Supporting Information

Synthesis of phosphatase-stable, cell-permeable peptidomimetic pro-drugs that target the SH2 domain of Stat3

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Experimental Methods

Fmoc-Haic was purchased from ChemImpex or was synthesized as described.[1] Fmoc-Gln-NHBn was prepared as described.[2] Other amino acids were from ChemImpex or NovaBiochem. Rink amide resin was from Advanced Chemtech. Solid phase peptide synthesis was carried out as described.[2,3] Peptides and prodrugs were tested for purity by reverse phase HPLC on an Agilent 1100 HPLC using a Phenomenex Luna 5μ C18(2) 4.6 × 250 mm column. For both peptide intermediates and prodrugs the gradient was 10 – 80% MeCN/30 min each solvent containing 0.1% TFA. Peptides or prodrugs were purified by reverse phase HPLC on a Varian Dynamax HPLC using a Phenomenex Luna 10μ C18(2) 21.2 × 250 mm column. Gradients of MeCN in H_2O at 20 mL/min were employed. For peptides 0.1% TFA was included in the eluents. For prodrugs no TFA was used in the mobile phase. NMR spectra were obtained on either a Bruker DPX 300 MHz spectrometer or a Bruker DRX 500 MHz spectrometer.

MDA-MB-468 breast tumor cells were acquired from the American Type Culture Collection and were maintained as monolayers in DMEM medium supplemented with 10% fetal bovine serum, 100 mM L-glutamine, 100 μM streptomycin and 100 U/mL penicillin. Cells were grown at 37°C in an atmosphere of 95% humidified air and 5% CO_2. Antibodies against Stat3 (Cat# 9132) and pStat3^{Y705} (Cat# 9145) were purchased from Cell Signaling Technology (Beverly, MA).

Synthesis of tert-butyl (E)-3-(4-iodophenyl)prop-2-enoate (2): A solution of *tert*-butyl diethylphosphonoacetate (10.0 g, 39.6 mmol), 4-iodobenzaldehyde (9.20g, 39.6mmol) and cesium carbonate (19.3 g, 59.4 mmol) in dry THF (15 mL) was stirred for 4 h. The solvent was removed *in vacuo*, the residue dissolved in 300 mL EtOAc, and the organic phase was washed with water (2 × 30 mL) followed by brine (1 × 30 mL) and dried ($MgSO_4$). After filtration and

concentration, the crude product was purified by silica gel column chromatography eluting with 10% EtOAc-hexanes. A white solid was obtained (**2**, 11.1 g, 86%). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.45 (s, 9H), 6.28 (d, 1H, J =16.0 Hz), 7.15 (d, 2H, J = 8.4 Hz), 7.41 (d, 1H, J=16.0 Hz), 7.62 (d, 1H, J = 8.4 Hz).

Synthesis of tert-butyl (E)-3-(4-diethoxyphosphoryldifluoromethyphenyl)prop-2-enoate (3): To a solution of diethyl bromodifluoromethylphosphonate (6.45 g, 24.1mmol) in dry DMF (100 mL) was added cadmium powder (5.41 g, 48.2 mmol). The suspension was stirred for 12 h under argon. Excess cadmium was removed by filtration under argon and the filtrate was treated with CuCl (2.86 g, 28.9 mmol) and **2** (5.00 g, 15.1 mmol) for 24 h at rt. Et_2O (300 mL), was added and the mixture stirred for 5 min and filtered. The organic layer was washed with saturated NH_4Cl (2 $\times$ 40 mL) and water (4 $\times$ 40 mL), dried ($MgSO_4$) and evaporated to give an oily residue. The crude product was purified by silica gel column chromatography with 40% EtOAc-hexanes as the eluant to give 4.92 g (83%) of **3** as colorless oil. HRMS (M + 1) calcd 391.1486, found 391.1441; ^{1}H NMR ($CDCl_3$, 300 MHz) δ1.32 (t, 6H, J = 7.2 Hz), 1.54 (s, 9H), 4.11-4.27 (m, 4H), 6.43 (d, 1H, J = 16.0 Hz), 7.56-7.64 (m, 5H). ^{19}F NMR ($CDCl_3$, 282.0 MHz) δ -108.8 (d, J = 115.0 Hz, 2F). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 16.2, 16.3, 28.1, 64.8, 64.9, 80.8, 122.2, 126.8, 127.8, 137.1, 142.1, 165.8.

Synthesis of pentachlorophenyl (E)-3-(4-diethoxyphosphoryldifluoromethyphenyl)prop-2-enoate (4): A solution of **7** (4.00 g, 10.2 mmol) in 5 mL dry dichloromethane was treated with 20 mL of trifluoroacetic acid for 1 h at room temperature. The TFA was removed *in vacuo* and residual acid was removed by addition and evaporation of toluene (2 $\times$ 10 mL). ^{1}H NMR (DMSO-d_6, 300 MHz) δ 1.31 (t, 6H, 6.9 Hz), 3.97-4.1 (m, 4H), 6.54 (d, 1H, J = 16.0 Hz), 7.47-7.57 (m, 3H), 7.76 (d, J = 8.1Hz, 2H). ^{19}F NMR (DMSO-d_6, 282.0 MHz) δ -107.7 (d, J = 122.8

Hz, 2F). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 16.5, 16.6, 65.1, 65.2, 121.9, 126.8, 126.9, 128.8, 133.5, 133.7, 137.4, 142.9, 167.7. The crude cinnamic acid derivative (3.5 g, 10.5 mmol), pentachlorophenol (3.1 g, 11.5 mmol), DCC (2.6 g, 12.6 mmol) and DMAP (1.3 g, 1.05 mmol) in 100 mL of EtOAc was stirred at room temperature for 24 h. The mixture was filtered through celite and the solvent removed *in vacuo*. The crude product was purified by silica gel chromatography eluting with 25% EtOAc-hexanes to give 5.1 g (84%) of **4** as a white solid. HRMS (M + 1) calcd 582.9195, found 582.9135; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.26 (t, 6H, J = 7.2 Hz), 4.1-4.2 (m, 4H), 6.67 (d, 1H, J = 16.0 Hz), 7.63 (s, 4H), 7.9 (d, J = 16.0 Hz, 1H). ^{19}F NMR ($CDCl_3$, 282.0 MHz) δ -109.06 (d, J = 114.2 Hz, 2F). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 16.3, 16.4, 64.9, 65.0, 116.7, 127.1, 127.8, 128.5, 131.6, 132.1, 135.9, 144.2, 147.6, 162.2.

Synthesis of Pentachlorophenyl (E)-3-(4-phosphoryldifluoromethyphenyl)prop-2-enoate (5): Iodotrimethylsilane (2.0 mL, 3.7 mmol) in 5 mL of dry CH_2Cl_2 was added dropwise to a solution of **4** (2.0 g, 3.43 mmol) and bis(trimethylsilyl)trifluoroacetamide (1.8 mL, 6.8 mmol) in 20 mL of dry CH_2Cl_2 at 0^{o}C under argon. Stirring was continued for 1 h at 0^{o}C and 1 h at room temperature. The solution was concentrated *in vacuo*. The residue was taken up in 20 mL MeCN/H_2O/AcOH (8:1:1), stirred for 45 min and concentrated *in vacuo*. Toluene (5 mL) was added and evaporated twice. On addition of ether solids separated, which were collected by filtration and washed with the same solvent to give 1.6 g of **5** as a white powder (89%). HPLC t_R 25.22, HRMS (M + H) calcd 526.8569, found 526.8542. 1H NMR (DMSO-d_6, 300 Hz) δ 7.13 (d, J = 15.9 Hz,1H), 7.60 (d, J = 8.1 Hz, 2H), 8.0 (d, J = 8.1 Hz, 1H), 8.08 (d, J =15.9 Hz, 2H). ^{13}C NMR (DMSO-d_6, 125 MHz) δ 116.4, 127.2, 127.8, 129.3, 131.2, 131.7, 135.4, 144.3, 148.9, 162.8. ^{19}F NMR (DMSO-d_6, 282 MHz) δ -108.52 (d, 2F, J= 104.3 Hz).

Synthesis of pentachlorophenyl (E)-3-(4-dipivaloyloxymethylphosphoryl-difluoromethyphenyl)prop-2-enoate (6): NaOH (144 mg, 3.6 mmol) in 2 mL of water was

added dropwise to a stirred suspension of **5** (1 g, 1.9 mmol) in 5 mL of water. When the mixture became clear (pH~10), $AgNO_3$ (807 mg, 4.75 mmol) was added. After 2 h at 4°C the gray precipitate was collected by filtration, dried, and pulverized in a mortar and pestle. The powder was suspended in dry toluene (10 mL) and pivyloxymethyl iodide (1.4 g, 5.7 mmol) was added and stirred for 24 h at room temperature. After filtration the solvent was removed *in vacuo* and the crude product was purified by silica gel column chromatography eluting with 30% EtOAc-hexanes to give a colorless sticky liquid of **6** (0.90 g, 64%) which solidified on storage at 4°C. ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.15 (s, 18H), 5.58 (dd, J = 12.3, 5.0 Hz, 2H), 5.67 (dd, J = 12.3, 5.0 Hz, 2H), 6.68 (d, J = 16.0 Hz, 1H), 7.6-7.64 (m, 4H), 7.9 (d, J = 16.0 Hz, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ 26.7, 38.7, 82.4, 82.5, 117.0, 127.2, 127.8, 128.6, 131.6, 132.1, 136.3, 144.1, 147.4, 162.2, 176.5. ^{19}F NMR ($CDCl_3$, 282 MHz) δ -109.54 (d, J = 123.4 Hz, 2F). ^{31}P NMR ($CDCl_3$ 202 MHz) δ 4.65 (m, 1P). Proton Decoupled ^{31}P NMR ($CDCl_3$ 202 MHz) δ 4.64 (t, J = 123.2 Hz, 1P). Anal. Calcd for $C_{28}H_{28}Cl_5F_2O_9P$: C, 44.56; H, 3.74; Cl, 23.49; F, 5.03. Found: C, 44.60; H, 3.76; Cl, 23.46; F, 4.95. HRMS (M + Na) calcd 776.9750, found 776.9718.

Synthesis of TFA·H-Haic-Gln-NHBn (7): Fmoc-Haic-Gln-NHBn was prepared on solid phase using 0.300 g (0.36 mmol) of rink resin as described by Mandal et al.[3] The Fmoc group was removed by treatment with 20% piperidine/DMF and the resin then treated with 3 $\times$ 10 mL of trifluoroacetic acid: triisopropylsilane: H_2O (TFA:TIS:H_2O) (95:2.5:2.5 v/v/v)[4] and filtered. The combined filtrates were evaporated to ca 5 mL and the product was isolated by precipitation in Et_2O and collected by centrifugation. The crude peptide was purified by reverse phase HPLC, using 0.1% TFA-water/acetonitrile, lyophilized, and dried *in vacuo* over P_2O_5 at 37°C to get 106 mg of a white solid. HPLC t_R16.28, (Purity, 100% at 230 nm, 95% at 275 nM). LC-MS (M + H) calcd 464.2298, found 464.1441. ^{1}H NMR (DMSO-d_6 500 MHz) δ 1.78-1.83 (m, 1H), 1.86-1.94 (m, 1H), 2.03-2.19 (m, 4H), 2.96 (d, J = 15.0 Hz, 1H), 3.14 (m, 2H), 3.44 (m, 1H), 4.22 (m, 2H),

4.28 (t, J = 6.0 Hz, 1H), 5.17 (dd, J = 2.5, 11.0 Hz, 1H), 6.8 (s, 1H), 7.02 (t, J = 7.5Hz, 1H), 7.08 (d, J = 7.5 Hz, 1H), 7.12 (d, J = 7.5 Hz, 1H), 7.21 -7.25 (m, 3H), 7.28-7.30 (m, 2H), 8.36 (m, 2H), 8.43 (m, 2H). ^{13}C NMR (DMSO-d_6 125 MHz) δ 28.5, 31.2, 31.9, 32.3, 52.9, 60.8, 124.4, 127.3, 127.5, 128.7, 171.1, 171.52.

Preparation of BP-PM6: To a stirred solution of **7** (0.050 g, 0.086 mmol), NMM (0.03 mL, 0.26 mmol) and DMAP (0.005 g, 0.04 mmol) in 2 mL of anhydrous NMP was added a solution of **6** (0.070 g, 0.091 mmol) in 2 mL of dry CH_2Cl_2 under argon. The reaction was monitored by HPLC. After completion, about 2 h, the reaction mixture was concentrated under vacuum and triturated with hexane-ether. The solid residue (0.102 g) was taken up in 2 mL of MeCN and purified by reverse phase HPLC using a gradient of MeCN in H_2O (no TFA in the eluant). Yield: 0.060 g (72%). HPLC t_R25.80 (purity>98%), HRMS (M + H) calcd 952.3709, found 952.3412. ^{19}F NMR (Acetone-d_6 282 MHz) δ -108.0 (d, J =123.0, 2F). Proton Decoupled ^{31}P NMR (Acetone-d_6 202 MHz) δ 4.1 (tr, J = 123.0 Hz, 1P). ^{1}H NMR (Acetone-d_6 500 MHz) δ 1.2 (s, 18H), 1.95-2.00 (m, 1H), 2.1-2.2 (m, 1H), 2.24-2.34 (m, 4H), 3.13-3.22 (m, 3H), 3.5 (m, 1H), 4.4 (d, J = 5.5Hz, 2H), 4.44 (m, 1H),4.65-4.7 (m, 1H), 5.24 (m, 1H), 5.72-5.8 (m, 4H), 6.95-7.02 (m, 2H), 7.1 (m, 1H), 7.2-7.21 (m, 0.8H), 7.25-7.28 (m, 3H), 7.6-7.66 (m, 3H), 7.8 (d, J = 8.0 Hz, 1H), 8.2 (d, J = 8.0Hz, 1H), 8.28 (t, J = 6.0 Hz, 1H), 8.4 (d, J = 7.0 Hz, 0.2 H). ^{13}C NMR (Acetone-d_6 125 MHz) δ 26.1, 27.9, 30.8, 31.3, 38.4, 42.5, 52.8, 53.7, 61.6, 82.7, 123.2, 123.6, 124.3, 126.5, 126.9, 127.3, 128.1, 128.4, 129.5, 132.8, 138.5, 138.9, 139.0,165.0, 170.4, 171.5, 171.7, 175.5, 176.3.

Synthesis of TFA·H-Leu-Pro-Gln-NHBn (8): Fmoc-Leu-Pro-Gln-NHBn was prepared on solid phase using 0.200 g (0.24 mmol) of rink resin as described by Mandal et al.[3] The Fmoc group was removed by treatment with 20% piperidine/DMF and the peptide was cleaved and isolated as in the case of TFA·H-Haic-Gln-NHBn. The crude peptide was purified by reverse phase

HPLC, using a gradient of 0.1%TFA-water/acetonitrile, to get 88 mg of white solid after drying *in vacuo* over P_2O_5 at 37°C for 24 hr. HPLC t_R9.17, (Purity, 95%) HRMS (M + H) calcd 446.2767, found 446.3924. ^{1}H NMR (DMSO-d_6 500 MHz) δ 0.9-0.95 (m, 7H), 1.55 (m, 2H), 1.78-1.98 (m, 7H), 2.01-2.13 (m, 3H), 3.42 (m, 1H), 3.72 (m, 1H), 4.12 (m, 1H), 4.22 (m, 1H), 4.28-4.3 (m, 2H), 4.42-4.45(m 1H), 6.8 (s, 1H), 7.23-7.33 (m, 7H), 8.15-8.2 (m, 4H), 8.37 (m, 1H).^{13}C NMR (DMSO-d_6 125 MHz) δ 21.6, 23.6, 23.8, 25.1, 28.4, 29.5, 31.9, 42.5, 47.3, 49.9, 53.0, 60.1, 127.2, 127.5, 128.7, 139.8, 158.4, 158.7,168.2, 171.3, 171.6, 174.3.

Preparation of BP-PM279G: To a stirred solution of **8** (0.042 g, 0.075 mmol), NMM (0.025 mL, 0.22 mmol) and DMAP (0.005 g, 0.04 mmol) in 5 mL of 50:50 anhydrous MeCN/CH_2Cl_2, was added **6** (0.060 g, 0.078 mmol) under inert atmosphere. The reaction was monitored by HPLC. After completion, about 2 h, the reaction mixture was concentrated under vacuum and triturated with ether. The solid residue (0.092 g) was taken up in 2 mL of MeCN and purified by reverse phase HPLC using a gradient of MeCN in H_2O (no TFA in the eluant). Yield: 53 mg (75%). HPLC t_R26.29 (purity >98%), HRMS (M + H) calcd 934.4179, found 934.4190. ^{19}F NMR (Acetone-d_6 282 MHz) δ -109.43 (d, J =124.0, 2F). Proton Decoupled ^{31}P NMR (Acetone-d_6 202 MHz) δ 4.3 (t, J = 121.0 Hz, 1P). ^{1}H NMR (Acetone-d_6 500 MHz) δ 0.93 (d, J = 6.0 Hz, 3H), 0.97 (d, J = 6.0 Hz, 3H), 1.22 (s, 18H), 1.53-1.58 (m, 1H), 1.67--1.74 (m, 2H), 1.93-2.0 (m, 2H), 2.18-2.23 (m, 1H), 2.3-2.4 (m, 2H), 3.85 (m, 1H), 3.94 (m, 1H), 4.3-4.37 (m, 3H), 4.43-4.49 (m, 1H), 4.93 (m, 1H), 5.72-5.81 (m, 4H), 6.5 (s, 0.5H), 6.9 (d, J = 16.0 Hz, 1H), 7.14 (s, 0.5), 7.23 (t, J = 7.0 Hz, 1H), 7.27-7.35 (m, 4H), 7.57 (d, J = 7.5 Hz, 1H), 7.62-7.7 (m, 4H), 7.77 (d, J = 8.0 Hz, 2H), 7.86 (m, 1H), 8.21(d, J = 7.0 Hz, 1H). ^{13}C NMR (Acetone-d_6 125 MHz) δ 21.1, 22.8, 24.5, 24.9, 26.1, 26.7, 31.5, 38.4, 40.4, 42.4, 47.4, 49.7, 53.5, 61.6, 82.6, 123.6, 126.6, 126.8, 127.2, 127.3, 127.9, 128.2, 138.2, 138.7, 139.7, 164.6, 171.1, 171.5, 172.4, 175.5, 176.0.

Synthesis of F2PmCinn-Haic-Gln-NHBn (NP-PM6): Fmoc-Haic-Gln-NHBn was assembled on 0.2 g (0.12 mmol) of Rink resin using the general procedures described above. Compound **5** (0.095 g, 0.18 mmol), HOBt (0.027 g, 0.18 mmol) and DIEA (31 μL, 0.18 mmol) in 5 mL of DMF was added. After 3 hr the ninhydrin test was negative. The peptide was cleaved from the resin with trifluoroacetic acid: triisopropylsilane: H_2O (95:2.5:2.5 v/v/v) and purified as above to give 43 mg of final peptide. T_R (min.) (0.1%TFA/MeCN-H_2O): 13.64 (Purity: > 99%). HRMS (M+H) Cald: 724.2348. Found 724.2620. ^{1}H NMR (DMSO-*d*6 500 MHz) δ 1.38-1.48 (m, 2H), 1.67 (m, 1H), 1.80 (m, 1H), 1.98-2.04 (m, 4H), 2.87 (d, J = 16.0 Hz, 1H), 2.95-3.06 (m, 2H), 3.32 (m, 2H), 4.12-4.22 (m, 3H), 4.42 (m, 1H), 5.03 (d, J = 8.5 Hz, 1H), 6.66 (s, 1H), 6.8 (d, J = 15.5 Hz, 1H), 6.88 (t, J =7.0 Hz, 1H), 6.99 (m, 1H), 7.11-7.2 (m, 5H), 7.36 (d, J = 16.0 Hz, 1H), 7.48 (s, 4H), 8.18 (d, J = 8.0 Hz, 1H), 8.32 (m, 1H), 8.50 (d, J = 7.0 Hz, 1H). ^{13}C NMR (DMSO-*d*6 125 MHz) δ 22.4, 22.7, 28.5, 29.5, 31.5, 31.9, 32.1, 42.5, 44.1, 52.9, 53.4, 61.0, 123.0, 124.0, 125.9, 127.1, 127.2, 127.5, 128.7, 129.7, 133.1, 135.7, 139.1, 139.4, 139.7, 164.9, 169.6, 171.0, 171.6, 174.1. ^{31}P NMR (DMSO-*d*6 202 MHz) δ 0.59 (t, J = 90.9 Hz, 1P).

Synthesis of MP-PM6: From failed solid phase synthesis of **BP-PM6**. 22 mg of desired material was obtained after HPLC purification. T_R (min) (0.1%TFA/MeCN-H_2O): 19.78 (Purity: > 97%). HRMS (M+H) Cald: 838.3029. Found 838.3032. ^{1}H NMR (DMSO-*d*6 500 MHz) δ1.06 (s, 9H), 1.68 (m, 1H), 1.80 (m, 1H), 1.94-2.08 (m, 4H), 2.87 (d, J = 16.5 Hz, 1H), 2.95-3.1 (m, 2H), 3.35 (m, 1H), 4.11-4.22 (m, 3H), 4.43 (t, J = 8.0 Hz, 1H), 5.04 (m, 1H), 5.38 (d, J = 11.5 Hz, 2H), 6.84 (d, J = 16.0 Hz, 1H), 6.9 (t, J = 7.0 Hz, 1H), 7.00 (t, J = 8.0 Hz, 1H), 7.11-7.21 (m, 5H), 7.38 (d, J = 16.0 Hz, 1H), 7.47 (d, J = 8.0 Hz, 2H), 7.55 (d, J = 8.0 Hz, 2H), 8.15 (d, J = 8.0 Hz, 1H), 8.3 (t, J = 6.0 Hz, 1H), 8.51 (d, J = 7.5 Hz, 1H). ^{13}C NMR (DMSO-*d*6 125 MHz) δ 27.1, 28.5, 29.5, 31.5, 31.8, 38.6, 42.5, 52.8, 53.4, 54.6, 61.0, 83.4, 117.9, 123.5, 125.9, 127.2, 127.5, 128.7,

129.7, 133.1, 136.7, 138.7, 139.4, 139.7, 169.6, 171.6, 174.1, 176.8, 197.7. ^{31}P NMR (DMSO-*d*6 202 MHz) δ 0.61 (t, J = 101.0 Hz, 1P).

Inhibition of Stat3 tyrosine 705 phosphorylation in MDA-MB-468 cells.

MDA-MB-468 breast tumor cells (0.4×10^6) were plated onto 35 mm culture dishes and were allowed to grow overnight. Prodrugs were prepared as 10 mM stock solutions in DMSO and aliquots were added to the culture media to give the correct final concentrations. After 2 h the cells were washed with ice cold phosphate buffered saline. Washed cells were treated with lysis buffer (50 mM Hepes, pH 7.4, 150 mM NaCl, 1.5 mM $MgCl_2$, 1 mM EGTA, 100 mM NaF, 10 mM sodium pyrophosphate, 10% glycerol, 1% Triton X-100, 1 mM PMSF, 1 mM Na_3VO_4, 10 μg/mL leupeptin and 10 μg/mL aprotinin). Cell-free detergent extracts were centrifuged at 15,000 rpm in a microcentriguge for 30 min at 4°C and the protein concentrations of the supernatants determined. Aliquots containing 12 μg of protein were separated on 8% SDS-PAGE and were transferred to PVDF filters. The filters were blocked with 5% bovine serum albumin and were probed with pStat3^{Y705} antibody followed by secondary antibody, whose signal was detected with an enhanced chemiluminescence kit (ECL, Amersham, Chicago, IL). Filters were stripped with stripping buffer (62.5 mM Tris, pH 6.8, 2% SDS, and 0.1 M 2-mercaptoethanol) at 50°C for 30 min. Filters were then probed with total Stat3 antibody and visualized with chemiluminescence as above.

References for the Supporting Information

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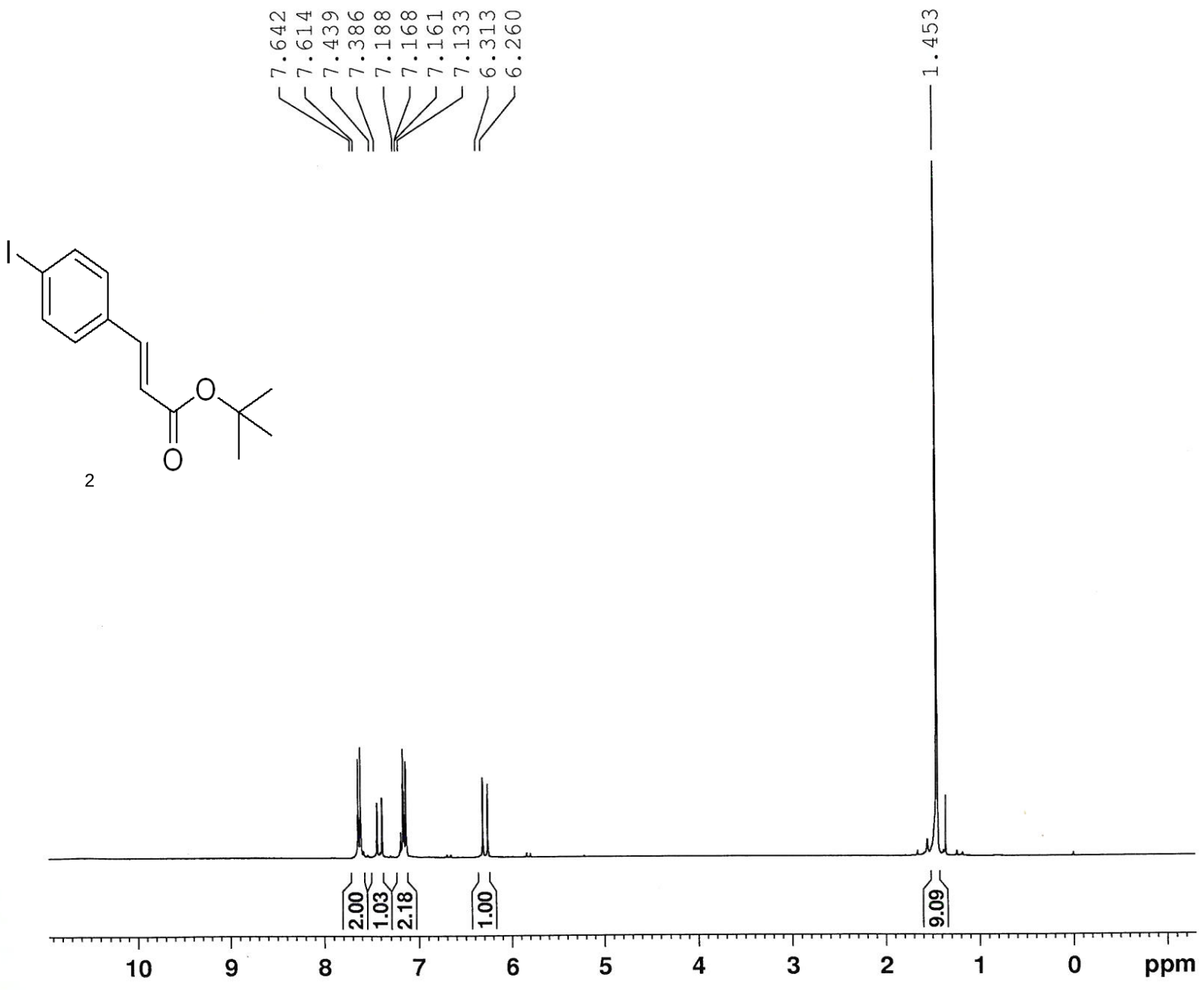


