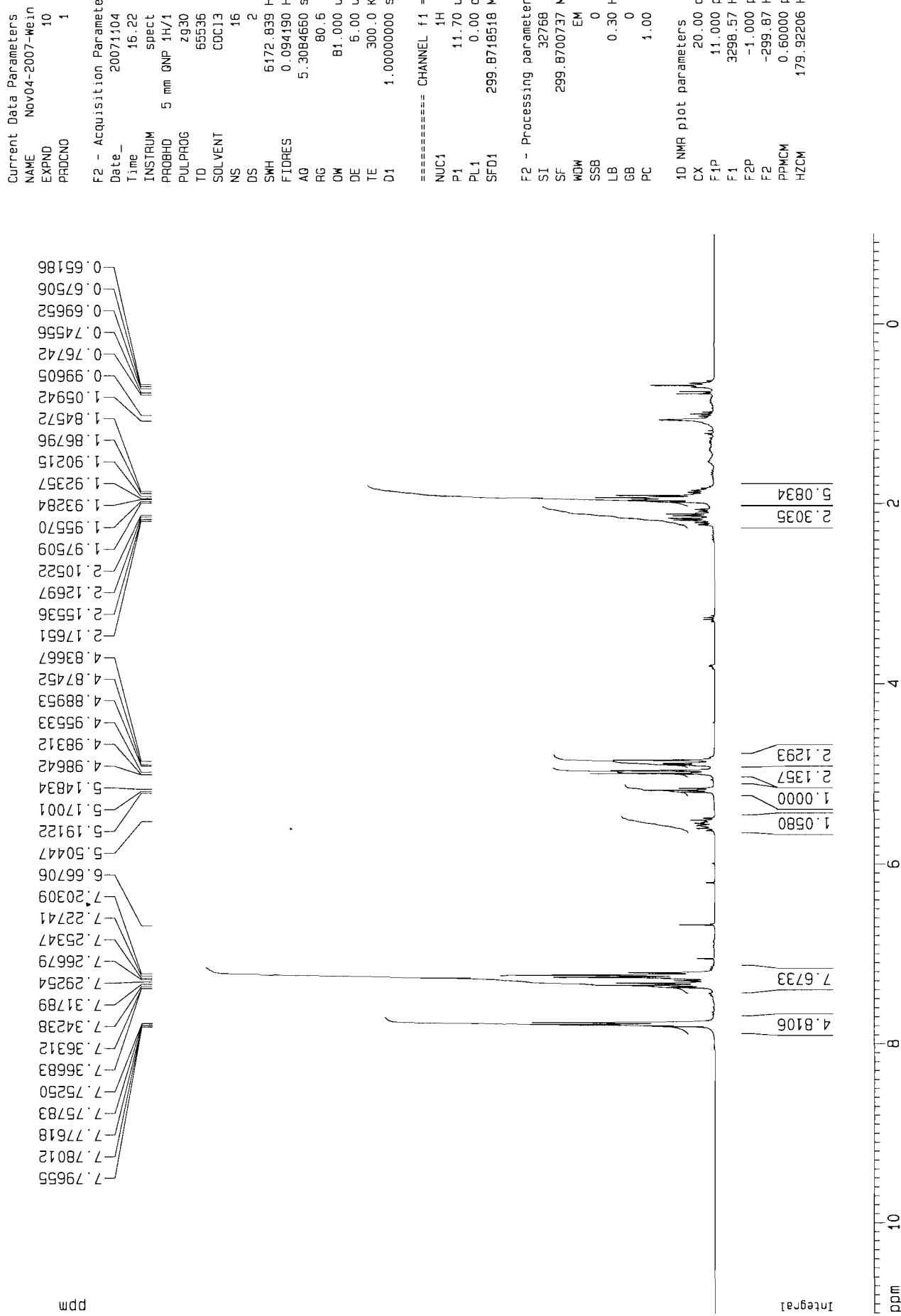


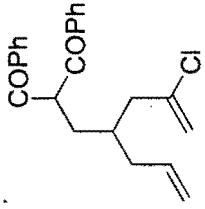
13





15a





15a

Current Data Parameters

NAME Nov04-2007-Wein
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters

Date 2007/10/04
Time 16:32
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 166
DS 4
SWH 18796.992 Hz
FIDRES 0.286819 Hz
AQ 1.7433076 sec
RG 512
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

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

NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SF01 75.4106357 MHz

===== CHANNEL f2 =====

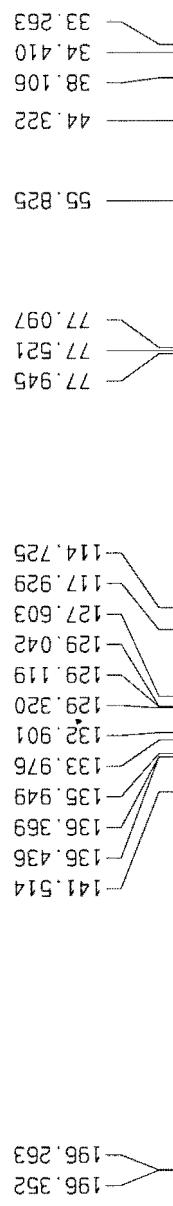
CPDPRG2 waltz16
NUC2 1H
PCPD02 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SF02 299.8711995 MHz

F2 - Processing parameters

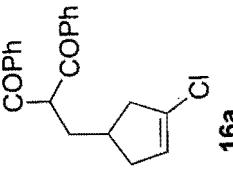
SI 32768
SF 75.4023410 MHz
WDM EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

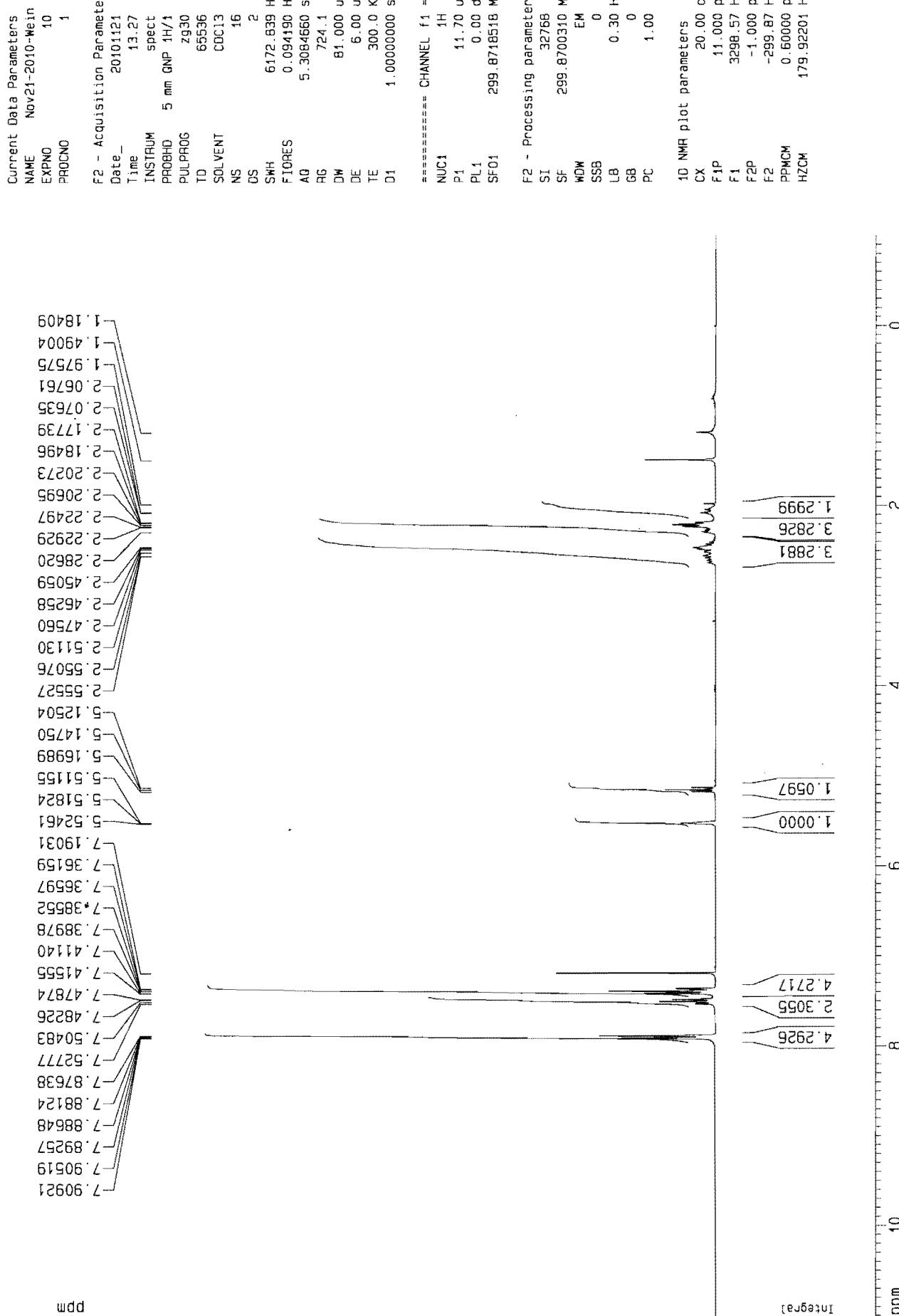
CX 20.00 cm
F1P 215.000 ppm
F1 16211.50 Hz
F2P -5.000 ppm
F2 -75.01 Hz
PPMCM 11.00000 ppm/cm
H2CM 829.49578 Hz/cm

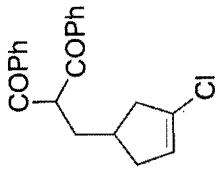


ppm

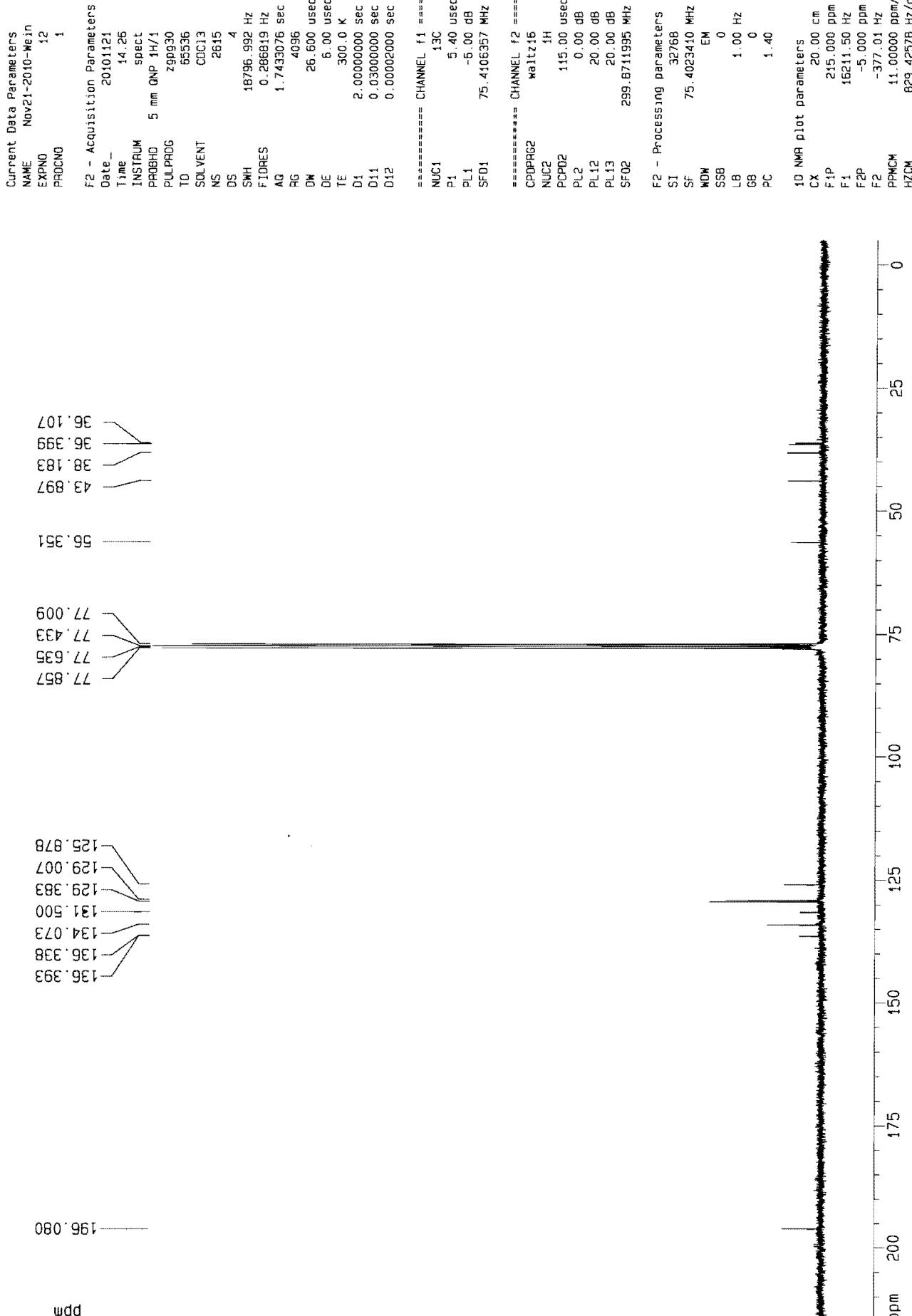
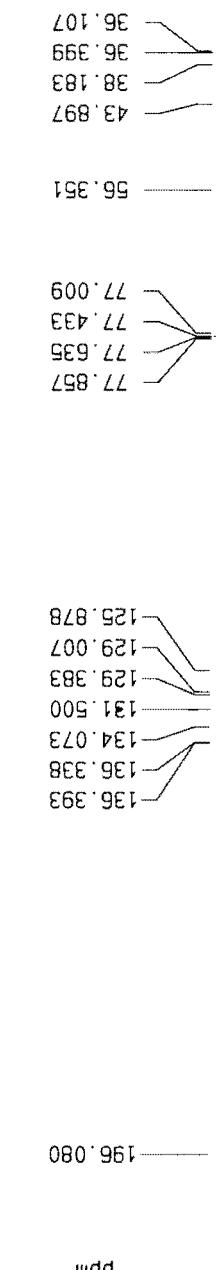


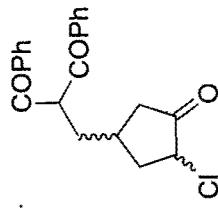
16a



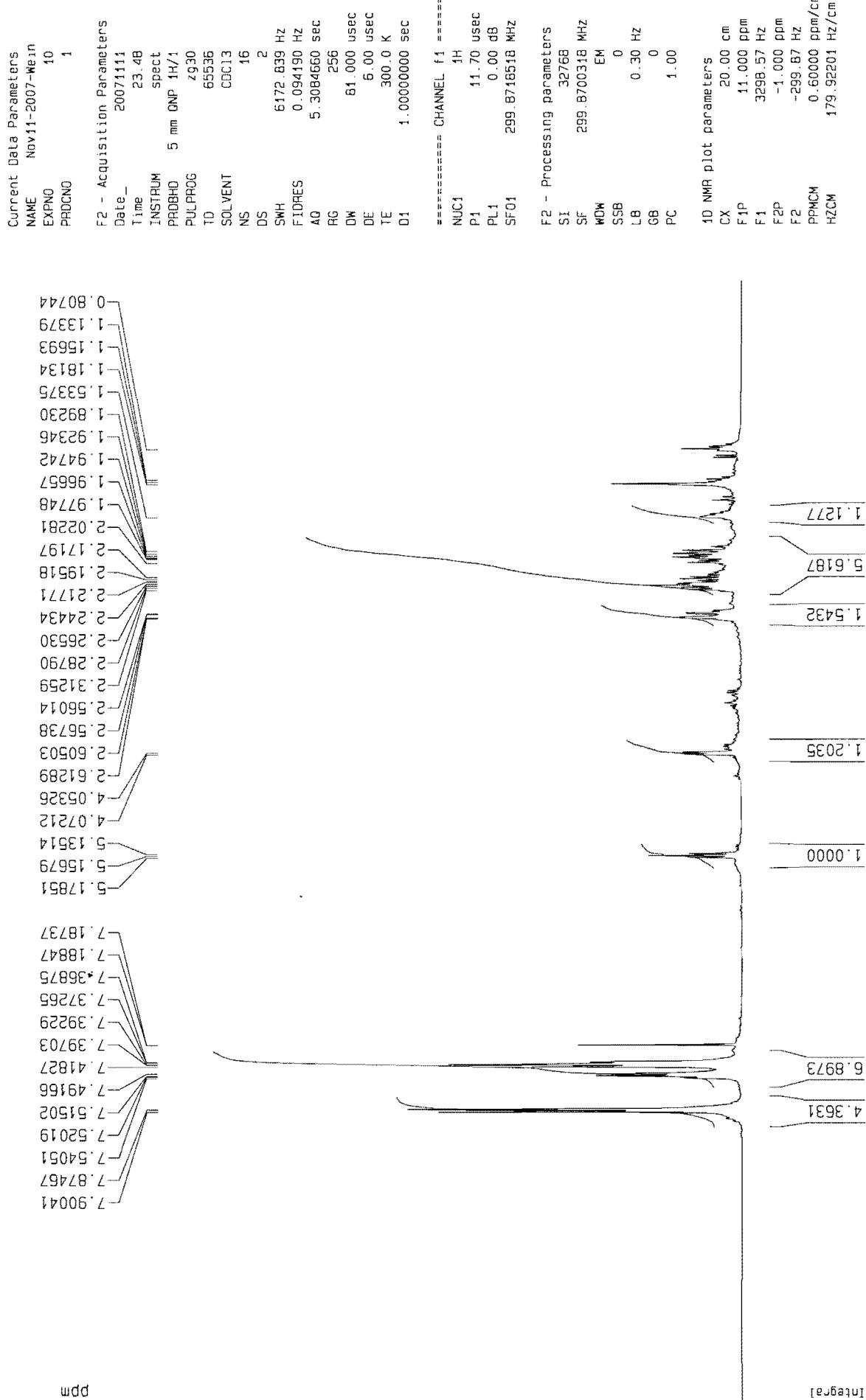


16a

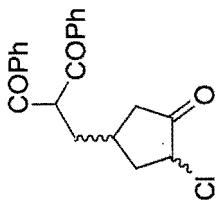




17a



ppm



17a

Current Data Parameters

NAME Nov11-2007-Wein
EXPND 12
PRCNO 1

F2 - Acquisition Parameters

Date .. 20071112
Time 9.36
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpp30
TO 65336
SOLVENT CDCl3
NS 9093
DS 4
SWH 18796.992 Hz
FIDRES 0.286849 Hz
AQ 1.7433076 sec
RG 512
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec
D11 0.0300000 sec
D12 0.0000200 sec

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

NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SF01 75.4106357 MHz

===== CHANNEL f2 =====

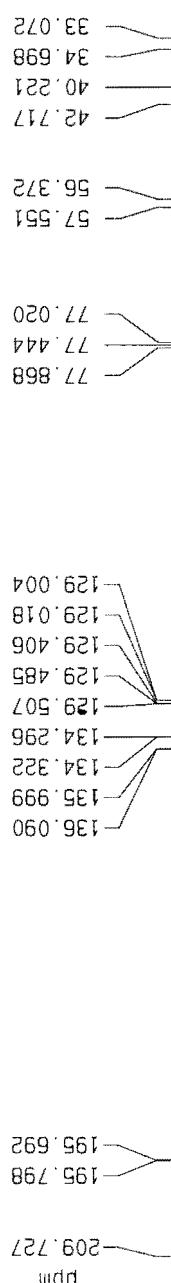
CPDPFG2
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SF02 299.8711995 MHz

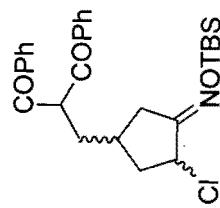
F2 - Processing Parameters

SI 32768
SF 75.4023410 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

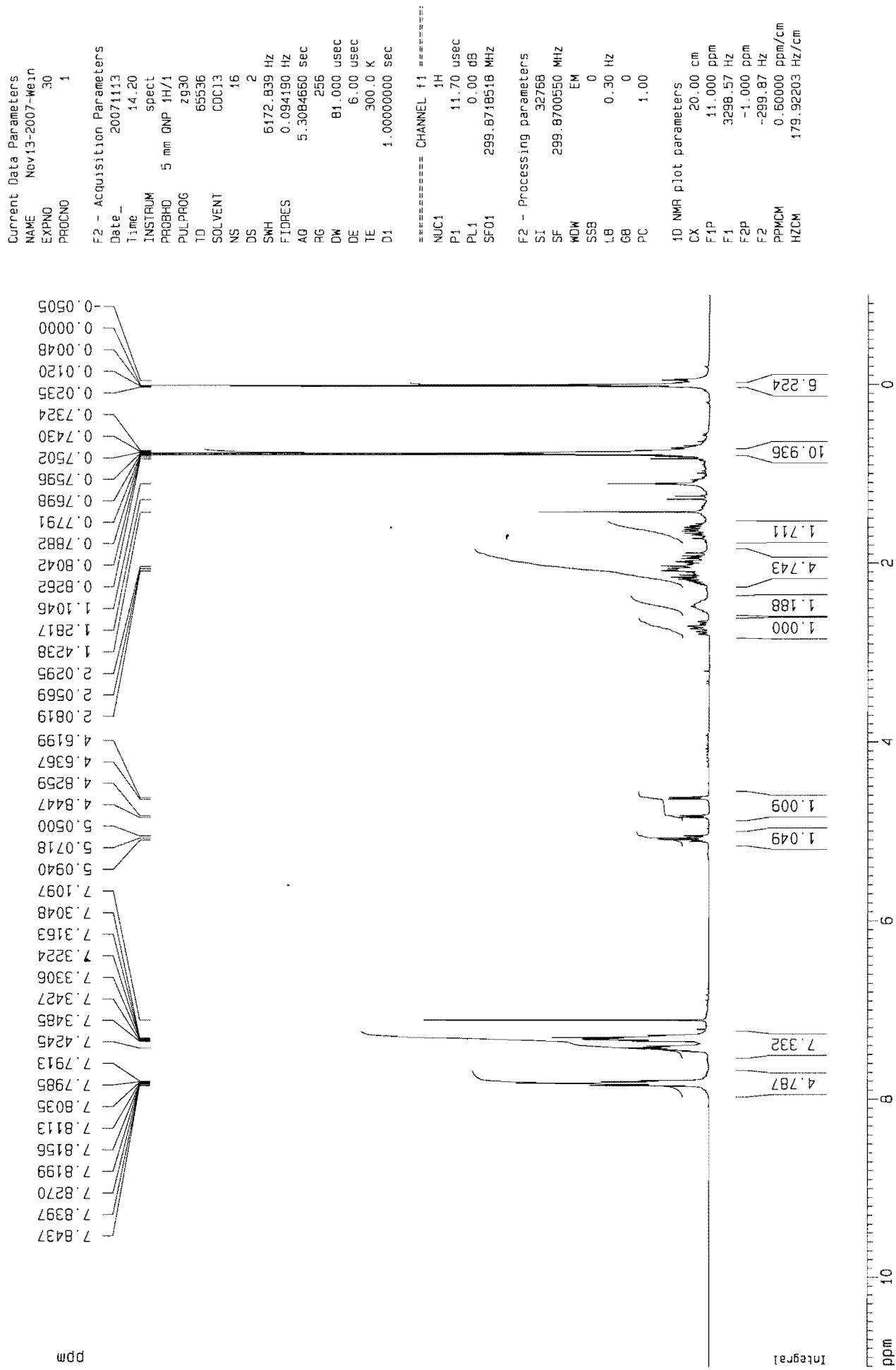
1D NMR plot parameters

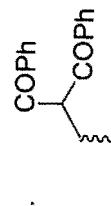
CX 20.00 cm
F1P 215.000 ppm
F1 162.1150 Hz
F2P -5.000 ppm
F2 -377.01 Hz
PPCM 11.00000 ppm/cm
HZCM 829.42578 Hz/cm



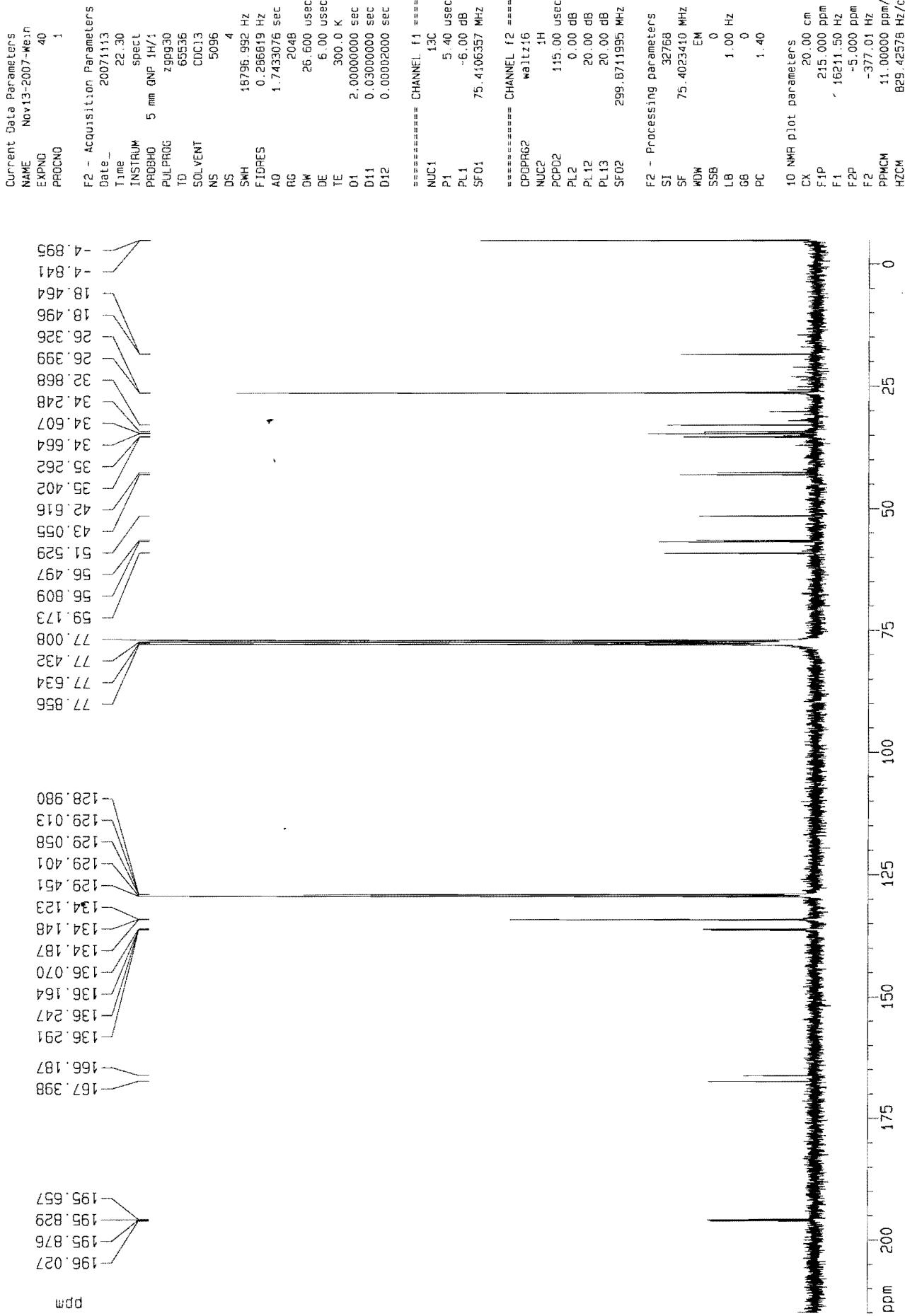


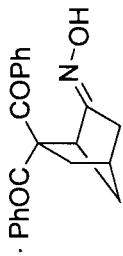
18a



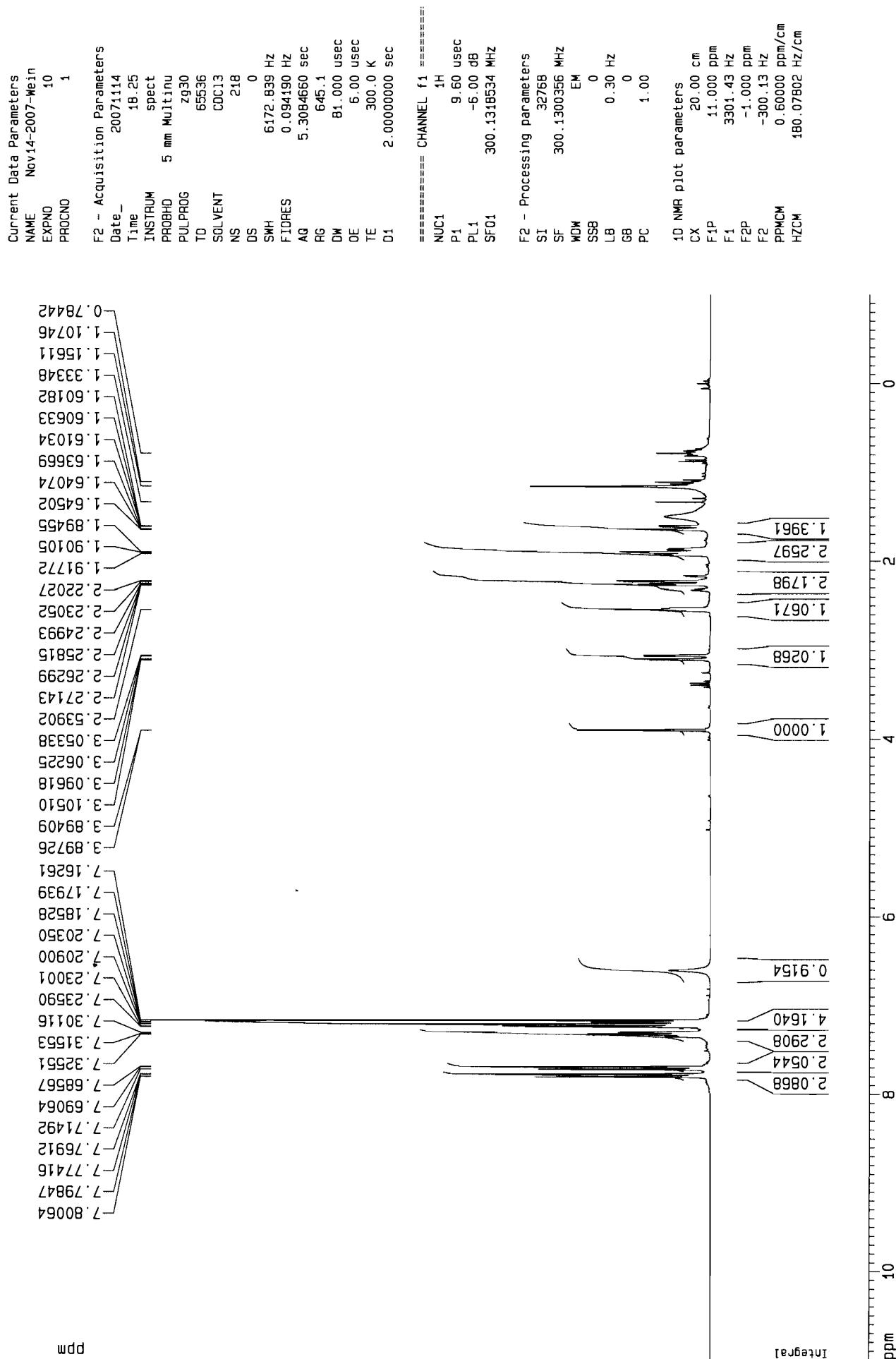


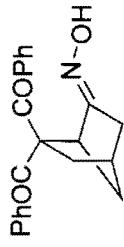
18a



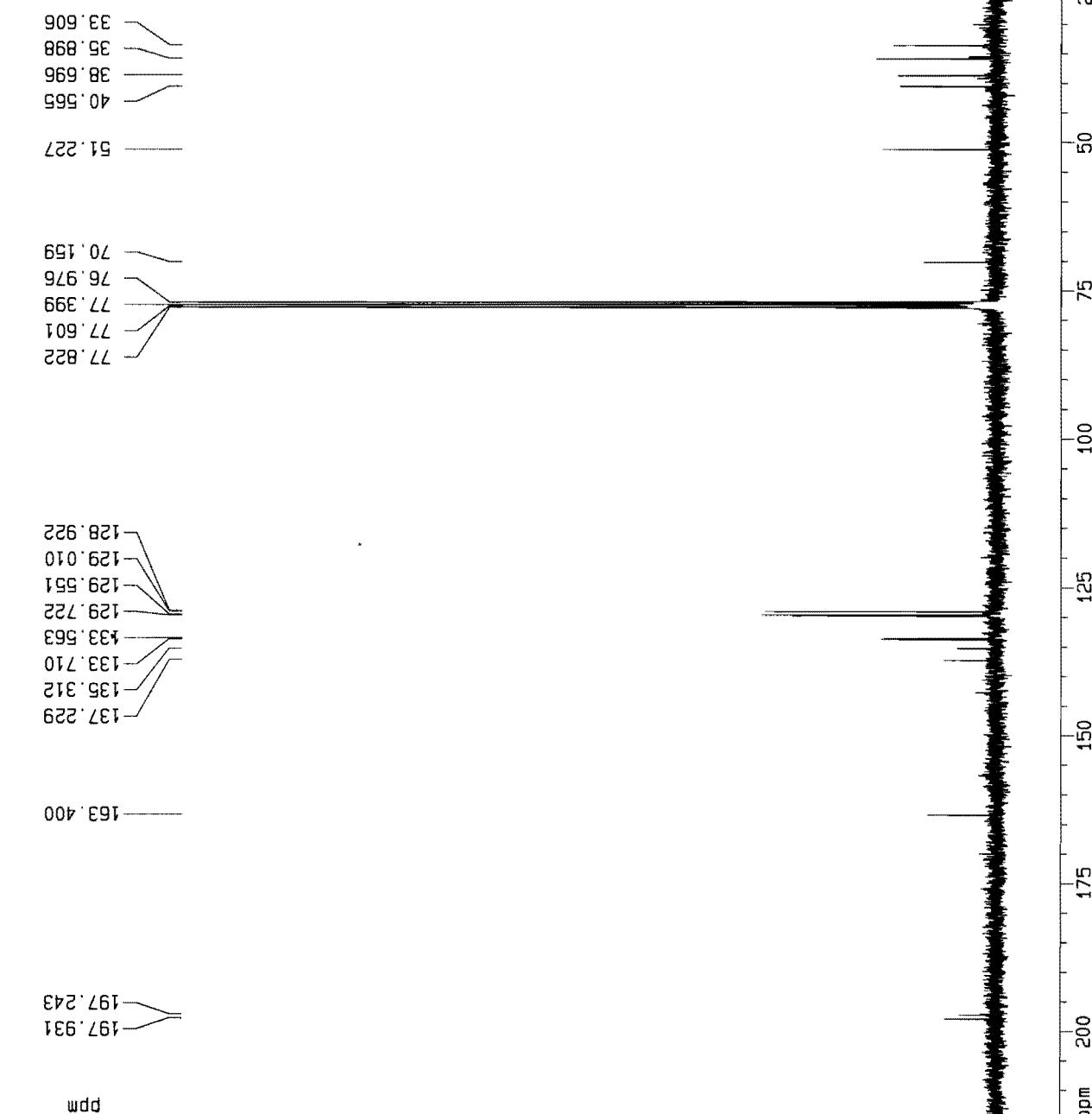


19





19



Current Data Parameters

NAME	Nov14-2007-Mein
EXPNO	12
PROCNO	1

F2 - Acquisition Parameters

Date	2007/11/14
Time	20:16
INSTRUM	spec
PROBHD	5 mm Multinu
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl ₃
NS	2339
DS	4
SWH	18832.393 Hz
FIDRES	0.287360 Hz
AQ	1.740308 sec
RG	16384
DW	26.550 usec
DE	6.00 usec
TE	300.0 K
D1	2.0000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

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

NUC1	13C
P1	11.80 usec
PL1	0.00 dB
SFO1	75.4760200 MHz

===== CHANNEL f2 =====

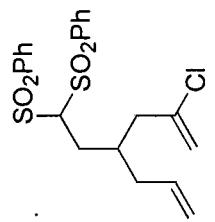
CPDPAGE2	Mertz16
NUC2	1H
PCPD2	110.00 usec
PL2	0.00 dB
PL12	17.50 dB
PL13	17.50 dB
SF02	300.1312005 MHz

F2 - Processing parameters

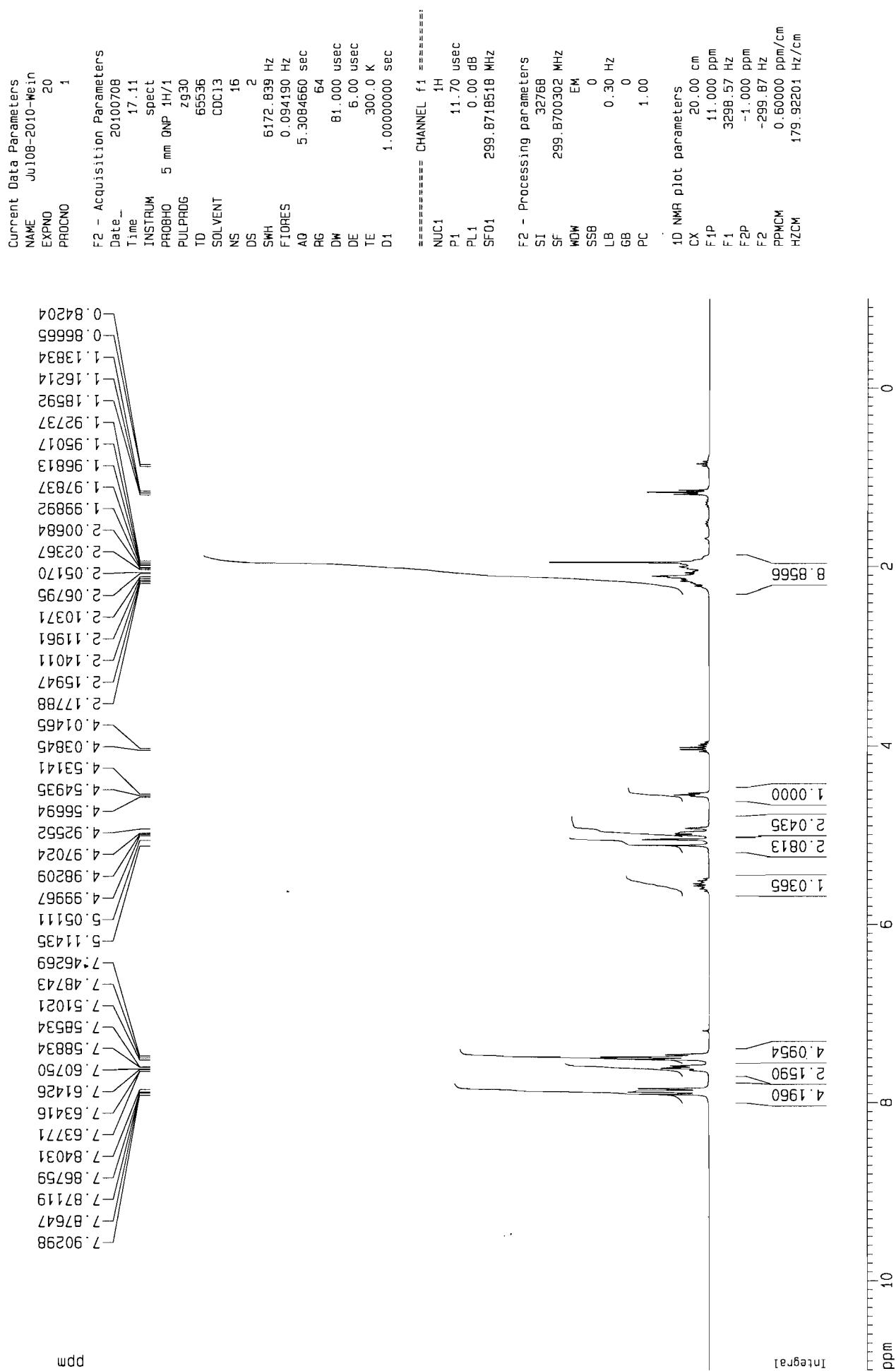
SI	32768
SF	75.4677190 MHz
SWW	EM
SSB	0
LB	1.00 Hz
6B	0
PC	1.40

1D NMR pilot parameters

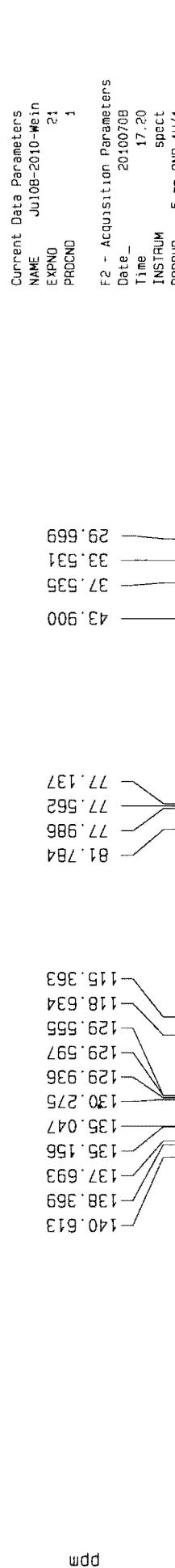
CX	20.00 cm
F1P	215.000 ppm
F1	16225.56 Hz
F2P	-5.000 ppm
F2	-377.34 Hz
PPMCM	11.00000 ppm/cm
HZCM	830.14490 Hz/cm

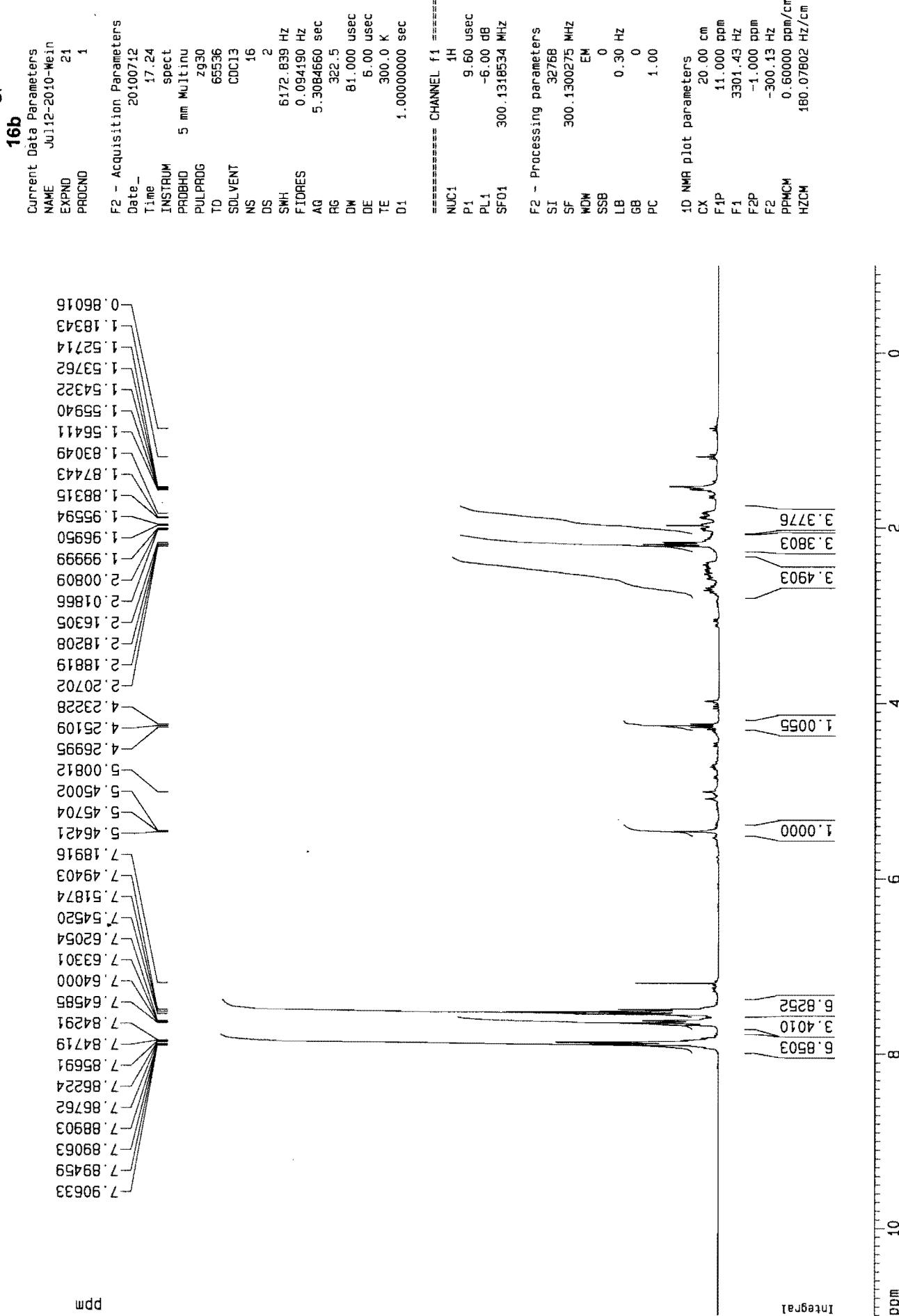
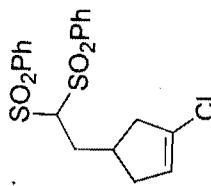


