

Supporting Information

A metal-free one-pot cascade synthesis of highly functionalized biaryl-2-carbaldehydes†

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General experimental methods:

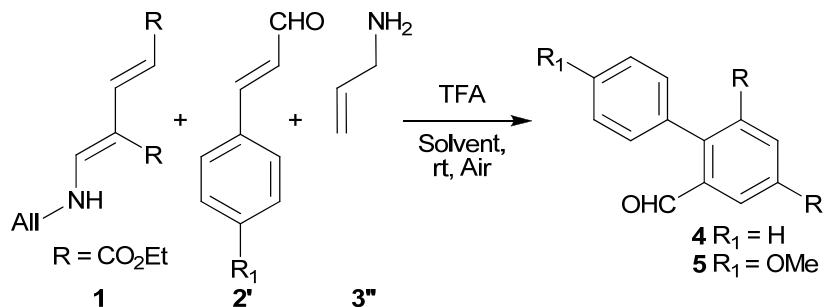
All the solvents were used without distillation and all biaryl syntheses were carried out at room temperature under inert-free aerobic atmosphere. Silica gel G-60 F₂₅₄ aluminum TLC plates were used to monitor the reactions with short wavelength ultraviolet light to visualize the spots. Flash column chromatography was performed on silica gel 230-400 mesh. ¹H and ¹³C NMR spectra were recorded at 500 and 125 MHz, respectively. Chemical shifts are given in ppm using solvent residual peak of chloroform δ 7.26 ppm as reference, and coupling constants in Hz. ESI- HRMS analysis was recorded using electrospray ionization with ions given in m/z.

General procedure for synthesis of cinnamaldehydes: Cinnamaldehydes were synthesized by following a known procedure employing Wittig reaction.¹ To a 50 mL round bottom flask equipped with a magnetic bar were added toluene (10 mL), pertinent aromatic aldehyde (1.5 mmol), (triphenylphosphoranylidene)acetaldehyde (Wittig reagent) (500 mg, 1 mmol) and the resulting mixture was stirred at 85 °C for overnight. After complete consumption of the Wittig reagent as indicated on TLC, the reaction mixture was concentrated and subjected to flash column chromatography. The product was eluted with DCM/hexane solvent system to afford the desired cinnamaldehyde.

Procedure for synthesis of biaryl 4: To a solution of dienaminodiester **1** (31 mg, 1 eq) in CHCl₃/MeCN (1:1) were added cinnamaldehyde (46.20 μL, 3 eq), allyl amine (27.5 μL, 3 eq) and TFA (28.0 μL, 3 eq) in a sequential manner at room temperature. After immediate addition of TFA, the reaction mixture appears intense red in color indicating the formation of trienamine. After complete consumption of compound **1** as visualized on TLC, the reaction mixture was quenched with saturated aqueous NaHCO₃ (10 mL) and extracted with DCM (1 x 10 mL). The organic layer was dried over anhydrous MgSO₄, concentrated and the crude mixture was subjected to flash column chromatography by eluting with DCM/hexane solvent system to afford

the desired biaryl **4** (24 mg, 60%). This general procedure was followed for the synthesis of the remaining biaryls.

Optimization of reaction conditions:



S.No	Ratio ^a TFA:2':3"	Solvent ^b	R ₁	Yield ^c
1	1:1:1	MeCN/DCM (1:2)	H	29%
2	3:2:2	MeOH	H	27%
3	3:2:2	THF	H	26%
4	3:2:2	toluene	H	32%
5	3:2:2	MeCN/toluene (1:1)	H	44%
6	3:3:3	DMF	H	25%
7	3:3:3	DMSO	OMe	trace
8	3:3:3	DME	OMe	nd
9	3:3:3	DMA	OMe	nd
10	3:3:3	MeCN/MeOH(1:1)	OMe	38%
11	2:1.5:2	MeCN/DCM (1:2)	H	31%
12	3:1.5:3	MeCN/DCM (1:2)	H	46%
13	3:1.5:3	MeCN/DCM (1:3)	H	43%
14	2:1.2:1.4	MeCN/DCM (1:1)	H	31%

^aequivalents of TFA, **2'** and **3''** respectively, ^bundistilled solvents, ^cisolated yields,

DMA = dimethylacetamide, DME = dimethoxyethane, nd = not detected

Diethyl-2-nitro-6-oxo-6H-benzo[c]chromene-8,10-dicarboxylate (8): To a solution of benzopyrone **7o** (14 mg, 1 eq) in DCM (1.5 mL) was added Dess-Martin periodinane (46 mg, 3 eq) at room temperature. After complete consumption of **7o** as indicated on TLC, the reaction mixture was quenched with saturated aqueous NaHCO₃ (6 mL) and extracted with DCM (10 mL × 2). The organic layer was dried over anhydrous Na₂SO₄, concentrated and purified by flash column chromatography (DCM/hexane 1:1) to afford the desired dibenzopyranone **8** (14 mg) in a quantitative yield: ¹H NMR (500 MHz, CDCl₃) δ 1.46 (m, 6H), 4.49 (q, 2H, *J* = 7.0), 4.63 (q, 2H, *J* = 7.0), 7.56 (d, 1H, *J* = 9.0), 8.42 (dd, 1H, *J* = 9.0, 2.0), 8.61 (s, 1H), 8.83 (d, 1H, *J* = 2.0), 9.17 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 13.9, 14.1, 62.3, 63.4, 116.6, 119.3, 119.8, 122.8, 123.2, 126.6, 131.3, 131.9, 133.8, 136.0, 143.9, 155.3, 158.5, 163.8, 168.2; ESI-HRMS [M+MeOH+Na]⁺ C₂₀H₁₉NO₉Na calcd for m/z 440.0957, found 440.0962.

Diethyl-4-(4,6-bis(ethoxycarbonyl)biphenyl-2-yl)-1-p-tolyl-1,4-dihydropyridine-3,5-dicarboxylate (9): To a solution of biaryl-2-carbaldehyde **4** (24 mg, 1 eq) in MeCN (1.5 mL) were added ethyl 3-(allylamino)acrylate (23 mg, 2 eq), *p*-toluidine (8 mg, 1 eq) and TFA (5.68 μL, 1 eq) in a sequential manner at room temperature. After complete consumption of biaryl **4** as indicated on TLC, the reaction mixture was quenched with saturated aqueous NaHCO₃ (5 mL) and extracted with EtOAc (10 mL × 2). The organic layer was dried over anhydrous Na₂SO₄, concentrated and purified by flash column chromatography (hexane/DCM 2:1) to produce the desired 1,4-DHP **9** (19 mg, 42%): ¹H NMR (500 MHz, CDCl₃) δ 0.89 (t, 3H, *J* = 7.0), 1.15 (t, 6H, *J* = 7.0), 1.39 (t, 3H, *J* = 7.0), 2.38 (s, 3H), 3.89 (q, 2H, *J* = 7.0), 4.05 (m, 4H), 4.39 (q, 2H, *J* = 7.0), 5.24 (s, 1H), 7.03 (d, 2H, *J* = 8.5), 7.22 (d, 2H, *J* = 8.5), 7.29 (s, 1H), 7.32 (t, 2H, *J* = 7.5), 7.36 (s, 2H), 7.45 (d, 2H, *J* = 7.5), 8.19 (s, 1H), 8.32 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 13.6, 14.2, 14.3, 20.8, 29.6, 60.1, 61.0, 61.1, 110.2, 120.7, 126.8, 127.2, 128.1, 129.0, 130.1,

130.3, 134.9, 136.2, 136.9, 138.2, 140.4, 144.7, 165.8, 166.6, 168.8; ESI-HRMS [M+Na]⁺ C₃₆H₃₇NO₈Na calcd for m/z 634.2416, found 634.2425.

Diethyl-2-(4,6-bis(ethoxycarbonyl)biphenyl-2-yl)-1-p-tolyl-1,2-dihdropyridine-3,5-dicarboxylate (10): To a solution of biaryl-2-carbaldehyde **4** (24.3 mg, 1.2 eq) in MeCN (2 mL) were added dienaminodiester **1** (15.7 mg, 1 eq), *p*-toluidine (8 mg, 1.2 eq) and TFA (5 μ L, 1 eq) in a sequential order at room temperature under aerobic atmosphere. After complete consumption of dienaminodiester **1** as observed on TLC, the reaction mixture was quenched with saturated aqueous NaHCO₃ (8 mL) and extracted with EtOAc (15 mL \times 2). The organic layer was dried over anhydrous Na₂SO₄, concentrated, and the crude mixture was subjected to flash column chromatography (hexane/DCM/EtOAc 4:2:0.2) to afford the desired 1,2-DHP **10** (19 mg, 50%): ¹H NMR (500 MHz, CDCl₃) δ 0.77 (t, 3H, *J* = 7.0), 1.31 (m, 6H), 1.39 (t, 3H, *J* = 7.0), 2.31 (s, 3H), 3.80 (m, 2H), 4.20 (m, 4H), 4.40 (q, 2H, *J* = 7.0), 5.76 (d, 1H, *J* = 7.5), 6.25 (s, 1H), 6.47 (d, 2H, *J* = 8.0), 6.80 (t, 1H, *J* = 7.5), 6.88 (d, 2H, *J* = 8.0), 7.20 (t, 1H, *J* = 7.5), 7.35 (t, 1H, *J* = 7.5), 7.47 (s, 1H), 7.80 (d, 1H, *J* = 7.5), 7.98 (s, 1H), 8.27 (d, 1H, *J* = 1.5), 8.61 (d, 1H, *J* = 1.5); ¹³C NMR (125 MHz, CDCl₃) δ 13.4, 13.6, 14.3, 14.5, 22.6, 58.2, 59.8, 60.3, 61.0, 61.1, 115.4, 125.1, 126.9, 127.1, 127.2, 128.9, 129.5, 130.0, 130.1, 130.2, 130.7, 133.7, 133.8, 136.9, 137.3, 140.1, 141.3, 143.3, 165.4, 165.5, 165.7, 168.4; ESI-HRMS [M+Na]⁺ C₃₆H₃₇NO₈Na calcd for m/z 634.2416, found 634.2421.

Diethyl-9-phenyl-9H-fluorene-2,4-dicarboxylate (11): To a solution of biaryl-2-carbaldehyde **4** (11 mg, 1 eq) in THF (1 mL) was added phenylmagnesium bromide (3M in ether, 56.2 μ L, 5 eq) at 0 °C under argon atmosphere. The reaction was allowed to attain room temperature slowly and after complete consumption of biaryl **4** as indicated on TLC, the reaction mixture was quenched with water (15 mL), 1M HCl (5 mL), and then extracted with EtOAc (10

mL × 2). The solvent was evaporated and the crude residue was directly treated with catalytic amount of *p*-TsOH in toluene (1.5 mL) under reflux conditions. After complete consumption of starting material as indicated on TLC, the reaction mixture was quenched with saturated aqueous NaHCO₃ (5 mL) and extracted with EtOAc (10 mL × 2). The organic layer was dried over anhydrous Na₂SO₄, concentrated and purified by flash column chromatography (hexane/DCM 4:1.5) to afford the desired fluorene **11** (13.6 mg) in a quantitative yield: ¹H NMR (500 MHz, CDCl₃) δ 1.37 (t, 3H, *J* = 7.0), 1.49 (t, 3H, *J* = 7.0), 4.30 (m, 2H), 4.55 (q, 2H, *J* = 7.0), 5.06 (s, 1H), 7.06 (d, 2H, *J* = 6.5), 7.25 (m, 3H), 7.31 (m, 2H), 7.40 (m, 1H), 8.04 (s, 1H), 8.42 (d, 1H, *J* = 7.5), 8.46 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 14.3, 54.1, 61.3, 61.6, 125.2, 125.5, 126.8, 127.2, 127.5, 128.4, 128.6, 128.9, 129.1, 130.7, 138.4, 140.3, 144.1, 149.8, 149.9, 165.9, 167.7; ESI-HRMS [M+1]⁺ C₂₅H₂₃O₄ calcd for m/z 387.1596, found 387.1587.

Diethyl-6-(di(1H-indol-3-yl)methyl)biphenyl-2,4-dicarboxylate (12): Catalytic amount of *p*-TsOH was added to a stirred solution of biaryl-2-carbaldehyde **4** (23.1 mg, 1 eq) and indole (16.5 mg, 2 eq) in ethanol (3 mL). The reaction mixture was kept at reflux temperature and stirred overnight. After complete consumption of biaryl **4** as indicated on TLC, the reaction mixture was quenched with saturated aqueous NaHCO₃ (2 x 5 mL) and extracted with EtOAc (10 mL × 2). The organic layer was dried over anhydrous Na₂SO₄, concentrated, and the crude mixture was subjected to flash column chromatography (hexane/DCM/EtOAc 4:2:1) to afford the desired BIM **12** (30.0 mg, 79%): ¹H NMR (500 MHz, CDCl₃) δ 0.91 (t, 3H, *J* = 7.0), 1.36 (t, 3H, *J* = 7.0), 4.00 (q, 2H, *J* = 7.0), 4.36 (q, 2H, *J* = 7.0), 5.68 (s, 1H), 6.35 (s, 2H), 6.93 (t, 2H, *J* = 7.0), 7.06 (d, 2H, *J* = 8.0), 7.13 (m, 4H), 7.24 (m, 5H), 8.00 (s, 2H), 8.16 (d, 1H, *J* = 1.5), 8.31 (d, 1H, *J* = 1.5); ¹³C NMR (125 MHz, CDCl₃) δ 13.6, 14.2, 36.3, 61.1, 61.5, 111.1, 118.9, 119.5,

121.7, 124.2, 126.5, 127.5, 127.8, 128.1, 128.5, 129.2, 132.6, 133.3, 136.7, 138.6, 144.0, 145.1, 166.5, 168.4; ESI-HRMS [M+Na]⁺ C₃₅H₃₀O₄N₂Na calcd for m/z 565.2103, found 565.2104.

Spectral data of Trienamine A & A', Biaryl B', 4-6 & 7a-7r

Diethyl 2-(4-nitrostyryl)-1-*p*-tolyl-1,2-dihydropyridine-3,5-dicarboxylate (Trienamine A):

¹H NMR (CDCl₃) δ 1.25 (t, 3H, *J* = 7.0), 1.37 (t, 3H, *J* = 7.0), 2.28 (s, 3H), 4.16 (m, 2H), 4.30 (q, 2H, *J* = 7.0), 5.11 (s, 1H), 6.55 (d, 1H, *J* = 13.5), 6.74 (d, 2H, *J* = 8.0), 7.08 (d, 2H, *J* = 8.0), 7.23 (d, 1H, *J* = 13.5), 7.57 (s, 1H), 7.61 (d, 2H, *J* = 8.5), 7.72 (s, 1H), 8.14 (d, 2H, *J* = 8.5); ¹³C NMR (CDCl₃) δ 13.8, 14.2, 14.3, 14.4, 20.6, 21.0, 29.6, 40.7, 60.5, 60.6, 61.6, 61.8, 112.9, 114.9, 115.7, 120.8, 123.3, 123.6, 124.1, 128.5, 129.8, 130.2, 130.3, 132.5, 132.9, 133.5, 135.7, 136.9, 139.5, 143.3, 147.0, 150.3, 155.0, 165.7, 166.4; ESI-HRMS [M+Na]⁺ C₂₆H₂₆N₂O₆Na calcd for m/z 485.1688, found 485.1698.

Dimethyl 6-(4-nitrophenyl)-5-((prop-2-ynylamino)methylene)cyclohexa-1,3-diene-1,3-dicarboxylate (Trienamine A'):

¹H NMR (CDCl₃) δ 2.35 (app t, 1H), 3.67 (s, 3H), 3.81 (s, 3H), 3.90 (m, 2H), 4.78 (m, 1H), 4.93 (s, 1H), 6.82 (d, 1H, *J* = 13.5), 7.50 (s, 1H), 7.52 (d, 2H, *J* = 9.0), 7.68 (s, 1H), 8.09 (d, 2H, *J* = 8.5); ¹³C NMR (CDCl₃) δ 37.5, 40.5, 51.6, 74.1, 77.9, 111.2, 113.2, 122.6, 123.9, 128.5, 132.8, 144.2, 146.9, 147.2, 150.3, 166.2, 166.9.

Dimethyl 6-formyl-4'-nitrobiphenyl-2,4-dicarboxylate (Biaryl B'):

¹H NMR (CDCl₃) δ 3.71 (s, 3H), 4.02 (s, 3H), 7.47 (d, 2H, *J* = 8.5), 8.34 (d, 2H, *J* = 8.5), 8.80 (d, 1H, *J* = 1.5), 8.84 (d, 1H, *J* = 1.5), 9.71 (s, 1H); ¹³C NMR (CDCl₃) δ 52.6, 52.9, 123.2, 123.3, 130.1, 131.2, 132.0, 132.2, 135.0, 135.8, 142.4, 146.7, 147.9, 164.8, 165.5, 189.1.

Diethyl 6-formyl-biphenyl-2,4-dicarboxylate (4): Yield: 24.0 mg (60%)

¹H NMR (500 MHz, CDCl₃) δ 0.98 (t, 3H, *J* = 7.0), 1.44 (t, 3H, *J* = 7.0), 4.07 (q, 2H, *J* = 7.0), 4.45 (q, 2H, *J* = 7.0), 7.28 (m, 2H), 7.46 (m, 3H), 8.68 (d, 1H, *J* = 2.0), 8.74 (d, 1H, *J* = 1.5), 9.77 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 13.6, 14.3, 61.5, 61.7, 128.1, 128.5, 129.3, 130.4, 130.9, 133.8, 134.9, 135.0, 135.2, 148.6, 164.8, 166.7, 190.8. ESI-HRMS [M+MeOH+Na]⁺ C₂₀H₂₂O₆Na calcd for m/z 381.1314, found 381.1318.

Diethyl 6-formyl-4'-methoxybiphenyl-2,4-dicarboxylate (5): Yield: 32.8 mg (55%)

¹H NMR (500 MHz, CDCl₃) δ 1.06 (t, 3H, *J* = 7.0), 1.43 (t, 3H, *J* = 7.0), 3.87 (s, 3H), 4.12 (q, 2H, *J* = 7.0), 4.44 (q, 2H, *J* = 7.0), 6.98 (d, 2H, *J* = 8.5), 7.19 (d, 2H, *J* = 9.0), 8.63 (d, 1H, *J* = 1.5), 8.71 (d, 1H, *J* = 2.0), 9.80 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 13.8, 14.3, 55.3, 61.5, 61.7, 113.6, 127.1, 130.1, 130.7, 130.8, 134.1, 134.7, 135.3, 148.3, 159.9, 164.8, 166.9, 191.0. ESI-HRMS [M+MeOH+Na]⁺ C₂₁H₂₄O₇Na calcd for m/z 411.1419, found 411.1422.

Diethyl 6-formyl-4'-nitrobiphenyl-2,4-dicarboxylate (6): Yield: 25.0 mg (50%)

¹H NMR (500 MHz, CDCl₃) δ 1.11 (t, 3H, *J* = 7.0), 1.46 (t, 3H, *J* = 7.0), 4.14 (q, 2H, *J* = 7.0), 4.47 (q, 2H, *J* = 7.0), 7.48 (d, 2H, *J* = 8.5), 8.34 (d, 2H, *J* = 8.5), 8.78 (s, 1H), 8.81 (s, 1H), 9.72 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 13.8, 14.2, 61.9, 62.0, 123.3, 130.2, 131.6, 132.0, 132.6, 134.9, 135.7, 142.6, 146.2, 147.8, 164.4, 165.3, 189.3. ESI-HRMS [M+MeOH+Na]⁺ C₂₀H₂₁NO₈Na calcd for m/z 426.1164, found 426.1171.

Diethyl 6-formyl-4'-methylbiphenyl-2,4-dicarboxylate (7a): Yield: 40.0 mg (60%)

¹H NMR (500 MHz, CDCl₃) δ 1.03 (t, 3H, *J* = 7.0), 1.43 (t, 3H, *J* = 7.0), 2.43 (s, 3H), 4.10 (q, 2H, *J* = 7.0), 4.44 (m, 2H), 7.16 (d, 2H, *J* = 8.0), 7.27 (bs, 2H), 8.65 (d, 1H, *J* = 2.0), 8.72 (d, 1H, *J* = 1.5), 9.78 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 13.6, 14.3, 21.2, 29.6, 61.5, 61.7, 128.8,

129.3, 130.2, 130.8, 132.0, 133.9, 134.7, 135.1, 138.5, 148.8, 164.8, 166.7, 190.9. ESI-HRMS $[M+MeOH+Na]^+$ C₂₁H₂₄O₆Na calcd for m/z 395.1470, found 395.1471.

Diethyl 6-formyl-4'-chlorobiphenyl-2,4-dicarboxylate (7b): Yield: 29 mg (63%)

¹H NMR (500 MHz, CDCl₃) δ 1.07 (t, 3H, *J* = 7.0), 1.44 (t, 3H, *J* = 7.0), 4.12 (q, 2H, *J* = 7.0), 4.46 (q, 2H, *J* = 7.0), 7.24 (d, 2H, *J* = 8.5), 7.46 (d, 2H, *J* = 8.5), 8.70 (d, 1H, *J* = 1.5), 8.74 (d, 1H, *J* = 2.0), 9.76 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 13.7, 14.3, 61.7, 61.8, 128.4, 130.6, 130.8, 131.2, 133.5, 133.7, 134.8, 135.1, 135.2, 147.3, 164.6, 166.2, 190.2. ESI-HRMS $[M+MeOH+Na]^+$ C₂₀H₂₁ClO₆Na calcd for m/z 415.0924, found 415.0929.

Diethyl 6-formyl-4'-bromobiphenyl-2,4-dicarboxylate (7c): Yield: 88.5 mg (84%)

¹H NMR (500 MHz, CDCl₃) δ 1.07 (t, 3H, *J* = 7.0), 1.44 (t, 3H, *J* = 7.0), 4.11 (q, 2H, *J* = 7.0), 4.46 (q, 2H, *J* = 7.0), 7.16 (d, 2H, *J* = 8.5), 7.60 (d, 2H, *J* = 8.5), 8.71 (s, 1H), 8.74 (d, 1H, *J* = 2.0), 9.76 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 13.7, 14.3, 61.7, 61.8, 123.0, 130.8, 130.9, 131.2, 131.3, 133.4, 134.2, 135.0, 135.2, 147.3, 164.6, 166.2, 190.2. ESI-HRMS $[M+MeOH+Na]^+$ C₂₀H₂₁BrO₆Na calcd for m/z 459.0419, found 459.0417.

Diethyl 6-formyl-4'-cyanobiphenyl-2,4-dicarboxylate (7d): Yield: 38.6 mg (86%)

¹H NMR (500 MHz, CDCl₃) δ 1.08 (t, 3H, *J* = 7.0), 1.45 (t, 3H, *J* = 7.0), 4.13 (q, 2H, *J* = 7.0), 4.48 (q, 2H, *J* = 7.0), 7.43 (d, 2H, *J* = 8.0), 7.78 (d, 2H, *J* = 8.0), 8.77 (d, 1H, *J* = 1.5), 8.79 (d, 1H, *J* = 2.0), 9.70 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 13.7, 14.2, 61.8, 62.0, 112.5, 118.2, 130.0, 131.4, 131.8, 132.7, 134.8, 135.6, 140.6, 146.5, 164.4, 165.5, 189.4. ESI-HRMS $[M+MeOH+Na]^+$ C₂₁H₂₁NO₆Na calcd for m/z 406.1266, found 406.1271.

Diethyl 4',6-diformylbiphenyl-2,4-dicarboxylate (7e): Yield: 20.6 mg (56%)

¹H NMR (500 MHz, CDCl₃) δ 1.05 (t, 3H, *J* = 7.0), 1.45 (t, 3H, *J* = 7.0), 4.11 (q, 2H, *J* = 7.0), 4.47 (q, 2H, *J* = 7.0), 7.48 (d, 2H, *J* = 8.0), 8.01 (d, 1H, *J* = 8.5), 8.78 (s, 2H), 9.73 (s, 1H), 10.12

(s, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 13.7, 14.3, 61.7, 61.9, 129.3, 130.0, 131.2, 131.5, 132.9, 134.9, 135.5, 136.1, 141.9, 147.3, 164.5, 165.8, 189.8, 191.4. ESI-HRMS $[\text{M}+2\text{MeOH}+\text{Na}]^+$ $\text{C}_{22}\text{H}_{26}\text{O}_8\text{Na}$ calcd for m/z 441.1525, found 441.1520.

Diethyl 6-formyl-2'-bromobiphenyl-2,4-dicarboxylate (7f): Yield: 47.4 mg (74%)

^1H NMR (500 MHz, CDCl_3) δ 1.05 (t, 3H, $J = 7.0$), 1.44 (t, 3H, $J = 7.0$), 4.11 (m, 2H), 4.47 (q, 2H, $J = 7.0$), 7.23 (dd, 1H, $J = 7.5, 1.0$), 7.34 (m, 1H), 7.42 (m, 1H), 7.69 (dd, 1H, $J = 8.0, 0.5$), 8.80 (d, 1H, $J = 1.5$), 8.86 (d, 1H, $J = 2.0$), 9.66 (s, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 13.6, 14.3, 61.5, 61.8, 123.3, 127.1, 130.0, 130.6, 131.1, 131.5, 132.4, 132.6, 134.9, 135.9, 136.8, 147.3, 164.6, 165.3, 190.0. ESI-HRMS $[\text{M}+\text{MeOH}+\text{Na}]^+$ $\text{C}_{20}\text{H}_{21}\text{BrO}_6\text{Na}$ calcd for m/z 459.0419, found 459.0426.

Diethyl 6-formyl-2'-ethynylbiphenyl-2,4-dicarboxylate (7g): Yield: 30.7 mg (69%)

^1H NMR (500 MHz, CDCl_3) δ 1.04 (t, 3H, $J = 7.0$), 1.45 (t, 3H, $J = 7.0$), 2.94 (s, 1H), 4.11 (q, 2H, $J = 7.0$), 4.46 (q, 2H, $J = 7.0$), 7.25 (m, 1H), 7.46 (m, 2H), 7.64 (m, 2H), 8.80 (s, 1H), 8.83 (s, 1H), 9.70 (s, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 13.6, 14.3, 61.4, 61.7, 81.3, 82.1, 122.1, 128.4, 128.5, 129.4, 130.8, 131.2, 132.5, 133.1, 135.2, 135.6, 138.7, 147.4, 164.8, 165.8, 190.2. ESI-HRMS $[\text{M}+\text{MeOH}+\text{Na}]^+$ $\text{C}_{22}\text{H}_{22}\text{O}_6\text{Na}$ calcd for m/z 405.1314, found 405.1310.

Diethyl 6-formyl-3',5'-dimethoxybiphenyl-2,4-dicarboxylate (7h): Yield: 50.0 mg (67%)

^1H NMR (500 MHz, CDCl_3) δ 1.06 (t, 3H, $J = 7.0$), 1.43 (t, 3H, $J = 7.0$), 3.80 (s, 6H), 4.13 (q, 2H, $J = 7.0$), 4.44 (q, 2H, $J = 7.0$), 6.42 (d, 2H, $J = 2.0$), 6.54 (d, 1H, $J = 2.0$), 8.64 (d, 1H, $J = 2.0$), 8.72 (d, 1H, $J = 1.5$), 9.81 (s, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 13.7, 14.3, 55.4, 61.5, 61.7, 100.3, 105.5, 107.9, 130.4, 130.6, 133.6, 134.7, 134.9, 137.0, 148.3, 160.5, 164.7, 166.6, 190.8. ESI-HRMS $[\text{M}+\text{MeOH}+\text{Na}]^+$ $\text{C}_{22}\text{H}_{26}\text{O}_8\text{Na}$ calcd for m/z 441.1525, found 441.1520.

Diethyl 6-formyl-5'-bromo-2'-methoxybiphenyl-2,4-dicarboxylate (7i): Yield: 60.2 mg (85%)

¹H NMR (500 MHz, CDCl₃) δ 1.09 (t, 3H, *J* = 7.0), 1.43 (t, 3H, *J* = 7.0), 3.71 (s, 3H), 4.15 (m, 2H), 4.46 (q, 2H, *J* = 7.0), 6.86 (d, 1H, *J* = 9.0), 7.24 (d, 1H, *J* = 2.0), 7.55 (dd, 1H, *J* = 2.0, 9.0), 8.75 (s, 2H), 9.73 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 13.8, 14.3, 55.8, 61.5, 61.7, 112.1, 112.8, 126.3, 130.8, 131.0, 132.9, 133.0, 133.5, 135.0, 135.5, 143.8, 155.7, 164.7, 166.0, 190.5. ESI-HRMS [M+MeOH+Na]⁺ C₂₁H₂₃BrO₇Na calcd for m/z 489.0524, found 489.0535.

Diethyl 6-formyl-4'-chloro-2'-fluorobiphenyl-2,4-dicarboxylate (7j): Yield: 21.2 mg (68%)

¹H NMR (500 MHz, CDCl₃) δ 1.15 (t, 3H, *J* = 7.0), 1.45 (t, 3H, *J* = 7.0), 4.18 (q, 2H, *J* = 7.0), 4.46 (q, 2H, *J* = 7.0), 7.15 (t, 1H, *J* = 7.0), 7.26 (m, 2H), 8.78 (d, 1H, *J* = 2.0), 8.83 (d, 1H, *J* = 2.0), 9.79 (d, 1H, *J* = 1.0); ¹³C NMR (125 MHz, CDCl₃) δ 13.7, 14.3, 61.8, 61.9, 116.2, 116.4, 124.5, 131.5, 131.7, 131.9, 133.2, 135.3, 135.8, 141.2, 160.3, 164.5, 165.4, 189.3. ESI-HRMS [M+MeOH+Na]⁺ C₂₀H₂₀ClFO₆Na calcd for m/z 433.0830, found 433.0831.

Diethyl 6-formyl-2',6'-difluorobiphenyl-2,4-dicarboxylate (7k): Yield: 53 mg (68%)

¹H NMR (500 MHz, CDCl₃) δ 1.15 (t, 3H, *J* = 7.0), 1.45 (t, 3H, *J* = 7.0), 4.21 (q, 2H, *J* = 7.0), 4.47 (q, 2H, *J* = 7.0), 7.04 (m, 2H), 7.47 (m, 1H), 8.83 (d, 1H, *J* = 2.0), 8.93 (d, 1H, *J* = 2.0), 9.83 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 13.7, 14.3, 30.9, 61.7, 61.9, 111.1-111.3, 112.6, 130.9, 131.0, 131.1, 131.8, 132.2, 133.1, 135.4, 136.0, 136.2, 158.7, 160.7, 164.5, 165.0, 189.5. ESI-HRMS [M+MeOH+Na]⁺ C₂₀H₂₀F₂O₆Na calcd for m/z 417.1125, found 417.1114.

Diethyl 6-formyl-3'-bromo-4'-hydroxy-5'-methoxybiphenyl-2,4-dicarboxylate (7l): Yield: 19 mg (38%)

¹H NMR (500 MHz, CDCl₃) δ 1.11 (t, 3H, *J* = 7.0), 1.44 (t, 3H, *J* = 7.0), 3.89 (s, 3H), 4.16 (q, 2H, *J* = 7.0), 4.46 (q, 2H, *J* = 7.0), 6.12 (bs, 1H), 6.73 (s, 1H), 7.04 (s, 1H), 7.26 (s, 1H), 8.64 (s, 1H), 8.71 (s, 1H), 9.83 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 13.8, 14.2, 56.5, 61.7, 108.2,

111.3, 125.6, 127.5, 130.6, 130.9, 134.0, 134.8, 135.2, 143.5, 146.7, 164.6, 166.4, 190.5. ESI-HRMS [M+MeOH+Na]⁺ C₂₁H₂₃BrO₈Na calcd for m/z 505.0474, found 505.0470.

Diethyl 4-(5-bromothiophene-2-yl)-5-formylisophthalate (7m): Yield: 54 mg (49%)

¹H NMR (500 MHz, CDCl₃) δ 1.19 (t, 3H, *J* = 7.0), 1.43 (t, 3H, *J* = 7.0), 4.23 (q, 2H, *J* = 7.0), 4.46 (q, 2H, *J* = 7.0), 6.83 (d, 1H, *J* = 3.5), 7.11 (d, 1H, *J* = 3.0), 8.65 (s, 1H), 8.71 (s, 1H), 9.98 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 13.8, 14.2, 61.9, 114.7, 130.0, 130.2, 130.9, 131.6, 134.8, 134.9, 136.3, 139.4, 164.4, 166.0, 189.8. ESI-HRMS [M+MeOH+Na]⁺ C₁₈H₁₉BrO₆SNa calcd for m/z 464.9983, found 464.9978.

Diethyl 4-(5-iodofuran-2-yl)-5-formylisophthalate (7n): Yield: 38.0 mg (55%)

¹H NMR (500 MHz, CDCl₃) δ 1.26 (t, 3H, *J* = 7.0), 1.43 (t, 3H, *J* = 7.0), 4.28 (q, 2H, *J* = 7.0), 4.44 (q, 2H, *J* = 7.0), 6.52 (d, 1H, *J* = 3.5), 6.76 (d, 1H, *J* = 3.0), 8.63 (d, 1H, *J* = 1.5), 8.71 (d, 1H, *J* = 1.5), 10.06 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 14.1, 14.2, 24.6, 36.6, 61.9, 62.1, 90.5, 116.9, 122.2, 131.2, 131.4, 134.0, 134.6, 134.9, 135.0, 151.4, 162.4, 166.5, 189.9. ESI-HRMS [M+MeOH+Na]⁺ C₁₈H₁₉IO₇Na calcd for m/z 497.0073, found 497.0072.

