Supporting Information

Practical Asymmetric Synthesis of a Non-Peptidic $\alpha_v\beta_3$ Antagonist

Stephen P. Keen,* Cameron J. Cowden,* Brian C. Bishop, Karel M. J. Brands, Antony J. Davies, Ulf H. Dolling, David R. Lieberman, and Gavin W. Stewart

Table of Contents

Additional Experimental Procedures

General. All reactions were carried out under a nitrogen atmosphere, unless stated otherwise. All commercially available reagents and solvents were used as received. Melting points are uncorrected. Chemical shifts are reported in ppm and referenced to residual protons in the deuterated solvent. Flash column chromatography was carried out using 60Å silica gel (Davisil® 40–63 micron).

***N*-Hex-5-yn-1-yl-5-methyl-1,2,4-triazin-3-amine (5).** 5-Methyl-3-(methylthio)-1,2,4-triazine (**4**) (200 mg, 1.42 mmol), hex-5-yn-1-ylamine (152 mg, 1.56 mmol) and *N*,*N*-dimethylacetamide (2 mL) were placed in a heavy-walled glass tube. The tube was sealed and the reaction mixture was heated to 146 ºC (oil bath temperature) for 21 h. The resulting solution was allowed to cool to rt and then

partitioned between ^{i}PrOAc (5 mL) and water (2 mL). The two layers were separated and the organic phase was concentrated in vacuo. The crude product was then purified by flash column chromatography (1:4 EtOAc/hexane) to afford triazine **5** (150 mg, 56%): ^{1}H NMR (250 MHz, CD_2Cl_2) δ 8.40 (s, 1 H), 5.7–5.1 (br, 1 H), 3.50 (q, *J* = 6.7 Hz, 2 H), 2.32 (s, 3 H), 2.29–2.20 (m, 2 H), 1.99 (t, *J* = 2.6 Hz, 1 H), 1.82–1.55 (m, 4 H).

***N*-Hex-5-en-1-yl-*N*-(4-methyl-1,3-oxazol-2-yl)acetamide (7).** Cesium carbonate (3.25 g, 10 mmol) was added to a stirred solution of *N*-(4-methyl-1,3-oxazol-2-yl)acetamide (**6**) (0.70 g, 5.0 mmol) in 2:1 DMF/THF (15 mL) and the resulting reaction mixture was heated to 60 °C. After 20 min, 6-bromo-1-hexene (0.80 mL, 6.0 mmol) was added and stirring at 60 °C was continued for a further 1 h. The reaction mixture was left to age at rt overnight and then partitioned between EtOAc (50 mL) and water (50 mL). The two layers were separated and the aqueous phase was extracted with EtOAc (50 mL). The combined organic layers were dried (Na_2SO_4) and then concentrated in vacuo. The crude oil was purified by flash column chromatography (1:4 EtOAc/hexane) to afford oxazole **7** (0.85 g, 77%): ^{1}H NMR (250 MHz, CD_2Cl_2) δ 7.26 (q, *J* = 1.3 Hz, 1 H), 5.78 (ddt, *J* = 17.1, 10.2, 6.7 Hz, 1 H), 5.03–4.89 (m, 2 H), 3.76 (dd, *J* = 7.5, 7.2 Hz, 2 H), 2.18 (s, 3 H), 2.13 (d, *J* = 1.3 Hz, 3 H), 2.09–1.99 (m, 2 H), 1.63–1.48 (m, 2 H), 1.43–1.30 (m, 2 H); ^{13}C NMR (63 MHz, CD_2Cl_2) δ 170.4, 156.3, 139.2, 137.5, 132.7, 114.9, 47.0, 33.9, 28.2, 26.4, 23.8, 12.1.

***N*-Benzyl-*N*-(4-methyl-1,3-oxazol-2-yl)hex-5-enamide (9).** To a stirred solution of 5-hexenoic acid (1.03 g, 9.0 mmol) and oxalyl chloride (0.95 mL, 10.9 mmol) in DCM (9 mL) was carefully added DMF (0.24 mL) at 0 °C. The resulting reaction mixture was stirred at rt overnight and then concentrated in vacuo. To the brown residue obtained was added a small volume of hexane (~3 mL) and, after stirring for 5 min, the mixture was filtered. The filtrate was then concentrated in vacuo to afford 5-hexenoyl chloride (0.66 g) as a colorless oil. This material was used as follows without further purification.