15b



15b





16b

Current Data Parameters

NAME Jul12-2010-Wein
EXPNO 31
PROCNO 1

F2 - Acquisition Parameters

Date_ 2010/07/12
Time_ 19:06
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpp30
TD 65536
SOLVENT1 CDCl3
NS 553
DS 4
SWH 18796.592 Hz
FIDRES 0.286519 Hz
AQ 1.743376 sec
RG 2048
DW 26.00 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec
D11 0.0300000 sec
D12 0.00002000 sec

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

NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SF01 75.4106357 MHz

===== CHANNEL f2 =====

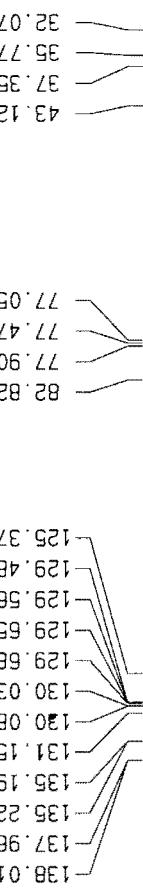
CPDPG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SF02 299.8711995 MHz

F2 - Processing parameters

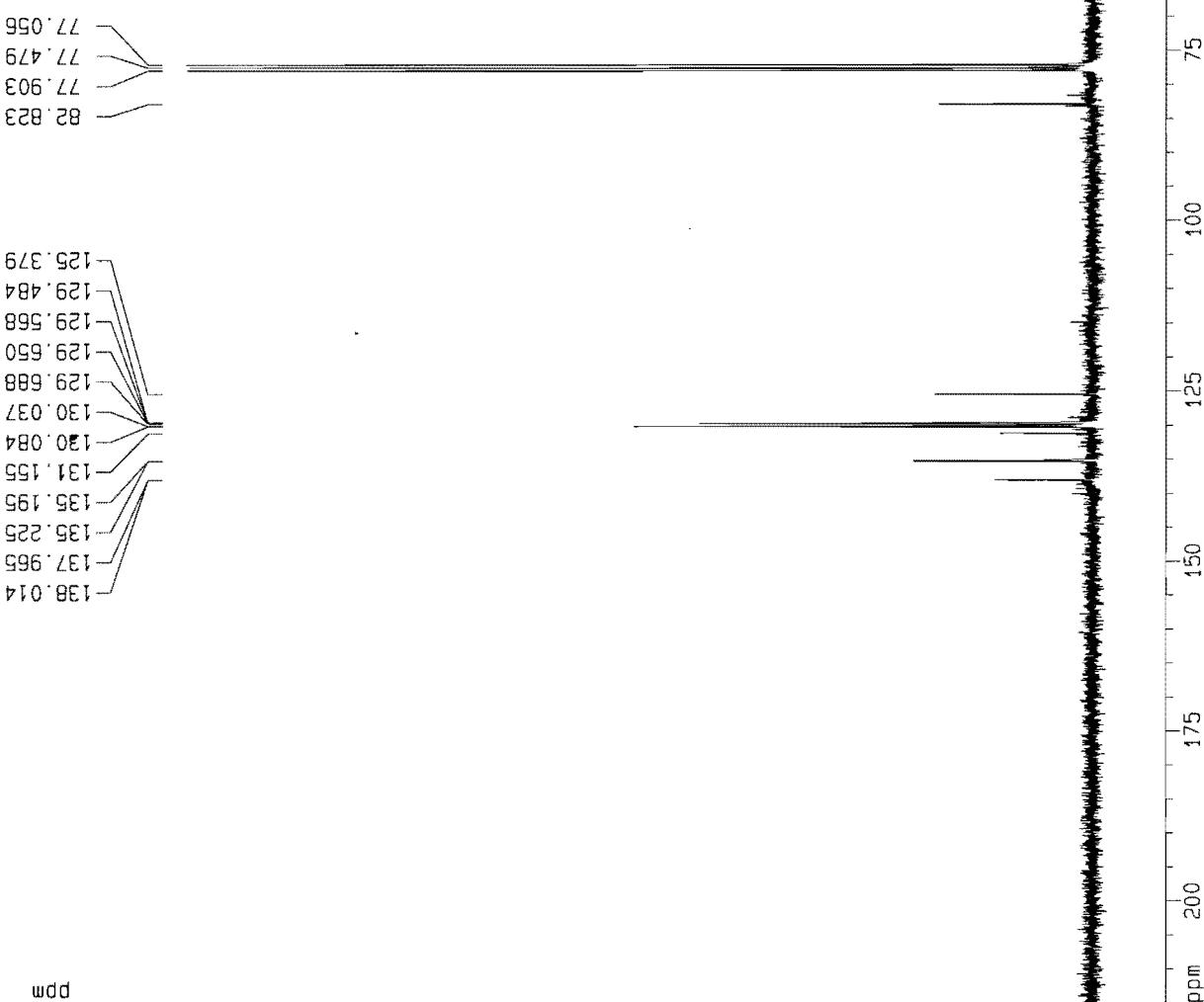
S1 32768
SF 75.402310 MHz
WM EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

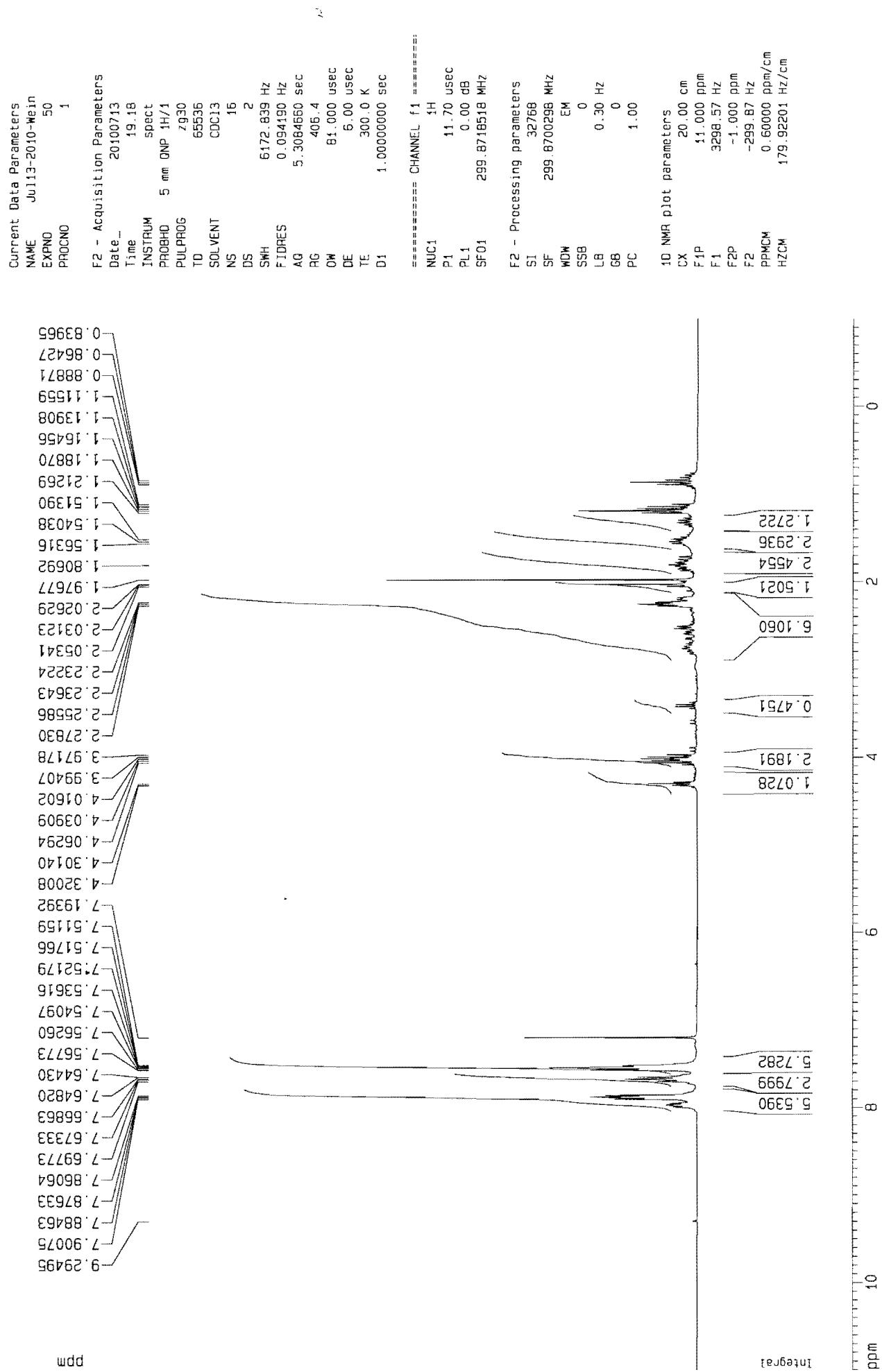
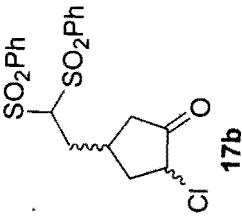
1D NMR pilot parameters

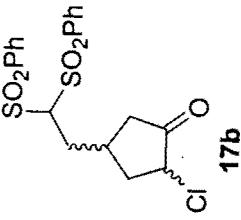
CX 20.00 cm
F1P 215.000 ppm
F1 16.11.50 Hz
F2P -5.000 ppm
F2 -377.01 Hz
PPMCH 11.00000 ppm/cm
HCCM 829.42578 Hz/cm



ppm







Current Data Parameters
 NAME JU113-2010-Mein
 EXPNO 52
 PROCN0 1

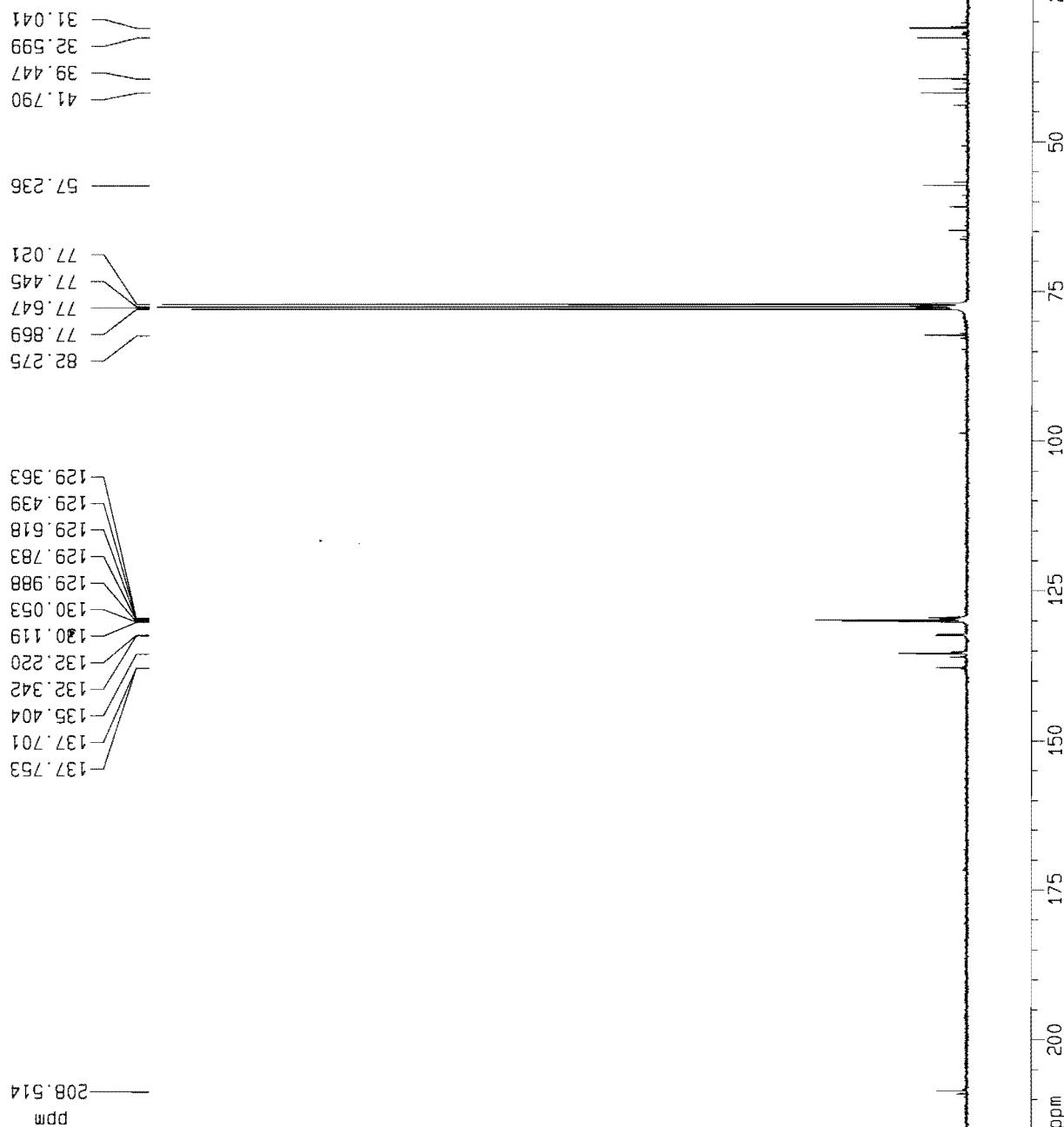
F2 - Acquisition Parameters
 Date_ 20100113
 Time 20:38
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zgpp30
 TO 65536
 SOLVENT CDCl3
 NS 1257
 DS 4
 SWH 18796.992 Hz
 FIDRES 0.286519 Hz
 AQ 1.7433076 sec
 RG 2048
 DW 600 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 D12 0.0000200 sec

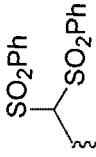
===== CHANNEL f1 =====:
 NUC1 13C
 P1 5.40 usec
 PL1 -6.00 dB
 SF01 75.4106357 MHz

===== CHANNEL f2 =====:
 CPDPBG2 walt16
 NUC2 1H
 PCPD2 115.00 usec
 PL2 0.00 dB
 PL12 20.00 dB
 PL13 20.00 dB
 SF02 299.8711995 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4023410 MHz
 NDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

10 NMR print parameters
 CX 20.00 cm
 F1P 215.000 ppm
 F1 16511.50 Hz
 F2P -5.000 ppm
 F2 -377.01 Hz
 PPMCM 11.00000 ppm/cm
 HZCM 829.42578 Hz/cm





NOTES

18b

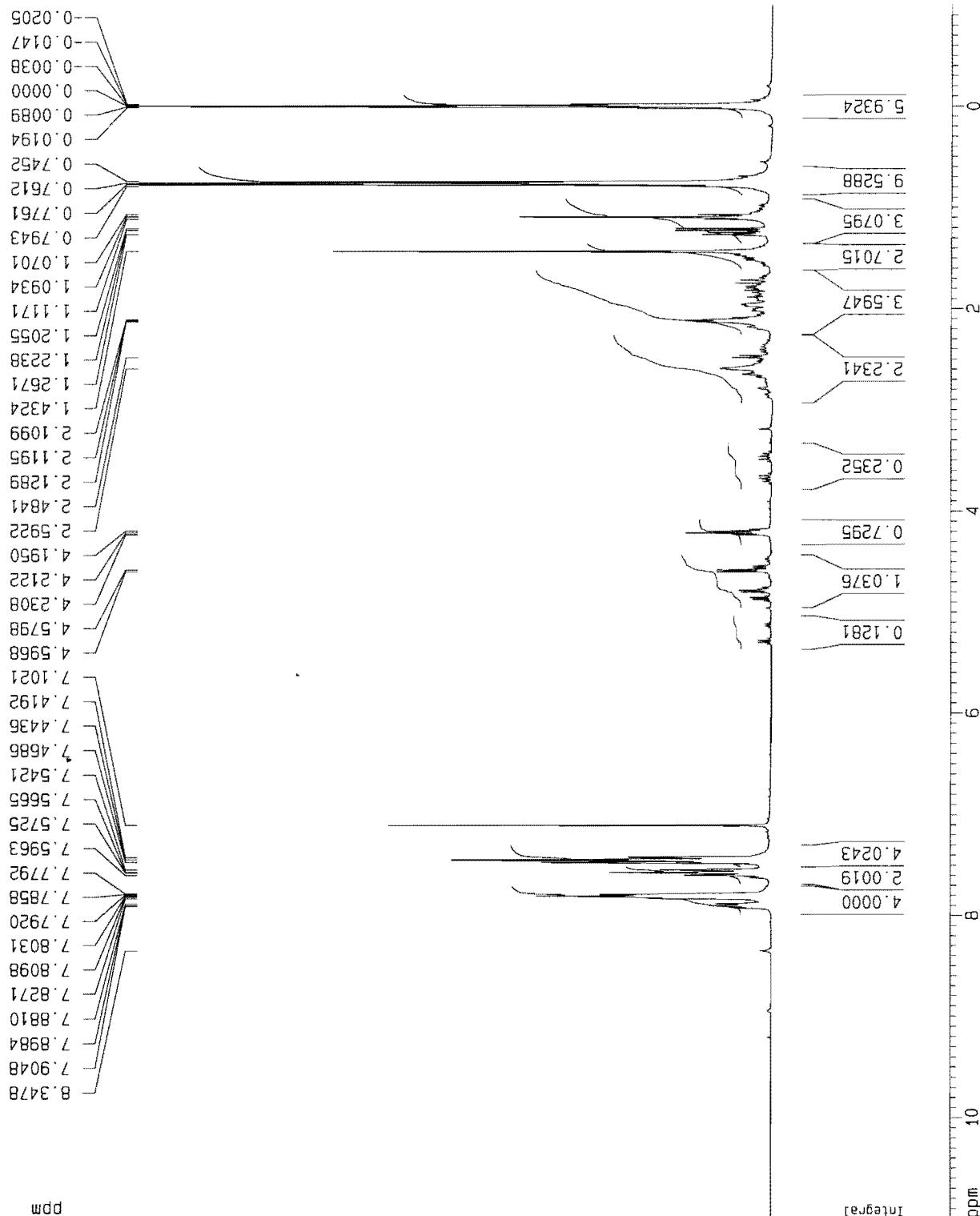
Current Data Parameters
NAME Jul15-2010-Wein
EXPNO 20
PROCNO 1

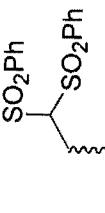
F2 - Acquisition Parameters
Date_ 20100715
Time_ 16.54
INSTRUM Spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 362
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.0000000 sec

===== CHANNEL f1 =====
NUC1 1H
SI 32768
SF 299.8700576 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

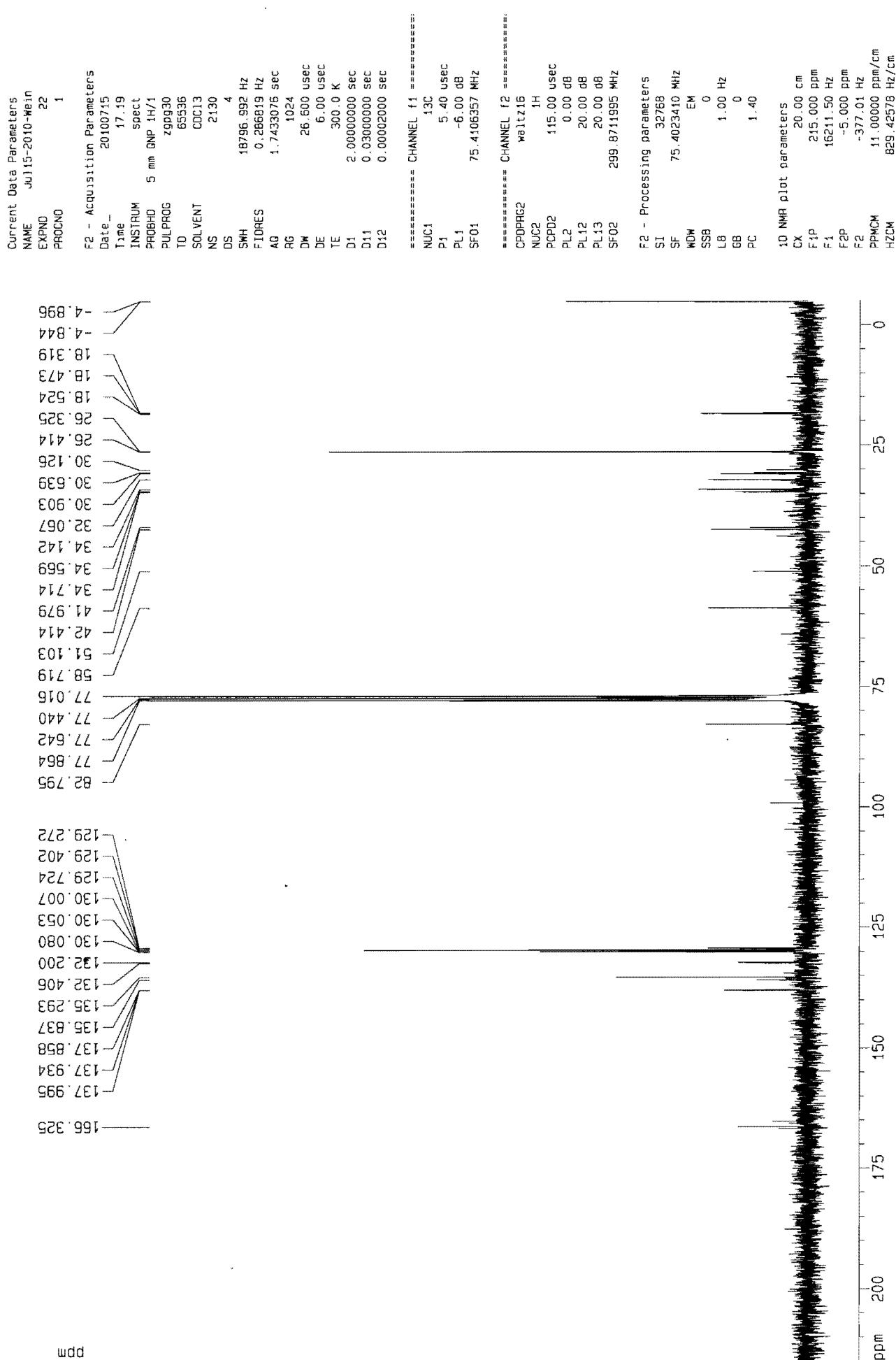
F2 - Processing parameters
SI 32768
SF 299.8700576 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

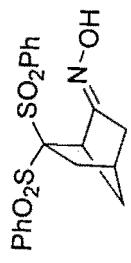
1D NMR pilot parameters
CX 20.00 cm
F1P 11.000 ppm
F1 3298.57 Hz
F2P -1.000 ppm
F2 -299.87 Hz
PPMCM 0 ppm/cm
H2DM 179.92203 Hz/cm





18b





20

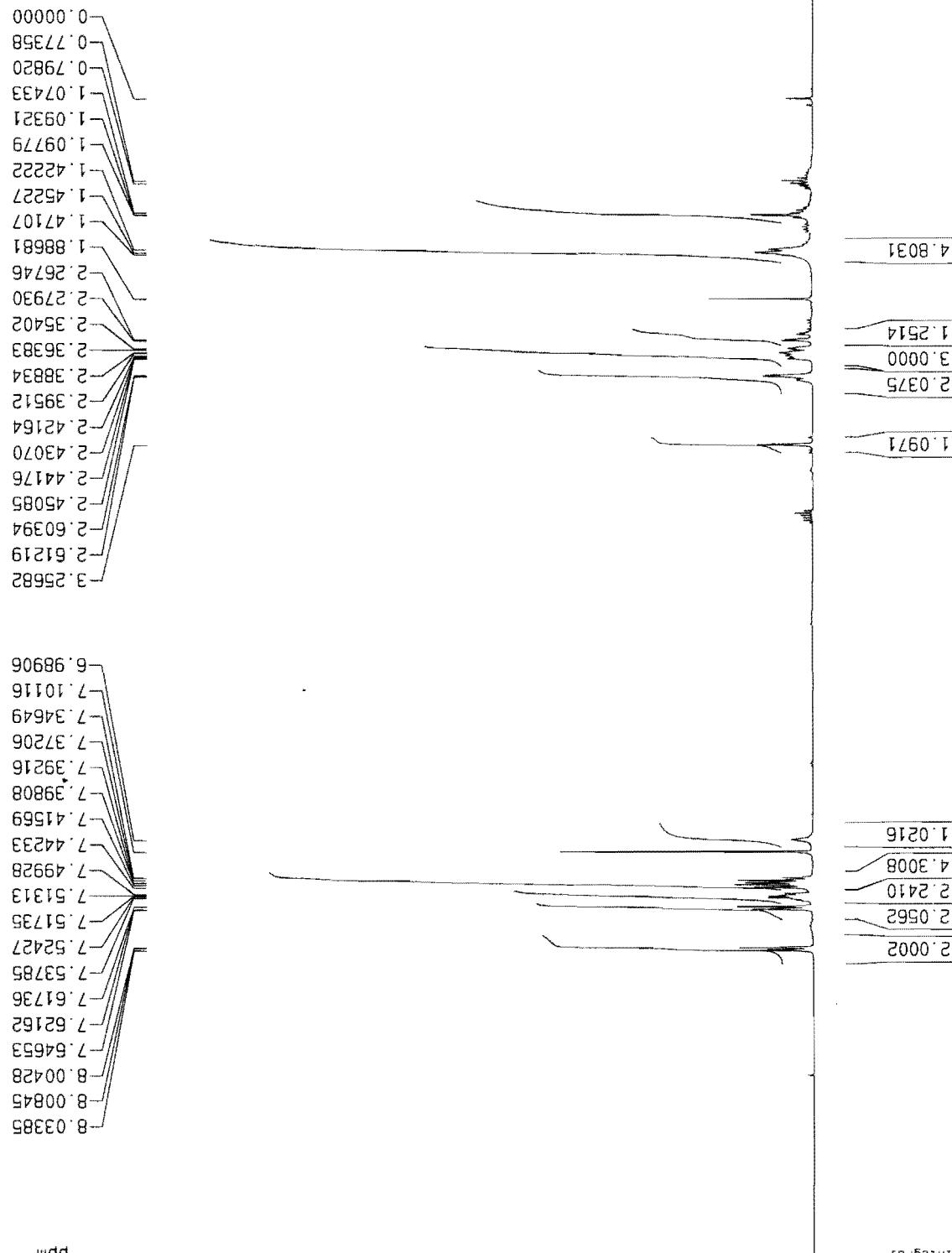
Current Data Parameters
 NAME Jul16-2010-Wein
 EXPNO 10
 PROCND 1

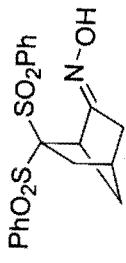
F2 - Acquisition Parameters
 Date 20100716
 Time 18.21
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG PULPROG
 TD 65536
 SOLVENT CDCl3
 NS 36
 DS 0
 SWH 6172.839 Hz
 FIDRES 0.091190 Hz
 AQ 5.3084660 sec
 RG 724.1
 DW 81.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 11.70 usec
 PL1 0.00 dB
 SF01 299.8718518 MHz

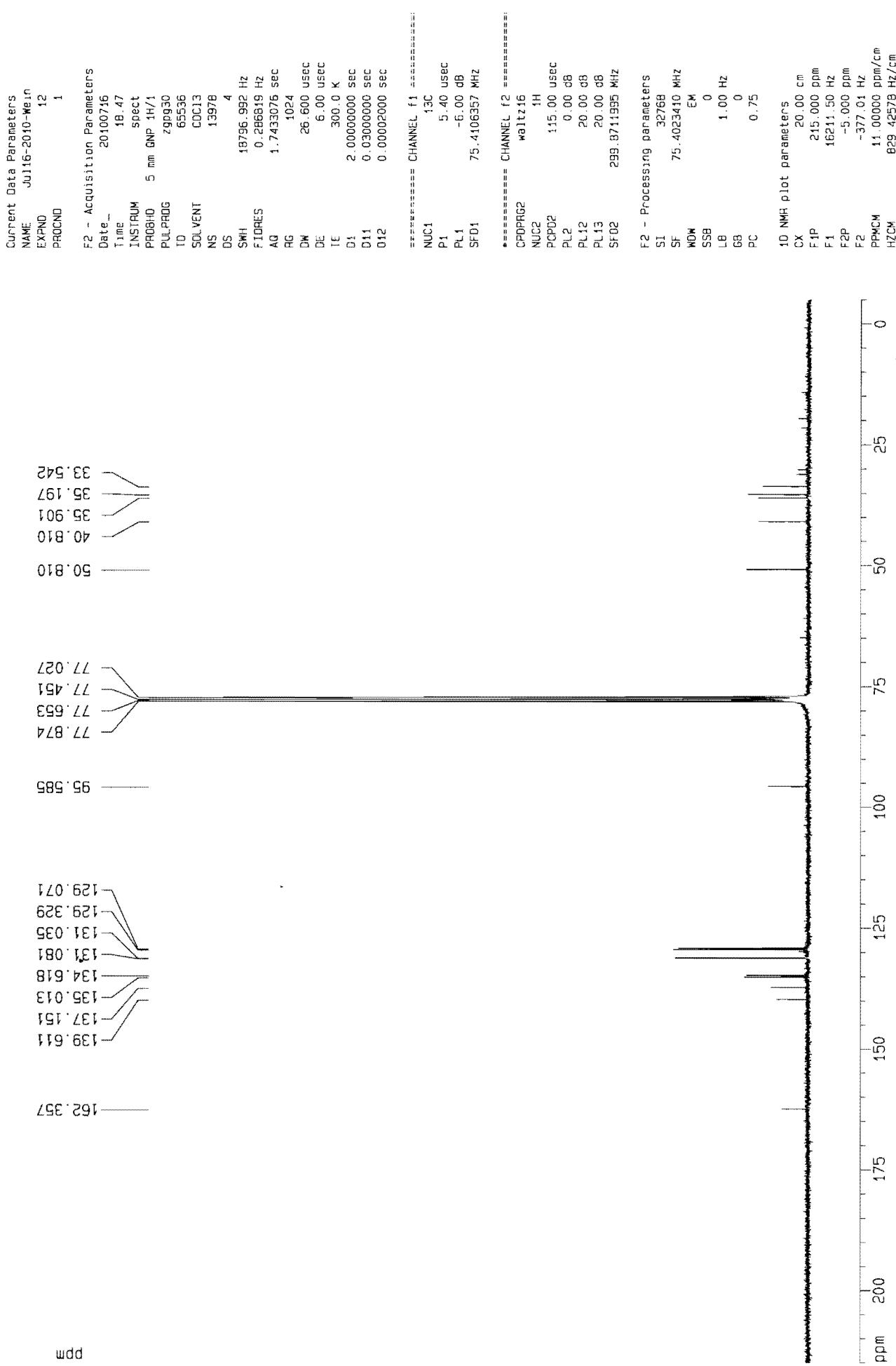
F2 - Processing parameters
 SI 32768
 SF 299.8700576 MHz
 WM EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

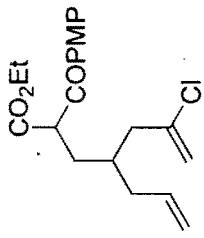
1D NMR plot parameters
 CX 20.00 cm
 F1P 11.000 ppm
 F1 3298.57 Hz
 F2P -1.000 ppm
 F2 -299.87 Hz
 PPMCM 0.66000 ppm/cm
 HZCM 179.92203 Hz/cm



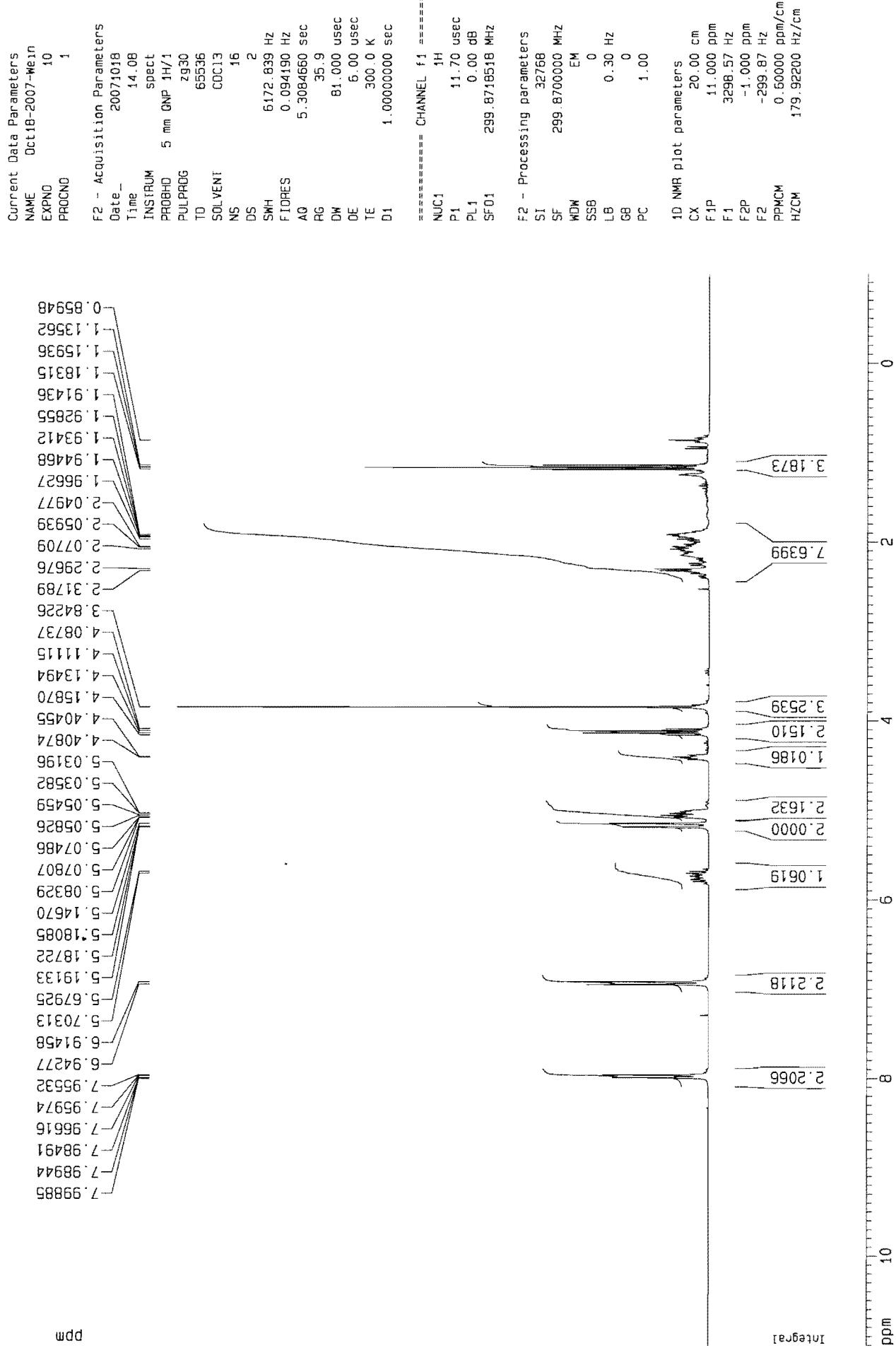


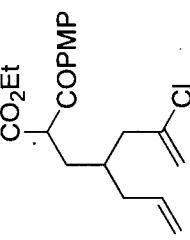
20



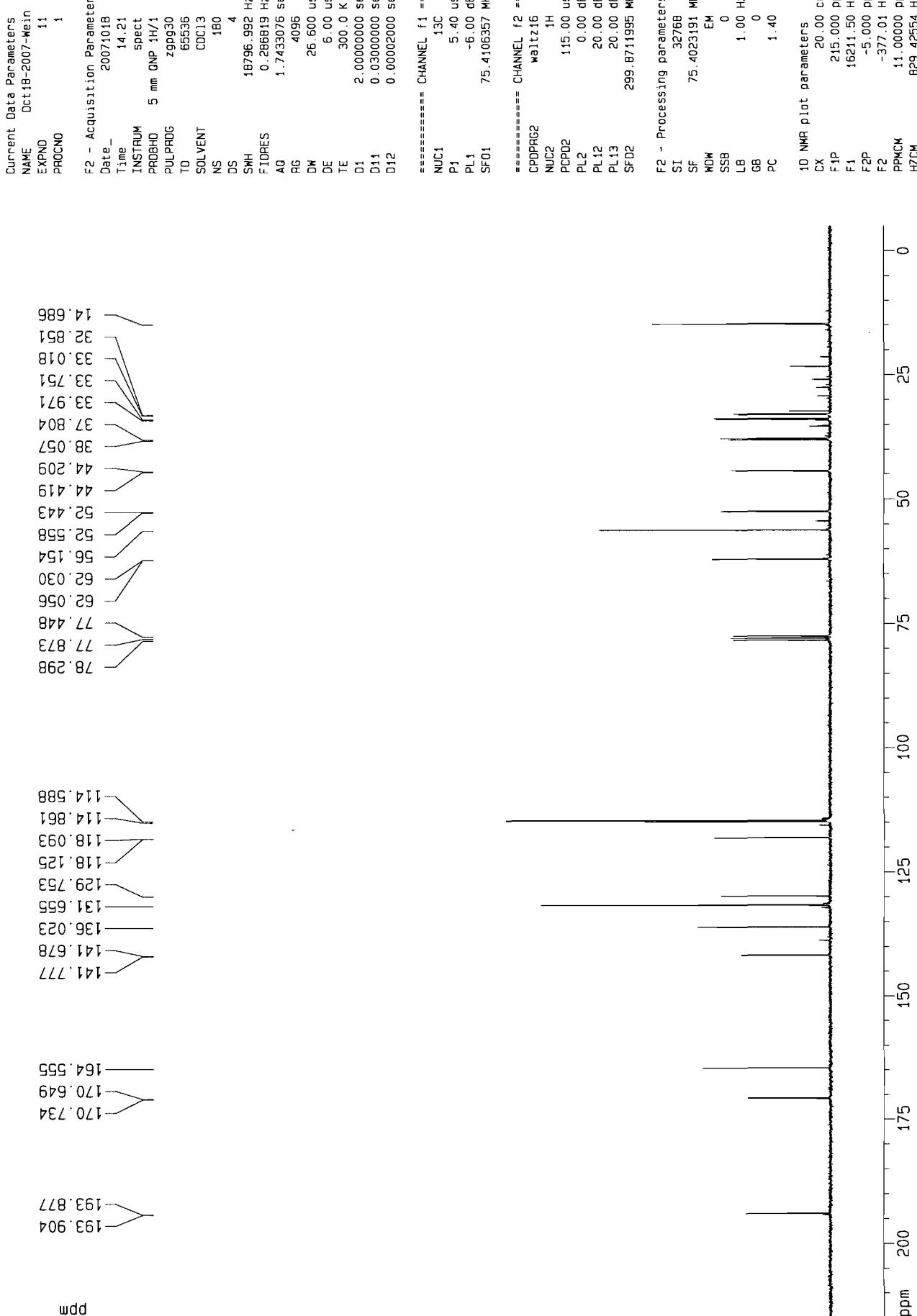


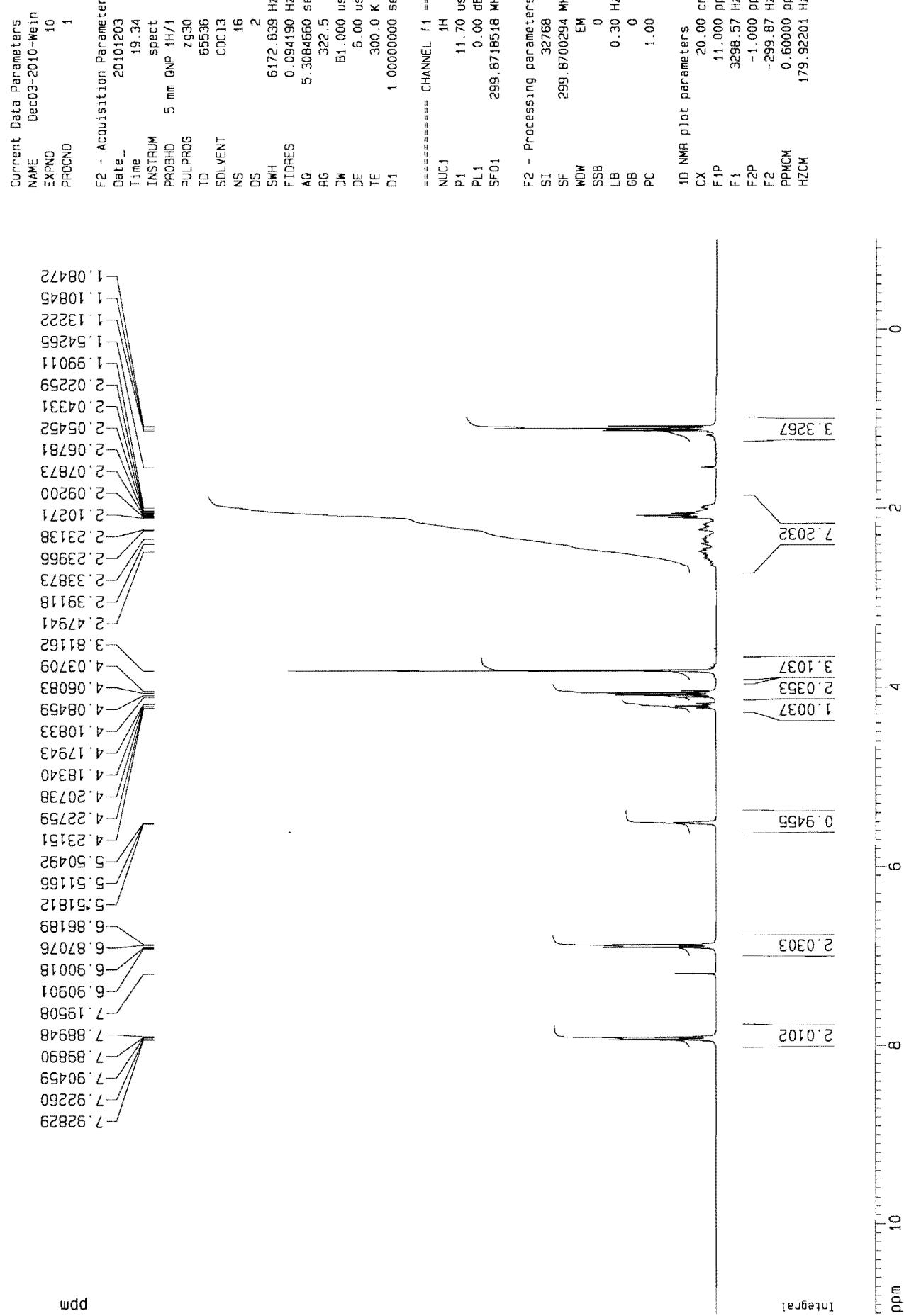
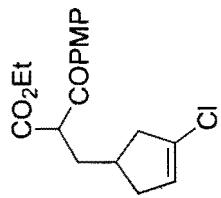
15c

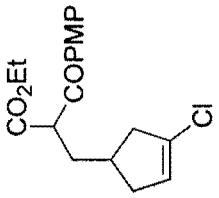




15c







16c

Current Data Parameters
NAME Dec03-2010-Mein
EXPNO 12
PROCND 1

F2 - Acquisition Parameters
Date 2010/03/20
Time 20:37
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg3030
TD 65336
SOLVENT CDCl3
NS 12867
DS 4
SWH 18796.992 Hz
FIDRES 0.286619 Hz
AQ 1.7433076 sec
RG 8192
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
T1 2.0000000 sec
D1 0.03000000 sec
D11 0.00000000 sec
D12 0.00002000 sec

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

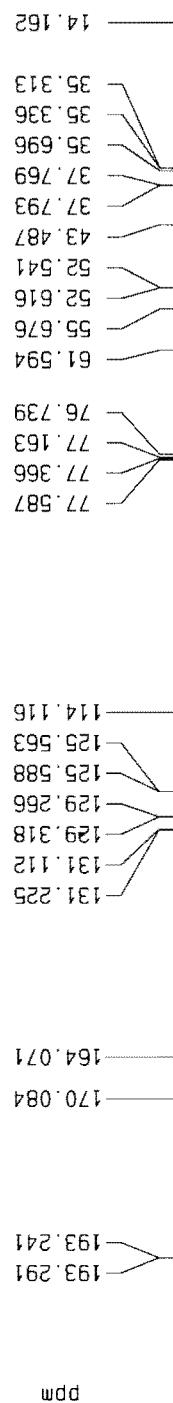
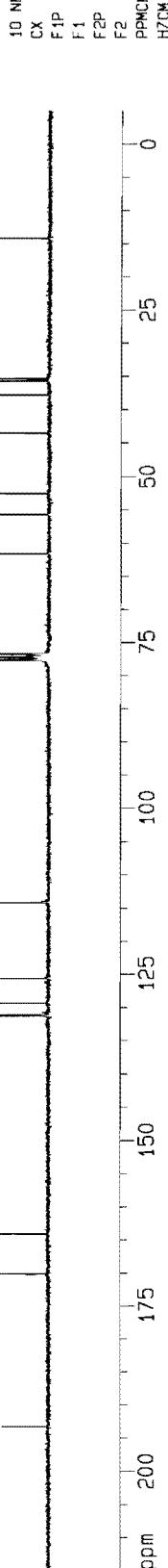
NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SF01 75.4106357 MHz

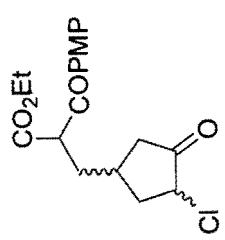
===== CHANNEL f2 =====

CPDPFG2 WALT16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SF02 299.8711995 MHz

F2 - Processing parameters

S1 326B
SF 75.402320 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





८

Current Data Parameters

NAME: NO. 22-5551, REC'D.
EXPNO: 10
RBOCNO: 1

The figure displays a proton NMR spectrum (f1) with the x-axis representing chemical shift in ppm, ranging from 0.00 to 10.00. The spectrum shows several peaks, with some being very sharp and others more broad. To the left of the spectrum, a vertical list of chemical shifts is provided, likely corresponding to the peaks. On the right side, two tables of parameters are shown: one for acquisition and one for processing.