Diethyl 6-hydroxy-2-nitro-6H-benzo[c]chromene-8,10-dicarboxylate (7o): Yield: 36.6 mg (68%)

¹H NMR (500 MHz, CDCl₃) δ 1.36 (t, 3H, *J* = 7.0), 1.43 (t, 3H, *J* = 7.0), 3.85 (d, 1H, *J* = 5.0), 4.44 (m, 3H), 4.55 (m, 1H), 6.52 (d, 1H, *J* = 4.5), 7.28 (s, 1H), 8.20 (d, 1H, *J* = 1.0), 8.25 (dd, 1H, *J* = 9.0, 2.5), 8.34 (d, 1H, *J* = 1.5), 8.52 (d, 1H, *J* = 2.5); ¹³C NMR (125 MHz, CDCl₃) δ 13.9, 14.2, 61.8, 62.7, 93.1, 119.8, 119.9, 120.7, 123.2, 126.0, 128.6, 129.2, 130.4, 130.6, 131.5, 133.5, 142.4, 156.8, 164.7, 168.7. ESI-HRMS [M+Na]⁺ C₁₉H₁₇NO₈Na calcd for m/z 410.0851, found 410.0848.

Diethyl 4-bromo-6-hydroxy-2-methoxy-6H-benzo[c]chromene-8,10-dicarboxylate (7p):

Yield: 18.4 mg (77%)

¹H NMR (500 MHz, CDCl₃) δ 1.34 (t, 3H, *J* = 7.0), 1.41 (t, 3H, *J* = 7.0), 3.49 (bs, 1H), 3.93 (s, 3H), 4.41 (m, 4H), 6.48 (s, 1H), 7.08 (s, 1H), 7.26 (s, 1H), 8.17 (d, 1H, *J* = 1.5), 8.28 (d, 1H, *J* = 1.5); ¹³C NMR (125 MHz, CDCl₃) δ 13.9, 14.2, 30.9, 56.4, 61.6, 62.3, 92.8, 114.0, 116.1, 121.6, 121.9, 129.1, 129.9, 130.3, 131.2, 133.9, 140.2, 150.7, 164.9, 169.2. ESI-HRMS [M+Na]⁺ C₂₀H₁₉BrO₇Na calcd for m/z 473.0211, found 473.0206.

Diethyl 2,6-dimethoxy-6H-benzo[c]chromene-8,10-dicarboxylate (7q): Yield: 20 mg (60%)

¹H NMR (500 MHz, CDCl₃) δ 1.32 (t, 3H, *J* = 7.0), 1.42 (t, 3H, *J* = 7.0), 3.52 (s, 3H), 3.78 (s, 3H), 4.40 (m, 4H), 5.88 (s, 1H), 6.94 (dd, 1H, *J* = 9.0, 3.5), 7.07 (d, 1H, *J* = 3.0), 7.12 (d, 1H, *J* = 9.0), 8.10 (d, 1H, *J* = 1.5), 8.25 (d, 1H, *J* = 1.5); ¹³C NMR (125 MHz, CDCl₃) δ 13.9, 14.3, 30.9, 55.7, 61.4, 62.0, 98.7, 111.9, 117.2, 119.3, 120.6, 129.2, 129.3, 130.0, 131.1, 131.5, 133.4, 145.3, 154.5, 165.1, 169.5. ESI-HRMS [M+Na]⁺ C₂₁H₂₂O₇Na calcd for m/z 409.1263, found 409.1260.

Diethyl 6-butoxy-2-methoxy-6H-benzo[c]chromene-8,10-dicarboxylate (7r): Yield: 15.2 mg (66%)

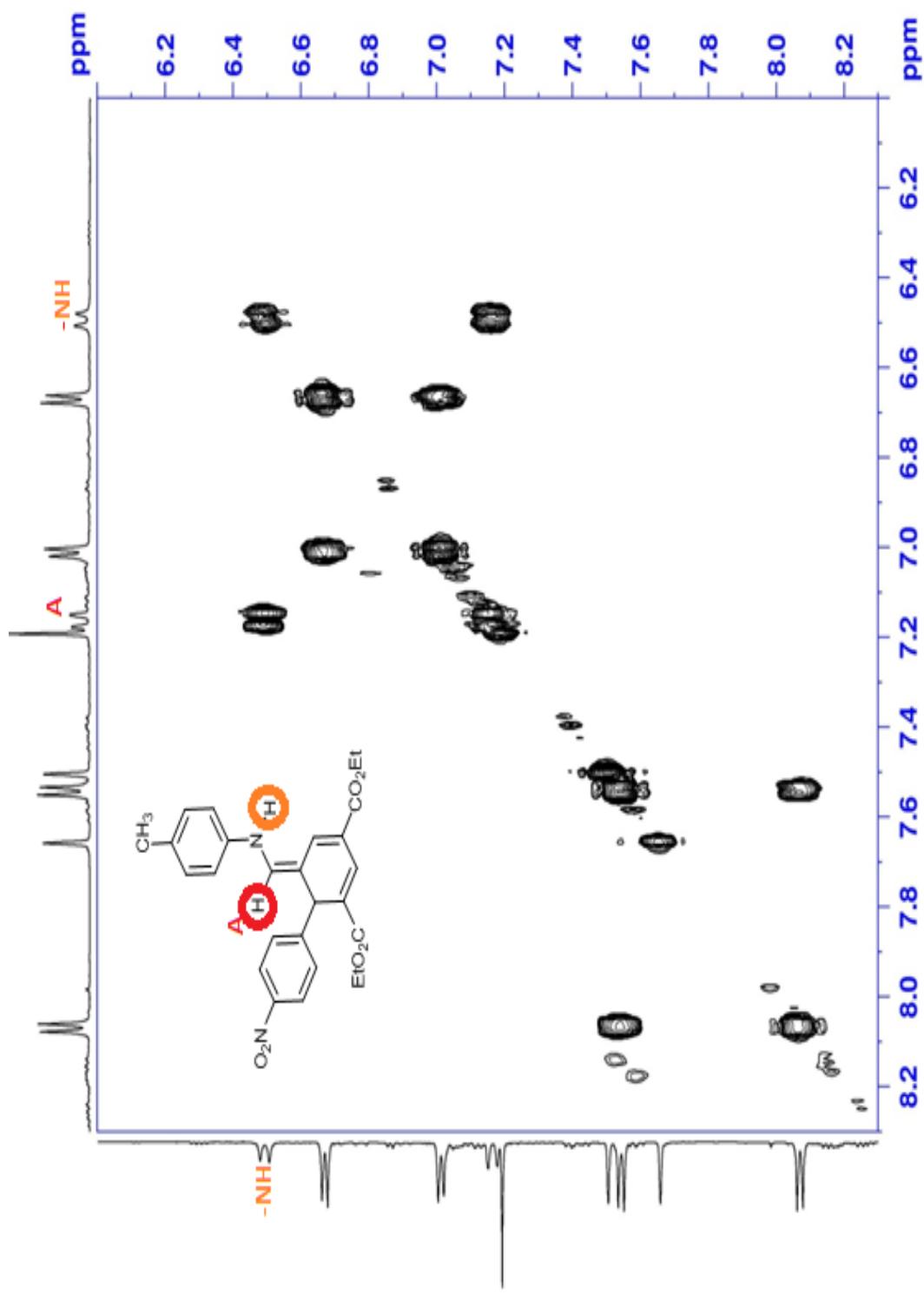
¹H NMR (500 MHz, CDCl₃) δ 0.84 (t, 3H, *J* = 7.0), 1.25 (m, 3H), 1.32 (t, 3H, *J* = 7.0), 1.42 (t, 3H, *J* = 7.0), 1.51 (m, 2H), 3.70 (m, 1H), 3.78 (s, 3H), 3.84 (m, 1H), 4.42 (m, 4H), 5.96 (s, 1H), 6.92 (dd, 1H, *J* = 9.0, 3.0), 7.06 (m, 2H), 8.08 (d, 1H, *J* = 1.5), 8.23 (d, 1H, *J* = 1.5); ¹³C NMR (125 MHz, CDCl₃) δ 13.9, 14.3, 19.1, 22.6, 29.6, 31.3, 55.7, 61.4, 61.9, 68.5, 97.7, 111.8, 117.1, 119.3, 120.6, 129.1, 129.2, 130.0, 130.9, 131.6, 133.7, 145.6, 154.3, 165.2, 169.6. ESI-HRMS [M+Na]⁺ C₂₄H₂₈O₇Na calcd for m/z 451.1732, found 451.1732.

References:

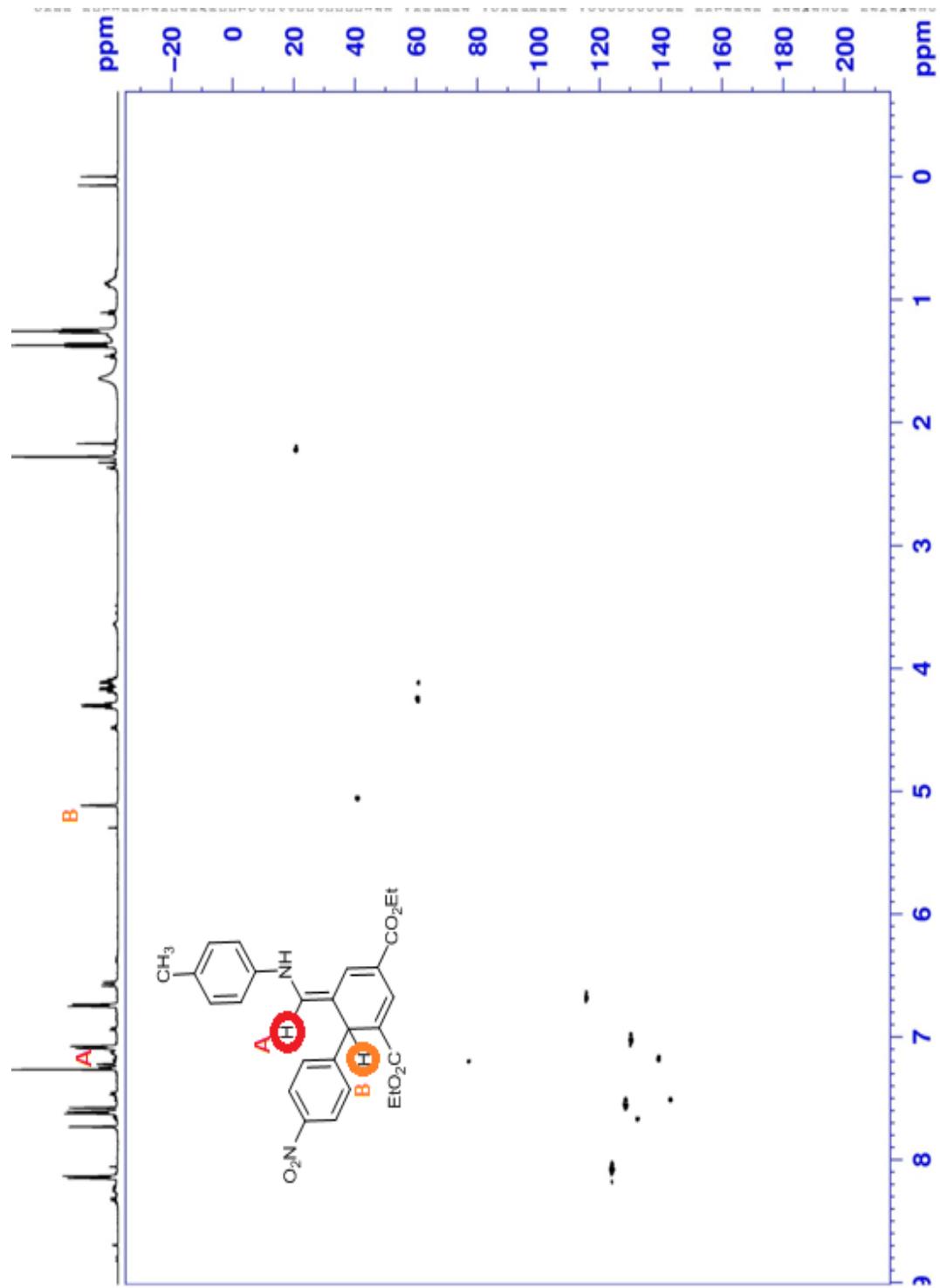
1. J. Kulhnek, S. Bures, O. Pytela, T. Mikysek and J. Ludvk, *Chem. Asian J.* 2011, **6**, 1604–1612.

Structural evidence for Trienamine A and A' by 2D NMR experiments:

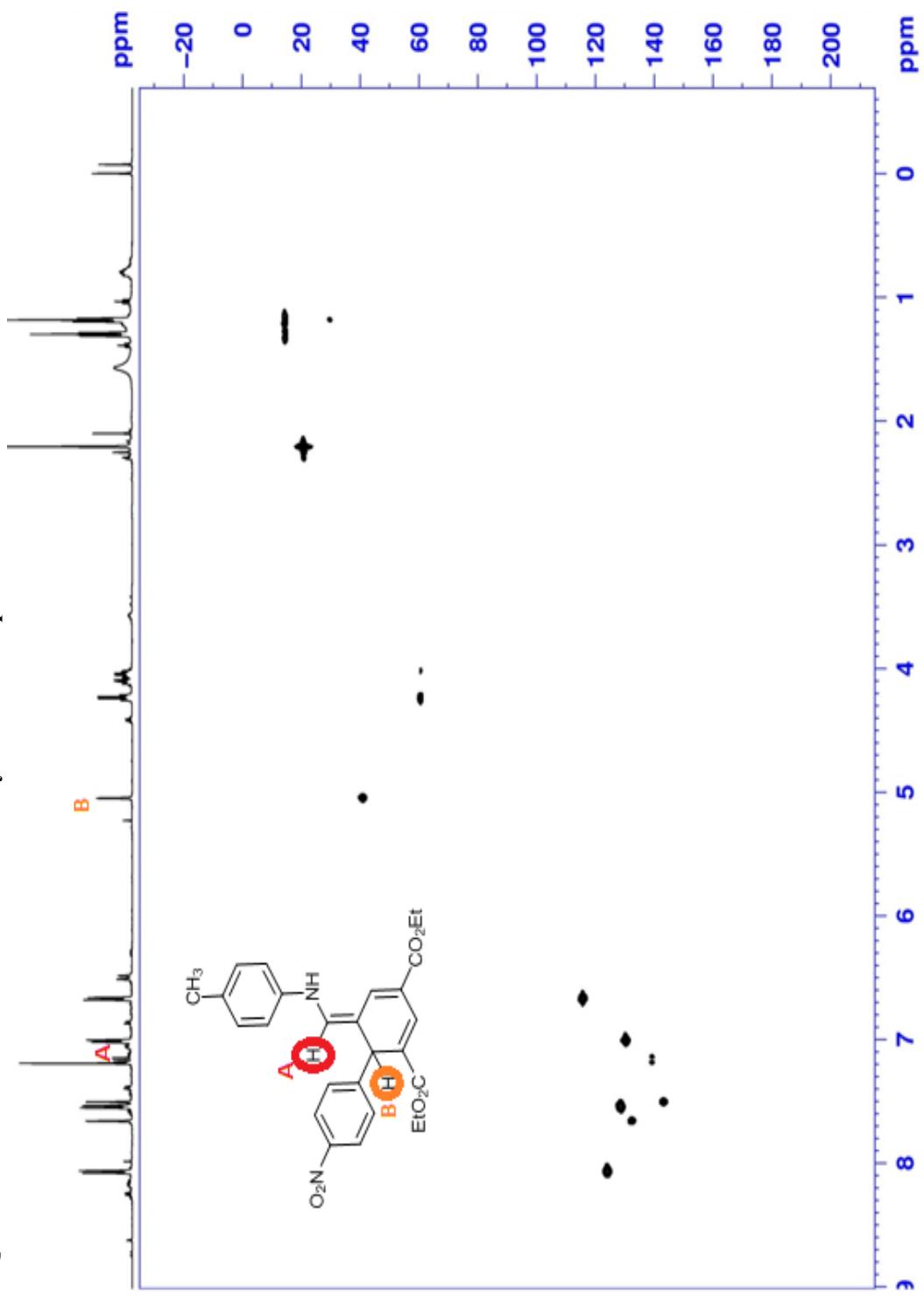
(A) COSY correlation for Trienamine A:



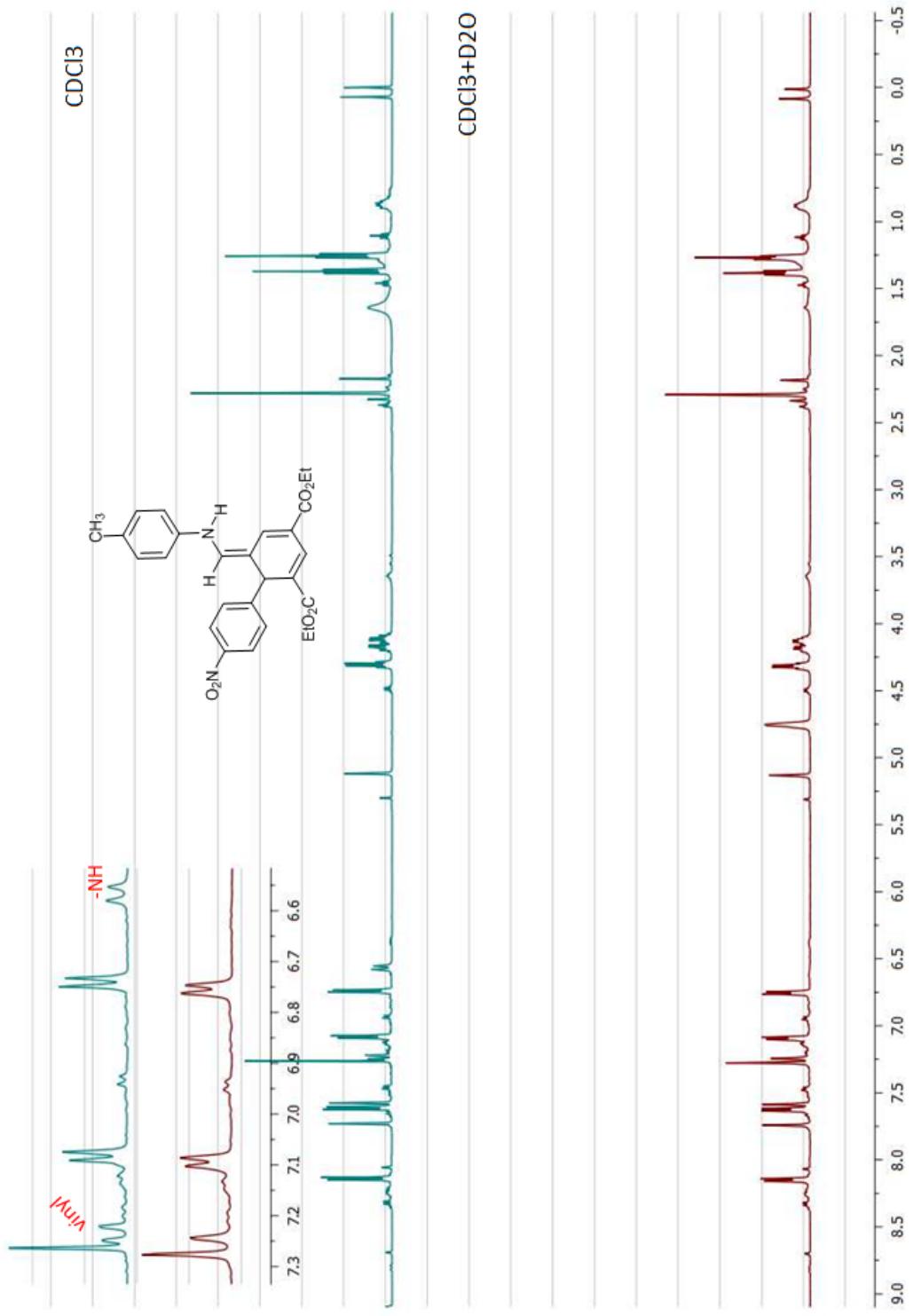
(B) HSQC correlation for Trienamine A with only one olefinic proton



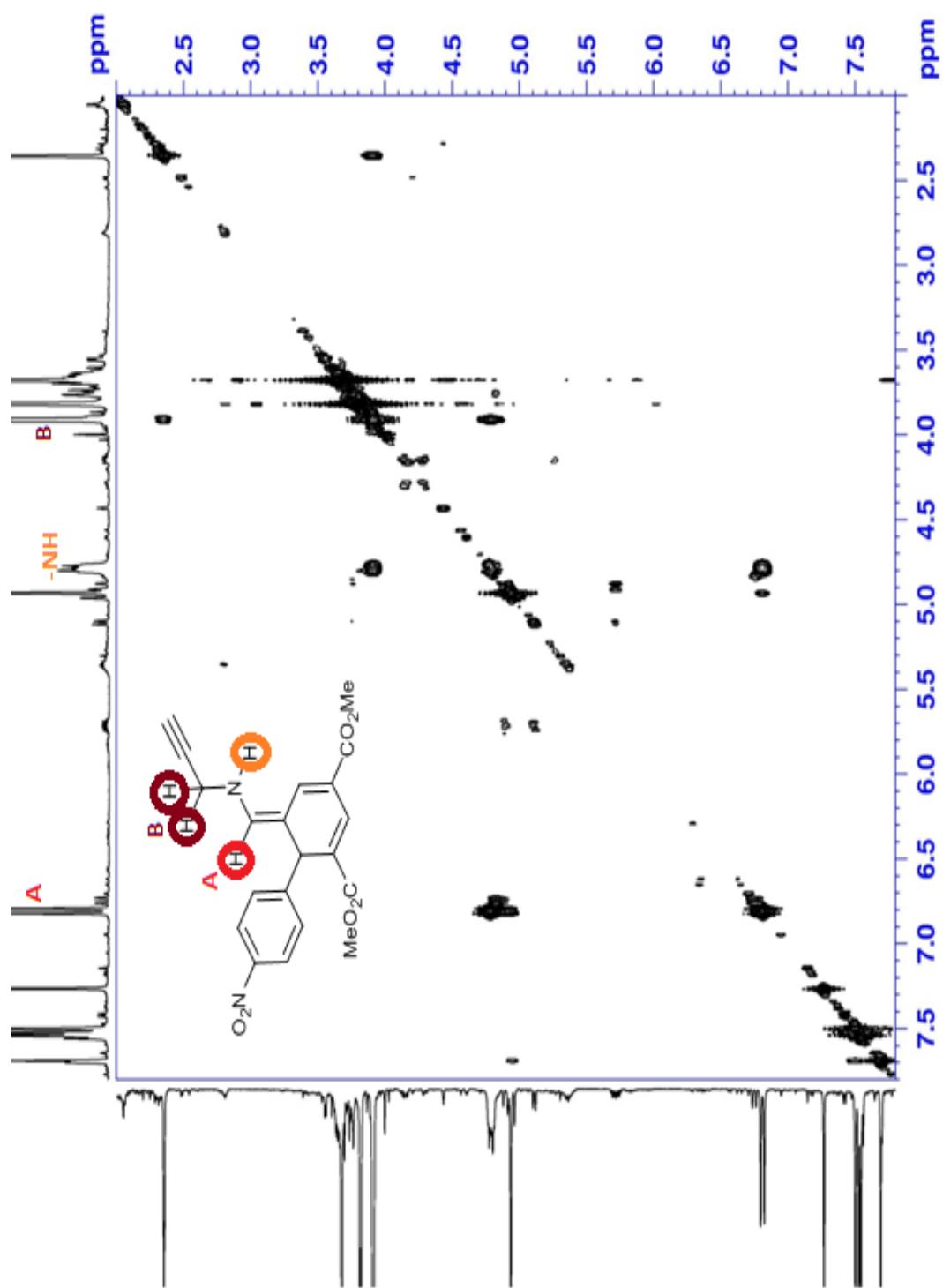
(C) HMQC correlation for Trienamine A with only one olefinic proton



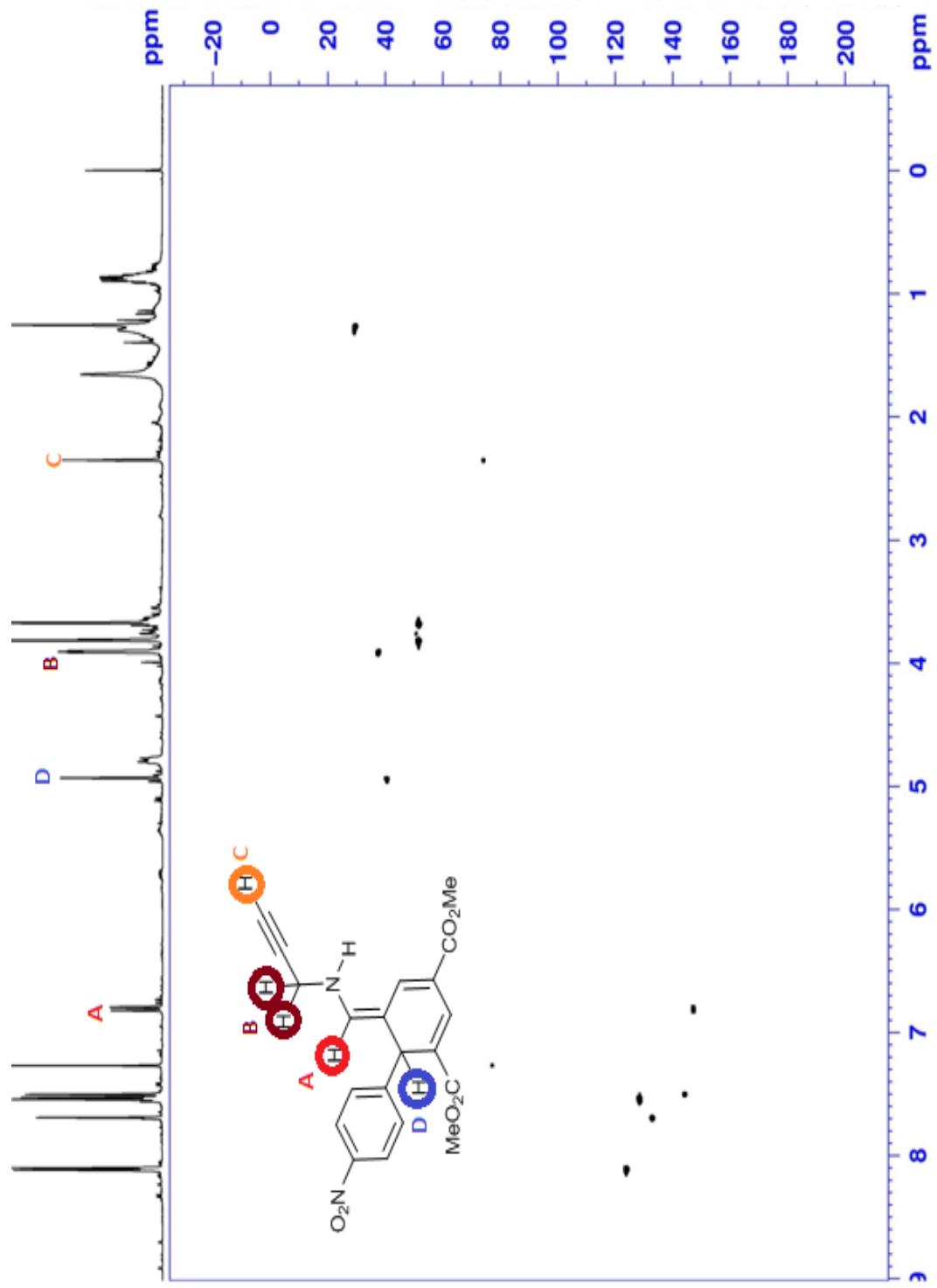
(D) -NH proton exchange experiment by addition of a drop of D₂O in CDCl₃ for Trienamine A:



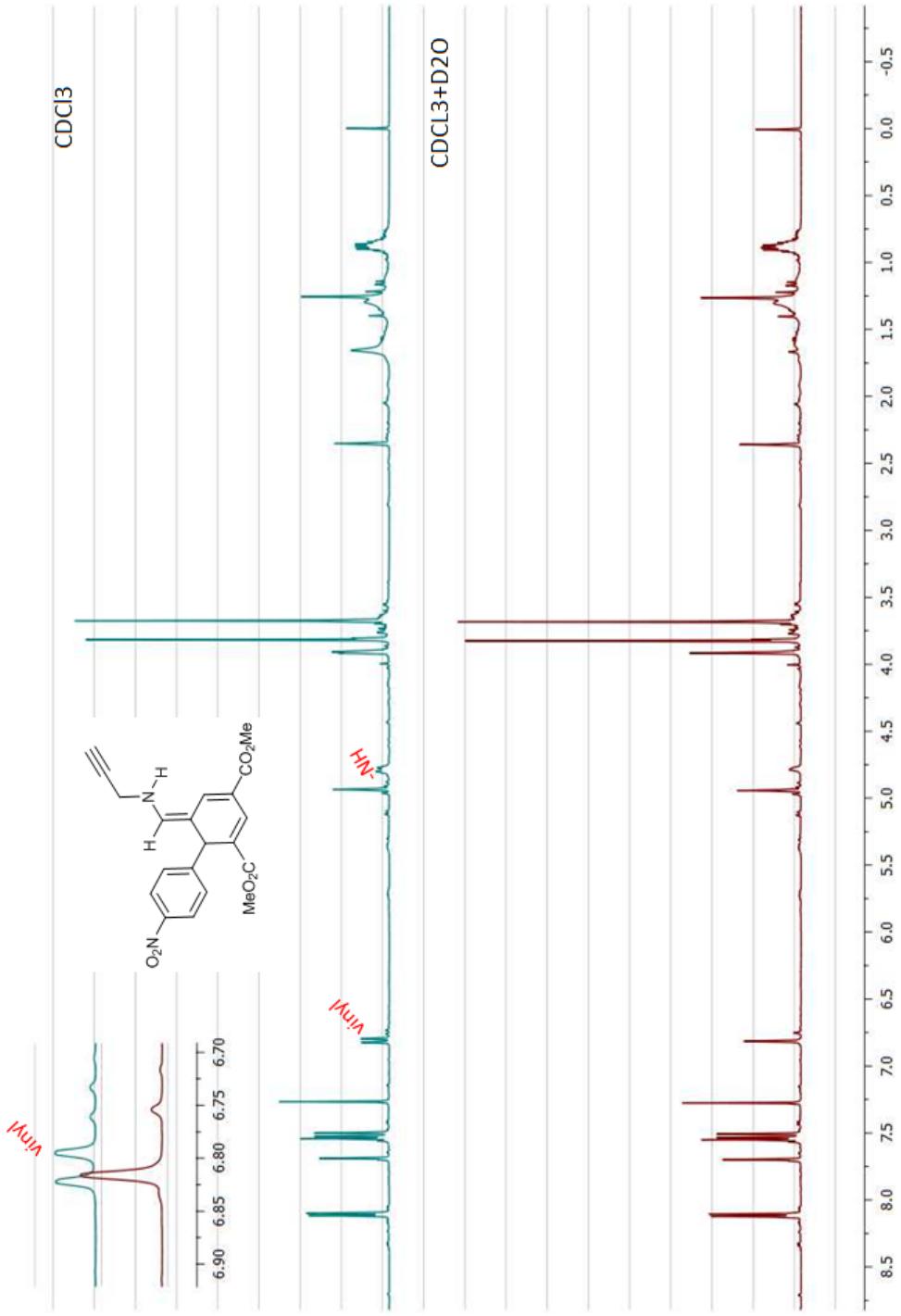
(a) COSY correlation for Trienamine A':



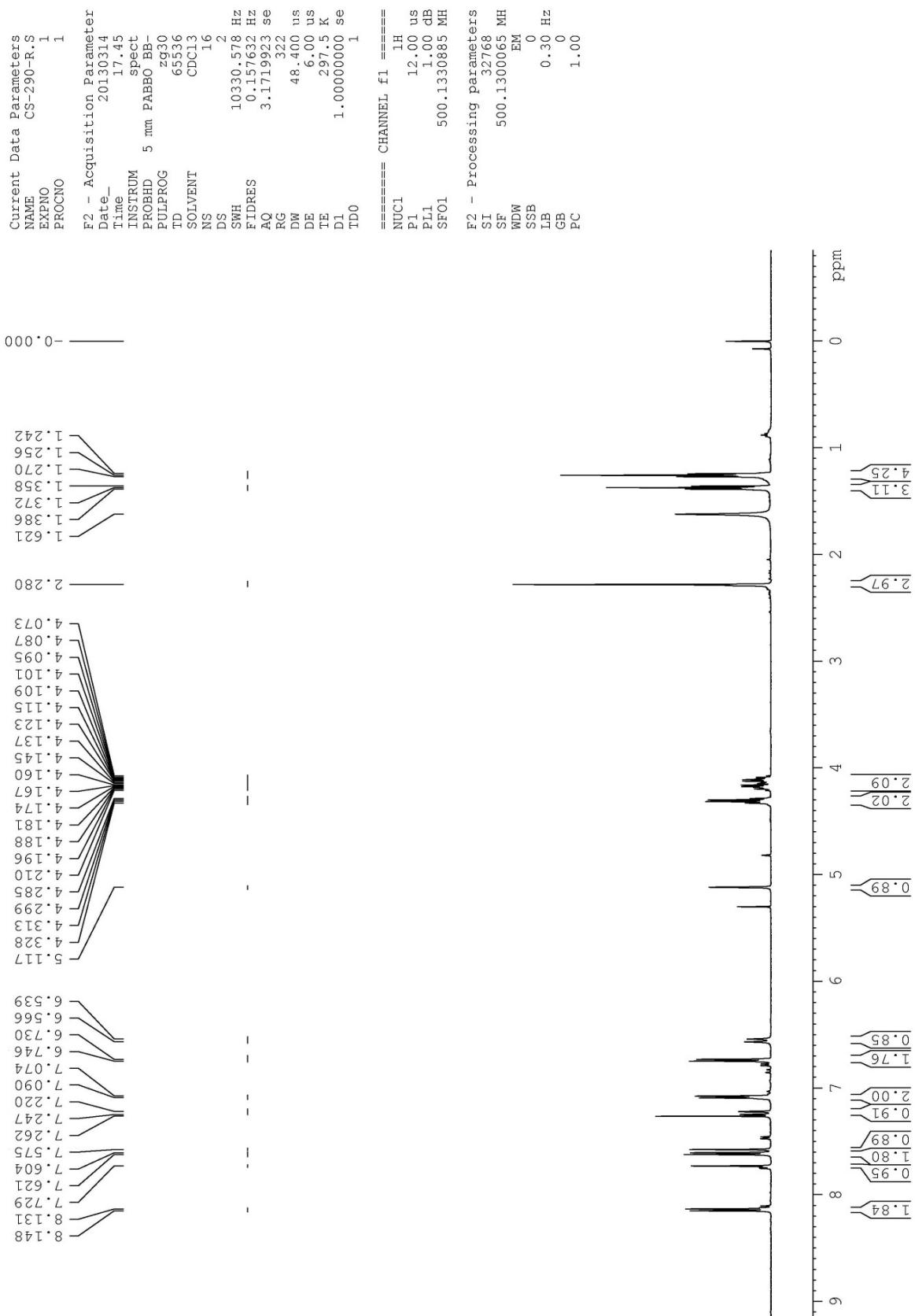
(b) HSQC correlation for Trienamine A' with only one olefinic proton