Current Data Parameters

NAME	BPPM-6_OL
EXPNO	1
PROCNO	1

F2 - Acquisition Paramet

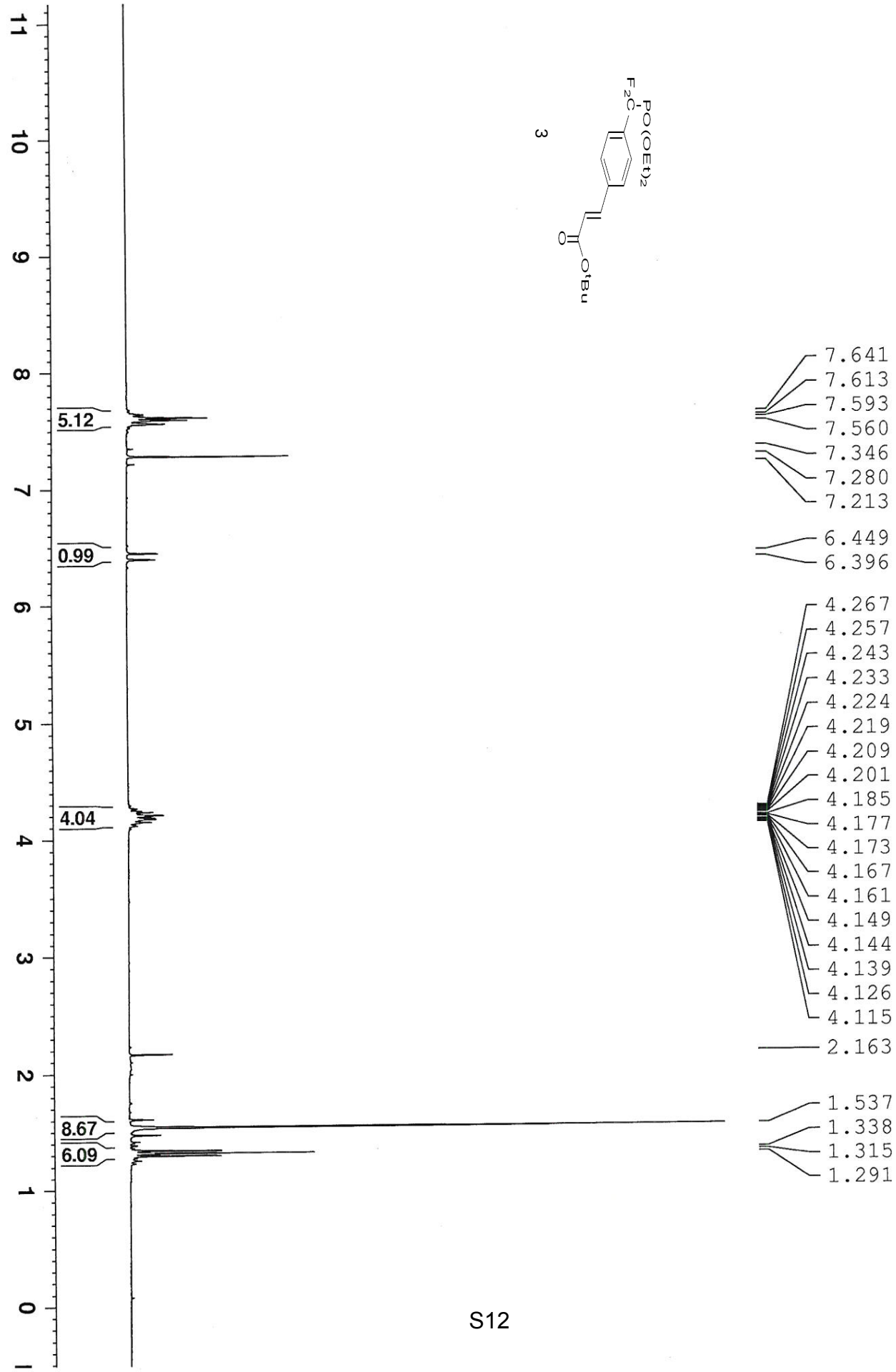
Date_	20090417
Time	12.45
INSTRUM	spect
PROBHD	5 mm QNP 1H/19
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	6172.839
FIDRES	0.094190
AQ	5.3084660
RG	161.3
DW	81.000
DE	6.00
TE	301.2
D1	1.00000000
TD0	1

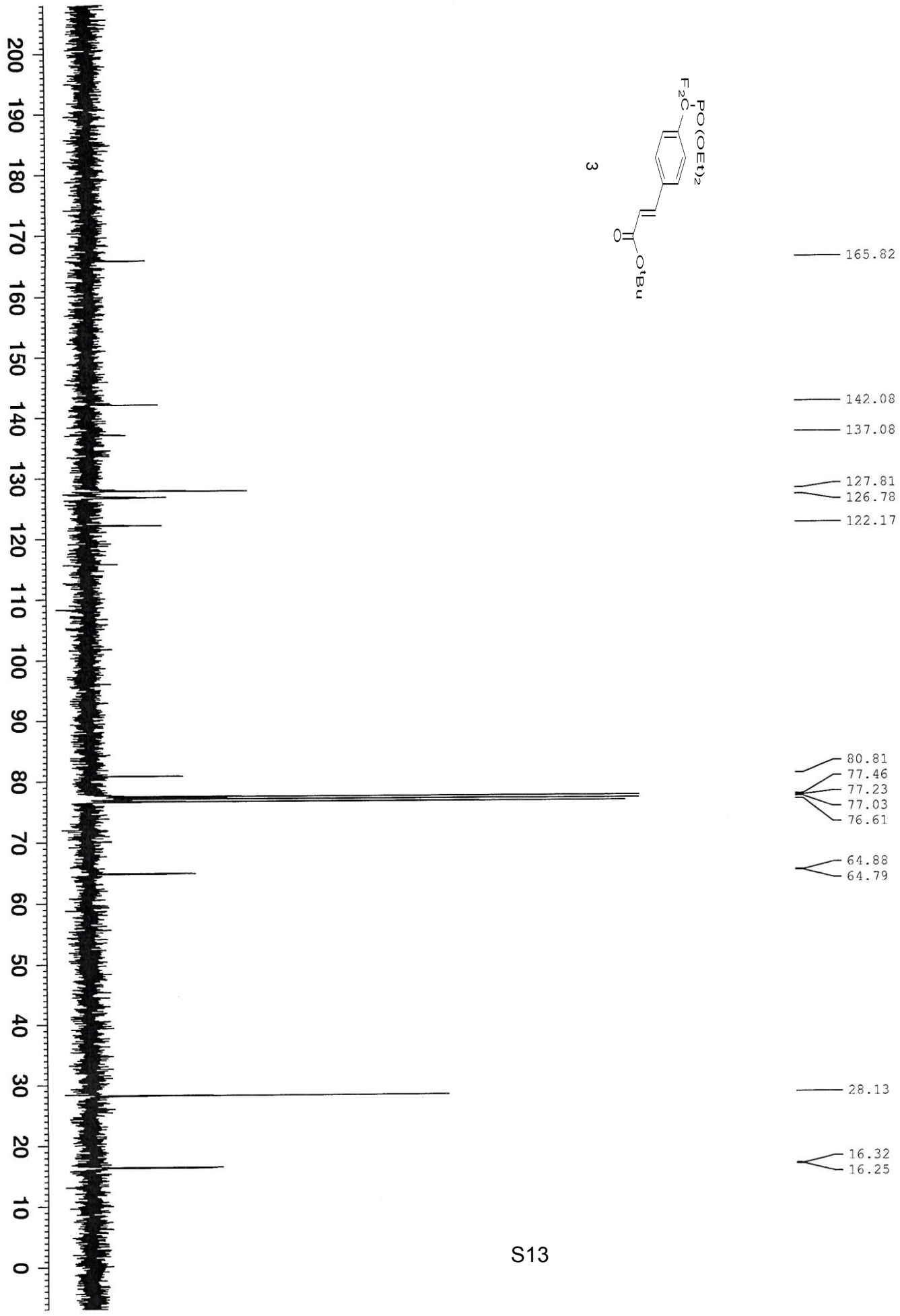
======== CHANNEL f1 ====

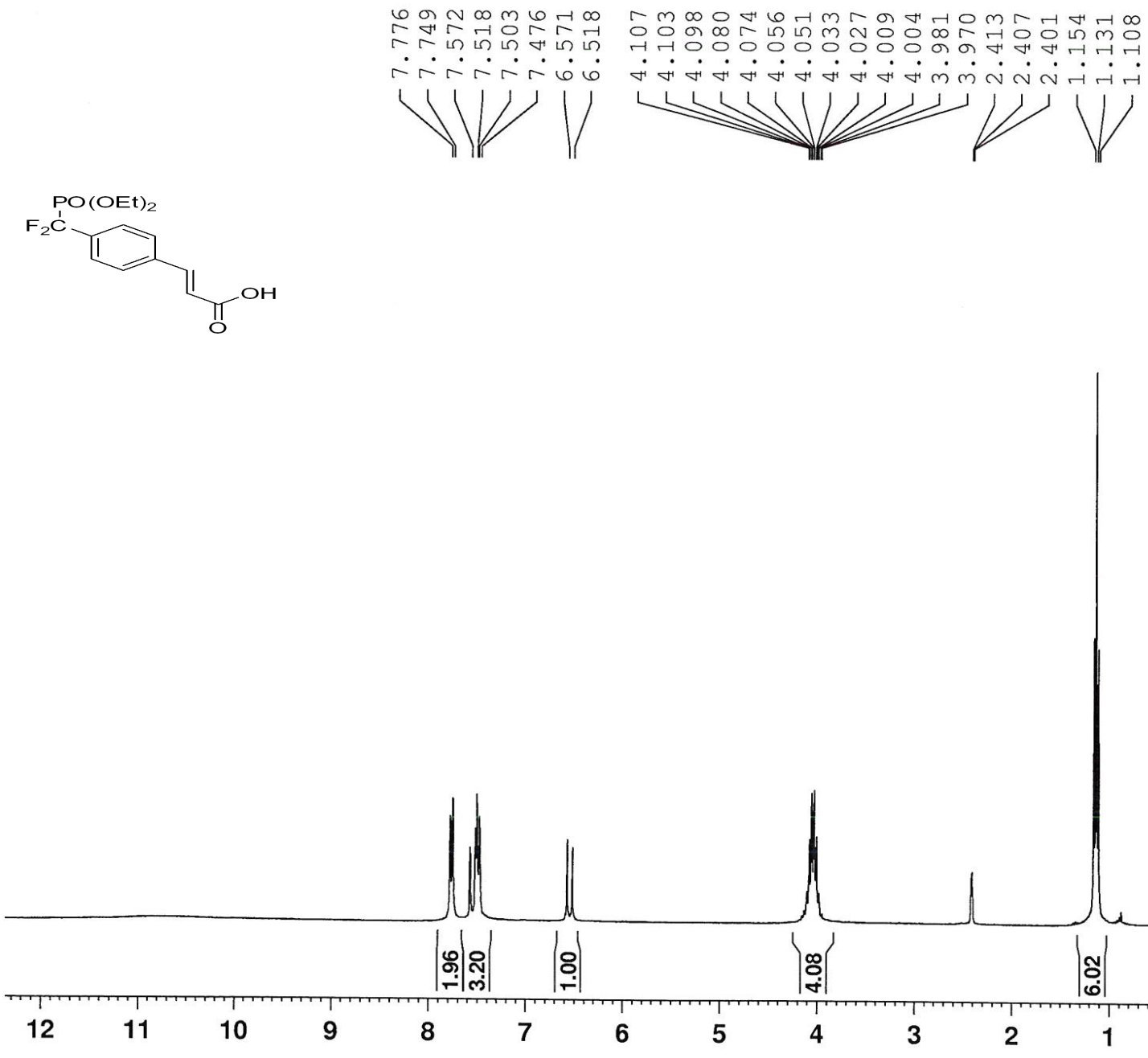
NUC1	1H
P1	10.25
PL1	-1.00
SFO1	300.1318534

F2 - Processing paramete

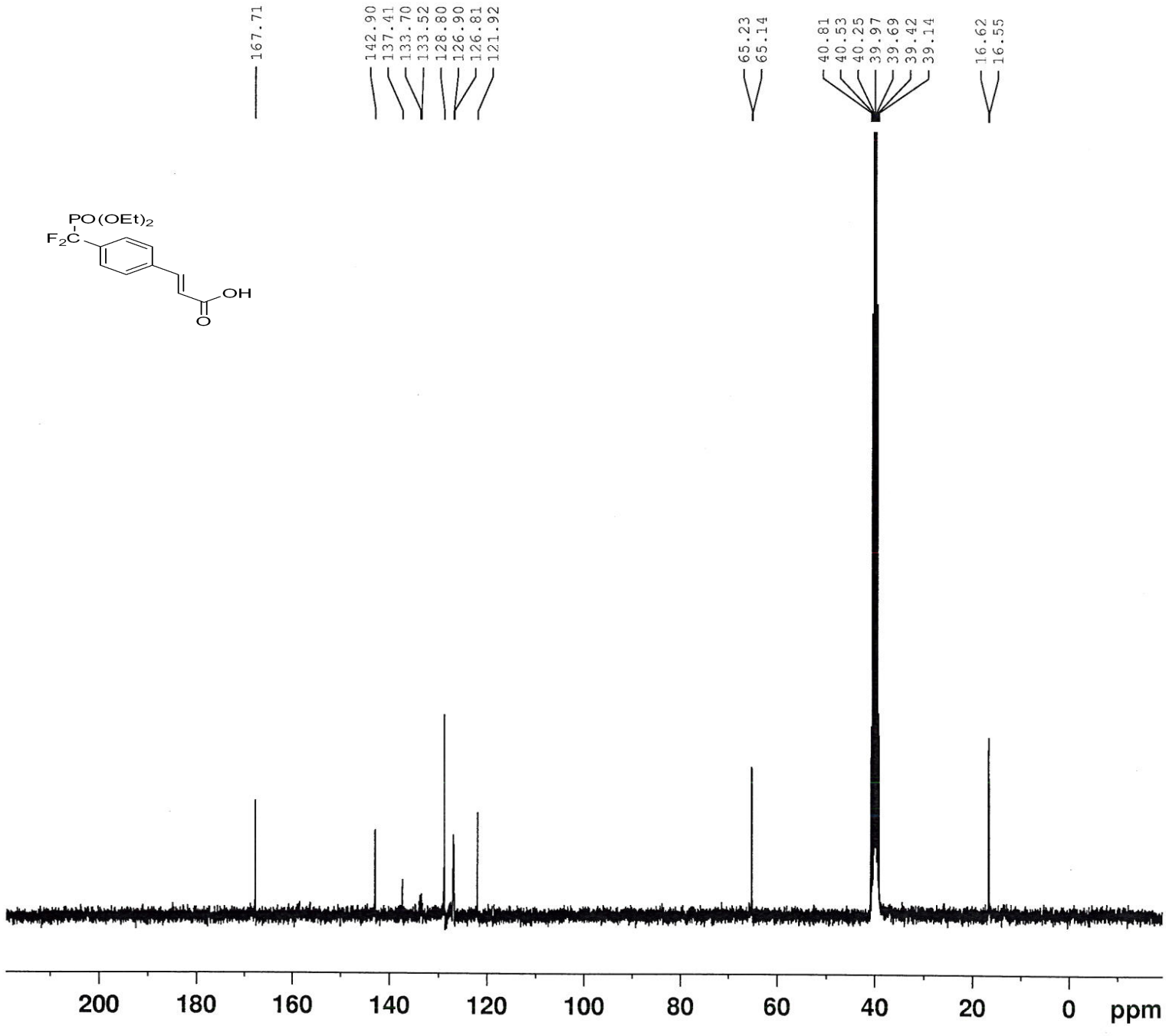
SI	32768
SF	300.1300275
WDW	EM
SSB	0
LB	0.30
GB	0
PC	1.00


3
F2C
PO(OEt)2
O
OtBu
7.641
7.613
7.593
7.560
7.346
7.280
7.213
6.449
6.396
4.267
4.257
4.243
4.233
4.224
4.219
4.209
4.201
4.185
4.177
4.173
4.167
4.161
4.149
4.144
4.139
4.126
4.115
2.163
1.537
1.338
1.315
1.291
5.12
0.99
4.04
8.67
6.09
11
10
9
8
7
6
5
4
3
2
1
0

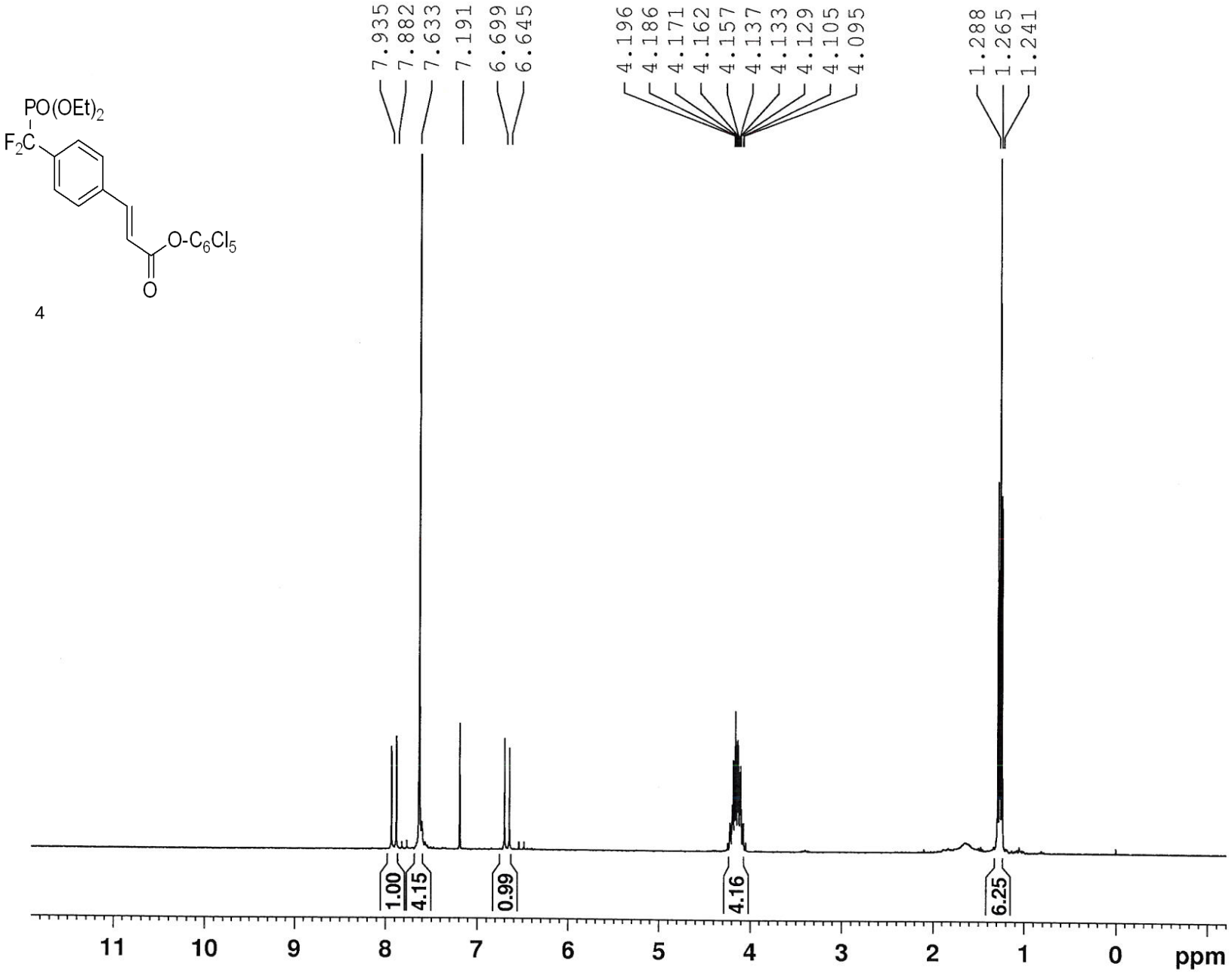

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
3
PO(OEt)2
F2C
O
O'Bu
165.82
142.08
137.08
127.81
126.78
122.17
80.81
77.46
77.23
77.03
76.61
64.88
64.79
28.13
16.32
16.25



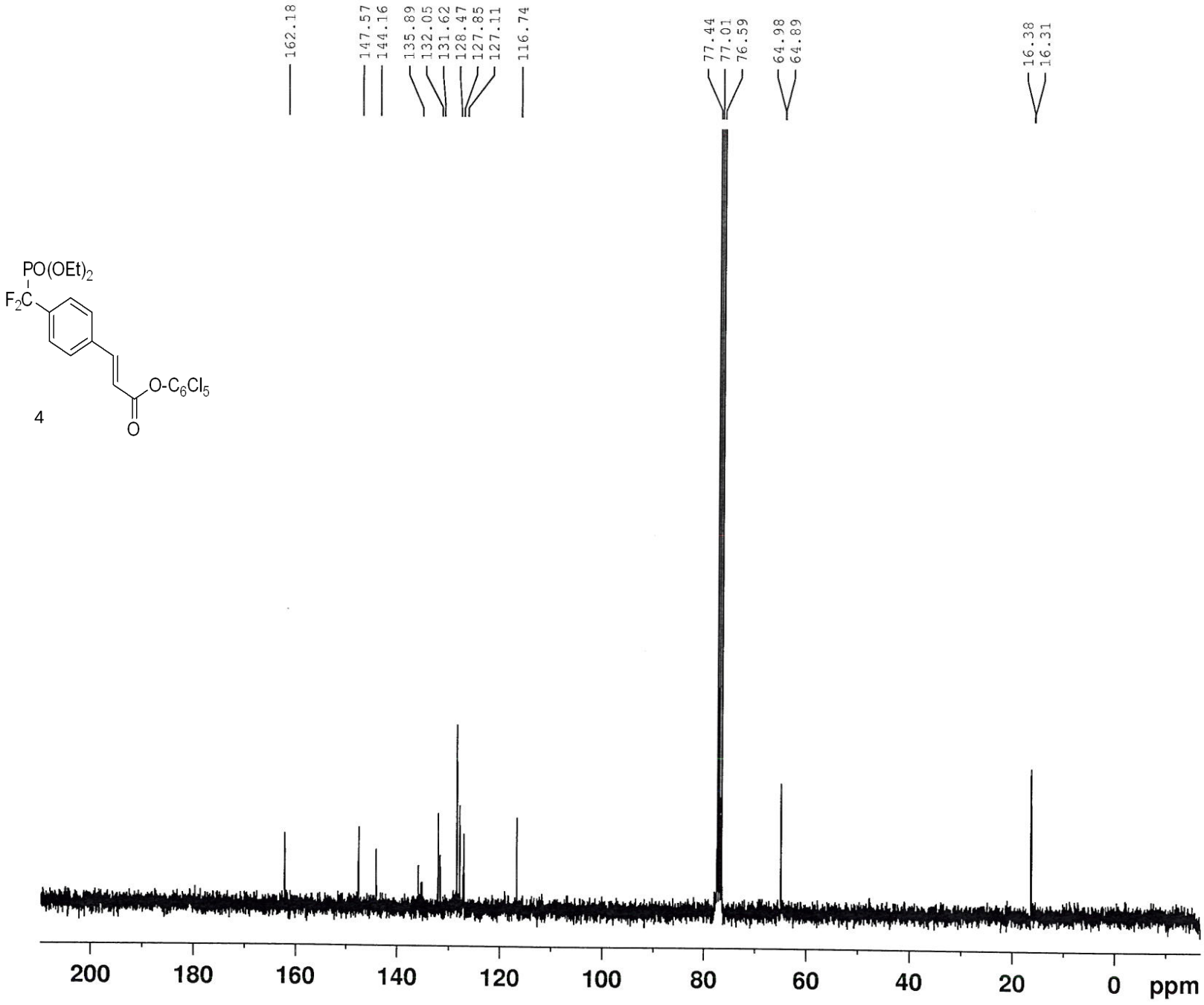

```
Current Data Parameter
NAME                 BPPM-6_C
EXPNO
PROCNO

F2 - Acquisition Param
Date_                2009041
Time                    12.5
INSTRUM                 spec
PROBHD    5 mm QNP 1H/1
PULPROG                 zg3
TD                     6553
SOLVENT                 DMS
NS
DS
SWH                 6172.83
FIDRES              0.09419
AQ                  5.308466
RG                      161.
DW                    81.00
DE                      6.0
TE                      301.
D1                1.0000000
TD0

======== CHANNEL f1 ==
NUC1                       1
P1                      10.2
PL1                     -1.0
SFO1              300.131853

F2 - Processing parame
SI                       3276
SF                300.130027
WDW                         E
SSB
LB                        0.3
GB
PC                        1.0
```



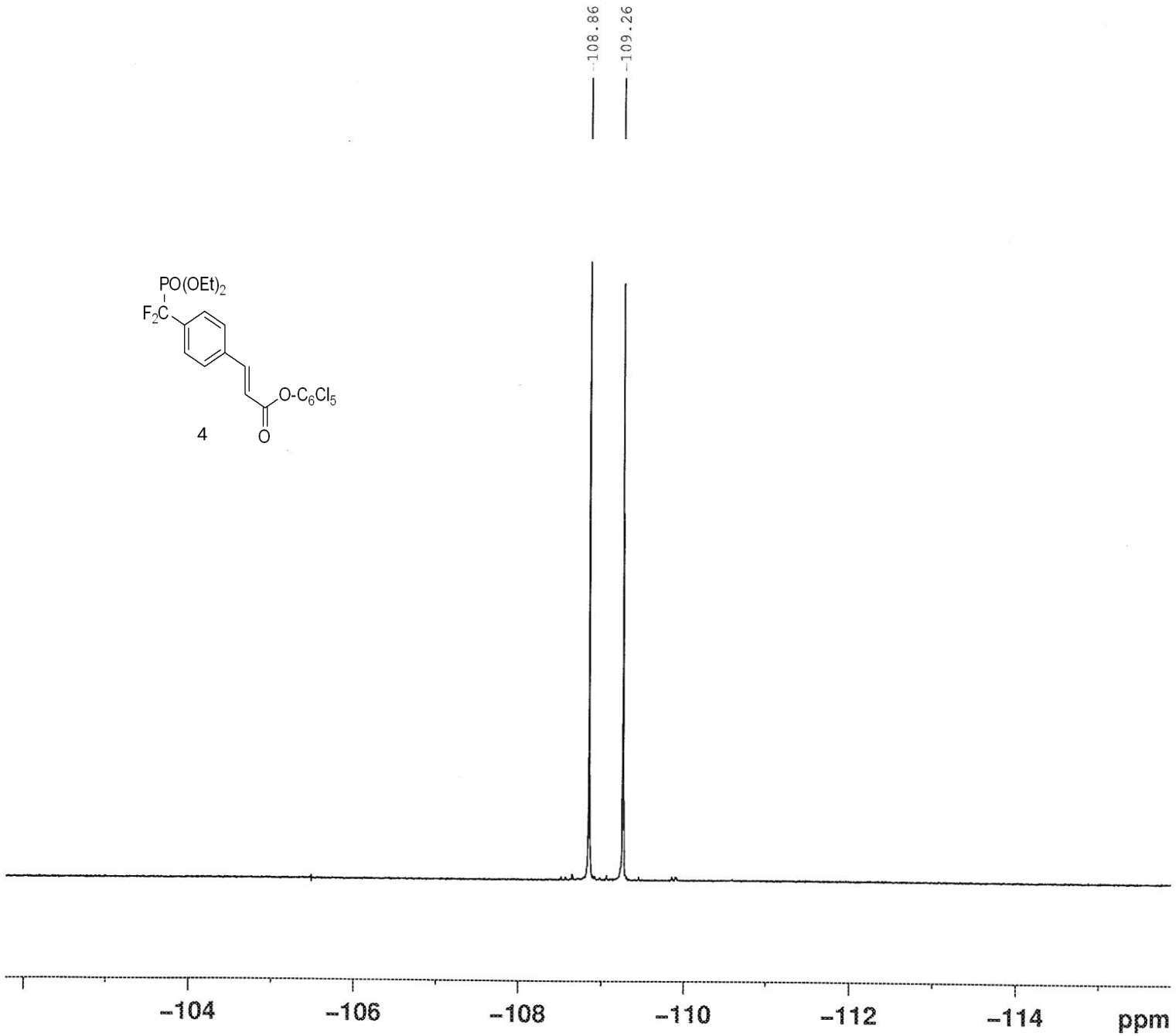

```
Current Data Parameters
NAME                  BPPM-6_OL
EXPNO                         4
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20090417
Time                      13.07
INSTRUM                   spect
PROBHD    5 mm QNP 1H/19
PULPROG                 zgpg30
TD                         65536
SOLVENT                  CDCl3
NS                           735
DS                             4
SWH                  17985.611 Hz
FIDRES                0.274439 Hz
AQ                    1.8219508 sec
RG                        645.1
DW                       27.800 use
DE                        6.00 use
TE                       301.2 K
D1                   2.00000000 sec
d11                  0.03000000 sec
DELTA                1.89999998 sec
TD0                           1

======== CHANNEL f1 =======
NUC1                        13C
P1                       10.00 use
PL1                       1.30 dB
SFO1                75.4752953 MHz

======== CHANNEL f2 =======
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                   100.00 use
PL2                      -1.00 dB
PL12                     19.00 dB
PL13                     19.00 dB
SFO2               300.1312005 MHz

F2 - Processing parameters
SI                         32768
SF                  75.4677490 MHz
WDW                           EM
SSB                            0
LB                        1.00 Hz
GB                             0
PC                         1.40
```