F2 - Acquisition Parameters

EXPO	10
PRONO	1
Date	2001108
Time	17.00
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg30
TD	65536
SOLVENT	CDC13
NS	16
DS	2
SWH	6172.839 Hz
FLDRES	0.034190 Hz
AQ	5.3084650 sec
RG	203.2
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.0000000 sec

==== CHANNEL f1 =====

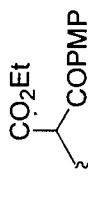
NUC1	1H
P1	11.70 usec
PL1	0.00 dB
SF01	299.8718518 MHz

F2 - Processing parameters

S1	32768
SF	299.8700287 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

10 NMR plot parameters

CX	20.00 cm
F1P	11.000 ppm
F1	3298.57 Hz
F2P	-1.000 ppm
F2	-299.87 Hz
PRMCM	0.60000 ppm/cm
HZCM	179.98201 Hz/cm



17c

Current Data Parameters

NAME Nov08-2007-Wein
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters

Date 2007/11/08
Time 17:23
INSTRUM spect
PROBHD 5 mm GNP 1H/1
PULPROG zg9930
TD 65536
SOLVENT COCl3
NS 4314
DS 4
SWH 18796.992 Hz
FIDRES 0.286819 Hz
AQ 1.7433076 sec
RG 4096
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec
D11 0.0300000 sec
D12 0.0000000 sec

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

NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SF01 75.4106357 MHz

===== CHANNEL f2 =====

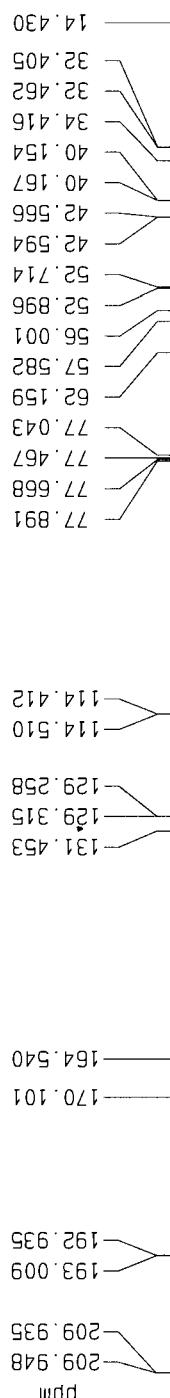
CP09PG2 Wallz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SF02 299.8711995 MHz

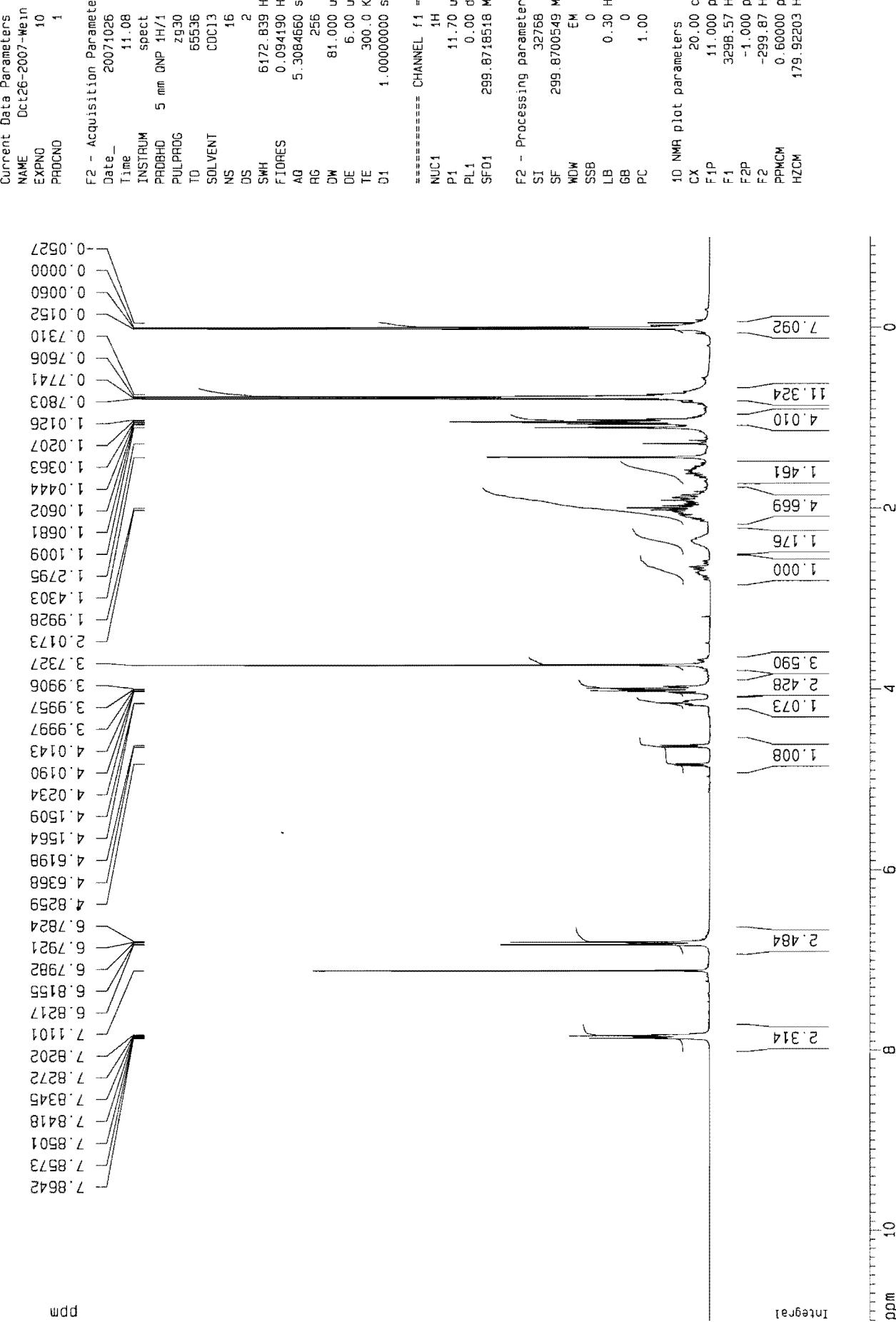
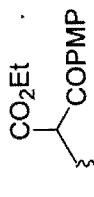
F2 - Processing parameters

SI 32768
SF 75.4023410 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

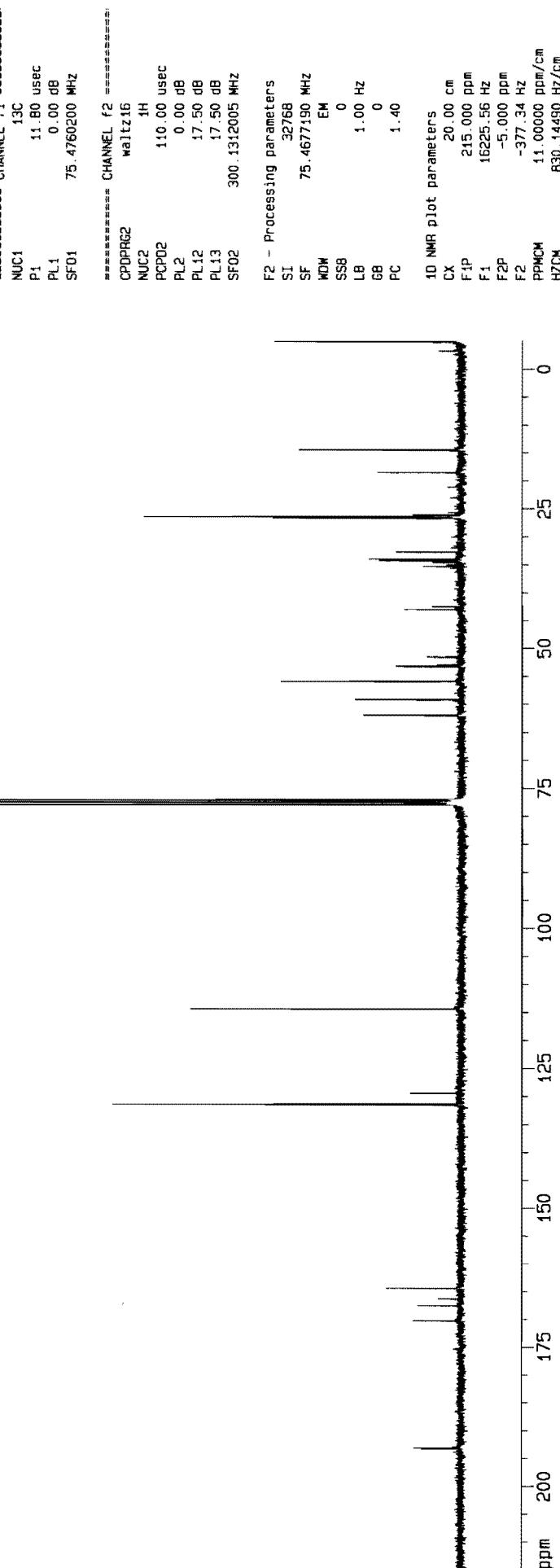
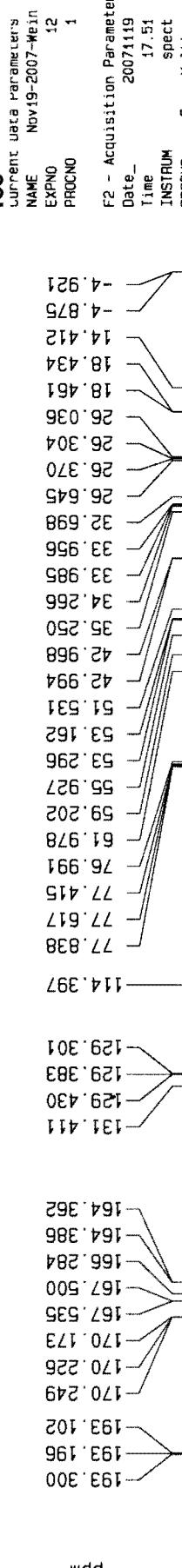
CX 20.00 cm
F1P 215.00 ppm
F1 16211.50 Hz
F2P -5.00 ppm
F2 -377.01 Hz
PPMCM 11.00000 ppm/cm
H2CM 829.42578 Hz/cm

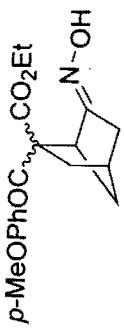




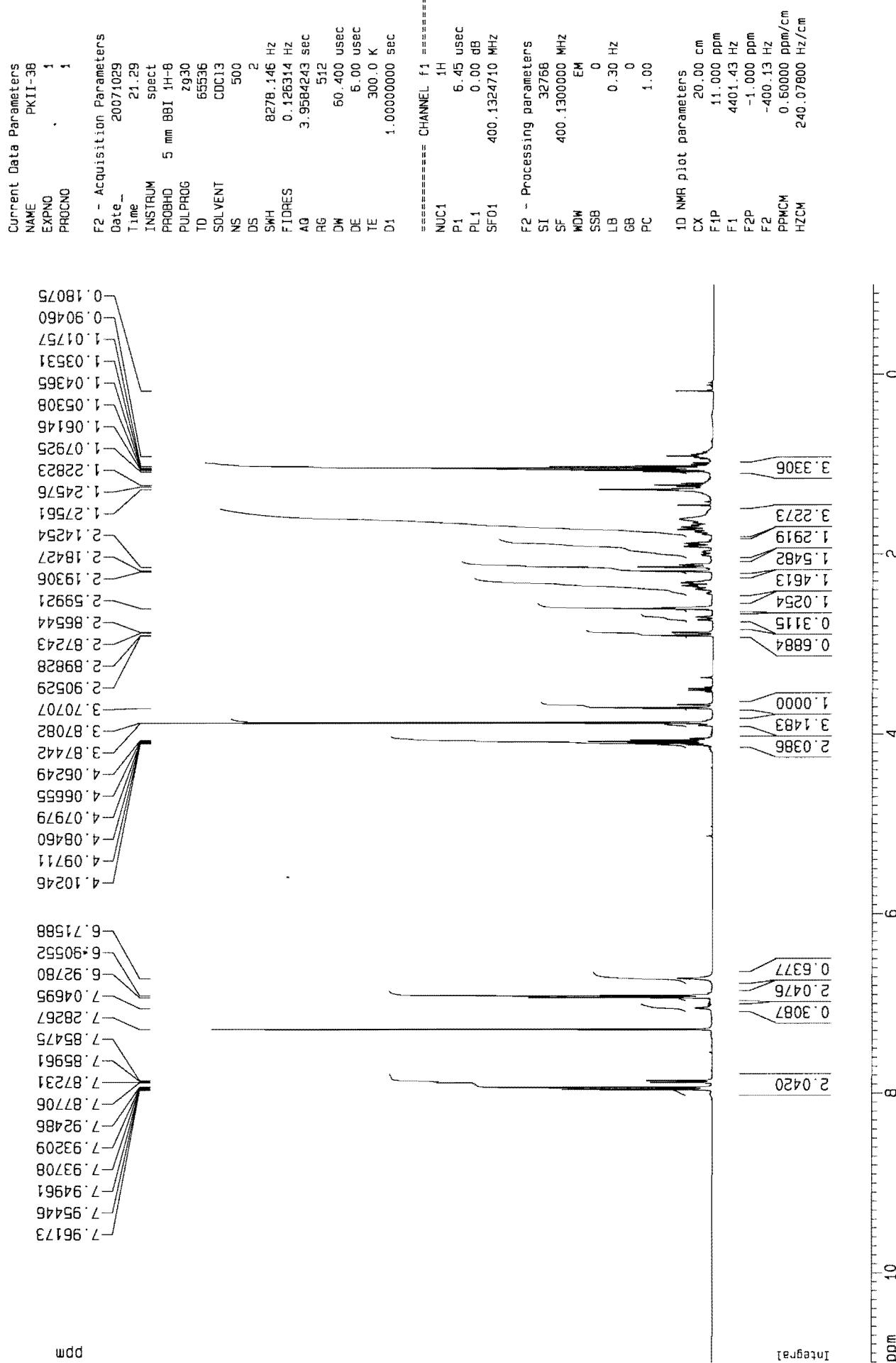


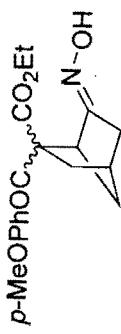
18c



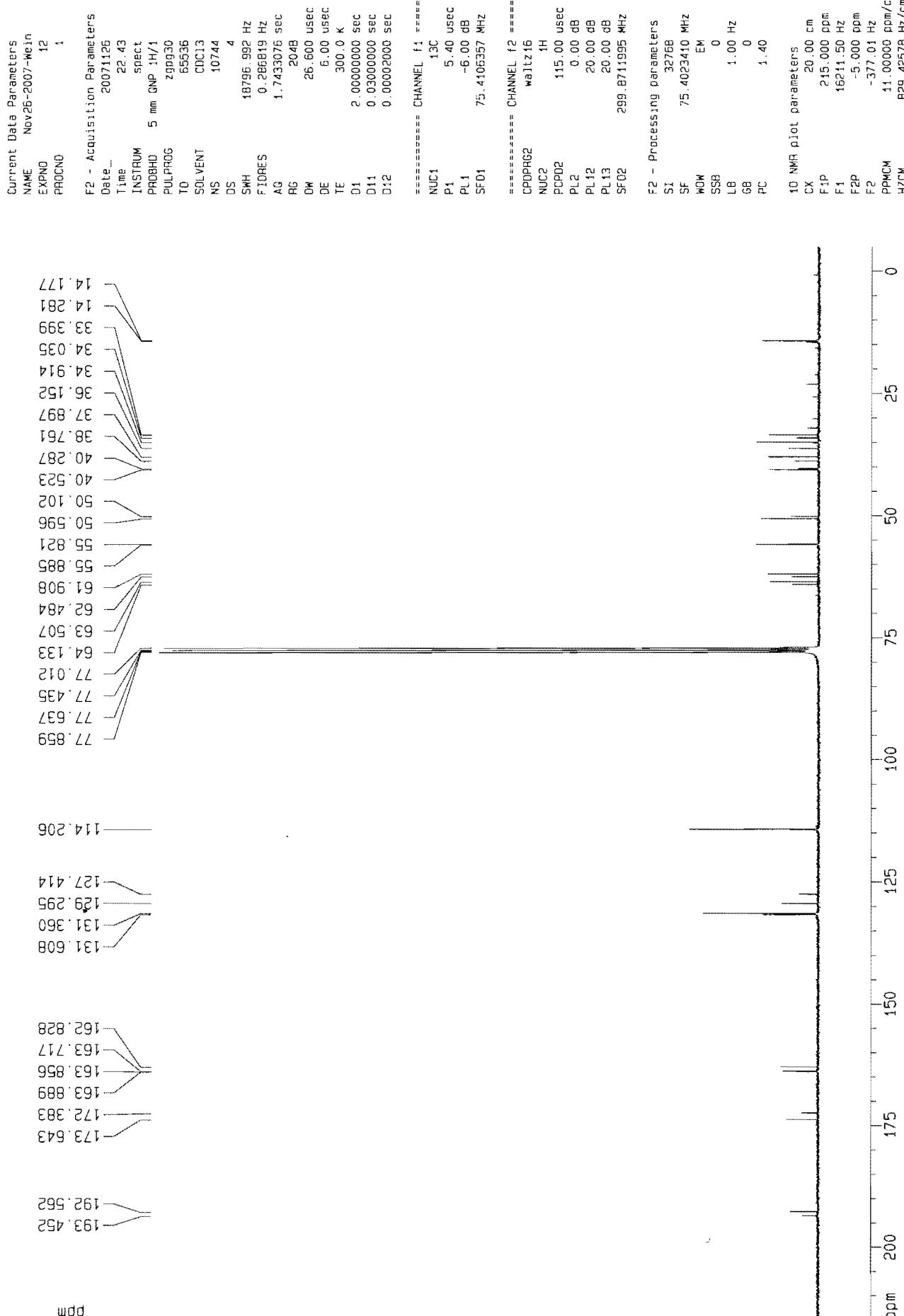


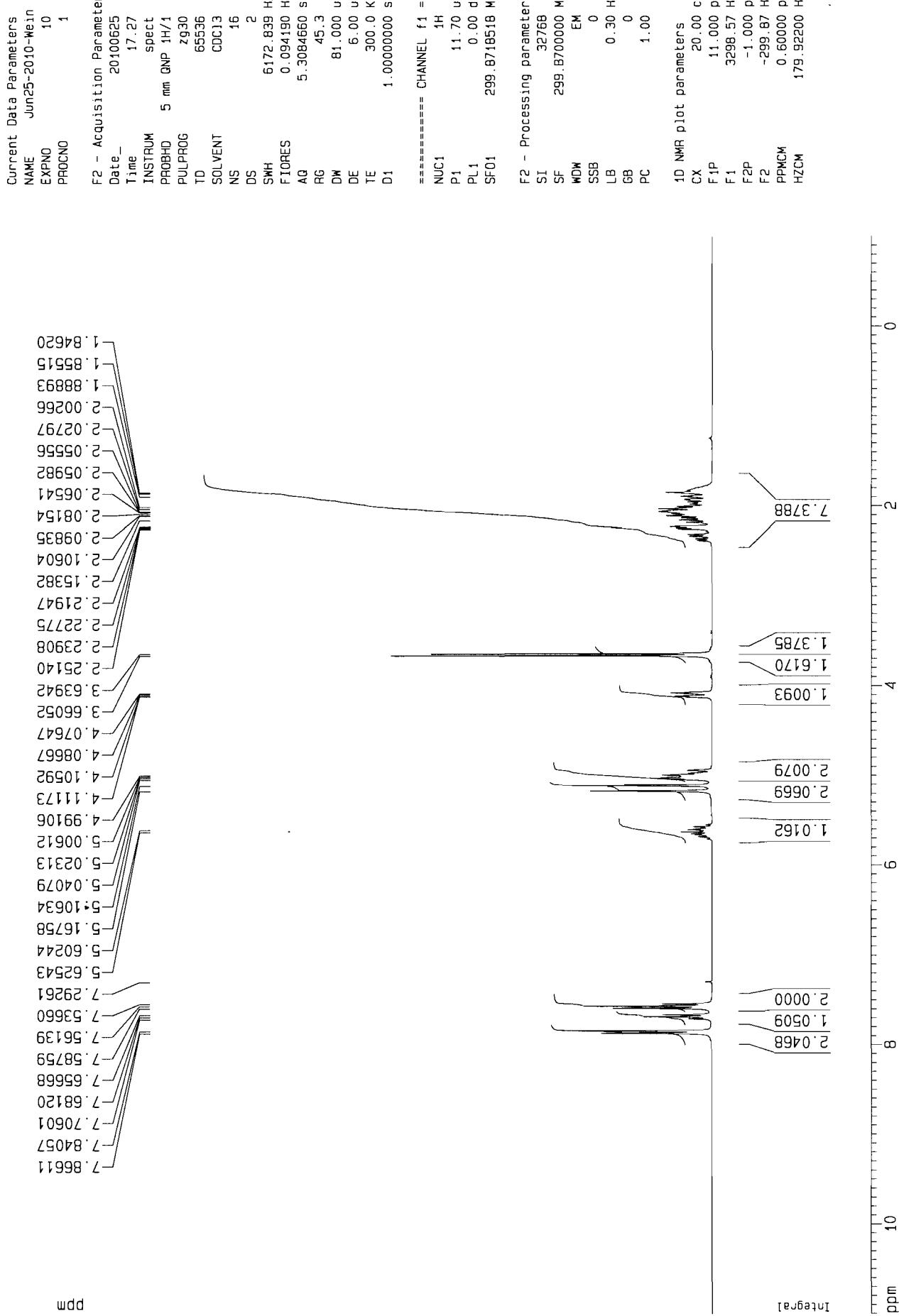
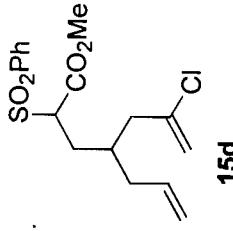
25

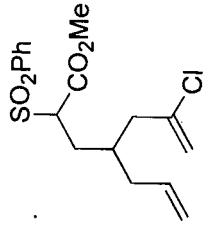




25

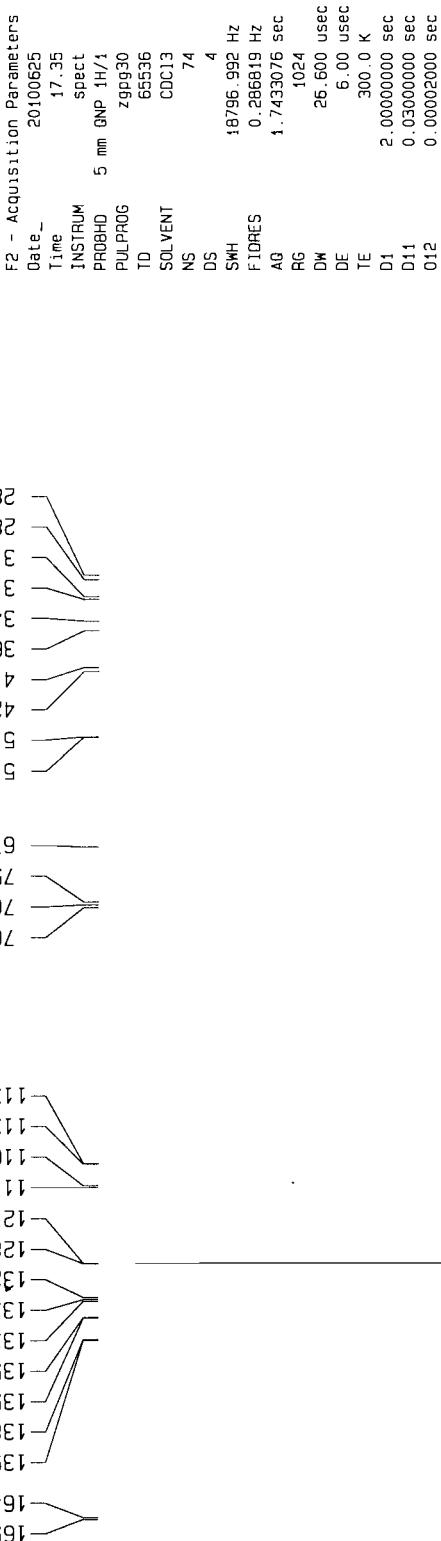




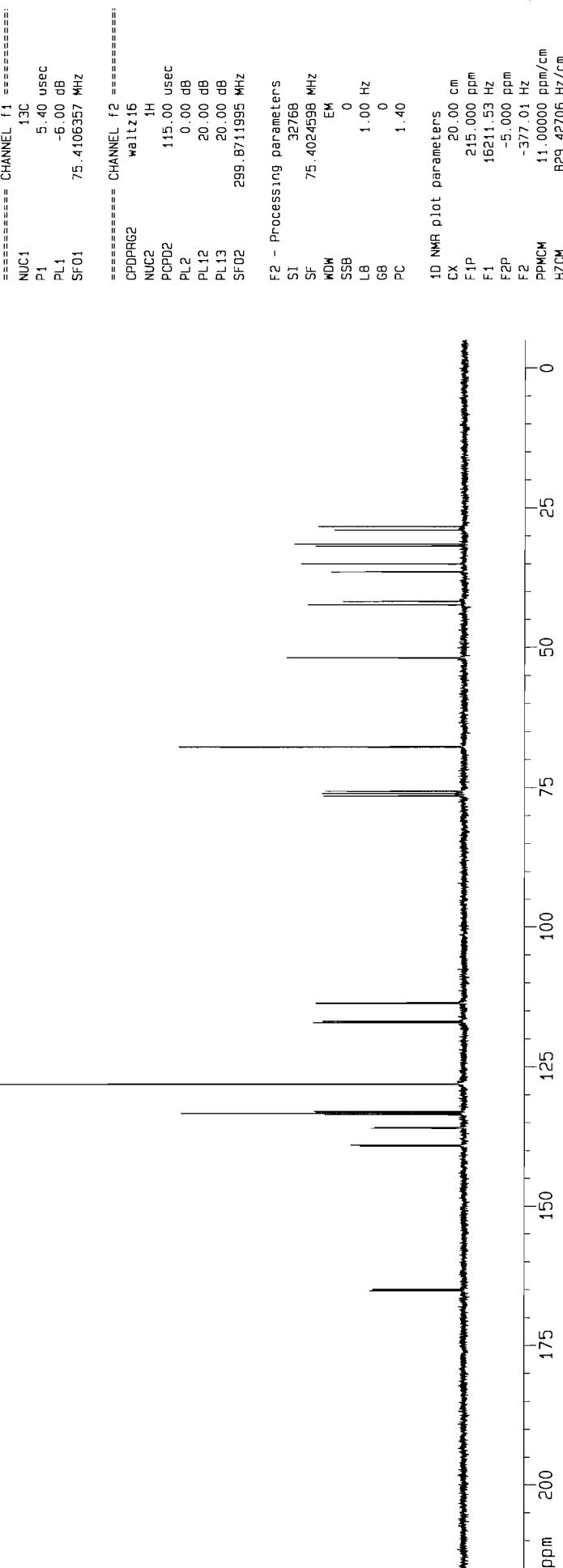


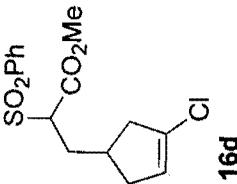
15d

Current Data Parameters
 NAME Jun25-2010-Mein
 EXPNO 11
 PROCNO 1

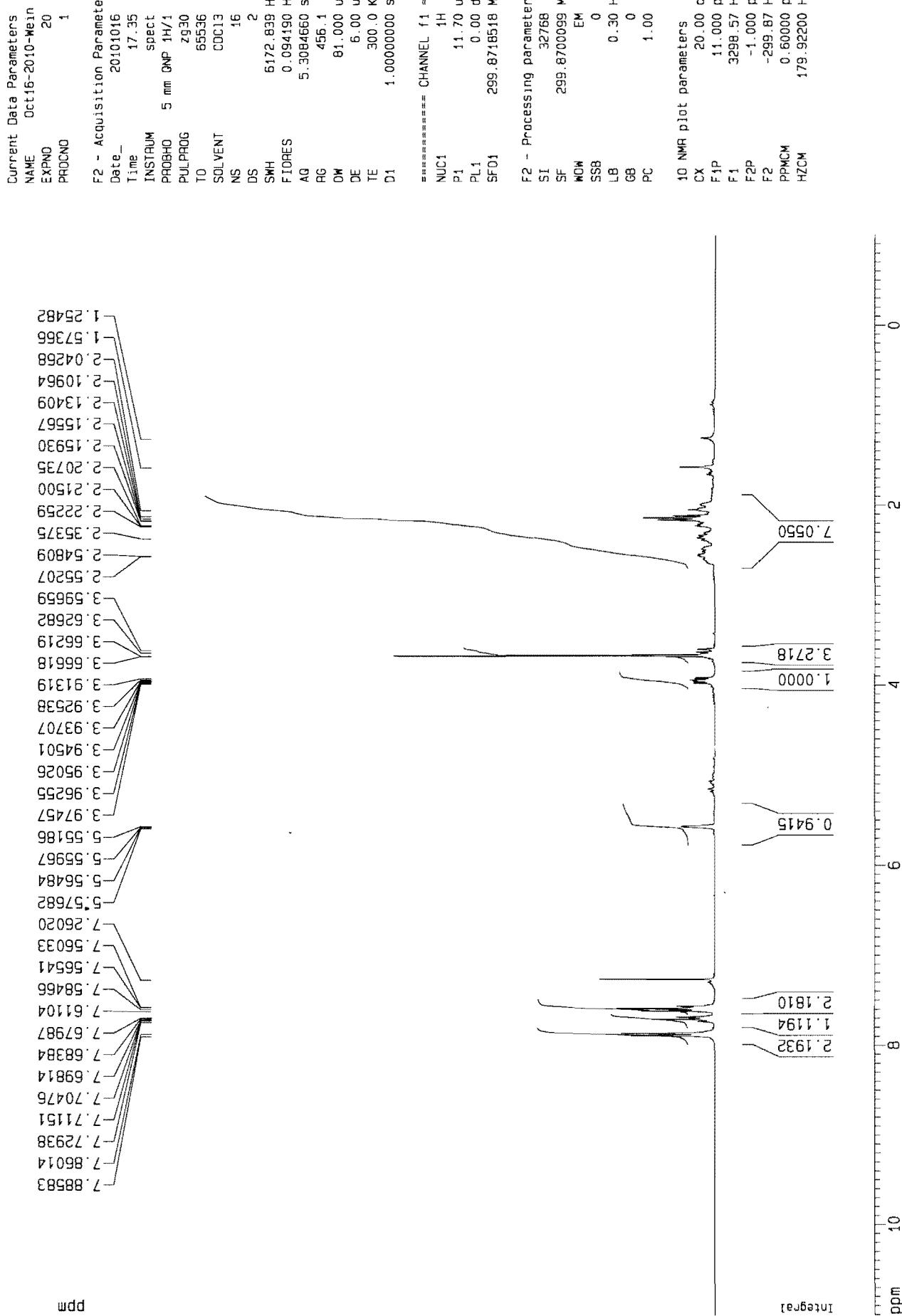


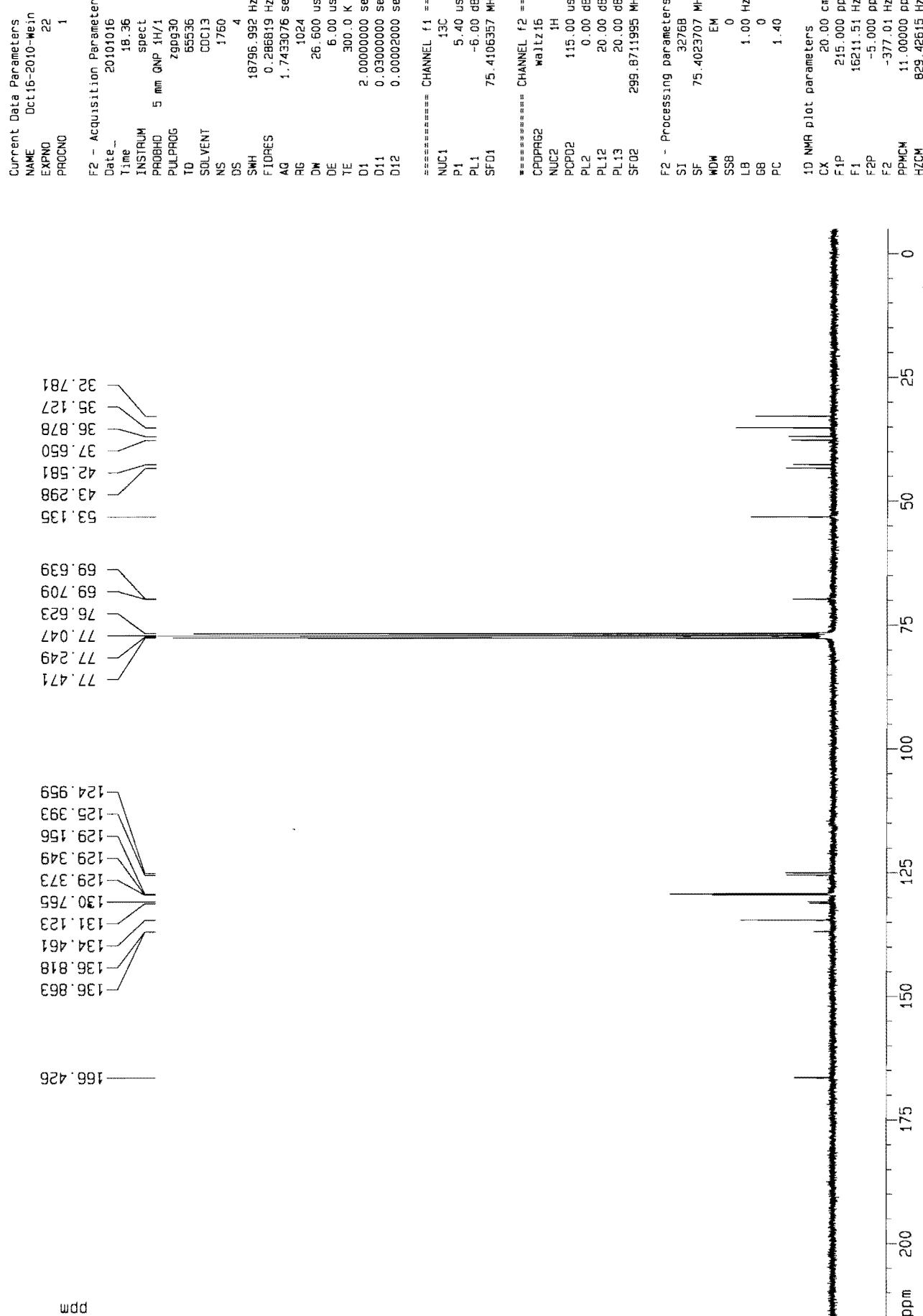
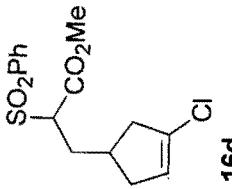
ppm

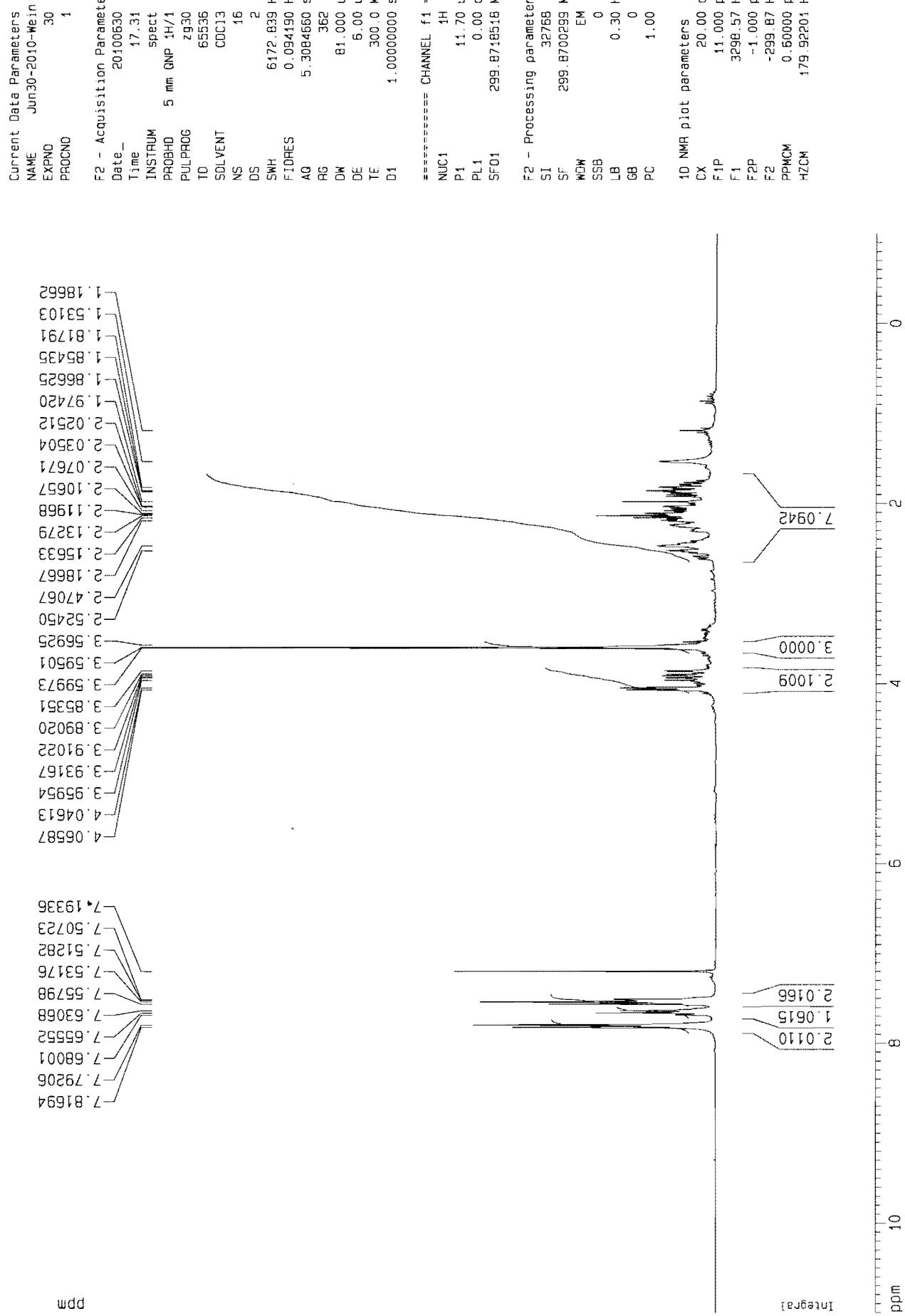
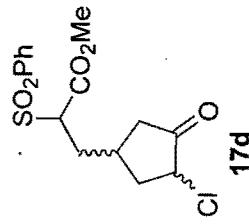


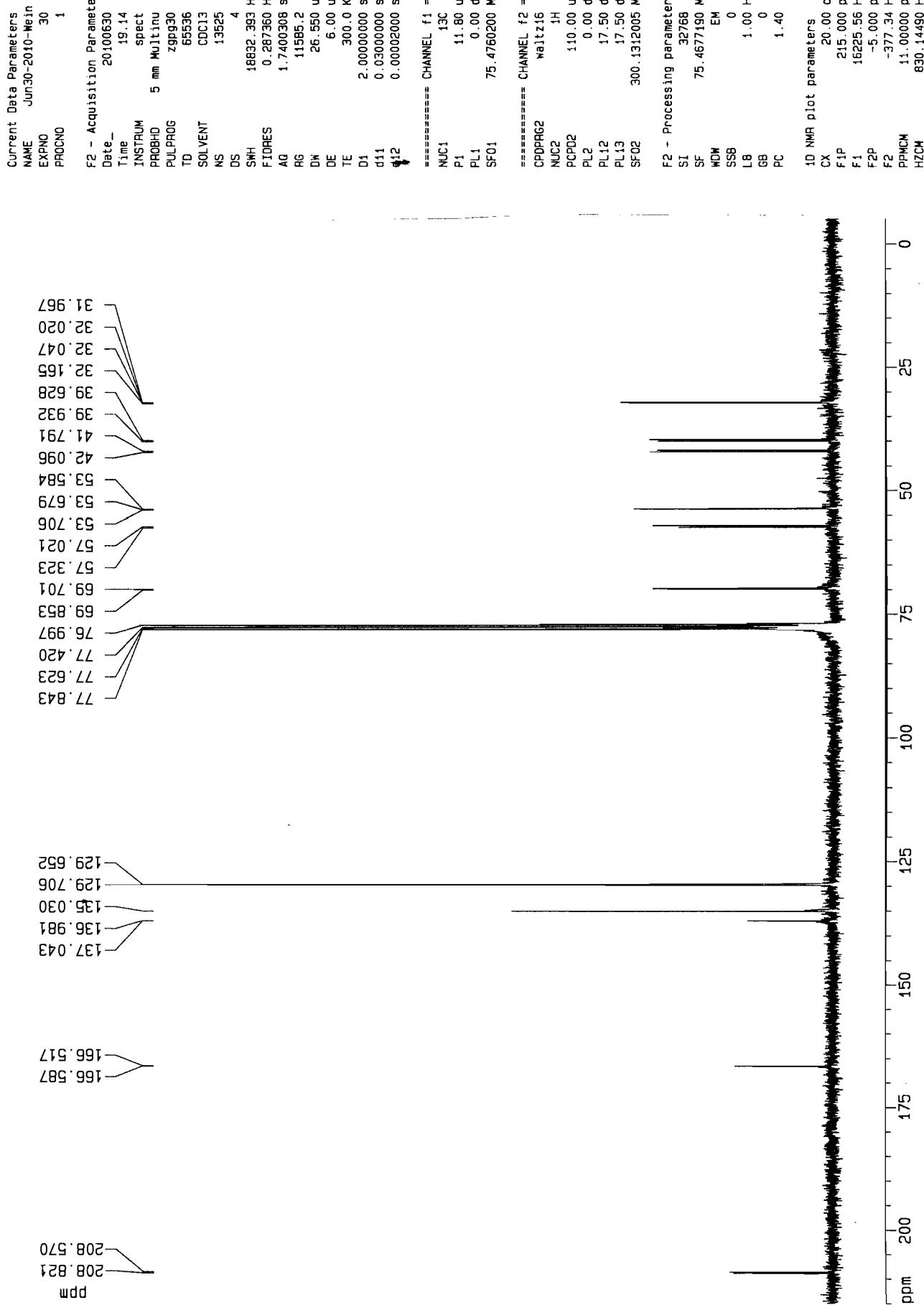
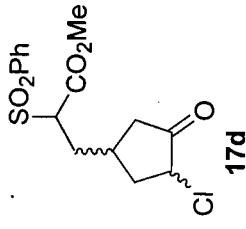