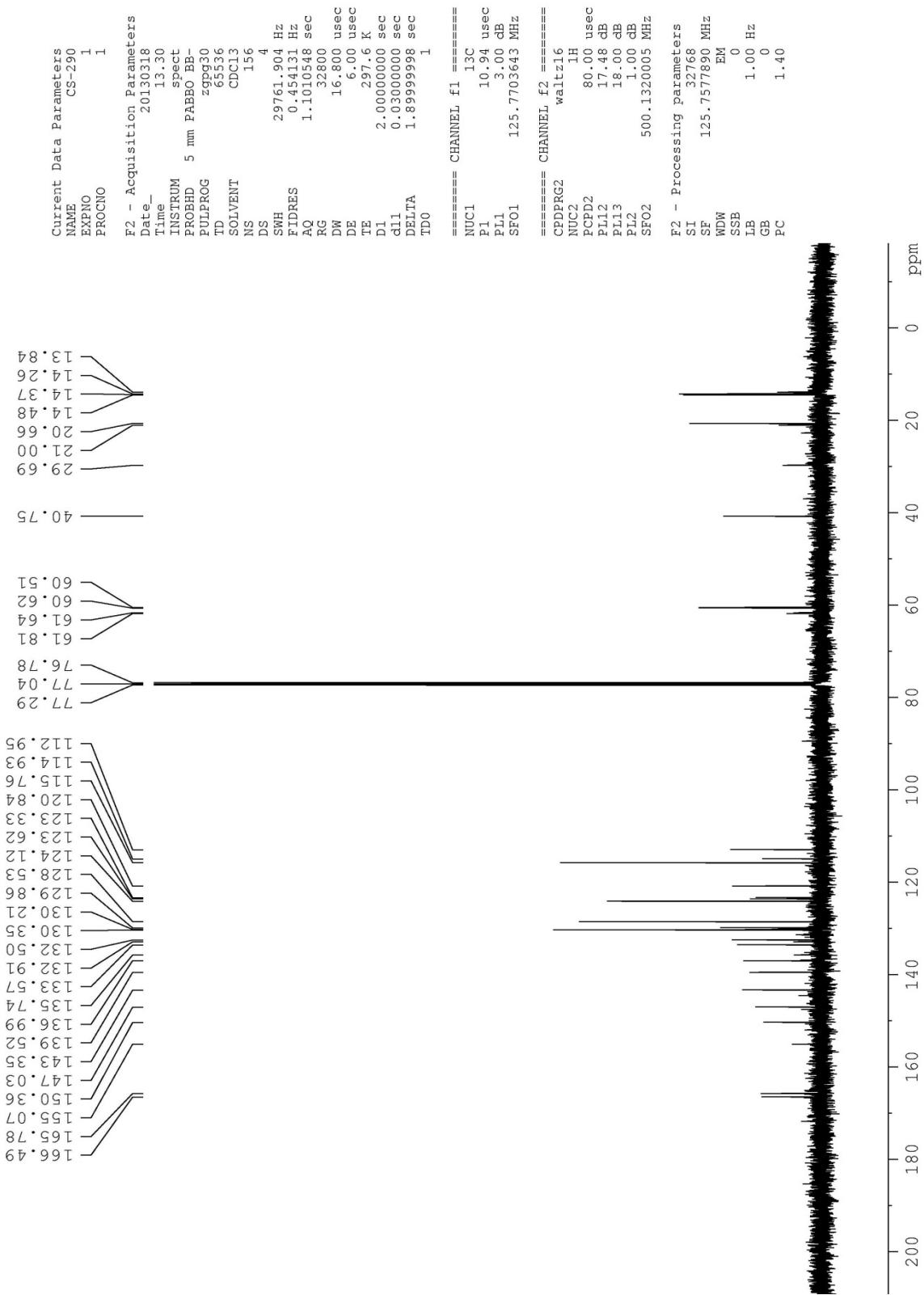


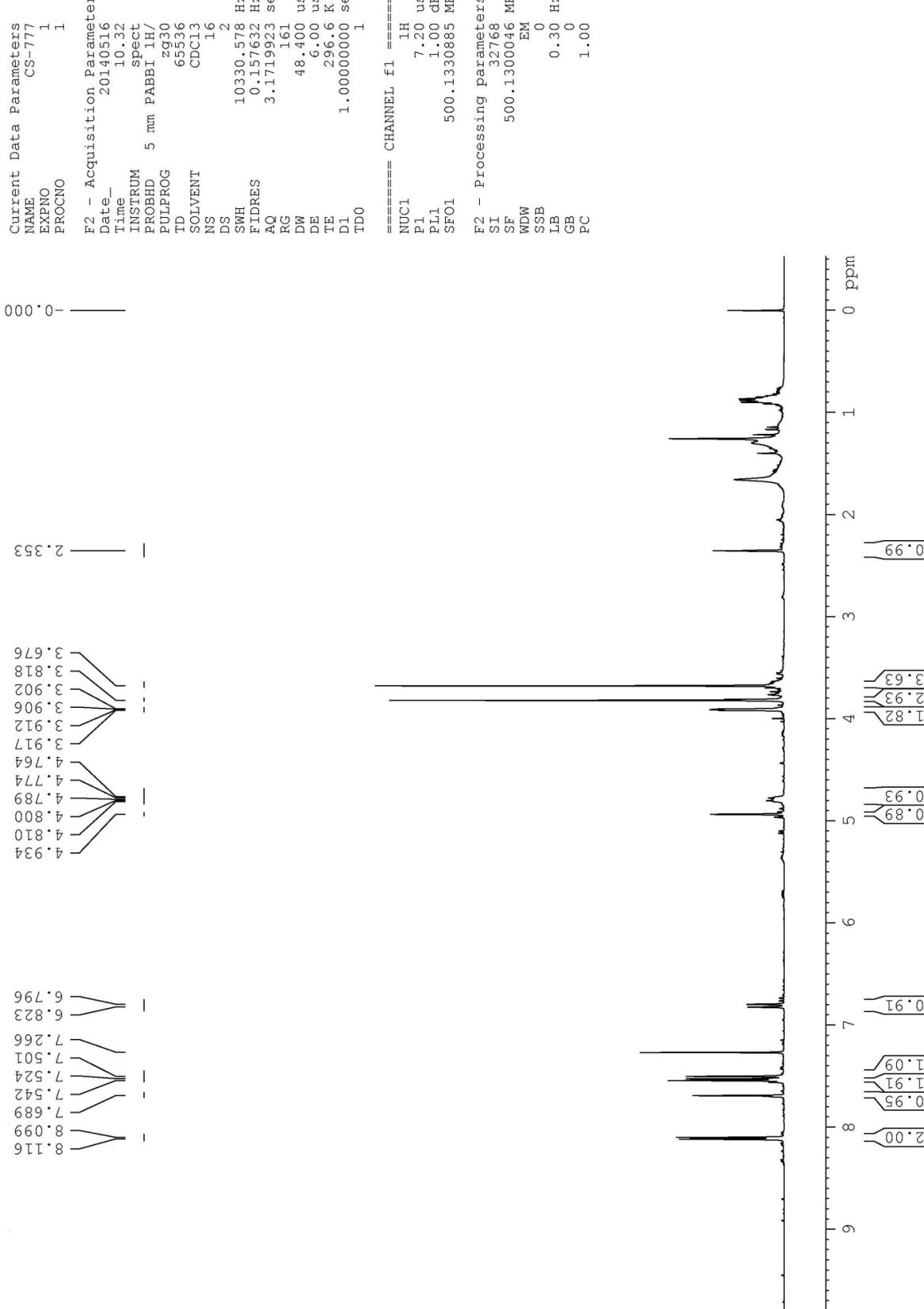
(c) NH proton exchange experiment by addition of a drop of D₂O in CDCl₃ for trienamine A':

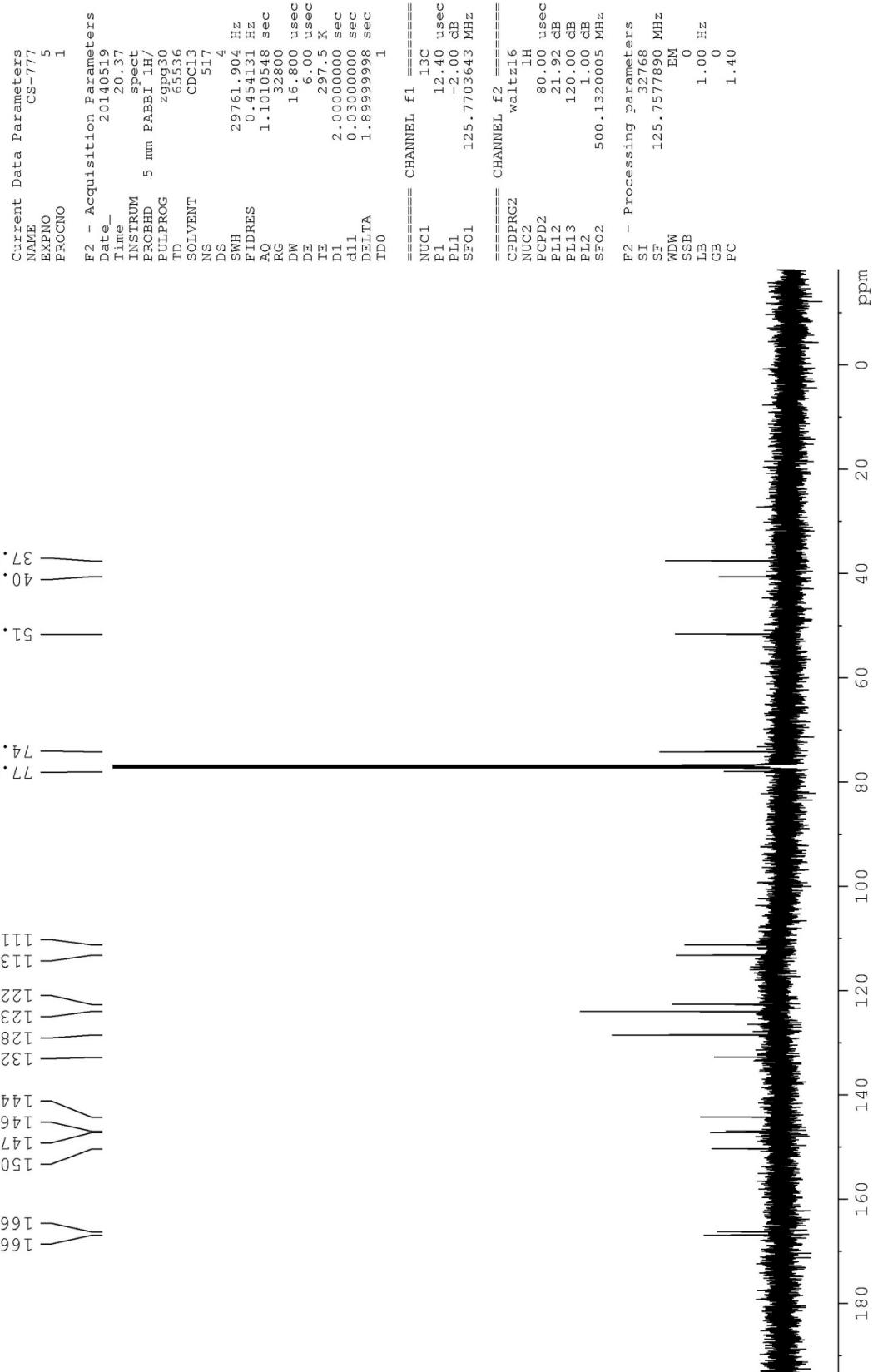


¹H and ¹³C NMR Copies of Trienamine A & A', Biaryl B', 4-6, 7a-7r & 8-12:









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Current Data Parameters
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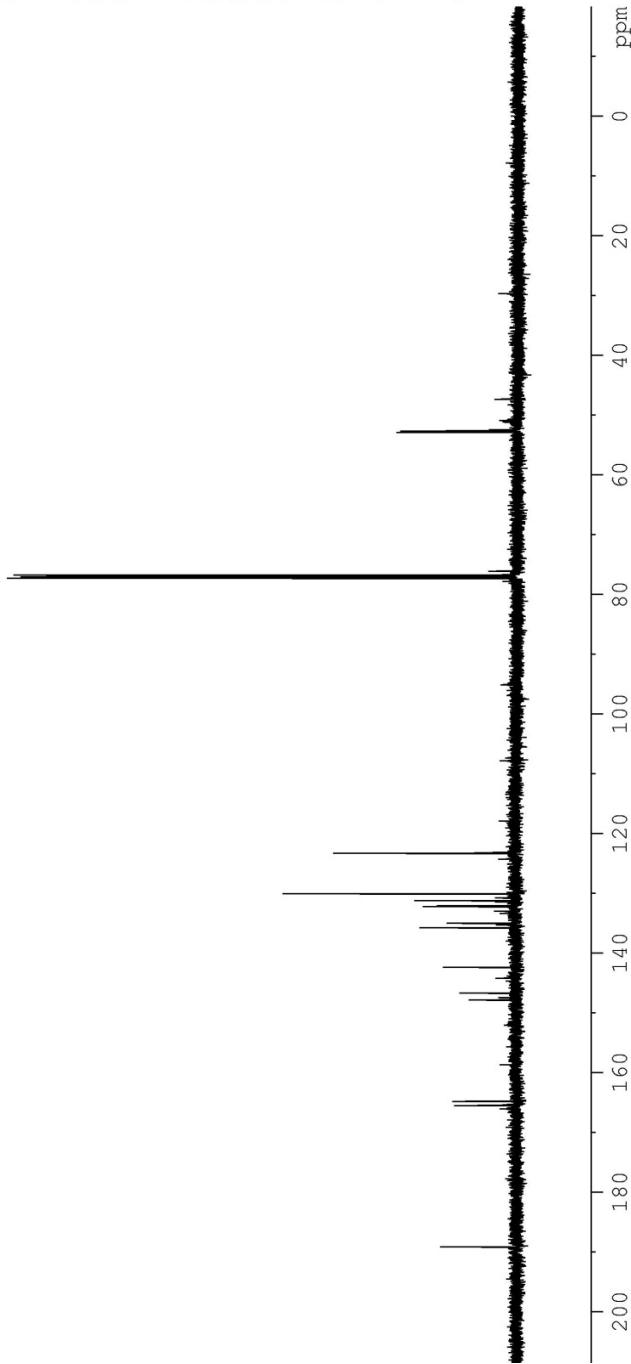
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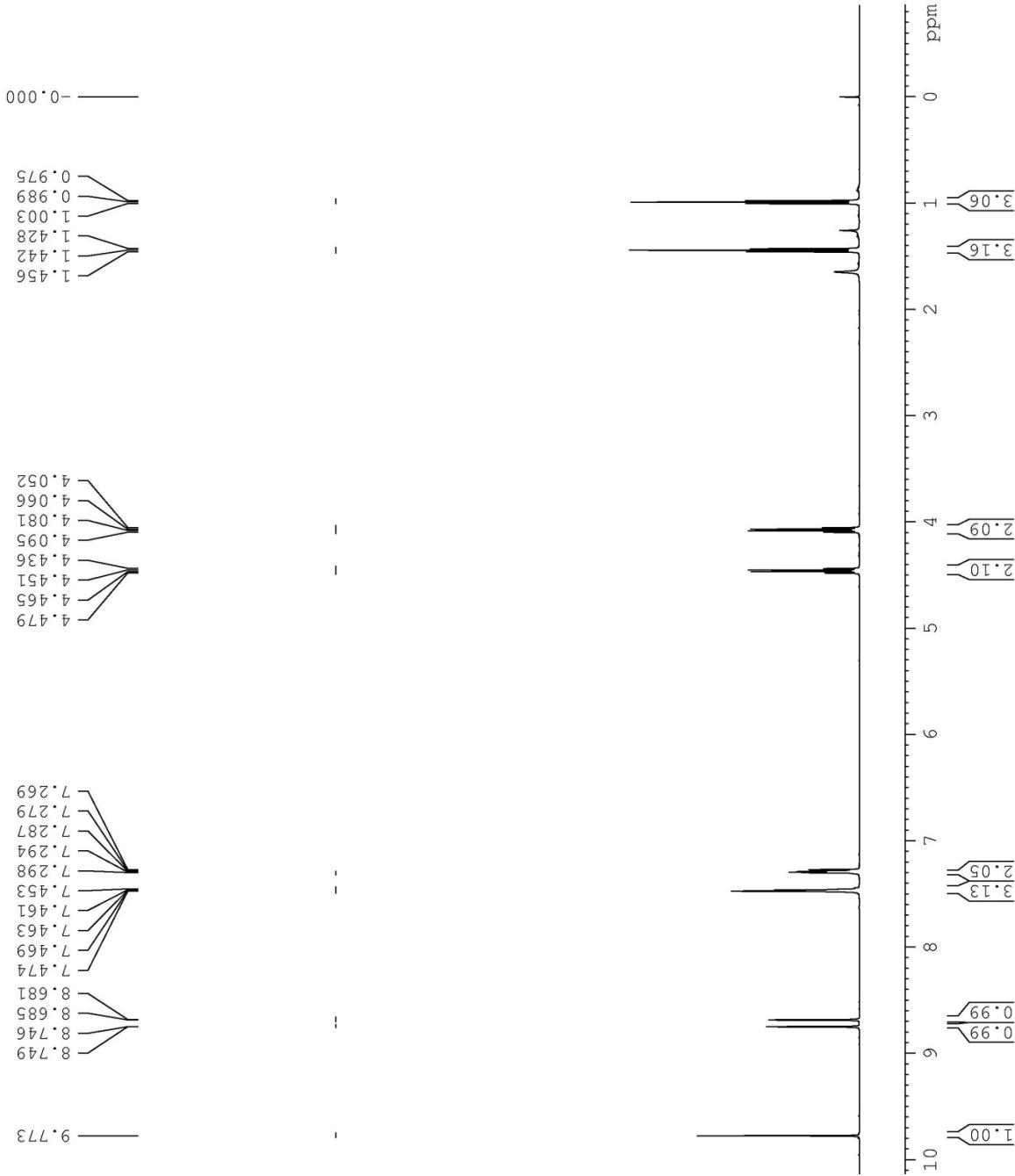
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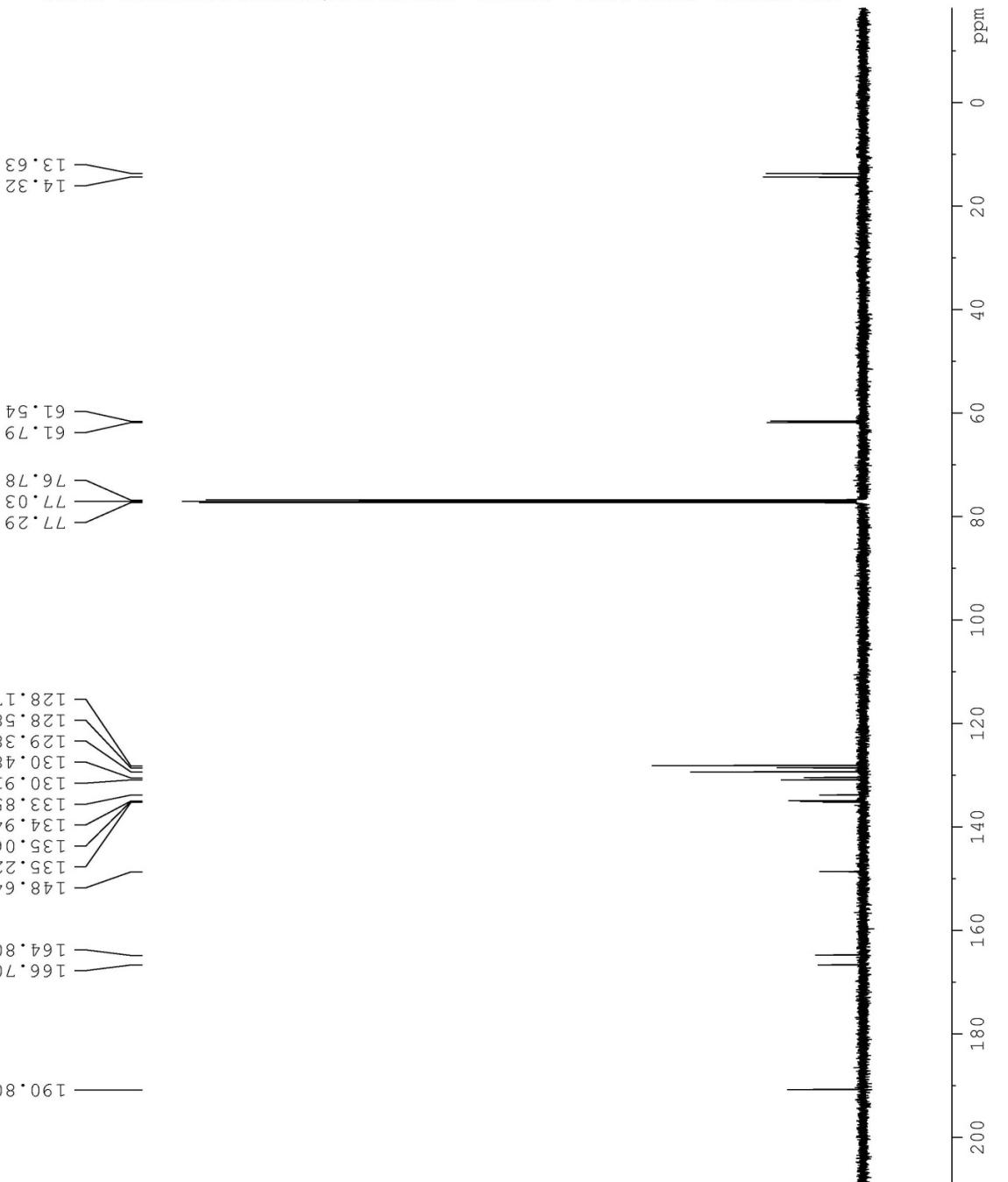
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3.927

3.941

3.955

3.969

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6.983

7.007

7.021

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7.315

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7.343

7.357

7.371

7.385

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7.983

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8.927

8.941

8.955

8.969

8.983

9.007

9.021

9.035

9.049

9.063

9.077

9.091

9.105

9.119

9.133

9.147

9.161

9.175

9.189

9.203

9.217

9.231

9.245

9.259

9.273

9.287

9.301

9.315

9.329

9.343

9.357

9.371

9.385

9.399

9.413

9.427

9.441

9.455

9.469

9.483

9.497

9.511

9.525

9.539

9.553

9.567

9.581

9.595

9.609

9.623

9.637

9.651

9.665

9.679

9.693

9.707

9.721

9.735

9.749

9.763

9.777

9.791

9.805

9.819

9.833

9.847

9.861

9.875

9.889

9.899

9.913

9.927

9.941

9.955

9.969

9.983

```

===== CHANNEL f1 =====
NUC1          1 H
P1           12.00 us
PL1          1.00 dB
SF01        500.1330885 MH

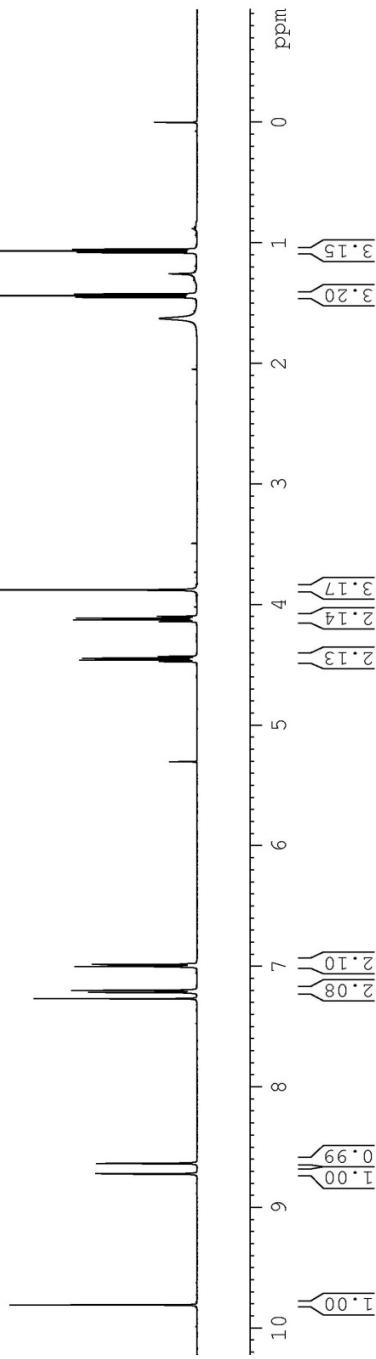
```

F2 - Processing parameters

SI	32768
SF	500.1300063
WDW	MH
SSB	EM
LB	0
GB	0.30 Hz
PC	0
	1.00

F2 - Processing parameters

SI	32768
SF	500.1300053
WDW	EM
SSB	0
LB	0.30
GB	Hz
PC	1.00



191.09
 166.95
 164.87
 159.97
 148.39
 135.31
 134.73
 130.89
 130.77
 127.11
 127.11
 113.69
 77.27
 76.77
 61.75
 61.56
 55.38
 31.80
 31.43

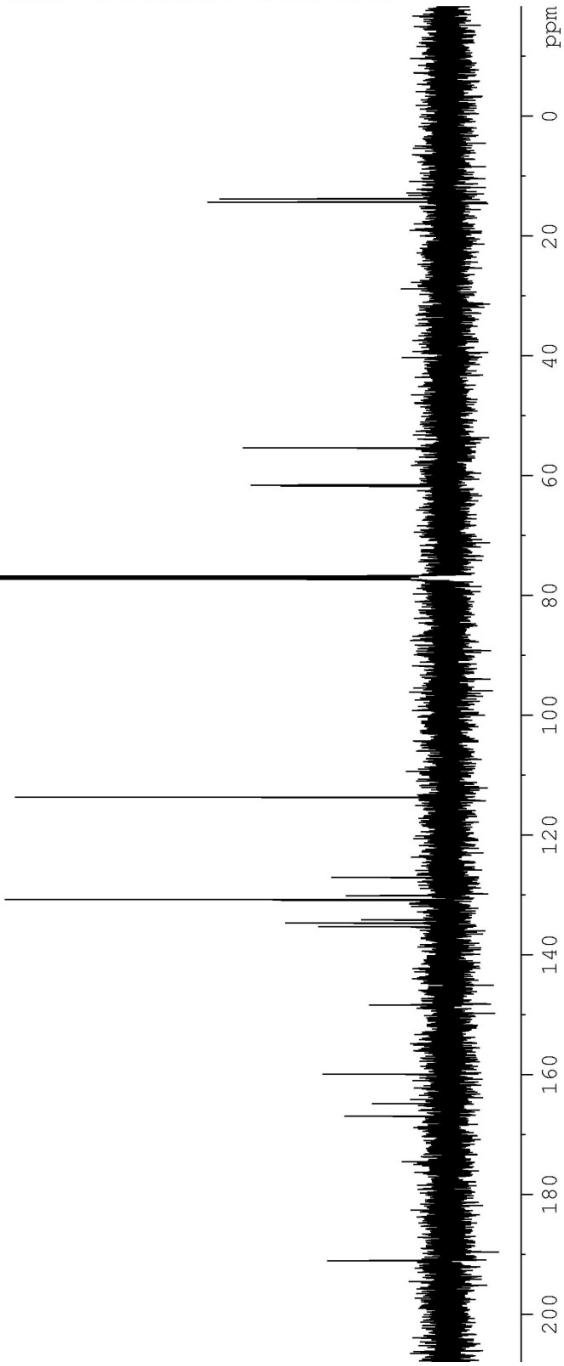
Current Data Parameters
 NAME CS-401
 EXPNO 2
 PROCN0 1

 F2 - Acquisition Parameters
 Date_ 20130617
 Time 12:46
 INSTRUM 5 mm PABBO BB-
 PROBHD spect
 PULPROG zgppg30
 TD 65536
 SOLVENT CDCl3
 NS 400
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.154131 Hz
 AQ 1.1010548 sec
 RG 32800
 DW 16.800 usec
 DE 6.00 usec
 TE 297.5 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.89999998 sec
 TDO 1

===== CHANNEL f1 ======
 NUC1 13C
 P1 10.94 usec
 PL1 3.00 dB
 SFO1 125.7703643 MHz

===== CHANNEL f2 ======
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL12 17.48 dB
 PL13 18.00 dB
 PL2 1.00 dB
 SF02 500.1320005 MHz

F2 - Processing parameters
 SI 32768
 SP 125.7577890 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

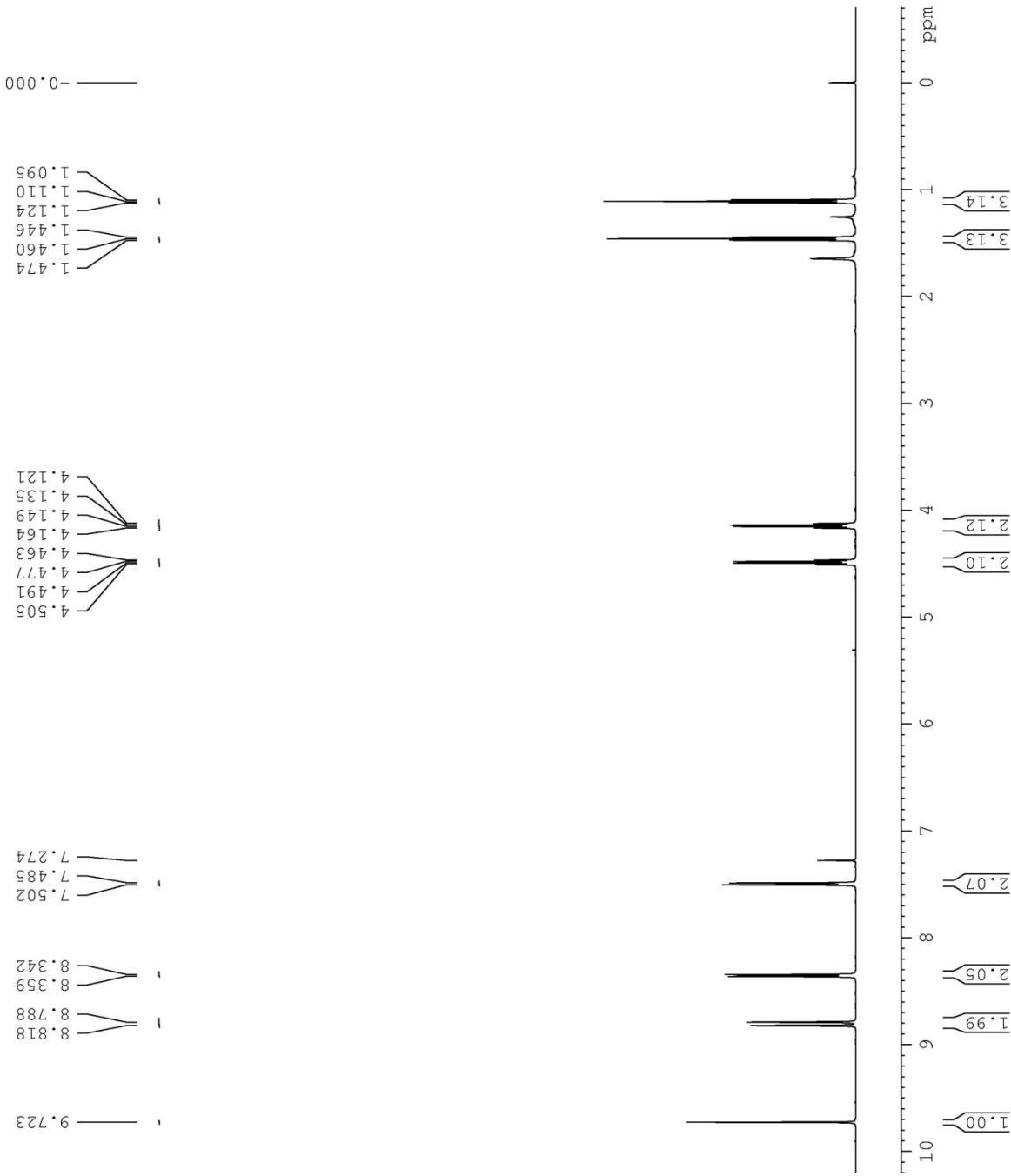


Current Data Parameters	
NAME	CS-387
EXPNO	1
PROCNO	1
F2 - Acquisition Parameters	
Date_	20130607
Time	9.40
INSTRUM	PABBO
PROBHD	5 mm BB-
PULPROG	2930
TD	65536
SOLVENT	CD13
NS	16
DS	
SWH	10330.578
FIDRES	0.157632
AQ	3.1719923
RG	203
DW	48.100
DE	6.00
TE	297.3
DI	1
1.00000000	
DFO	1

```

===== CHANNEL f1 =====
NUC1          1.00 us
P1            12.00 us
PL1           1.00 dB
SFO1          500..1330885 MH
F2 - Processing parameters
SI             32768
SF            500..1300010 MH
WDW           EM
SSB            0
LB            0..30 Hz
GB            0
PC            0