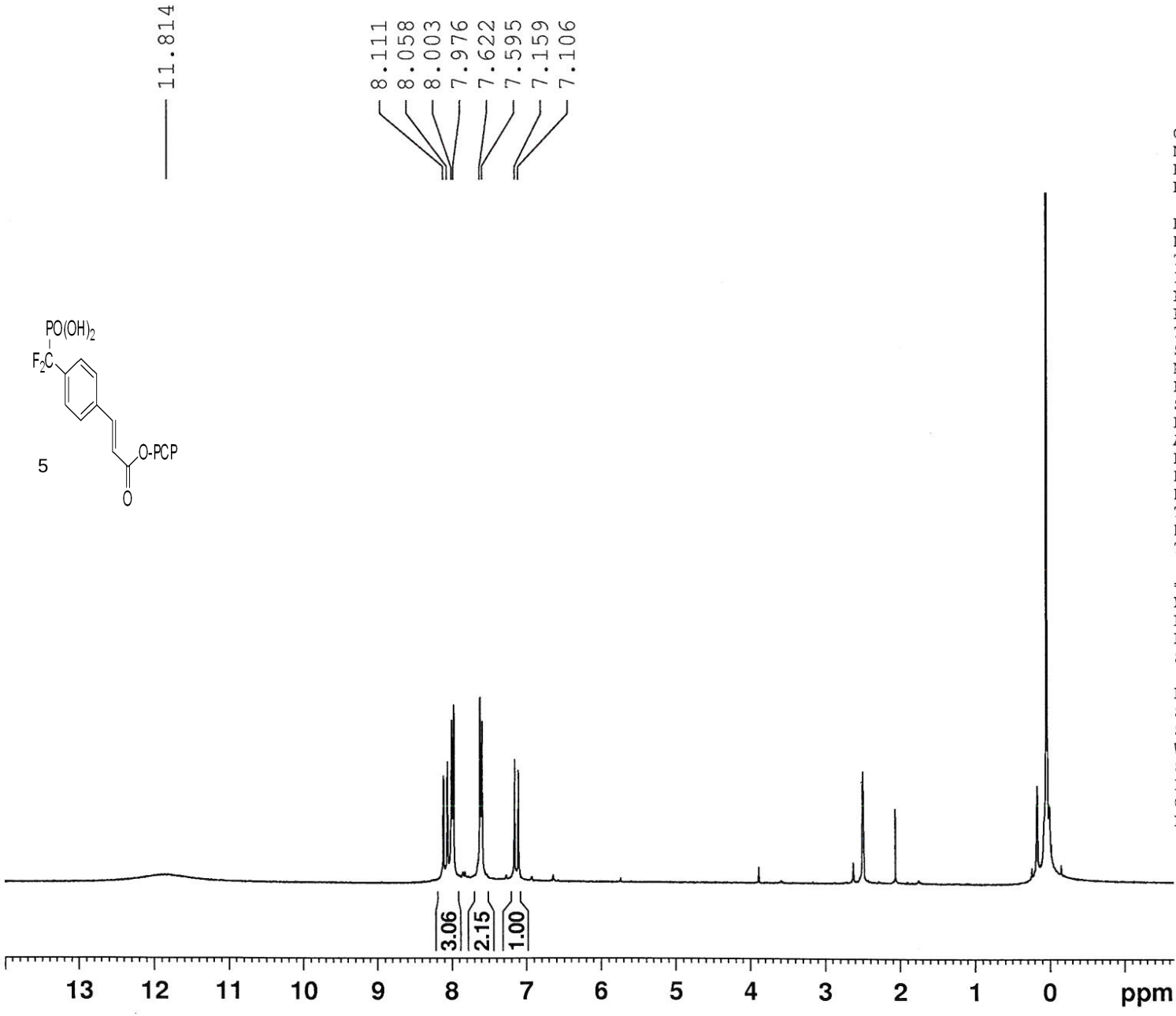

```
Current Data Parameters
NAME        DEDFPCP_ OL-1
EXPNO                  1
PROCNO                 1

F2 - Acquisition Paramet
Date_           20090417
Time               15.41
INSTRUM            spect
PROBHD   5 mm QNP 1H/19
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     4
DS                     2
SWH             6172.839
FIDRES          0.094190
AQ              5.3084660
RG                   362
DW                81.000
DE                 6.00
TE                300.2
D1            1.00000000
TD0                    1

======== CHANNEL f1 ====
NUC1                  1H
P1                 10.25
PL1                -1.00
SFO1          300.1318534

F2 - Processing paramete
SI                 32768
SF          300.1300268
WDW                   EM
SSB                    0
LB                  0.30
GB                     0
PC                  1.00
```



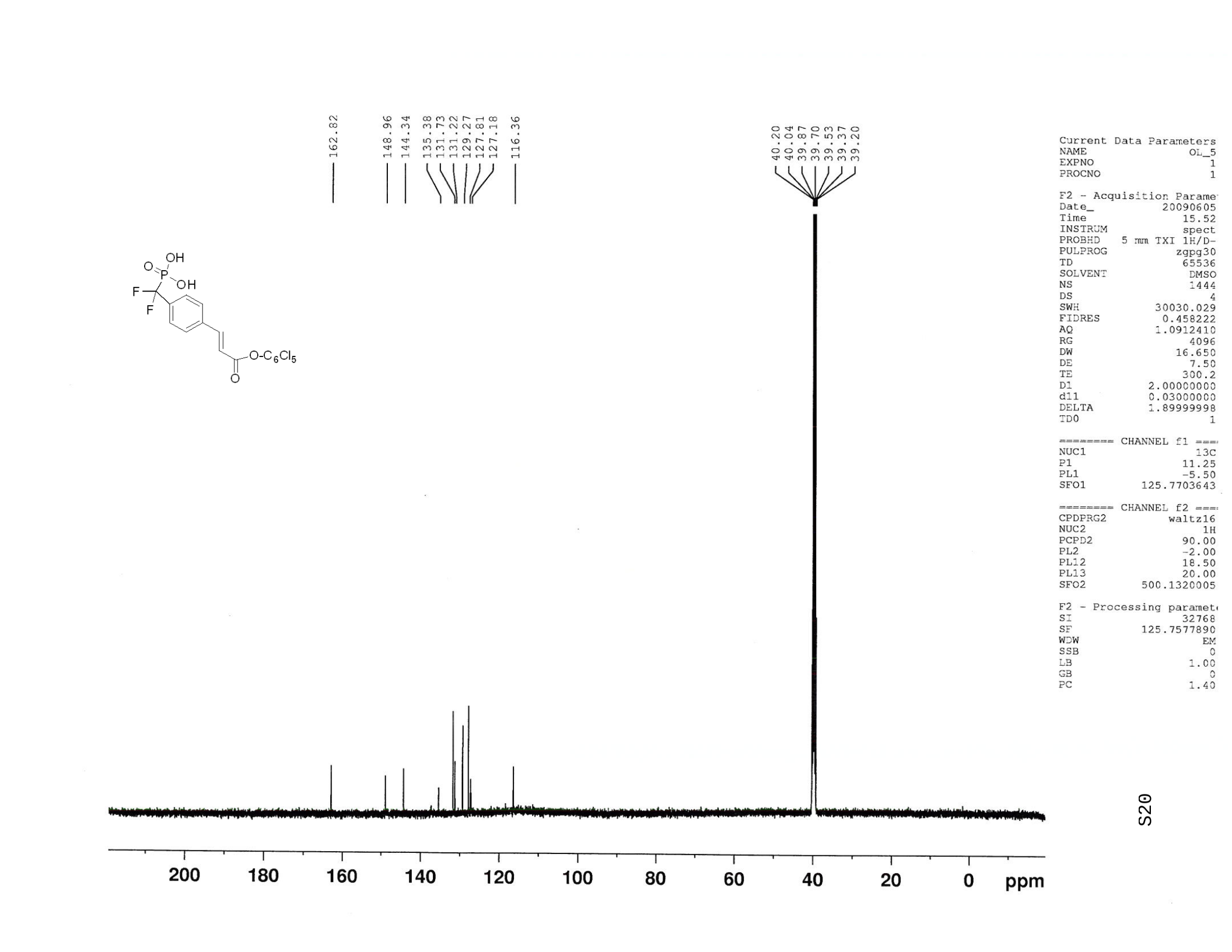

```
Current Data Parameters
NAME       DEDFPCP_ OL-1
EXPNO                 3
PROCNO                1

F2 - Acquisition Paramet
Date_          20090417
Time              16.51
INSTRUM           spect
PROBHD   5 mm QNP 1H/19
PULPROG         zgpg30
TD              65536
SOLVENT          CDCl3
NS                1024
DS                   4
SWH          17985.611
FIDRES        0.274439
AQ            1.8219508
RG                456.1
DW                27.800
DE                 6.00
TE                301.2
D1           2.00000000
d11          0.03000000
DELTA        1.89999998
TD0                   1

======== CHANNEL f1 ====
NUC1                13C
P1                10.00
PL1                1.30
SFO1         75.4752953

======== CHANNEL f2 ====
CPDPRG2         waltz16
NUC2                1H
PCPD2           100.00
PL2              -1.00
PL12             19.00
PL13             19.00
SFO2        300.1312005

F2 - Processing paramete
SI                32768
SF         75.4677490
WDW                EM
SSB                 0
LB               1.00
GB                  0
PC               1.00
```




```
Current Data Parameters
NAME       DEDFPCP_ OL-1
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20090417
Time              15.43
INSTRUM           spect
PROBHD   5 mm QNP 1H/19
PULPROG         zgflqn
TD              131072
SOLVENT           C6D6
NS                  16
DS                   4
SWH           67567.570 Hz
FIDRES        0.515500 Hz
AQ            0.9699828 sec
RG                912.3
DW                7.400 usec
DE                6.00 usec
TE                301.2 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 19F
P1                12.00 usec
PL1               -4.00 dB
SFO1        282.3761148 MHz

F2 - Processing parameters
SI                65536
SF          282.4043550 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```




```
Current Data Parameters
NAME                  BPPM-6_OL
EXPNO                         6
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20090417
Time                      13.55
INSTRUM                   spect
PROBHD    5 mm QNP 1H/19
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                            6
DS                            2
SWH                    6172.839 Hz
FIDRES                 0.094190 Hz
AQ                     5.3084660 sec
RG                        287.4
DW                       81.000 usec
DE                        6.00 usec
TE                        300.2 K
D1                    1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                       10.25 usec
PL1                      -1.00 dB
SFO1                300.1318534 MHz

F2 - Processing parameters
SI                           32768
SF                  300.1300000 MHz
WDW                             EM
SSB                             0
LB                        0.30 Hz
GB                              0
PC                        1.00
```



Current Data Parameters

NAME	OL_5
EXPNO	1
PROCNO	1

F2 - Acquisition Parame

Date_	20090605
Time	15.52
INSTRUM	spect
PROBHD	5 mm TXI 1H/D-
PULPROG	zgpg30
TD	65536
SOLVENT	DMSO
NS	1444
DS	4
SWH	30030.029
FIDRES	0.458222
AQ	1.0912410
RG	4096
DW	16.650
DE	7.50
TE	300.2
D1	2.00000000
d11	0.03000000
DELTA	1.89999998
TD0	1

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

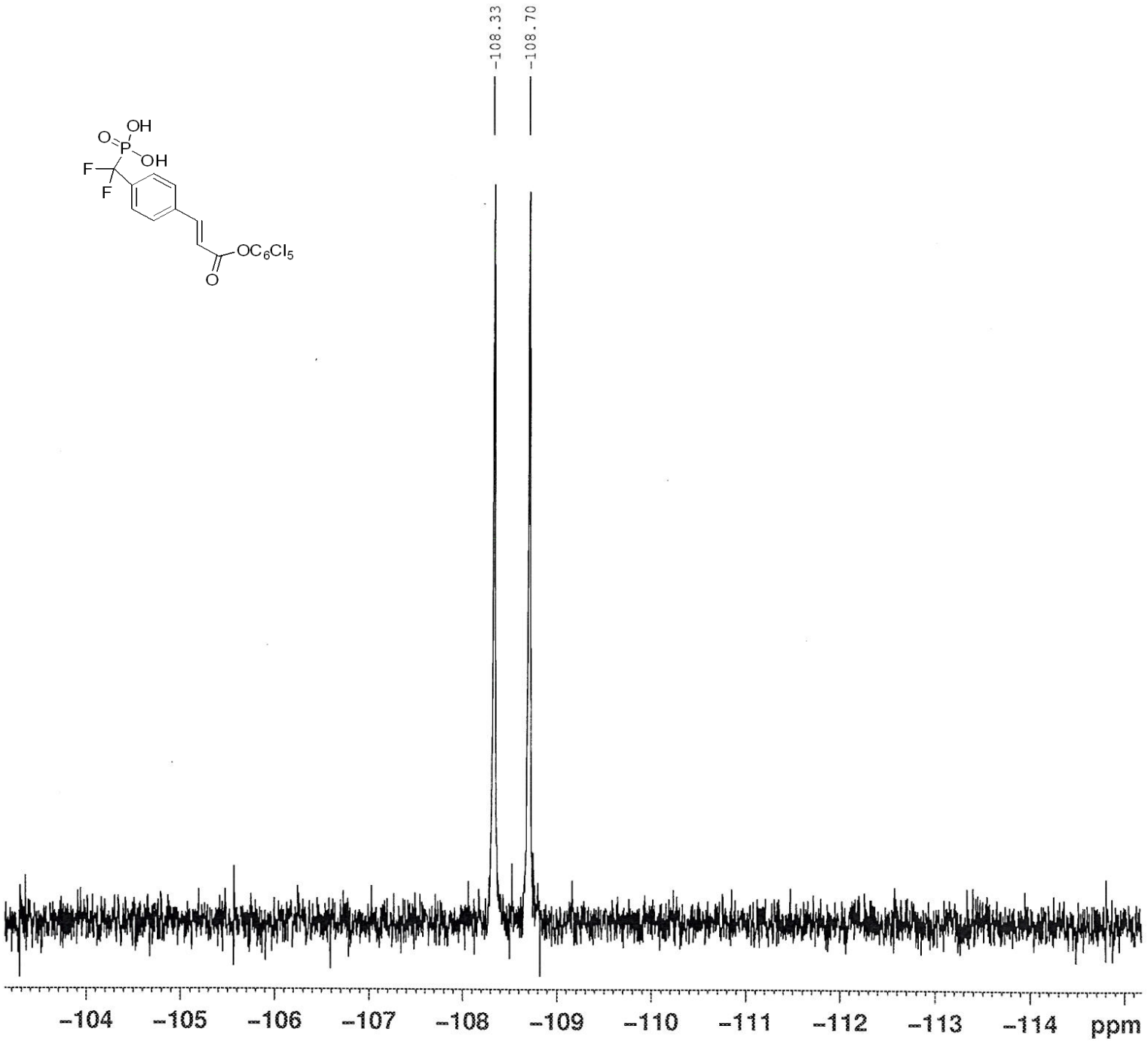
NUC1	13C
P1	11.25
PL1	-5.50
SFO1	125.7703643

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

CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00
PL2	-2.00
PL12	18.50
PL13	20.00
SFO2	500.1320005

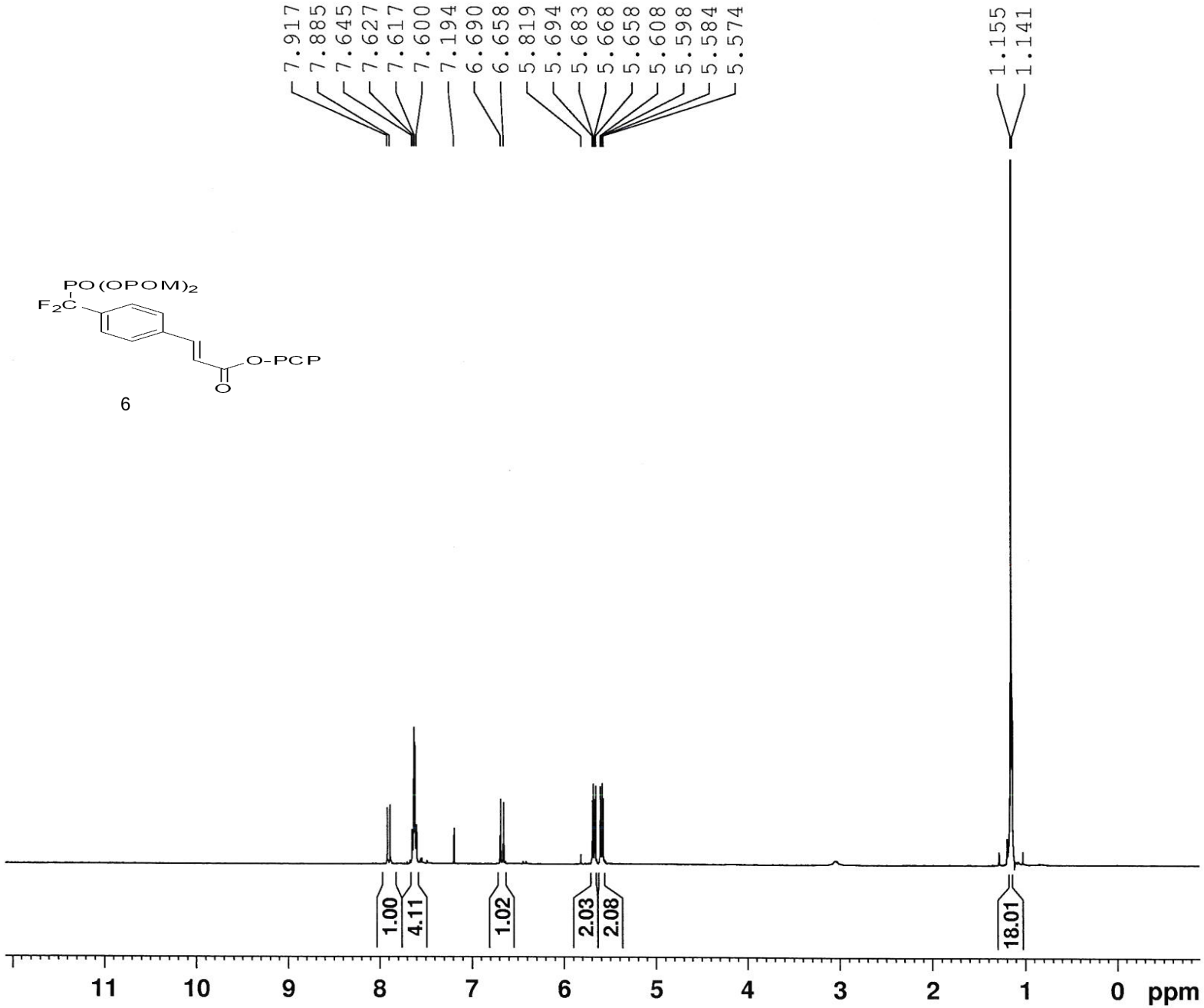
F2 - Processing paramet

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

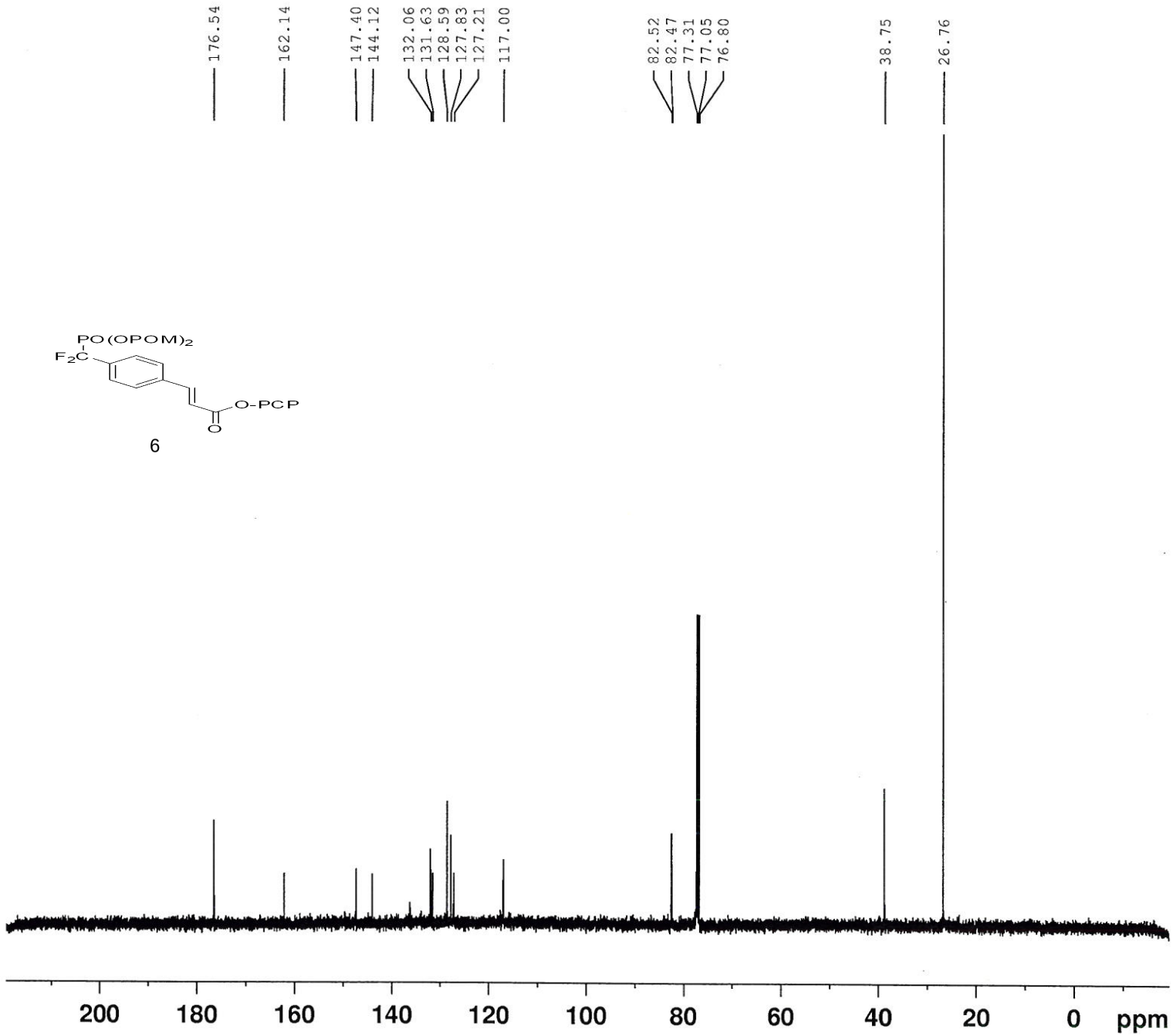



```
Current Data Parameters
NAME                  bppm6-5
EXPNO                       4
PROCNO                      1

F2 - Acquisition Parameters
Date_              20090605
Time                 10.51
INSTRUM              spect
PROBHD    5 mm QNP 1H/19
PULPROG           zgfhigqn
TD                  131072
SOLVENT               CDCl3
NS                       64
DS                        4
SWH               67567.570 Hz
FIDRES             0.515500 Hz
AQ                 0.9699828 sec
RG                     4096
DW                    7.400 usec
DE                    6.00 usec
TE                   300.2 K
D1                1.00000000 sec
d11               0.03000000 sec
d12               0.00002000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     19F
P1                    12.00 usec
PL1                   -4.00 dB
SFO1           282.3761148 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                100.00 usec
PL2                   -1.00 dB
PL12                  19.00 dB
SFO2           300.1312005 MHz

F2 - Processing parameters
SI                       65536
SF             282.4043550 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

6

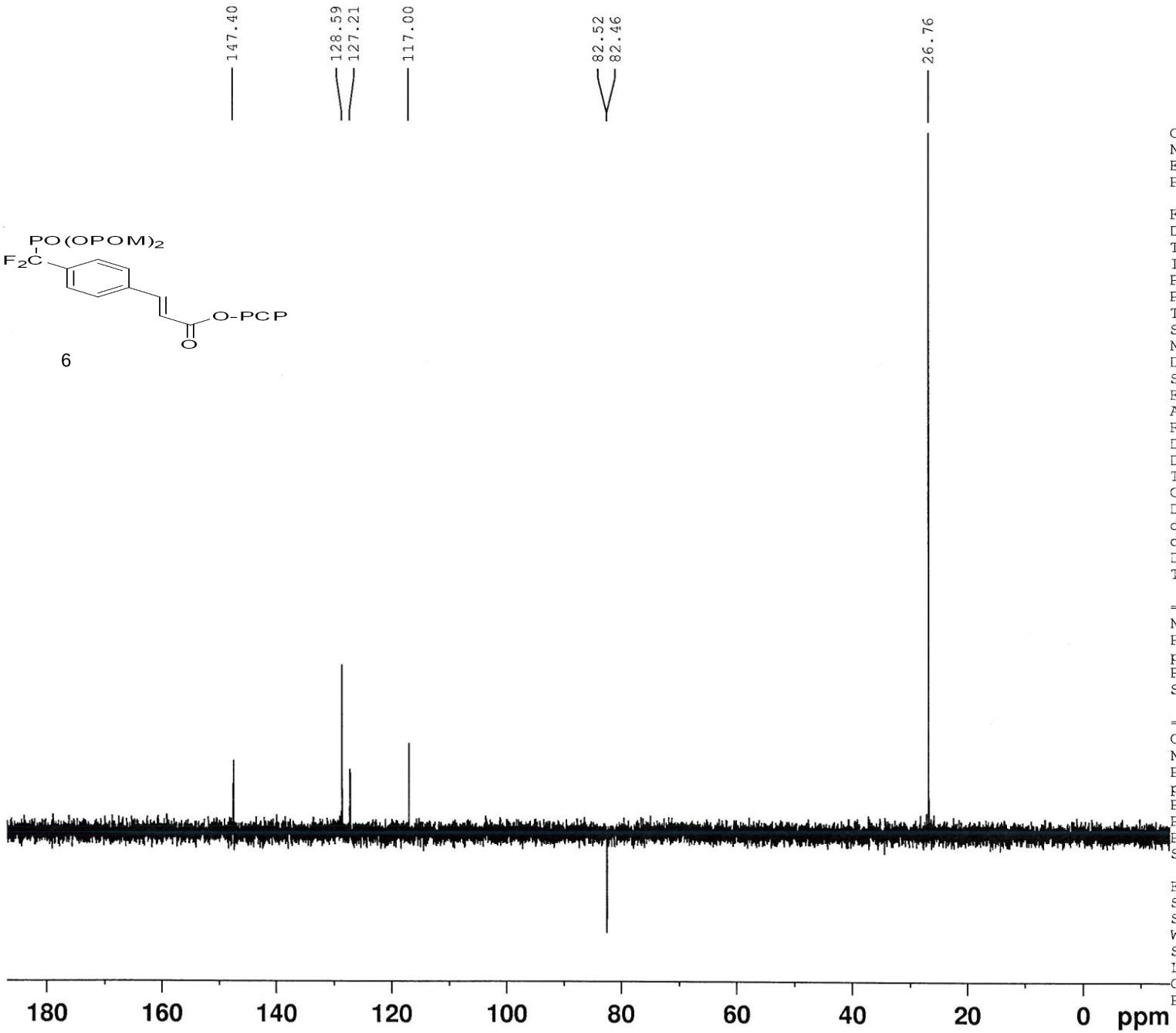



```
Current Data Parameters
NAME                 BPPCPCINN
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parame
Date_                 20090421
Time                      8.34
INSTRUM                  spect
PROBHD     5 mm TXI 1H/D-
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                           4
DS                           2
SWH                  10330.578
FIDRES                0.157632
AQ                    3.1720407
RG                         40.3
DW                       48.400
DE                        7.50
TE                       298.2
D1                  1.00000000
TD0                          1

======== CHANNEL f1 ===
NUC1                        1H
P1                        8.00
PL1                      -2.00
SFO1               500.1330885

F2 - Processing paramet
SI                       32768
SF                 500.1300463
WDW                         EM
SSB                          0
LB                        0.30
GB                           0
PC                        1.00
```



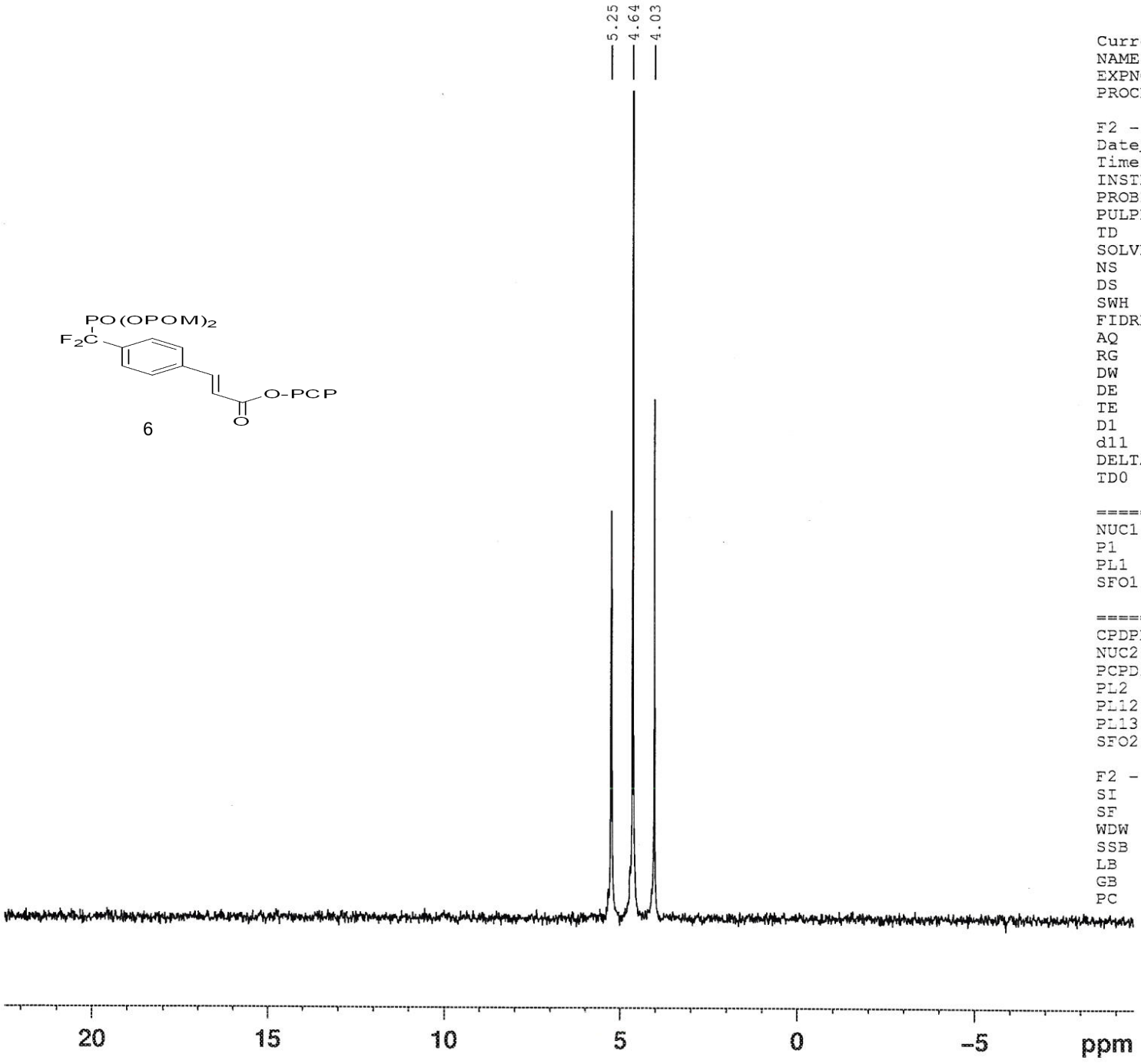

```
Current Data Parameters
NAME                  BPPCPCINN
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameter
Date_                  20090421
Time                       8.44
INSTRUM                   spect
PROBHD    5 mm TXI 1H/D-
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         150
DS                           4
SWH                  30030.029 Hz
FIDRES                0.458222 Hz
AQ                    1.0912410 se
RG                       1448.2
DW                       16.650 us
DE                        7.50 us
TE                       298.2 K
D1                 2.00000000 se
d11                0.03000000 se
DELTA              1.89999998 se
TD0                          1

======== CHANNEL f1 ======
NUC1                       13C
P1                       11.25 us
PL1                      -5.50 dB
SFO1               125.7703643 MH

======== CHANNEL f2 ======
CPDPRG2                waltz16
NUC2                       1H
PCPD2                    90.00 us
PL2                      -2.00 dB
PL12                     18.50 dB
PL13                     20.00 dB
SFO2               500.1320005 MH

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

6

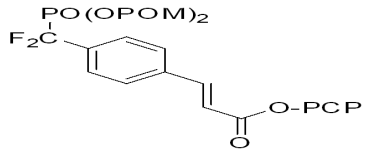