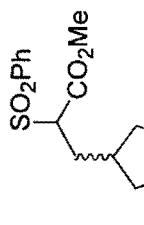
1g^h



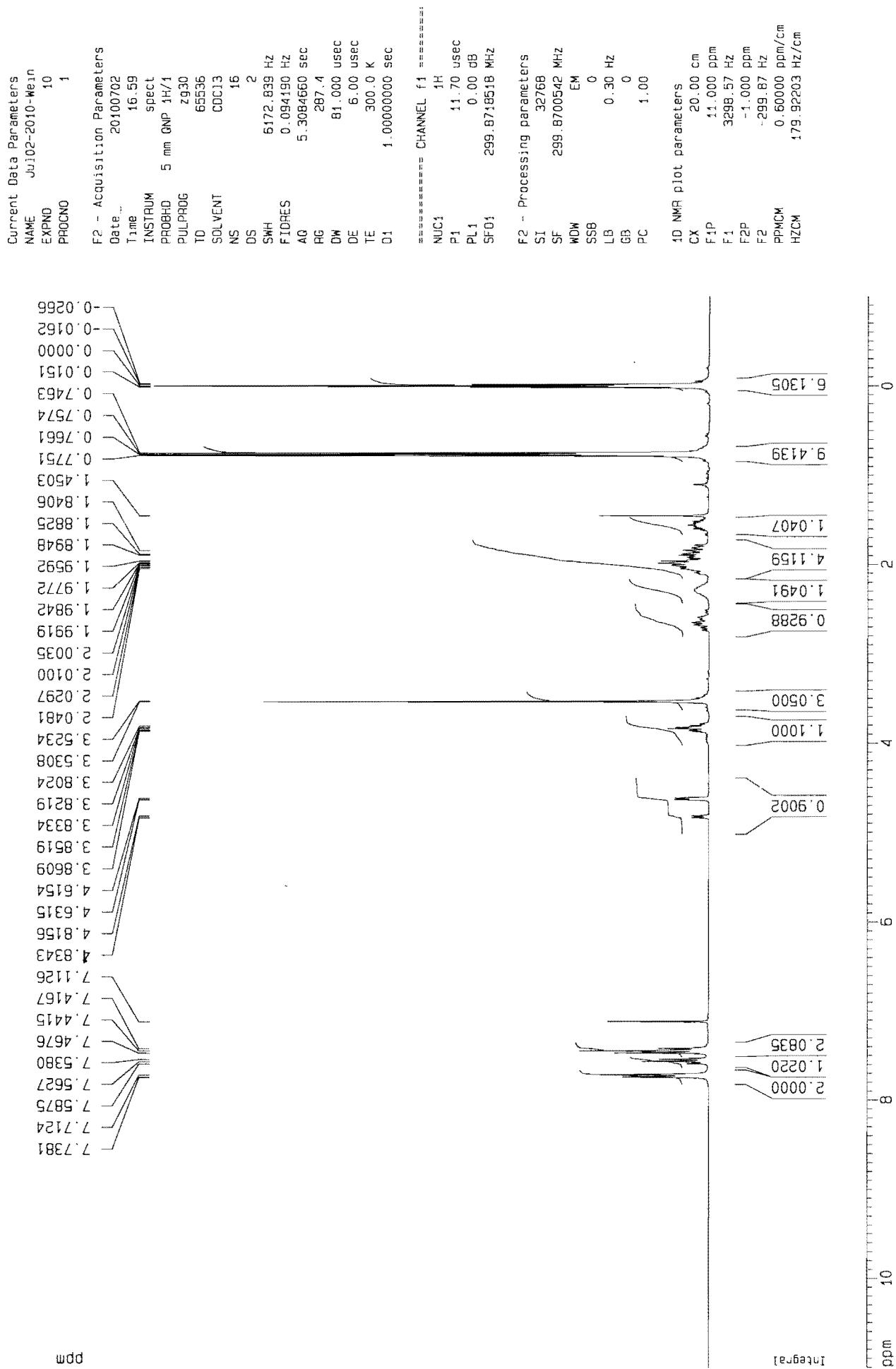


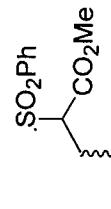




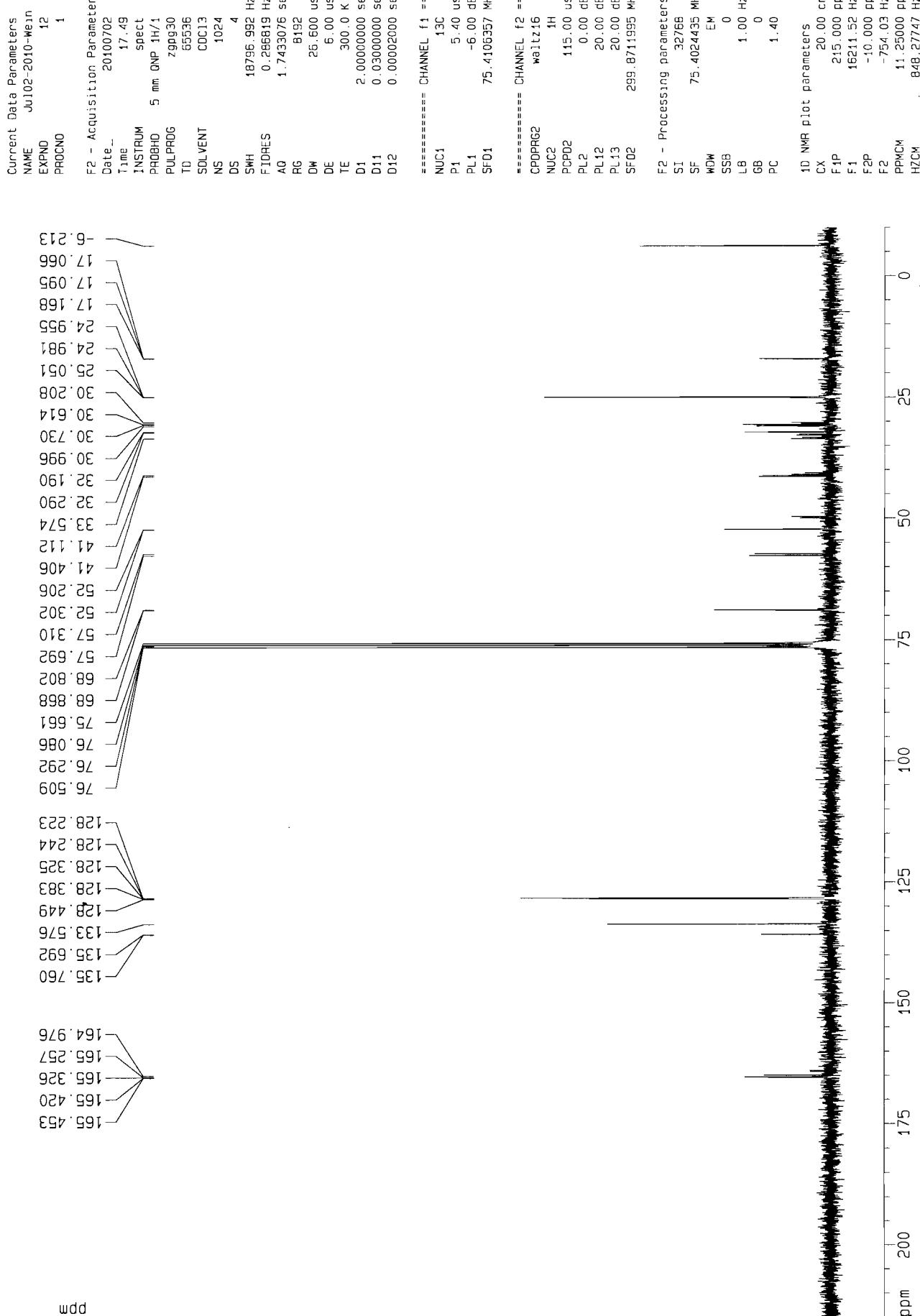


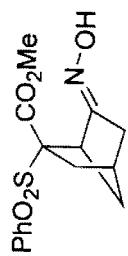
18d



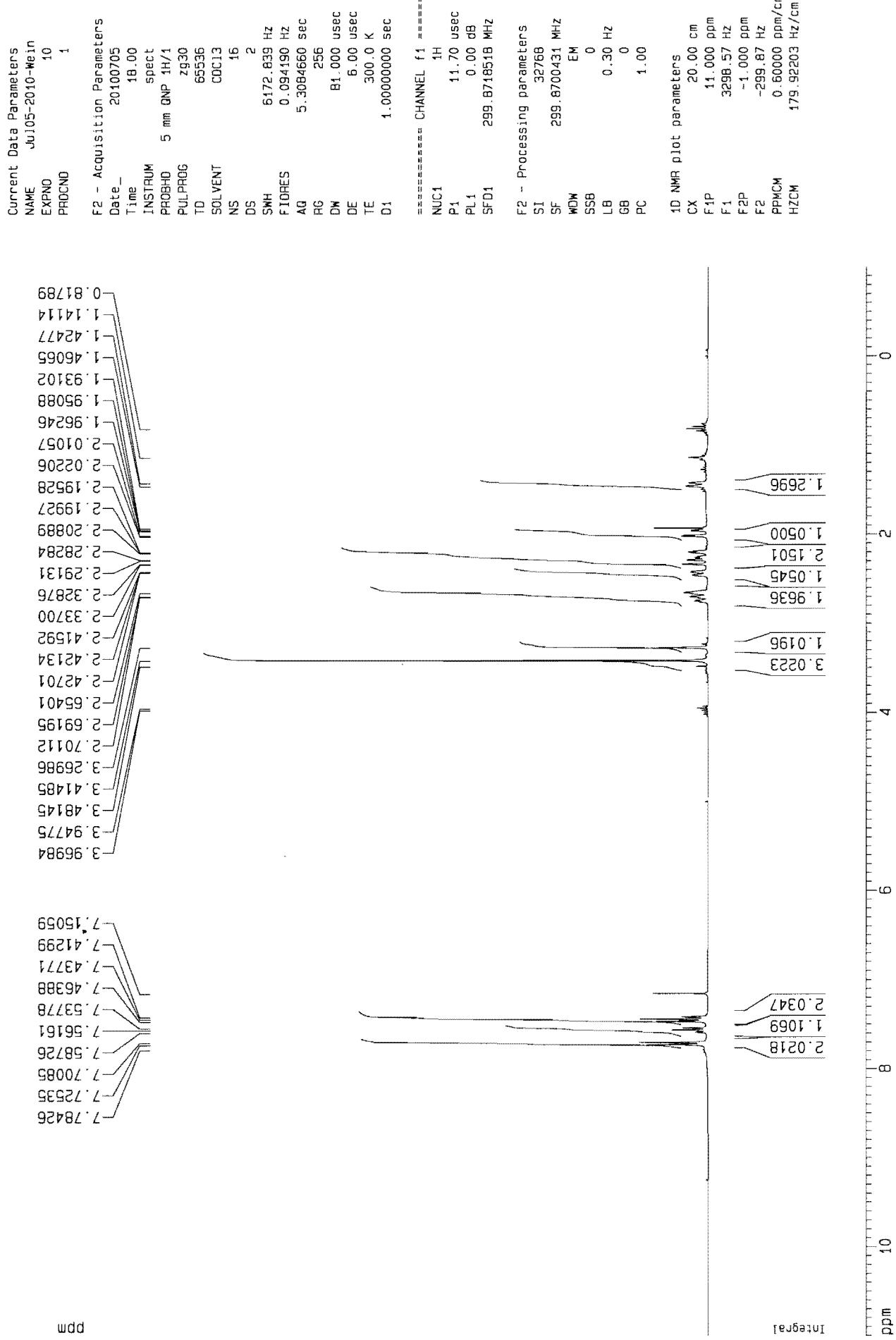


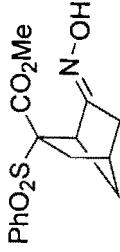
18d



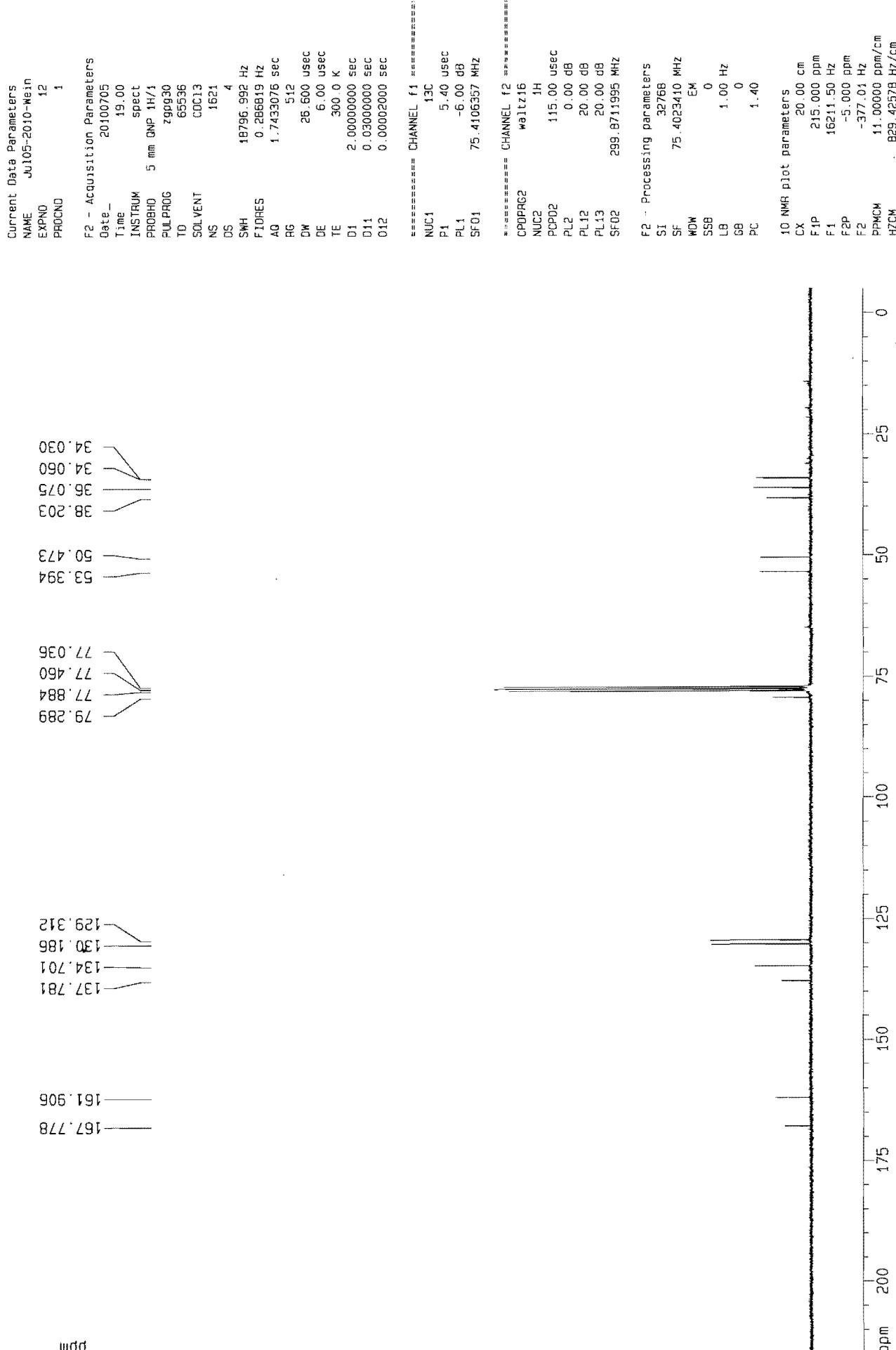


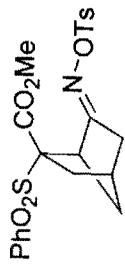
22a



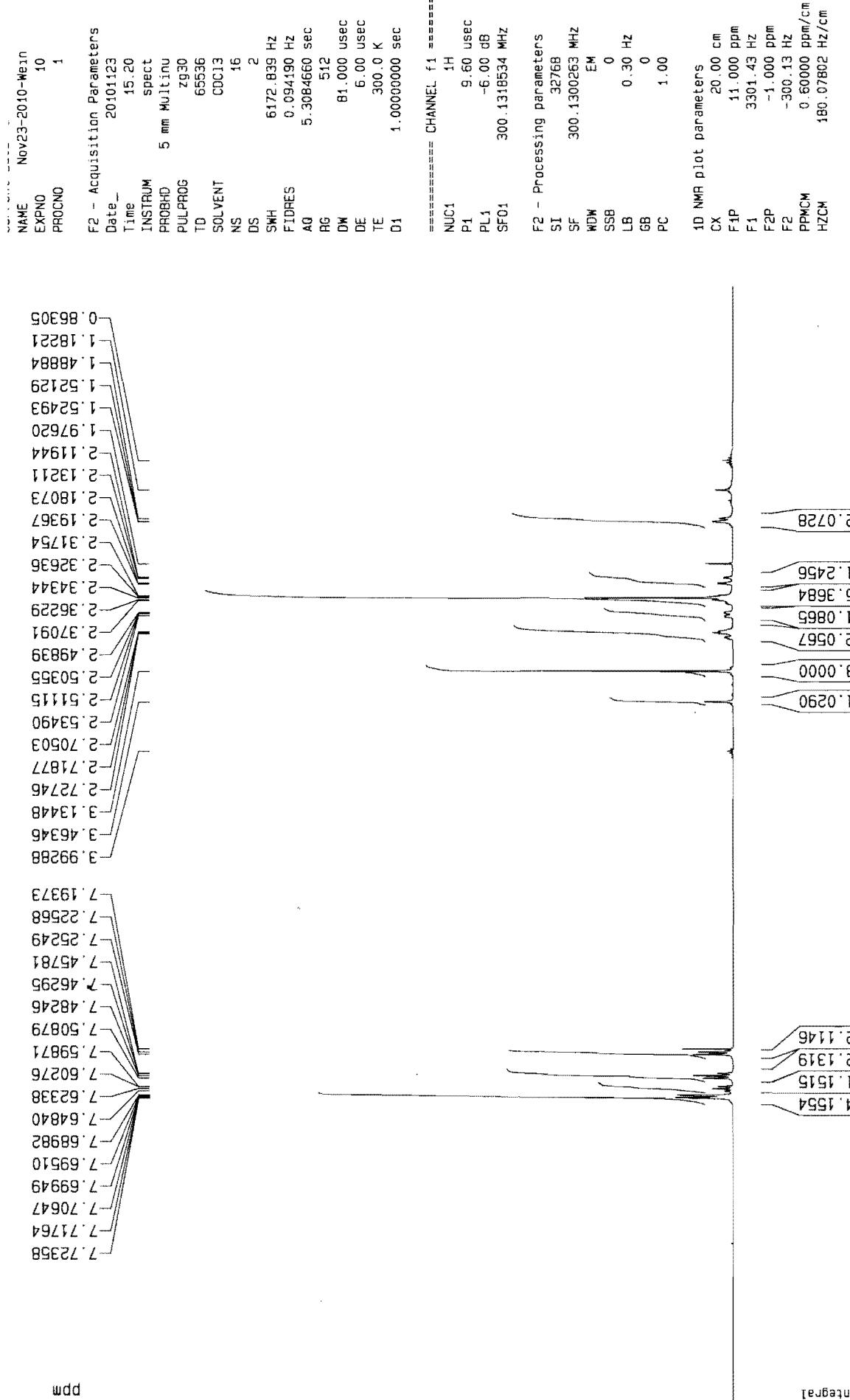


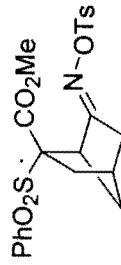
22a



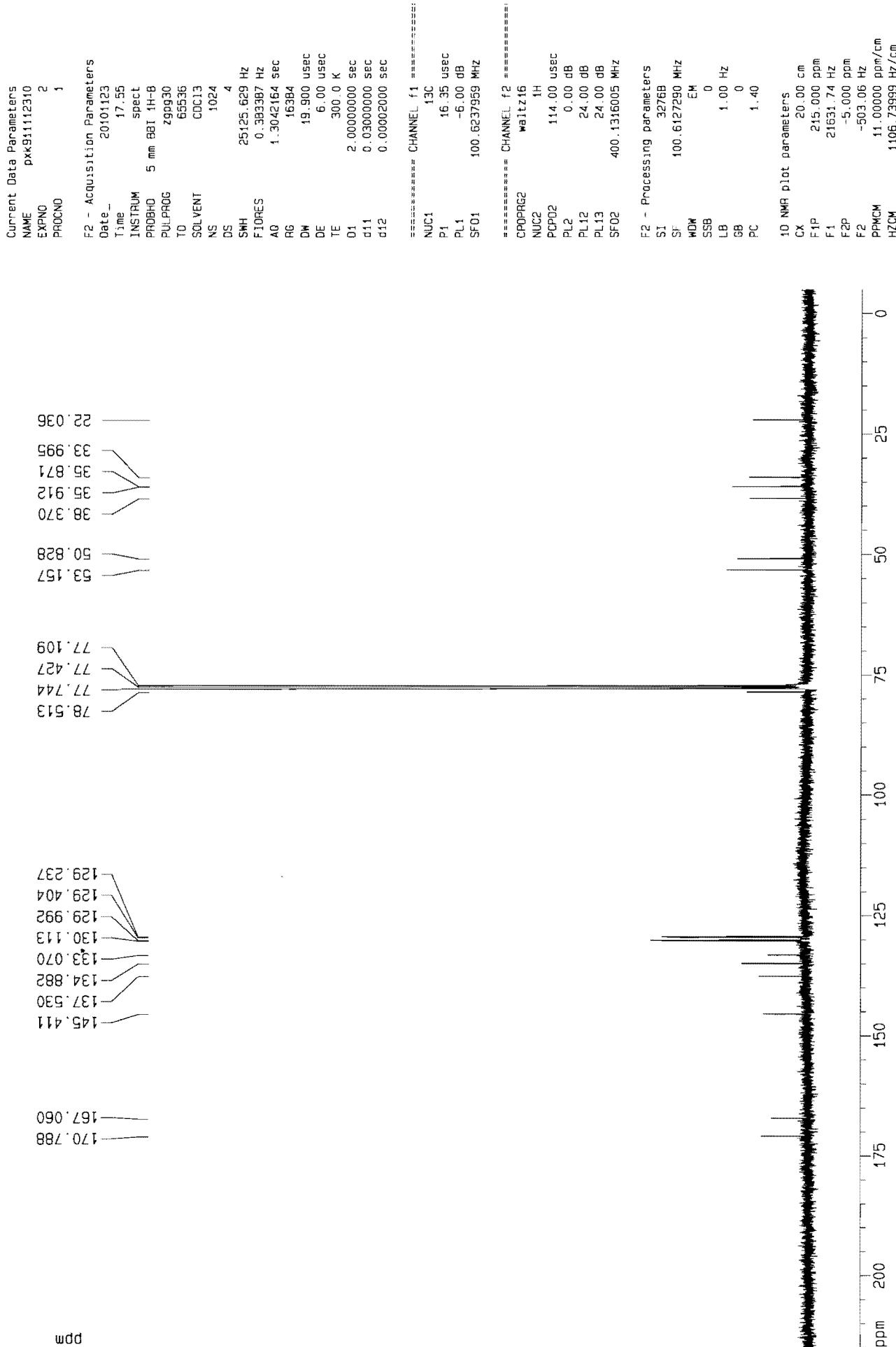


22b

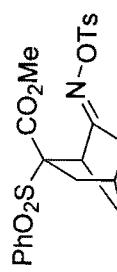




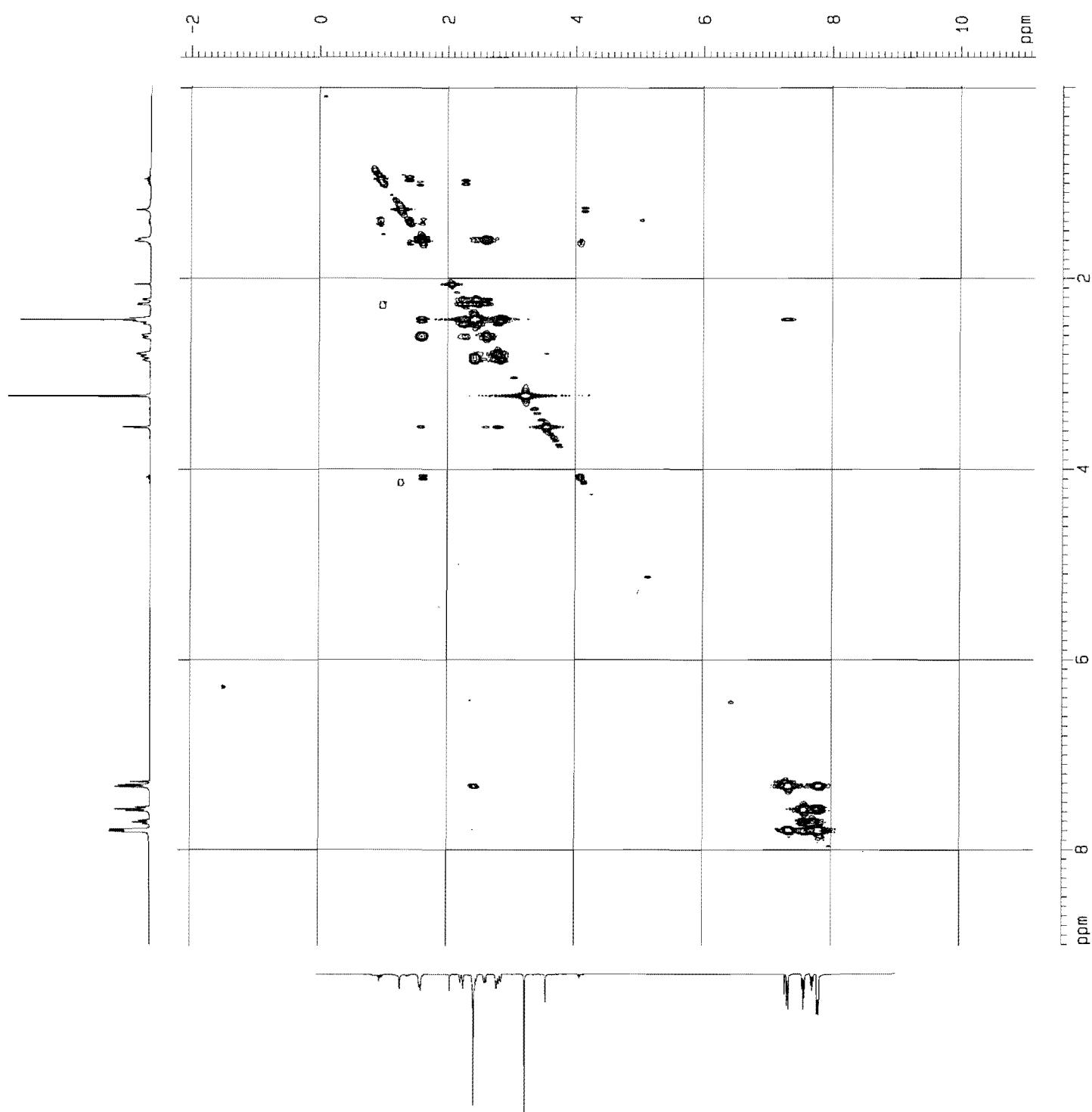
22b

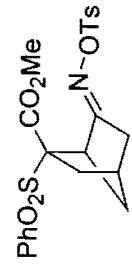


COSY

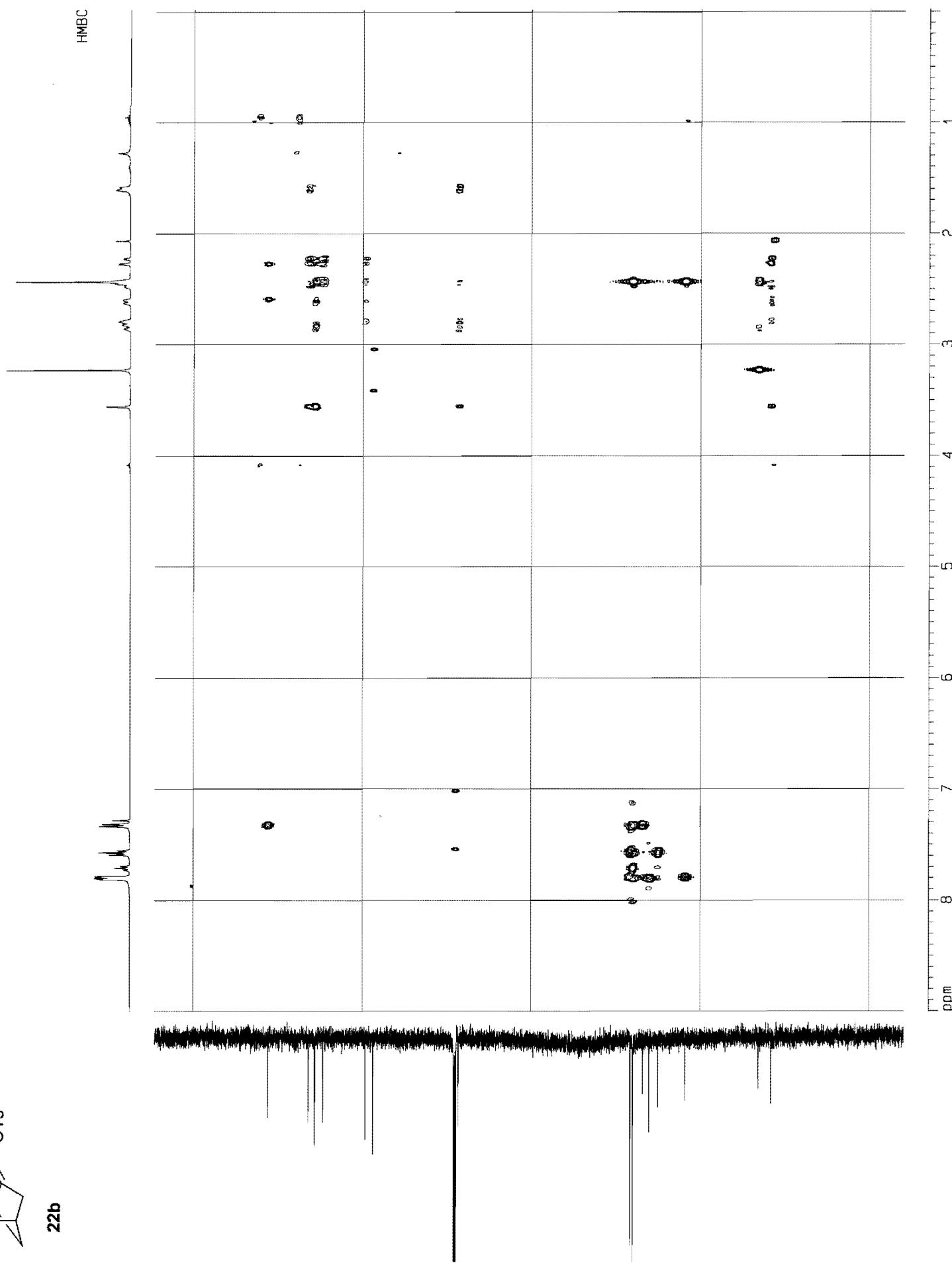


22b

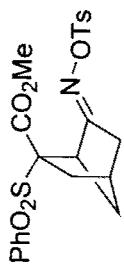
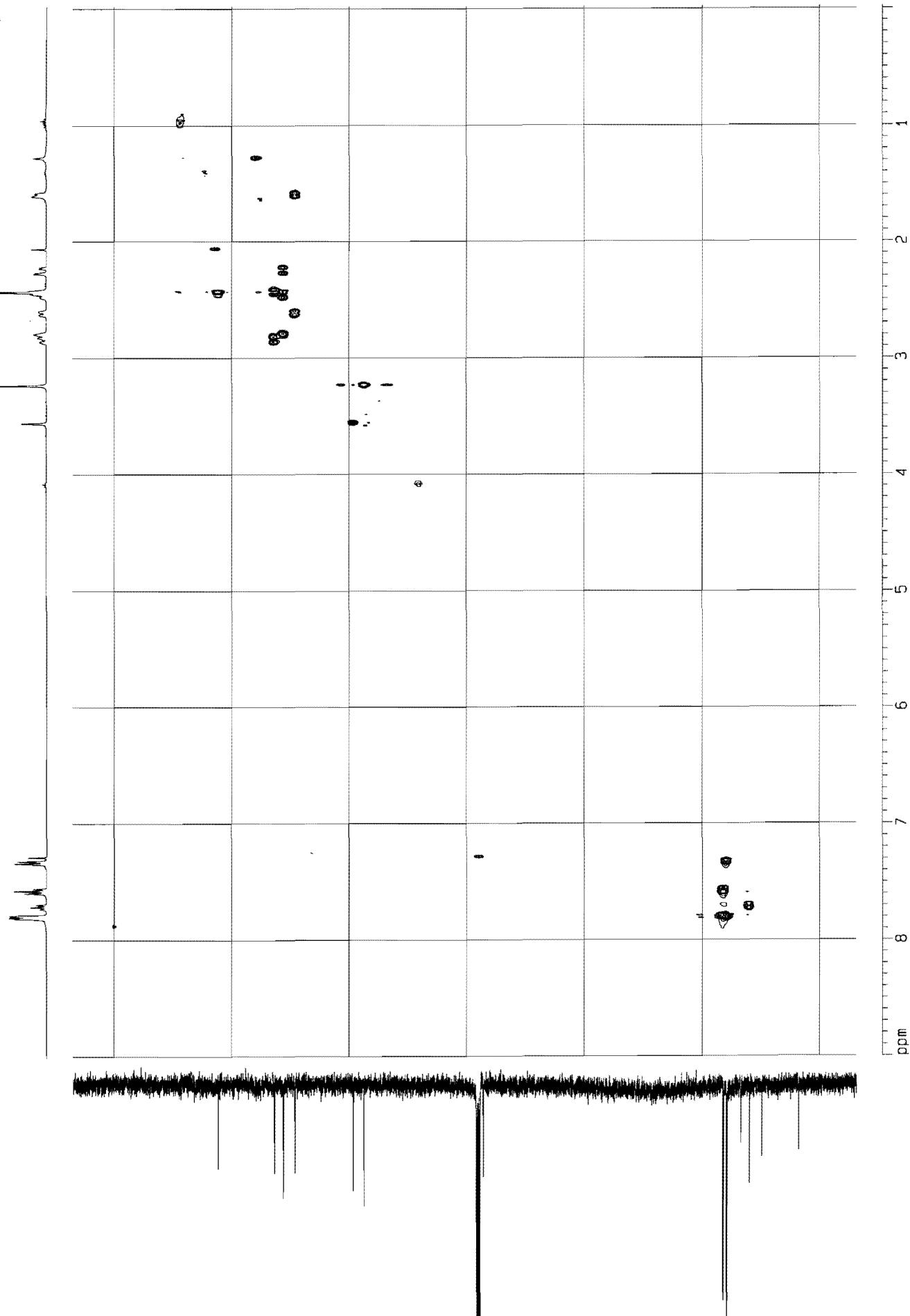


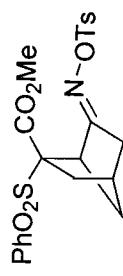


22b

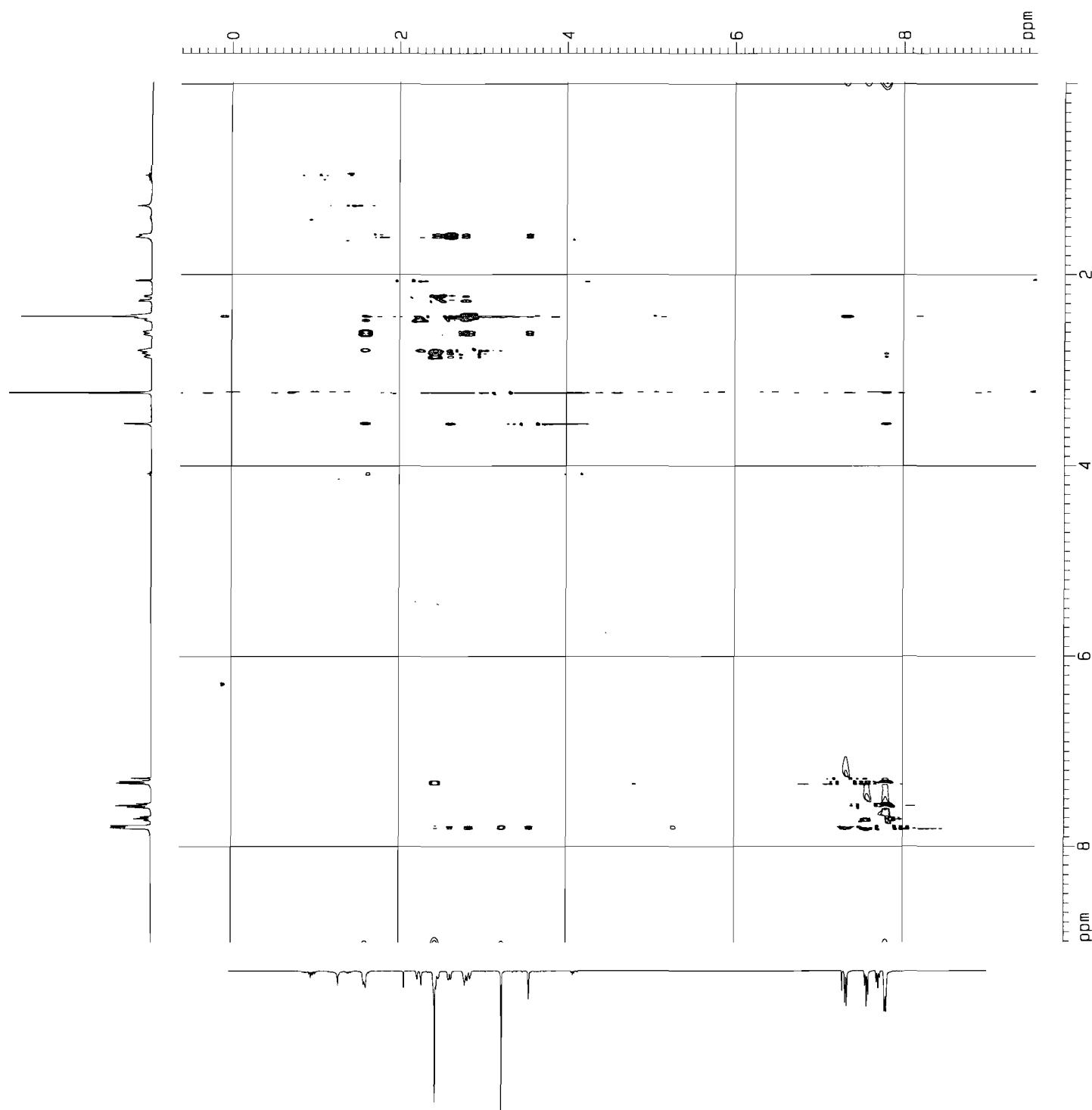


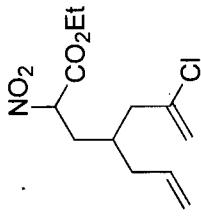
HMQC



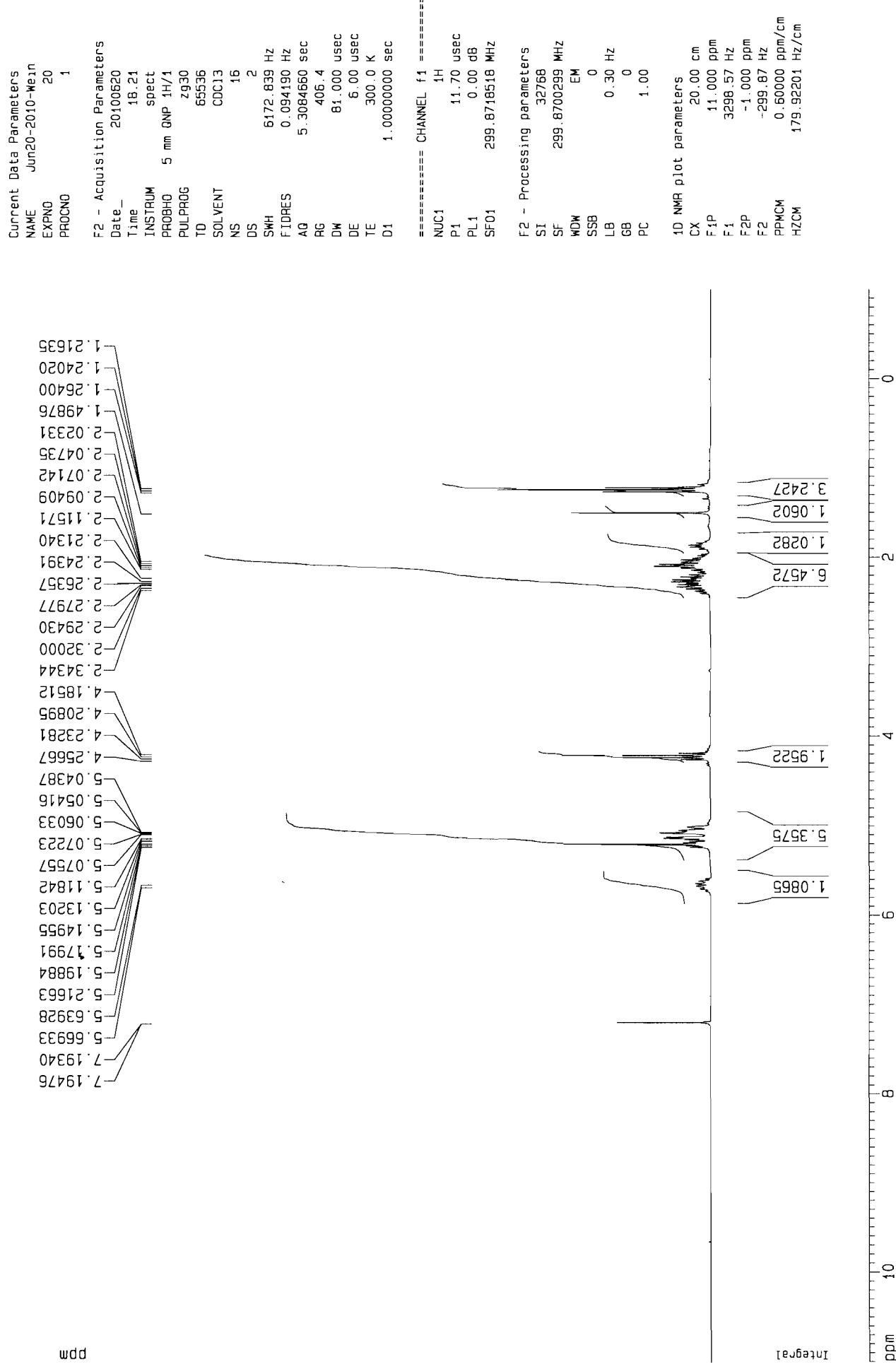


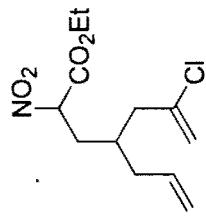
22b NoSEY



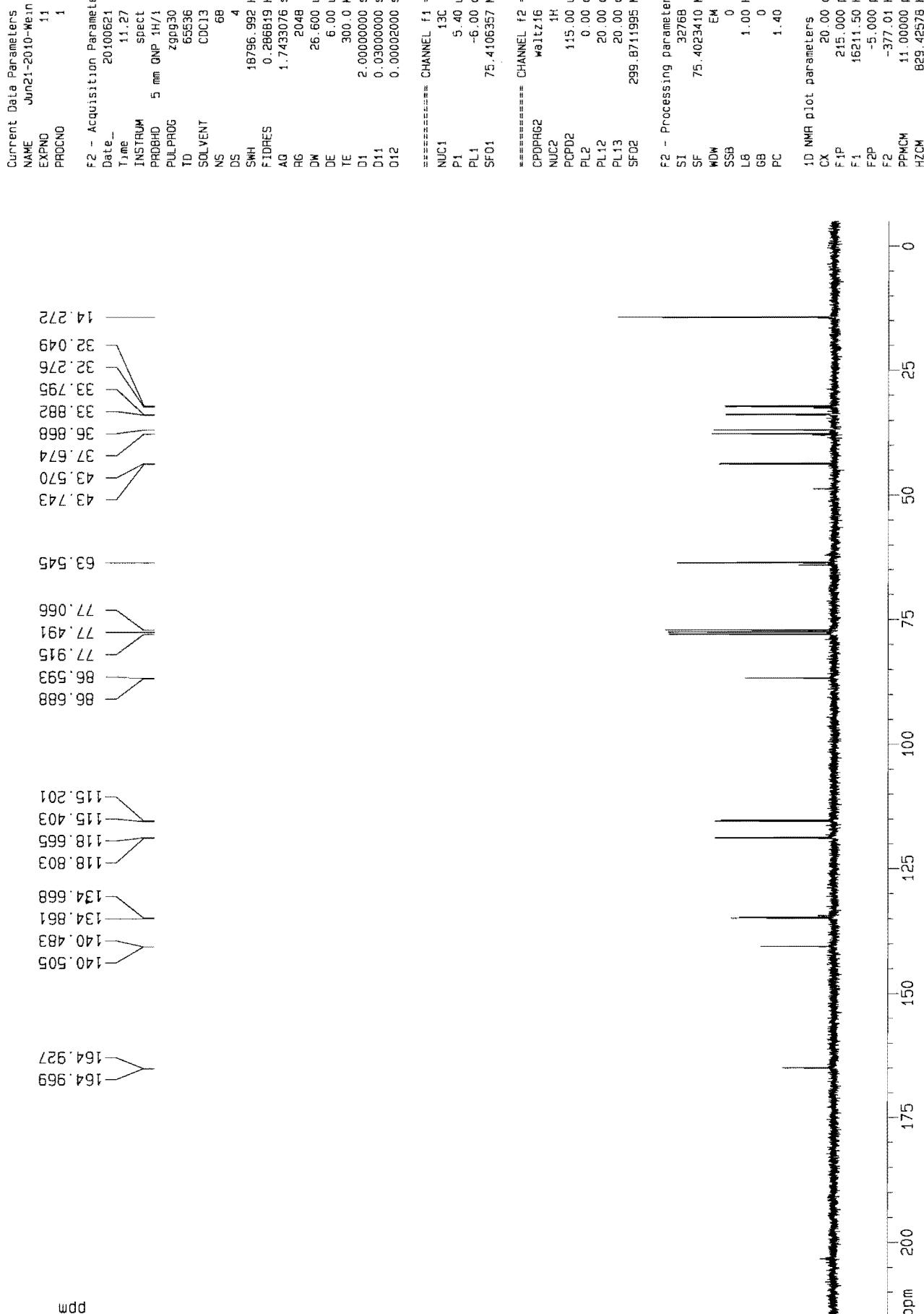


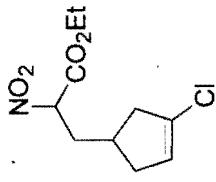
15e



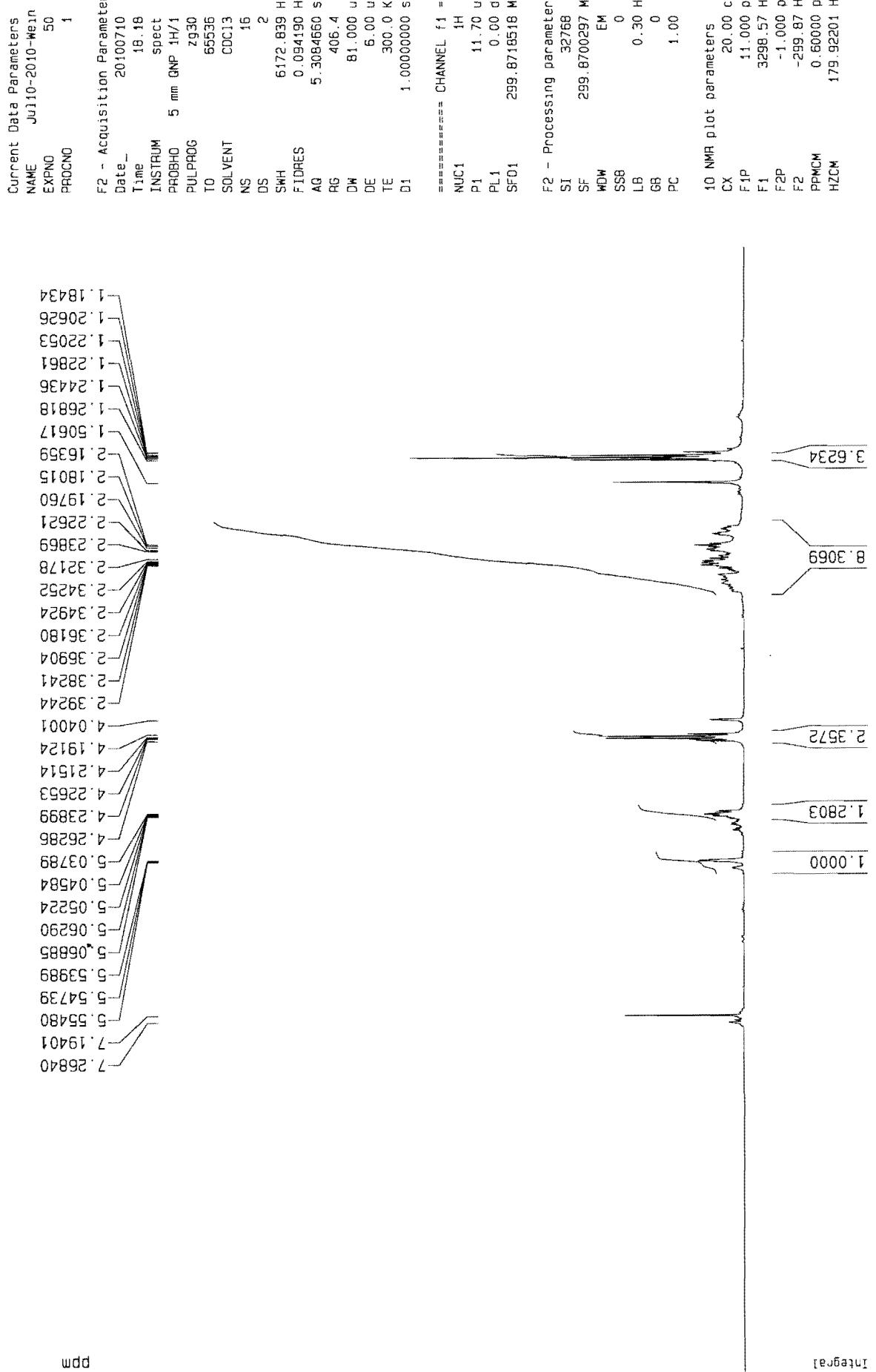


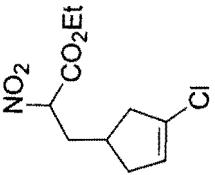
15e





16e





16e

Current Data Parameters

NAME JU110-2010-Main
EXPNO 41
PROCNO 1

F2 - Acquisition Parameters

Date 20100710
Time 18:10
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1123
DS 4
SWH 18795.982 Hz
FIDRES 0.286819 Hz
AQ 1.7433016 sec
RG 2048
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec
D11 0.0300000 sec
D12 0.0000200 sec