```



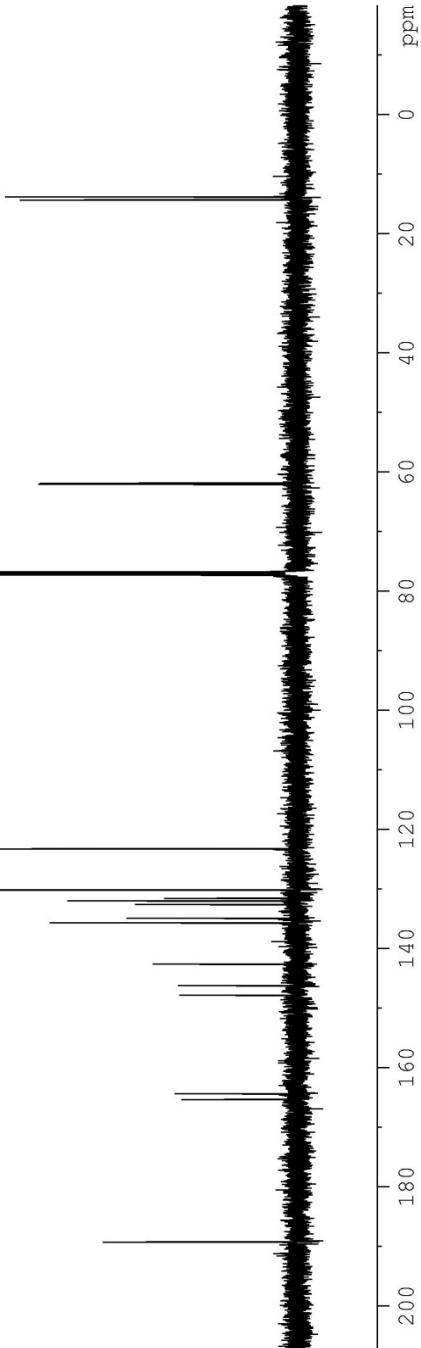
189.31
 165.38 164.41
 147.88 146.27 142.67 135.71 134.92 132.65 132.04 131.61 130.21 123.30
 77.29 77.03 76.78 62.06 61.90
 14.29 14.21

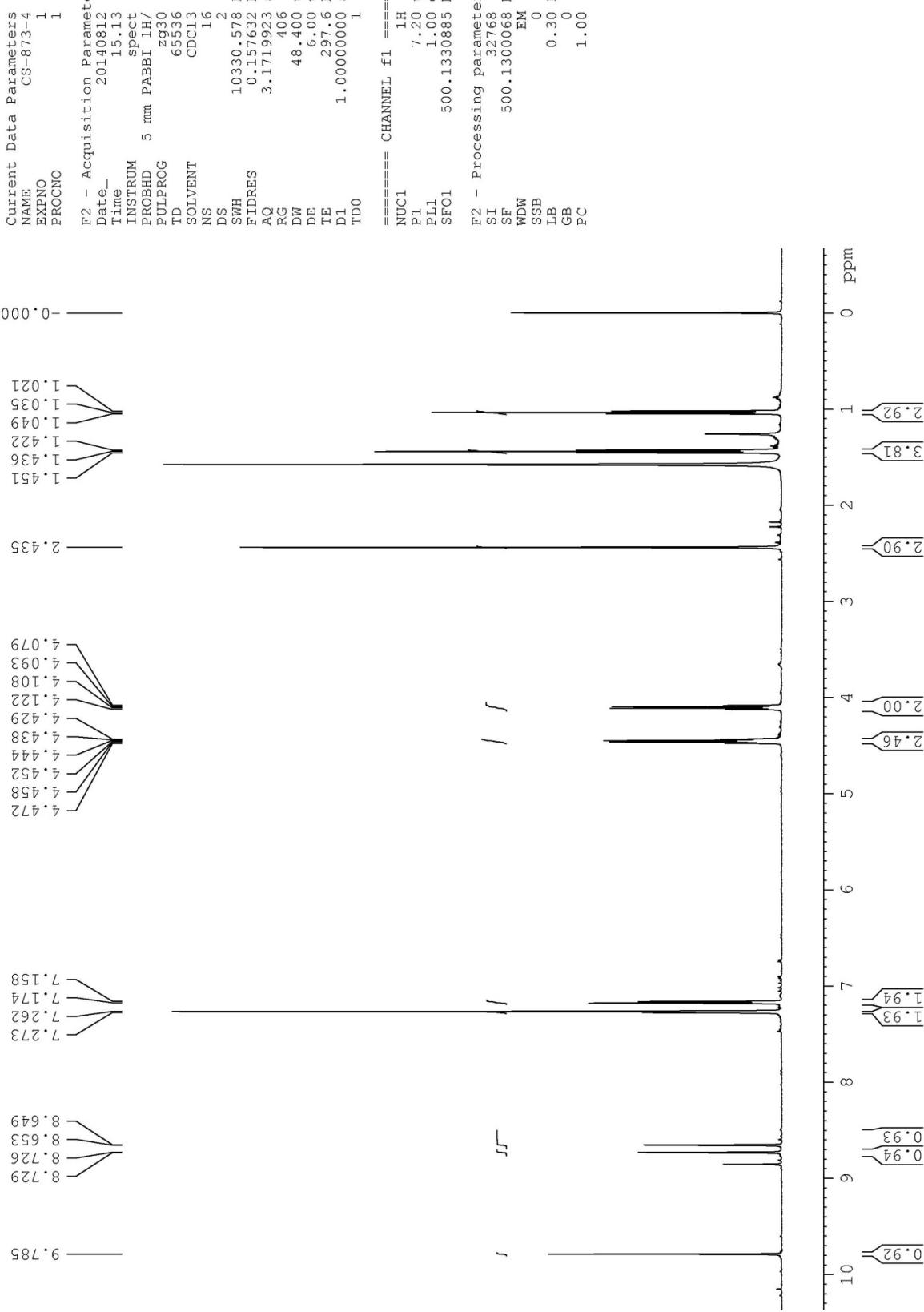
Current Data Parameters
 NAME CS-387
 EXPNO 2
 PROCN0 1

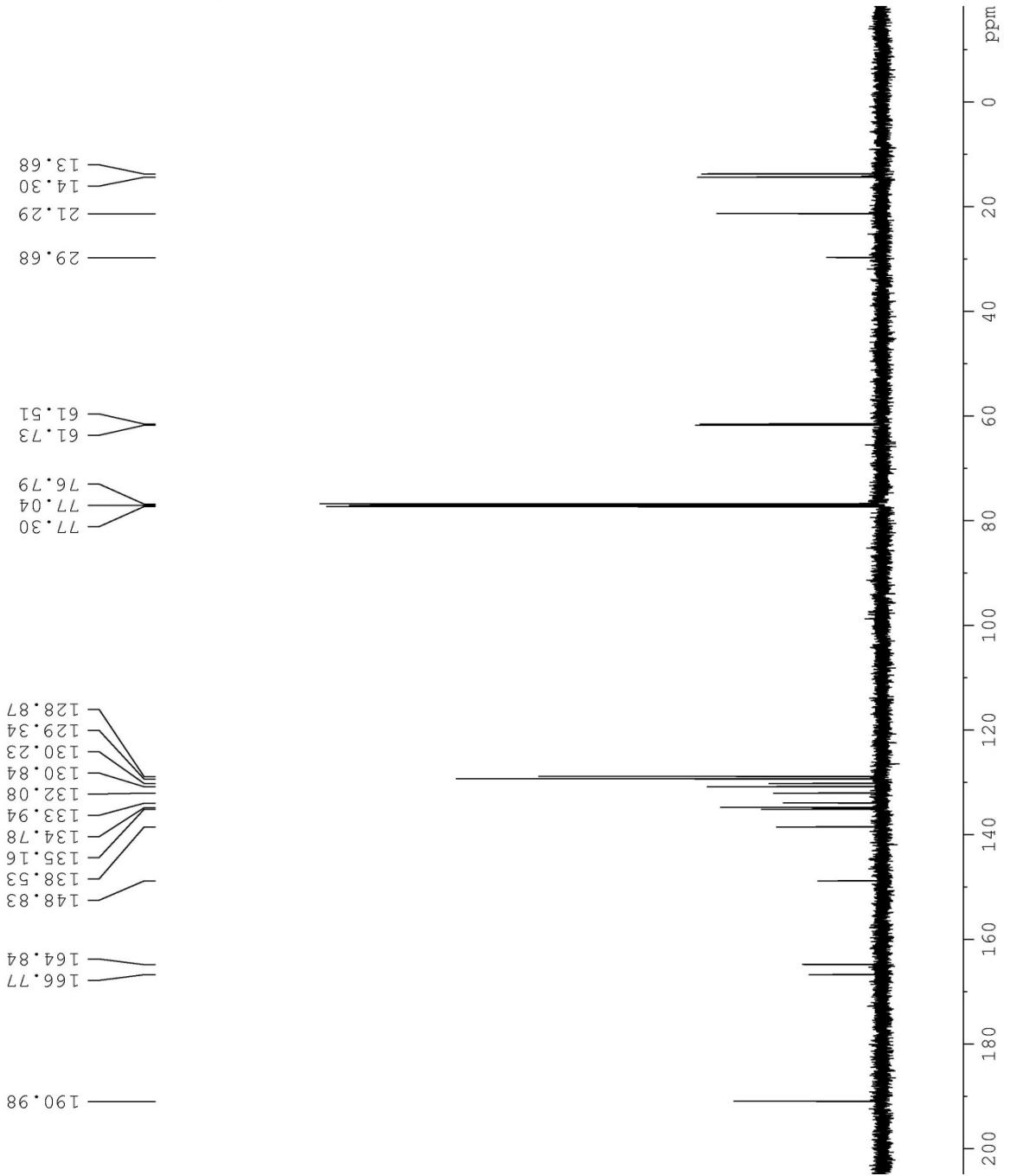
F2 - Acquisition Parameters
 Date_ 20130607
 Time 9.57
 INSTRUM spect
 PROBHD 5 mm FABBO BB-
 PULPROG zgppg30
 TD 65536
 SOLVENT CDC13
 NS 296
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010548 sec
 RG 32800
 DW 16.800 usec
 DE 6.00 usec
 TE 2.98.2 K
 D1 2.0000000 sec
 d11 0.1300000 sec
 DELTA 1.8999998 sec
 TDO 1

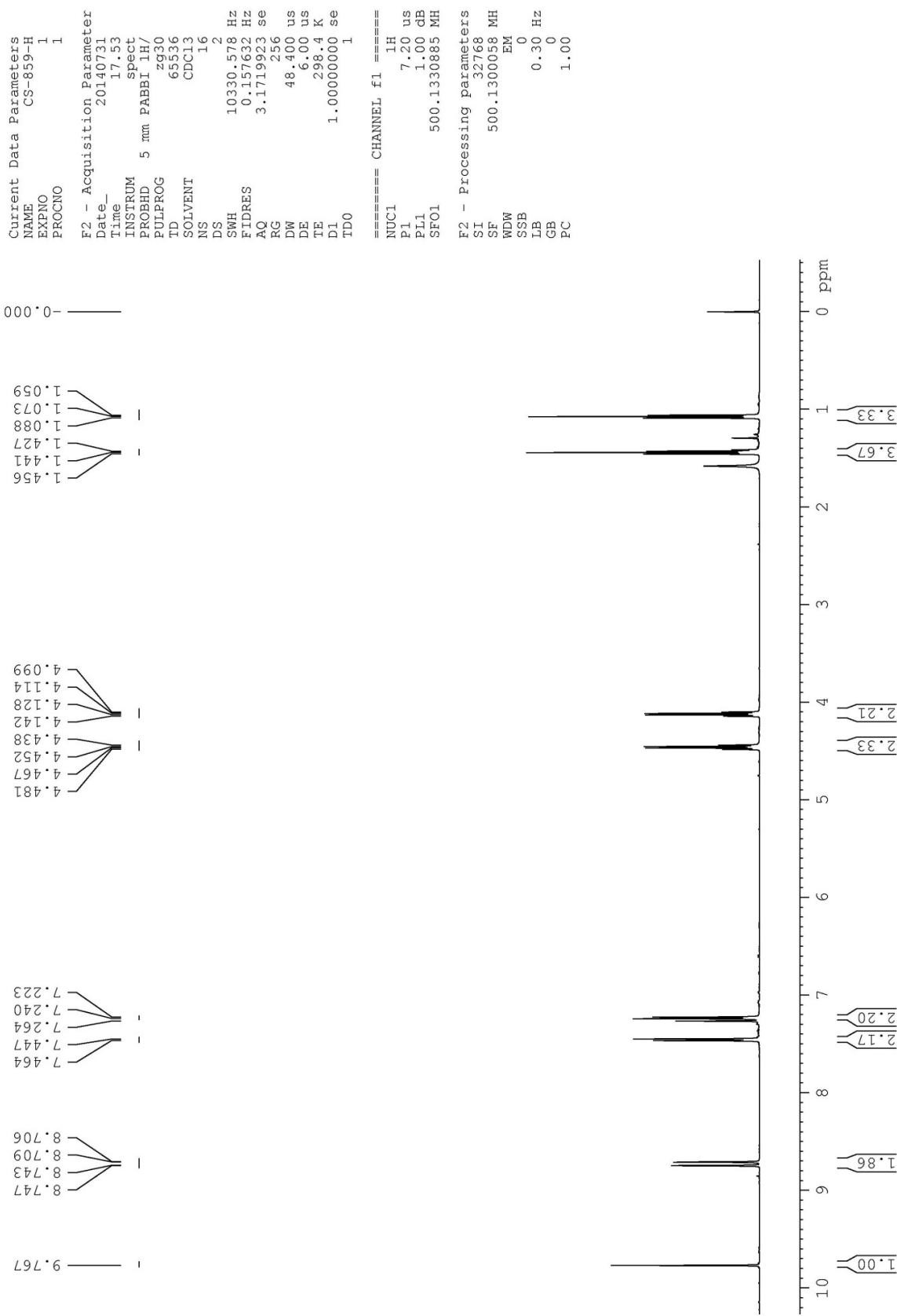
===== CHANNEL f1 ======
 NUC1 13C
 P1 10.94 usec
 PL1 3.00 dB
 SFO1 125.7703643 MHz
 ===== CHANNEL f2 ======
 CPDPRG2 waltz16
 NUO2 1H
 PCPD2 80.00 usec
 PL12 17.48 dB
 PL13 18.00 dB
 PL2 1.00 dB
 SFO2 500.1320005 MHz

F2 - Processing parameters
 SI 32768
 SF 125.7577890 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40







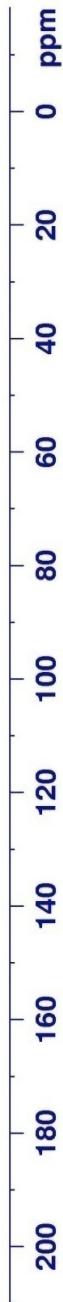


Current Data Parameters
NAME CS-530
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130917
Time 19.21
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgppg30
TD 65536
SOLVENT CDC13
NS 256
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 32800
DW 16.800 usec
DE 6.00 usec
TE 297.2 K
D1 2.0000000 sec
d11 0.0300000 sec
DELTA 1.8999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.40 usec
PL1 125.7703643 MHz
SFO1
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL1.2 21.92 dB
PL1.3 120.00 dB
PL2 1.00 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

147.35
166.24
164.65
190.26
133.71
135.20
135.10
134.89
133.71
133.54
131.22
130.83
130.65
128.45
77.28
77.03
76.77
61.87
61.71
13.30
14.30
13.73

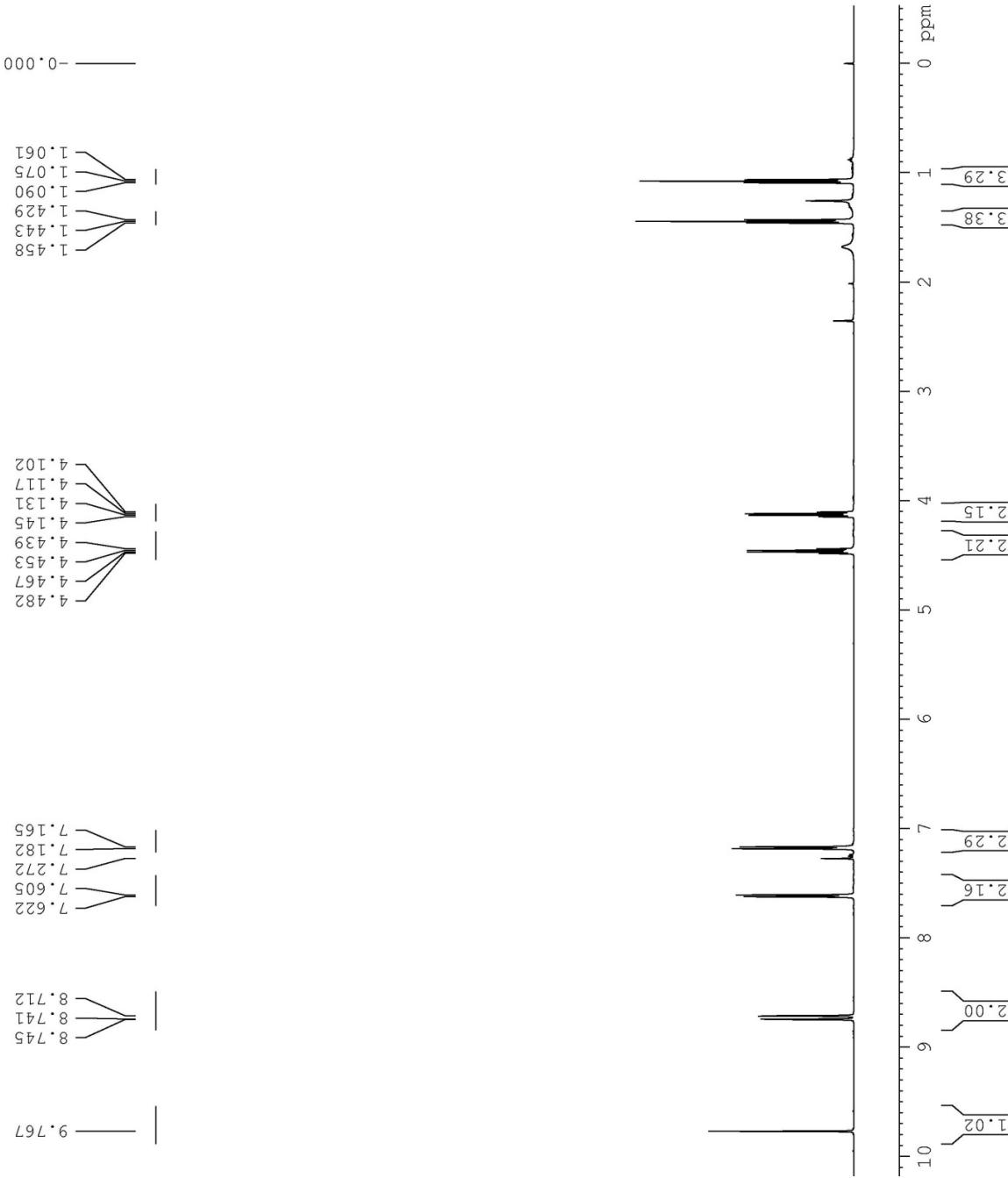


Current Data Parameters
 NAME CS-834-D
 EXPNO 2
 PROCN0 1

F2 - Acquisition Parameter
 Date_ 20140725
 Time_ 10.29
 INSTRUM spect
 PROBHD 5 mm PABBI 1H/
 PULPROG 2930
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 se
 RG 71.8
 DW 48.400 us
 DE 6.00 us
 TE 295.5 K
 D1 1.00000000 se
 TDO 1

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

NUC1 1H
 P1 7.20 us
 PL1 1.00 dB
 SF01 500.1330885 MH



Current Data Parameters
NAME CS-535
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130924
Time 12.55

INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgppg30
TD 65536

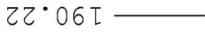
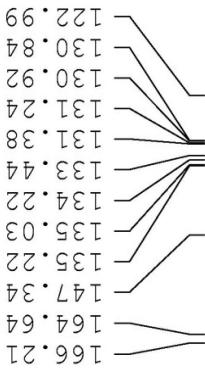
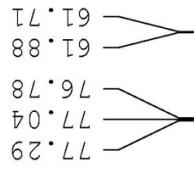
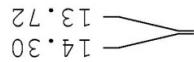
SOLVENT CDC13
NS 256
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec

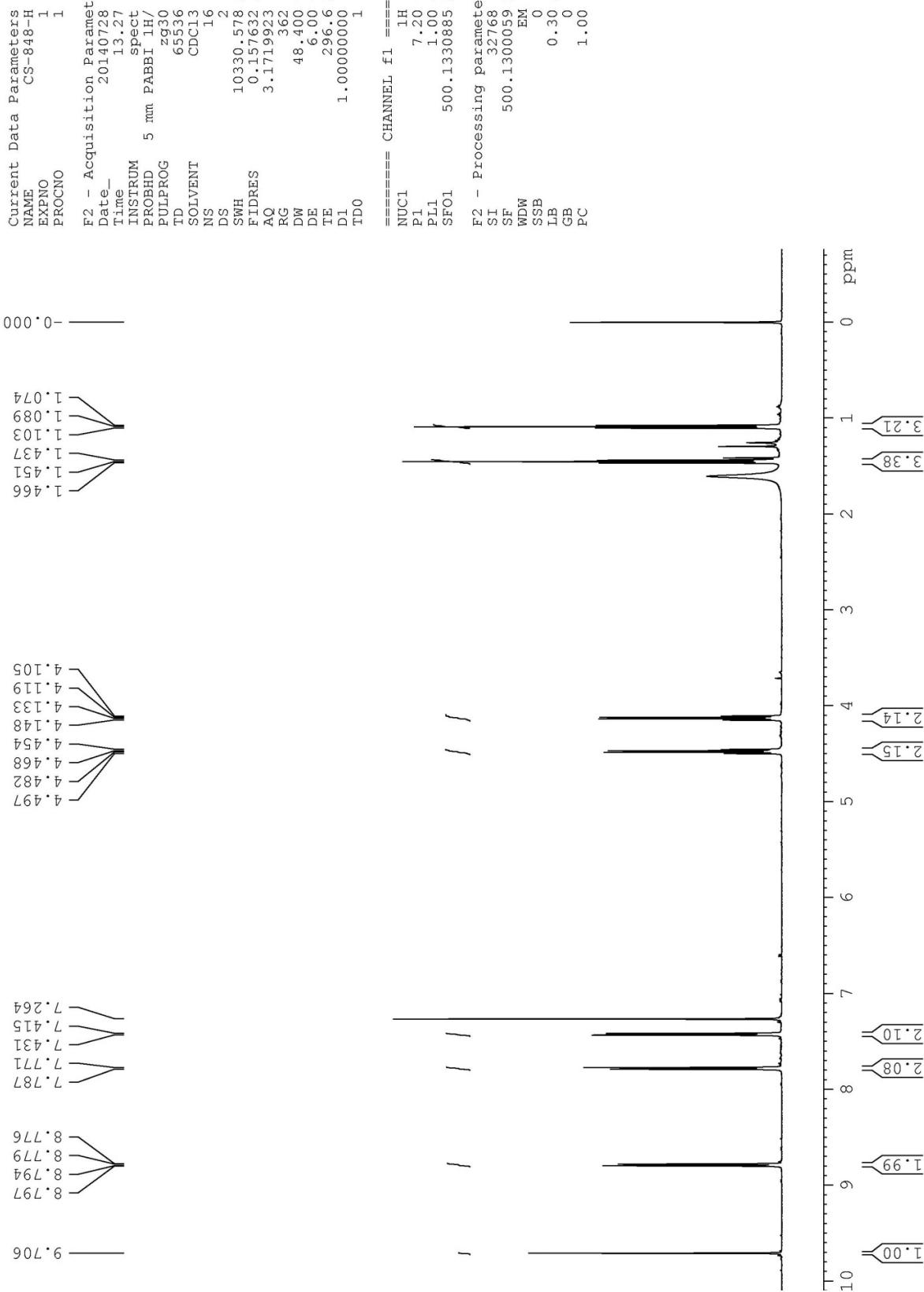
RG 32800
DW 16.800 usec
DE 6.00 usec
TE 297.0 K
D1 2.0000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.40 usec
PL1 125.7703643 MHz
SF01

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 21.92 dB
PL13 120.00 dB
PL12 1.00 dB
SF02 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





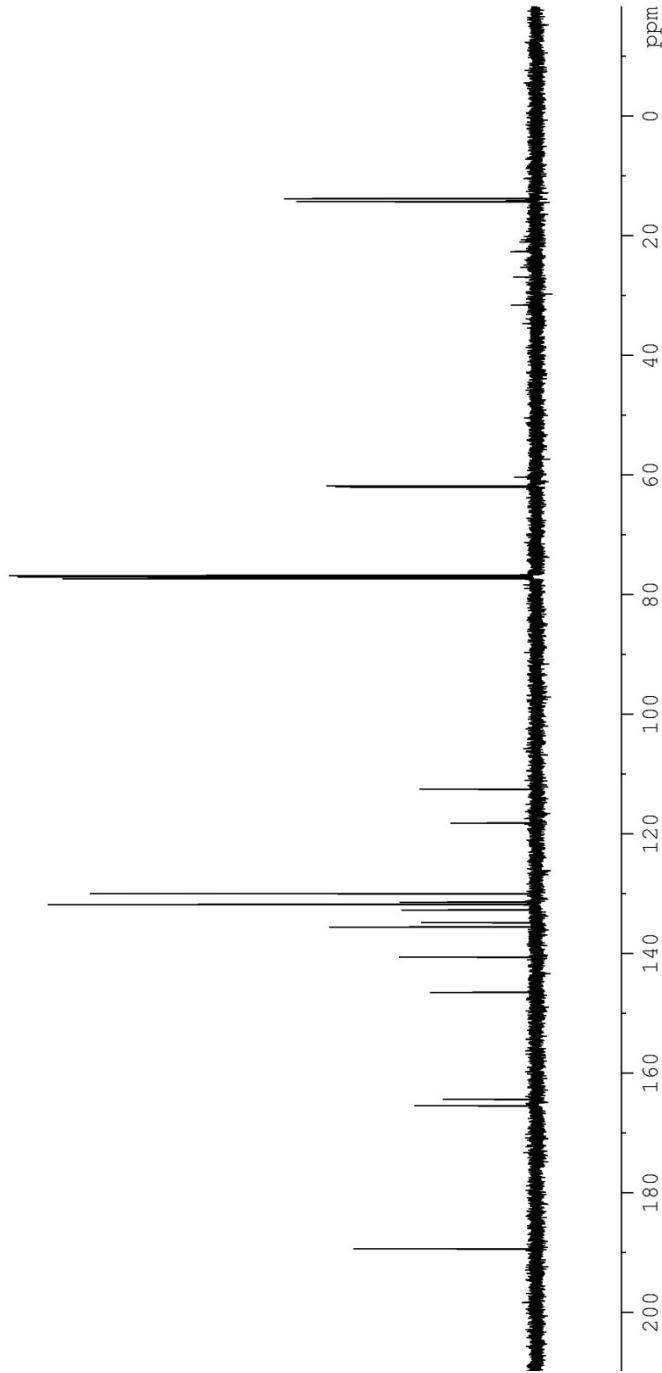


Current Data Parameters
NAME CS-428
EXNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130709
Time_ 15.44
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 120
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 32800
DW 16.800 usec
DE 6.00 usec
TE 297.8 K
D1 2.0000000 sec
d11 0.0300000 sec
DELT1A 1.8999998 sec
TD0 1

===== CHANNEL f1 ======
NUC1 13C
P1 10.94 usec
PL1 3.00 dB
SF01 125.7703643 MHz

===== CHANNEL f2 ======
CPDPRG2 watzl16
NUC2 1H
PCPD2 80.00 usec
PL1,2 17.48 dB
PL1,3 18.00 dB
PL2 1.00 dB
SFQ2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



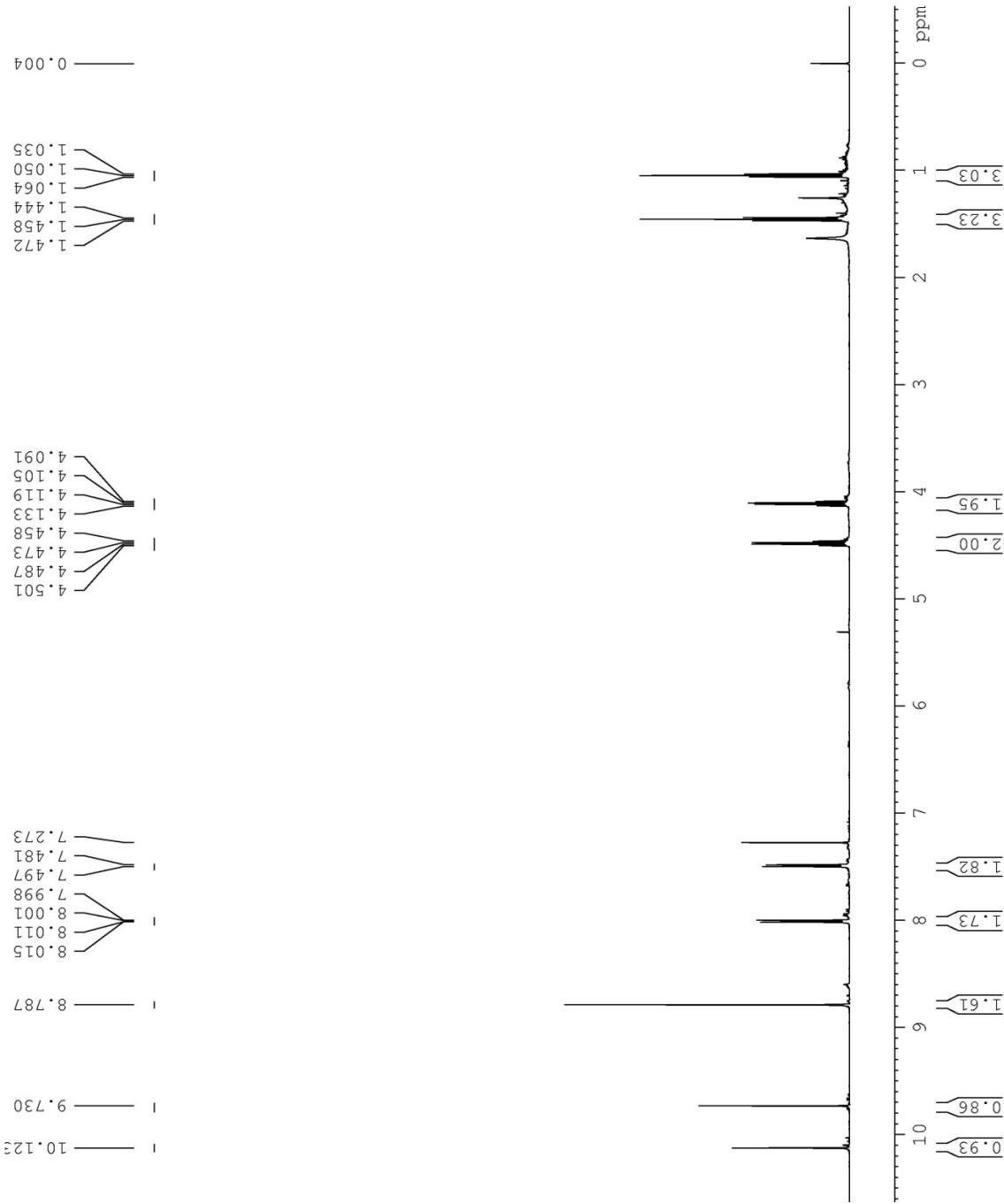
```

Current Data Parameters          F2 - Acquisition Parameters
NAME      CS-562           Date_   2013\01\06
EXPNO     1                 Time_  13:30
PROCNO    1

F2 - Processing parameters
CHANNEL f1 =====
NUC1L    1H
P1       7.20 u
PL1      1.00 d
SFO1    500.1330885 M

F2 - Processing parameter
SI      32768
SF      500.1300000 M
WDW     EM
SSB     0
LB      0.30 H
GB      1.00
PC      1.00

```





Current Data Parameters
NAME CS-562
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters

Date 20131017
Time 14.06
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgpp30
TD 65536
SOLVENT CDC13
NS 256
DS 4
SWH 29761.904 Hz
FIDRES 0.451131 Hz
AQ 1.1010548 sec
RG 32800
DW 16.800 usec
DE 6.00 usec
TE 297.0 K
D1 2.000000 sec
d1 0.10300000 sec
DELT A 1.8999998 sec
TD0 1