```
Current Data Parameters
NAME                 BPPCPCINN
EXPNO                        5
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20090421
Time                     10.02
INSTRUM                  spect
PROBHD    5 mm TXI 1H/D-
PULPROG                dept135
TD                       65536
SOLVENT                  CDCl3
NS                          31
DS                           4
SWH                30030.029 Hz
FIDRES             0.458222 Hz
AQ                 1.0912410 sec
RG                    9195.2
DW                   16.650 usec
DE                    7.50 usec
TE                    298.2 K
CNST2             145.0000000
D1                 2.00000000 sec
d2                 0.00344828 sec
d12                0.00002000 sec
DELTA              0.00001432 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                      13C
P1                     11.25 usec
p2                     22.50 usec
PL1                    -5.50 dB
SFO1              125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
P3                      8.50 usec
p4                     17.00 usec
PCPD2                  90.00 usec
PL2                    -2.00 dB
PL12                   18.50 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
```

6

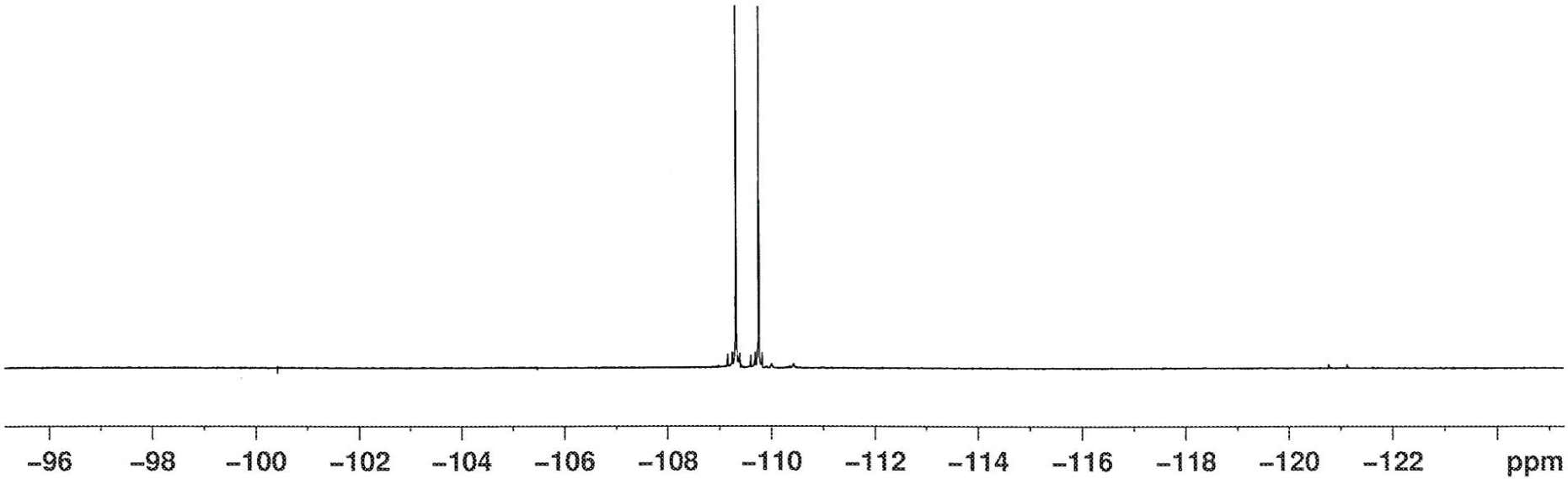


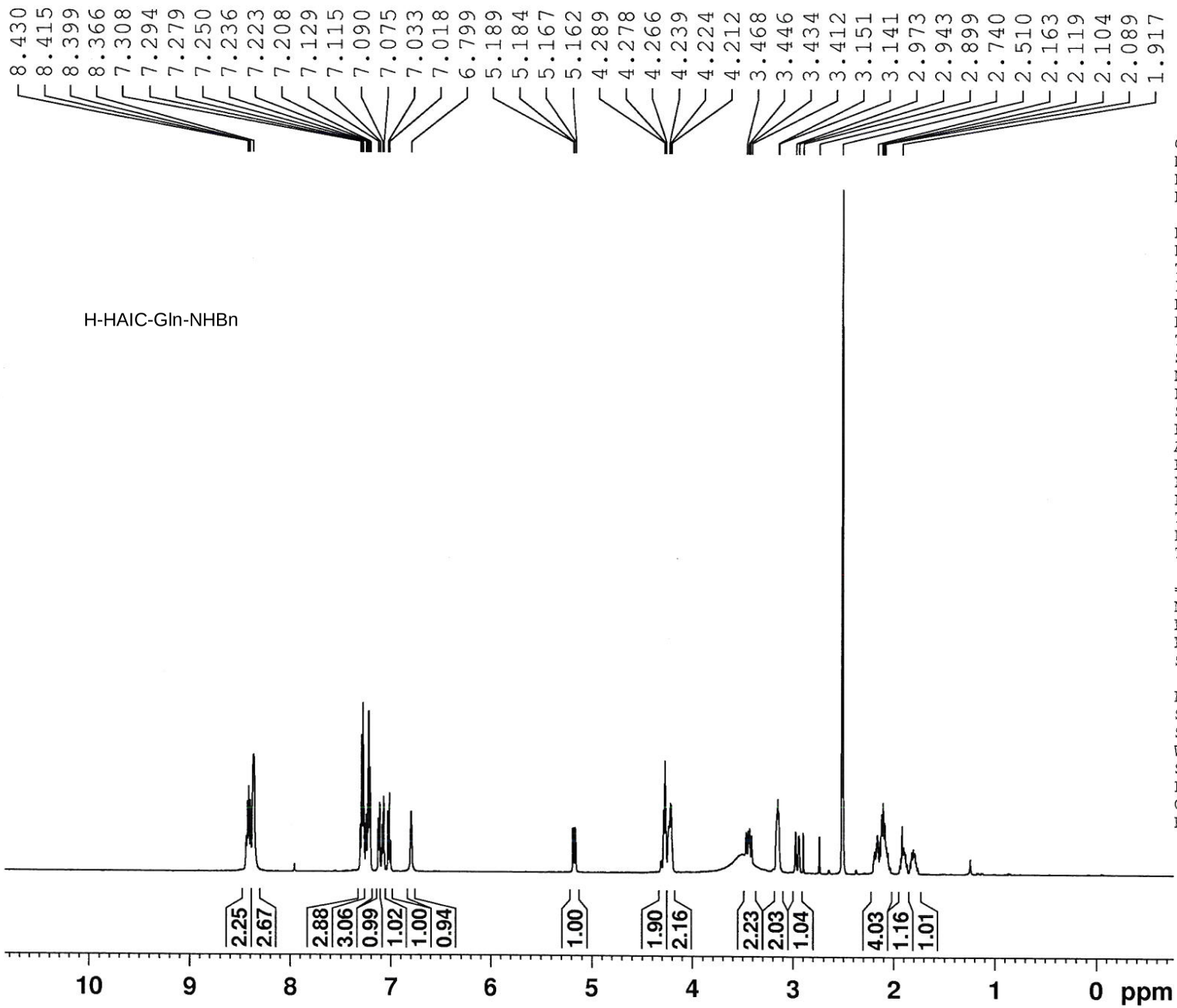

```
Current Data Parameters
NAME            BPPCPCINN
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_            20090421
Time                9.58
INSTRUM            spect
PROBHD   5 mm TXI 1H/D-
PULPROG           zgpg30
TD                 65536
SOLVENT          Acetone
NS                    16
DS                     4
SWH            80645.164 Hz
FIDRES          1.230548 Hz
AQ             0.4063794 sec
RG               20642.5
DW                 6.200 usec
DE                 7.50 usec
TE                298.2 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  31P
P1                 15.00 usec
PL1                -4.50 dB
SFO1         202.4462121 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -2.00 dB
PL12               18.50 dB
PL13               20.00 dB
SFO2         500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF           202.4563350 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



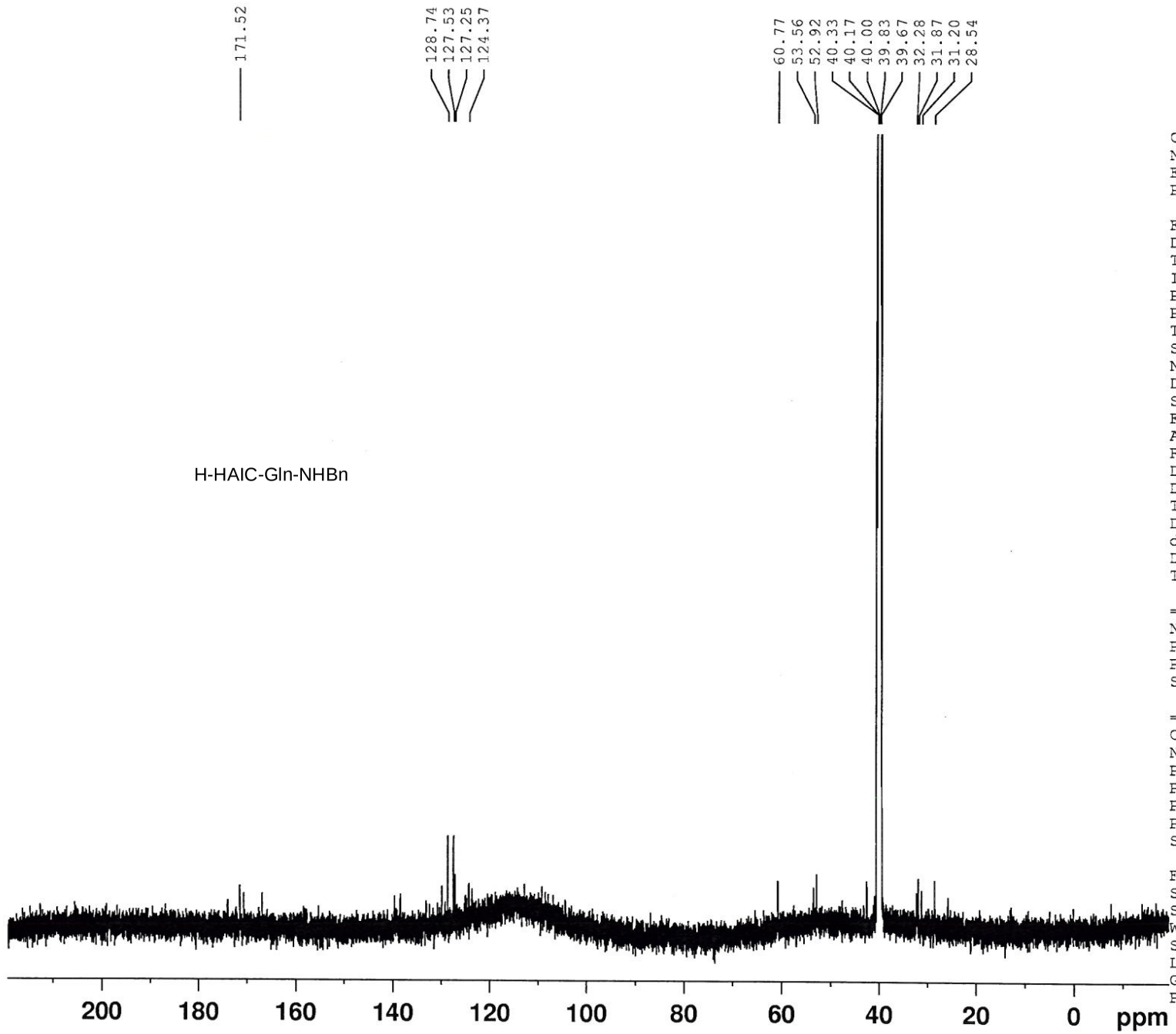
6

-30872.09
-30995.53



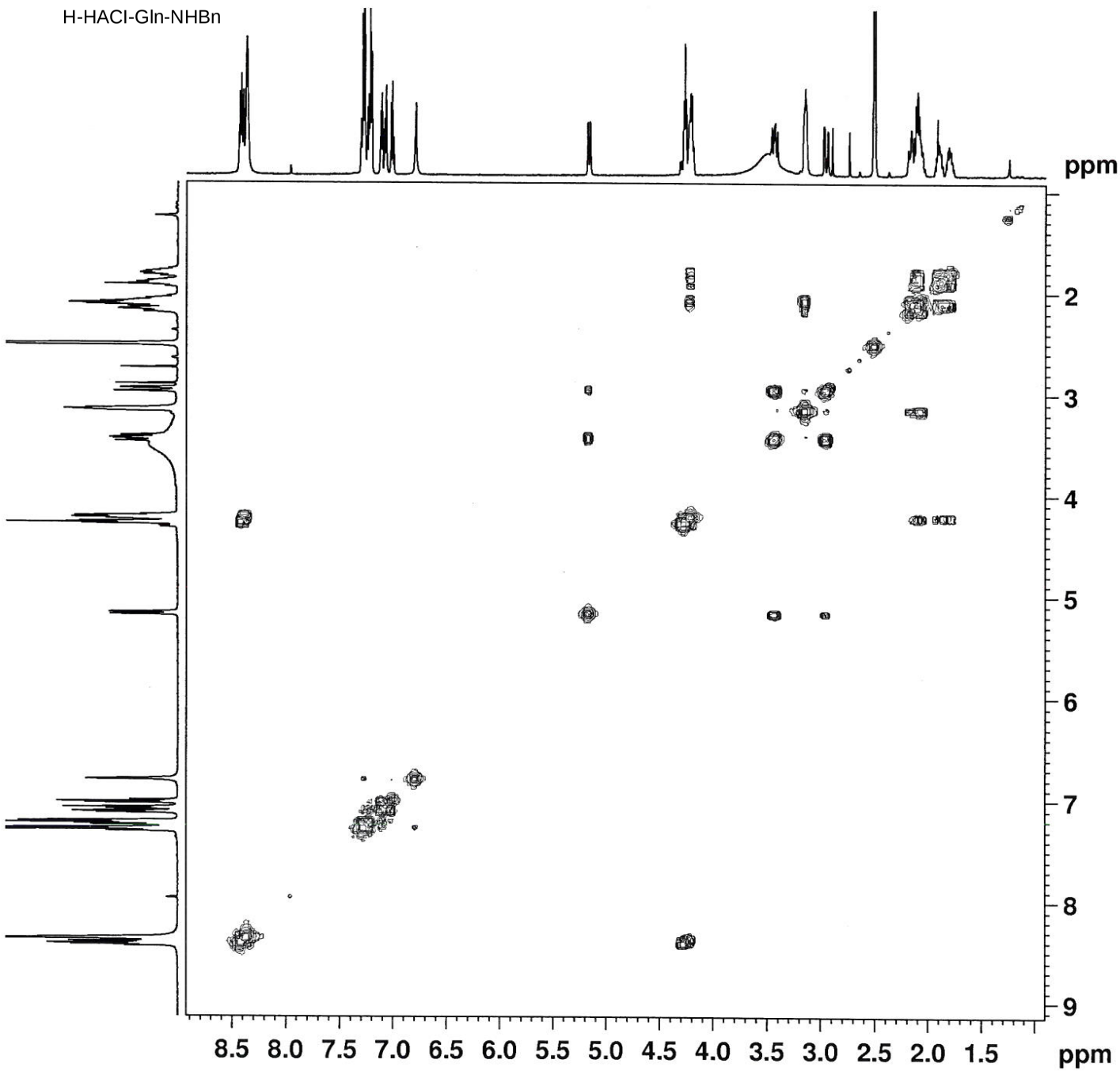



```
Current Data Parameters
NAME                      69G
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20090414
Time                  16.50
INSTRUM               spect
PROBHD    5 mm TXI 1H/D-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH              10330.578 Hz
FIDRES            0.157632 Hz
AQ                3.1720407 sec
RG                      256
DW                 48.400 usec
DE                  7.50 usec
TE                  299.2 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                    8.75 usec
PL1                  -2.00 dB
SFO1          500.1330885 MHz

F2 - Processing parameters
SI                    32768
SF            500.1300000 MHz
WDW                      EM
SSB                       0
LB                  0.30 Hz
GB                        0
PC                     1.00
```

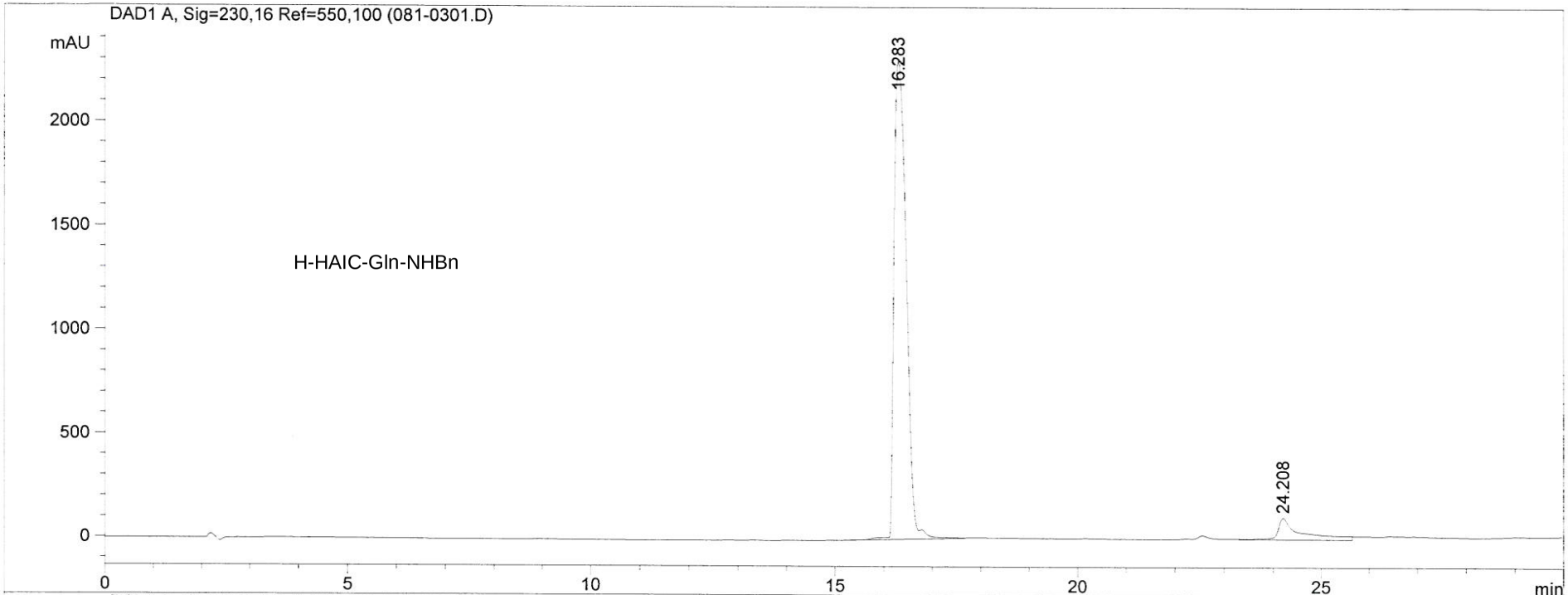


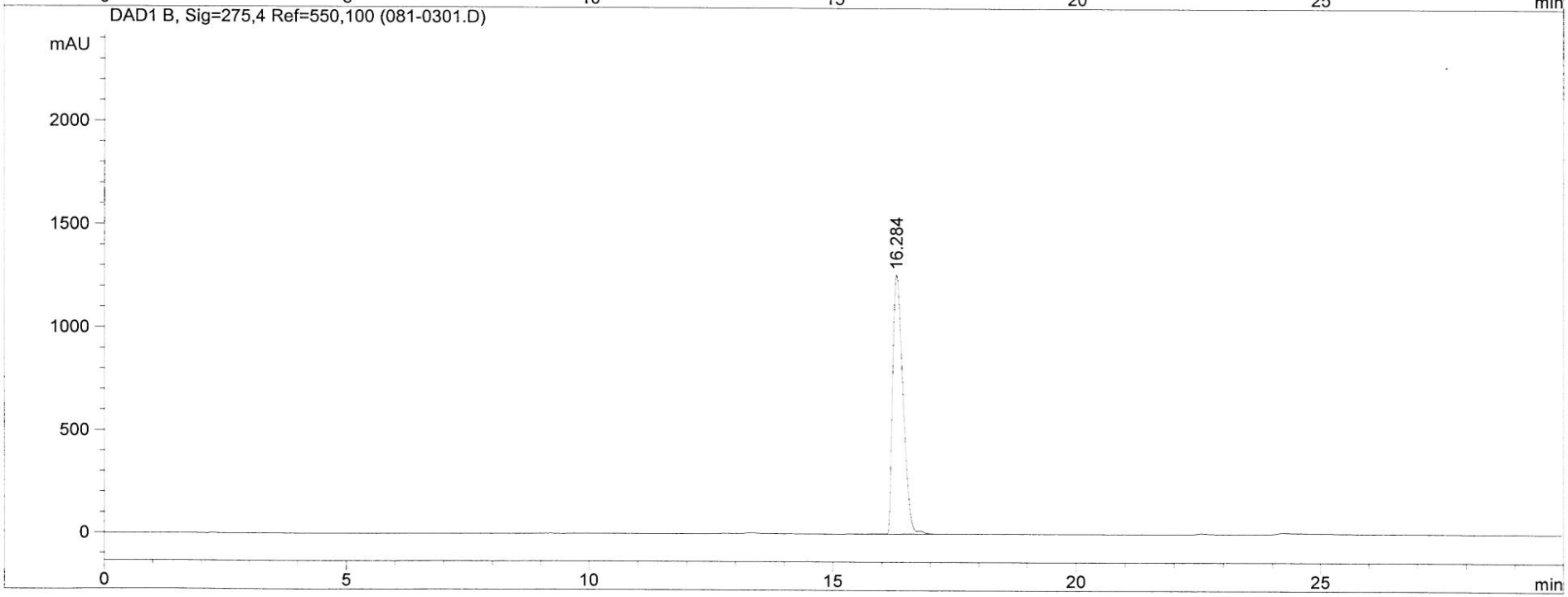

```
Current Data Parameters
NAME                      69G
EXPNO                       3
PROCNO                      1

F2 - Acquisition Parameters
Date_              20090414
Time                  17.03
INSTRUM               spect
PROBHD    5 mm TXI 1H/D-
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                    15360
DS                        4
SWH              30030.029 Hz
FIDRES            0.458222 Hz
AQ                1.0912410 sec
RG                   2896.3
DW                  16.650 usec
DE                   7.50 usec
TE                  299.2 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                  11.25 usec
PL1                 -5.50 dB
SFO1          125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2               90.00 usec
PL2                 -2.00 dB
PL12                18.50 dB
PL13                20.00 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
```

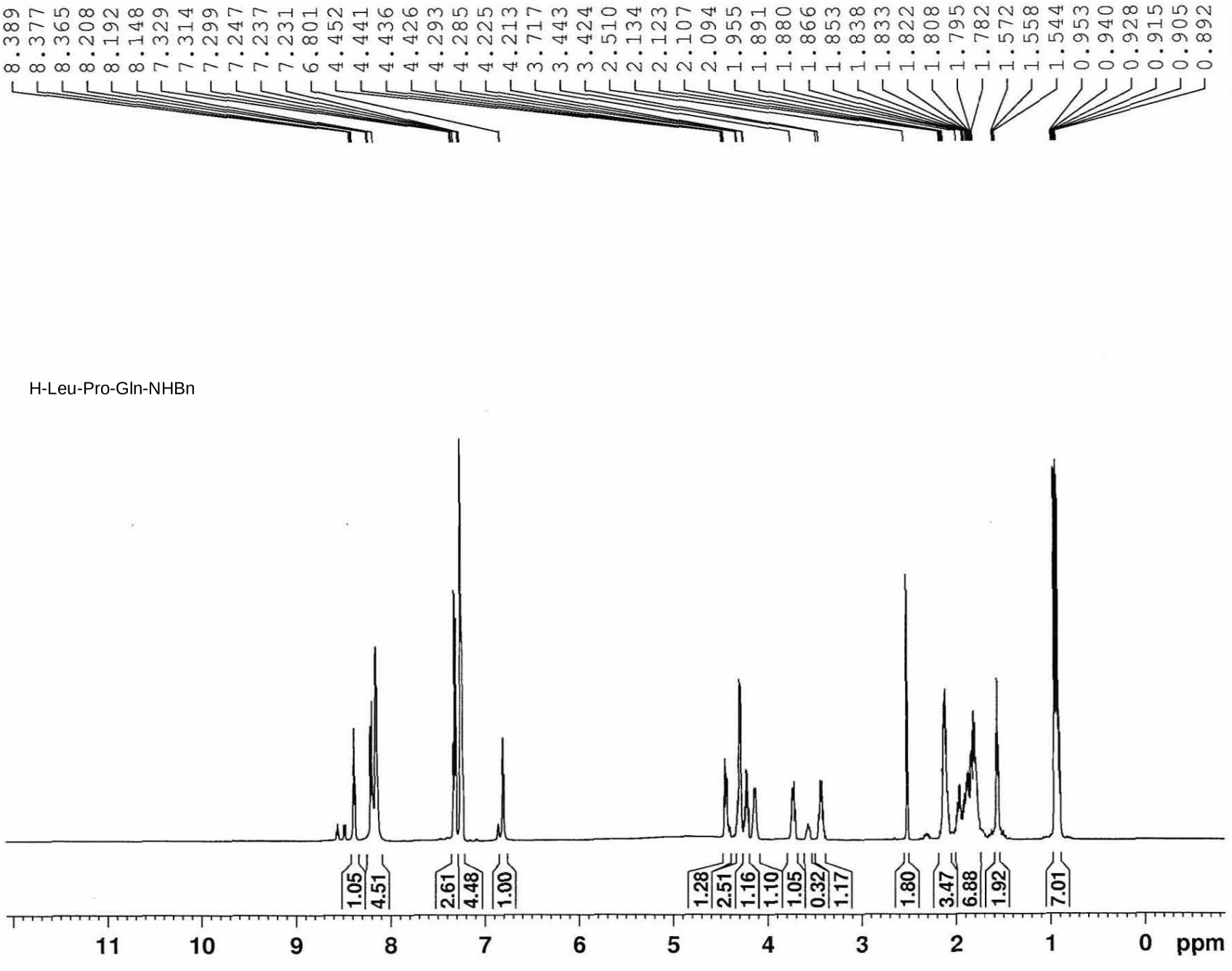
H-HACl-Gln-NHBn

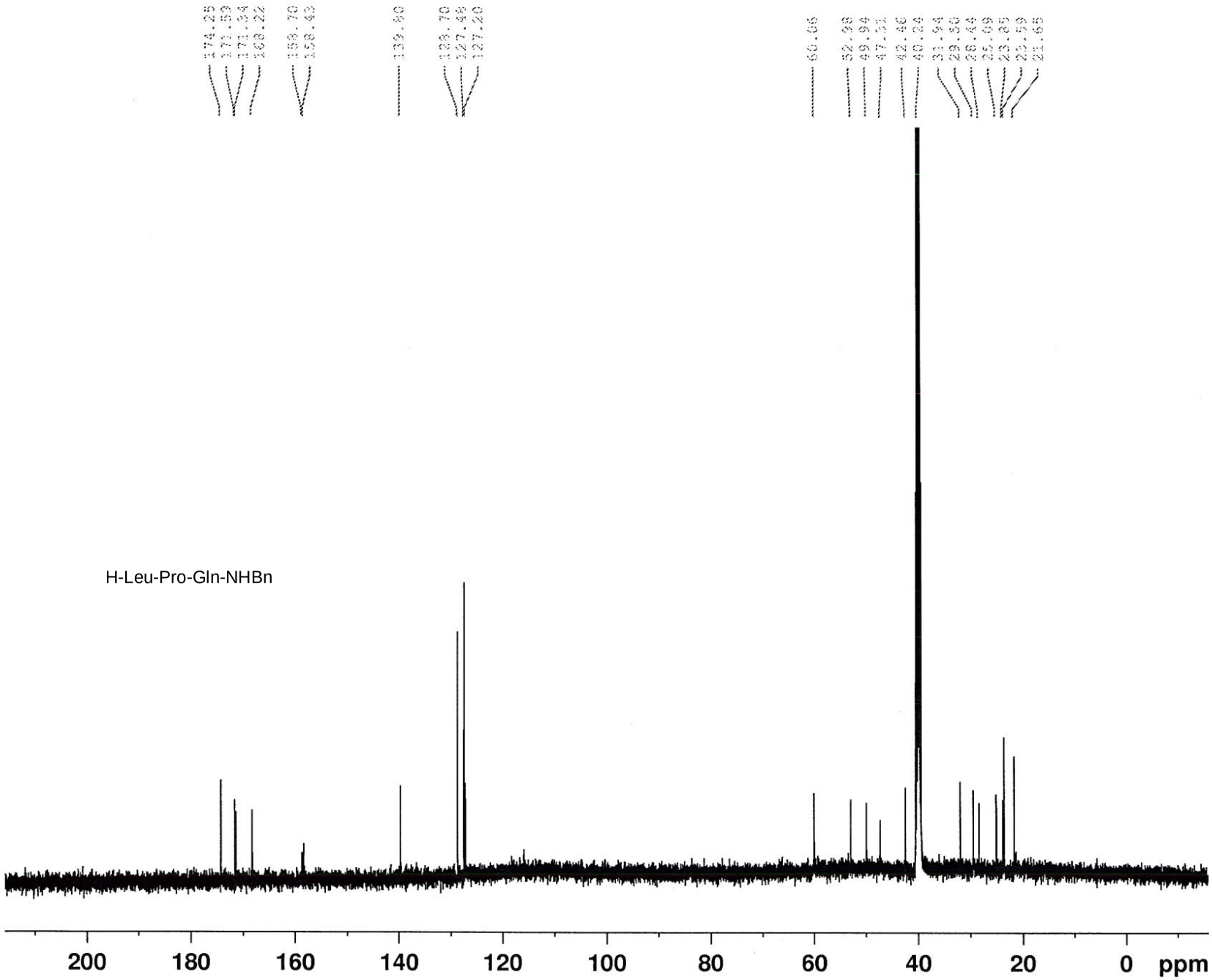



```
Current Data Parameters
NAME                      69G
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20090414
Time                  16.51
INSTRUM                spect
PROBHD    5 mm TXI 1H/D-
PULPROG           cosygpqf
TD                    2048
SOLVENT                DMSO
NS                        2
DS                        8
SWH                6666.667 Hz
FIDRES             3.255208 Hz
AQ                 0.1537250 sec
RG                       64
DW                   75.000 usec
DE                    7.50 usec
TE                   299.2 K
d0                 0.00000300 sec
D1                 1.48689198 sec
d13                0.00000400 sec
D16                0.00020000 sec
IN0                0.00015000 sec

======== CHANNEL f1 ========
NUC1                      1H
P0                    8.75 usec
P1                    8.75 usec
PL1                  -2.00 dB
SFO1           500.1330069 MHz

====== GRADIENT CHANNEL =====
GPNAM1            SINE.100
GPNAM2            SINE.100
GPZ1                 10.00 %
GPZ2                 10.00 %
P16                1000.00 usec

F1 - Acquisition parameters
ND0                       1
TD                      128
SFO1             500.133 MHz
FIDRES          52.083332 Hz
SW                13.330 ppm
FnMODE                 QF

F2 - Processing parameters
SI                     1024
SF            500.1300000 MHz
WDW                    SINE
SSB                       0
LB                     0.00 Hz
GB                        0
PC                     1.00

F1 - Processing parameters
SI                     1024
MC2                      QF
SF            500.1300000 MHz
WDW                    SINE
SSB                       0
LB                     0.00 Hz
GB                        0
```