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

NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SF01 75.4106337 MHz

===== CHANNEL f2 =====

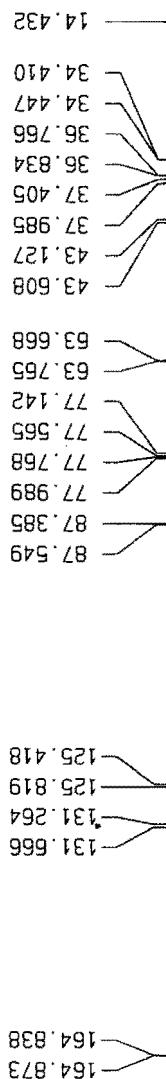
CPOPG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SF02 299.8711985 MHz

F2 - Processing parameters

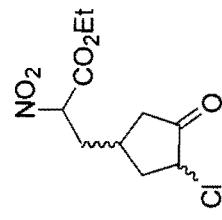
SI 32768
SF 75.4023316 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR pilot parameters

CX 20.00 cm
F1P 215.000 ppm
F1 16211.50 Hz
-5.000 ppm
F2P -37.01 Hz
F2 11.00000 ppm/cm
PPMCM 829.42560 Hz/cm



ppm



17e

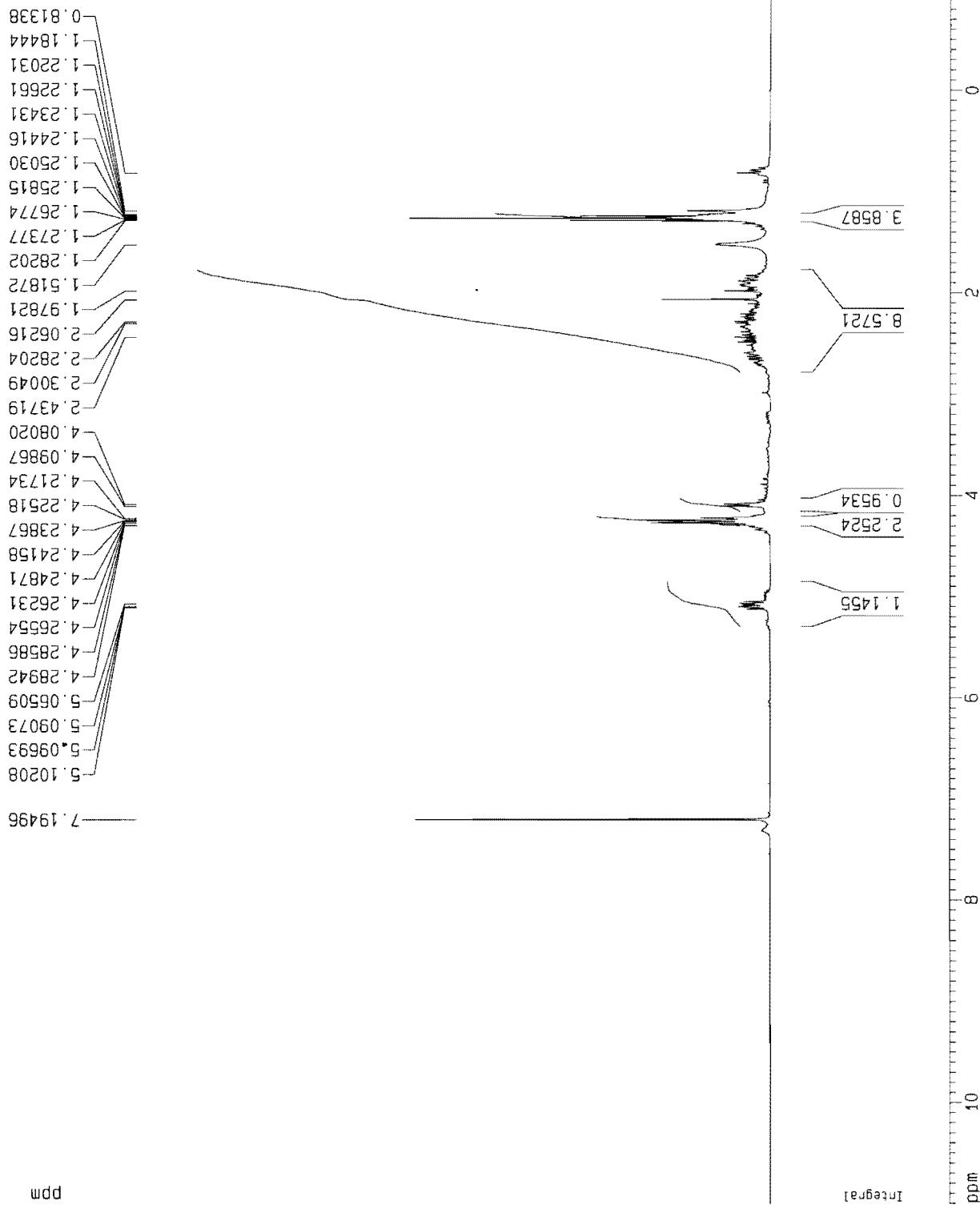
Current Data Parameters
NAME Ju111-2010-Main
EXDNO 20
PROCNO 1

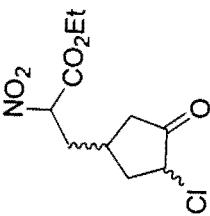
F2 - Acquisition Parameters
Date 2010/07/11
Time 15:21
INSTRUM Spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094150 Hz
AQ 5.3084660 sec
RG 574.7
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.0000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 1.170 usec
PL1 0.00 dB
SF01 299.8718518 MHz

F2 - Processing parameters
SI 32768
SF 299.870296 MHz
NDW EM
SSB 0
LB 0.30 Hz
BB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 11.000 ppm
F1 3298.57 Hz
-1.000 ppm
F2P -299.87 Hz
PPMCM 0.60000 ppm/cm
HZCM 179.92201 Hz/cm





17e

Current Data Parameters

NAME Jul11-2010-Wein
Date 22
PROGNO 1

F2 - Acquisition Parameters

Date 20100711
Time 16:59
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 15972
DS 4
SWH 18796.992 Hz
FIDRES 0.286519 Hz
AQ 1.7433076 sec
RG 4096
DW 26.600 usec
DE 6.00 usec
TE 360.0 K
D1 2.0000000 sec
D11 0.03000000 sec
D12 0.000002000 sec

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

NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SF01 75.4106357 MHz

===== CHANNEL f2 =====

CPDPRG2 Watt16
NUC2 1H
PCP02 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SF02 299.8711995 MHz

F2 - Processing parameters

SI 32768
SF 75.4023410 MHz
WM EM
SSB 0
LB 1.00 Hz
GS 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm

F1P 215.00 ppm

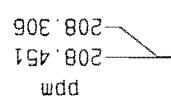
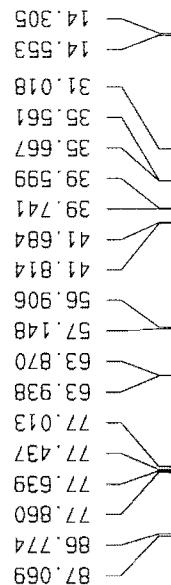
F1 16211.50 Hz

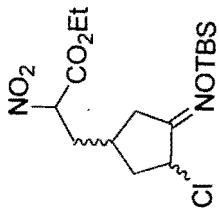
F2P -5.000 ppm

F2 -377.01 Hz

PPMCK 11.00000 ppm/cm

HZCM 829.42578 Hz/cm





18e

Current Data Parameters
NAME JU113-2010-Mein
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters

Date_ 20100713
Time 12.34
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.308460 sec
RG 512
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.0000000 sec

==== CHANNEL f1 =====

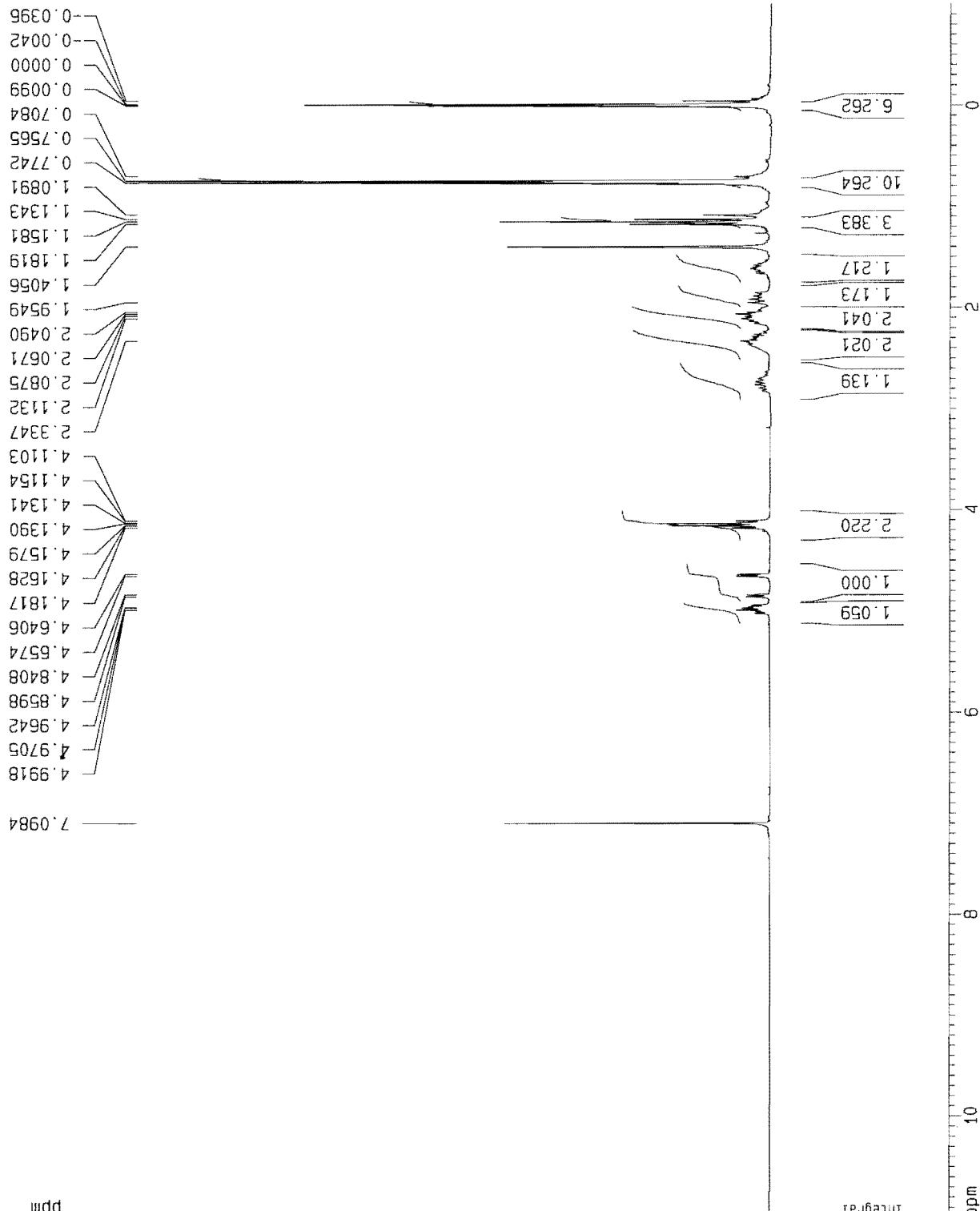
NUC1 1H
P1 11.70 usec
PL1 0.00 dB
SF01 299.8718518 MHz

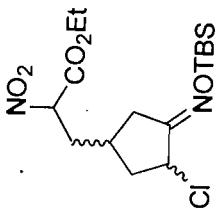
F2 - Processing parameters

CX 32768
SF 299.8700586 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 11.000 ppm
F1 3298.57 Hz
F2P -1.000 ppm
F2 -299.87 Hz
PPMCH 0.60000 ppm/cm
HZCM 179.93203 Hz/cm





18e

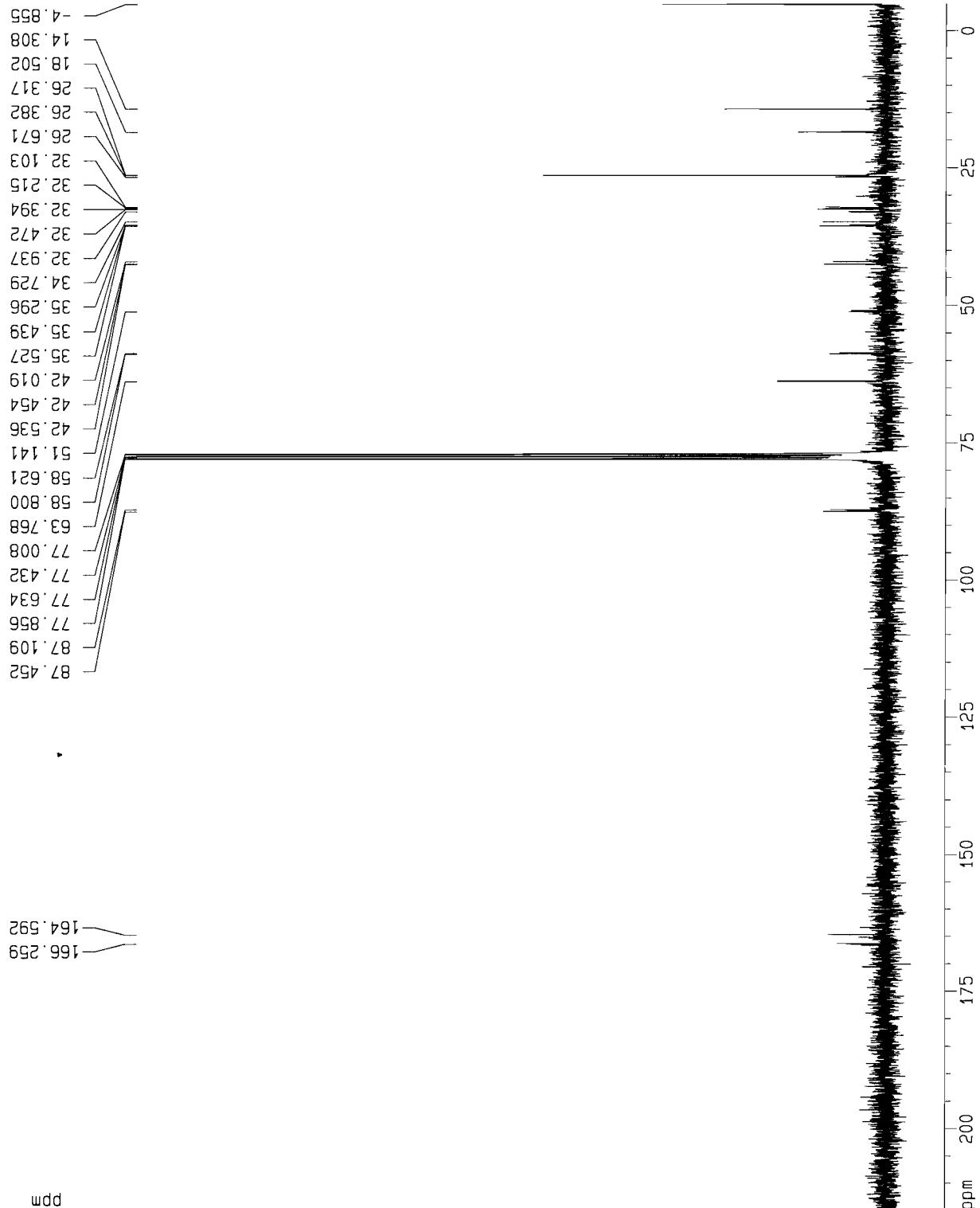
Current Data Parameters
JUL13-2010-ME
NAME 40
EXPNO 1
PROCNO

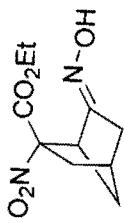
F2 - Acquisition Parameters
Date_ 20100713
Time_ 16.02
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg930
TD 65536
SOLVENT CDCl3
NS 2321
DS 4
SWH 18796.992 Hz
FIDRES 0.286619 Hz
AG 1.7433076 sec
RG 1024
DW 25.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec
D11 0.03600000 sec
D12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SF01 75.4106357 MHz
===== CHANNEL f2 =====
CPDPG2 walt16
NUC2 1H
CPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SF02 299.8711995 MHz

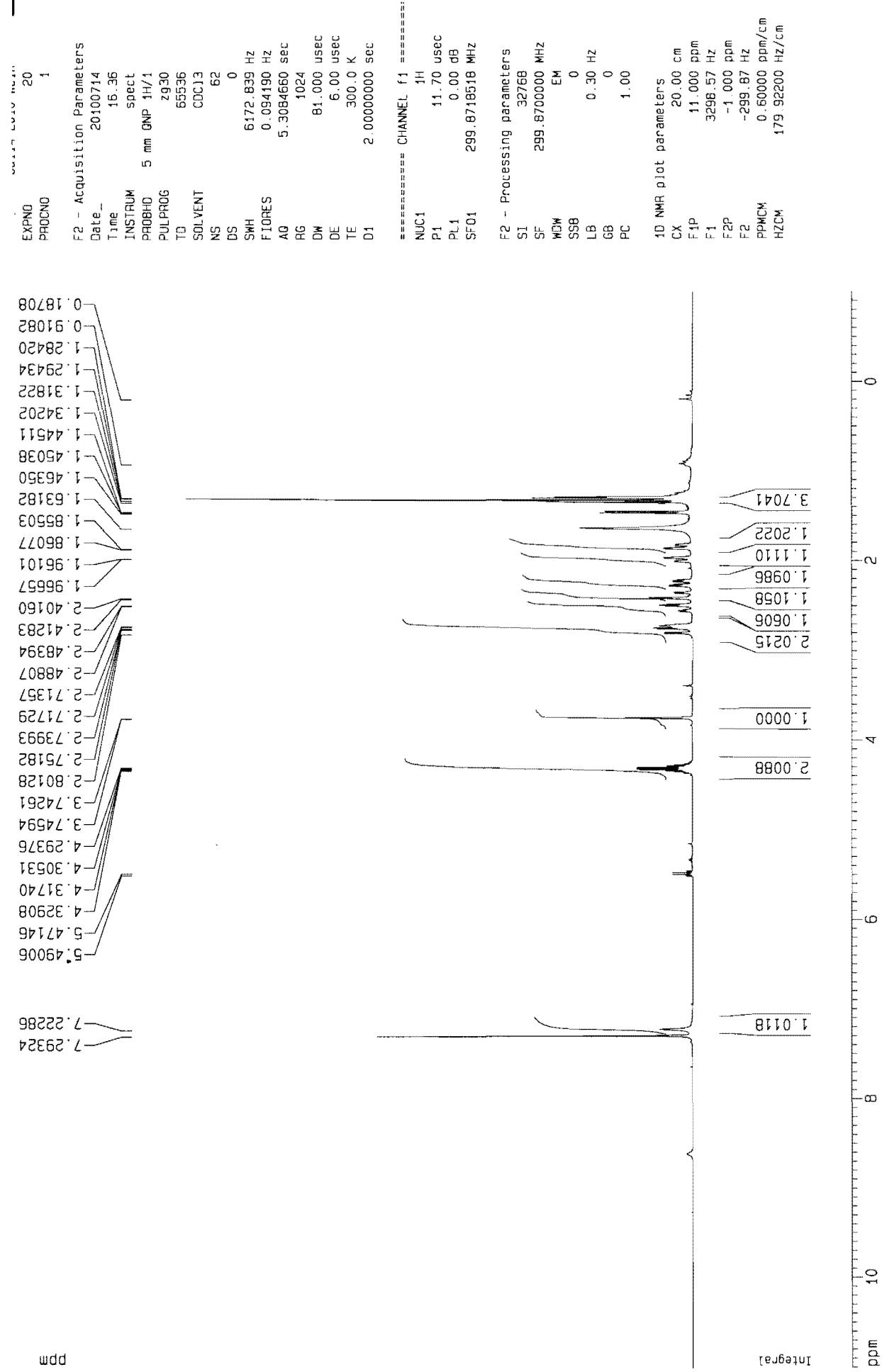
F2 - Processing parameters
SI 32768
SF 75.402310 MHz
WM EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

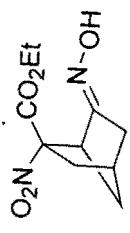
10 NMR plot parameters
CX 20.00 cm
F1P 215.000 ppm
F1 16211.50 Hz
F2P -5.000 ppm
F2 -377.01 Hz
PPMCM 11.00000 ppm/cm
HzCM 829.42378 Hz/cm



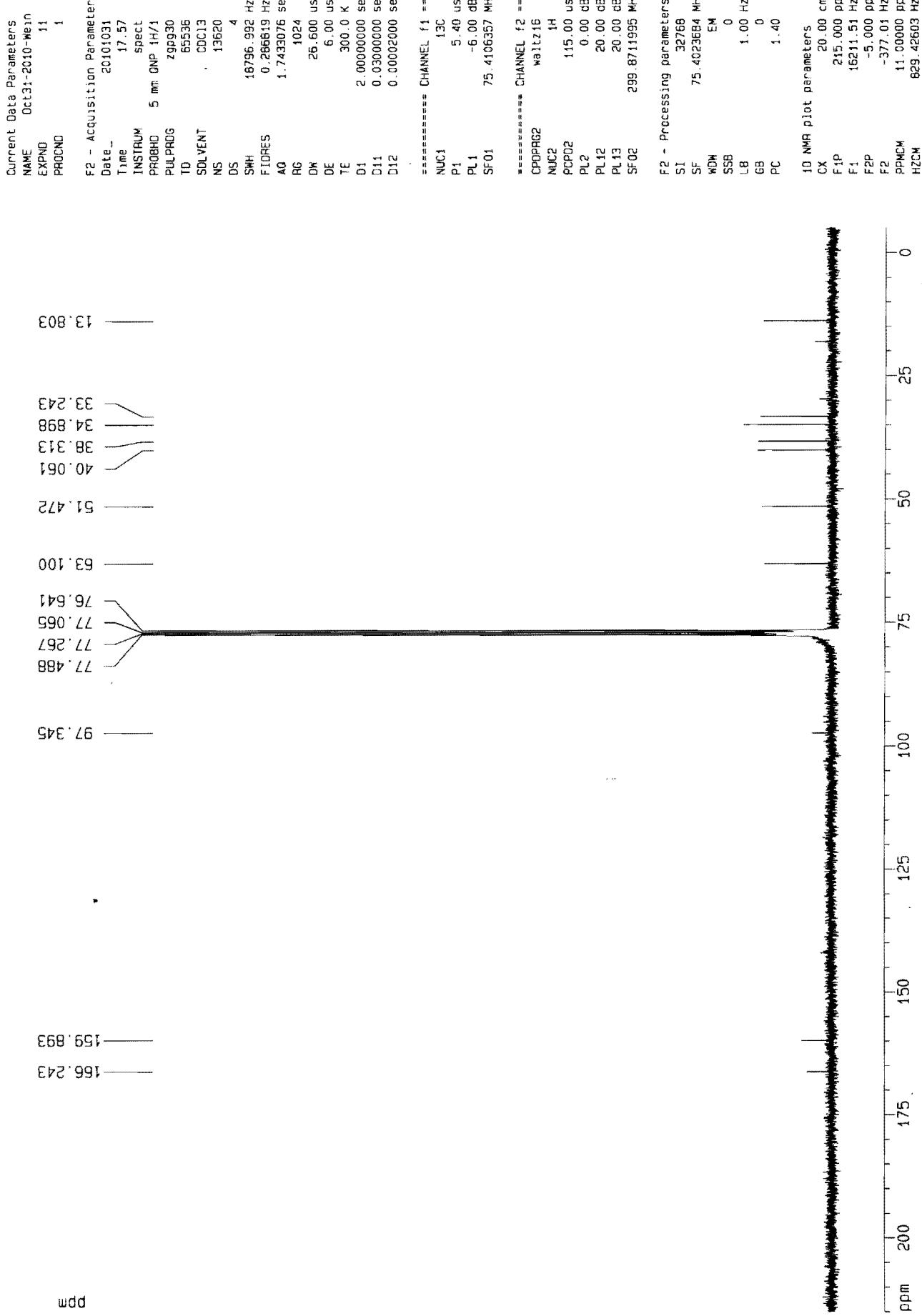


24a

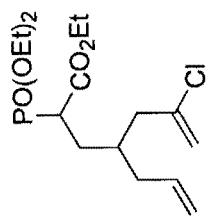




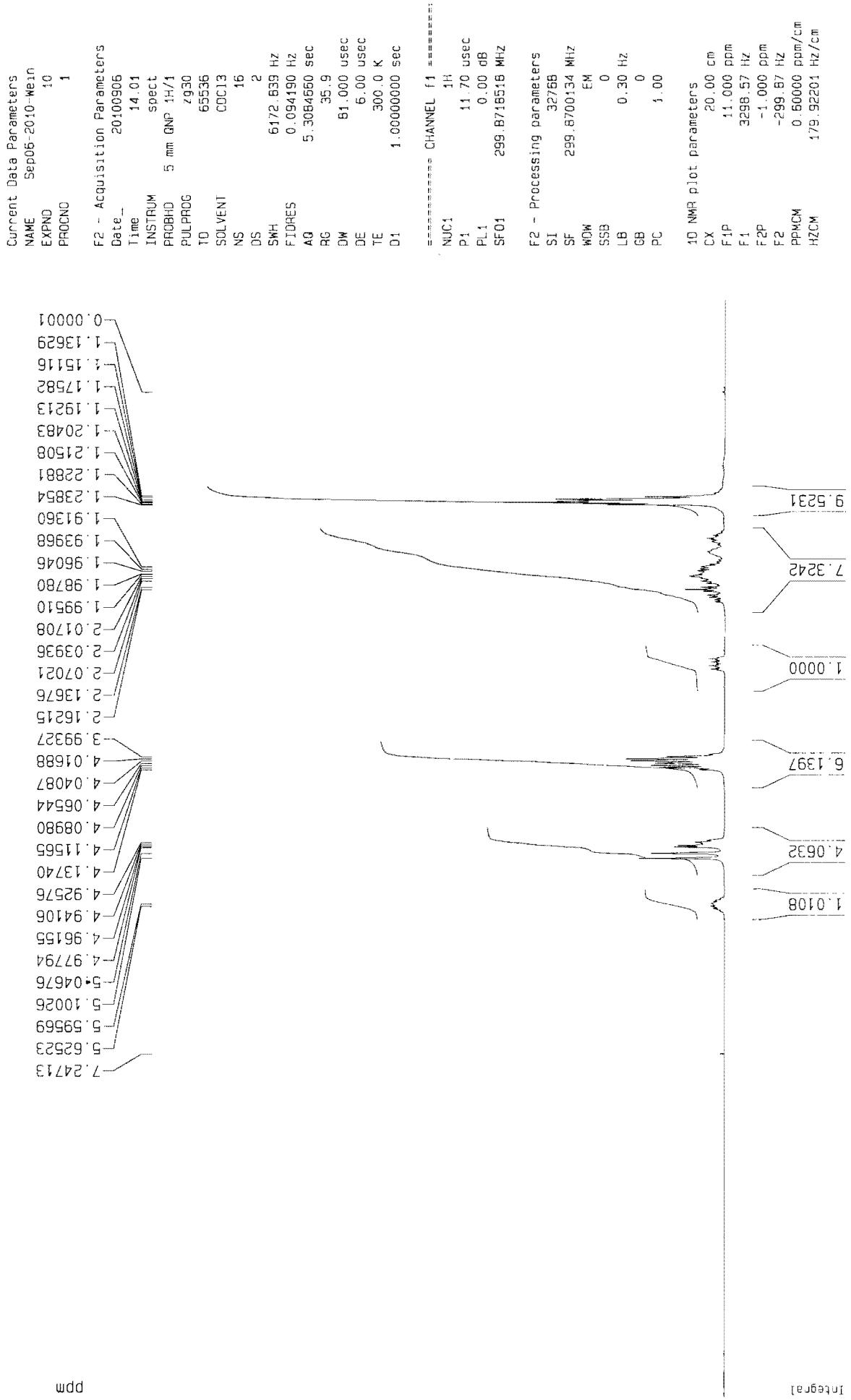
24a

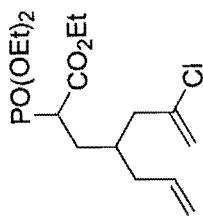


ppm

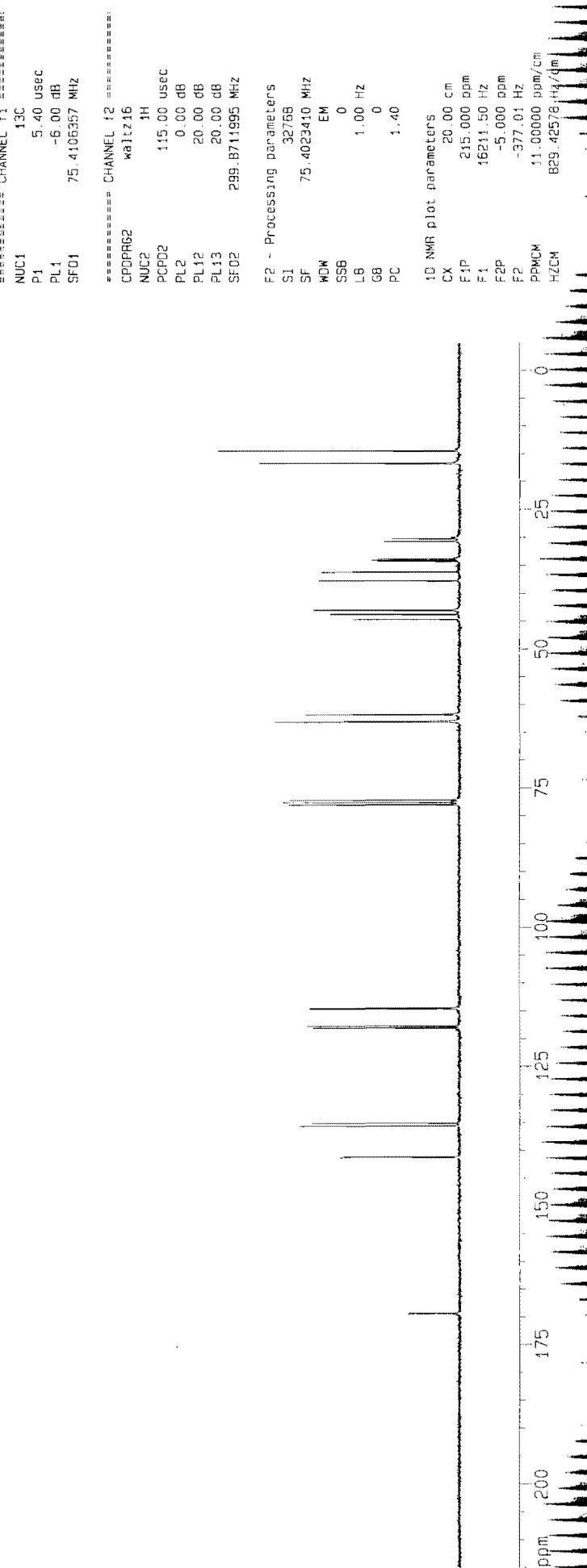
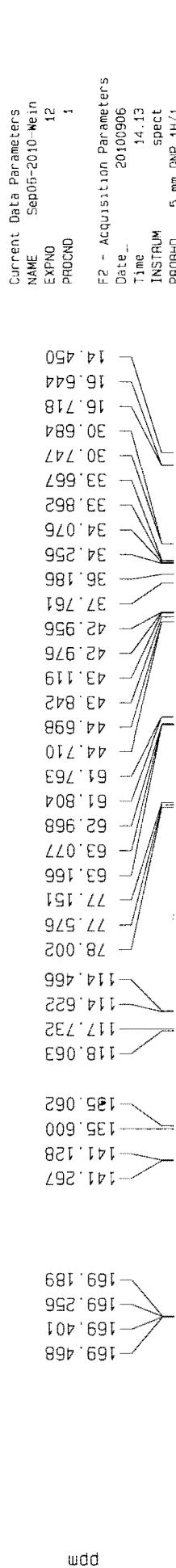


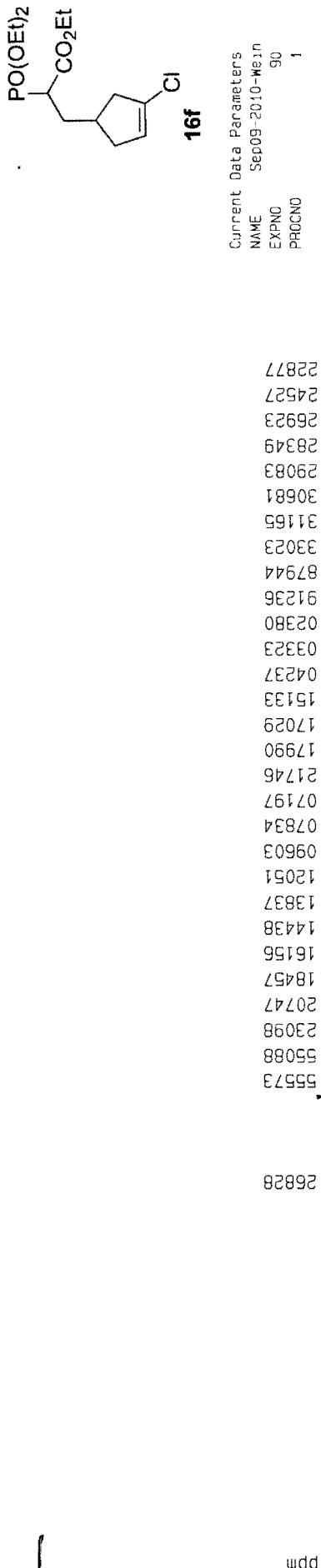
15f





15f





Current Data Parameters
NAME Sep09-2010-Wein
EXPNO 90
PROCNO 1

F2 - Acquisition Parameters

Date 20100909
Time 16:15
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG 2930
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.308460 sec
RG 90.5
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.0000000 sec

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

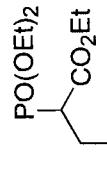
NUC1 1H
P1 11.70 usec
PL1 0.00 dB
SF01 299.8718518 MHz

F2 - Processing parameters

SI 32768
SF 299.8700071 MHz
WDW EM
SSB 0
tB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 11.00 ppm
F1 3298.57 Hz
F2P -1.000 ppm
F2 -299.87 Hz
SPMCM 0.60000 ppm/cm
HZCM 179.922500 Hz/cm



16f

Current Data Parameters
NAME Sep09-2010-Mein
EXPNO 92
PROCND 1

F2 - Acquisition Parameters
Date_ 20100909
Time_ 20:03
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpp30
TD 65336
SOLVENT CDCl3
NS 1532
DS 4
SWH 18795.992 Hz
FIDRES 0.286319 Hz
AG 1.7433076 sec
RG 1024
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec
D11 0.0300000 sec
D12 0.00002000 sec

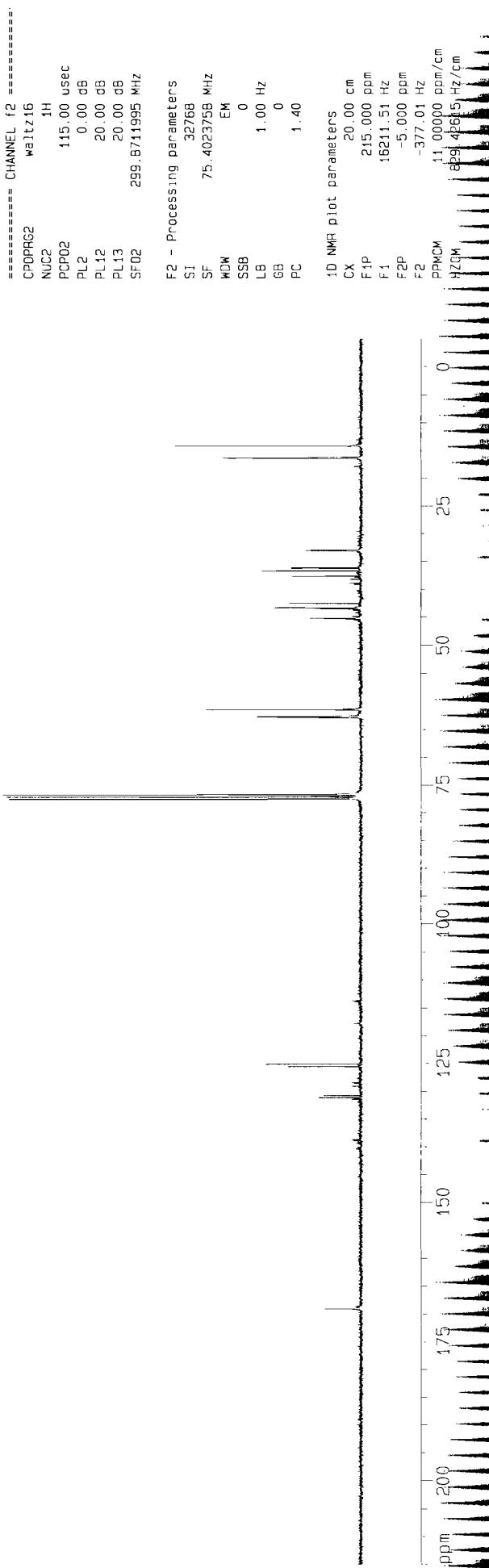
===== CHANNEL f1 =====

NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SF01 75.4106357 MHz

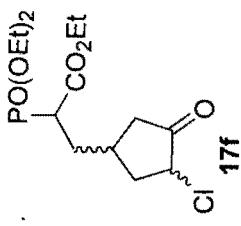
===== CHANNEL f2 =====

COPPRG2
NUC2 1H
PCP02 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SF02 299.8711995 MHz

F2 - Processing parameters
SI 32768
SF 75.4023358 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