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

NUC1 13C
P1 12.40 usec
PL1 -2.00 dB
SFO1 125.7703643 MHz

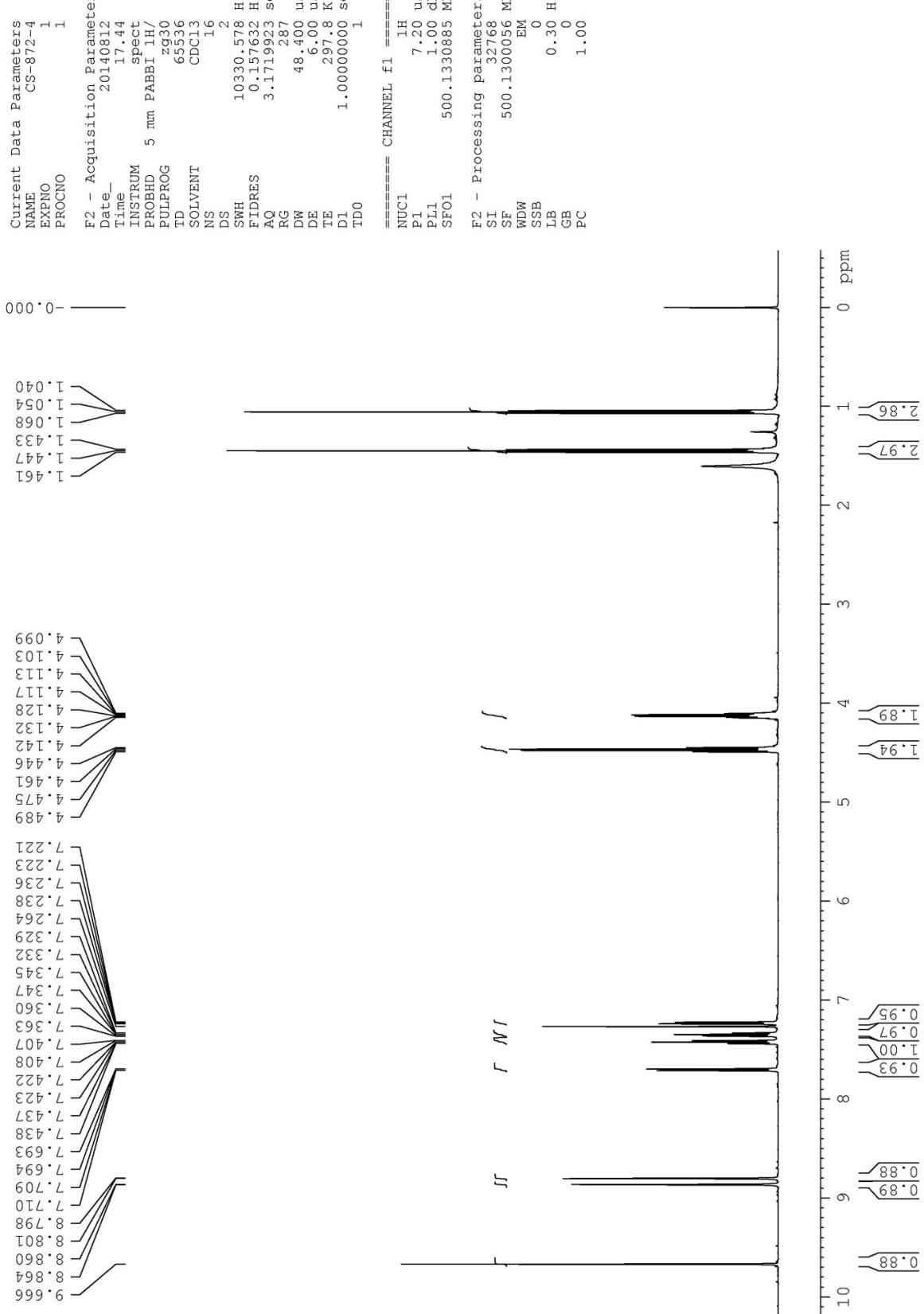
===== CHANNEL f2 =====

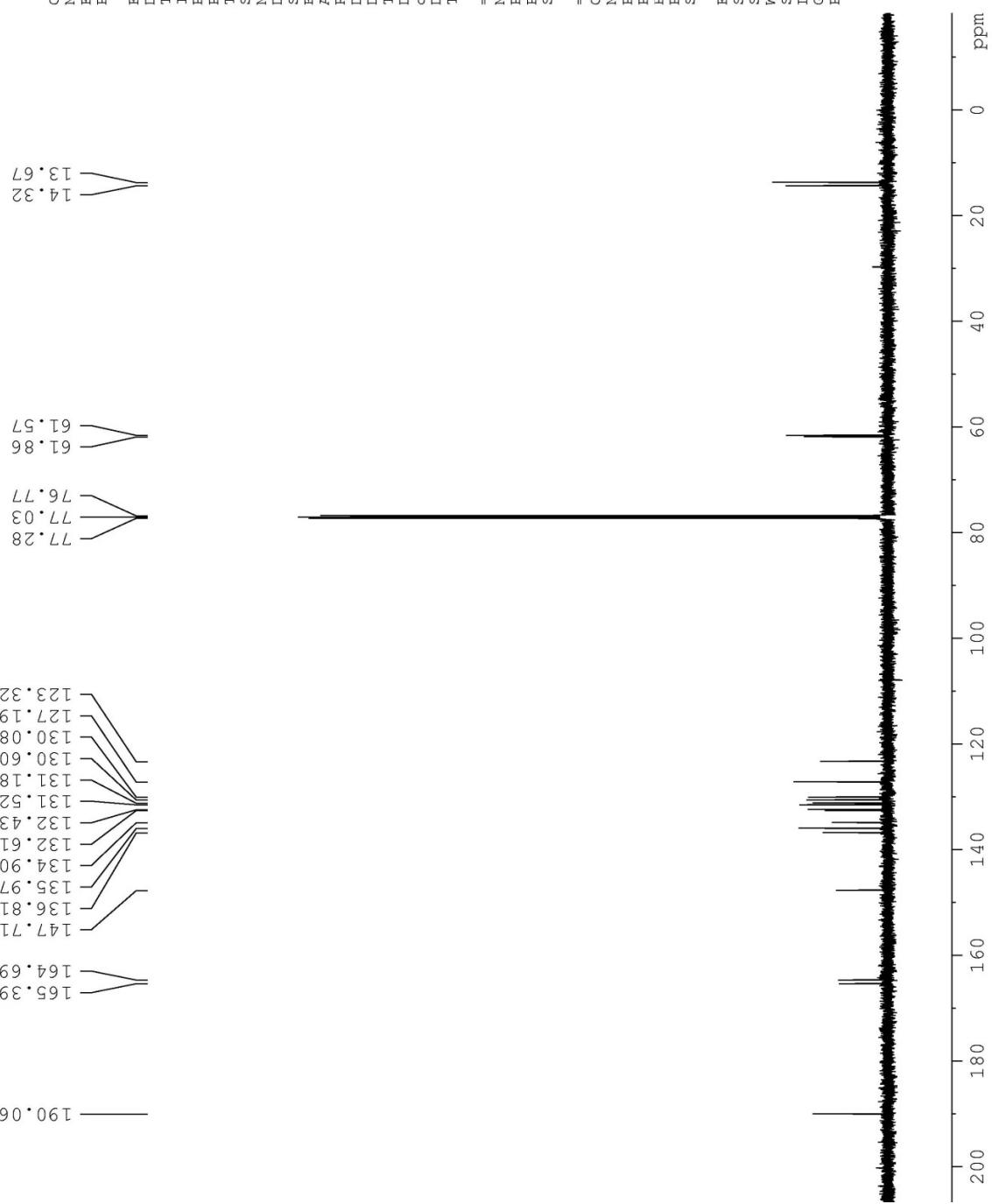
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL1.2 21.92 dB
PL1.3 120.00 dB
PL2 1.00 dB
SFO2 500.1320005 MHz

F2 - Processing parameters

SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40







Current Data Parameters
NAME CS-600
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20131115
Time_ 11:30
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgppg30
TD 65536
SOLVENT CDCl3
NS 272
DS 4
SWH 29751.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 32800
DW 16.800 usec
DE 6.00 usec
TE 297.0 K
D1 2.0000000 sec
d11 0.0300000 sec
DELT1A 1.8999999 sec
TDO 1

===== CHANNEL F1 =====
NUC1 13C
P1 12.40 usec
PL1 125.7703643 MHz
SFO1 125.7703643 MHz

===== CHANNEL F2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL1.2 21.92 dB
PL1.3 120.00 dB
PL2 1.00 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Current Data Parameters

NAME NAH-3-1

EXPNO 1

PROCNO 1

F2 - Acquisition Parameter

Date_ 20140228

Time_ 17.00

INSTRUM spect

PROBHD 5 mm PABBI 1H/

PULPROG zg30

TD 65336

SOLVENT CDCl3

NS 16

DS 2

SWH 10330.578 Hz

FIDRES 0.157632 Hz

AQ 3.1719923 se

RG 406

DW 48.000 us

DE 6.000 us

TE 298.5 K

D1 1.00000000 se

TDO 1

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

NUC1 1H

P1 7.20 us

PL1 1.00 dB

SF01 500.1330885 MH

EM

F2 - Processing parameters

SI 32768

SF 500.1300000 MH

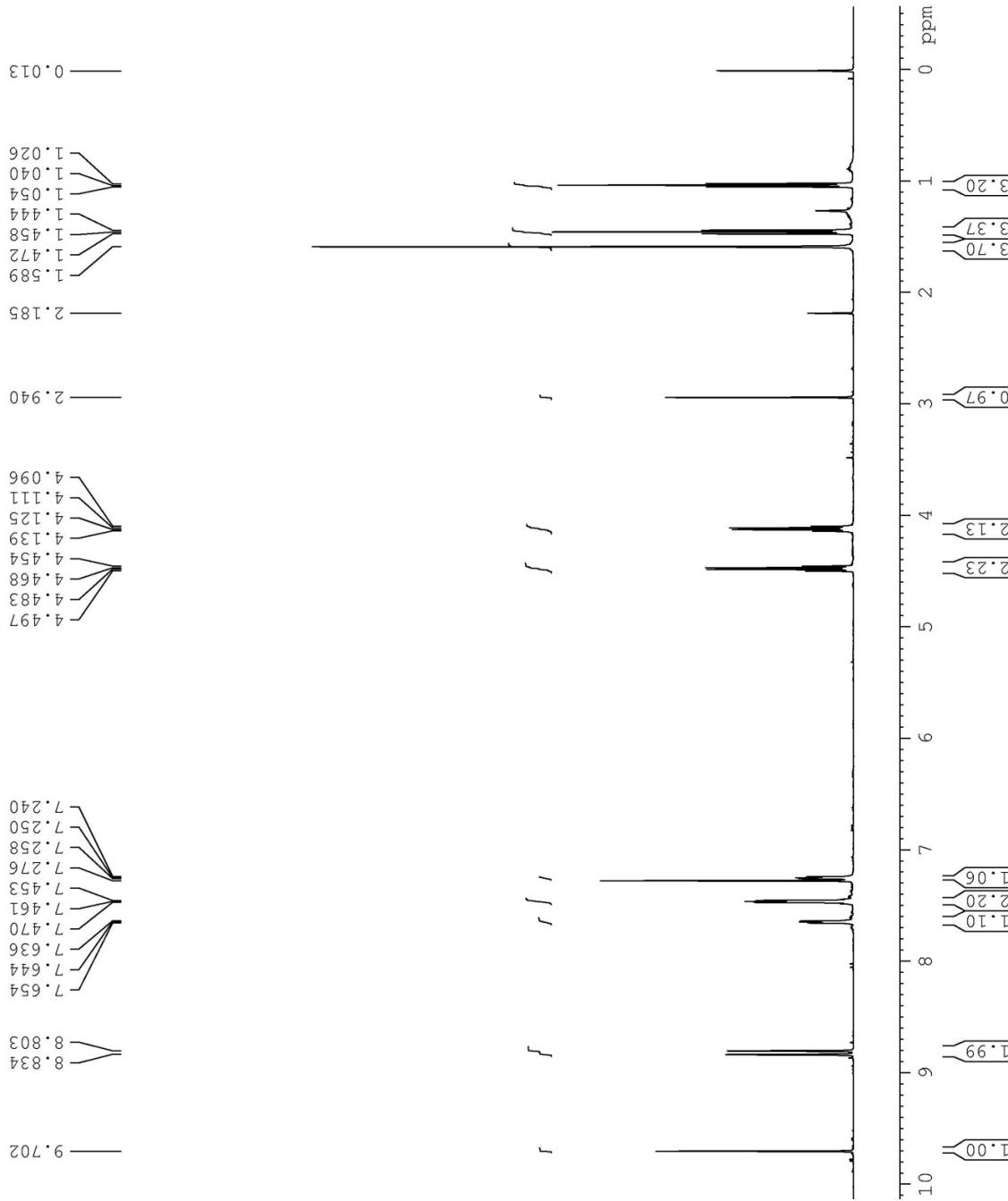
WDW

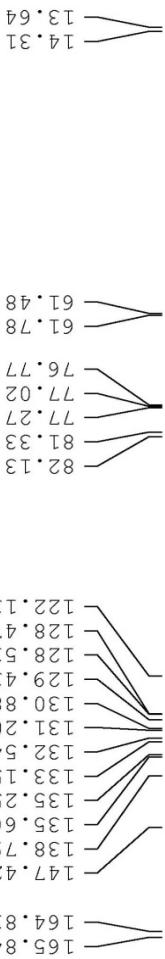
SSB 0

LB 0.30 Hz

GB 0

PC 1.00





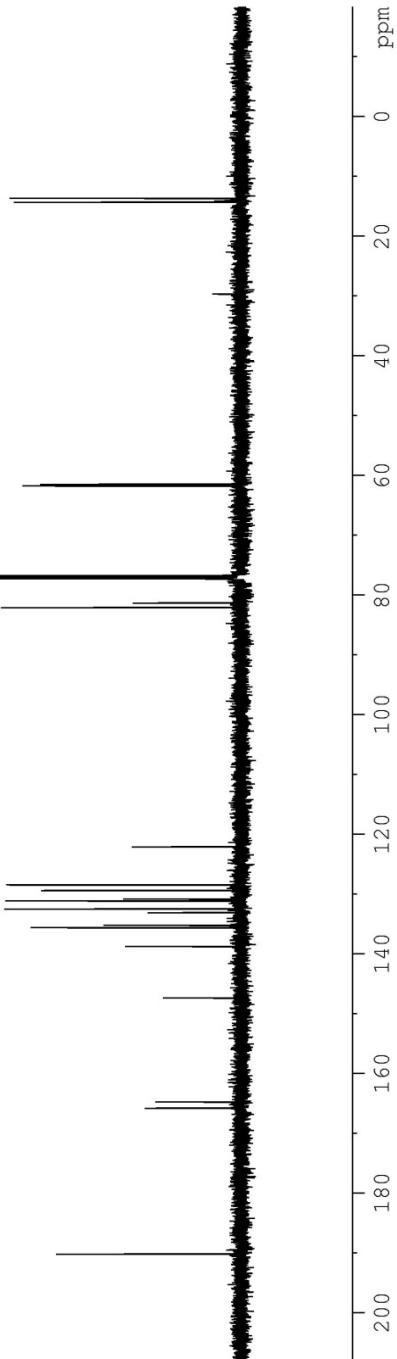
Current Data Parameters
NAME CS-436
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130716
Time 16.34
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgr930
TD 65536
SOLVENT CDCl3
NS 219
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 32800
DW 16.800 usec
DE 6.00 usec
TE 299.9 K
D1 2.0000000 sec
d11 0.3000000 sec
DETA 1.8999998 sec
TDO 1

===== CHANNEL f1 =====
NUC1 13C
P1 10.94 usec
PL1 3.00 dB
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL1.2 17.48 dB
PL1.3 18.00 dB
PL2 1.00 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577390 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



```

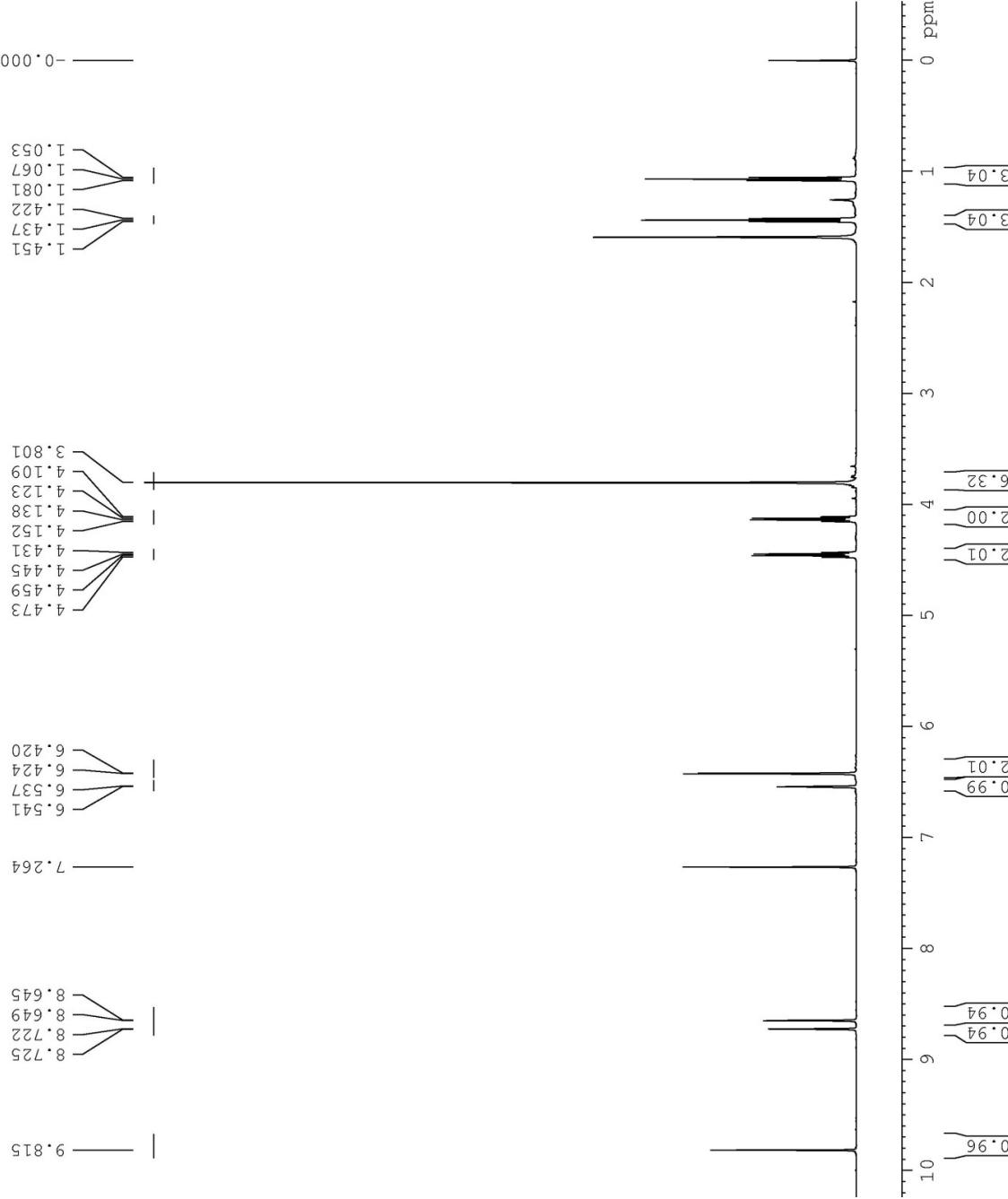
Current Data Parameters
  NAME CS-851-1
  EXPNO 1
  PROCNO 1

F2 - Acquisition Parameter
Date_ 20140730
Time_ 14.00
INSTRUM spect
PROBID 5 mm PABBI 1H/
PULPROG zg30
TD 65336
SOLVENT CDC13
NS 2
DS 2
SWH 10330 578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 se
RG 287
DW 48.400 us
DE 6.00 us
TE 297.2 K
D1 1.0000000 se
TDO 1

=====
CHANNEL f1 =====
NUC1 1H
P1 7.20 us
PL1 1.00 dB
SF01 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.13300005 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

```



14.30
13.73

55.43
55.47
61.77
77.28
77.02
76.77

100.37
101.94
105.50
107.36
107.96

130.48
130.65
133.68
134.77
134.97
137.02
148.35

161.06
164.78
166.62

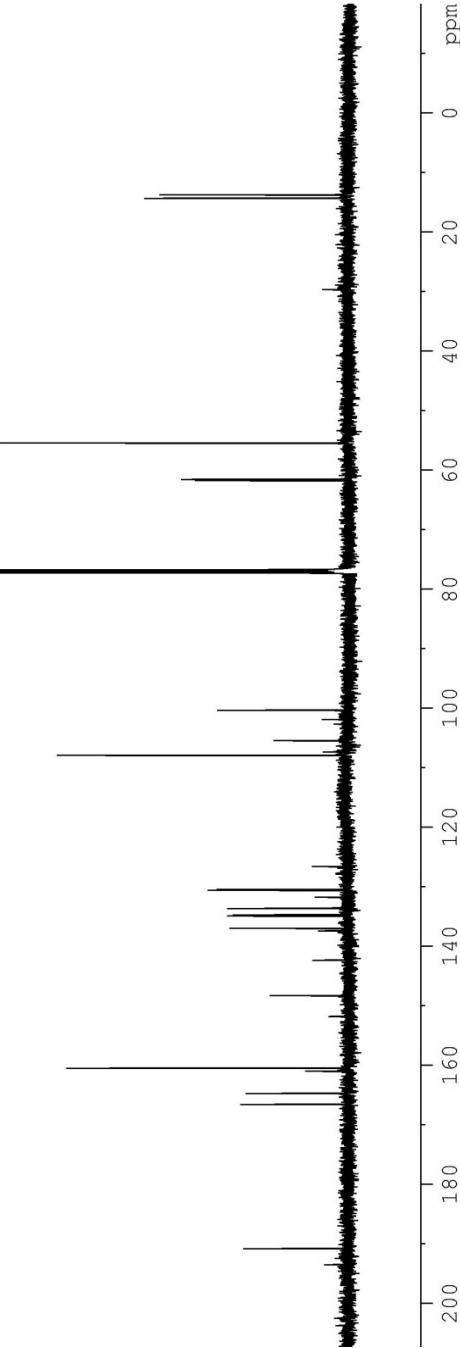
190.87

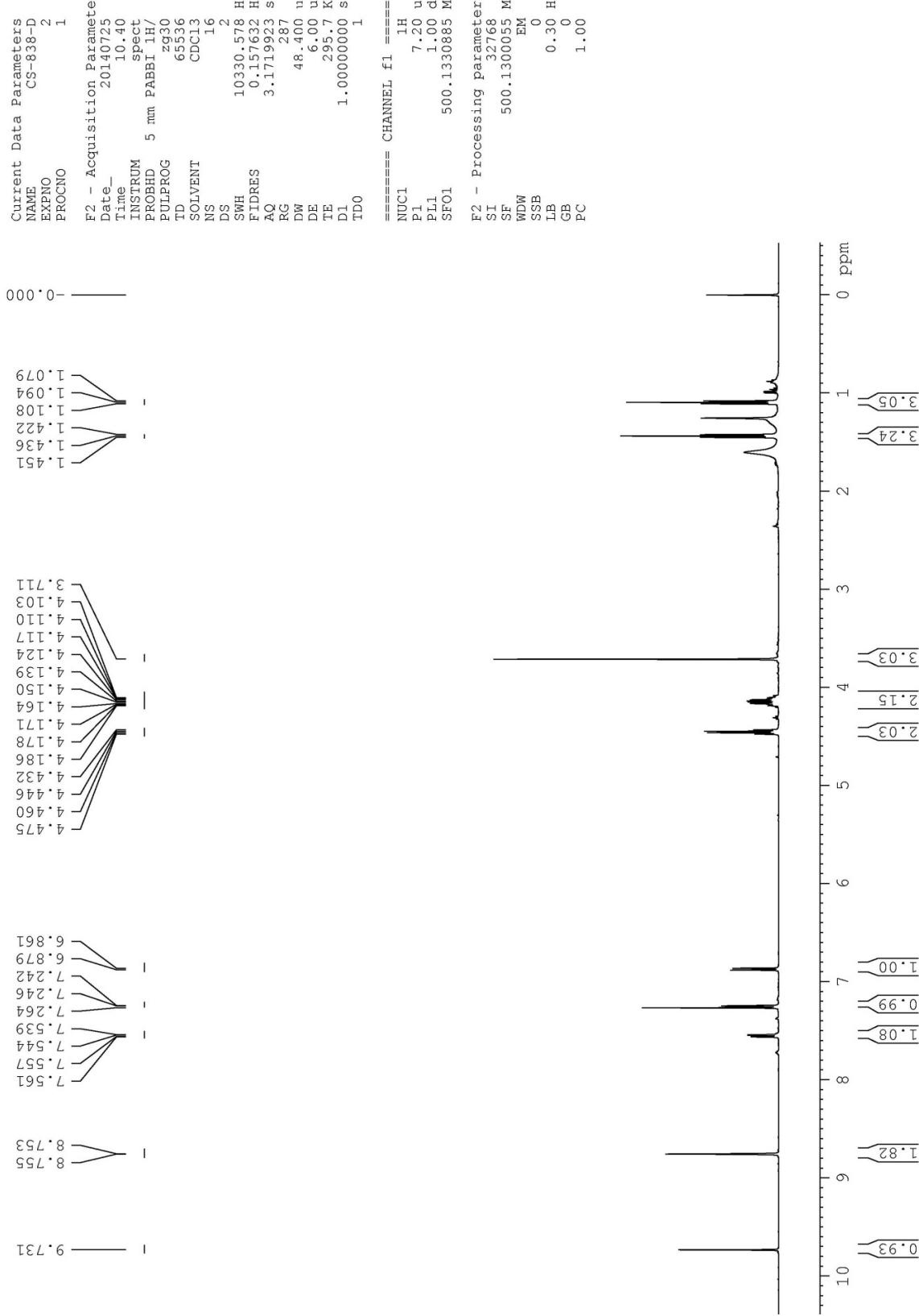
Current Data Parameters
NAME CS-56-1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20131014
Time_ 16.56
INSTRUM spect
PROBID 5 mm PABBI 1H/
PULPROG zppg30
TD 65336
SOLVENT C6D13
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 32800
DW 16.800 usec
DE 6.00 usec
TE 296.9 K
D1 2.0000000 sec
d11 0.0300000 sec
DELTa 1.8999998 sec
TDO 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.40 usec
PL1 125.7703643 MHz
SFO1
===== CHANNEL f2 =====
CPDPFG2 wait:16
NUC2 1H
PCPD2 80.00 usec
PL12 21.92 dB
PL13 120.00 dB
PL2 1.00 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





13.80
14.32

55.84
61.50
61.78

77.28
77.02
76.77

112.15
112.80
112.38
130.80
131.09
132.95
133.03
135.53
135.52
143.87
155.78
164.78
166.01

190.58

Current Data Parameters
NAME CS-558
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20131015
Time 12.30
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgppg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 29751.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 32800
DW 16.800 usec
DE 6.00 usec
TE 296.6 K
D1 2.0000000 sec
d11 0.03000000 sec
DELTAt 1.8999998 sec
TDO 1

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

NUC1 13C
P1 12.40 usec
PL1 125.7703643 MHz
SFO1 1

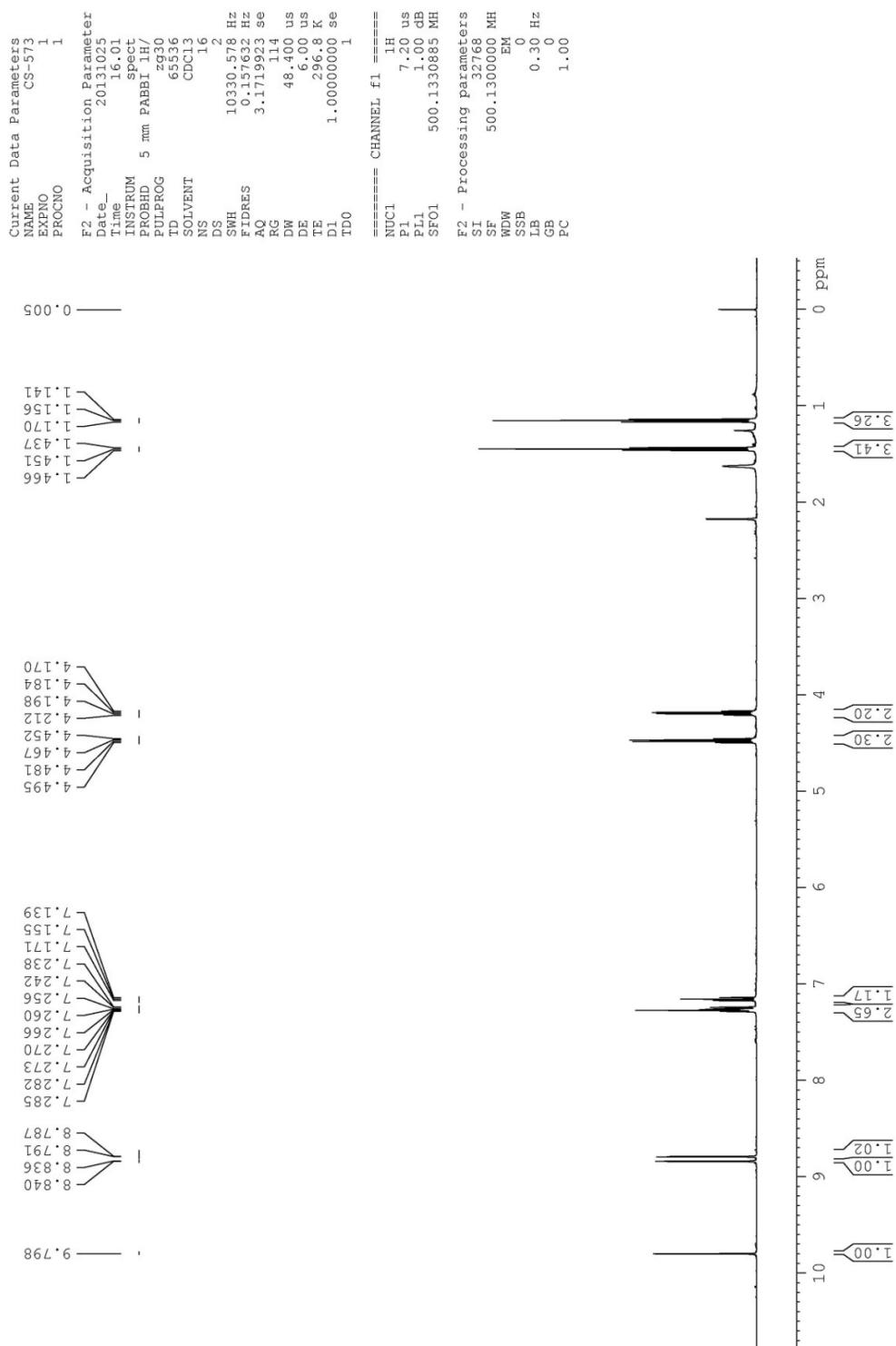
===== CHANNEL f2 =====

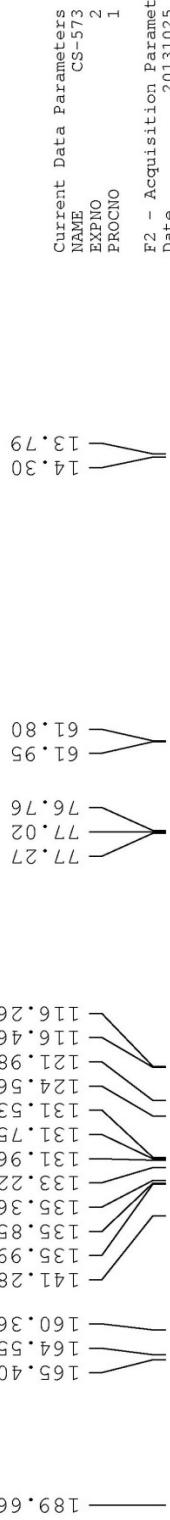
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 21.92 dB
PL13 120.00 dB
PL2 1.00 dB
SFO2 500.1320005 MHz

F2 - Processing parameters

SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40







Current Data Parameters
NAME CS-573
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20131025
Time 16.09
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgppg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 29751.904 Hz
FIDRES 0.454131 Hz
AQ 1.101058 sec
RG 32800
DW 16.800 usec
DE 6.00 usec
TE 296.9 K
D1 2.0000000 sec
q1 0.0300000 sec
DELTA A 1.89999998 sec
TD0 1

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

NUC1 13C
P1 12.40 usec
PL1 125.7703643 MHz
SFO1

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

CPDPFG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 21.32 dB
PL13 120.00 dB
PL12 1.00 dB
SFO2 500.1320005 MHz