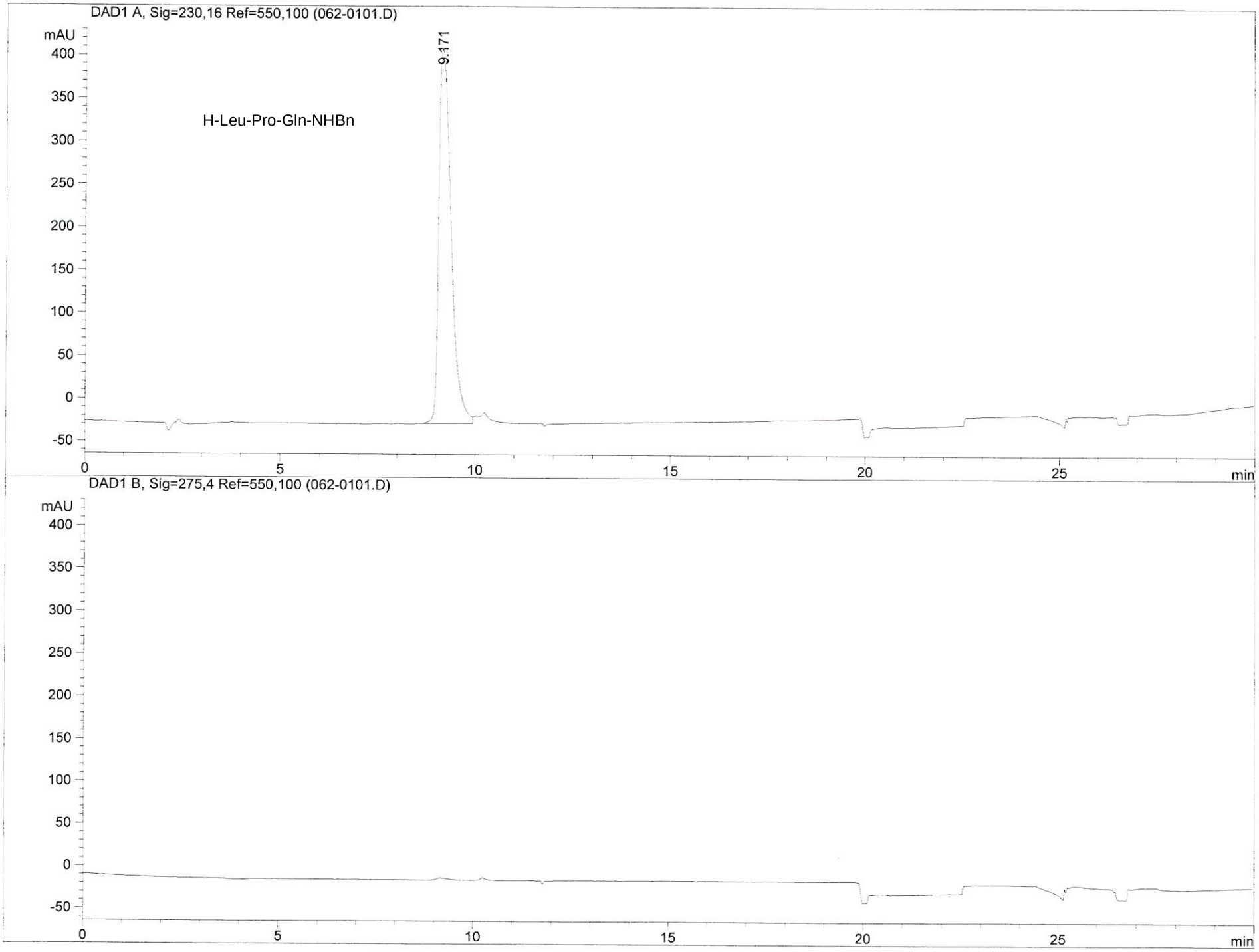

DAD1 A, Sig=230,16 Ref=550,100 (081-0301.D)
mAU
2000
1500
1000
500
0
16.283
24.208
H-HAIC-Gln-NHBn
0
5
10
15
20
25
min


DAD1 B, Sig=275,4 Ref=550,100 (081-0301.D)
mAU
2000
1500
1000
500
0
16.284
0
5
10
15
20
25
min

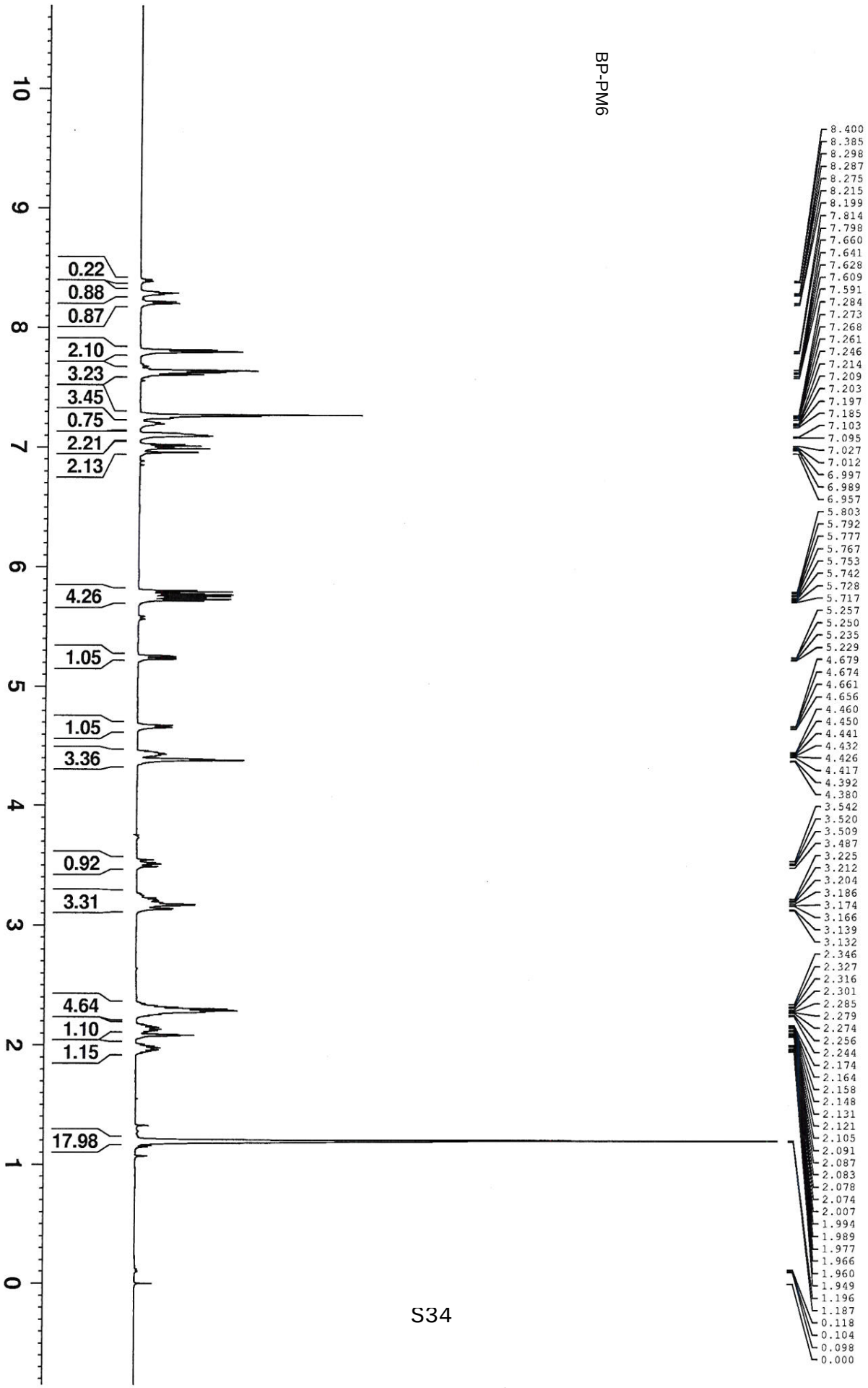

8.389
8.377
8.365
8.208
8.192
8.148
7.329
7.314
7.299
7.247
7.237
7.231
6.801
4.452
4.441
4.436
4.426
4.293
4.285
4.225
4.213
3.717
3.443
3.424
2.510
2.134
2.123
2.107
2.094
1.955
1.891
1.880
1.866
1.853
1.838
1.833
1.822
1.808
1.795
1.782
1.572
1.558
1.544
0.953
0.940
0.928
0.915
0.905
0.892
Current Data Parameter
NAME 27
EXPNO
PROCNO
F2 - Acquisition Parar
Date_ 200905
Time 11.
INSTRUM spe
PROBHD 5 mm TXI 1H/
PULPROG zg
TD 655
SOLVENT CDC
NS
DS
SWH 10330.5
FIDRES 0.1576
AQ 3.17204
RG 101.
DW 48.4
DE 7.
TE 298.
D1 1.000000
TD0
======== CHANNEL f1 ==
NUC1
P1 8.
PL1 -2.
SFO1 500.13308
F2 - Processing parame
SI 327
SF 500.13000
WDW
SSB
LB 0.
GB
PC 1.
H-Leu-Pro-Gln-NHBn
1.05
4.51
2.61
4.48
1.00
1.28
2.51
1.16
1.10
1.05
0.32
1.17
1.80
3.47
6.88
1.92
7.01
11
10
9
8
7
6
5
4
3
2
1
0
ppm



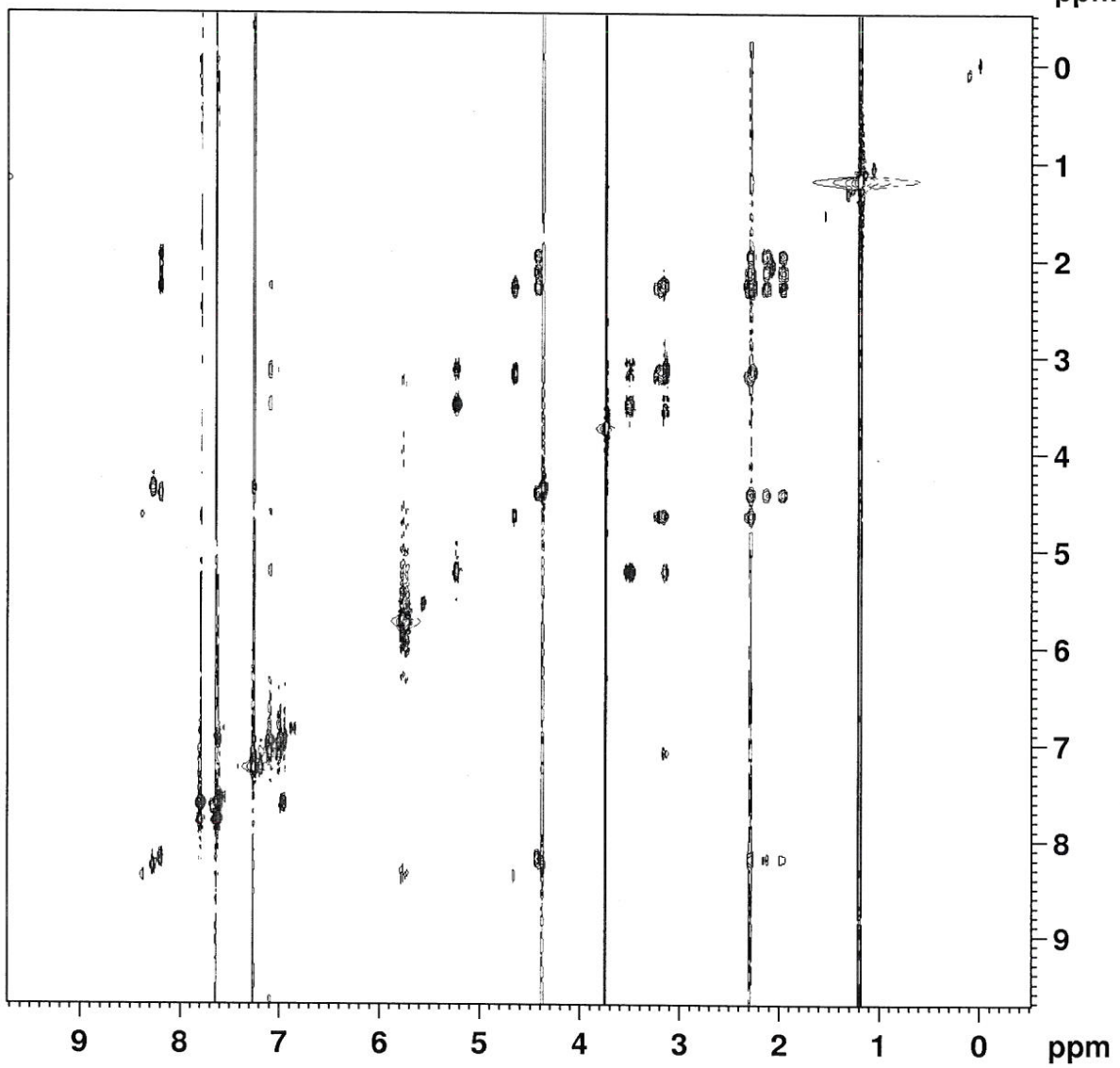

```
Current Data Parameters
NAME                  276G
EXPNO                    5
PROCNO                   1

F2 - Acquisition Paramete
Date_              20090514
Time                 17.15
INSTRUM              spect
PROBHD    5 mm TXI 1H/D-
PULPROG            zgpg30
TD                  65536
SOLVENT               DMSO
NS                    1285
DS                       4
SWH              30030.029 H
FIDRES           0.458222 H
AQ               1.0912410 s
RG                  4597.6
DW                  16.650 u
DE                   7.50 u
TE                  299.2 K
D1             2.00000000 s
d11            0.03000000 s
DELTA          1.89999998 s
TD0                      1

======== CHANNEL f1 =====
NUC1                   13C
P1                   12.00 u
PL1                  -5.50 d
SFO1          125.7703643 M

======== CHANNEL f2 =====
CPDPRG2            waltz16
NUC2                    1H
PCPD2                90.00 u
PL2                  -2.00 d
PL12                 18.50 d
PL13                 20.00 d
SFO2          500.1320005 M

F2 - Processing parameter
SI                   32768
SF          125.7577890 M
WDW                     EM
SSB                      0
LB                    1.00 H
GB                       0
PC                    1.40
```


DAD1 A, Sig=230,16 Ref=550,100 (062-0101.D)
mAU
400
350
300
250
200
150
100
50
0
-50
9.171
H-Leu-Pro-Gln-NHBn
0
5
10
15
20
25
min
DAD1 B, Sig=275,4 Ref=550,100 (062-0101.D)
mAU
400
350
300
250
200
150
100
50
0
-50
0
5
10
15
20
25
min

S33

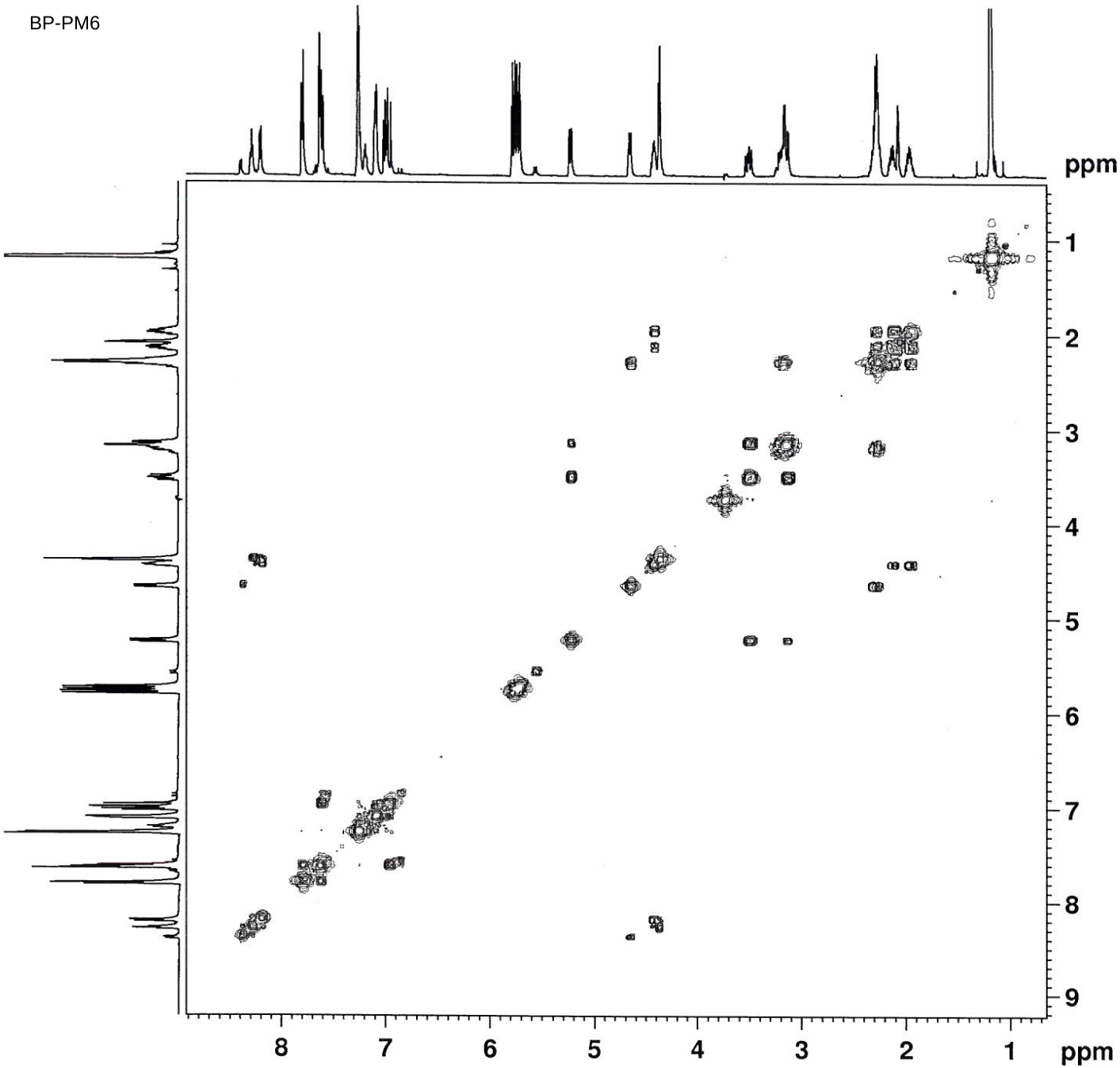

BP-PM6
0.22
0.88
0.87
2.10
3.23
3.45
0.75
2.21
2.13
4.26
1.05
1.05
3.36
0.92
3.31
4.64
1.10
1.15
17.98
8.400
8.385
8.298
8.287
8.275
8.215
8.199
7.814
7.798
7.660
7.641
7.628
7.609
7.591
7.284
7.273
7.268
7.261
7.246
7.214
7.209
7.203
7.197
7.185
7.103
7.095
7.027
7.012
6.997
6.989
6.957
5.803
5.792
5.777
5.767
5.753
5.742
5.728
5.717
5.257
5.250
5.235
5.229
4.679
4.674
4.661
4.656
4.460
4.450
4.441
4.432
4.426
4.417
4.392
4.380
3.542
3.520
3.509
3.487
3.225
3.212
3.204
3.186
3.174
3.166
3.139
3.132
2.346
2.327
2.316
2.301
2.285
2.279
2.274
2.256
2.244
2.174
2.164
2.158
2.148
2.131
2.121
2.105
2.091
2.087
2.083
2.078
2.074
2.007
1.994
1.989
1.977
1.966
1.960
1.949
1.196
1.187
0.118
0.104
0.098
0.000

BP-PM6

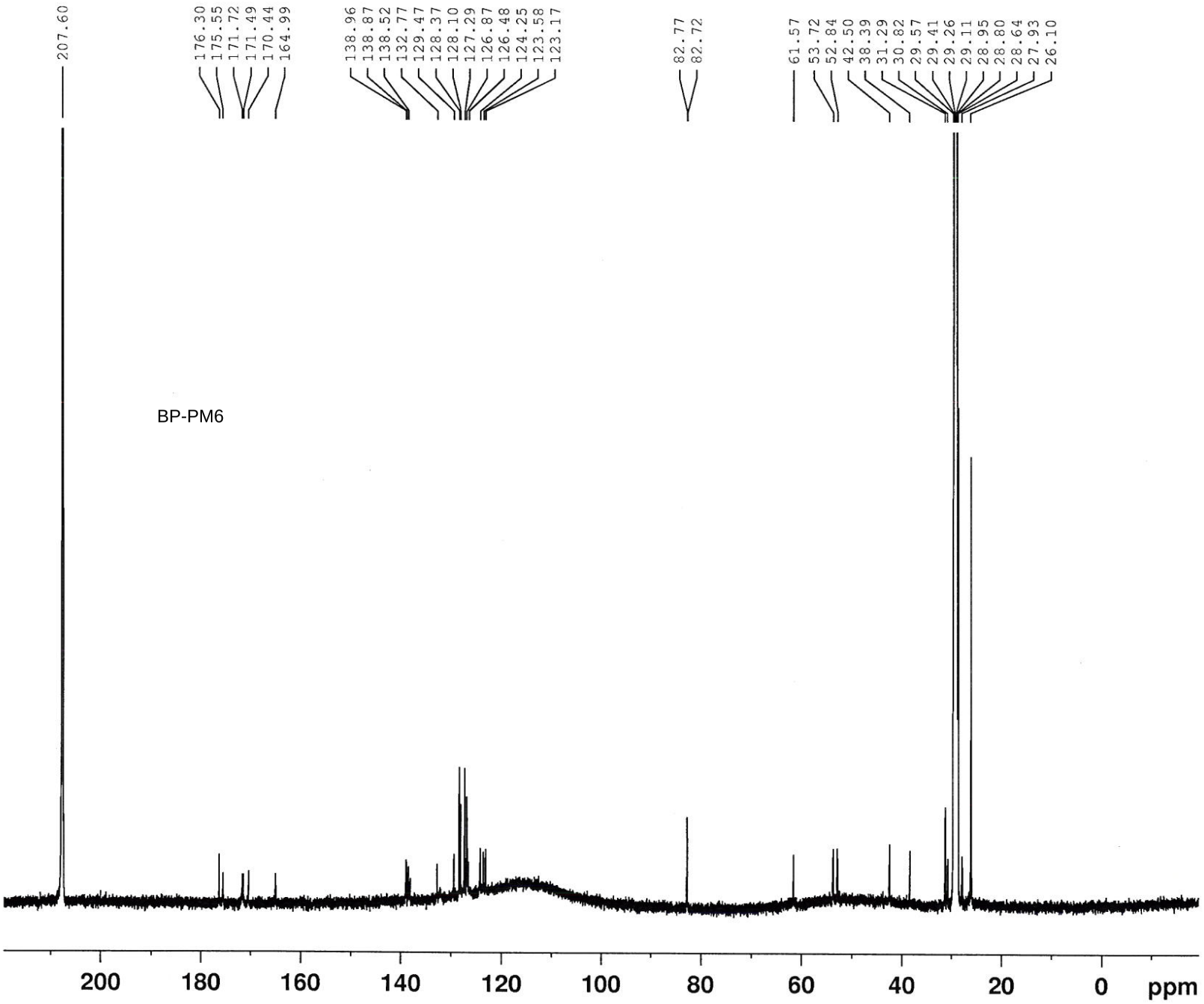



```
Current Data Parameters
NAME                 67G052909
EXPNO                        4
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20090529
Time                     11.28
INSTRUM                  spect
PROBHD    5 mm TXI 1H/D-
PULPROG               mlevetgp
TD                       4096
SOLVENT                  CDCl3
NS                           8
DS                          16
SWH                  5122.951 Hz
FIDRES               1.250720 Hz
AQ                   0.3999172 sec
RG                       203.2
DW                      97.600 usec
DE                       7.50 usec
TE                      300.2 K
d0                   0.00000300 sec
D1                   2.00000000 sec
D9                   0.07000000 sec
d11                  0.03000000 sec
D12                  0.00002000 sec
D16                  0.00020000 sec
DELTA                0.00119791 sec
DELTA1               0.00120800 sec
FACTOR1                      5
IN0                  0.00019520 sec
l1                          30
SCALEF                       6
ST1CNT                     128

======== CHANNEL f1 ========
NUC1                        1H
P1                       8.00 usec
p2                      16.00 usec
p5                      23.34 usec
P6                      35.00 usec
p7                      70.00 usec
P17                   2500.00 usec
PL1                     -2.00 dB
PL10                    10.82 dB
SFO1           500.1323022 MHz

====== GRADIENT CHANNEL =====
GPNAM1              SINE.100
GPNAM2              SINE.100
GPZ1                    30.00 %
GPZ2                    30.00 %
P16                   1000.00 usec

F1 - Acquisition parameters
ND0                          1
TD                         256
SFO1             500.1323 MHz
FIDRES           20.011526 Hz
SW                   10.243 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                        2048
SF             500.1300000 MHz
WDW                     QSINE
SSB                         2
LB                       0.00 Hz
GB                          0
PC                       1.00

F1 - Processing parameters
SI                        1024
MC2          echo-antiecho
SF             500.1300000 MHz
WDW                     QSINE
SSB                         2
LB                       0.00 Hz
GB                          0
```