PPM



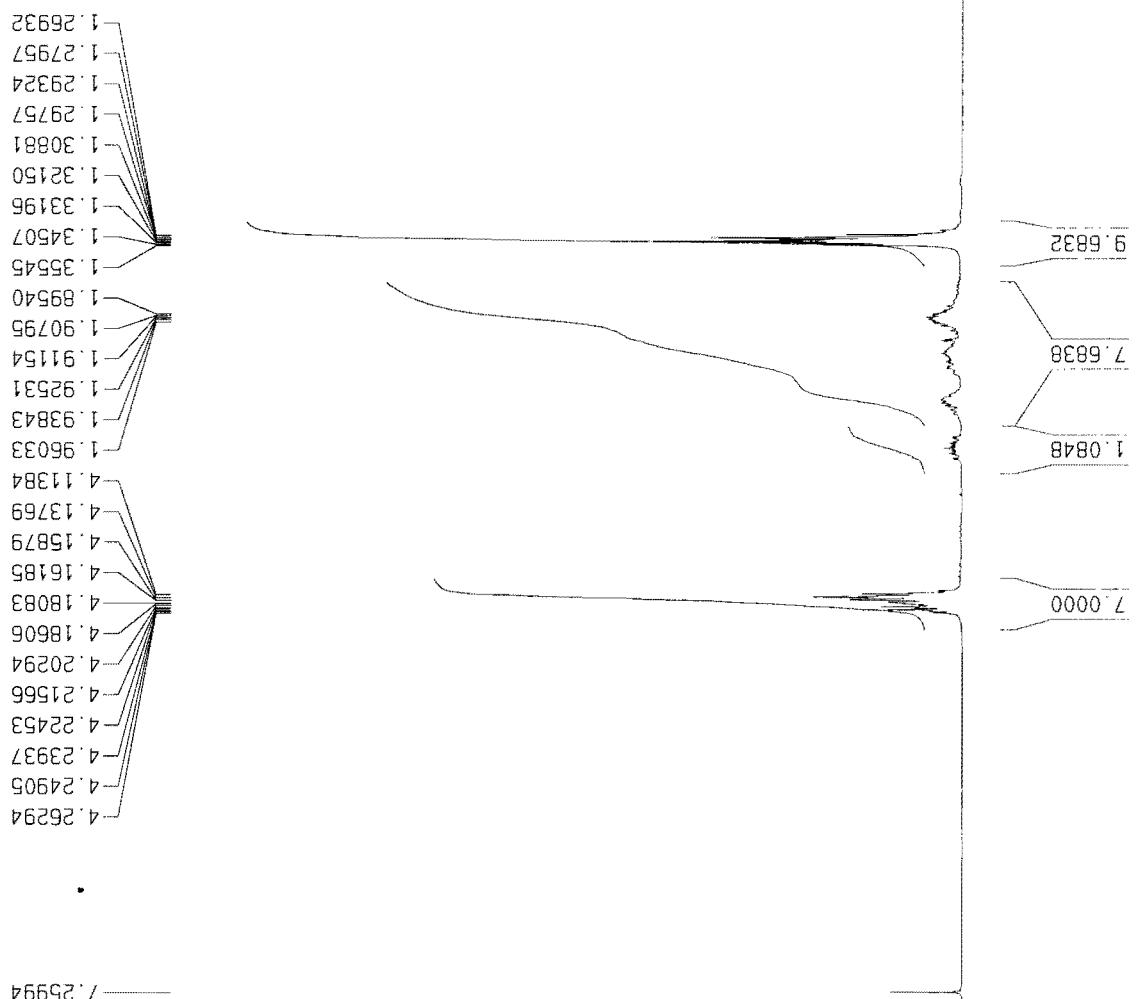
Current Data Parameters
 NAME Sep13-2010-Wen
 EXPND 10
 PROCNO 1

F2 - Acquisition Parameters
 Date .. 2010913
 Time 18.54
 INSTRUM Spect
 PROBHD 5 mm GNP 1H/1
 PULPROG zg30
 TD 55635
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 6172.839 Hz
 FIDRES 0.094190 Hz
 AQ 5.3064660 sec
 RG 256
 DW 81.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec

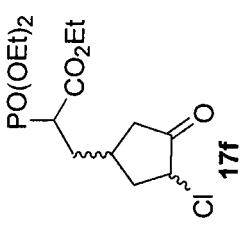
===== CHANNEL f1 =====
 NUC1 1H
 P1 11.70 usec
 PL1 0.00 0.08
 SF01 299.8718518 MHz

F2 - Processing parameters
 SI 32768
 SF 299.8700099 MHz
 WDM EM
 SS8 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 ppm
 F1P 11.000 ppm
 F1 3298.57 Hz
 -1.000 ppm
 F2P -299.87 Hz
 PPMCM 0.60000 ppm/cm
 HZCM 179.92200 Hz/cm



ppm



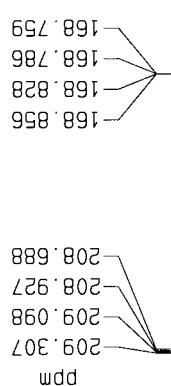
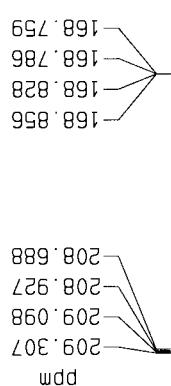
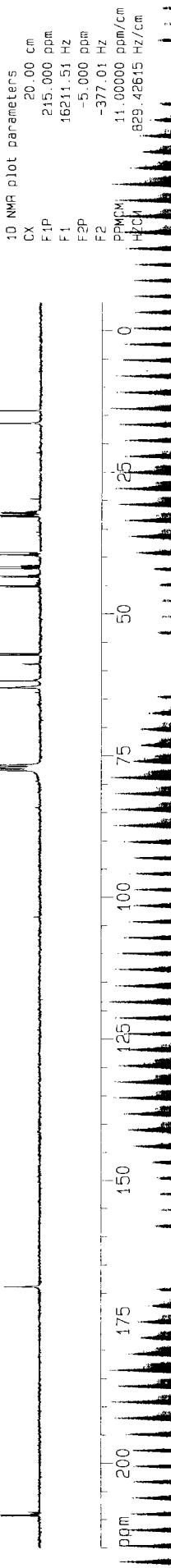
Current Data Parameters
 NAME Sep13-2010-Mein
 EXPNO 12
 PROBNO 1

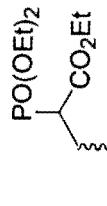
F2 - Acquisition Parameters
 Date 20100113
 Time 20:27
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zgpp30
 TD 65336
 SOLVENT CDCl3
 NS 1217
 DS 4
 SWH 18796.592 Hz
 FIDRES 0.286619 Hz
 AQ 1.7433076 sec
 RG 4096
 DW 26.00 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 D12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 5.40 usec
 PL1 -6.00 dB
 SF01 75.4106357 MHz

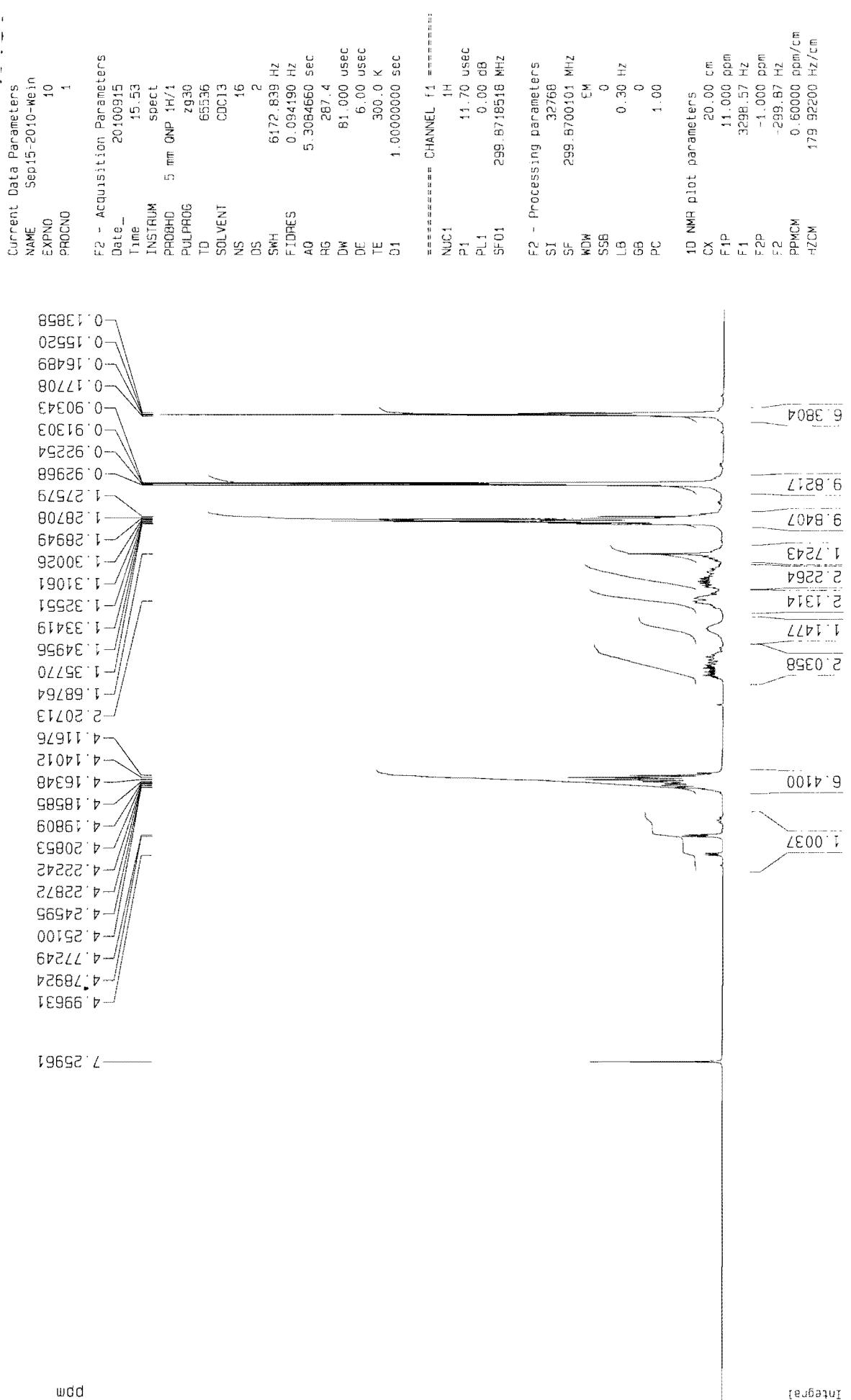
===== CHANNEL f2 =====
 CPDPFG2 Wait:16
 NUC2 1H
 PCPD2 115.00 usec
 PL2 0.00 dB
 PL12 20.00 dB
 PL13 20.00 dB
 SF02 299.8711995 MHz

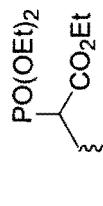
F2 - Processing Parameters
 SI 32768
 SF 75.4023247 MHz
 NOD 1 EM
 SSB 0
 LB 1.00 Hz
 GG 0
 PC 1.40





18f

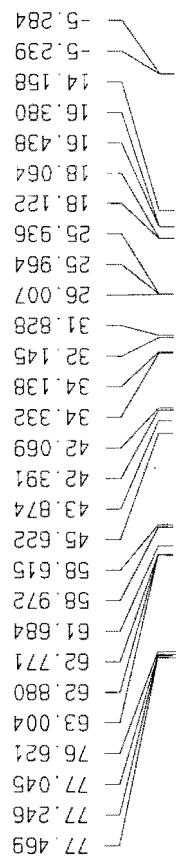




**Cl NOTBS
18f**

Current Data Parameters
NAME Sep15-2010-Wein
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date 20100915
Time 16:54
INSTRUM spect
PROBHD 5 mm GNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1674
DS 4
SWH 18796.932 Hz
FIDRES 0.266819 Hz
AQ 1.7435076 sec
RG 1024
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.000000 sec
D11 0.0300000 sec
D12 0.00002000 sec



PPM

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

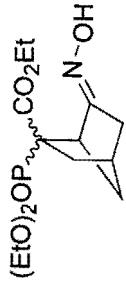
NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SF01 75.4106357 MHz

===== CHANNEL f2 =====

CPDPG2 waltz15
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SF02 299.8711995 MHz

F2 - Processing parameters
SI 32768
SF 75.4023707 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 15080.47 Hz
F2P -15.000 ppm
F2 -1131.04 Hz
PPM 10.75000 ppm/cm
Z 610.5750 Hz/cm



26

Current date = 17-Sep-2010 Mean

NAME Sep 17-2010.Mean
EXPNO 30
PROCNO 1

F2 - Acquisition Parameters

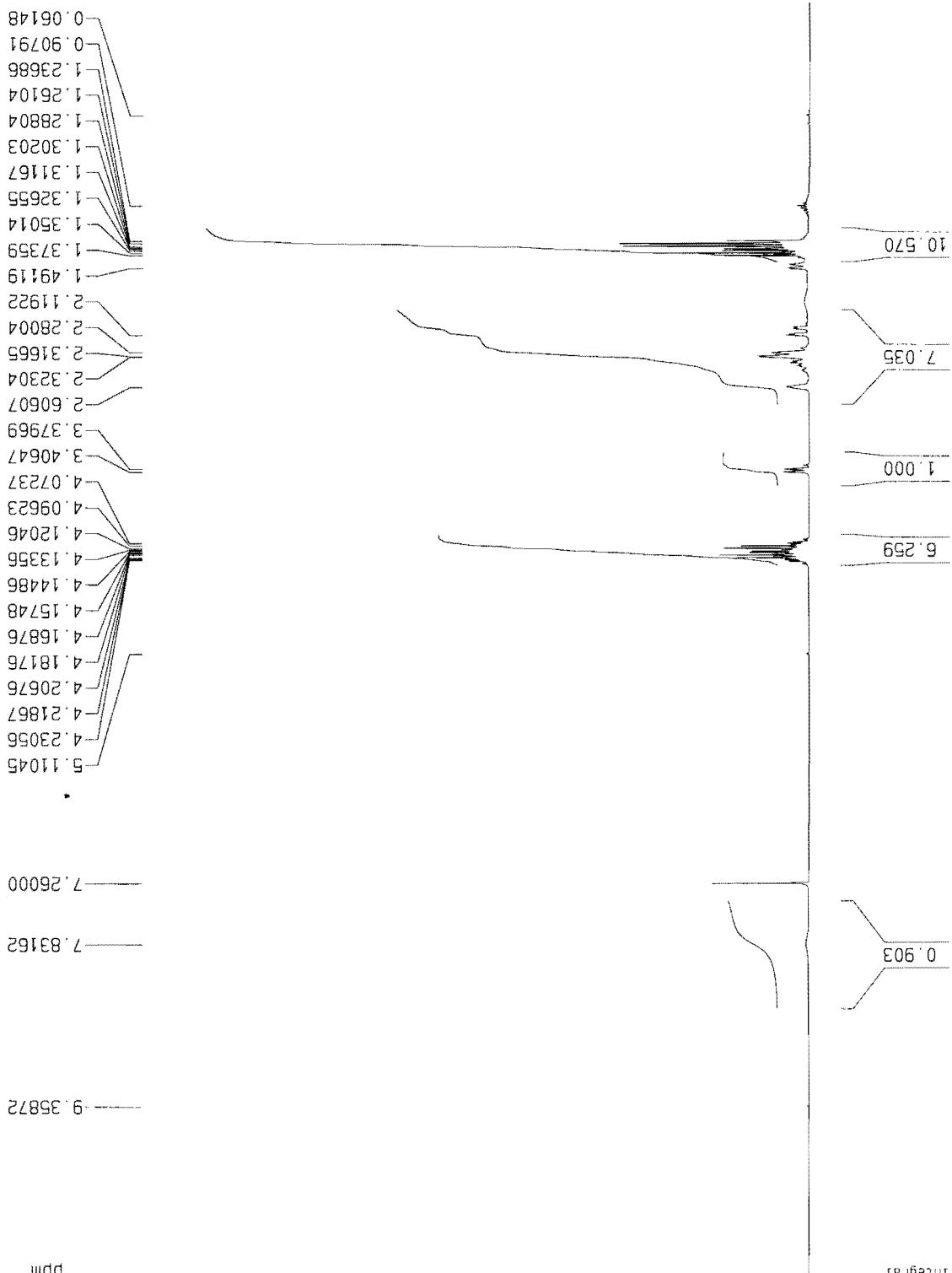
Date_	20100917
Time_	19.03
INSTRUM	spec
PROBHD	5 mm GNP 1H/1
PULPROG	2930
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIORES	0.094190 Hz
AQ	5.308660 sec
RG	456.1
DW	B1 0.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.0000000 sec

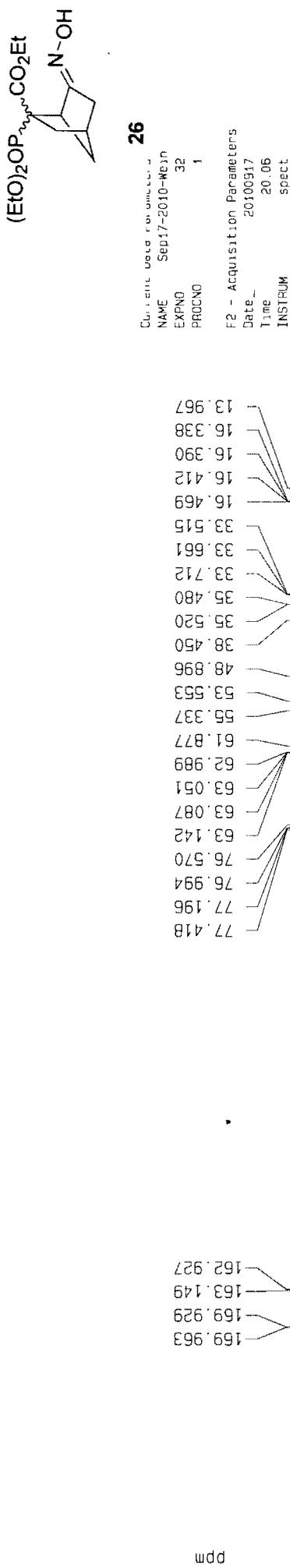
===== CHANNEL f1 =====

NUC1	1H
P1	11.70 usec
PL1	0.00 dB
SF01	299.8718516 MHz

F2 - Processing parameters

CX	20.00 cm
F1P	11.000 ppm
F1	3298.57 Hz
F2P	-1.000 ppm
F2	-299.87 Hz
PPMCM	0.50000 ppm/cm
HZCM	179.95200 Hz/cm





Chemical shift data for compound 26:

NAME	Sep17-2010-Wet
EXPO	32
PROCNO	1

F2 - Acquisition Parameters

Date	2010-05-17
Time	20:06
INSTRUM	spec
PROBHD	5 mm QNP 1H/1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl ₃
NS	14782
DS	4
SWH	18796.992 Hz
TDRES	0.286619 Hz
AG	1.7433076 sec
RG	1024
DW	26.000 usec
DE	6.00 usec
TE	300.0 K
D1	2.0000000 sec
D11	0.0300000 sec
D12	0.00002000 sec

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

NUC1	13C
P1	5.40 usec
PL1	-6.00 dB
SF01	75.4106357 MHz

===== CHANNEL f2 =====

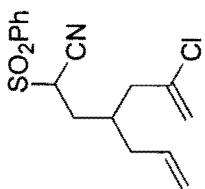
CPDPGR2	Walt16
NUC2	1H
PCPD2	115.00 usec
PL2	0.00 dB
PL12	20.00 dB
PL13	20.00 dB
SF02	299.8711995 MHz

F2 - Processing parameters

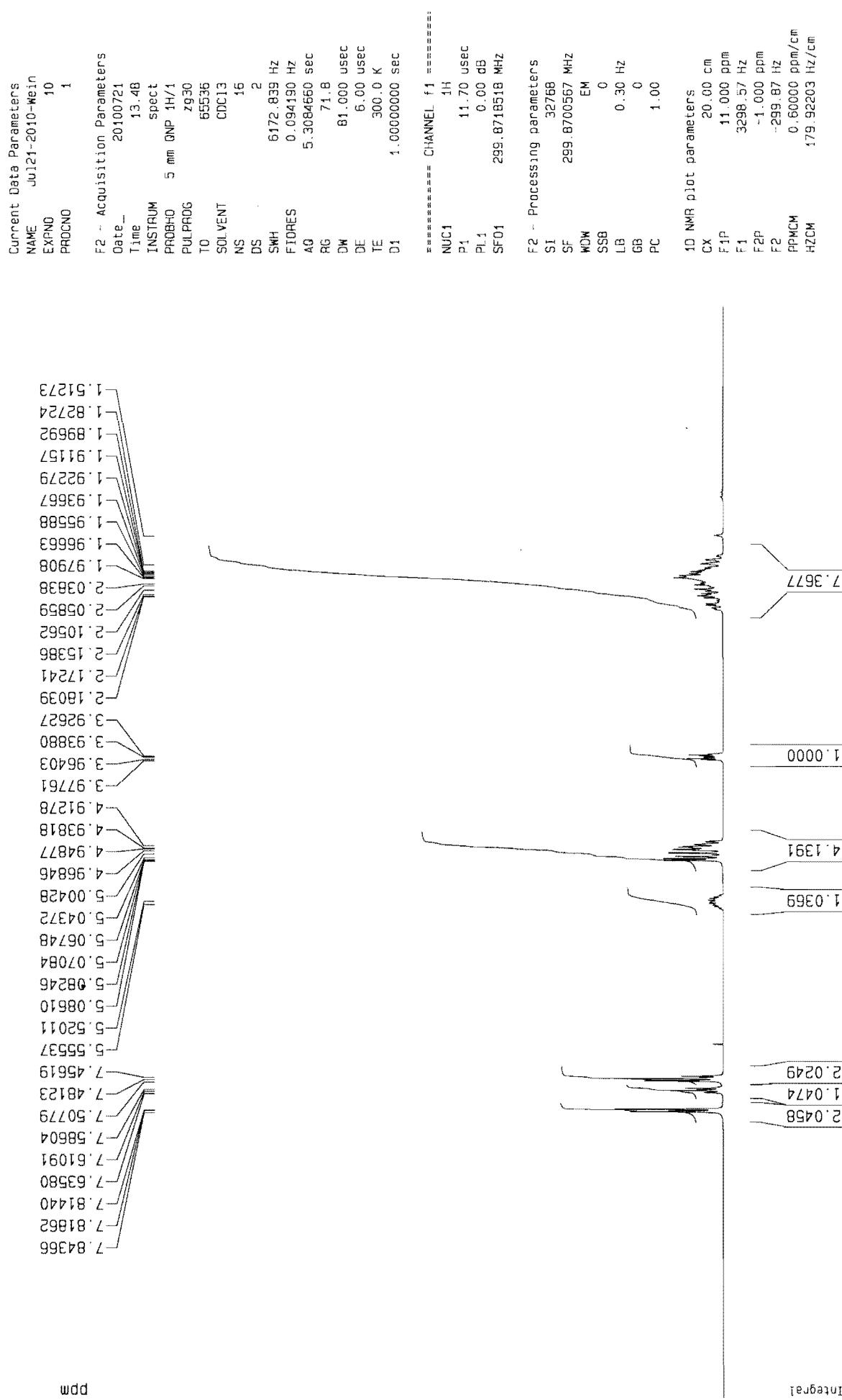
SI	32768
SF	75.4023147 MHz
NDDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

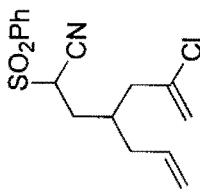
1D NMR Plot parameters

CX	20.00 cm
CP	215.00 ppm
F1P	16211.51 Hz
=2D	-5.000 ppm
F2	-377.01 Hz
PPMCK	11.0000 ppm/cm
HCM	829.42215 Hz



15g





15g

Current Data Parameters

NAME JU121-2010-Wein
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters

Date 20100721
Time 13.57
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG 290030
TD 65536
SOLVENT CDCl₃
NS 93
DS 4
SWH 18796.992 Hz
FIDRES 0.285619 Hz
AQ 1.7433076 sec
RG 4096
DW 25.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.000000 sec
D11 0.0300000 sec
D12 0.00002000 sec

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

NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SF01 75.4106357 MHz

===== CHANNEL f2 =====

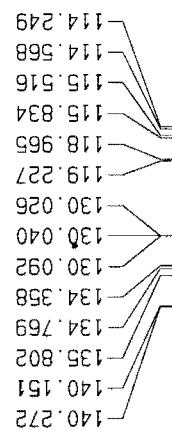
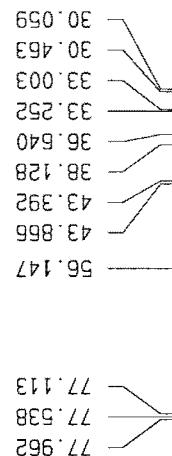
CPDPFG2
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SF02 299.8711995 MHz

F2 - Processing parameters

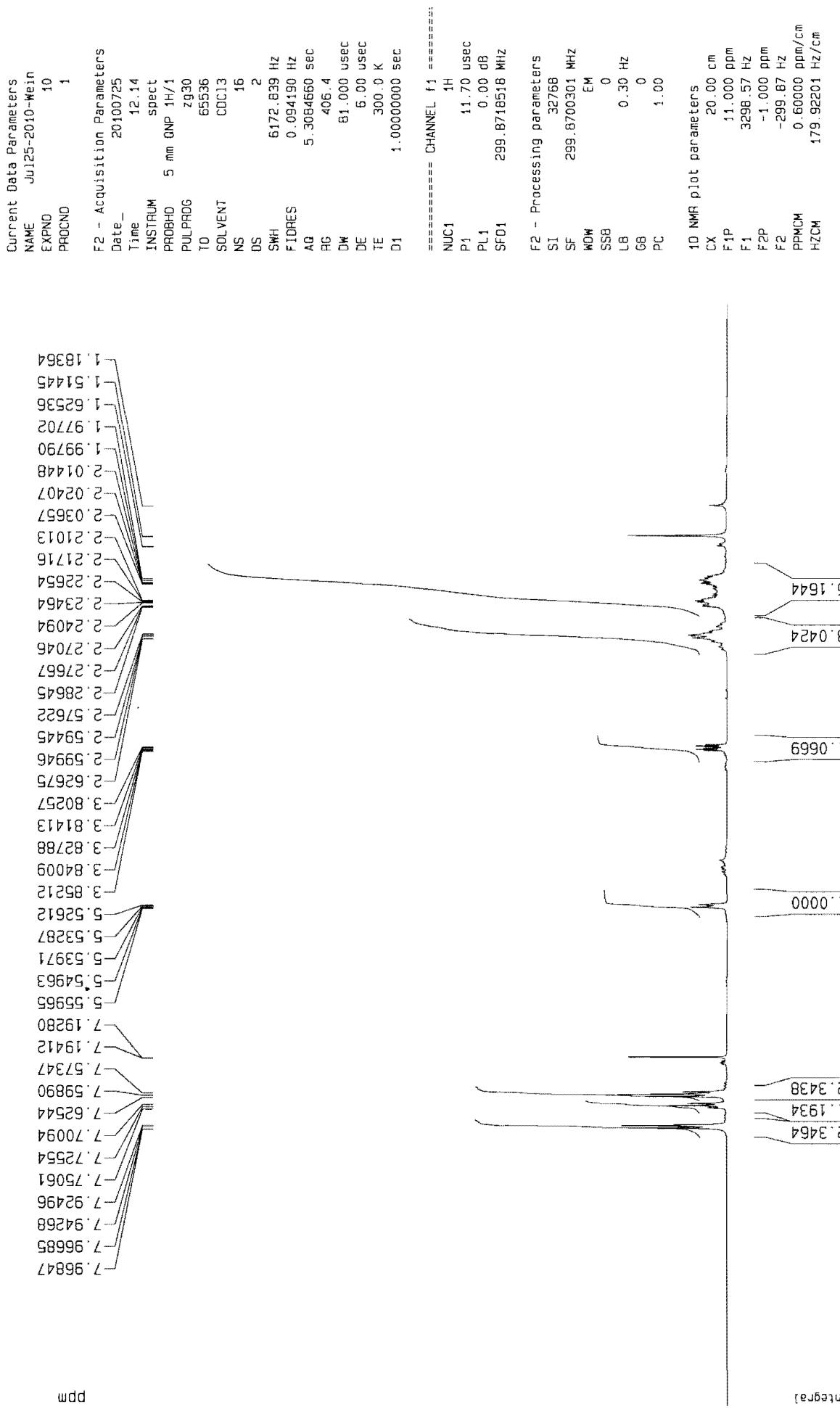
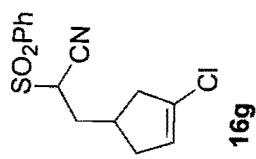
SI 32768
SF 75.4023410 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

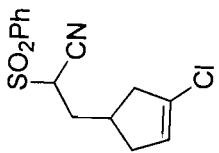
1D NMR plot parameters

CX 20.00 cm
F1P 215.00 ppm
F1 16211.50 Hz
F2P -5.000 ppm
F2 -377.01 Hz
PPMCM 11.00000 ppm/cm
HZCM 829.42578 Hz/cm

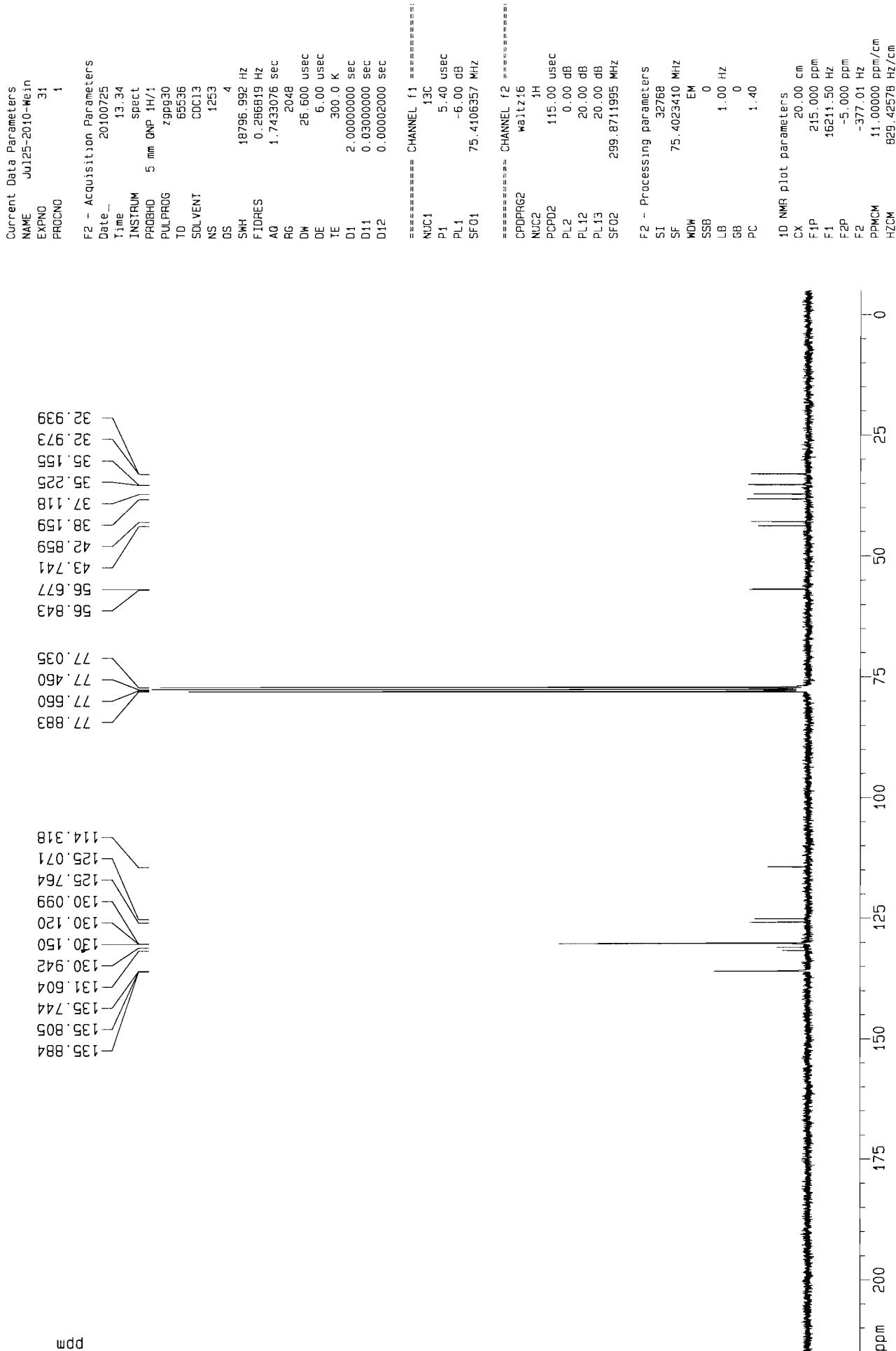


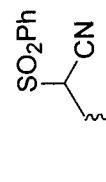
ppm





16g





17g

Current Data Parameters
NAME Ju12B-2010-Mein
EXPNO 10
PROCNO 1

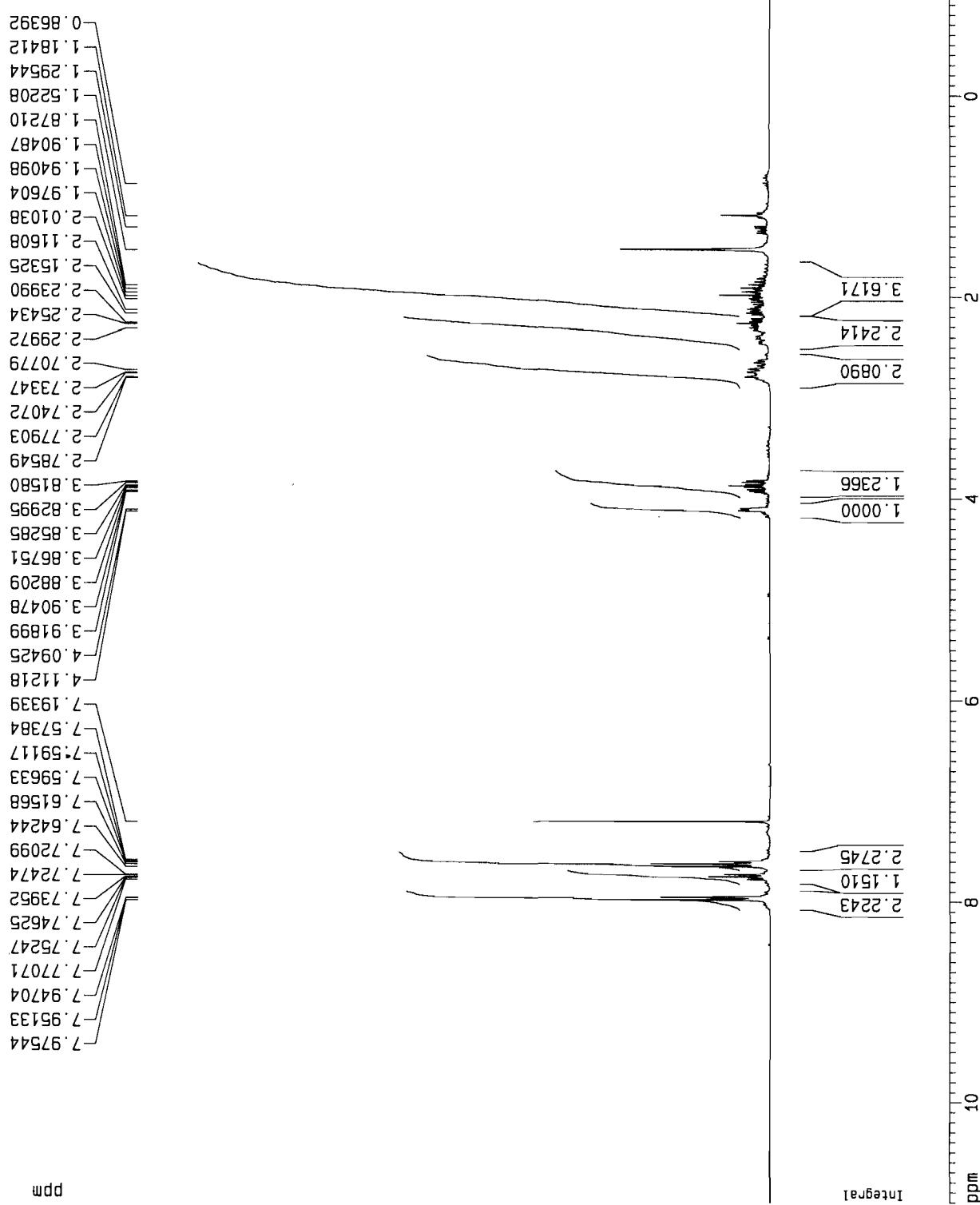
F2 - Acquisition Parameters
Date 20100728
Time 19:13

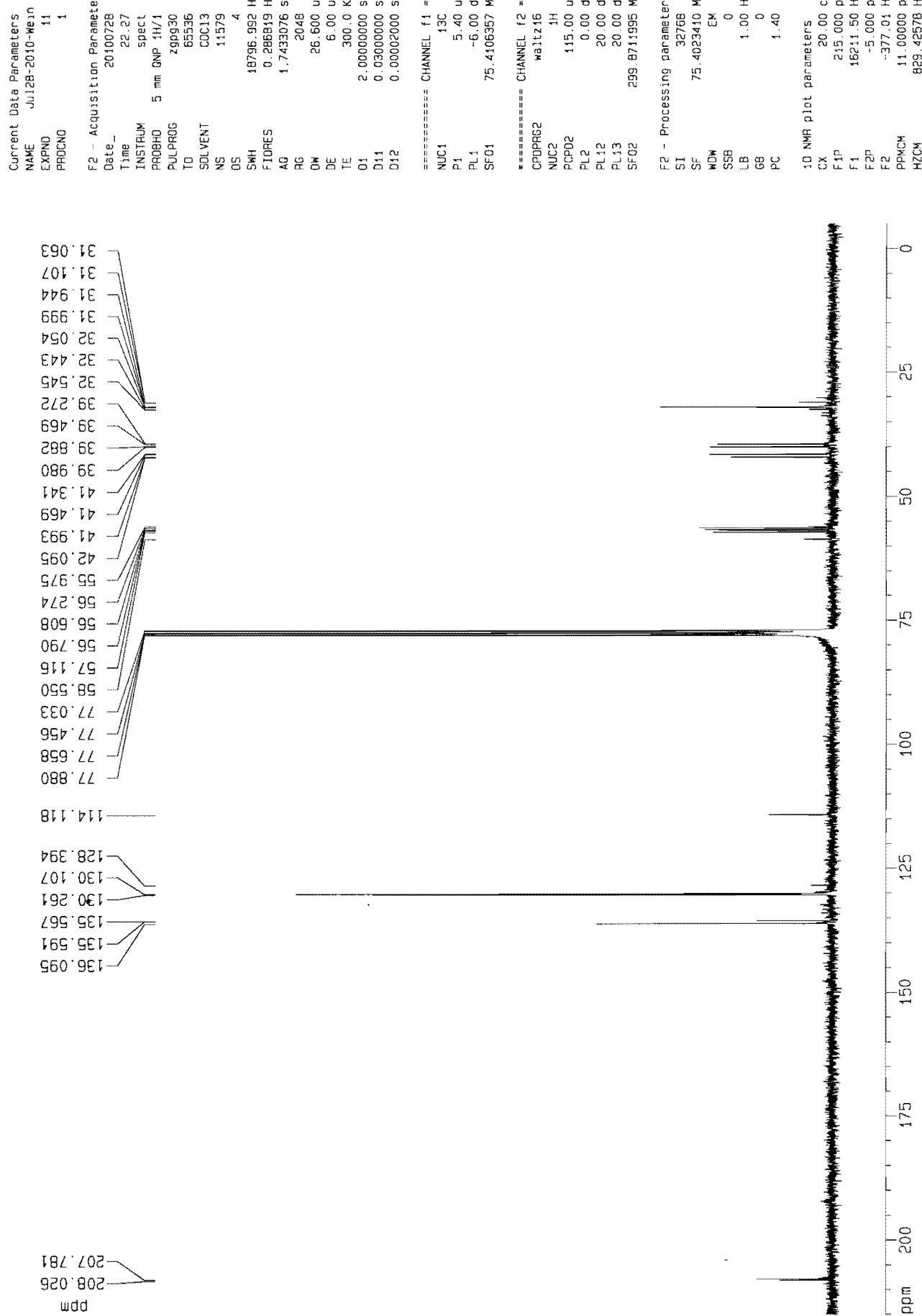
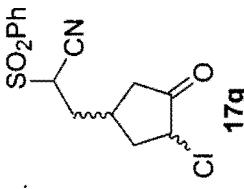
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084650 sec
RG 512
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.0000000 sec

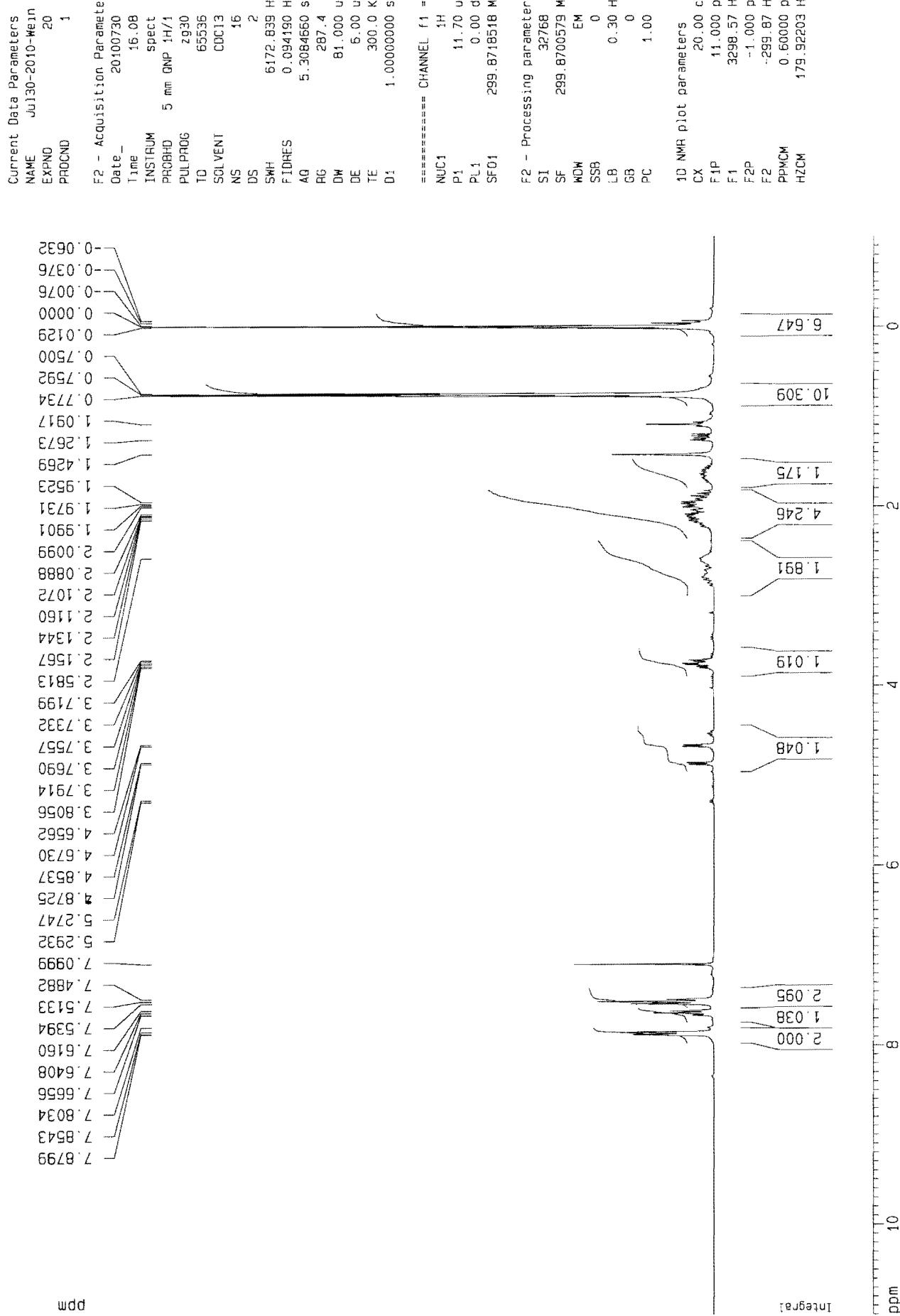
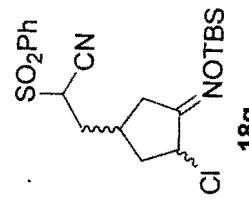
===== CHANNEL f1 =====
NUC1 1H
P1 9.60 usec
PL1 -6.00 dB
SF01 300.1319534 MHz