F2 - Processing parameters

SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



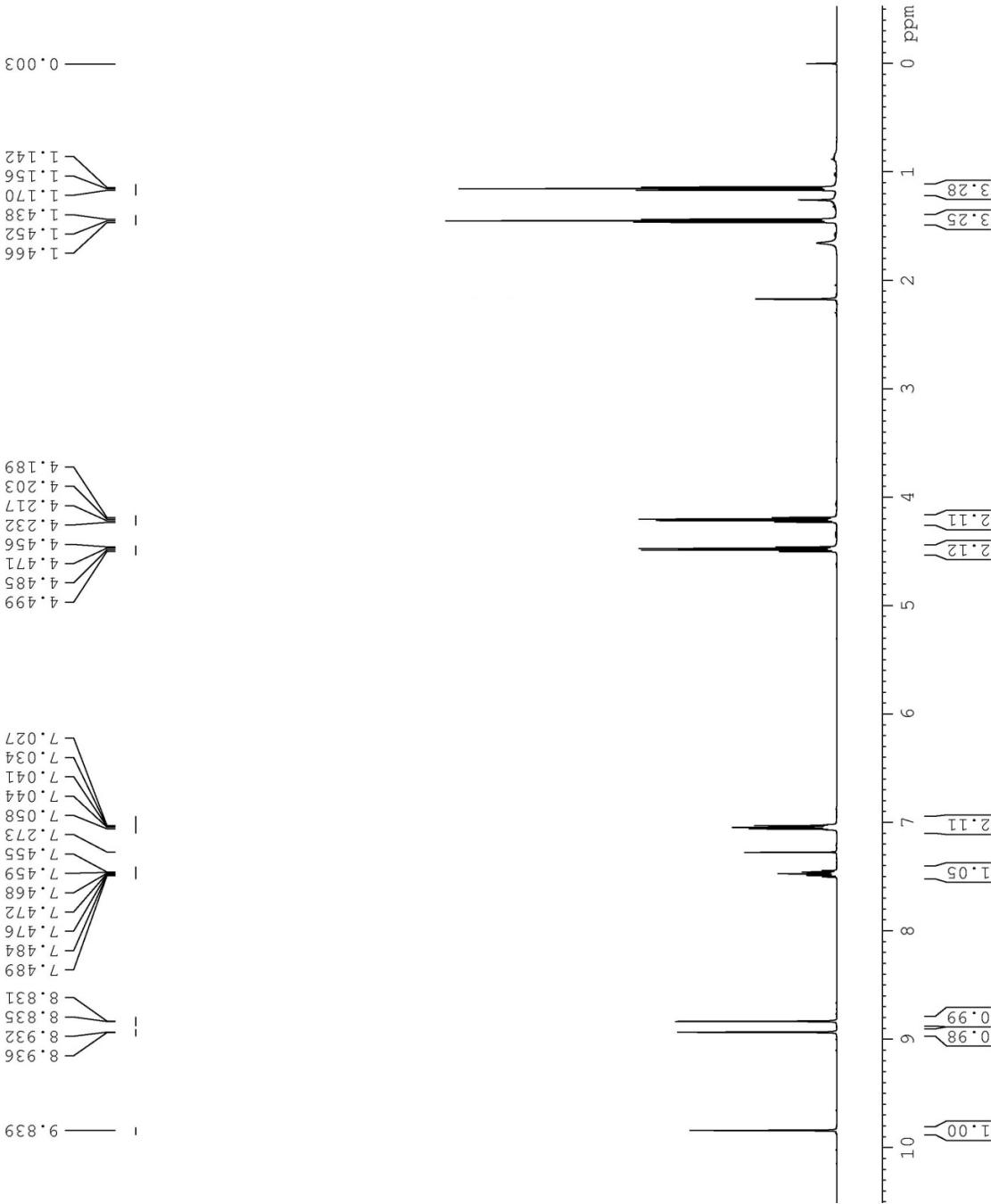
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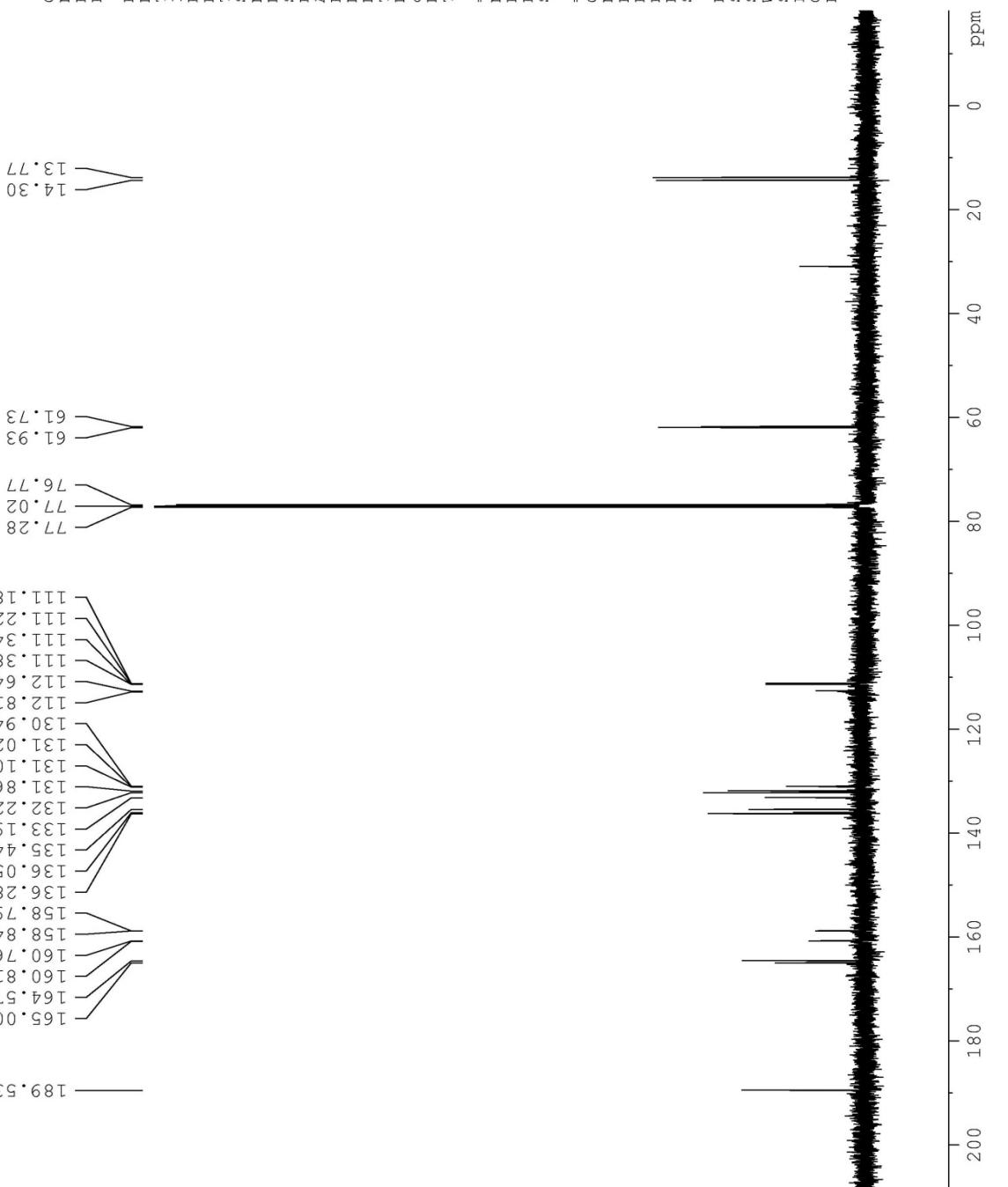
Current Data Parameters          F2 - Acquisition Parameter
NAME      CS-572             Date... 20131026
EXPHD    1                   TIME   12..33
INSTRUM spect              PROPHD  5 mm PABBI 1H/
PULPROG z930               TD     65536
          CDC13
SOLVENT 16
DS        2
SWH      10330.578 Hz
FIDRES  0.157532 Hz
        3.1719923 sec
AQ       90.5
RG       48.100 us
DW       6.00  us
DE       296.6 K
TE       1.0000000 s
D1       1
TDO      =====

===== CHANNEL f1 =====
NUC1      1H
P1        7.20 us
PLL      1.00 GB
SFO1     500.1330885 MH

F2 - Processing parameters
SI        32768
SF       500.1300000 MH

```



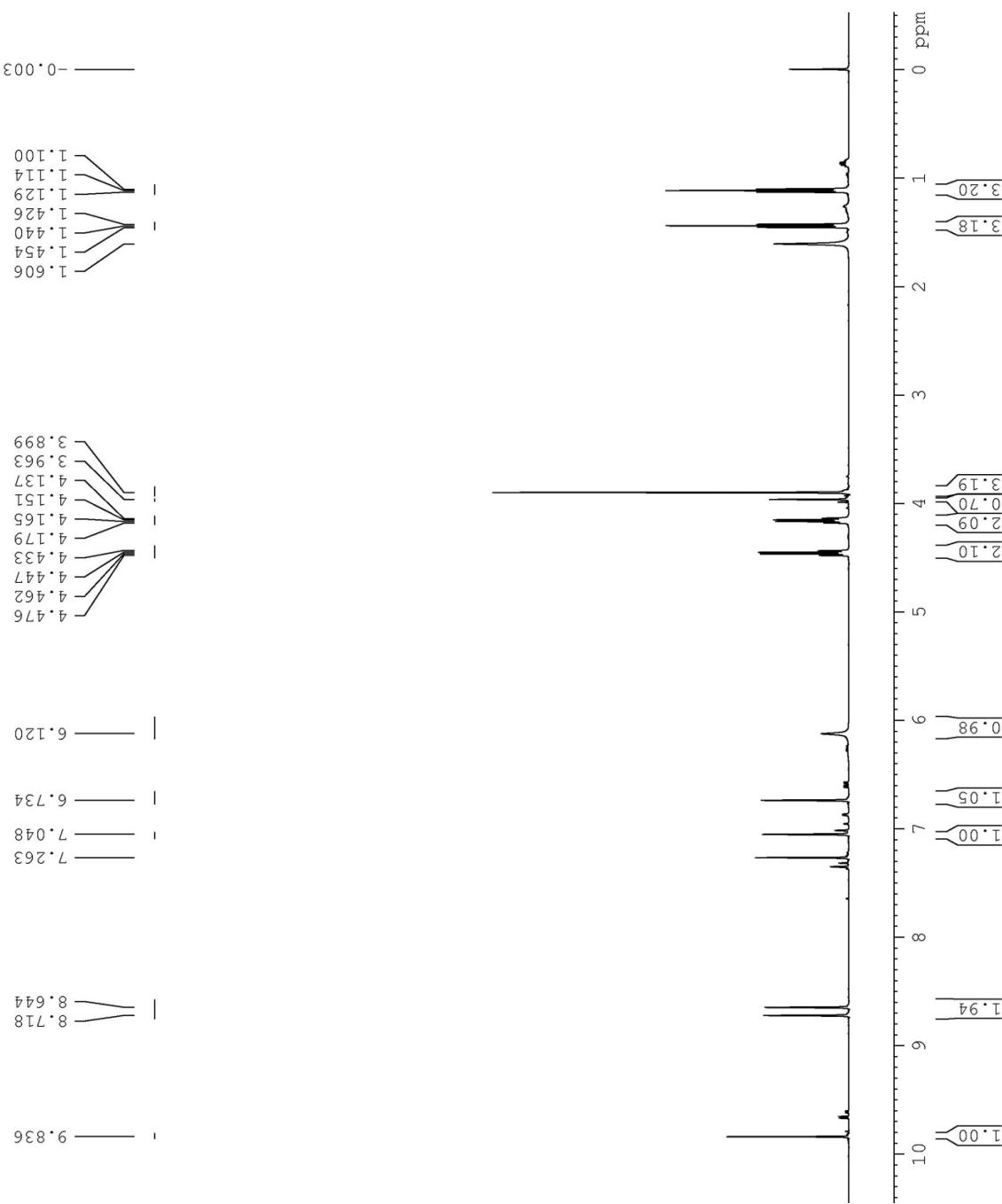


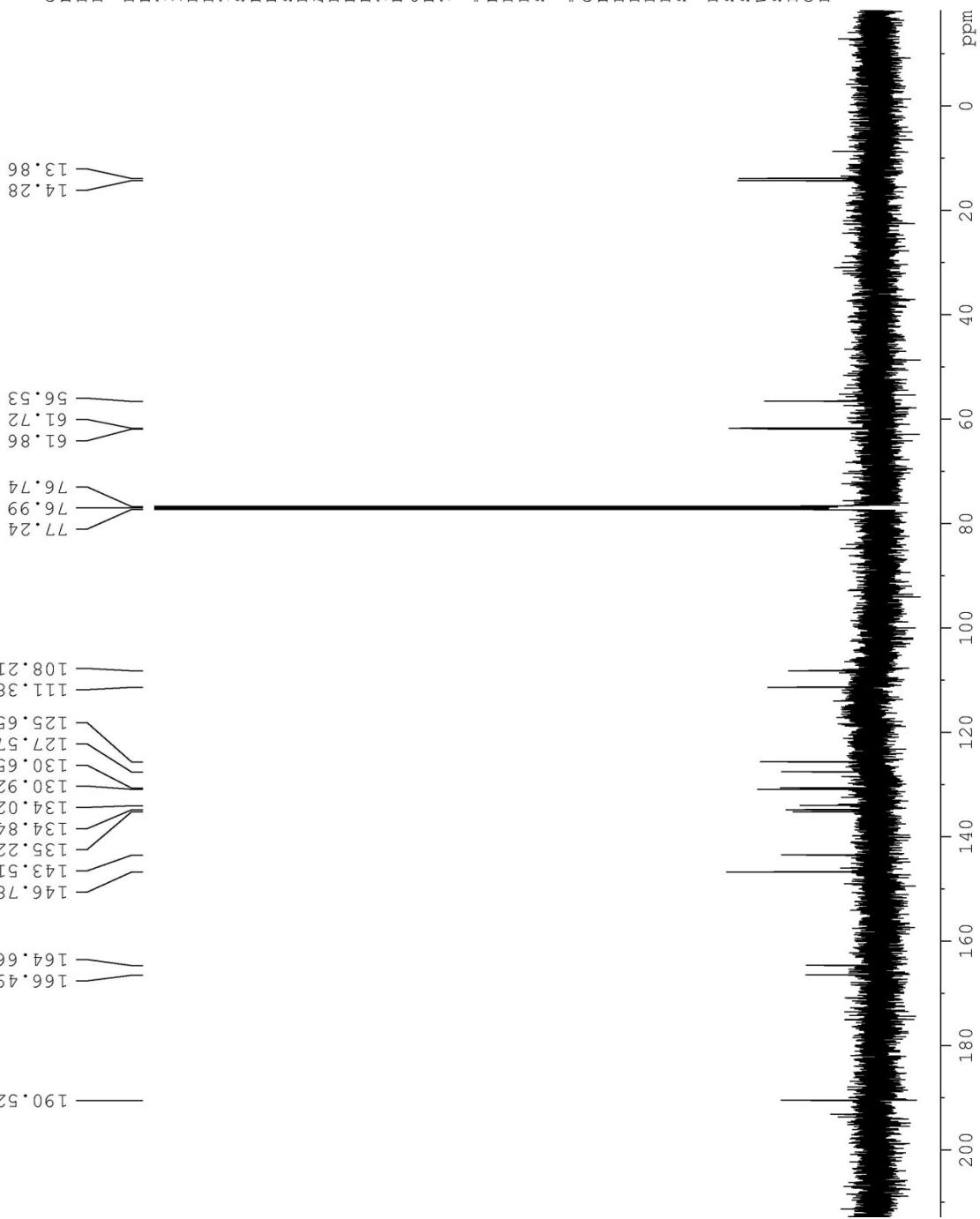
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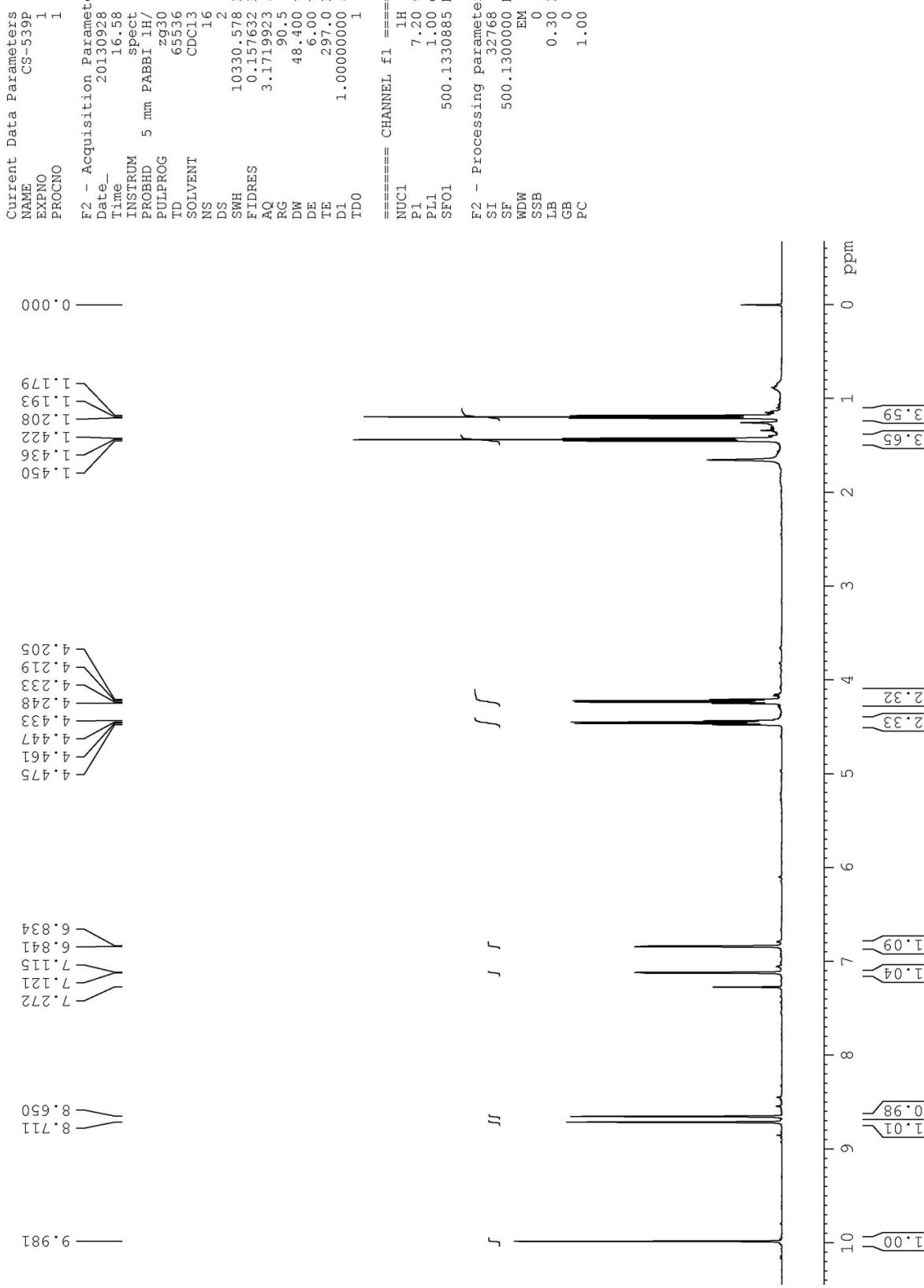
Current Data Parameters          F2 - Acquisition Parameter
NAME      CS-630-3             Date_    20131206
EXPNO     1                     TIMEFRM  18-18
PROCNO    1                     INSTRUM  spect
                           5 mm PABBI 1H/
                           PULPROG zg930
                           TD       65536
                           SOLVENT CD13
                           NS       16
                           DS       2
                           SWH    10330.578 Hz
                           FIDRES 0.157632 Hz
                           AO      3.1719943 s
                           RG      228
                           DW      48.400 us
                           DE      6.000 us
                           TE      297.3 K
                           D1      1.0000000 se
                           TDO      1

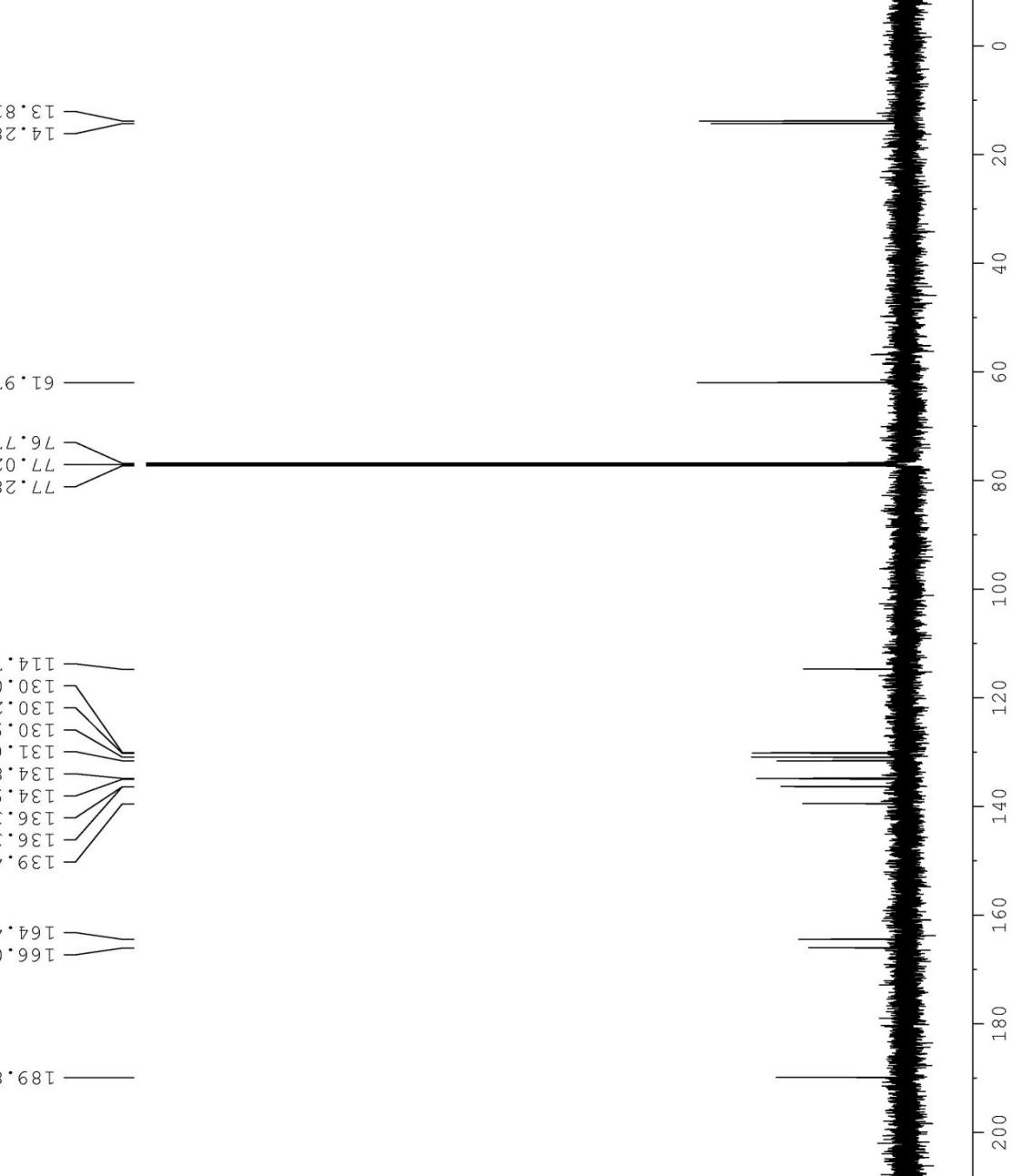
===== CHANNEL f1 =====
NUC1      1H
P1        7.20  us
PLL      1.00  dB
SF01    500.133085 MHz
                           S1      32768
                           SF      500.130000 MHz
                           WW      EM
                           SSB      0
                           LB      0.30 Hz
                           GB      0
                           PC      1.00

```









Current Data Parameters
NAME CS-539P
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130928
Time_ 17.13
INSTRUM spect
PROBID 5 mm PABBI-1H/
PULPROG zgppg30
TD 65536
SOLVENT CDCl3
NS 102
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 32800
DW 16.800 usec
DE 6.00 usec
TE 297.1 K
D1 2.0000000 sec
d11 0.0300000 sec
DELTAT 1.8999998 sec
TD0 1

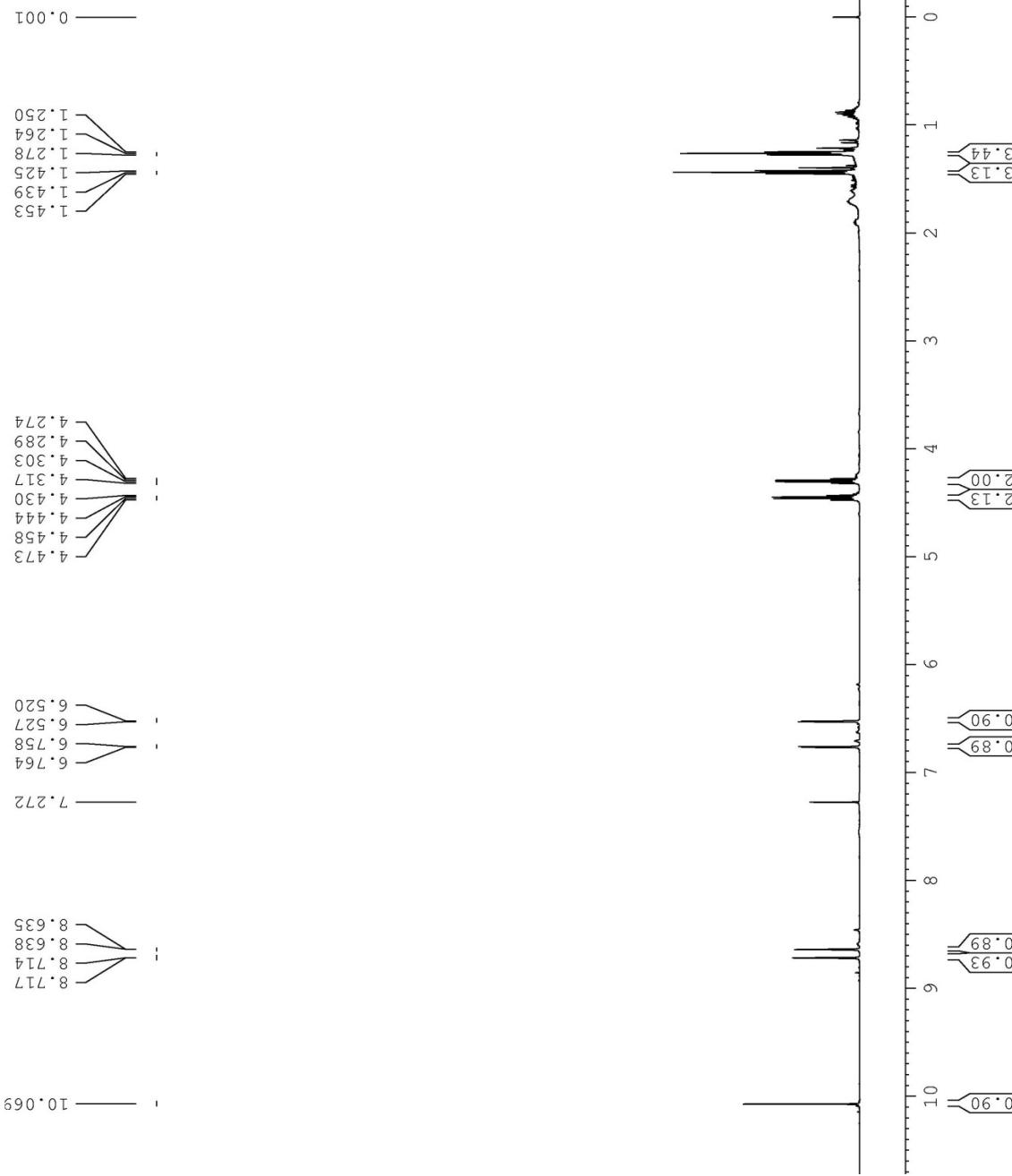
===== CHANNEL f1 =====
NUC1 13C
P1 12.40 usec
PL1 -2.00 dB
SFO1 125.7703643 MHz
===== CHANNEL f2 =====
CPDPG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL1.2 21.92 dB
PL1.3 120.00 dB
PL2 1.00 dB
SFO2 500.1320005 MHz

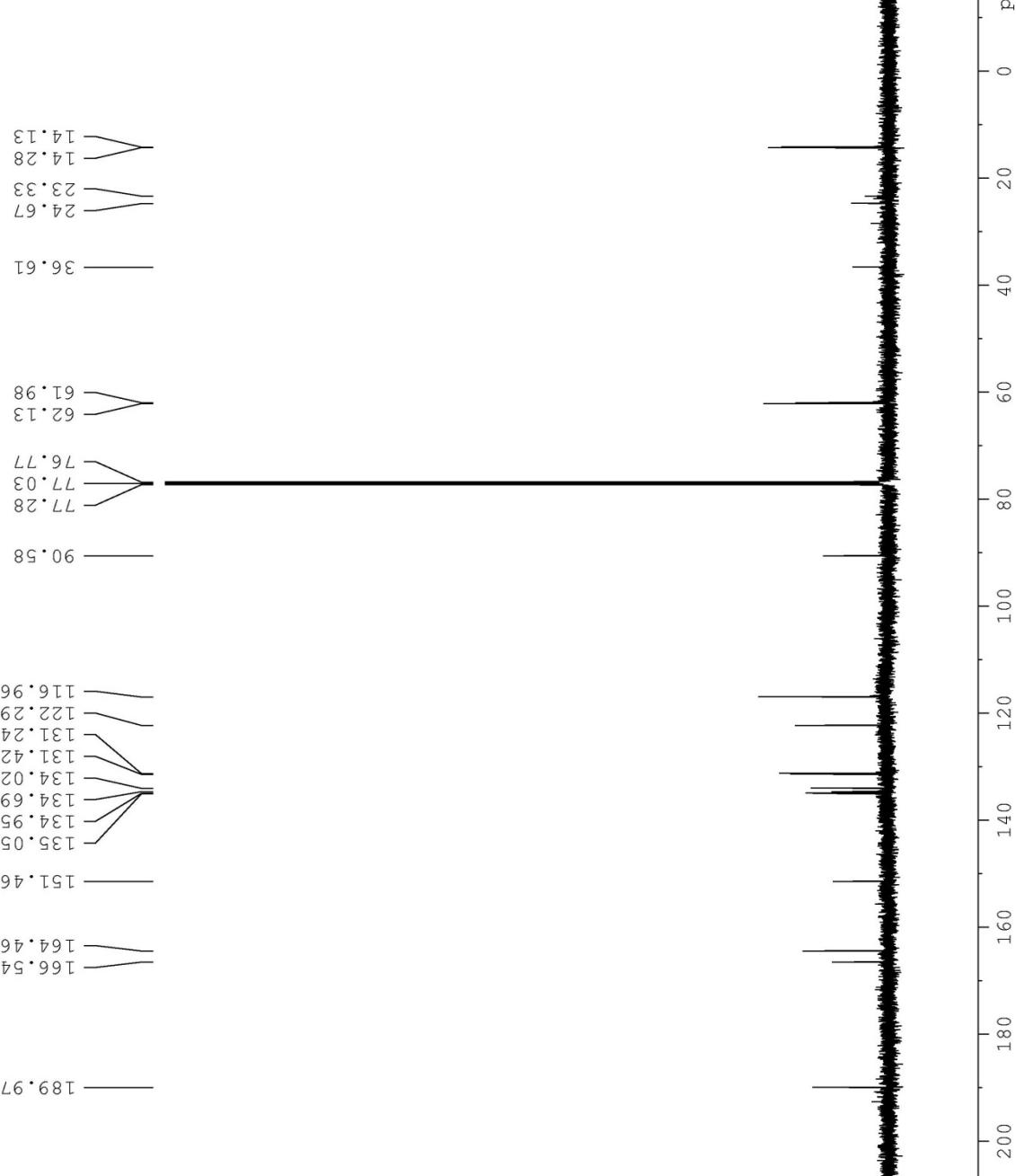
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Current Data Parameters
NAME CS-543
EXPNO 1
PROCNO

F2 - Acquisition Parameter
Date_ 20131002
Time_ 10.39
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG Z930
TD 65536
SCVOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 se
RG 90.5
DW 48.400 us
DE 6.00 us
TE 295.7 K
D1 1.0000000 se
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 7.20 us
PL1 1.00 dB
SF01 500.1330885 MH
F2 - Processing parameters
SI 32768
SF 500.1300000 MH
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