BP-PM6

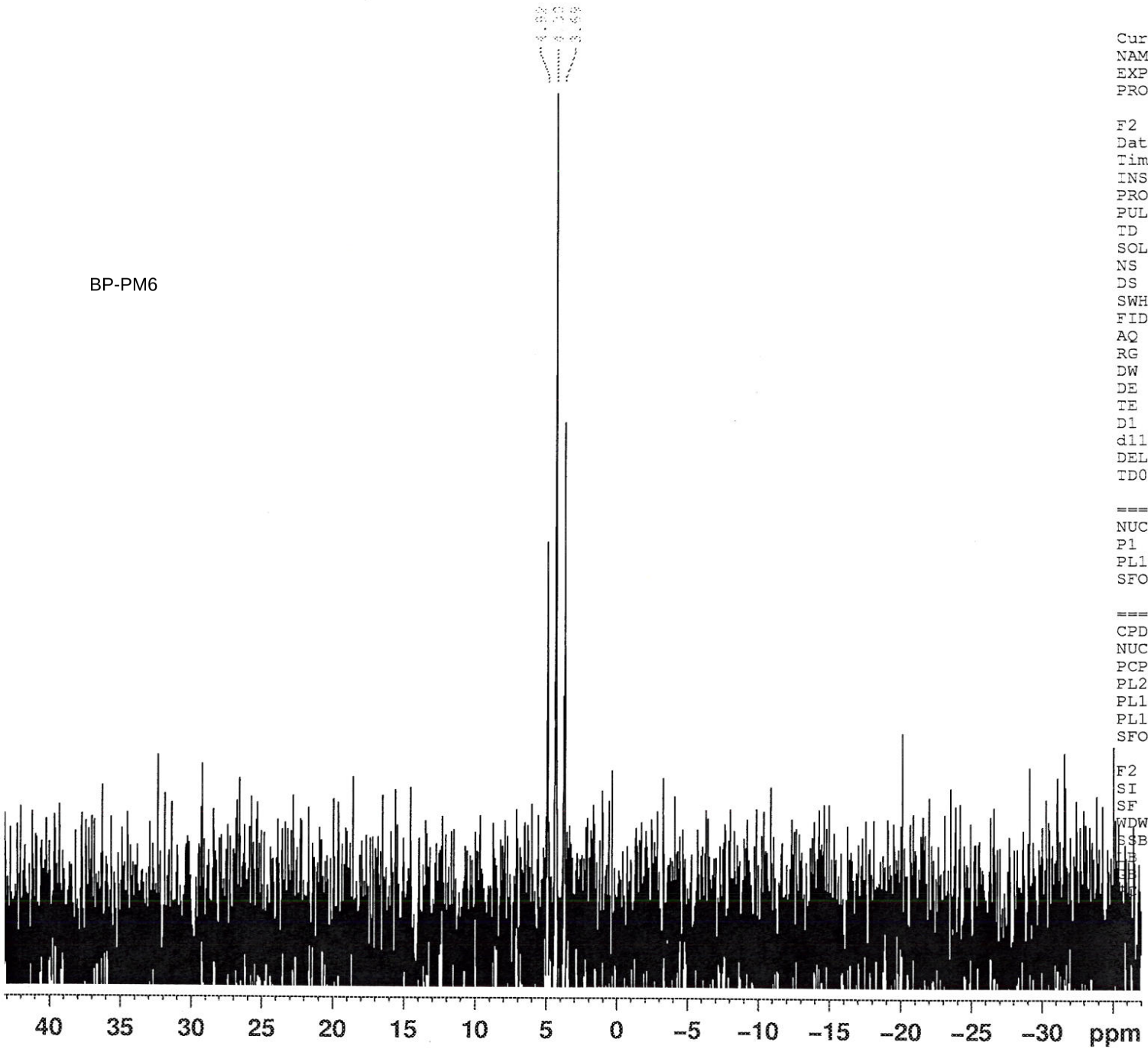



```
Current Data Parameters
NAME                 67G052909
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20090529
Time                     11.16
INSTRUM                  spect
PROBHD    5 mm TXI 1H/D-
PULPROG              cosygpqf
TD                       2048
SOLVENT                  DMSO
NS                          1
DS                          8
SWH                  6666.667 Hz
FIDRES               3.255208 Hz
AQ                   0.1537250 sec
RG                       203.2
DW                      75.000 usec
DE                       7.50 usec
TE                      300.2 K
d0                   0.00000300 sec
D1                   1.48689198 sec
d13                  0.00000400 sec
D16                  0.00020000 sec
IN0                  0.00015000 sec

======== CHANNEL f1 ========
NUC1                        1H
P0                        8.00 usec
P1                        8.00 usec
PL1                      -2.00 dB
SFO1                500.1330069 MHz

====== GRADIENT CHANNEL =====
GPNAM1                SINE.100
GPNAM2                SINE.100
GPZ1                     10.00 %
GPZ2                     10.00 %
P16                    1000.00 usec

F1 - Acquisition parameters
ND0                         1
TD                        128
SFO1                  500.133 MHz
FIDRES               52.083332 Hz
SW                       13.330 ppm
FnMODE                      QF

F2 - Processing parameters
SI                        1024
SF                  500.1300000 MHz
WDW                       SINE
SSB                          0
LB                        0.00 Hz
GB                           0
PC                        1.00

F1 - Processing parameters
SI                        1024
MC2                         QF
SF                  500.1300000 MHz
WDW                       SINE
SSB                          0
LB                        0.00 Hz
GB                           0
```



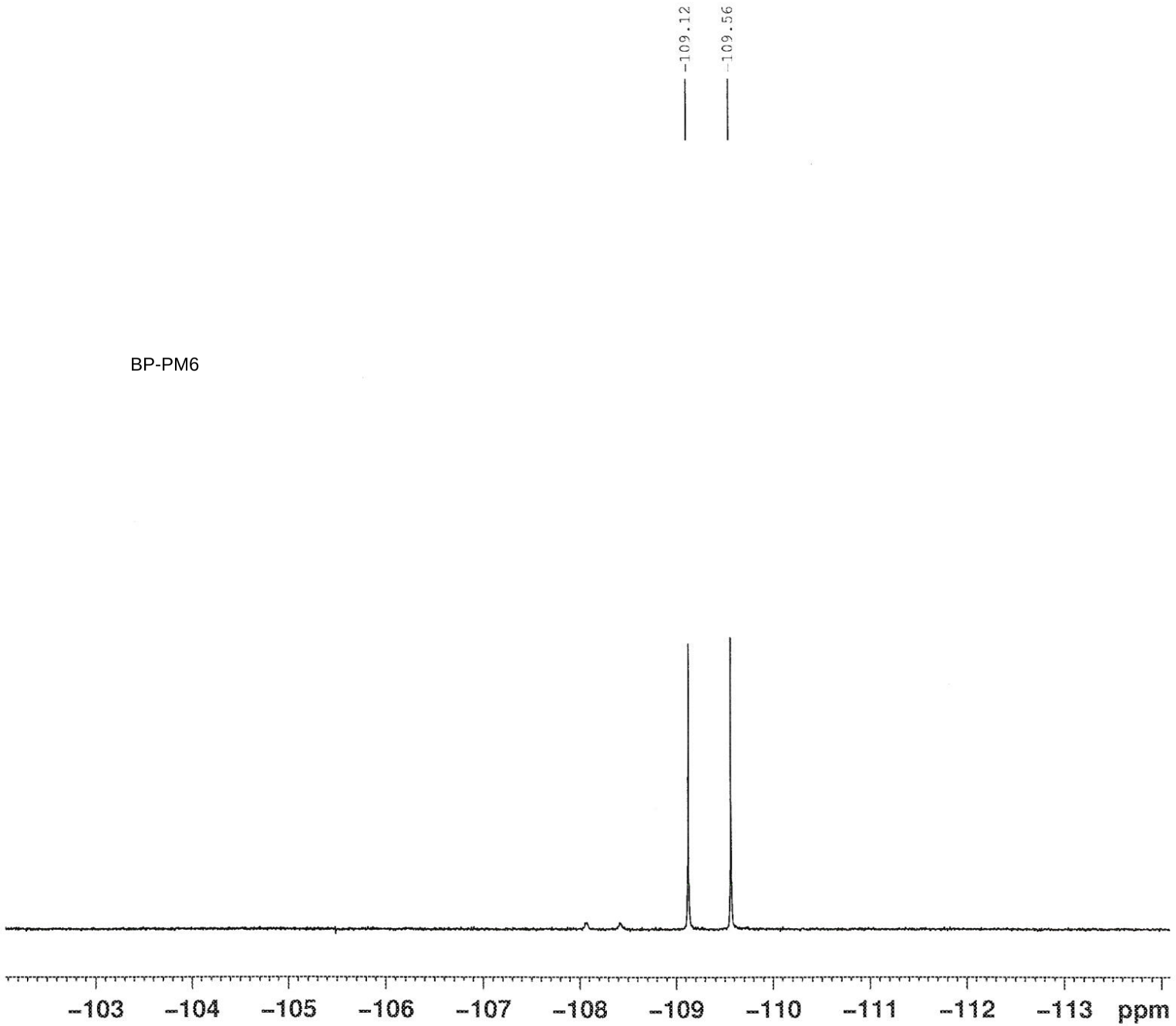

```
Current Data Parameters
NAME                 67G_3-1
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parame
Date_               2009052
Time                  11.27
INSTRUM                spect
PROBHD    5 mm TXI 1H/D-
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                    51200
DS                        4
SWH               30030.029
FIDRES             0.458222
AQ                1.091241
RG                    32768
DW                    16.650
DE                     7.50
TE                    298.2
D1                2.0000000
d11               0.0300000
DELTA             1.8999999
TD0                        1

======== CHANNEL f1 ===
NUC1                     13C
P1                     11.25
PL1                    -5.50
SFO1          125.7703643

======== CHANNEL f2 ===
CPDPRG2              waltz16
NUC2                     1H
PCPD2                  90.00
PL2                    -2.00
PL12                   18.50
PL13                   20.00
SFO2          500.1320005

F2 - Processing paramet
SI                    32768
SF            125.7577890
WDW                       EM
SSB                        0
LB                     1.00
GB                         0
PC                     1.40
```

BP-PM6

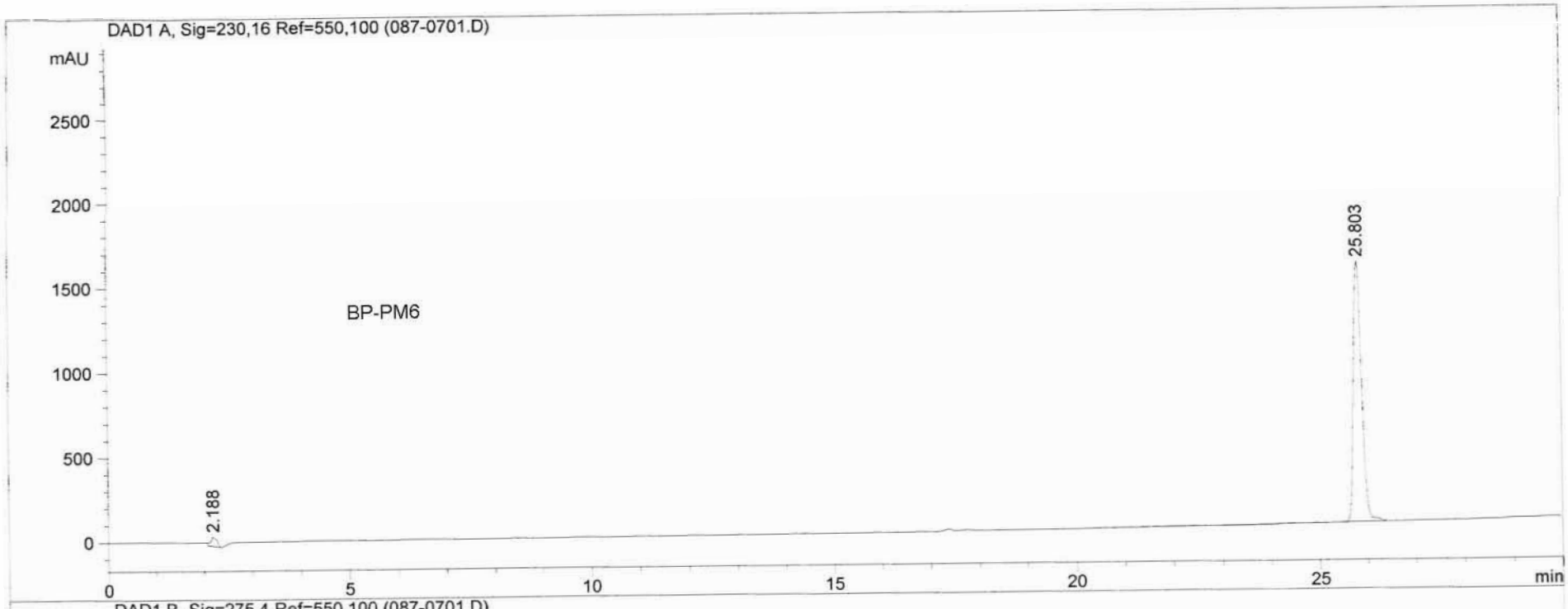


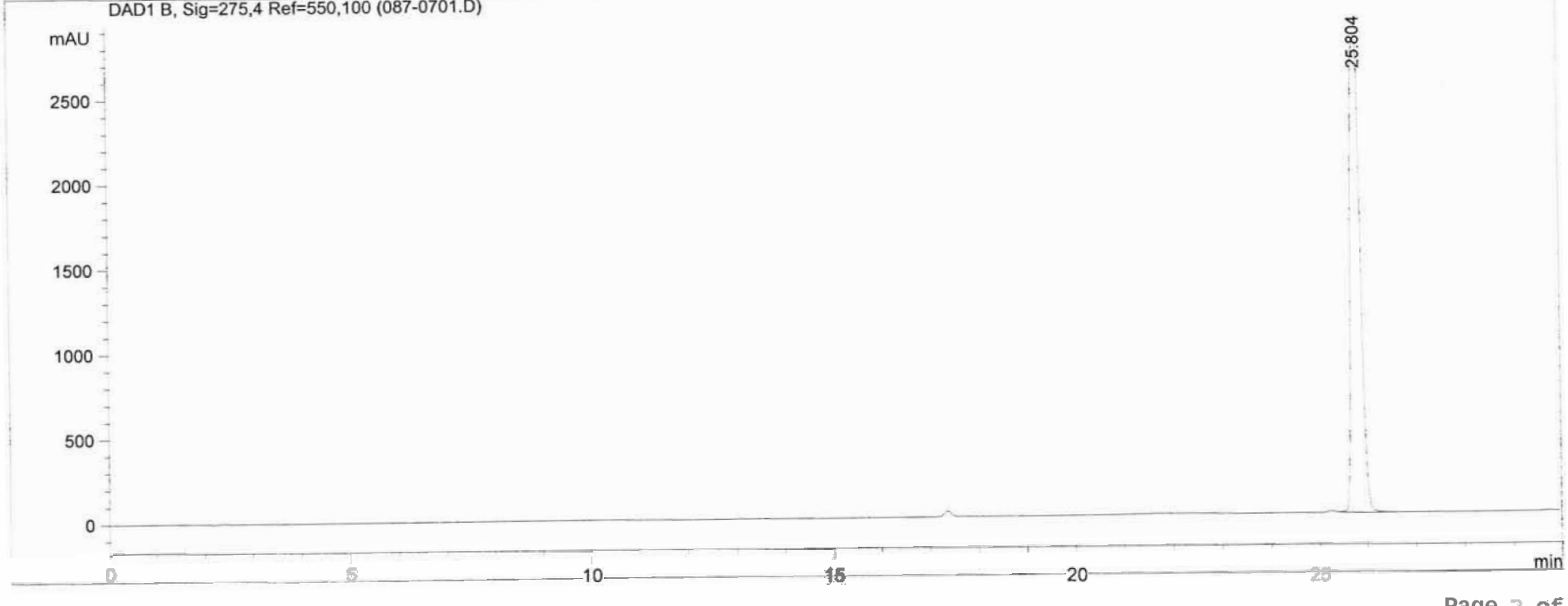

```
Current Data Parameters
NAME                  67G_3-1
EXPNO                       5
PROCNO                      1

F2 - Acquisition Parameters
Date_                20090525
Time                    10.43
INSTRUM                 spect
PROBHD    5 mm TXI 1H/D-
PULPROG                zgpg30
TD                      65536
SOLVENT               Acetone
NS                         16
DS                          4
SWH                 80645.164 Hz
FIDRES               1.230548 Hz
AQ                   0.4063794 sec
RG                     20642.5
DW                       6.200 usec
DE                       7.50 usec
TE                      298.2 K
D1                  2.00000000 sec
d11                 0.03000000 sec
DELTA               1.89999998 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       31P
P1                       15.00 usec
PL1                      -4.50 dB
SFO1             202.4462121 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                    90.00 usec
PL2                      -2.00 dB
PL12                     18.50 dB
PL13                     20.00 dB
SFO2             500.1320005 MHz

F2 - Processing parameters
SI                         32768
SF               202.4563350 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40
```

BP-PM6

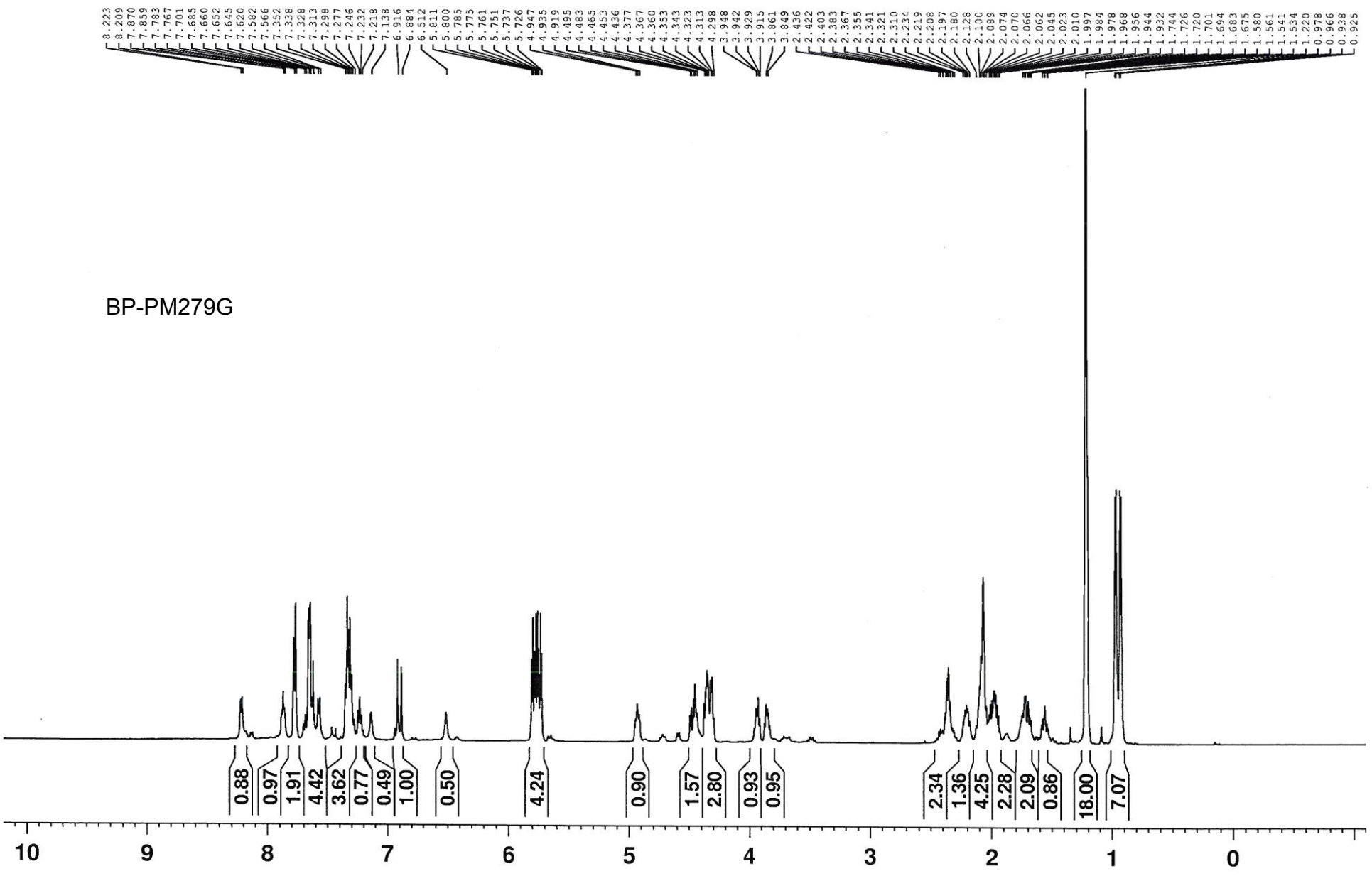



```
Current Data Parameters
NAME                  67G_1
EXPNO                     7
PROCNO                    1

F2 - Acquisition Parameters
Date_              20090525
Time                  11.15
INSTRUM               spect
PROBHD   5 mm QNP 1H/19
PULPROG           zgfhigqn
TD                  131072
SOLVENT               CDCl3
NS                       16
DS                        4
SWH              67567.570 Hz
FIDRES               0.515500 Hz
AQ                0.9699828 sec
RG                     4096
DW                   7.400 usec
DE                   6.00 usec
TE                   300.2 K
D1             1.00000000 sec
d11            0.03000000 sec
d12            0.00002000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    19F
P1                   12.00 usec
PL1                  -4.00 dB
SFO1          282.3761148 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2               100.00 usec
PL2                  -1.00 dB
PL12                 19.00 dB
SFO2          300.1312005 MHz

F2 - Processing parameters
SI                      65536
SF            282.4043550 MHz
WDW                        EM
SSB                         0
LB                   0.30 Hz
GB                          0
PC                       1.00
```

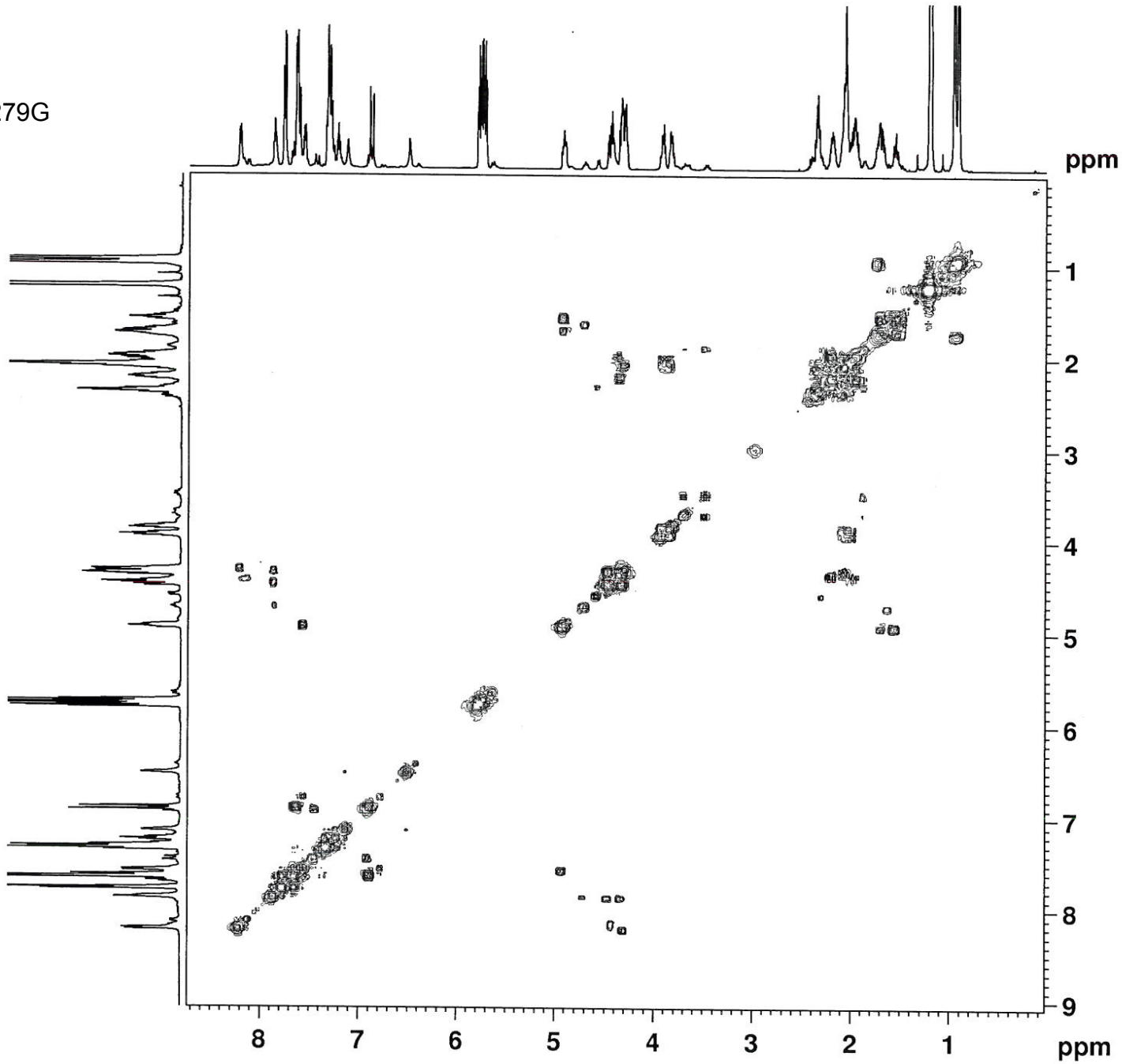

DAD1 A, Sig=230,16 Ref=550,100 (087-0701.D)
mAU
2500
2000
1500
1000
500
0
BP-PM6
2.188
25.803
0
5
10
15
20
25
min


DAD1 B, Sig=275,4 Ref=550,100 (087-0701.D)
mAU
2500
2000
1500
1000
500
0
25.804
0
5
10
15
20
25
min

S40

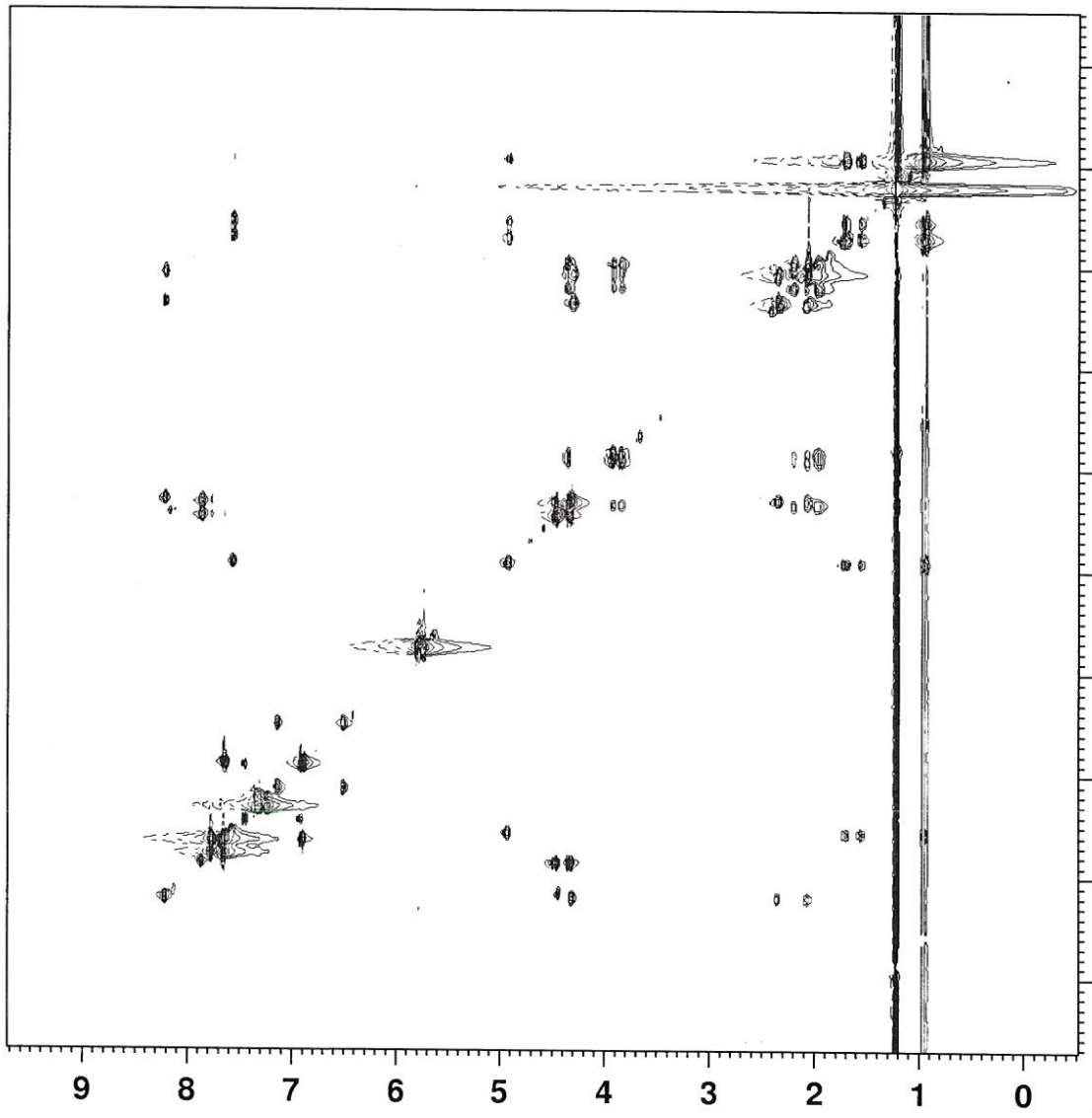

8.223
8.209
7.870
7.859
7.783
7.767
7.701
7.685
7.660
7.652
7.645
7.620
7.582
7.566
7.352
7.338
7.328
7.313
7.298
7.277
7.246
7.232
7.218
7.138
6.916
6.884
6.512
5.811
5.800
5.785
5.775
5.761
5.751
5.737
5.726
4.947
4.935
4.919
4.495
4.483
4.465
4.453
4.436
4.377
4.367
4.360
4.353
4.343
4.323
4.313
4.298
3.948
3.942
3.929
3.915
3.861
3.849
2.436
2.422
2.403
2.383
2.367
2.355
2.341
2.321
2.310
2.234
2.219
2.208
2.197
2.180
2.128
2.100
2.089
2.074
2.070
2.066
2.062
2.045
2.023
2.010
1.997
1.984
1.978
1.968
1.956
1.944
1.932
1.744
1.726
1.720
1.701
1.694
1.683
1.675
1.580
1.561
1.541
1.534
1.220
0.978
0.966
0.938
0.925
BP-PM279G
0.88
0.97
1.91
4.42
3.62
0.77
0.49
1.00
0.50
4.24
0.90
1.57
2.80
0.93
0.95
2.34
1.36
4.25
2.28
2.09
0.86
18.00
7.07
10
9
8
7
6
5
4
3
2
1
0