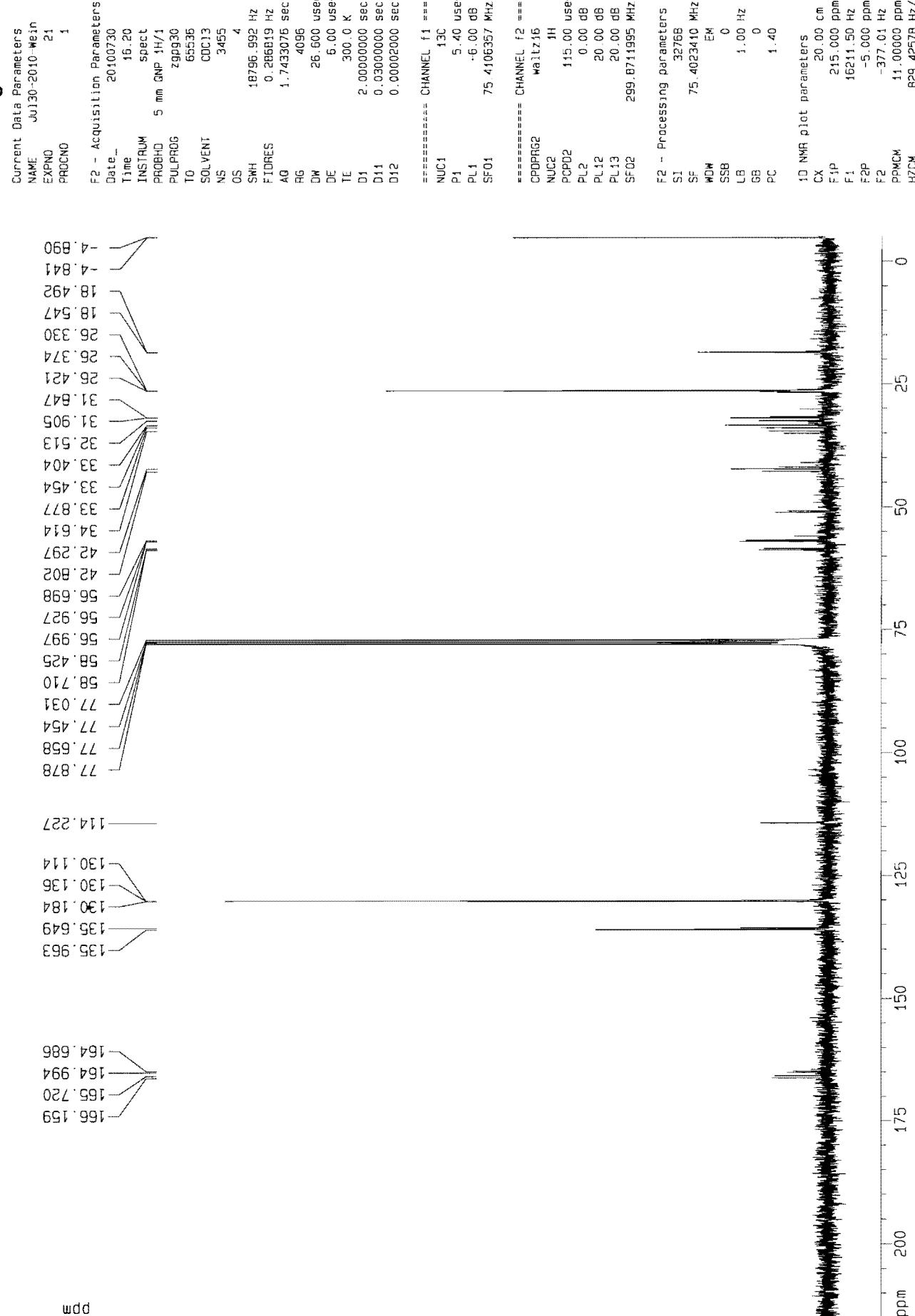
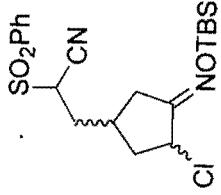
F2 - Processing parameters
SI 32768
SF 300.13000263 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

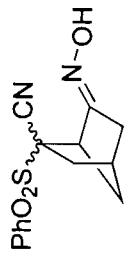
1D NMR plot parameters
CX 20.00 cm
F1P 11.000 ppm
F1 3301.43 Hz
F2P -1.000 ppm
F2 -300.13 Hz
PPMCM 0.60000 ppm/cm
HZCM 180.07802 Hz/cm











27

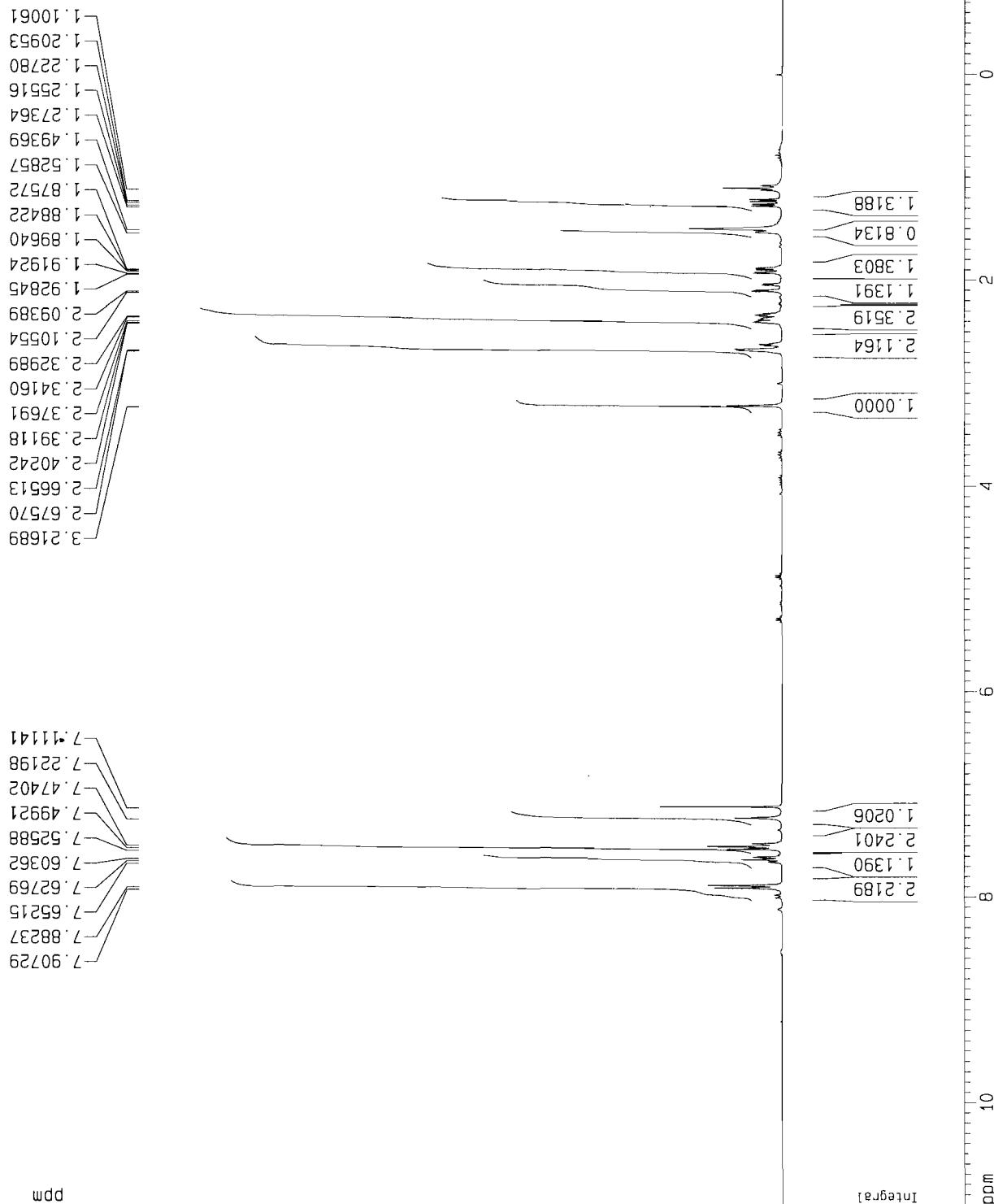
Current Data Parameters
NAME Aug01-2010-Ne1n
EXPNO 10
PROCNO 1

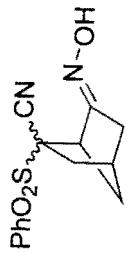
F2 - Acquisition Parameters
Date 20100801
Time 11.20
INSTRUM Spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 41
DS 0
SWH 6172.839 Hz
FIDRES 0.094150 Hz
AQ 5.3084650 sec
RG 574.7
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 11.70 usec
PL1 0.00 dB
SF01 299.8718518 MHz

F2 - Processing parameters
SI 32768
SF 299.8705055 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR pilot parameters
CX 20.00 cm
F1P 11.000 ppm
F1 3298.57 Hz
F2P -1.000 ppm
F2 -299.87 Hz
PPMCM 0.60000 ppm/cm
HZCM 179.92203 Hz/cm





Current Data Parameters

NAME Aug01-2010-Nein
EXPNO 12
PROCNO 1

F2 ~ Acquisition Parameters

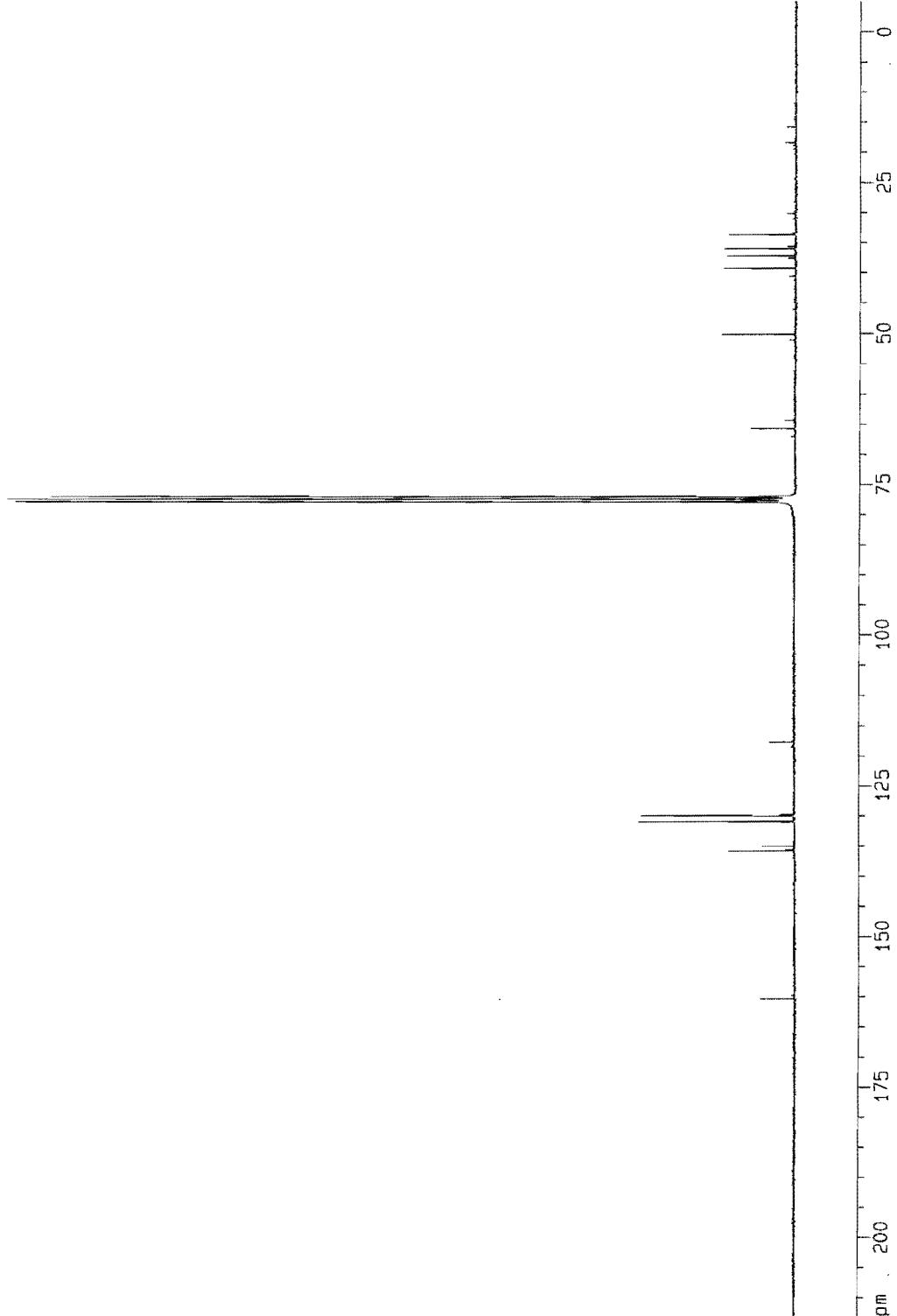
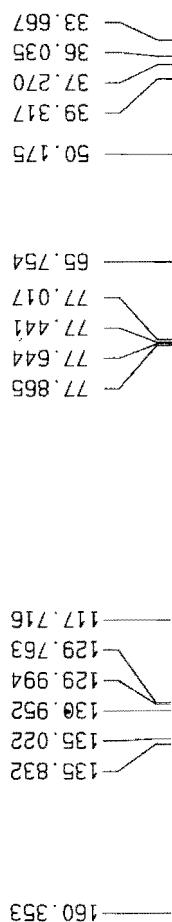
Date 20100801
Time 13:01
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
FIDRES 0.286819 Hz
AQ 1.7433076 SEC
RG 3649.1
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 SEC
D11 0.0300000 SEC
D12 0.00002000 SEC

===== CHANNEL f1 ======
CPDPAG2 13C
NUC2 1H
PCPD02 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SF01 75.4106357 MHz
SF02 299.8711595 MHz

F2 ~ Processing parameters

SI 32768
SF 75.4023410 MHz
DW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

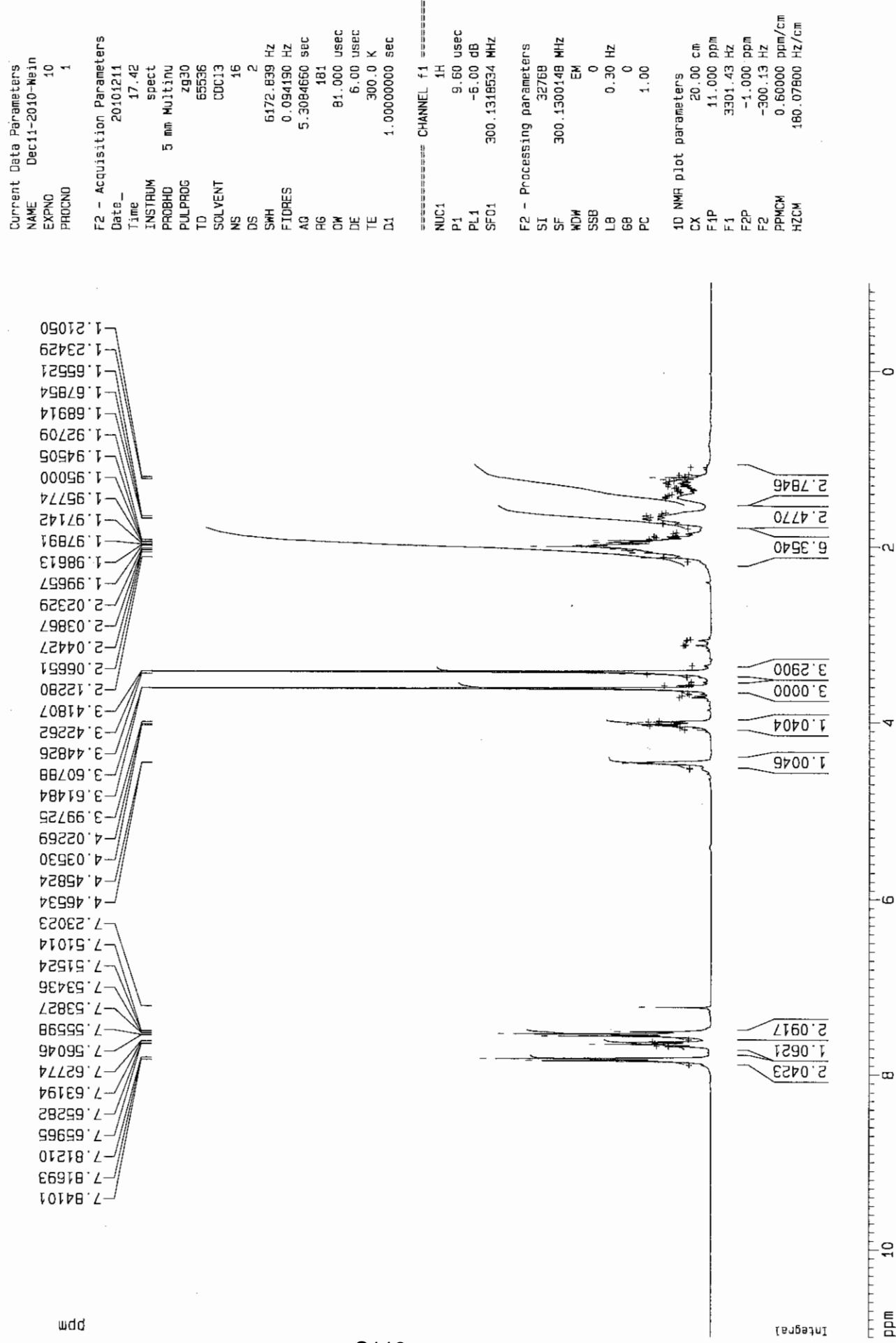
1D NMR plot parameters
CX 20.00 cm
F1P 215.00 ppm
F1 16241.50 Hz
F2P -5.000 ppm
F2 -377.01 Hz
PPCM 11.00000 ppm/cm
HZCM 829.42378 Hz/cm





30

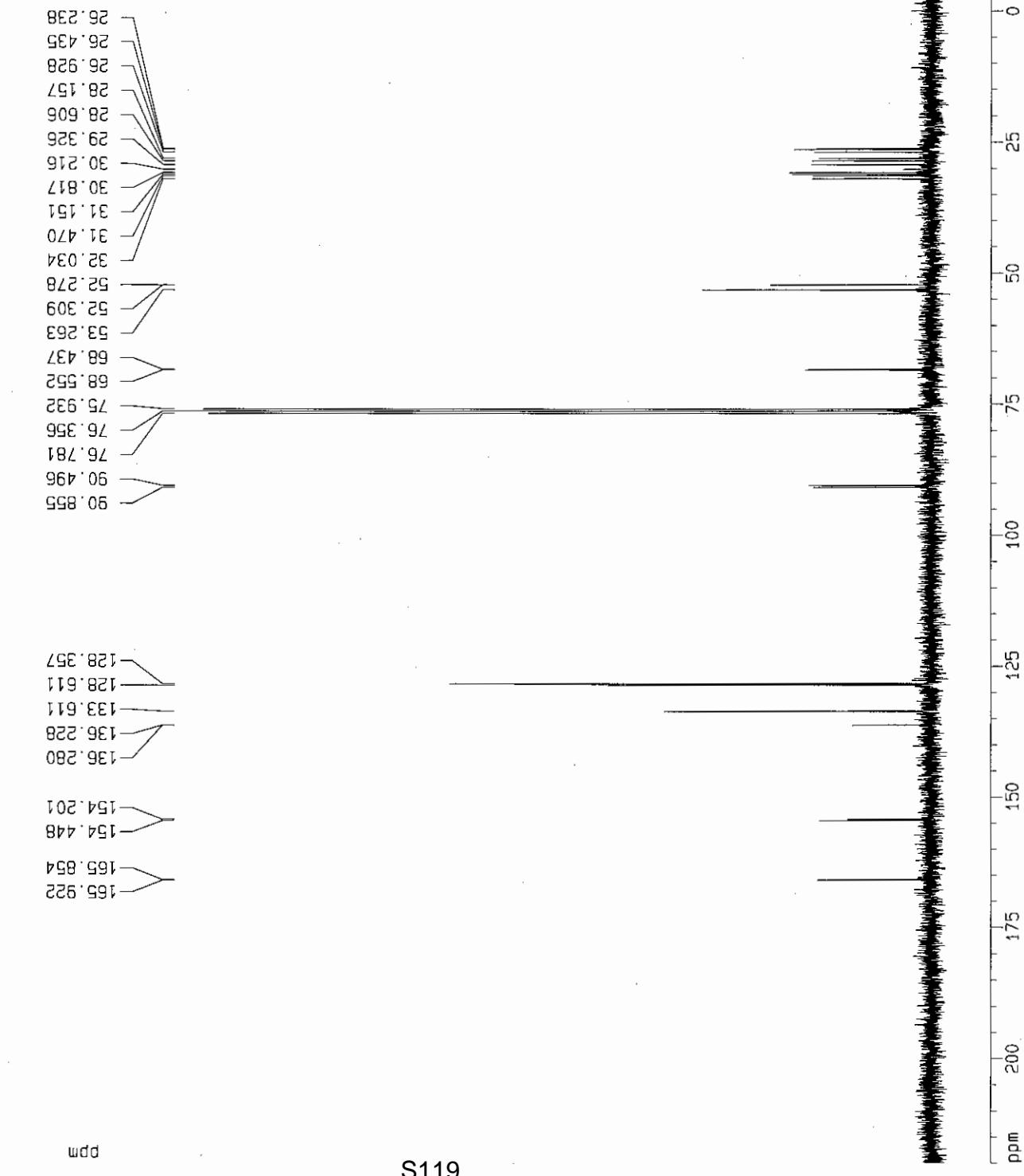
P_L - II - p211 - P

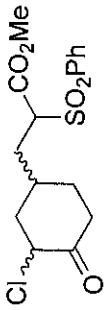




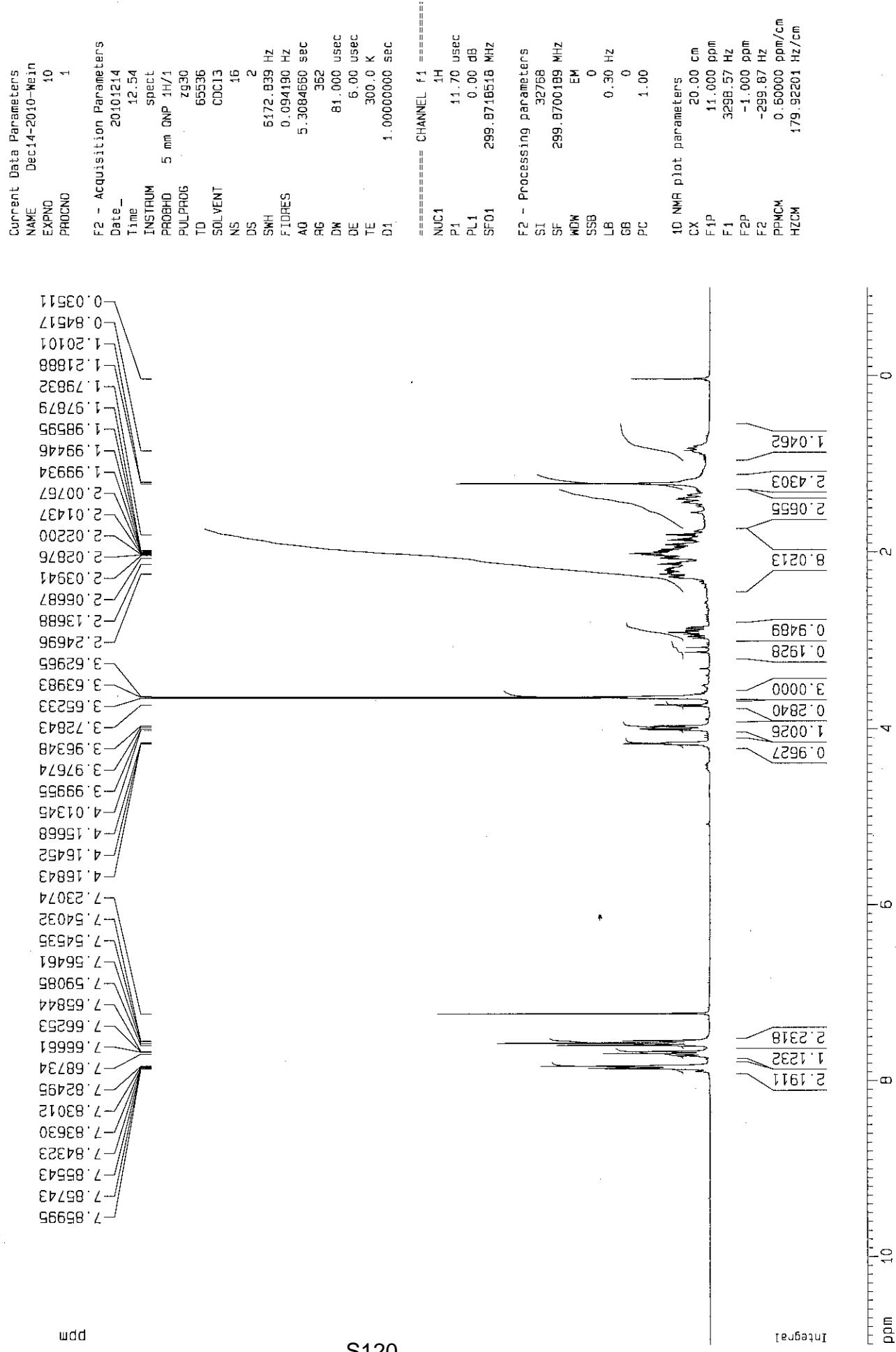
30

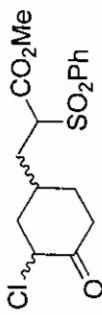
PL - II - p205 - P



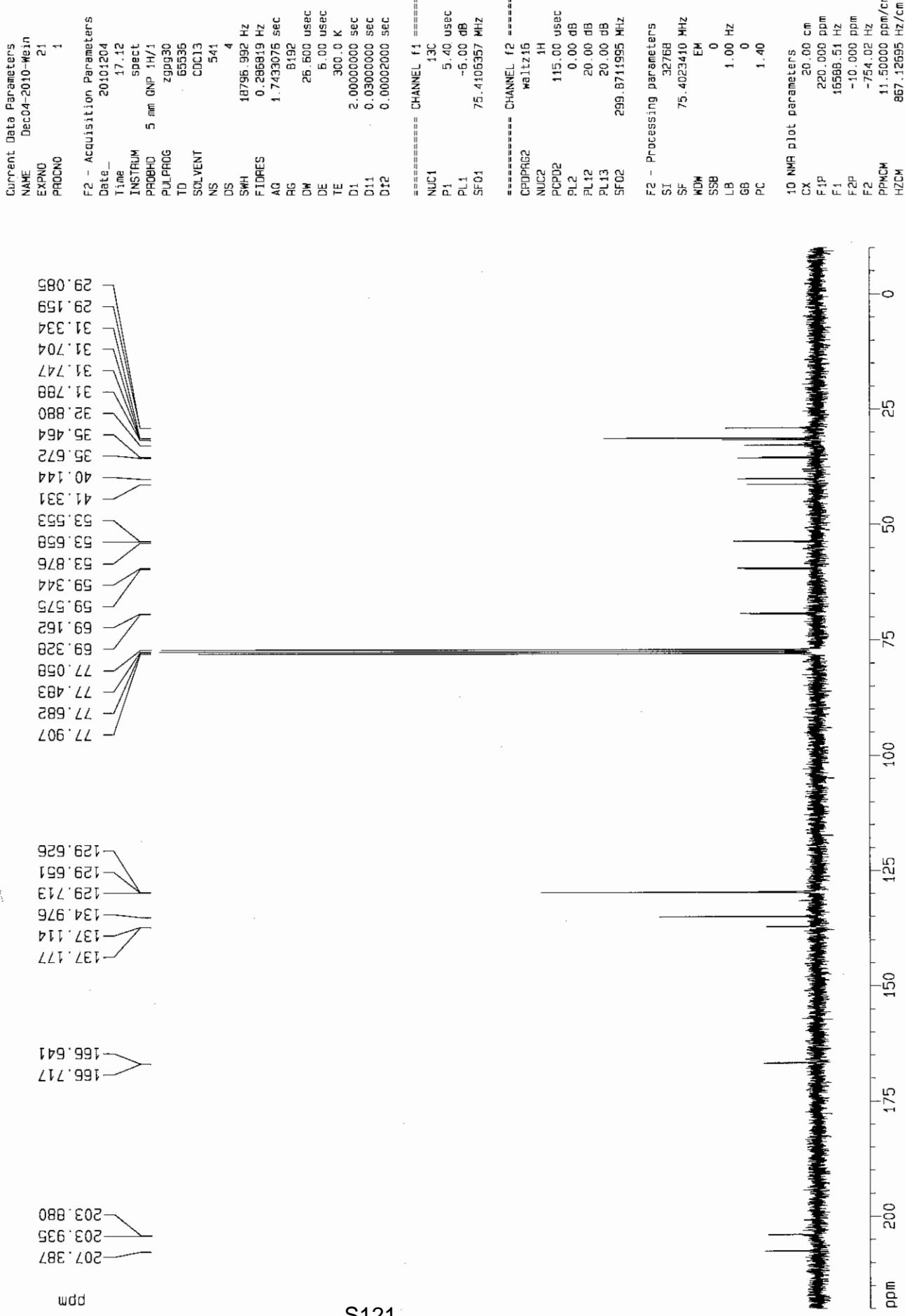


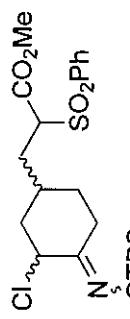
PL - II - p212 - P



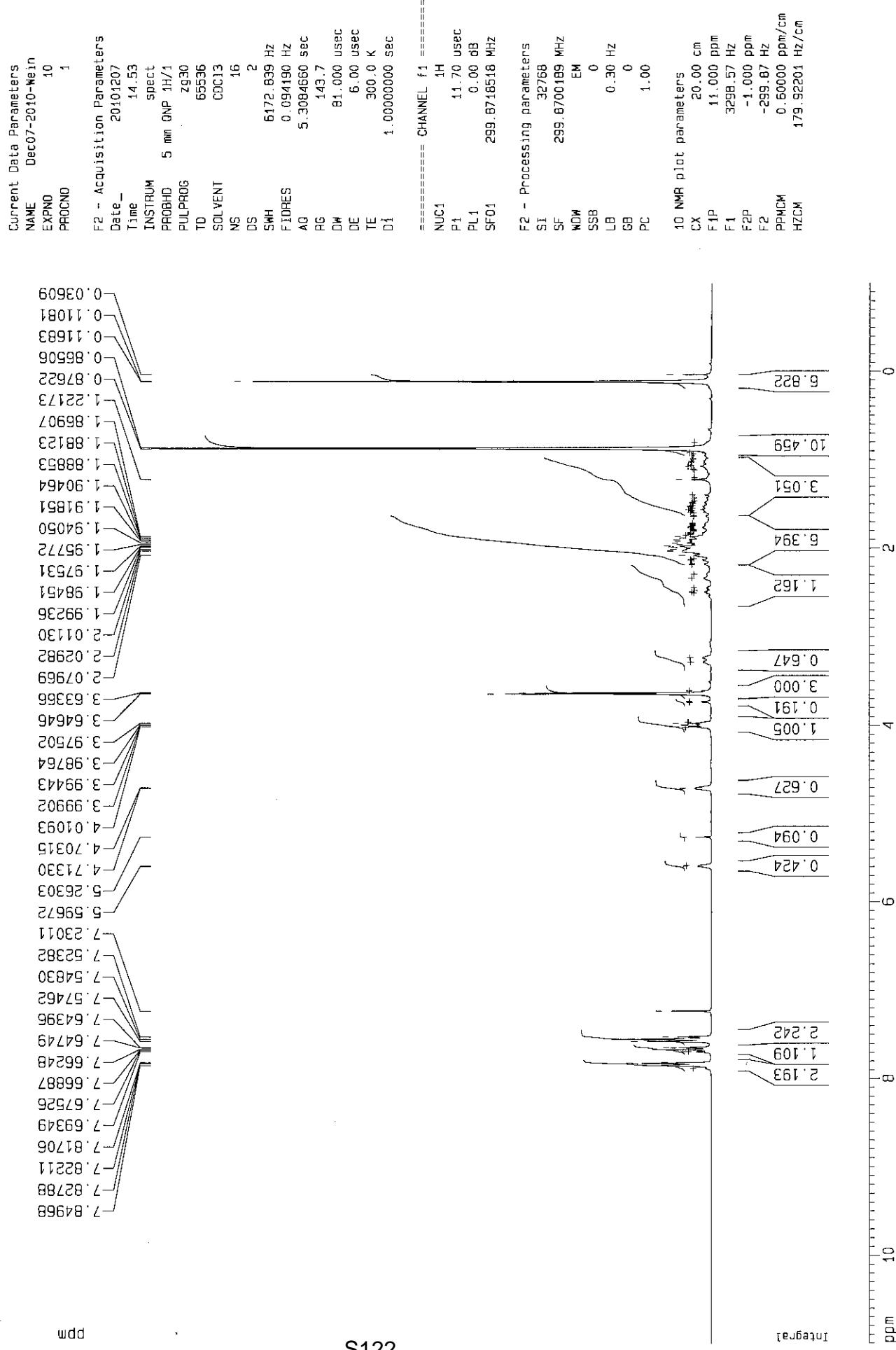


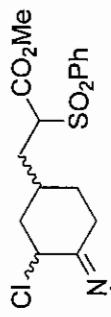
PL - II - p205 - P(mix)



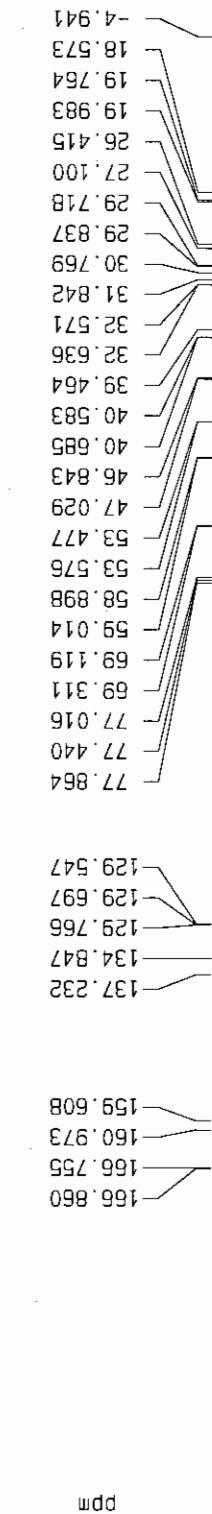


PL - II - p207 - P





PL - II - p207 - P



Current Data Parameters
NAME Dec07-2010-Mein
EXPNO 11
PROCNO 1

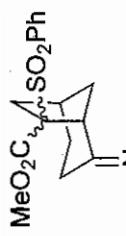
F2 - Acquisition Parameters
Date_ 20101207
Time 14.59
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg90f30
TD 65536
SOLVENT CDCl3
NS 620
DS 4
SWH 18795.992 Hz
FIDRES 0.286819 Hz
AQ 1.7433076 sec
RG 8192
DW 600 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec
D11 0.0300000 sec
D12 0.0000200 sec

===== CHANNEL f1 =====
NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SF01 75.4106357 MHz

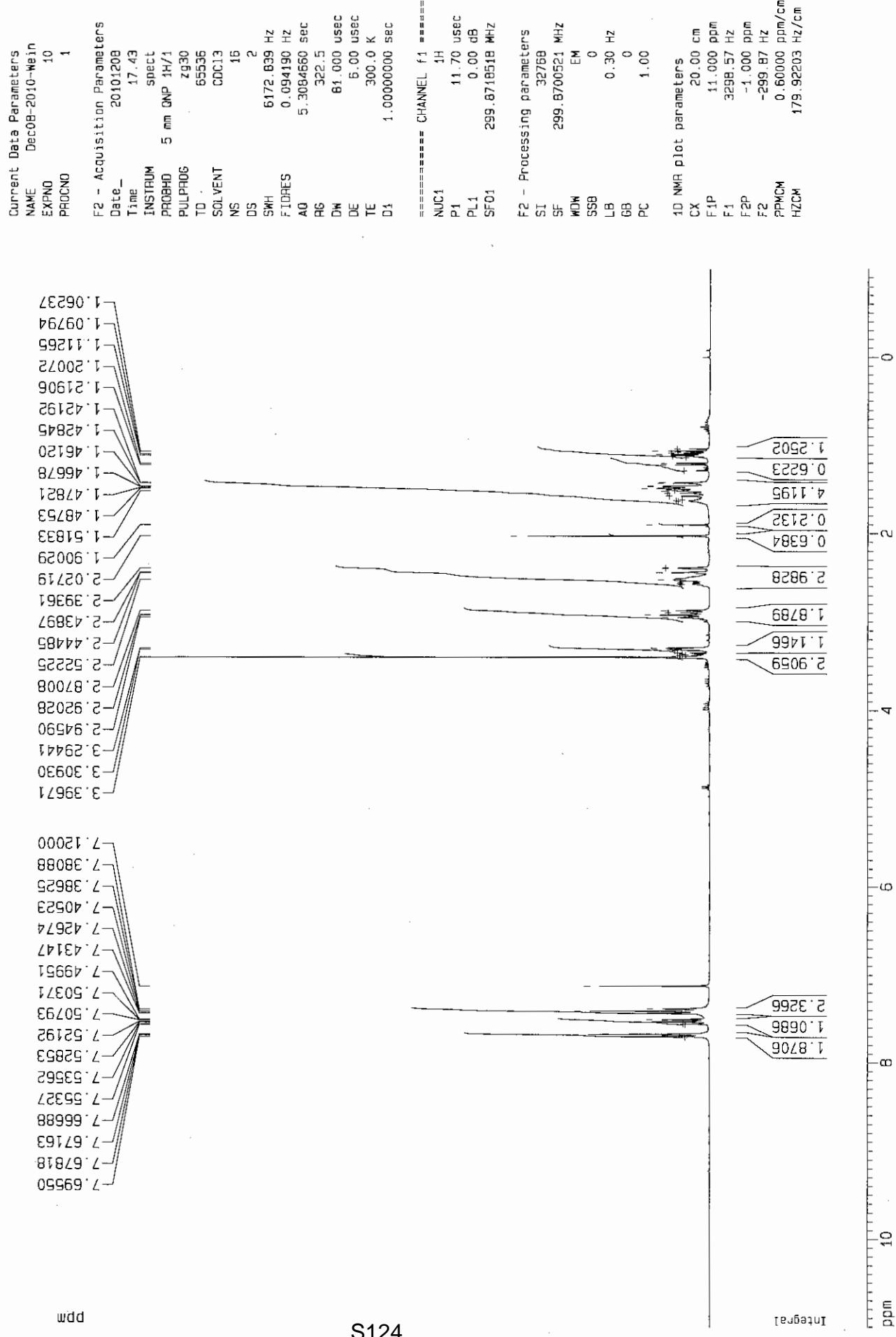
===== CHANNEL f2 =====
CPDPG2 W11216
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SF02 299.8711995 MHz

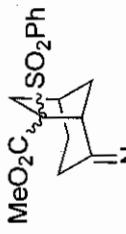
F2 - Processing parameters
SI 32768
SF 75.4023410 MHz
WIDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR pilot parameters
CX 20.00 cm
F1,P 220.000 ppm
F1 165.88 51 Hz
F2,P -10.000 ppm
F2 -75.4.02 Hz
PPMCM 11.50000 ppm/cm
HZCM 867.12695 Hz/cm



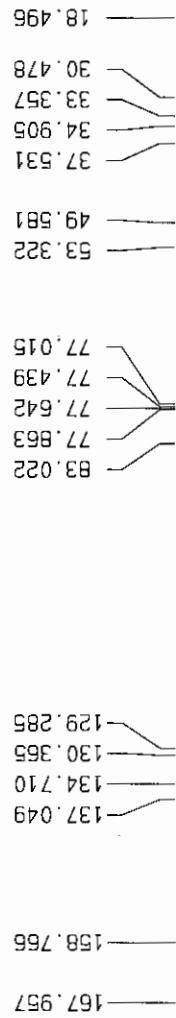
PL - II - p20B - P





HO⁻ 33a

PL - II - 208 - P



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Current Date Parameters
NAME Dec08-2010-Wein
EXPO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20101208
Time_ 17.47
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT DDC13
NS 1024
DS 4
SWH 18796.992 Hz
ETRIM 0.2866819 Hz
AQ 1.7433076 sec
RG 16384
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec
D11 0.0300000 sec
D12 0.00002000 sec

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===== CHANNEL f1 =====
NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SF01 75.4106357 MHz

===== CHANNEL f2 =====
CPDPG2 WALTz15
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SF02 299.8711995 MHz

F2 - Processing parameters
SI 32768
SF 75.4023410 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