Current NAME	Data EXPNO	Parameters PROCCNO	NAME CS-853-H
			1

F2 - Acquisition Parameters

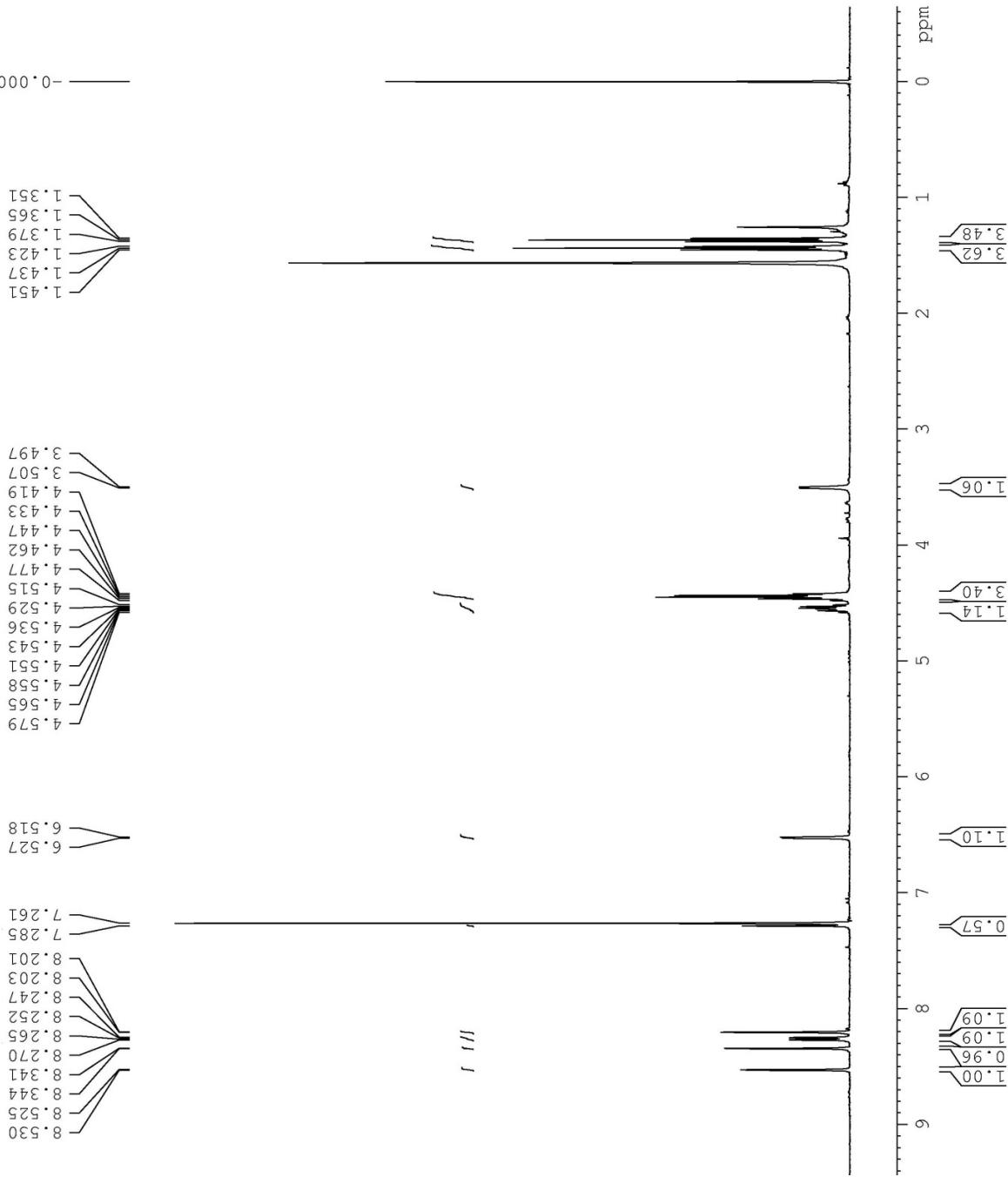
Date_	20140731
Time	17.59
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	zg30
TD	65536
SOLVENT	CDC13
NS	16
DS	2

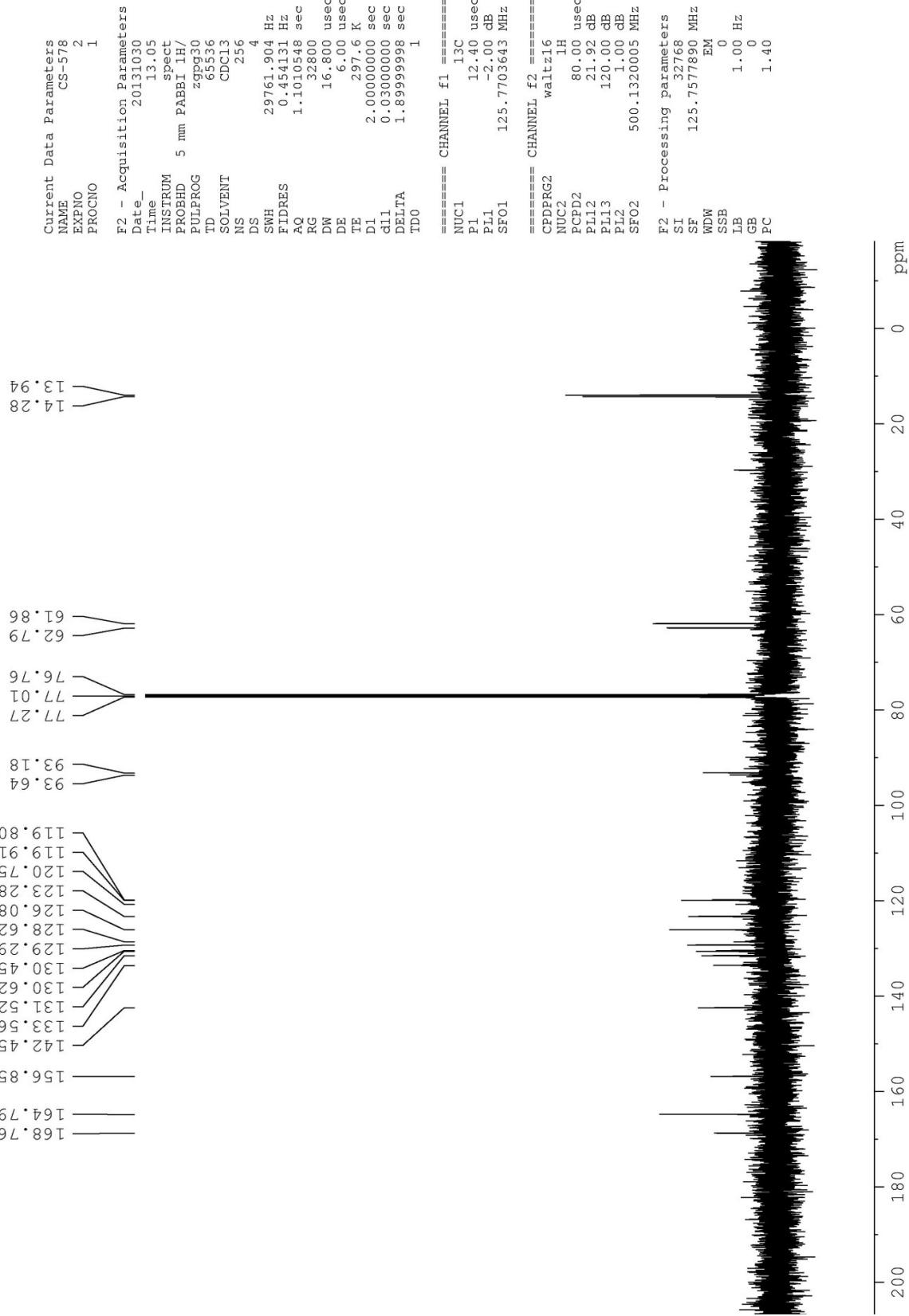
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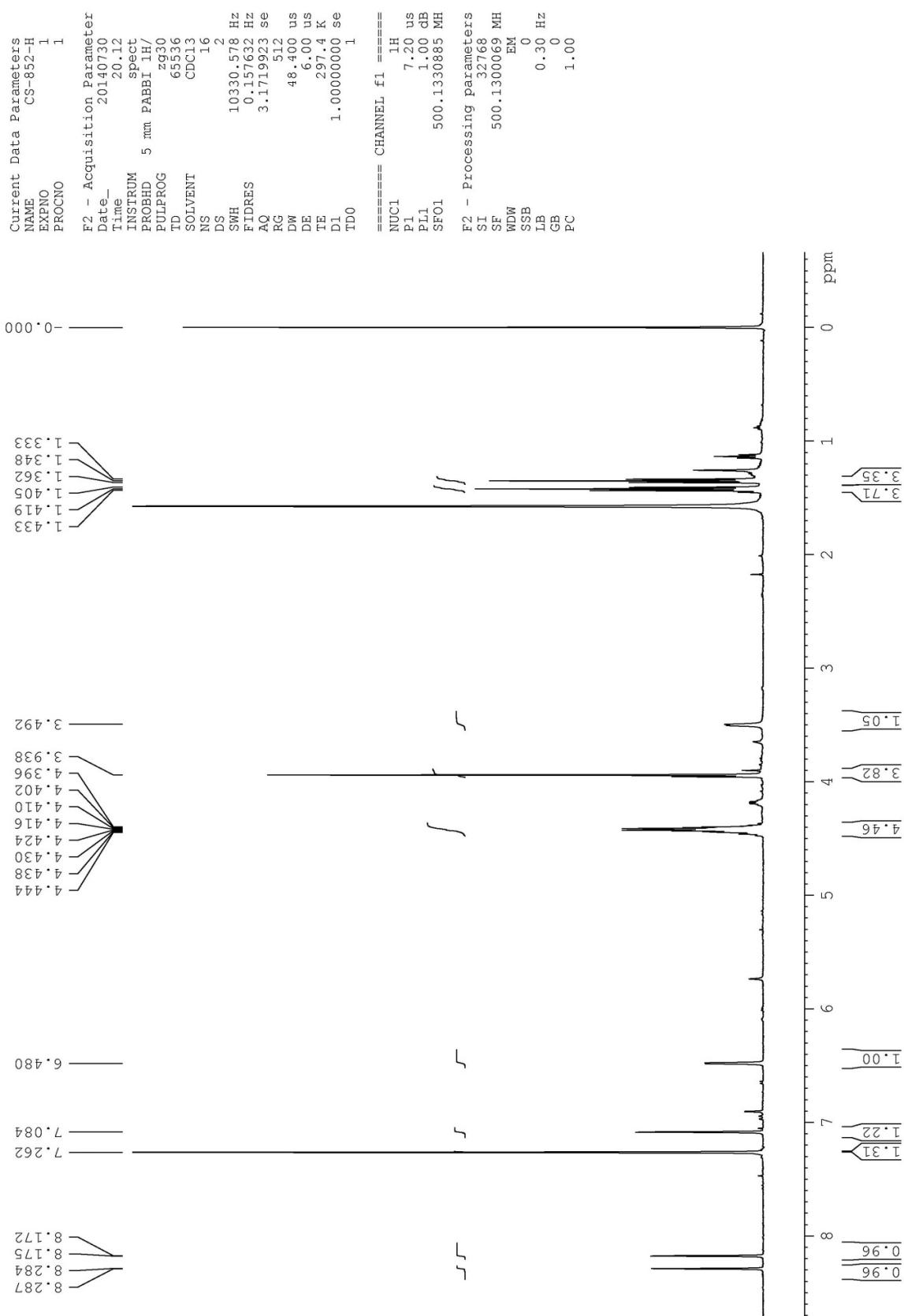
===== CHANNEL f1 =====
NUC1          7.20 us
P1           1.00 dB
PLL          500.1330885 MH
SFO1