BP-PM279G

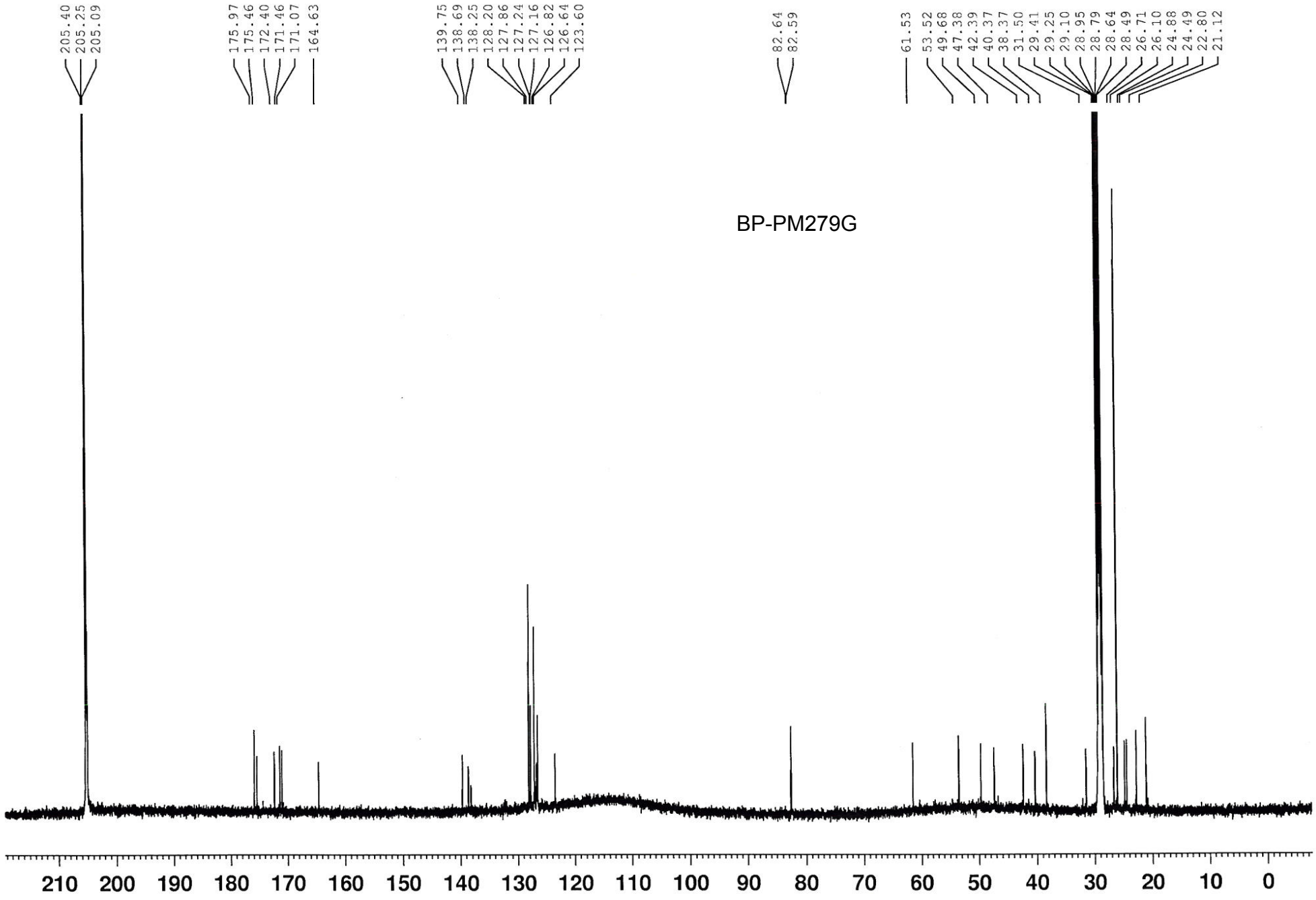



```
Current Data Parameters
NAME                 279G052709
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20090528
Time                      11.01
INSTRUM                   spect
PROBHD     5 mm TXI 1H/D-
PULPROG                cosygpqf
TD                         2048
SOLVENT                    DMSO
NS                            1
DS                            8
SWH                  6666.667 Hz
FIDRES               3.255208 Hz
AQ                   0.1537250 sec
RG                       101.6
DW                      75.000 usec
DE                       7.50 usec
TE                      300.2 K
d0                   0.00000300 sec
D1                   1.48689198 sec
d13                  0.00000400 sec
D16                  0.00020000 sec
IN0                  0.00015000 sec

======== CHANNEL f1 ========
NUC1                         1H
P0                       8.00 usec
P1                       8.00 usec
PL1                     -2.00 dB
SFO1                500.1330069 MHz

====== GRADIENT CHANNEL =====
GPNAM1                SINE.100
GPNAM2                SINE.100
GPZ1                     10.00 %
GPZ2                     10.00 %
P16                    1000.00 usec

F1 - Acquisition parameters
ND0                           1
TD                          128
SFO1                500.133 MHz
FIDRES              52.083332 Hz
SW                      13.330 ppm
FnMODE                      QF

F2 - Processing parameters
SI                         1024
SF                  500.1300000 MHz
WDW                       SINE
SSB                          0
LB                        0.00 Hz
GB                           0
PC                        1.00

F1 - Processing parameters
SI                         1024
MC2                          QF
SF                  500.1300000 MHz
WDW                       SINE
SSB                          0
LB                        0.00 Hz
GB                           0
```

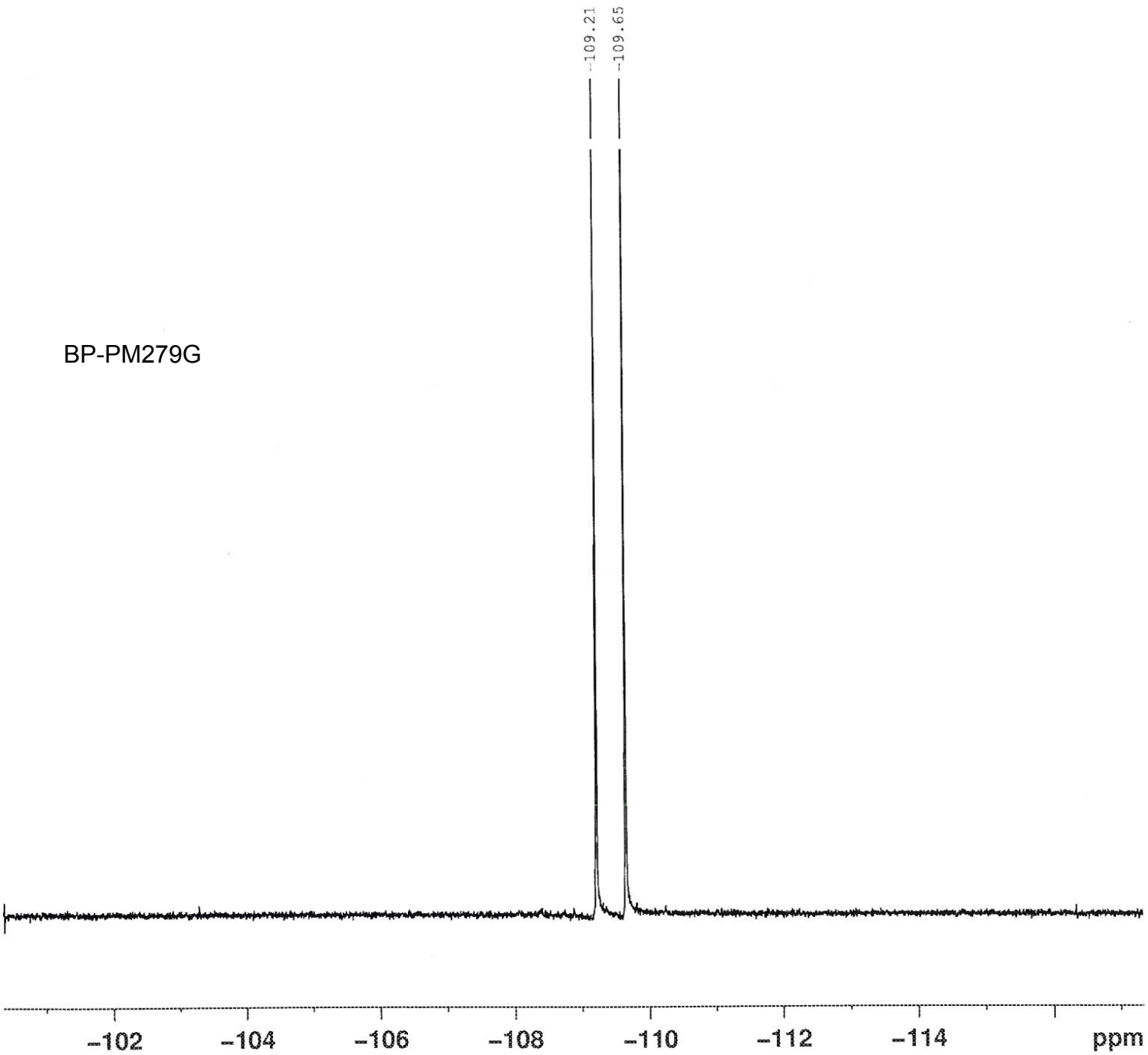
BP-PM279G




```
Current Data Parameters
NAME                  279G052709
EXPNO                          3
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20090528
Time                      11.07
INSTRUM                   spect
PROBHD    5 mm TXI 1H/D-
PULPROG                mlevetgp
TD                         4096
SOLVENT                   CDCl3
NS                             8
DS                            16
SWH                   5122.951 Hz
FIDRES                1.250720 Hz
AQ                    0.3999172 sec
RG                           128
DW                       97.600 usec
DE                        7.50 usec
TE                       300.2 K
d0                    0.00000300 sec
D1                    2.00000000 sec
D9                    0.07000000 sec
d11                   0.03000000 sec
D12                   0.00002000 sec
D16                   0.00020000 sec
DELTA                 0.00119791 sec
DELTA1                0.00120800 sec
FACTOR1                        5
IN0                   0.00019520 sec
l1                             30
SCALEF                         6
ST1CNT                       128

======== CHANNEL f1 ========
NUC1                          1H
P1                         8.00 usec
p2                        16.00 usec
p5                        23.34 usec
P6                        35.00 usec
p7                        70.00 usec
P17                     2500.00 usec
PL1                       -2.00 dB
PL10                      10.82 dB
SFO1                500.1323022 MHz

====== GRADIENT CHANNEL =====
GPNAM1                SINE.100
GPNAM2                SINE.100
GPZ1                      30.00 %
GPZ2                      30.00 %
P16                     1000.00 usec

F1 - Acquisition parameters
ND0                            1
TD                           256
SFO1                  500.1323 MHz
FIDRES               20.011526 Hz
SW                       10.243 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                          2048
SF                 500.1300000 MHz
WDW                       QSINE
SSB                           2
LB                         0.00 Hz
GB                            0
PC                         1.40

F1 - Processing parameters
SI                          1024
MC2                echo-antiecho
SF                 500.1300000 MHz
WDW                       QSINE
SSB                           2
LB                         0.00 Hz
GB                            0
```

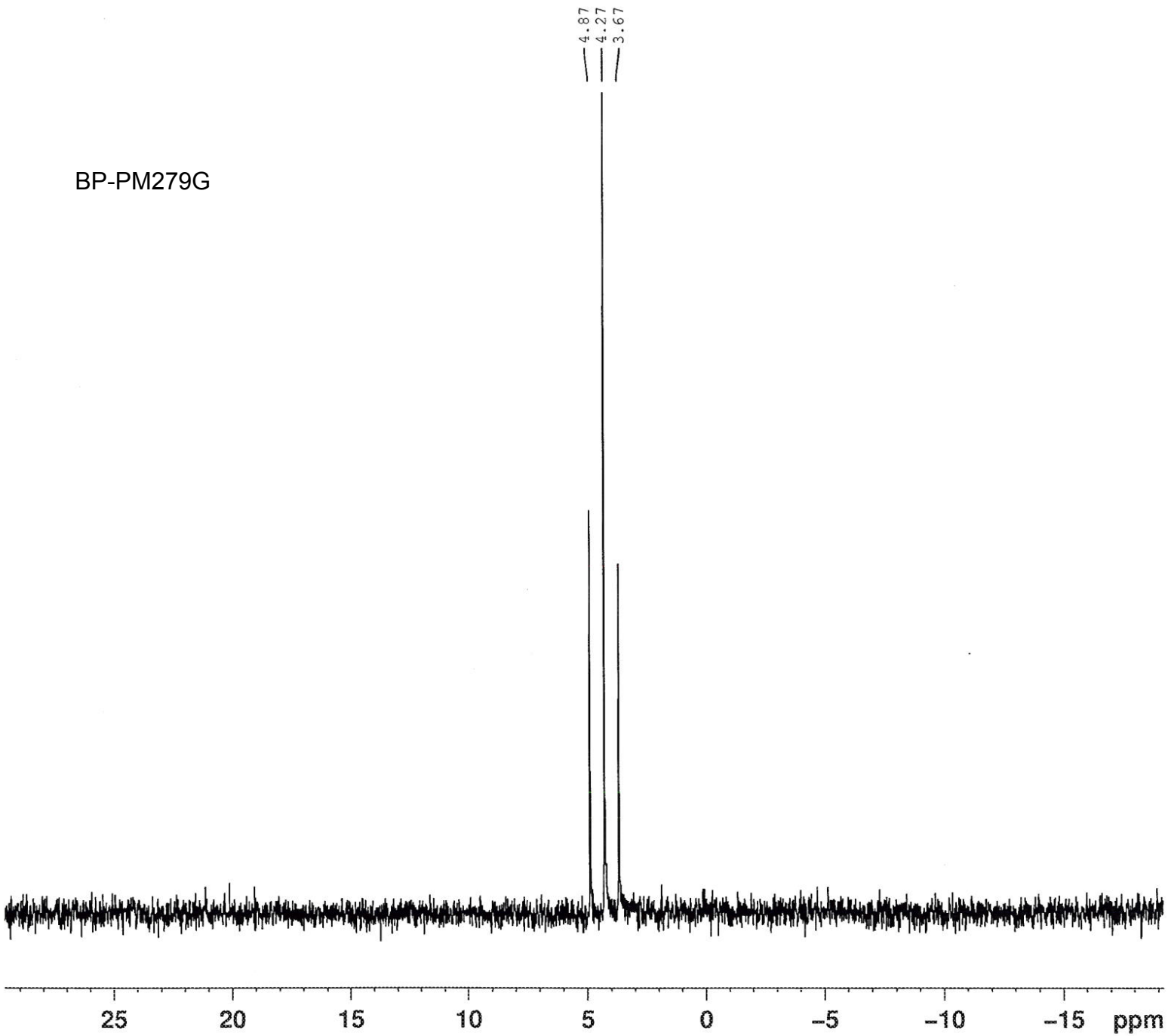

205.40
205.25
205.09
175.97
175.46
172.40
171.46
171.07
164.63
139.75
138.69
138.25
128.20
127.86
127.24
127.16
126.82
126.64
123.60
82.64
82.59
61.53
53.52
49.68
47.38
42.39
40.37
38.37
31.50
29.41
29.25
29.10
28.95
28.79
28.64
28.49
26.71
26.10
24.88
24.49
22.80
21.12
BP-PM279G
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

BP-PM279G

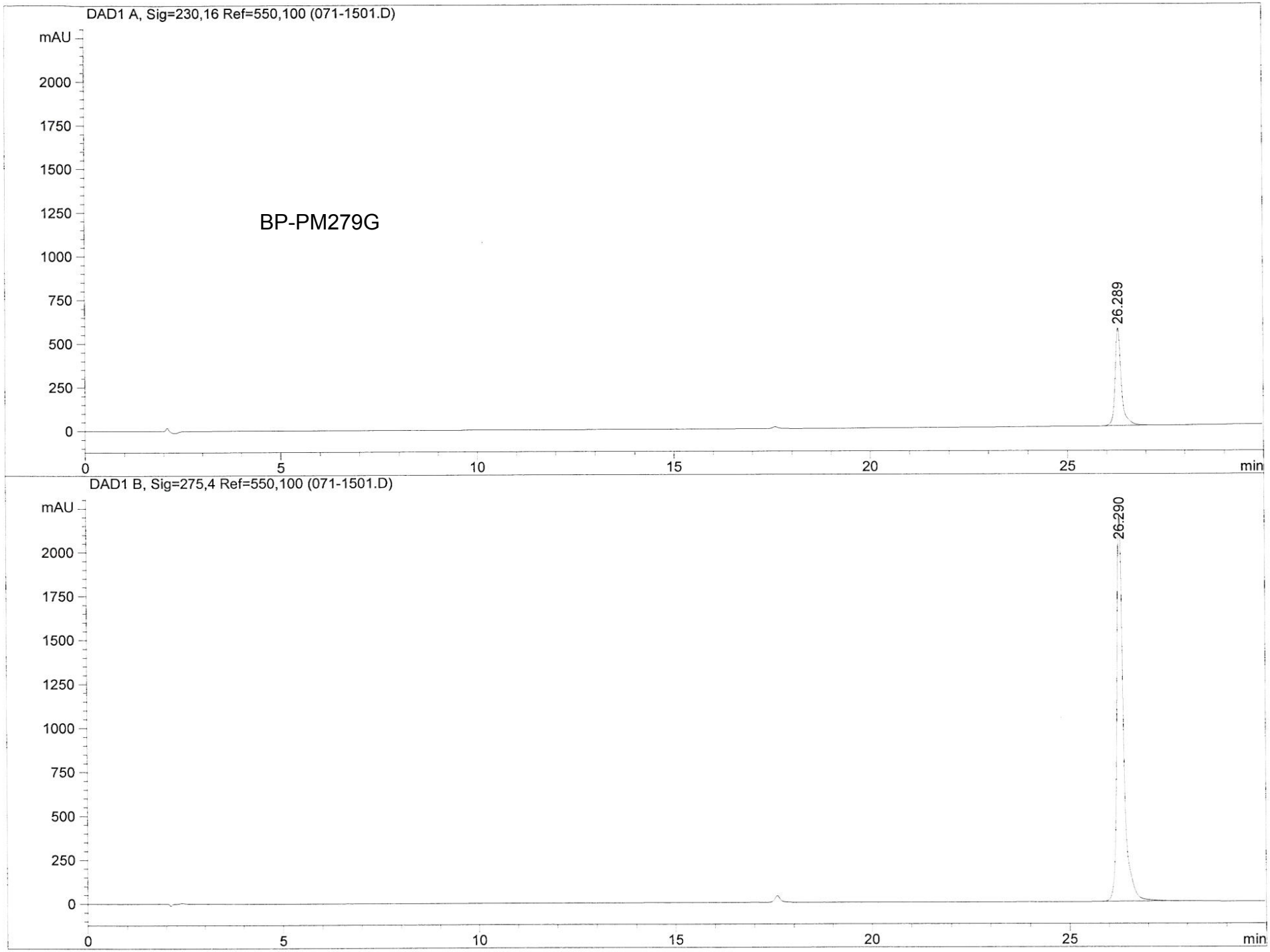


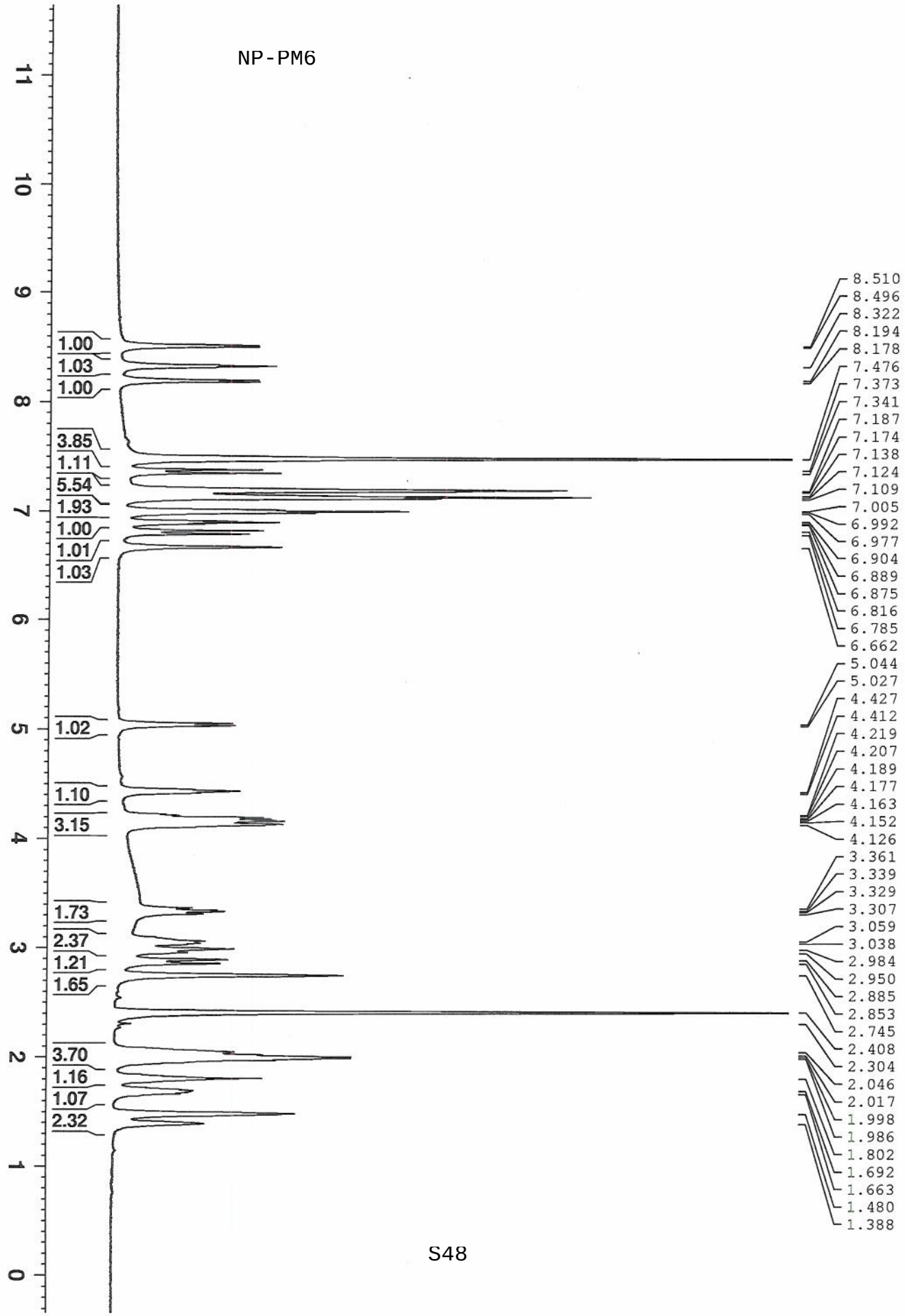

```
Current Data Parameters
NAME                  279G
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_              20090508
Time                  9.26
INSTRUM              spect
PROBHD    5 mm QNP 1H/19
PULPROG           zgfhigqn
TD                  131072
SOLVENT            Acetone
NS                       60
DS                        4
SWH               67567.570 Hz
FIDRES            0.515500 Hz
AQ                0.9699828 sec
RG                   1824.6
DW                  7.400 usec
DE                  6.00 usec
TE                  301.2 K
D1               1.00000000 sec
d11              0.03000000 sec
d12              0.00002000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    19F
P1                  12.00 usec
PL1                 -4.00 dB
SFO1          282.3761148 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2              100.00 usec
PL2                 -1.00 dB
PL12                19.00 dB
SFO2          300.1312005 MHz

F2 - Processing parameters
SI                    65536
SF            282.4043550 MHz
WDW                      EM
SSB                       0
LB                  0.30 Hz
GB                        0
PC                     1.00
```

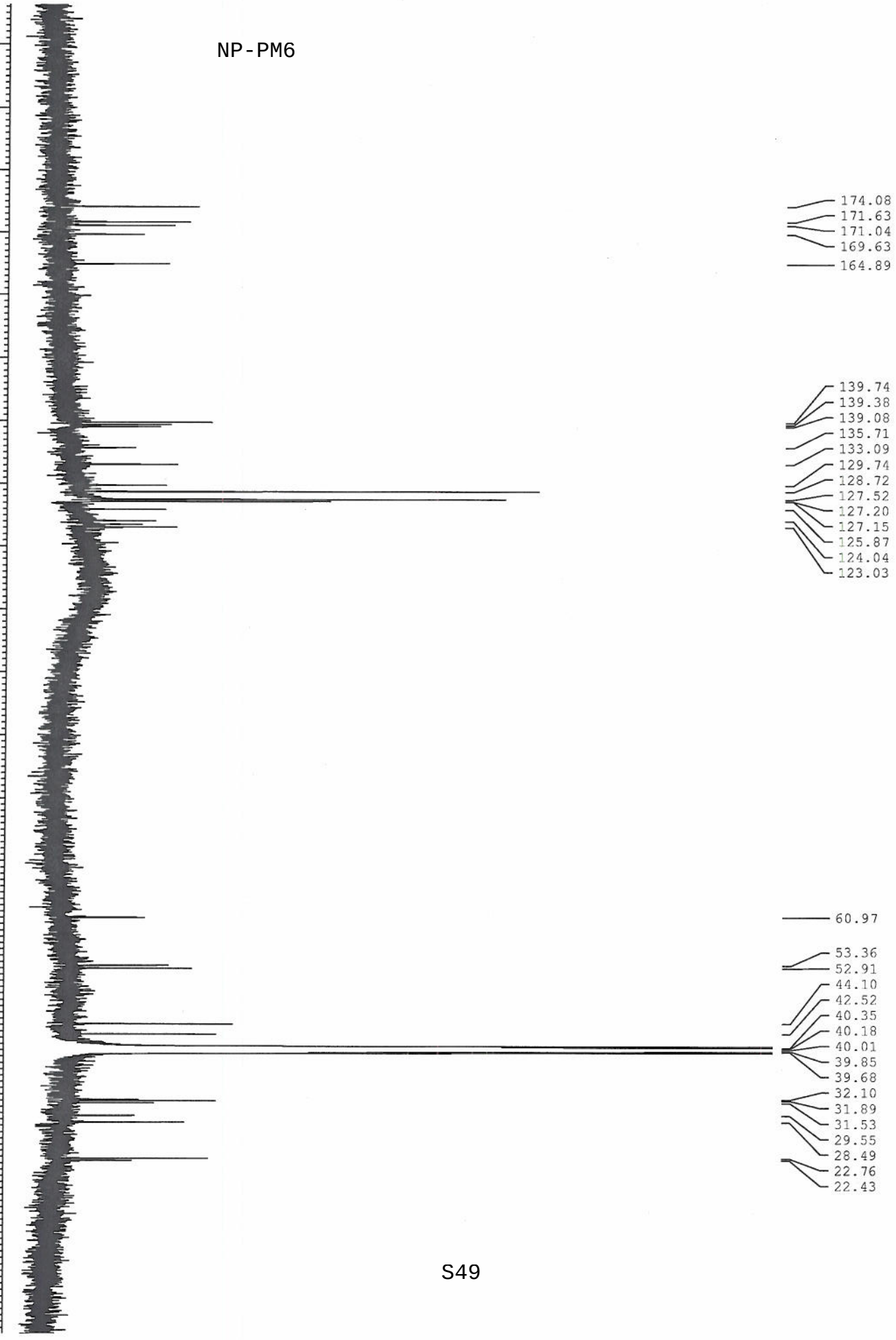
BP-PM279G

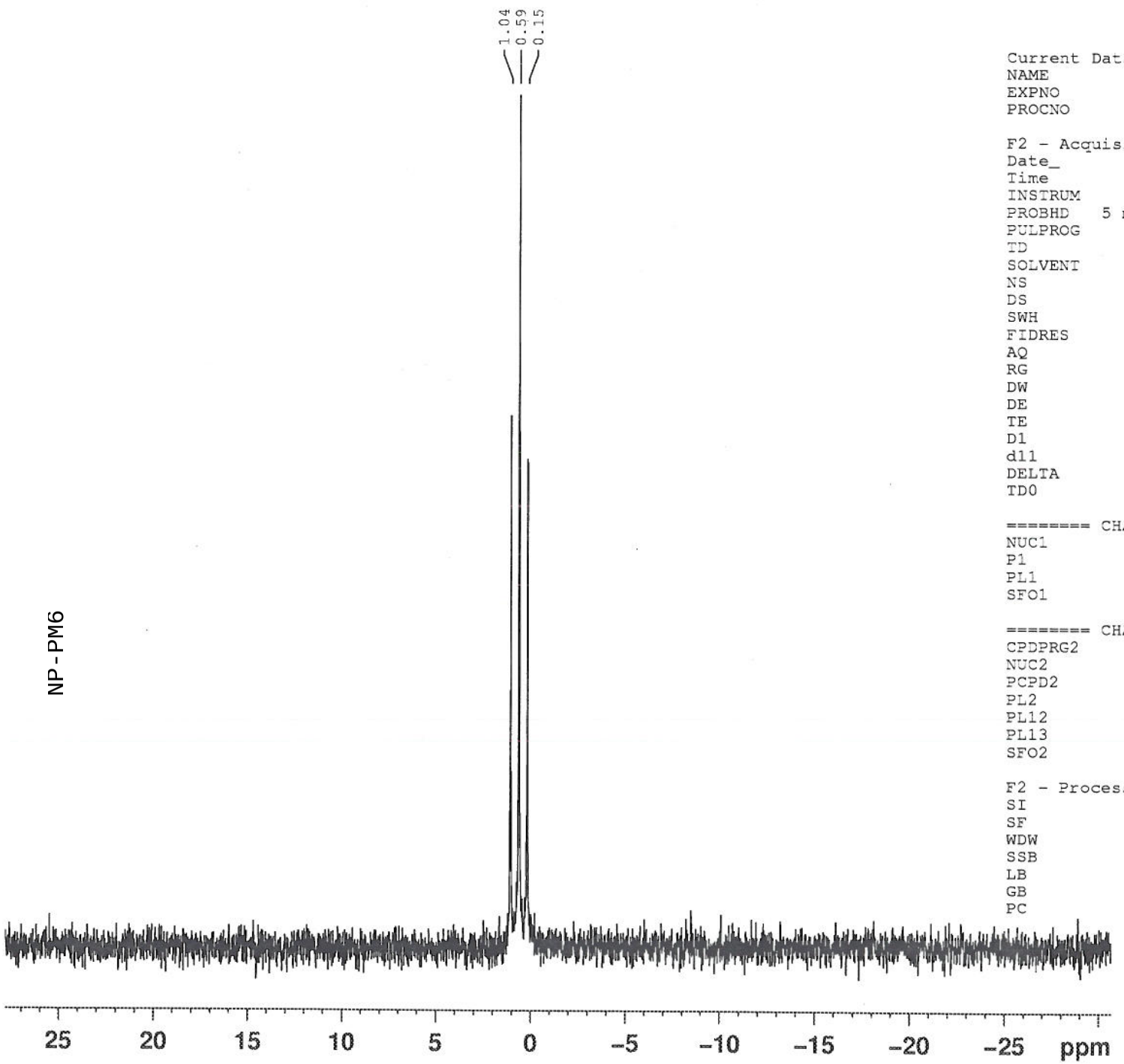



```
Current Data Parameters
NAME                  279G
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_              20090508
Time                  9.19
INSTRUM              spect
PROBHD   5 mm TXI 1H/D-
PULPROG              zgpg30
TD                    65536
SOLVENT             Acetone
NS                      103
DS                        4
SWH               80645.164 Hz
FIDRES             1.230548 Hz
AQ                0.4063794 sec
RG                   5792.6
DW                   6.200 usec
DE                   7.50 usec
TE                   298.2 K
D1                2.00000000 sec
d11               0.03000000 sec
DELTA             1.89999998 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    31P
P1                   15.00 usec
PL1                  -4.50 dB
SFO1           202.4462121 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                90.00 usec
PL2                  -2.00 dB
PL12                 18.50 dB
PL13                 20.00 dB
SFO2           500.1320005 MHz

F2 - Processing parameters
SI                      32768
SF             202.4563350 MHz
WDW                        EM
SSB                         0
LB                   1.00 Hz
GB                          0
PC                   1.40
```