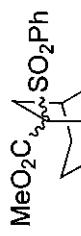
```

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1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 16500.51 Hz
-10.000 ppm
F2P -754.02 Hz
F2 11.50000 ppm/cm
PPCM 667.12695 Hz/cm
HZDN

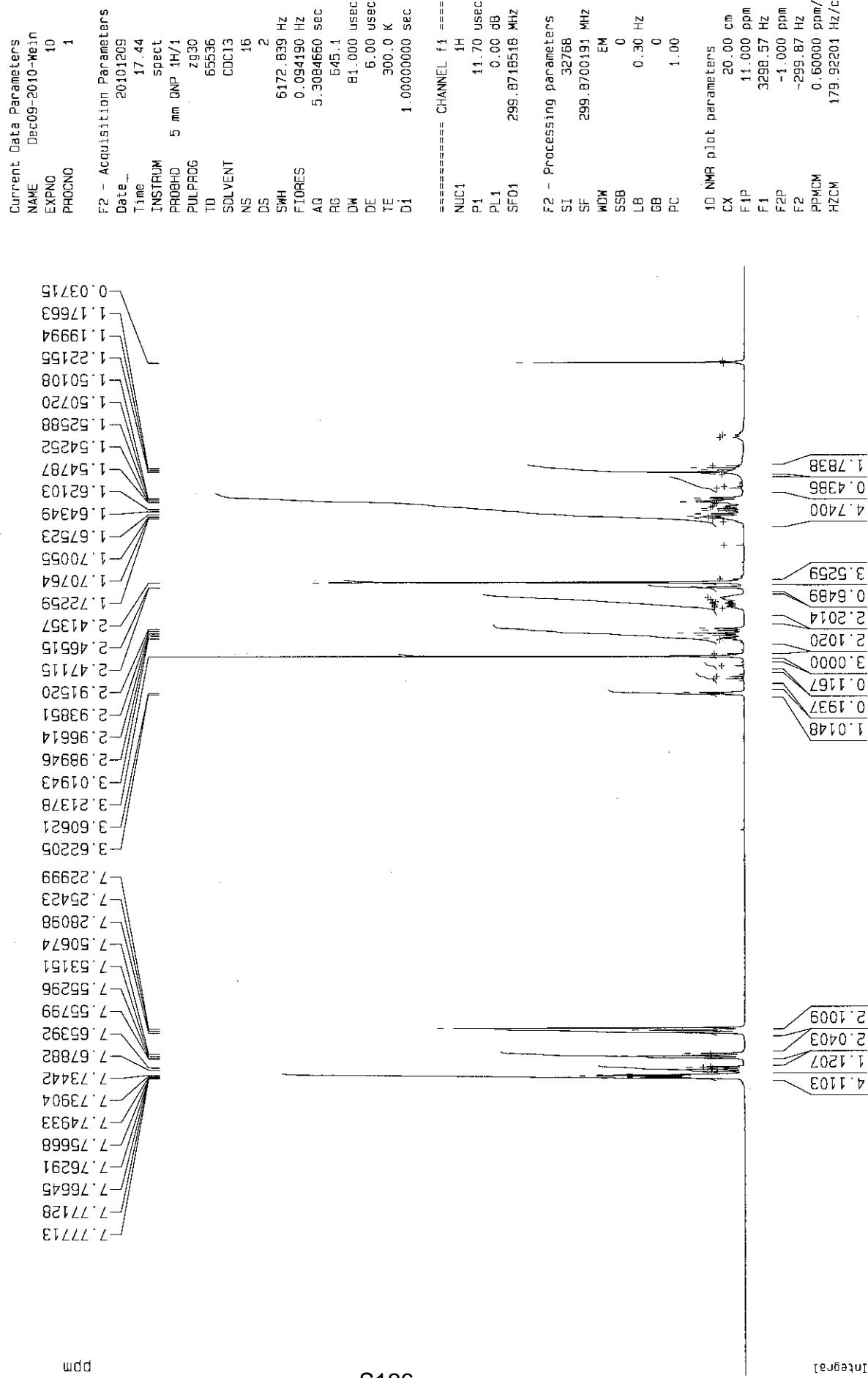
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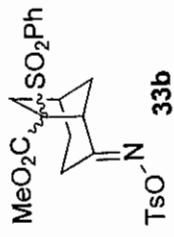
ppm



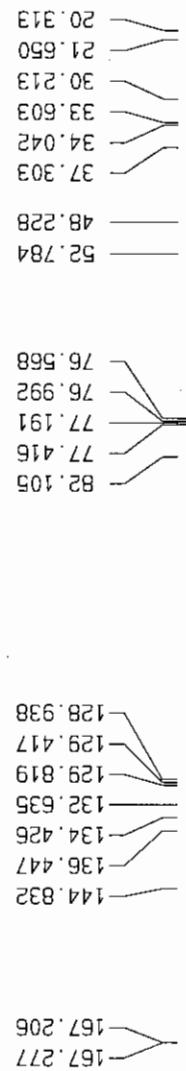
33b

PL - II - p209 -





Puhui Tosylate



Current Data Parameters
NAME Dec10-2010 Wein
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters

Date 20101210
Time 10:33
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg930
TD 65536
SOLVENT CDCl3
NS 3682
DS 4
SWH 18796.992 Hz
FIDRES 0.286819 Hz
AQ 1.7433076 sec
RG 16384
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec
D11 0.0300000 sec
D12 0.0000200 sec

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

NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SF01 75.4106357 MHz

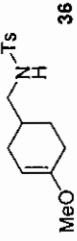
===== CHANNEL f2 =====

OPDPMG2 WALTZ16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SF02 299.8711995 MHz

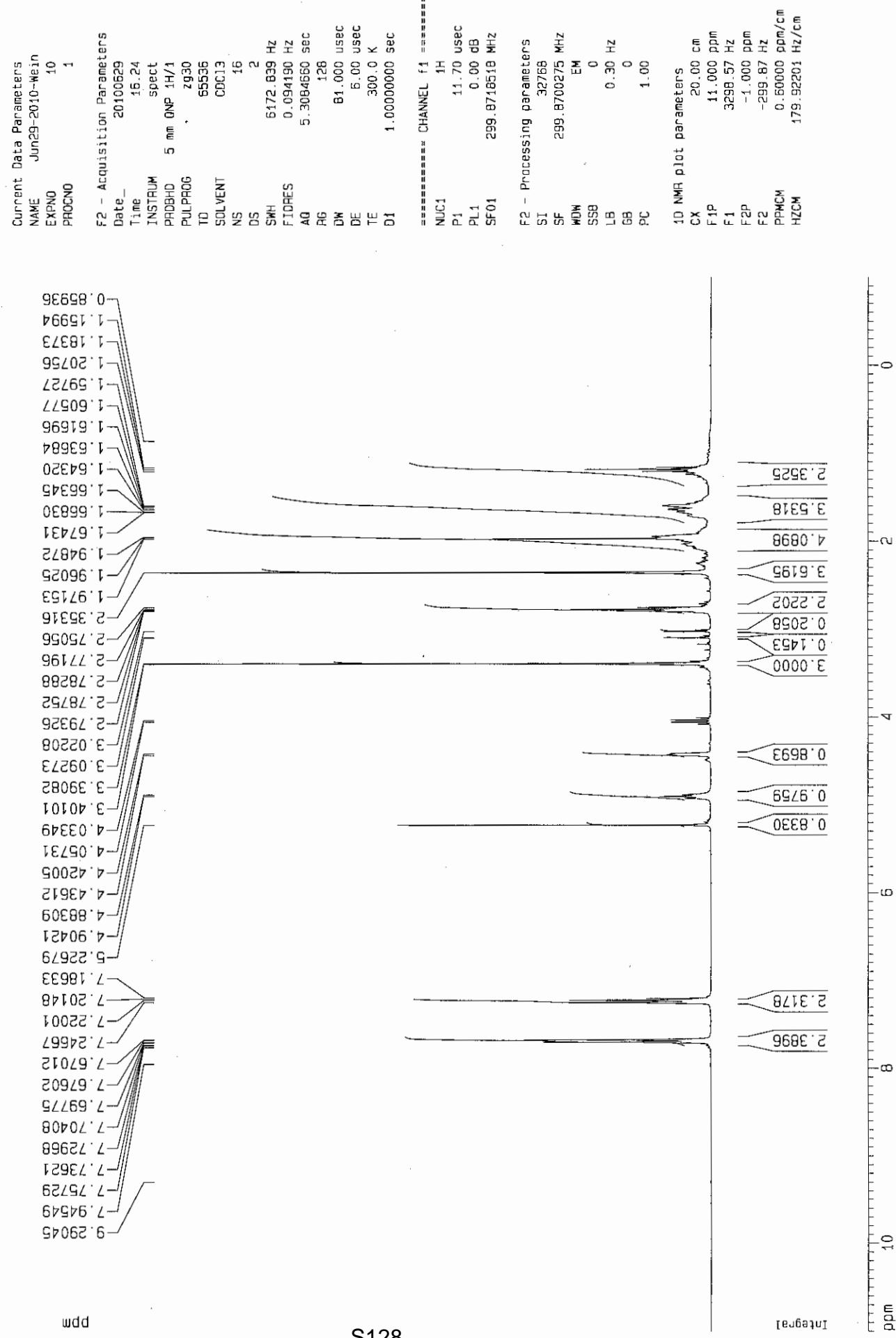
F2 - Processing parameters

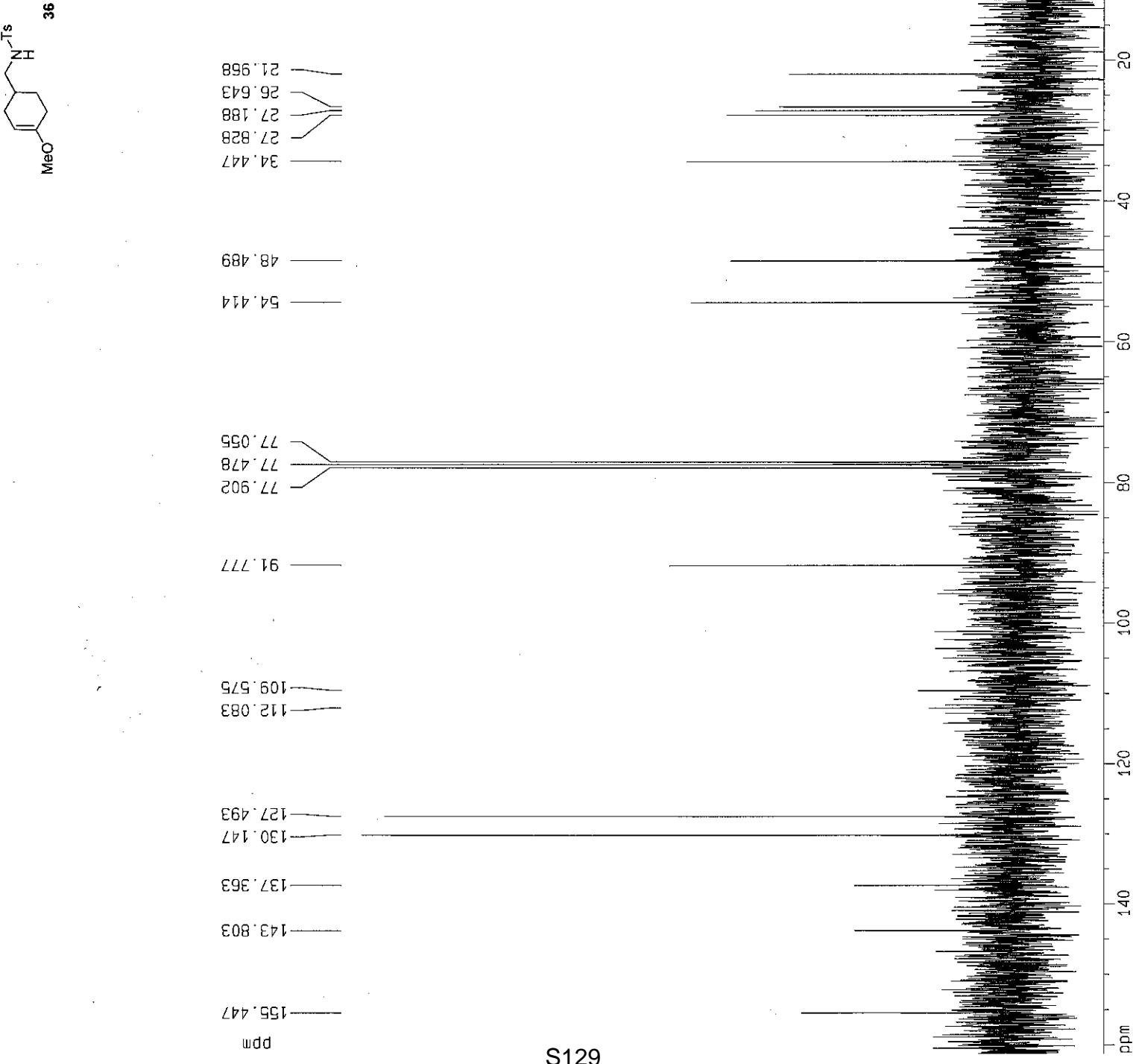
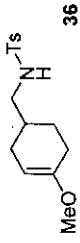
SI 32768
SF 75.4023741 MHz
NMW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
TX 20.00 cm
F1P 215.000 ppm
F1 16211.51 Hz
F2P -5.000 ppm
F2 -377.01 Hz
PPCM 11.00000 ppm/cm
HZ/CM 829.42615 Hz/cm



PL - II - p83 - P





Current Data Parameters
NAME TH-40c13
EXPNO 38201
PROCNO 1

F2 - Acquisition Parameters

Date 20090702
Time 17.26
INSTRUM spect
PROBHD 5 mm DNP 1H/1
PULPROG zgpp30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 18796.992 Hz
FIDRES 0.288819 Hz
AQ 1.7433076 sec
RG 1024
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec
D11 0.0300000 sec
D12 0.0000200 sec

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

NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SF01 75.4106357 MHz

===== CHANNEL f2 =====

CPDPG2 W11216
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SF02 299.8711995 MHz

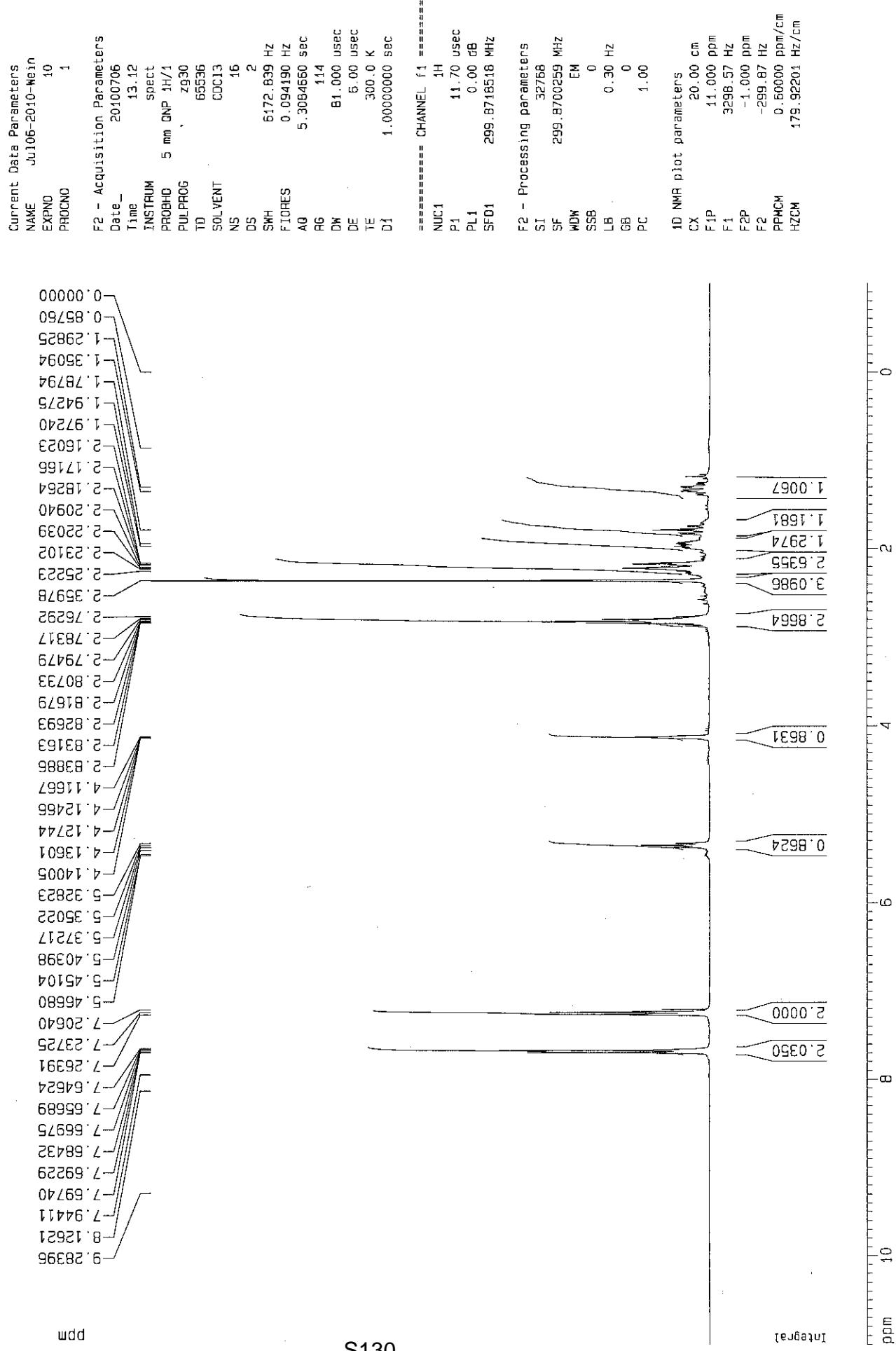
F2 - Processing parameters

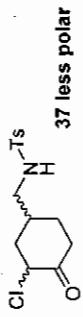
S1 32768
SF 75.4023410 MHz
NMW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR pilot parameters
CX 20.00 cm
F1P 161.310 ppm
F1 12163.12 Hz
F2P 0.565 ppm
F2 42.64 Hz
PPMCM 8.03720 ppm/cm
HZCM 606.02399 Hz/cm

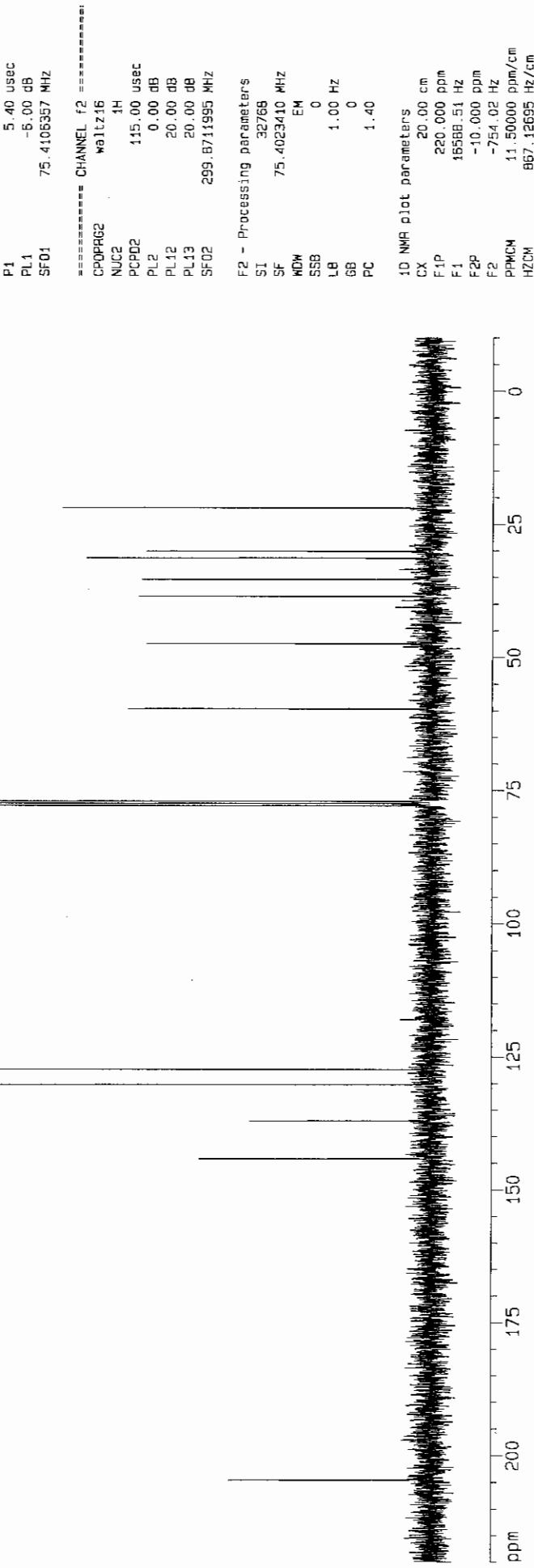
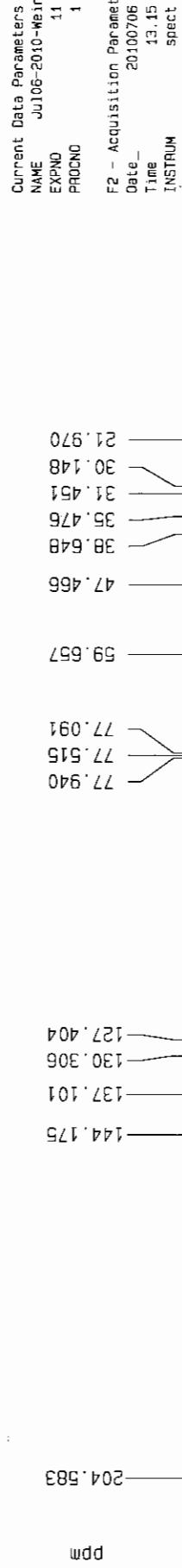


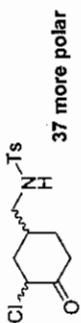
PL - II - p87 - P



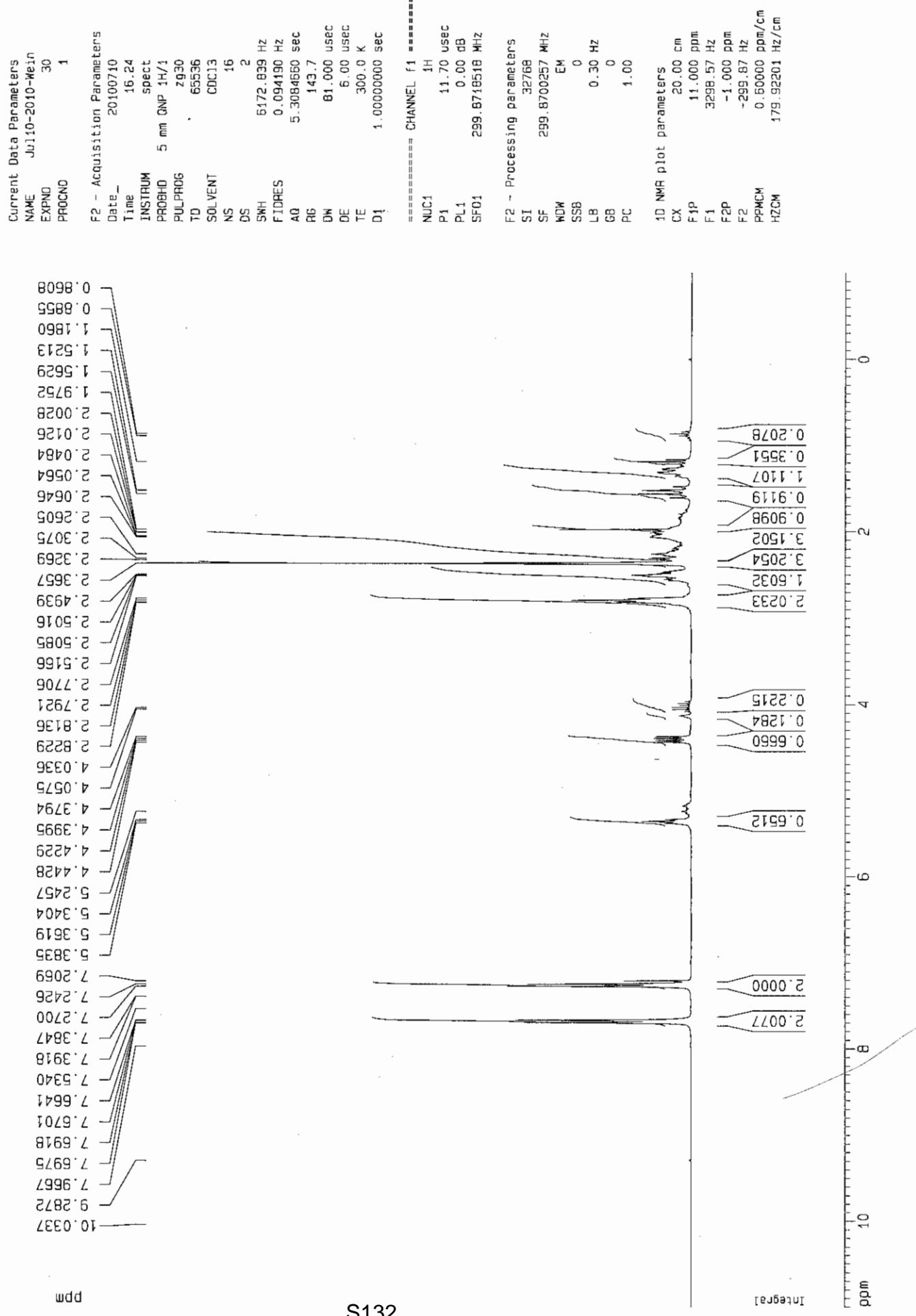


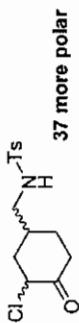
PL - II - p87 - P





PL - II - p88 - P(3)





PL - II - p88 - P(3)



ppm

S133

Current Data Parameters

NAME JU110-2010-Wein
EXPNO 34
PROCNO 1

F2 - Acquisition Parameters

Date 20100710
Time 16:41
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 15
DS 4
SWH 18796.992 Hz
FIDRES 0.28619 Hz
AQ 1.7433076 sec
RG 4096
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec
D11 0.0300000 sec
D12 0.0000200 sec

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

NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SF01 75.4103357 MHz

===== CHANNEL f2 =====

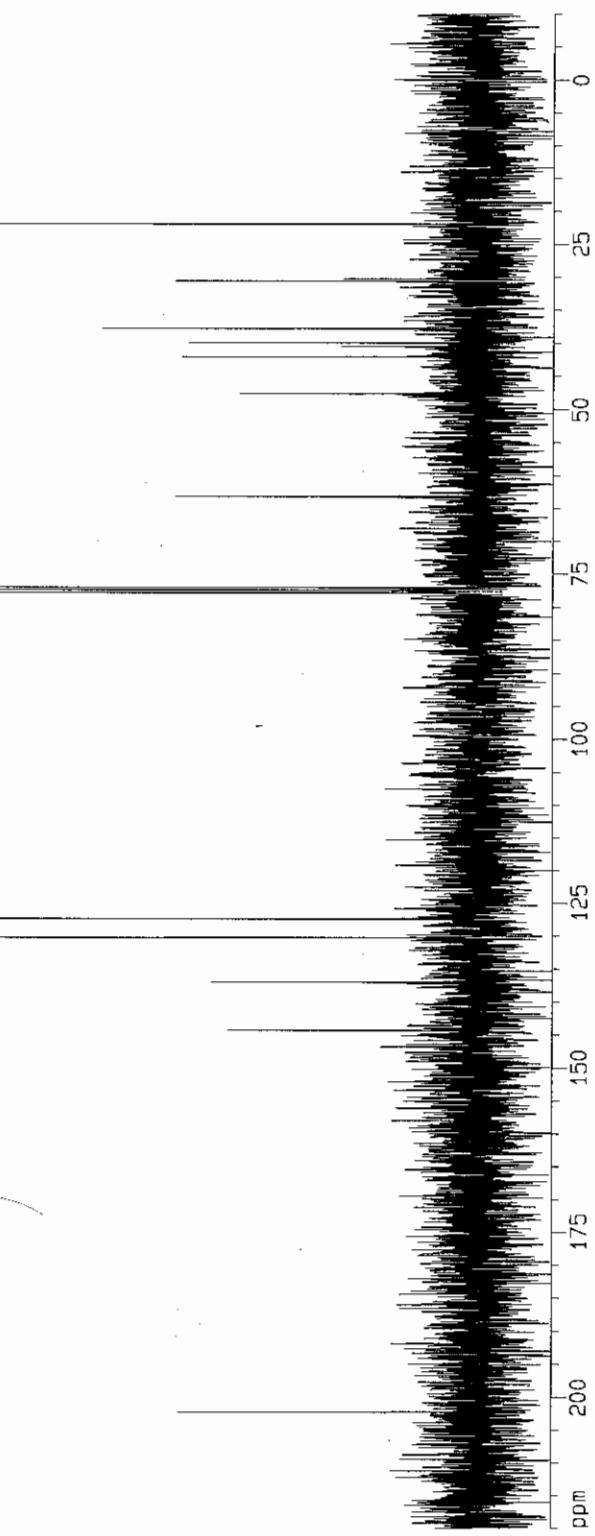
CPDPFG2 waltz15
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SF02 299.871995 MHz

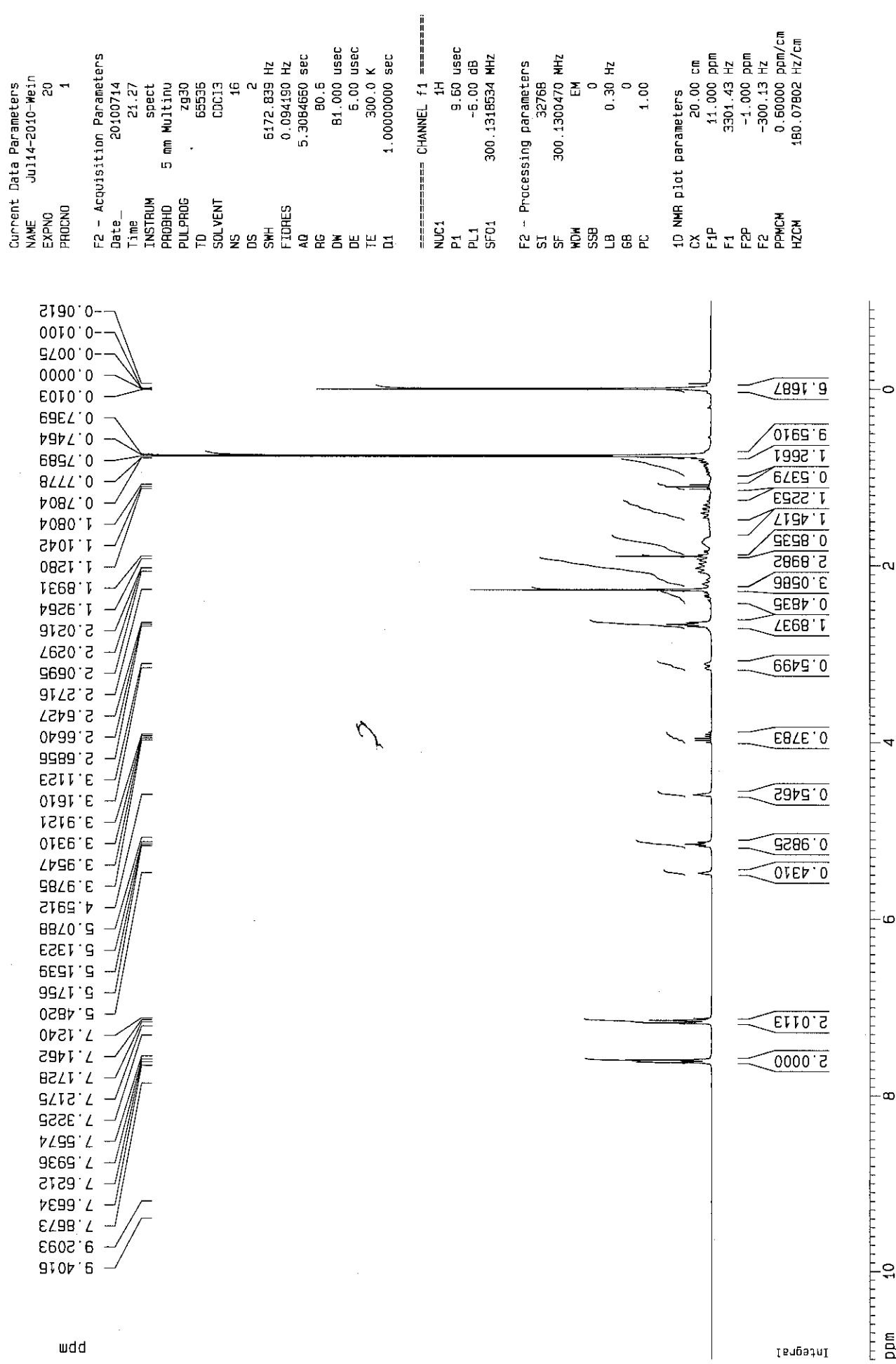
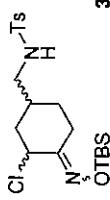
F2 - Processing parameters

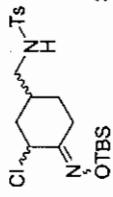
SI 32768
SF 75.4023410 MHz
WM EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR pilot parameters

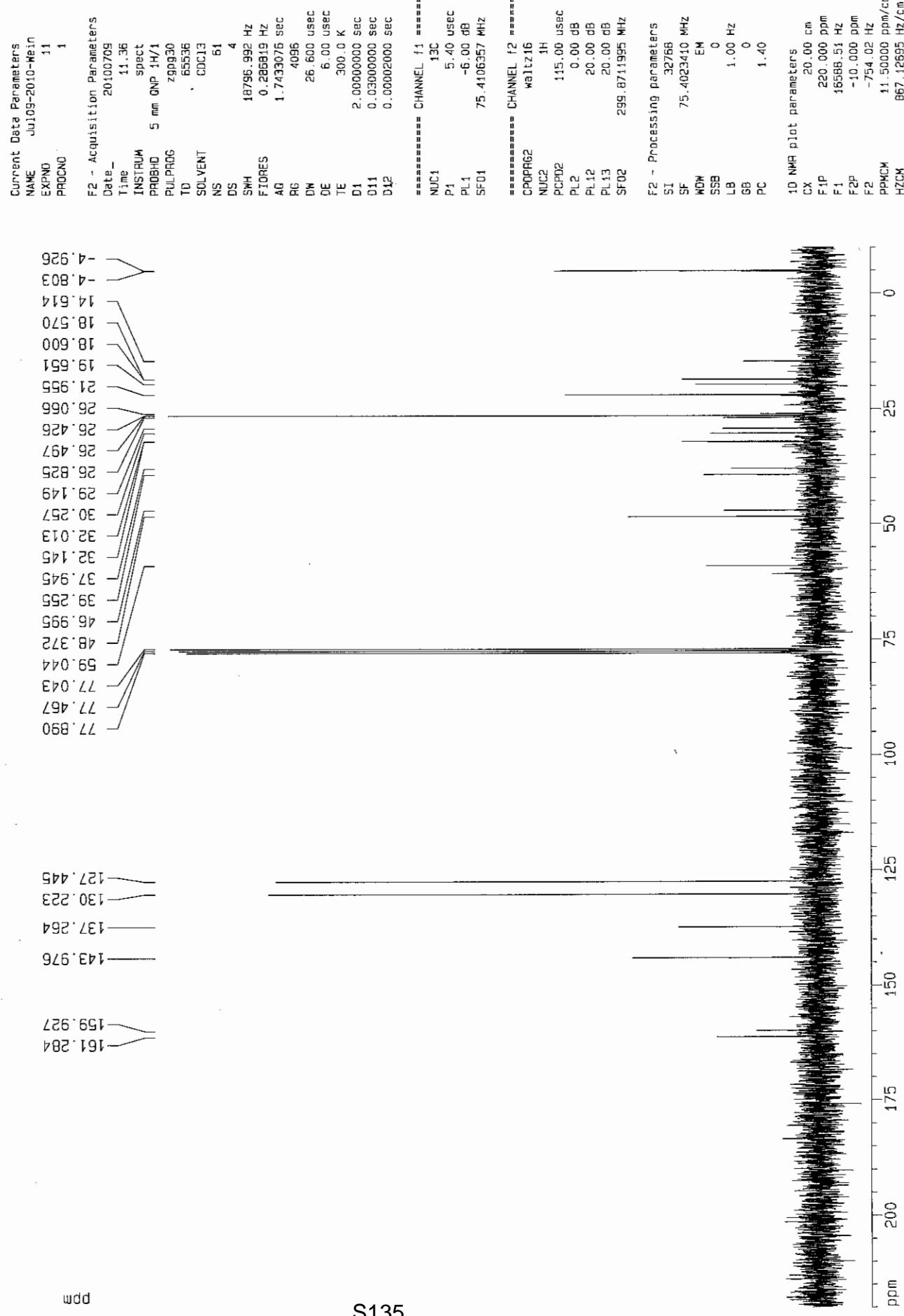
DX 20.00 cm
F1P 220.000 ppm
f1 165.08, 51 Hz
f2P -10.000 ppm
F2 -75.402 Hz
PPMCH 11.50000 ppm/cm
HZCM 867.12695 Hz/cm

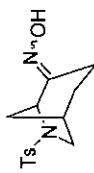






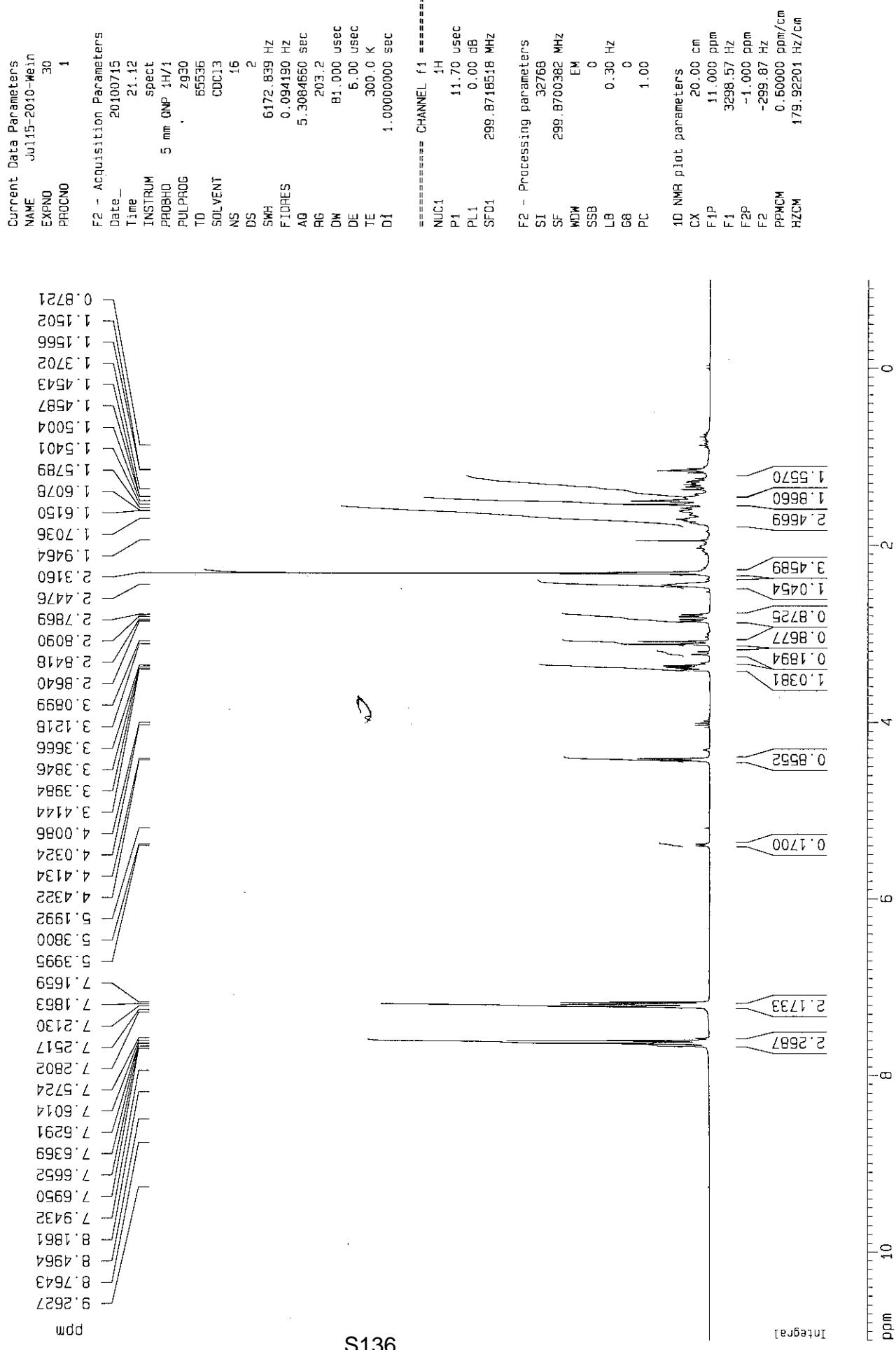
PL - II - p89 - P (2)

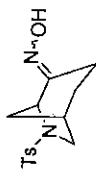




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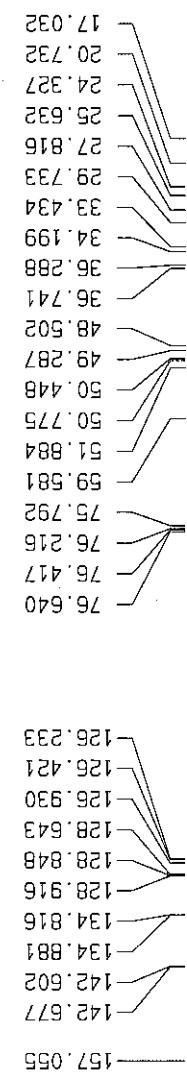
PL - II - p94 - P





39

PL - II - p94 - P



Current Data Parameters
NAME Jul15-2010-Mean
EXPNO 31
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100715
Time_ 22.08
INSTRUM spect
PROBID 5 mm QNP 1H/1
PULPROG zpg30
TD 65536
SOLVENT CDCl₃
NS 875
DS 4
SWH 18796.992 Hz
FIDRES 0.286819 Hz
AQ 1.7433076 sec
RG 2048
DM 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec
D11 0.0300000 sec
D12 0.00002000 sec

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

NUC1 13C
P1 5.40 uspc
PL1 -6.00 dB
SF01 75.4106357 MHz

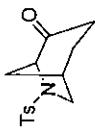
===== CHANNEL f2 =====

CPDPG2
NUC2 1H
POPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFQ2 299.8711995 MHz

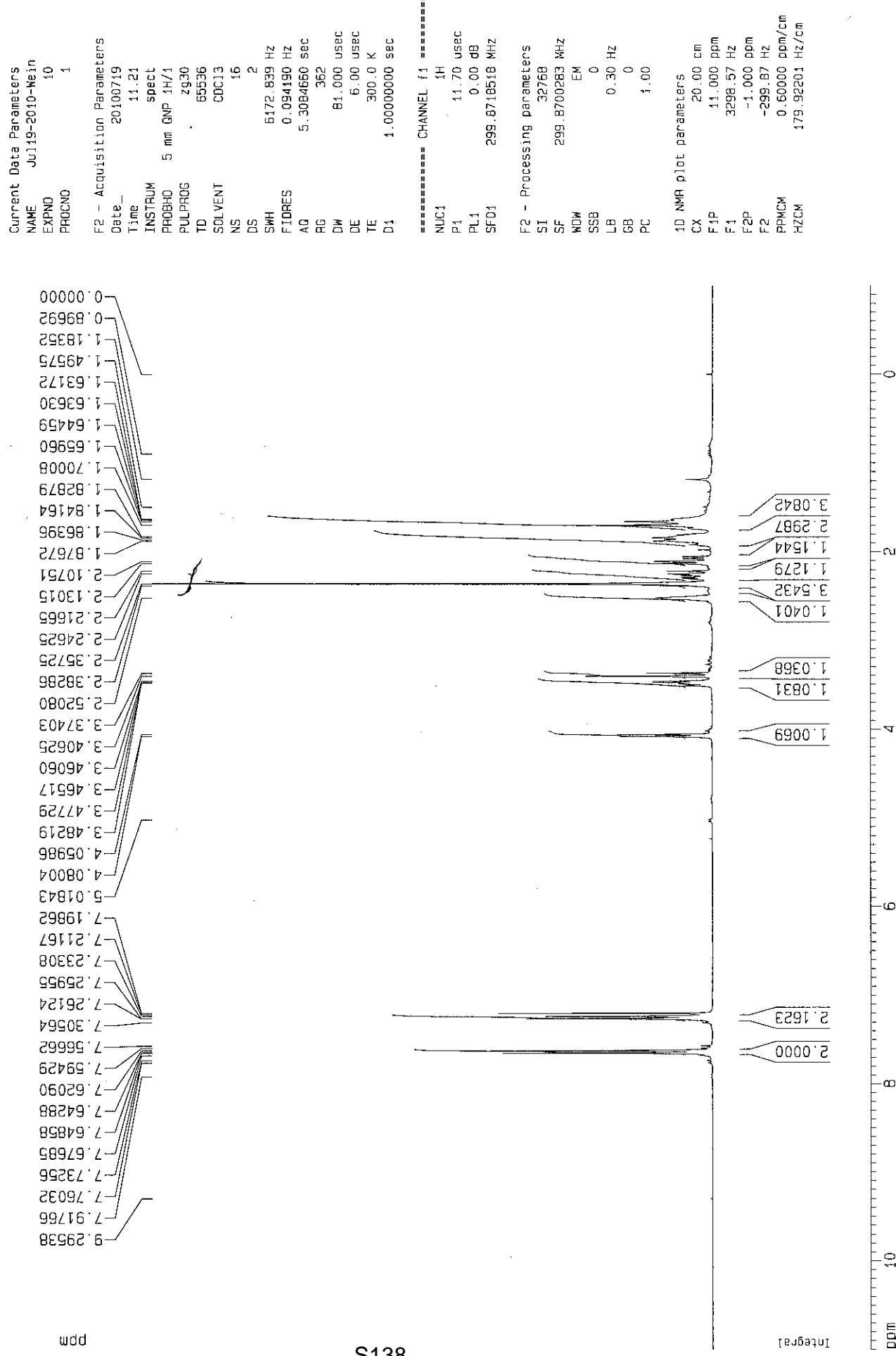
F2 - Processing parameters

CX 32768
SF 75.402348 MHz
NDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.00 ppm
F1 165.93 54 Hz
F2P -10.000 ppm
F2 -754.03 Hz
PPCM 11.50000 ppm/cm
HZCM 867.12799 Hz/cm



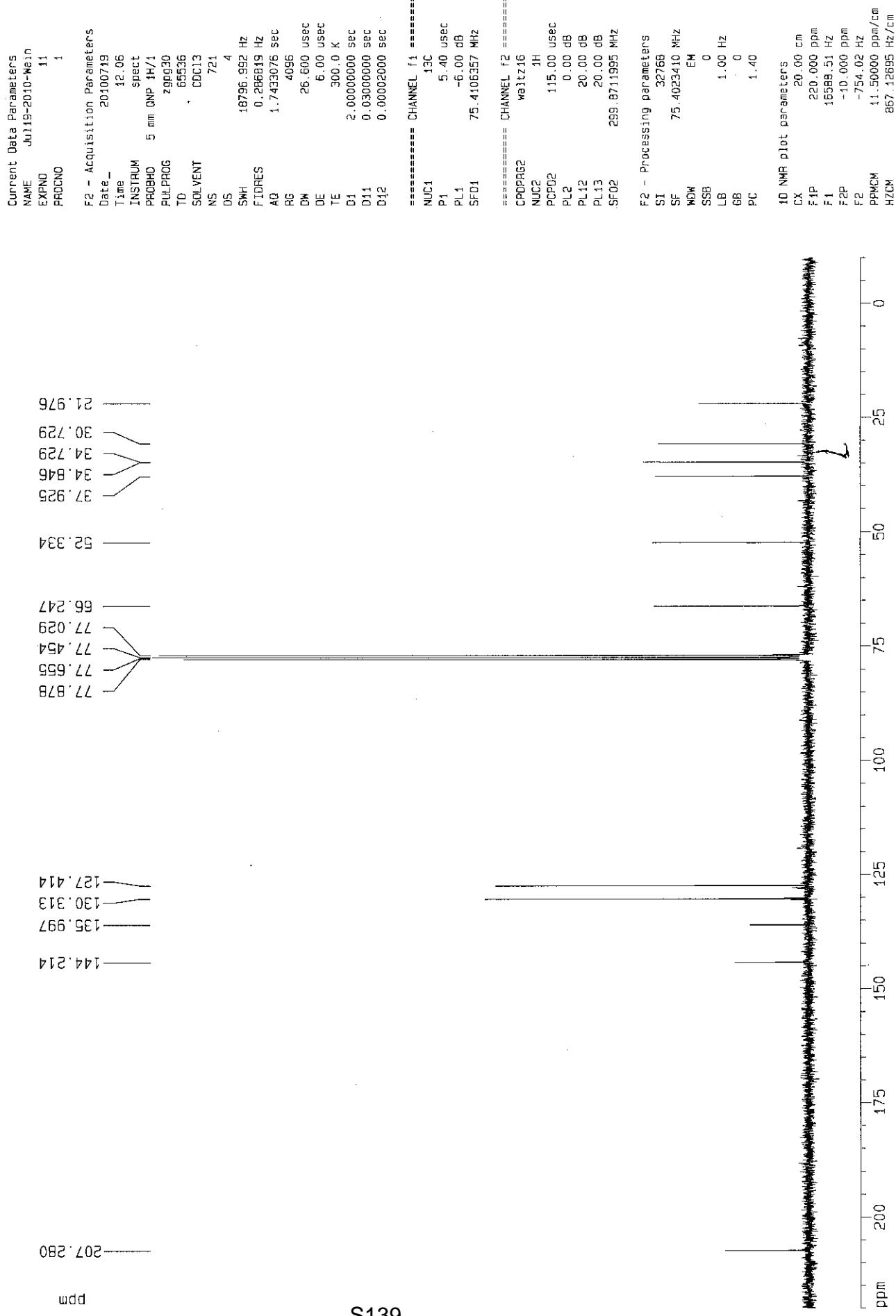
$$PL = II = 0.95 - P$$



PL - II - p95 - P



40



207.280

ppm

S139