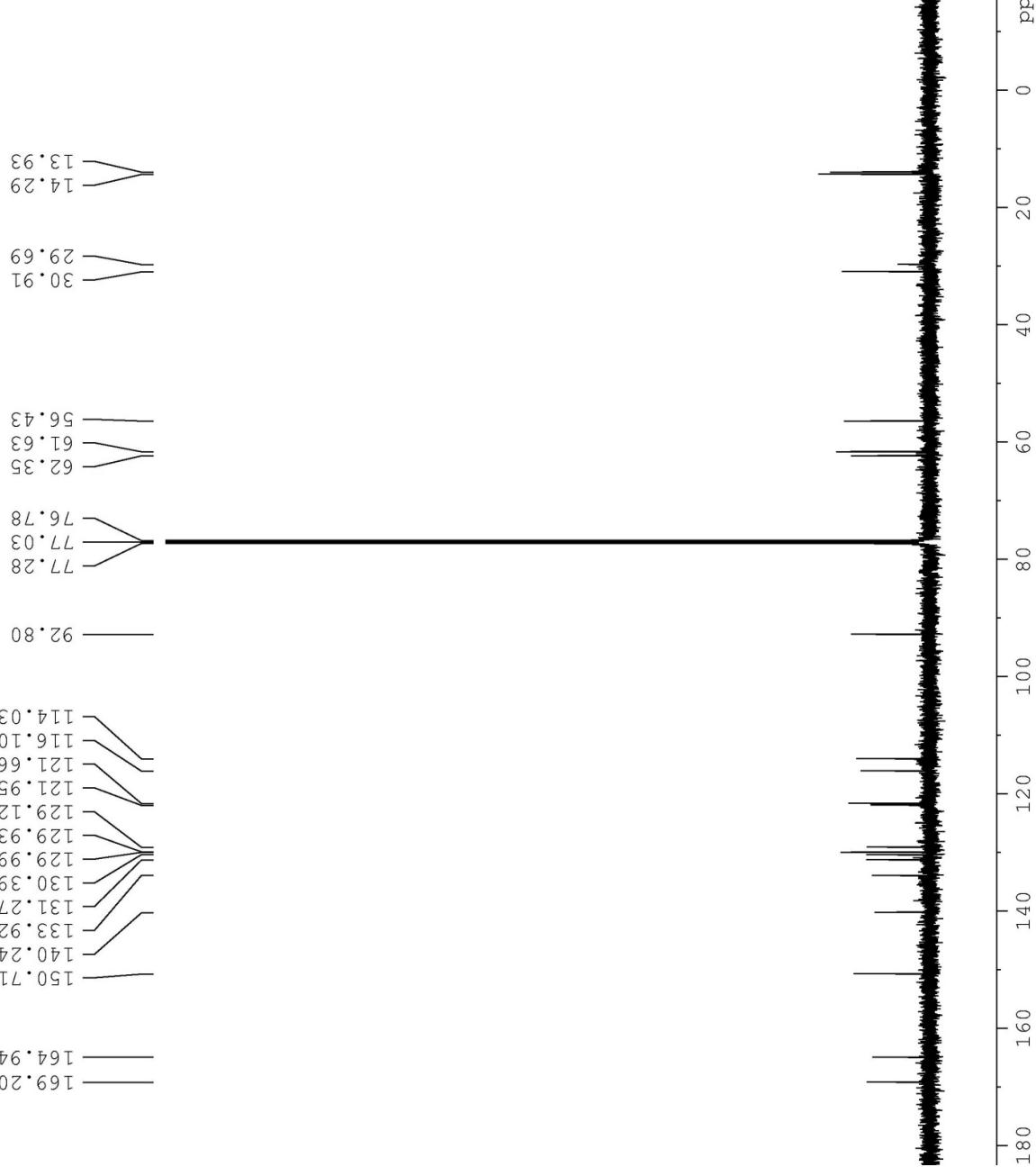
F2 - Processing parameters
SI            32768
SF          500.1300001 MH
WDW             EM
SSB              0
LB              0.30 Hz
GB              0
PC              0

```









Current Data Parameters
NAME CS-636-1
EXPNO 3
PROCNO 1

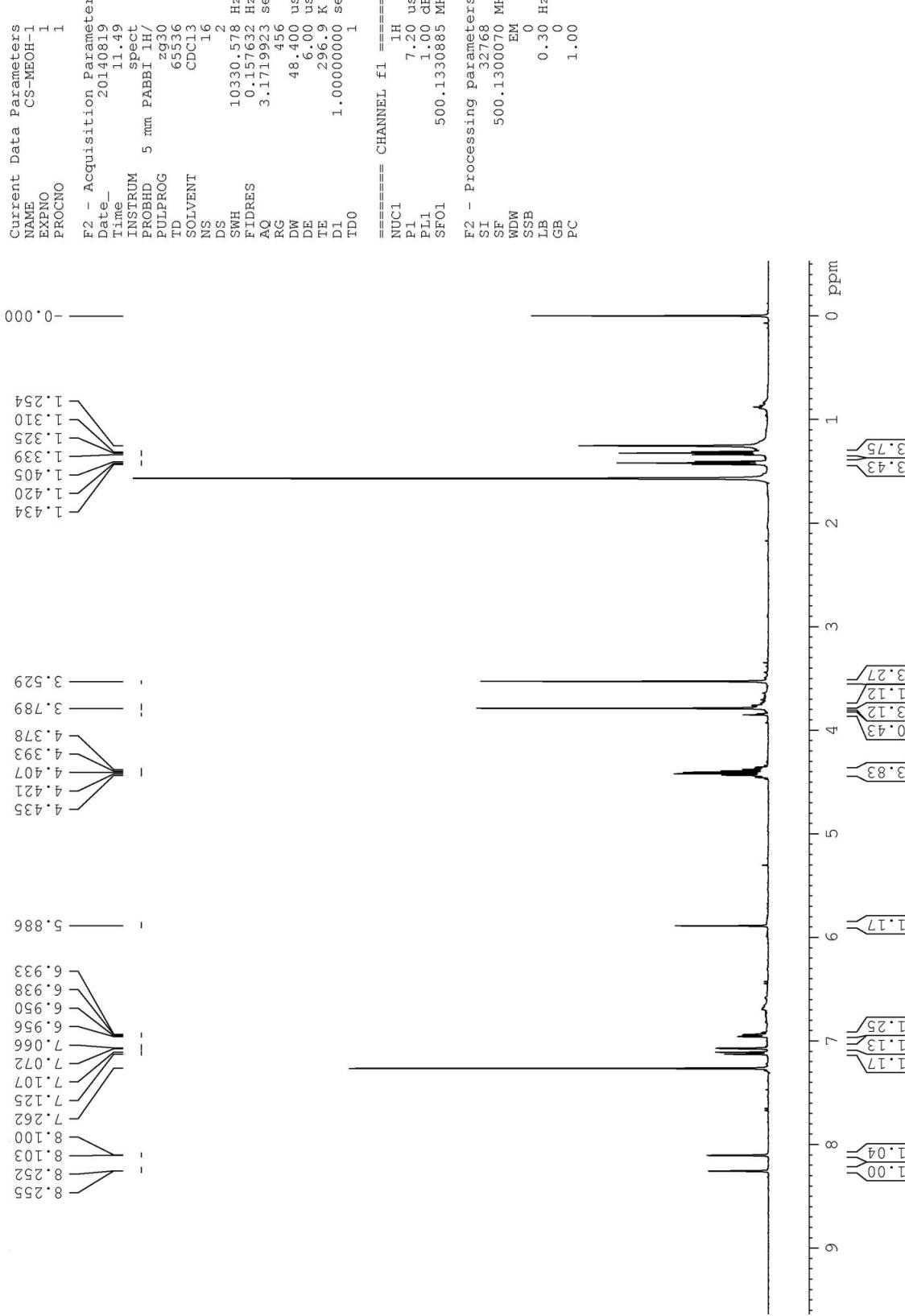
F2 - Acquisition Parameters
Date_ 20131220
Time 17.04

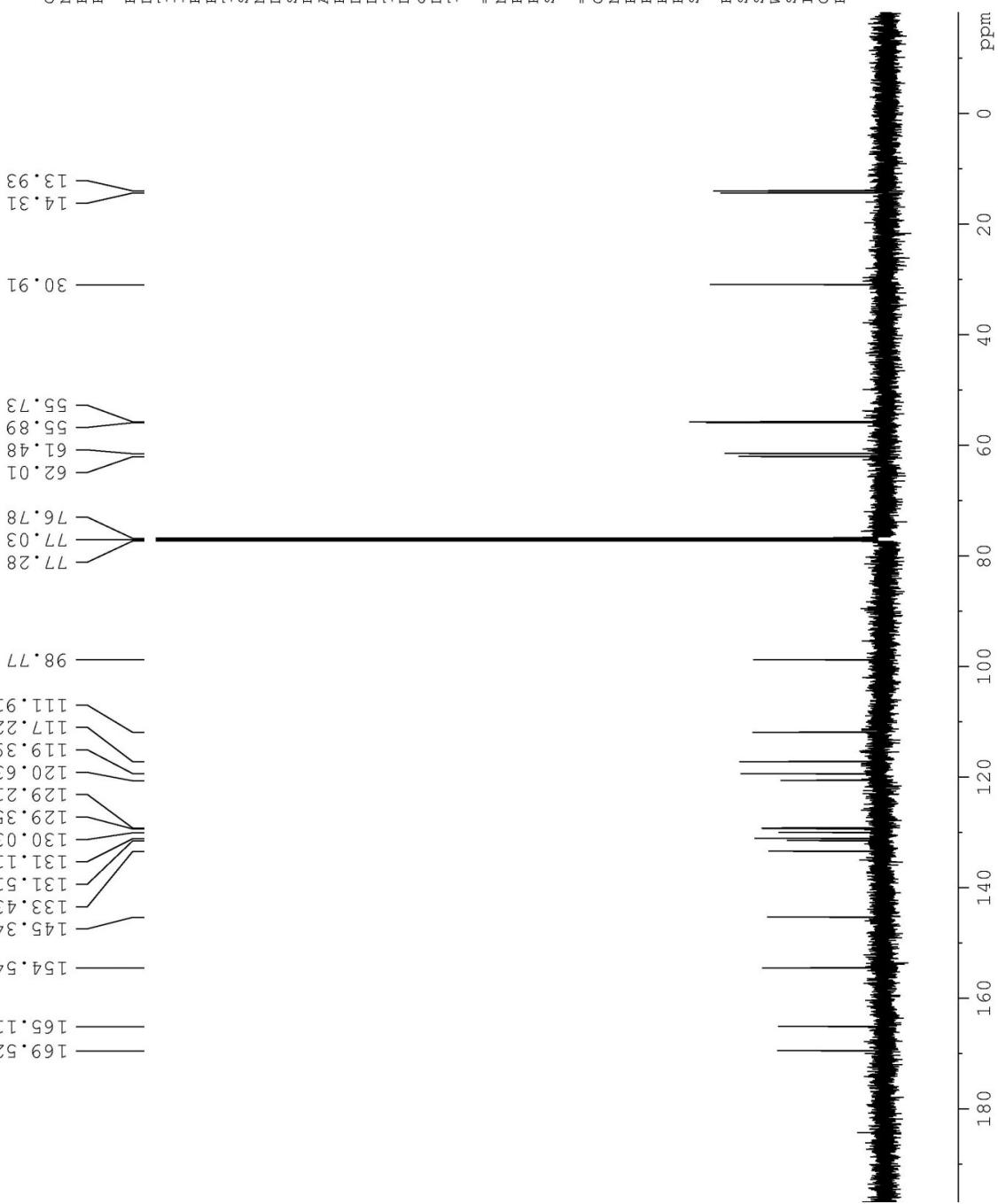
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 456
DS 4
SWH 29761.904 Hz
FIDRES 0.154131 Hz
AQ 1.1010548 sec
RG 32800
DW 16.800 usec
DE 6.00 usec
TE 297.4 K
D1 2.0000000 sec
d11 0.03000000 sec
DELT A 1.89999998 sec
TDO 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.40 usec
PL1 125.7703643 MHz
SFO1

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL1 21.92 dB
PL13 120.00 dB
PL2 1.00 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



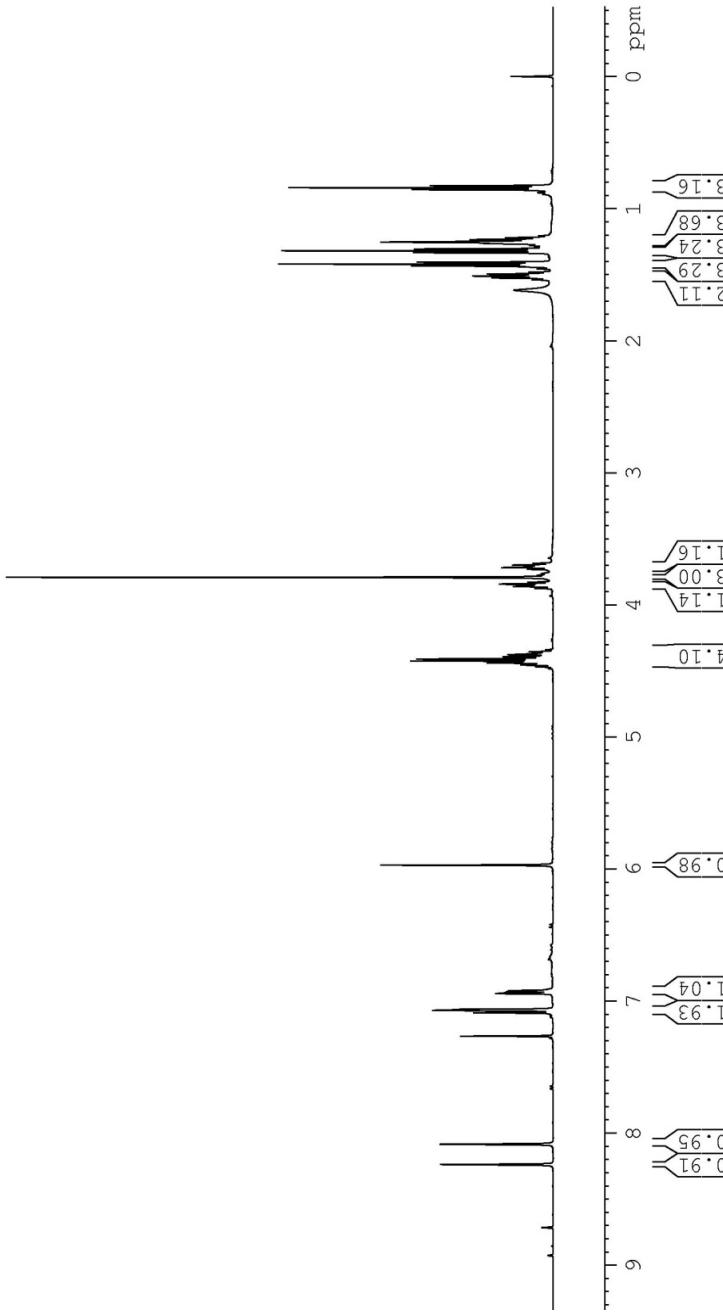


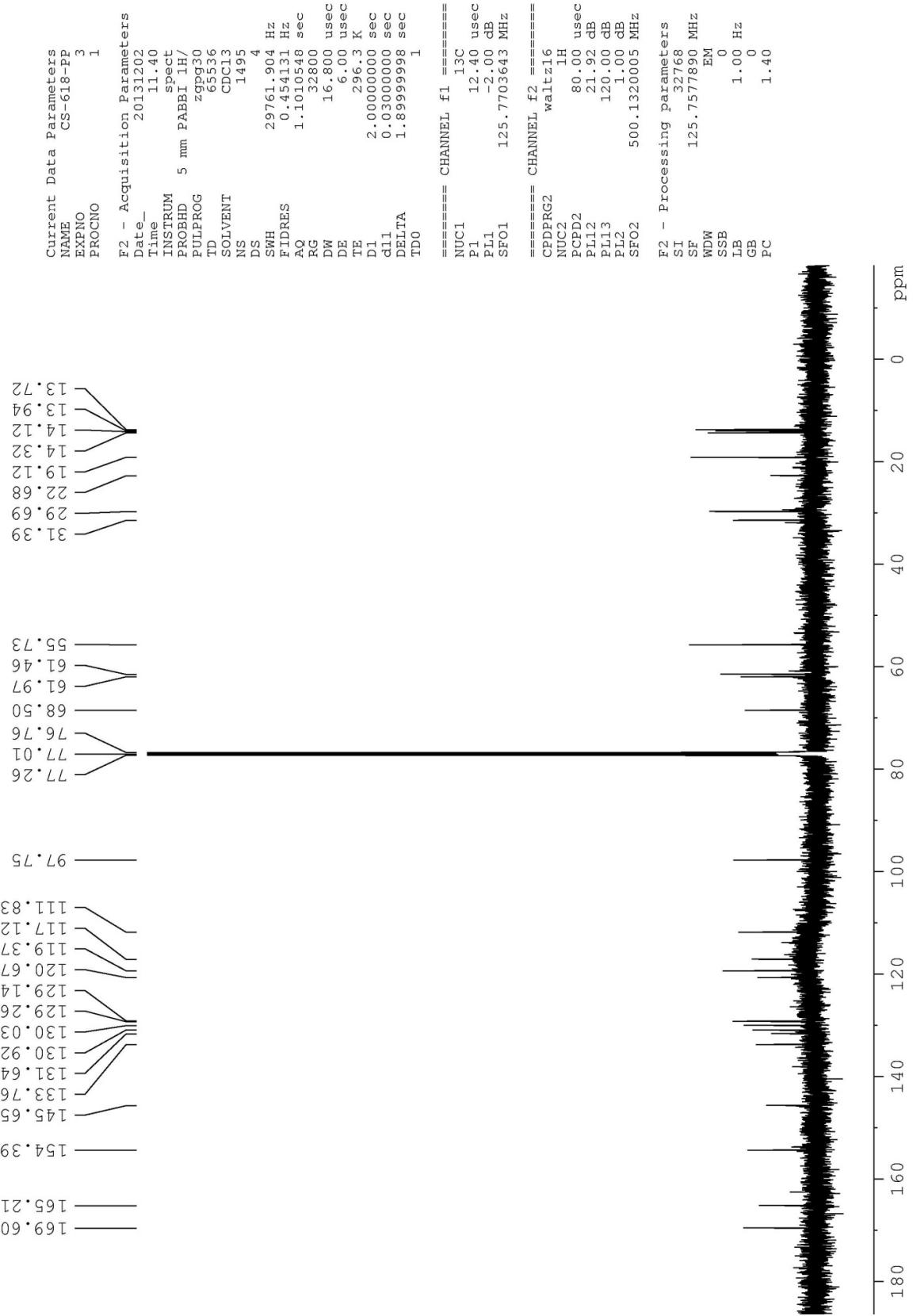
Current Data Parameters

NAME	CS-BUOH
EXPNO	1
PROCNO	1

F2 - Acquisition Parameter

Date_	20140819
Time	11.07
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	zg930
TD	65536
SOLVENT	CDCl3
NS	16
DS	1
SWH	10330.578
FIDRES	0.157632
AQ	3.1719923
RG	80.6
DW	48.400
DE	6.00
TE	296.5
D1	1.0000000000000001
TDD	1



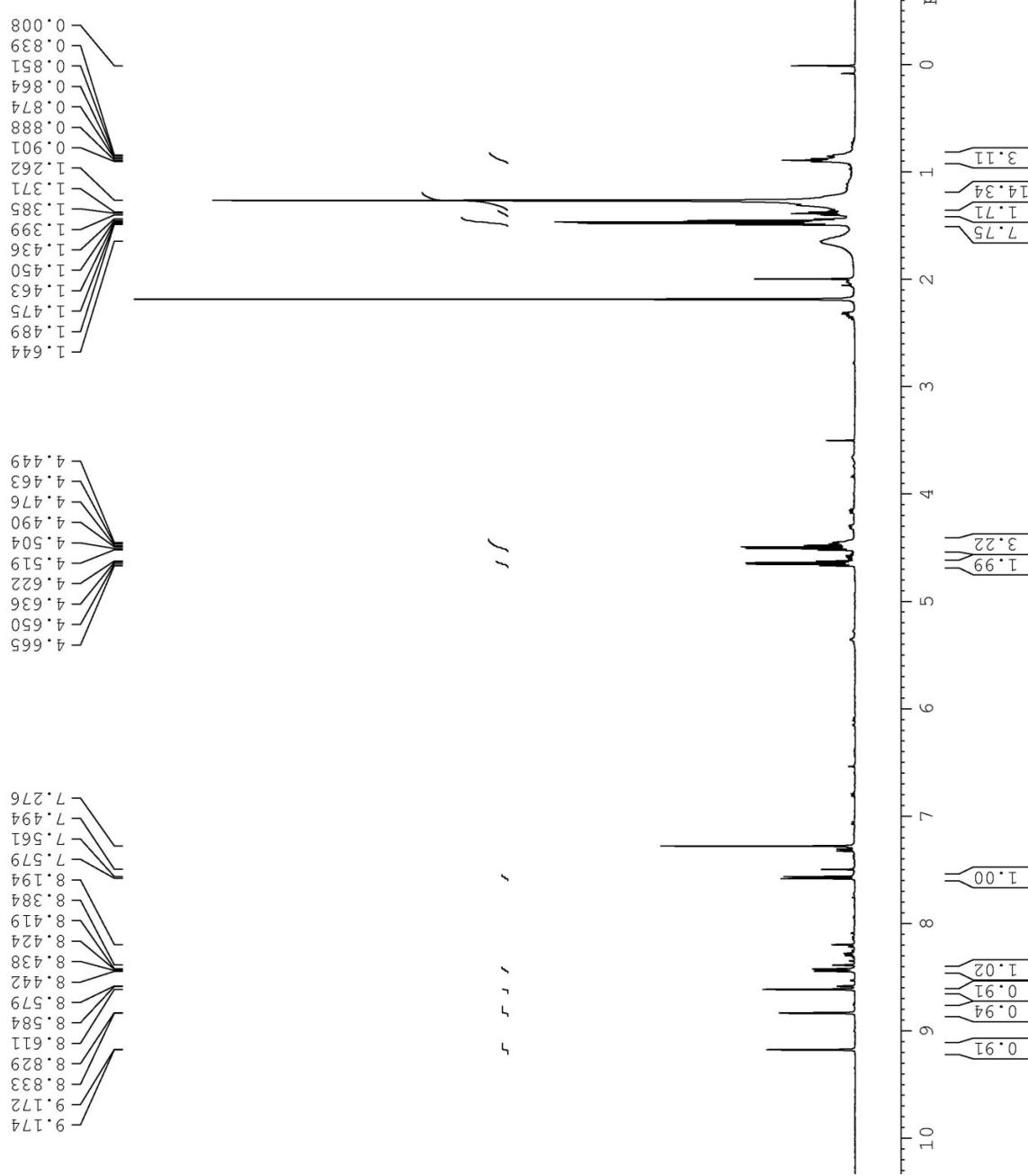


Current Data Parameters	NAME	CS-649
	EXPNO	1
	PROCNO	1

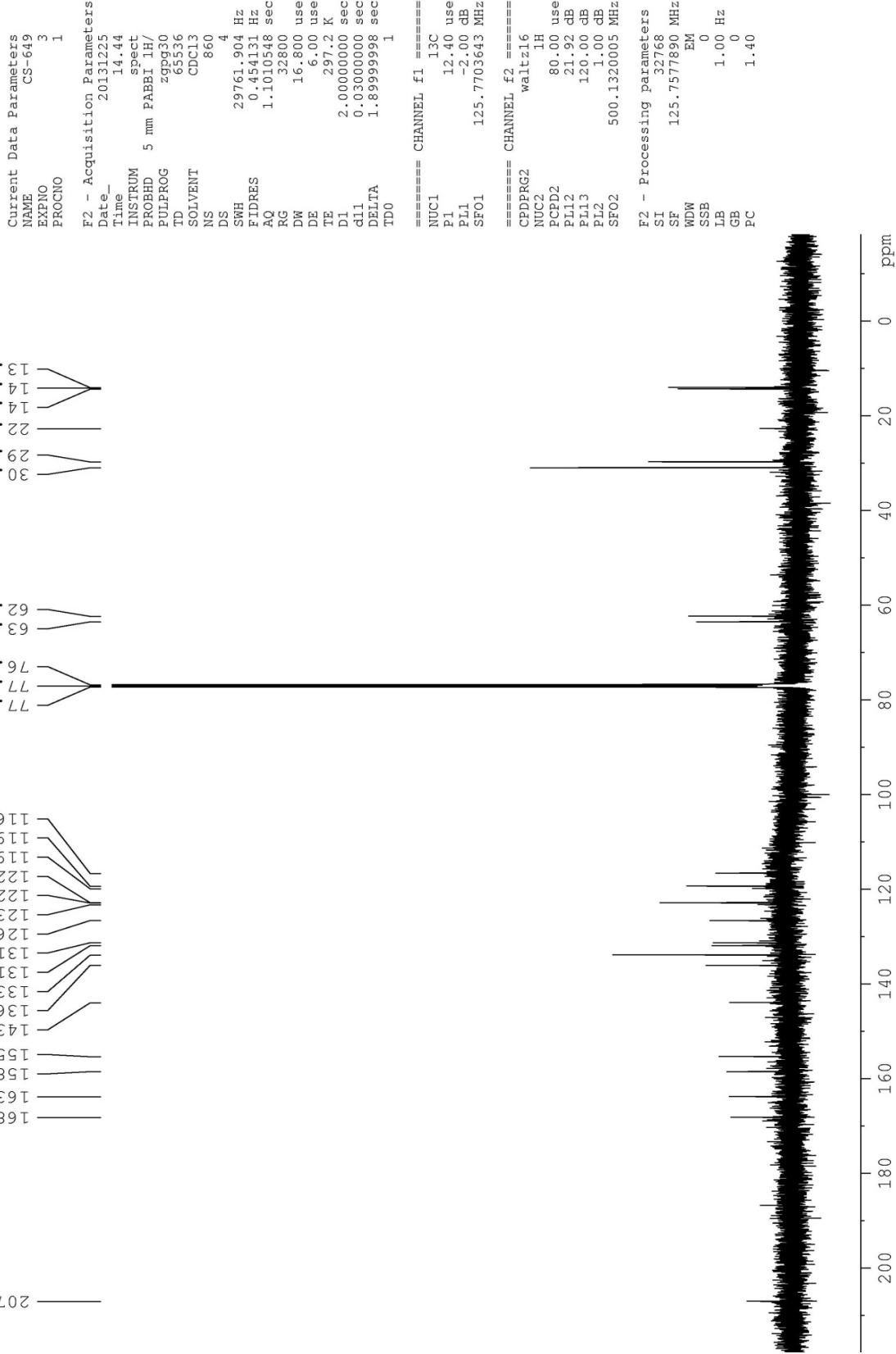
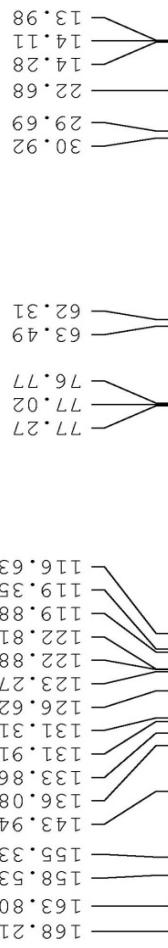
F12 - Acquisition Parameters

F2 - Acquisition Parameter
 Date- 20131219
 Time 11.39
 INSTRUM spect
 PROBHD 5 mm PABBI 1H/
 PULPROG zg30
 TD 655136
 SOLVENT CDC13
 NS 16
 DS 10330.578 Hz
 SWH 0.157632 Hz
 FIDRES 3.1719923 se
 AQ RG 114
 DW 48.400 us
 DE 6.00 us
 TE 296.1 K
 D1 1.00000000 se
 TDO 1

===== CHANNEL f1 =====	
NUC1	1.1 H
P1	7.20 us
PL1	1.00 dB
SFO1	500.1330885 MH
F2 - Processing parameters	
SI	32768
SF	500.1300000 MH
WDW	EM
SSB	0
LB	0.30 Hz
GB	1.00
PC	



207.07



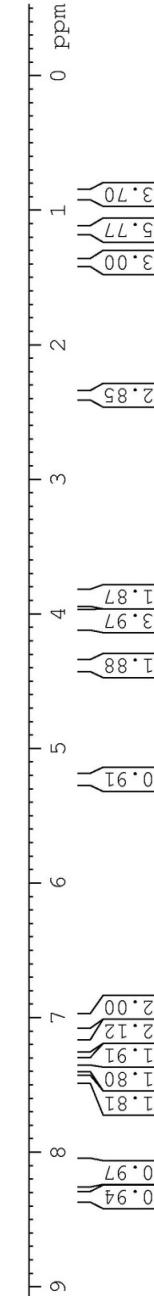
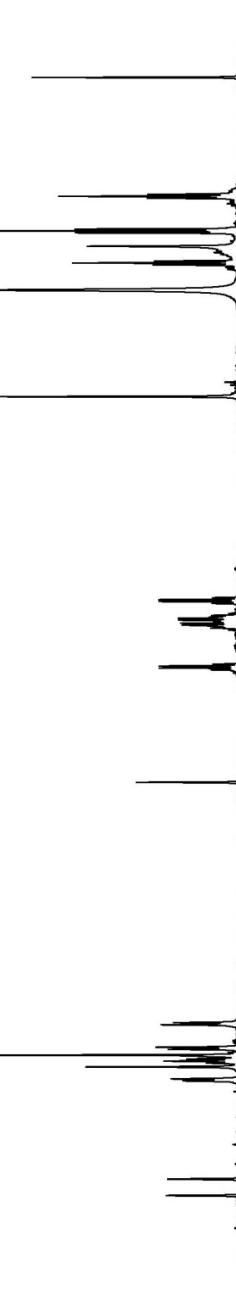
Current Data Parameters
NAME CS-656-TB
EXPNO 1
PROCNO 1

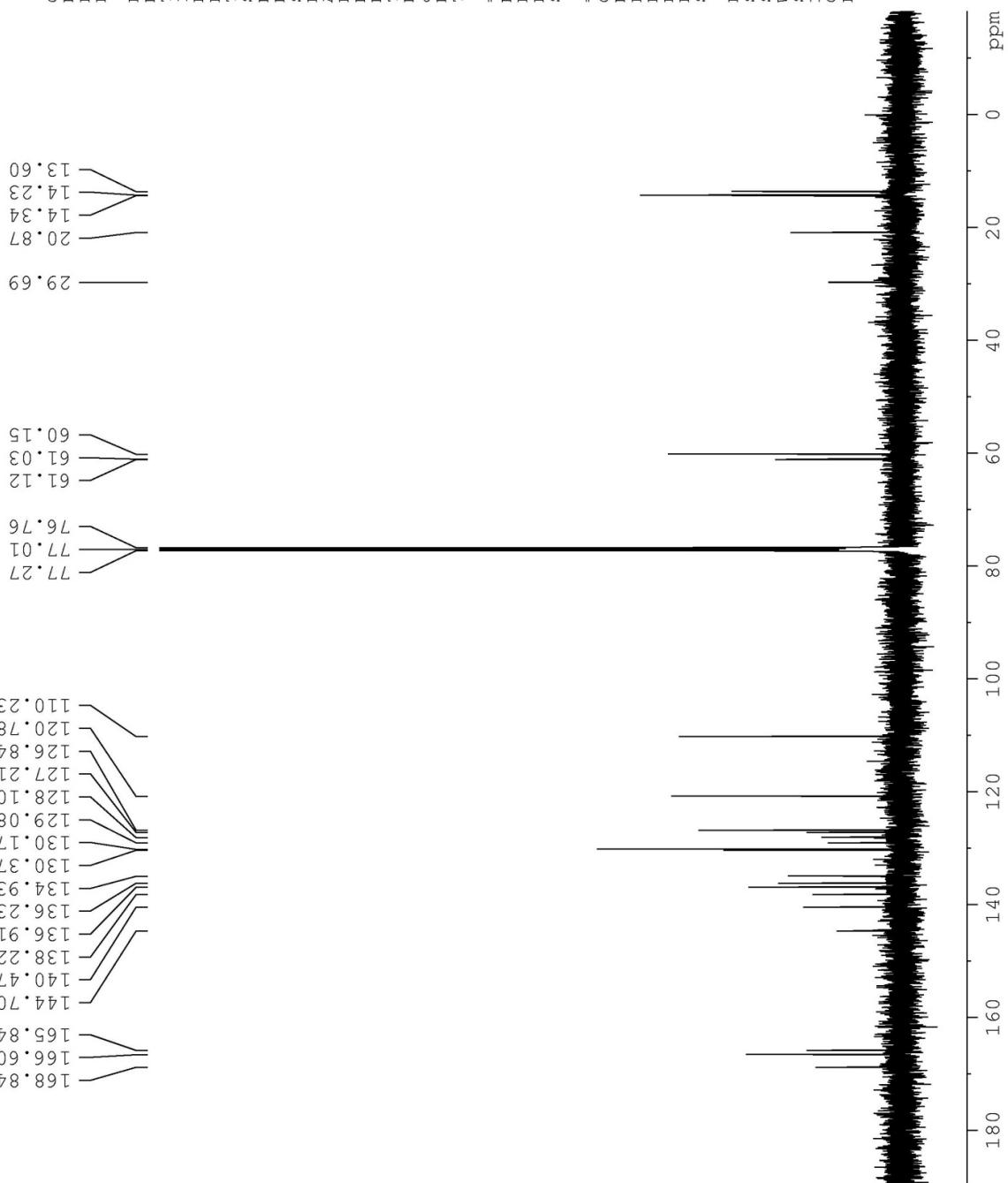
F2 - Acquisition Parameter
Date_ 20131226

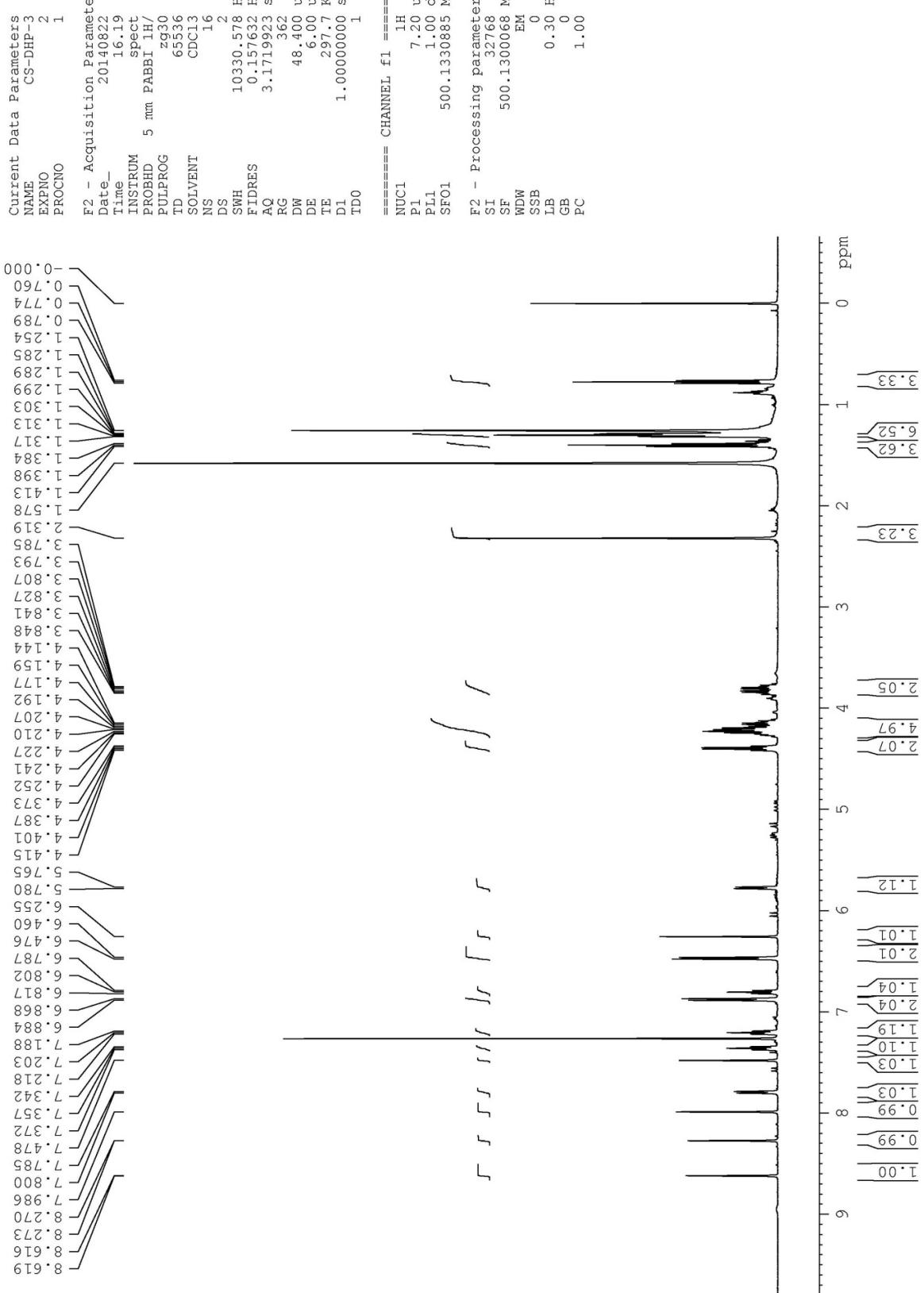
Time_ 20.17
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG 2930
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 se
RG 362
DW 48.400 us
DE 6.00 us
TE 296.5 K
D1 1.00000000 se
TDO 1

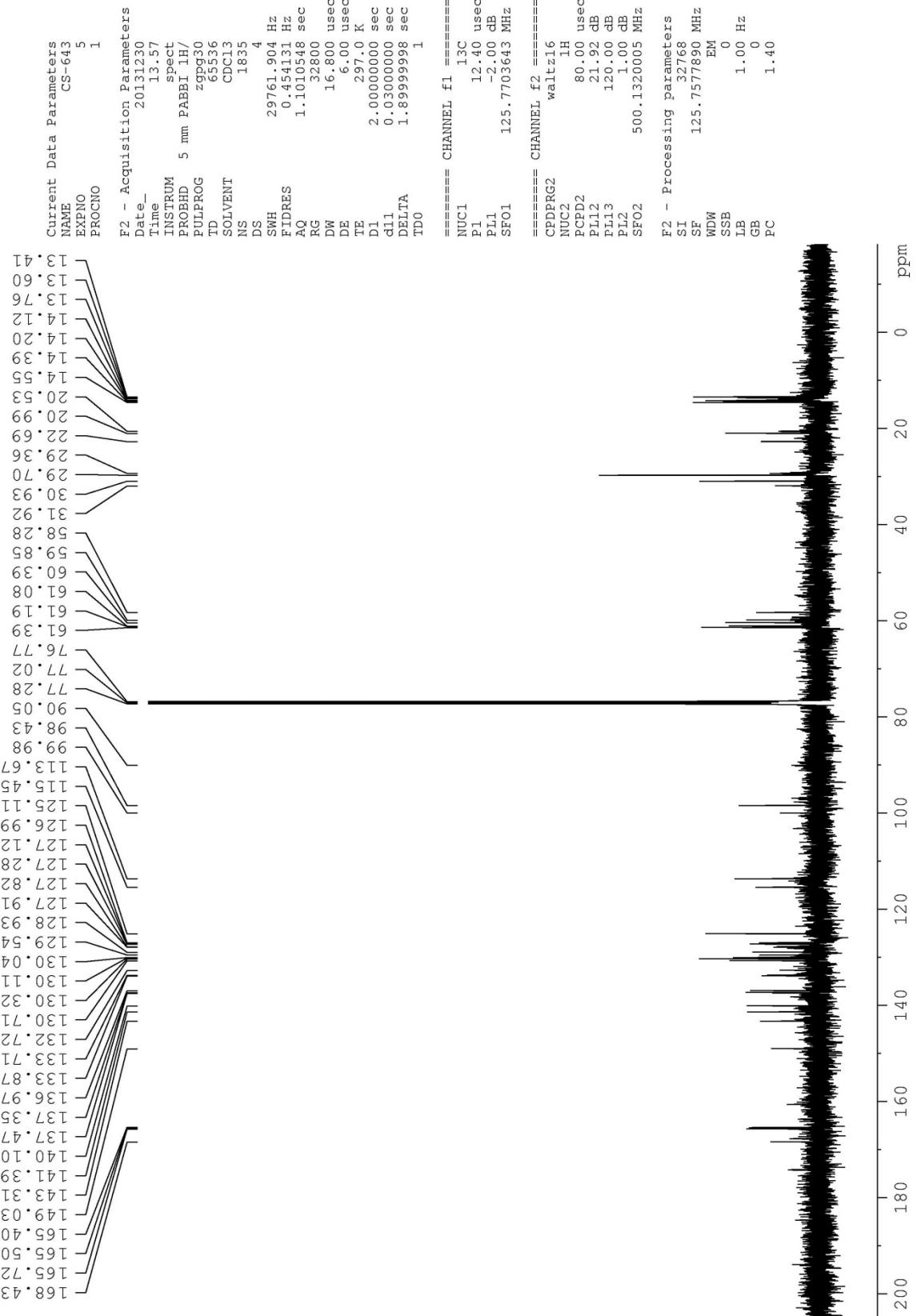
===== CHANNEL f1 =====

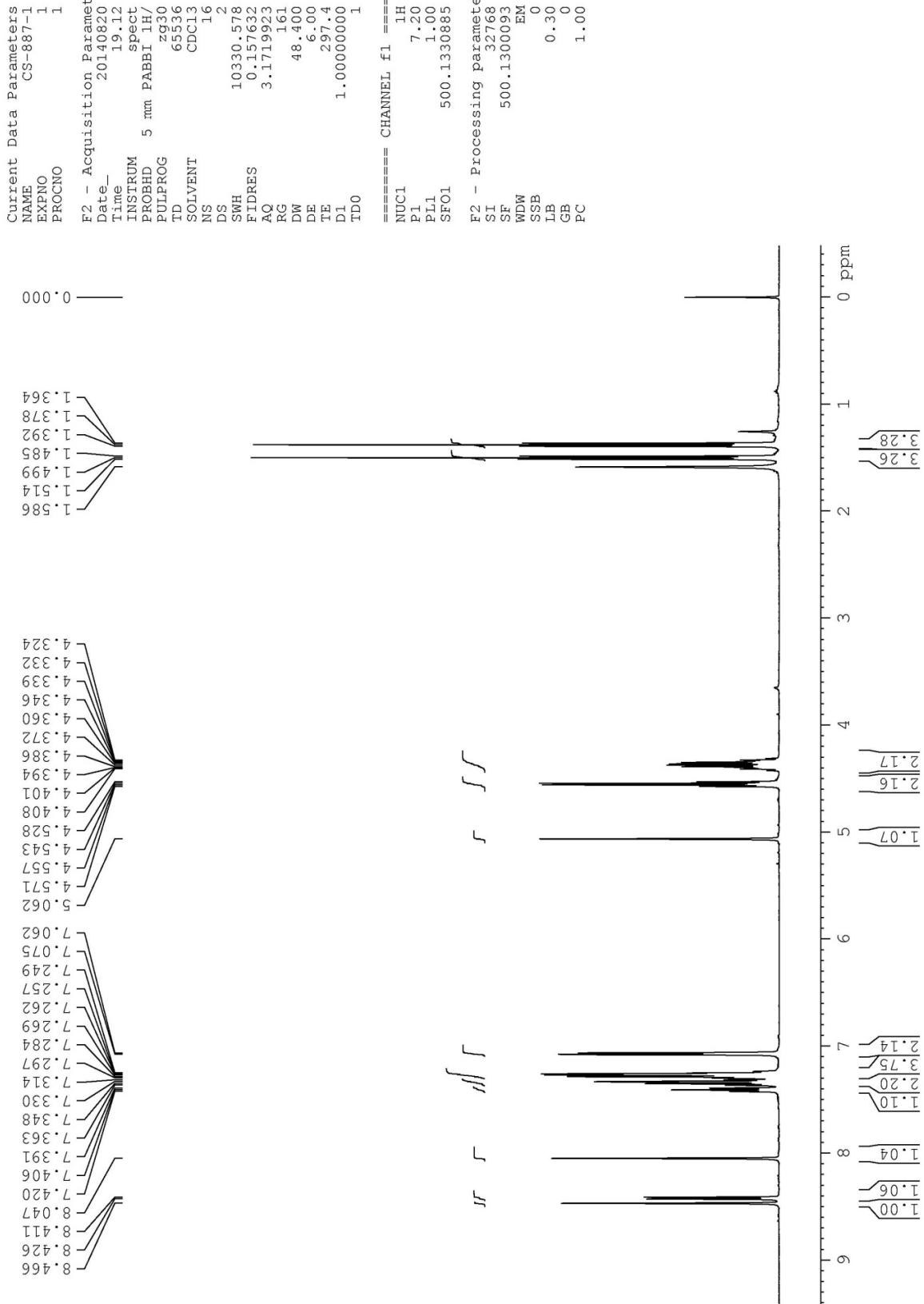
NUC1 1H
P1 7.20 us
PL1 1.00 dB
SFO1 500.1330885 MH

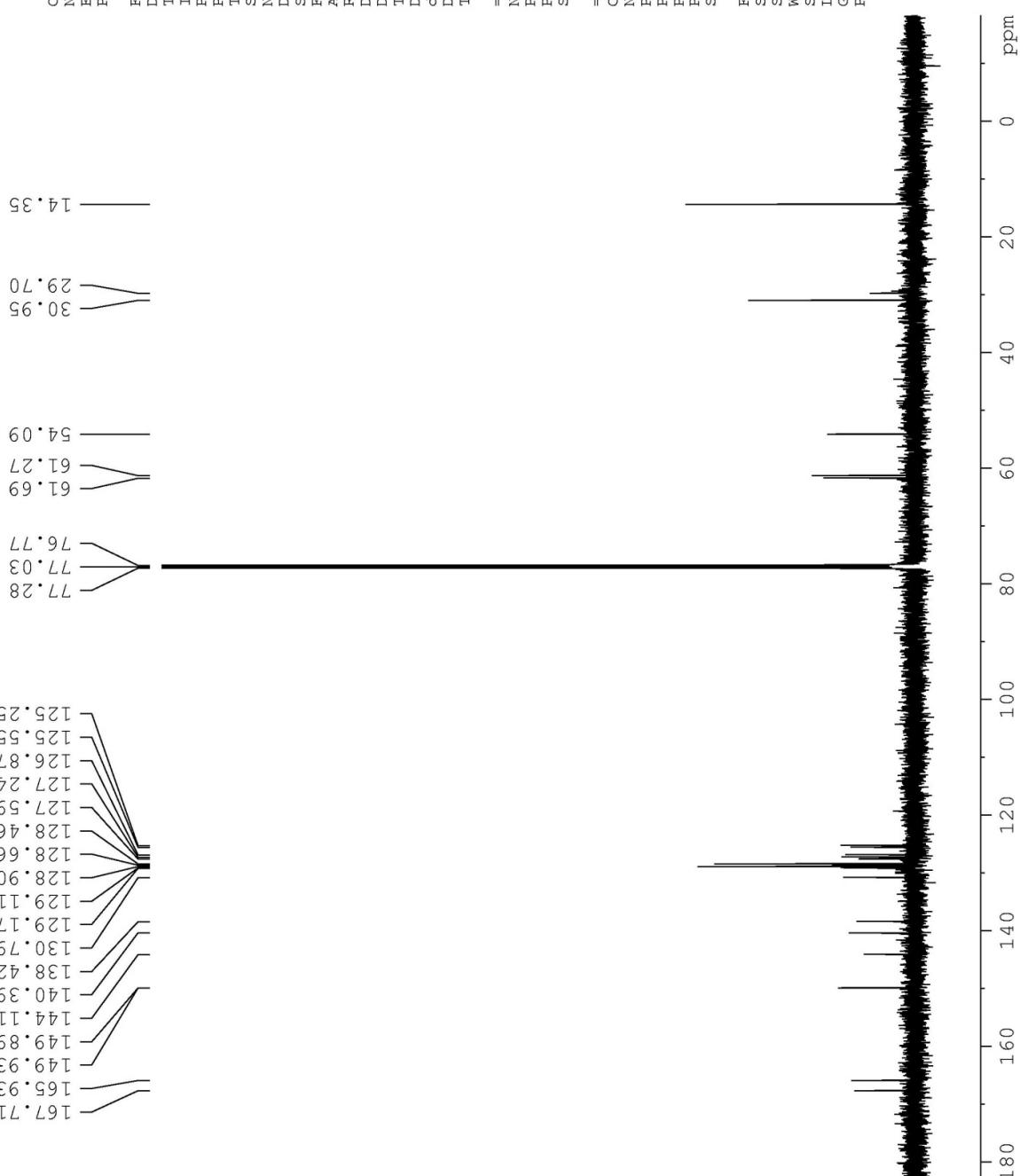


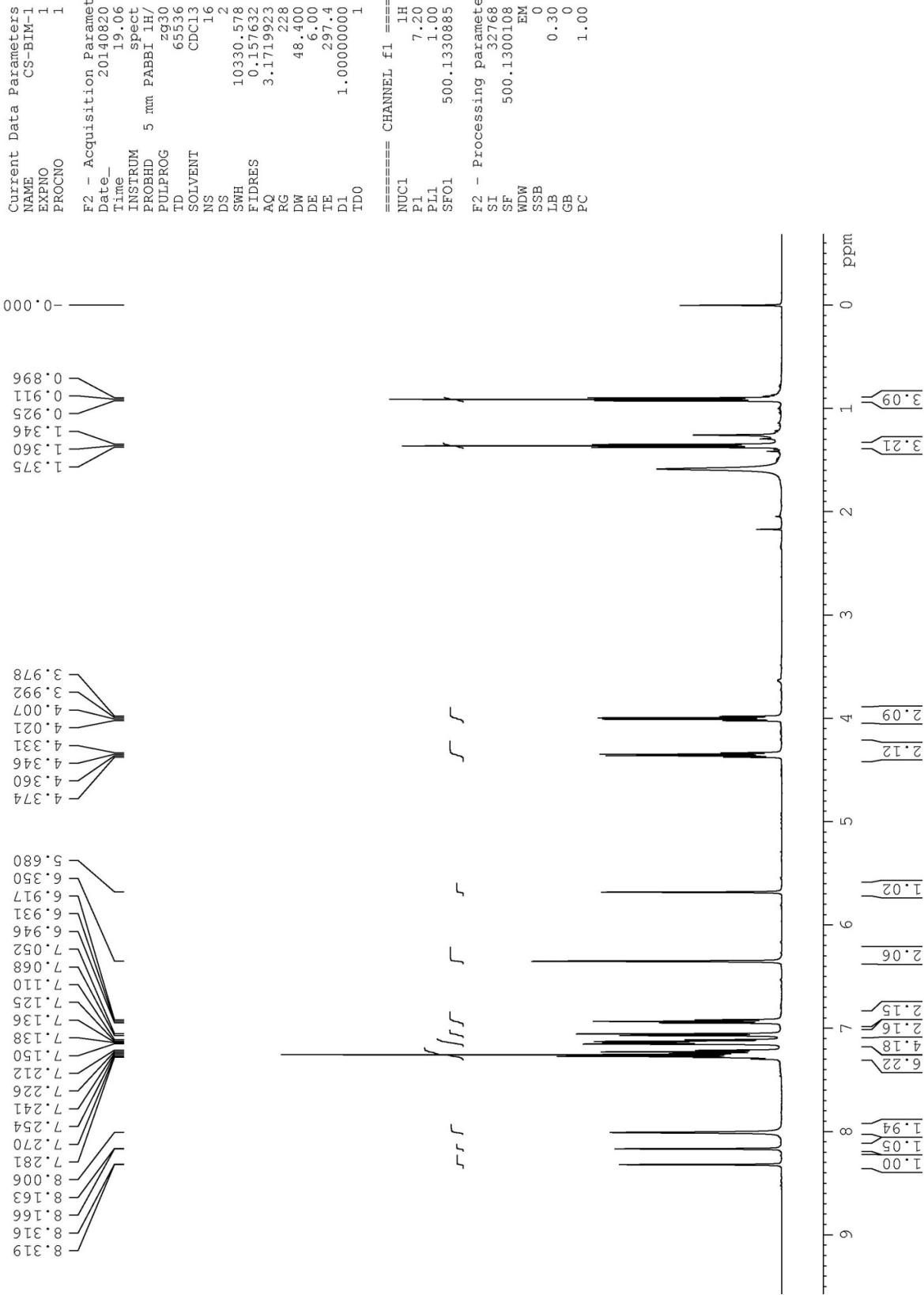






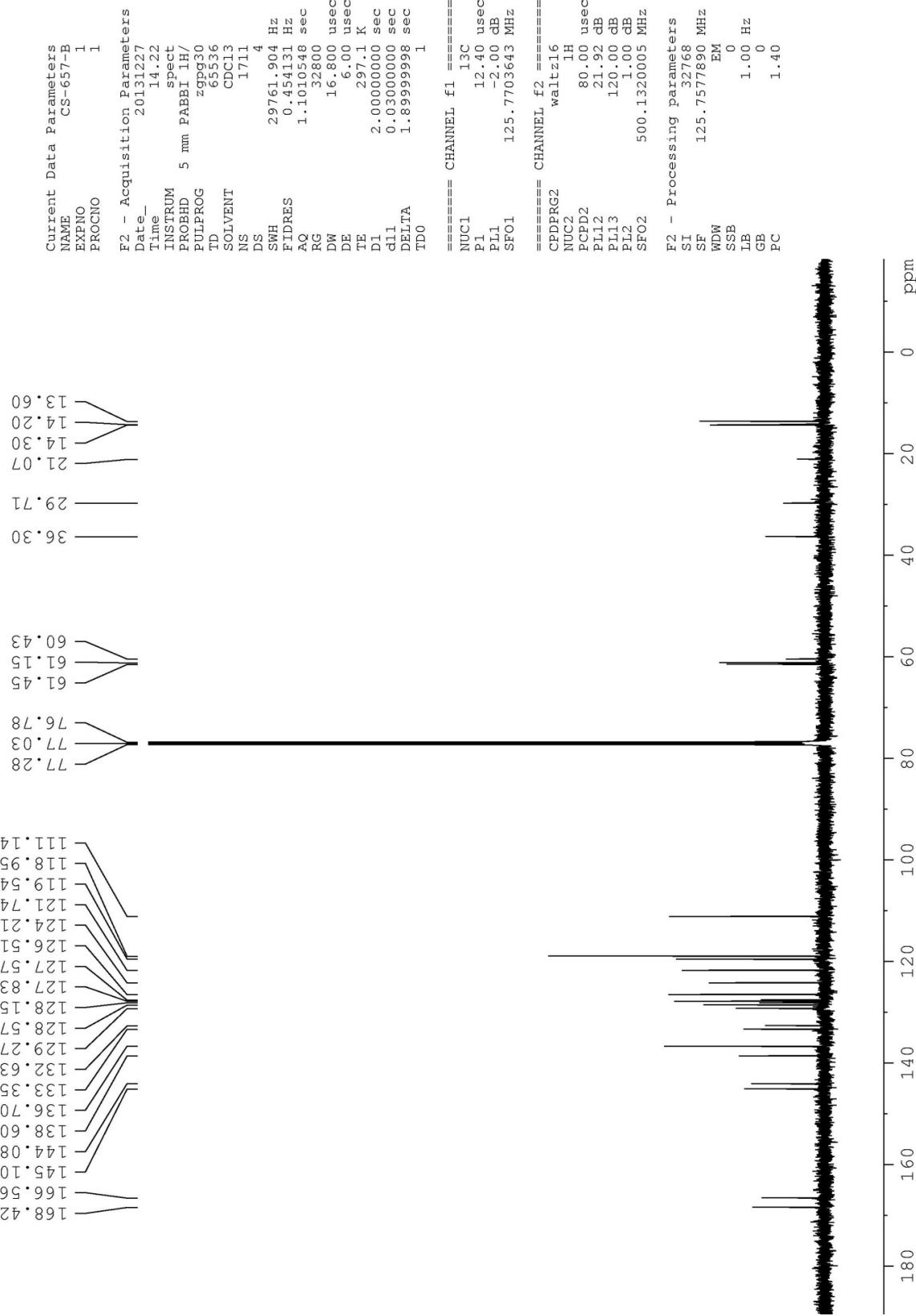






Current Data Parameters
 NAME CS-657.B
 EXPNO 1
 PROCNO 1

 F2 - Acquisition Parameters
 Date_ 20131227
 Time 14.22
 INSTRUM spect
 PROBHD 5 mm PABBI 1H/
 PULPROG zppg30
 TD 65536
 CDC13
 SOLVENT 1711
 DS 4
 SWH 29761.304 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010548 sec
 RG 32800
 DW 16.800 usec
 DE 6.00 usec
 TE 297.1 K
 D1 2.0000000 sec
 d11 0.03000000 sec
 DELTA 1.8999998 sec
 TDO 1



HMBC Spectra of 4, 6, 7a, 7e, 7g, 7l, 7m and 7q: Correlation between ortho H and carbonyl carbon is circled.