DAD1 A, Sig=230,16 Ref=550,100 (071-1501.D)
mAU
2000
1750
1500
1250
1000
750
500
250
0
BP-PM279G
26.289
0
5
10
15
20
25
min
DAD1 B, Sig=275,4 Ref=550,100 (071-1501.D)
mAU
2000
1750
1500
1250
1000
750
500
250
0
26.290
0
5
10
15
20
25
min

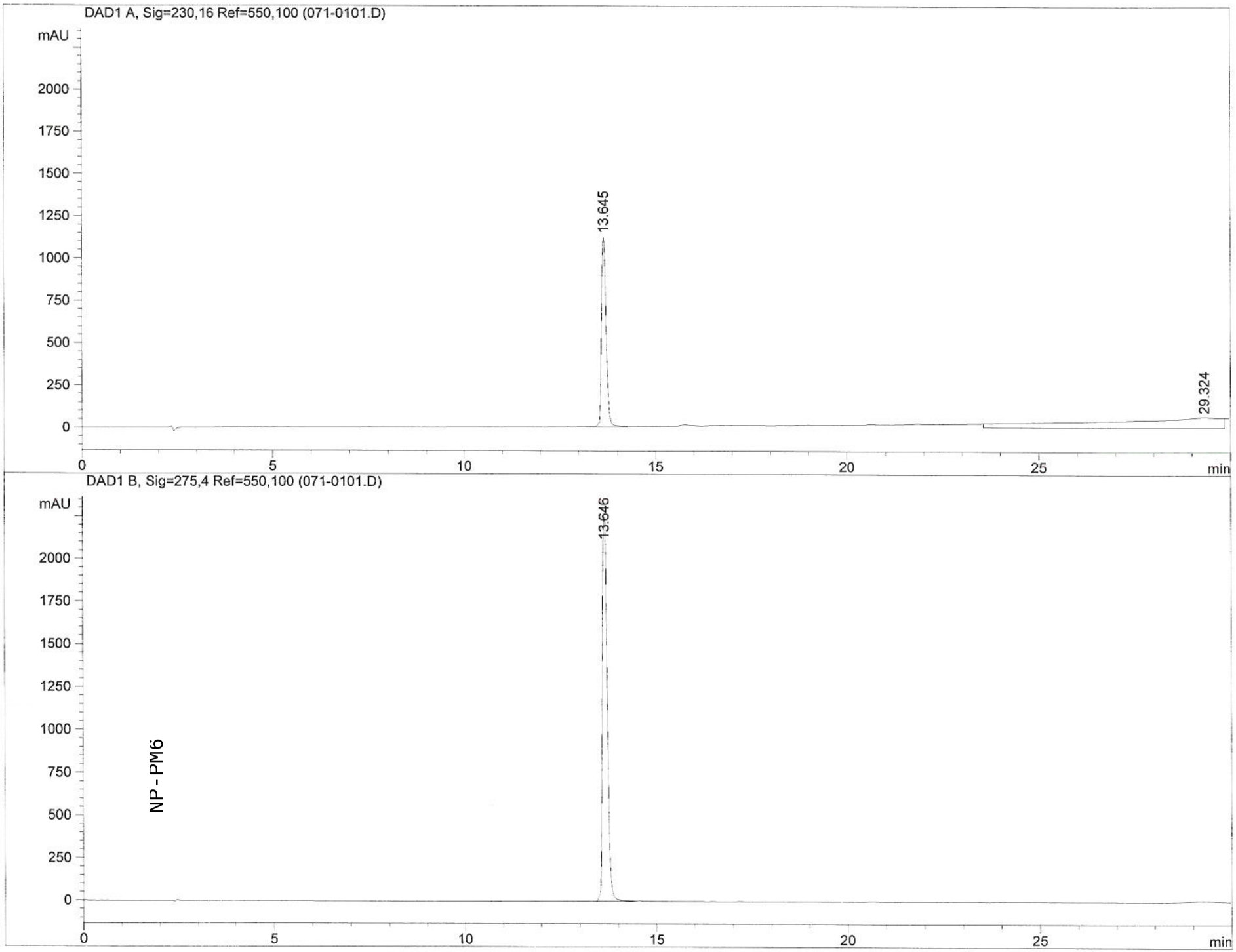

NP-PM6
11
10
9
8
7
6
5
4
3
2
1
0
1.00
1.03
1.00
3.85
1.11
5.54
1.93
1.00
1.01
1.03
1.02
1.10
3.15
1.73
2.37
1.21
1.65
3.70
1.16
1.07
2.32
8.510
8.496
8.322
8.194
8.178
7.476
7.373
7.341
7.187
7.174
7.138
7.124
7.109
7.005
6.992
6.977
6.904
6.889
6.875
6.816
6.785
6.662
5.044
5.027
4.427
4.412
4.219
4.207
4.189
4.177
4.163
4.152
4.126
3.361
3.339
3.329
3.307
3.059
3.038
2.984
2.950
2.885
2.853
2.745
2.408
2.304
2.046
2.017
1.998
1.986
1.802
1.692
1.663
1.480
1.388


NP-PM6
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
174.08
171.63
171.04
169.63
164.89
139.74
139.38
139.08
135.71
133.09
129.74
128.72
127.52
127.20
127.15
125.87
124.04
123.03
60.97
53.36
52.91
44.10
42.52
40.35
40.18
40.01
39.85
39.68
32.10
31.89
31.53
29.55
28.49
22.76
22.43

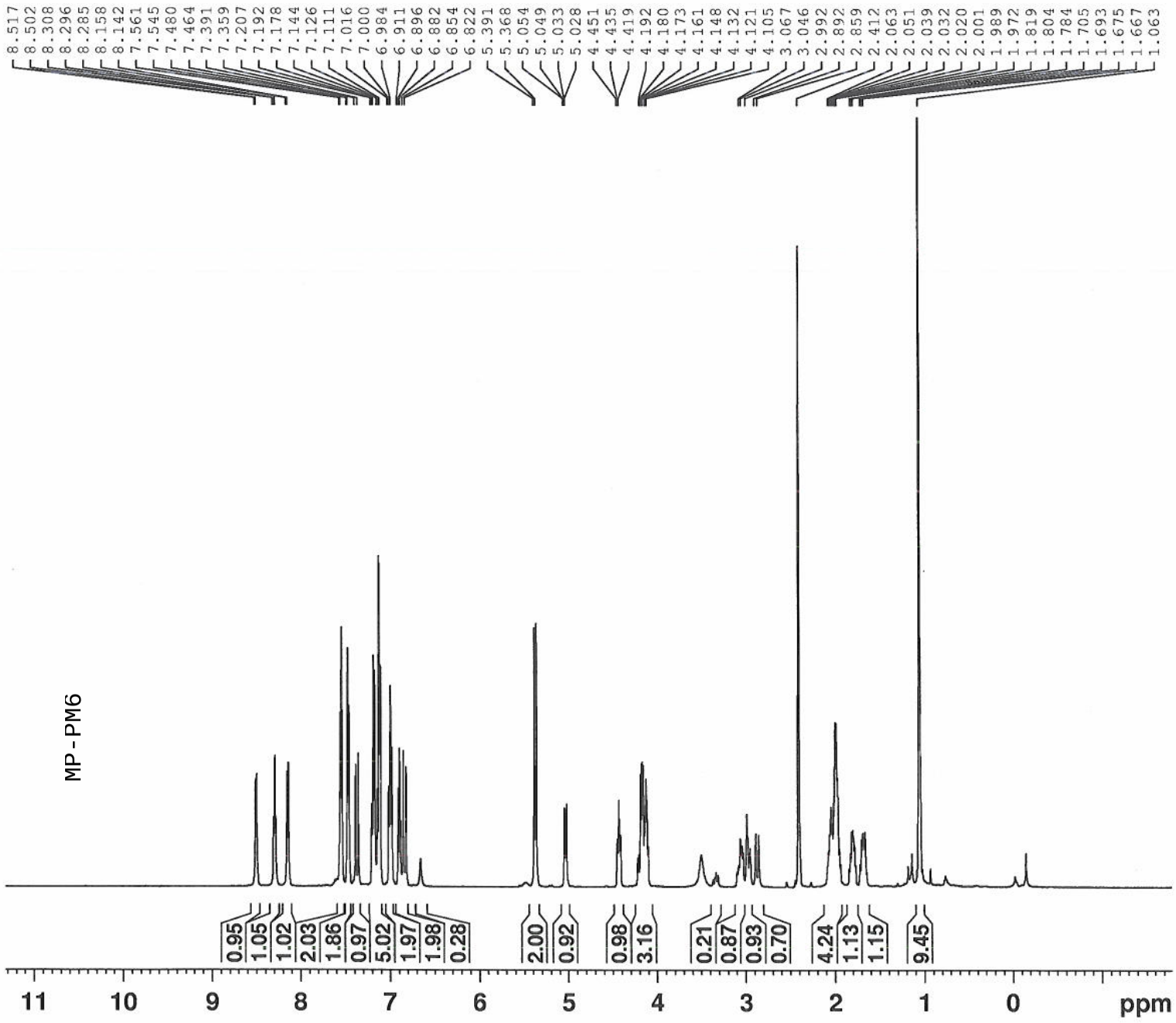


NP-PM6

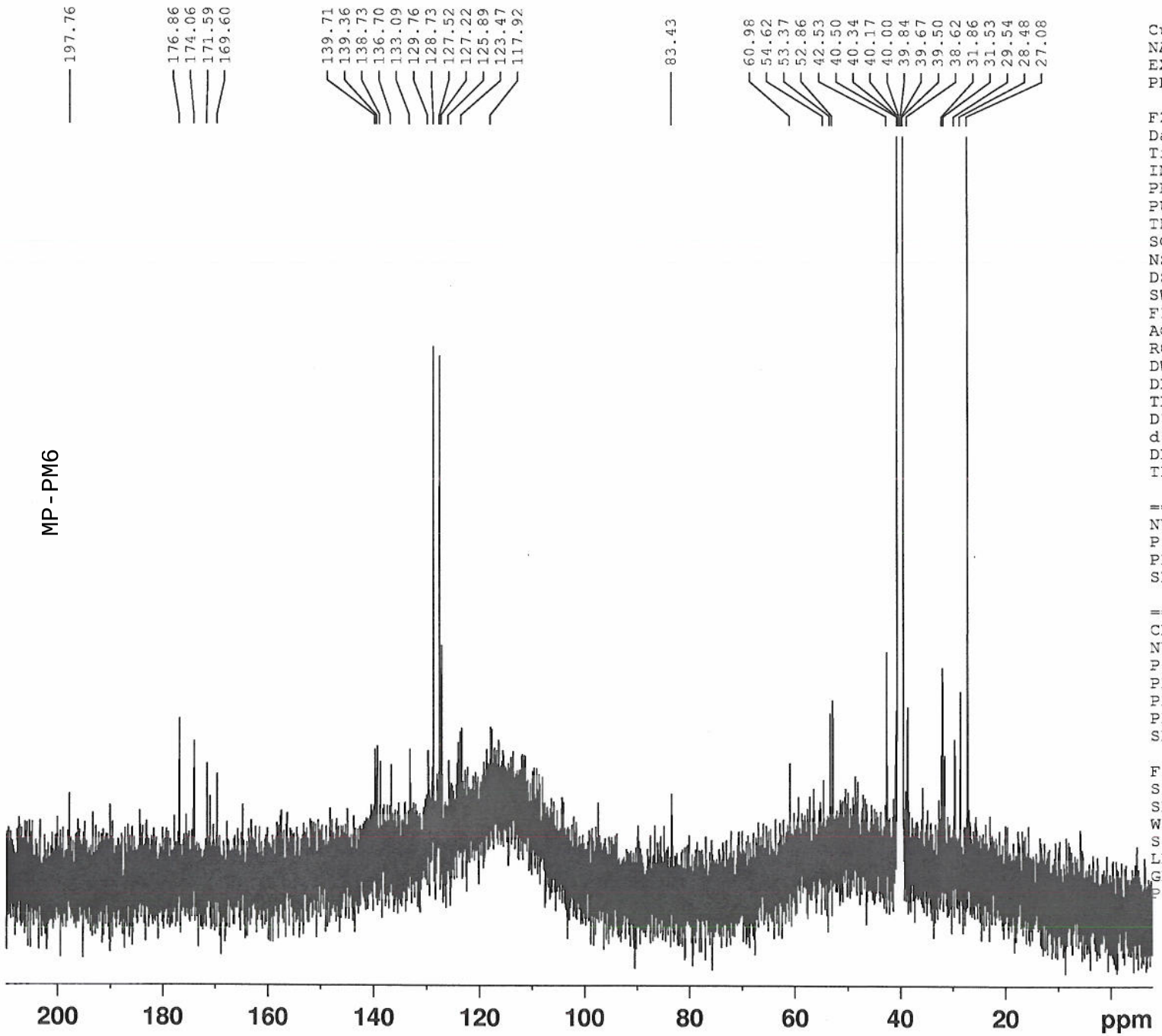
```
Current Data Parameters
NAME                  65G
EXPNO                   5
PROCNO                  1

F2 - Acquisition Parameters
Date_            20090706
Time                11.57
INSTRUM             spect
PROBHD    5 mm TXI 1H/D-
PULPROG            zgpg30
TD                  65536
SOLVENT           Acetone
NS                    516
DS                      4
SWH             80645.164 Hz
FIDRES           1.230548 Hz
AQ               0.4063794 sec
RG                 18390.4
DW                   6.200 usec
DE                   7.50 usec
TE                  300.2 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  31P
P1                  15.00 usec
PL1                 -4.50 dB
SFO1         202.4462121 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2               90.00 usec
PL2                 -2.00 dB
PL12                18.50 dB
PL13                20.00 dB
SFO2         500.1320005 MHz

F2 - Processing parameters
SI                    32768
SF           202.4563350 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```


DAD1 A, Sig=230,16 Ref=550,100 (071-0101.D)
mAU
2000
1750
1500
1250
1000
750
500
250
0
13.645
29.324
0
5
10
15
20
25
min
DAD1 B, Sig=275,4 Ref=550,100 (071-0101.D)
mAU
2000
1750
1500
1250
1000
750
500
250
0
13.646
NP-PM6
0
5
10
15
20
25
min

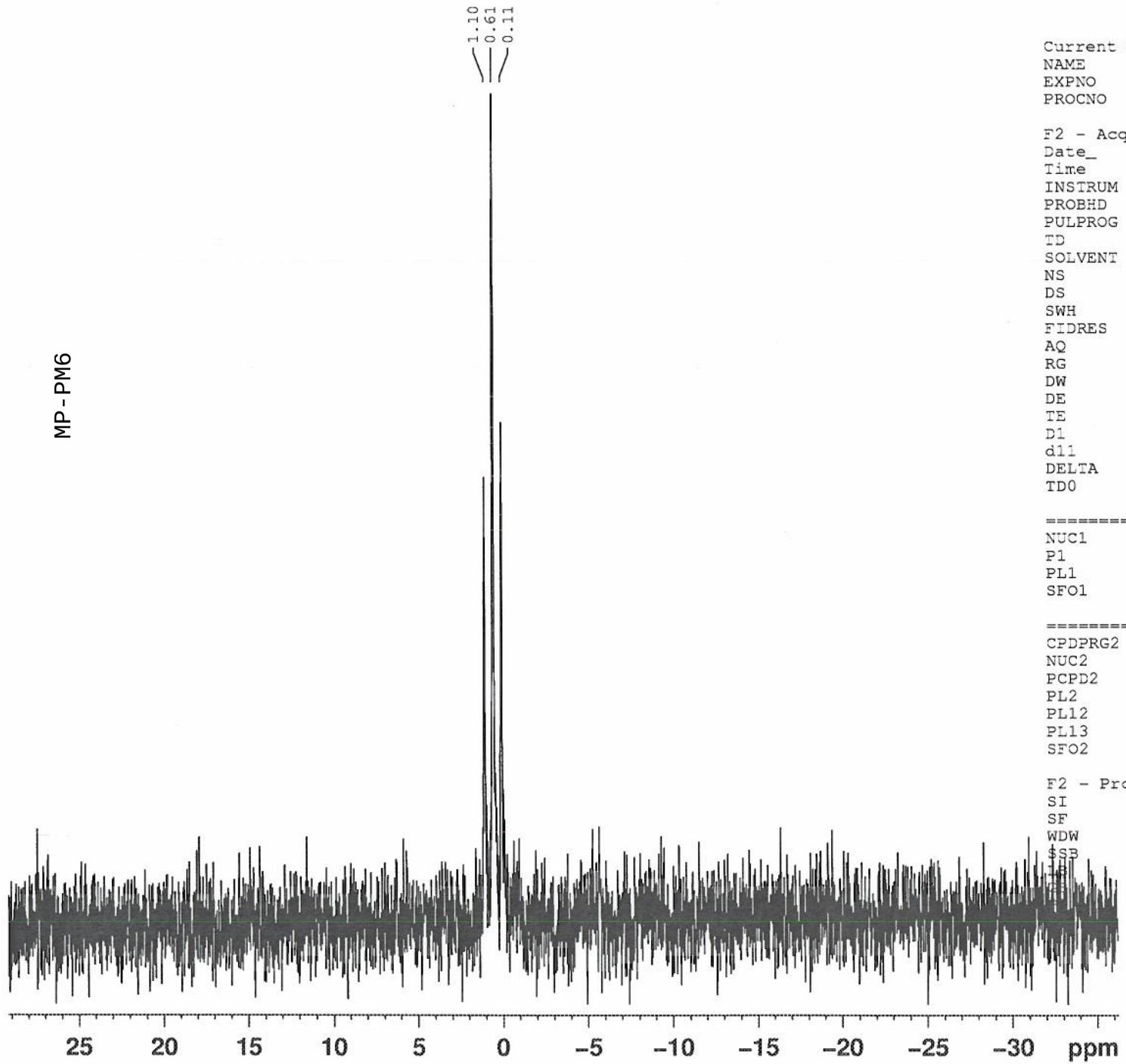
S51




```
Current Data Parameters
NAME                  BPPM-6-MP
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20090701
Time                      14.13
INSTRUM                   spect
PROBHD    5 mm TXI 1H/D-
PULPROG                    zgpr
TD                        65536
SOLVENT                    DMSO
NS                           16
DS                            2
SWH                   10330.578 Hz
FIDRES                0.157632 Hz
AQ                    3.1720407 sec
RG                        161.3
DW                       48.400 usec
DE                        7.50 usec
TE                       300.2 K
D1                  1.00000000 sec
d12                 0.00002000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                        8.00 usec
PL1                      -2.00 dB
PL9                      53.92 dB
SFO1                500.1317530 MHz

F2 - Processing parameters
SI                        32768
SF                  500.1300495 MHz
WDW                          EM
SSB                           0
LB                        0.30 Hz
GB                            0
PC                         1.00
```

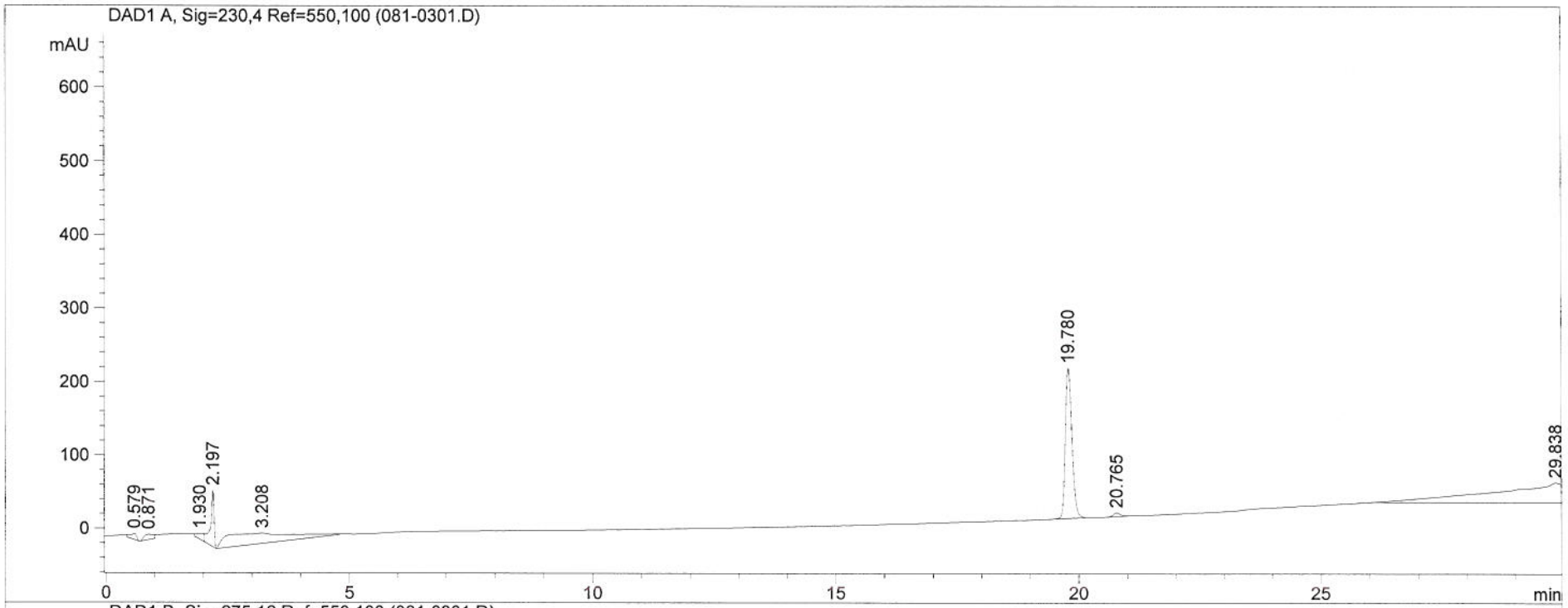


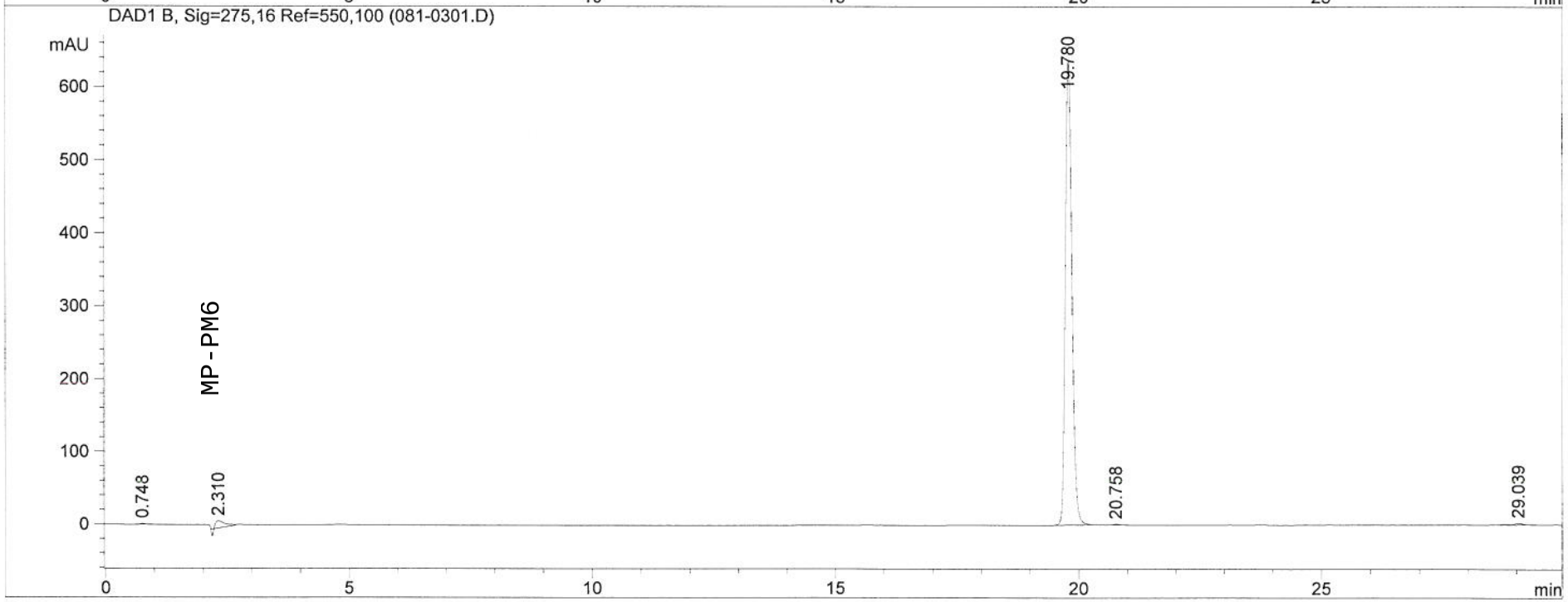

```
Current Data Parameters
NAME                  BPPM-6-MP
EXPNO                         5
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20090702
Time                       6.35
INSTRUM                   spect
PROBHD    5 mm TXI 1H/D-
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                       12288
DS                           4
SWH                 30030.029 Hz
FIDRES                0.458222 Hz
AQ                    1.0912410 sec
RG                       32768
DW                     16.650 usec
DE                      7.50 usec
TE                      301.2 K
D1                 2.00000000 sec
d11                0.03000000 sec
DELTA              1.89999998 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                     11.25 usec
PL1                     -5.50 dB
SFO1            125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  90.00 usec
PL2                     -2.00 dB
PL12                    18.50 dB
PL13                    20.00 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
```



MP-PM6

```
Current Data Parameters
NAME                 BPPM-6-MP
EXPNO                        7
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20090702
Time                     13.01
INSTRUM                  spect
PROBHD     5 mm TXI 1H/D-
PULPROG                 zgpg30
TD                       65536
SOLVENT                Acetone
NS                          1024
DS                             4
SWH                  80645.164 Hz
FIDRES                1.230548 Hz
AQ                    0.4063794 sec
RG                      20642.5
DW                        6.200 usec
DE                        7.50 usec
TE                       300.2 K
D1                   2.00000000 sec
d11                  0.03000000 sec
DELTA                1.89999998 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         31P
P1                        15.00 usec
PL1                       -4.50 dB
SFO1               202.4462121 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                         1H
PCPD2                     90.00 usec
PL2                       -2.00 dB
PL12                      18.50 dB
PL13                      20.00 dB
SFO2               500.1320005 MHz

F2 - Processing parameters
SI                           32768
SF                 202.4563350 MHz
WDW                             EM
SSB                              0
LB                            1.00 Hz
GB                               0
PC                            1.40
```


DAD1 A, Sig=230,4 Ref=550,100 (081-0301.D)
mAU
600
500
400
300
200
100
0
0.579
0.871
1.930
2.197
3.208
19.780
20.765
29.838
0
5
10
15
20
25
min


DAD1 B, Sig=275,16 Ref=550,100 (081-0301.D)
mAU
600
500
400
300
200
100
0
MP-PM6
0.748
2.310
19.780
20.758
29.039
0
5
10
15
20
25
min

S55