To a solution of *N*-benzyl-4-methyl-1,3-oxazol-2-amine (**8**) (100 mg, 0.53 mmol) in THF (10 mL) was added sodium hydride (23 mg, 60% in mineral oil, 0.58 mmol). After stirring for 10 min, the above 5-hexenoyl chloride (70 mg, 0.53 mmol) was added and the resulting reaction mixture was left to age at

rt overnight. The reaction was then quenched by addition of water (25 mL) and extracted twice with ^{i}PrOAc (2 × 25 mL). The combined extracts were concentrated in vacuo and the residue obtained was purified by flash column chromatography (1:4 EtOAc/hexane) to afford oxazole **9** (68 mg, 45%) as a light yellow oil: ^{1}H NMR (250 MHz, CD_2Cl_2) δ 7.35–7.21 (m, 6 H), 5.79 (ddt, *J* = 17.1, 10.2, 6.7 Hz, 1 H), 5.06–4.93 (m, 4 H), 2.58 (dd, *J* = 7.6, 7.3 Hz, 2 H), 2.14–2.02 (m, 2 H), 2.12 (d, *J* = 1.4 Hz, 3 H), 1.81–1.68 (m, 2 H).

***N*-Benzyl-*N*-hex-5-en-1-yl-4-methyl-1,3-oxazol-2-amine (10).** To a solution of *N*-benzyl-4-methyl-1,3-oxazol-2-amine (**8**) (0.22 g, 1.17 mmol) in THF (10 mL) was added KHMDS (0.30 g, 1.51 mmol) followed by 6-bromo-1-hexene (0.22 mL, 1.64 mmol). The resulting stirred reaction mixture was then heated to reflux for 16 h. After cooling to rt, water (25 mL) was added and the mixture was extracted three times with *tert*-butyl methyl ether (3 × 25 mL). The combined extracts were concentrated in vacuo to afford oxazole **10** (0.26 g, 82%) as a yellow oil, which was used without further purification: ^{1}H NMR (250 MHz, CD_2Cl_2) δ 7.40–7.22 (m, 5 H), 6.96 (q, *J* = 1.4 Hz, 1 H), 5.80 (ddt, *J* = 17.1, 10.2, 6.7 Hz, 1 H), 5.06–4.91 (m, 2 H), 4.61 (s, 2 H), 3.35 (dd, *J* = 7.5, 7.3 Hz, 2 H), 2.13–1.95 (m, 2 H), 2.04 (d, *J* = 1.4 Hz, 3 H), 1.68–1.53 (m, 2 H), 1.45–1.30 (m, 2 H); ^{13}C NMR (63 MHz, CD_2Cl_2) δ 162.2, 139.2, 138.7, 136.9, 129.0, 128.0, 127.8, 127.7, 114.9, 52.0, 48.3, 34.0, 27.6, 26.6, 12.3.

***tert*-Butyl 5,6,7,8-tetrahydro-9*H*-pyrido[2,3-*b*]azepine-9-carboxylate (15).** A stirred solution of chloride **14** (8.00 g, 28.2 mmol) in THF (160 mL) was cooled to 0 °C. KHMDS (56.3 mL, 0.5 M solution in toluene, 28.2 mmol) was then added and the resulting reaction mixture was warmed to reflux. After stirring at this temperature for 3 h, the reaction mixture was cooled to rt and diluted with water (100 mL). The THF was then removed in vacuo and the aqueous mixture was extracted twice with EtOAc (2 × 100 mL). The combined organic layers were washed with saturated brine (100 mL) and then concentrated in vacuo. The resulting yellow oil was purified by flash column chromatography (2:1 *tert*-butyl methyl ether/hexane) to afford pyridoazepine **15** (6.62 g, 95%) as a white solid: mp 70.5–72.0 °C; ^{1}H NMR (400 MHz, CD_2Cl_2) δ 8.29 (dd, *J* = 4.8, 1.8 Hz, 1 H), 7.55 (dd, *J* = 7.5, 1.7 Hz, 1 H), 7.11 (dd, *J* = 7.5, 4.8 Hz, 1 H), 4.1–2.8 (br, 2 H), 2.71 (dd, *J* = 6.4, 5.0 Hz, 2 H), 1.86–1.52 (m, 4 H),

1.39 (s, 9 H); ^{13}C NMR (101 MHz, CD_2Cl_2) δ 156.4, 154.3, 147.2, 139.0, 135.4, 122.9, 80.4, 47.4, 34.1, 30.1, 28.7, 26.6. Anal Calcd for $C_{14}H_{20}N_2O_2$: C, 67.71; H, 8.12; N, 11.28. Found: C, 67.74; H, 8.18; N, 11.40.

***N*-(6-Chloropyridin-2-yl)-2,2-dimethylpropanamide (17).** A stirred solution of 2-amino-6-chloropyridine (3.93 g, 30.6 mmol) and triethylamine (4.69 mL, 33.7 mmol) in toluene (20 mL) was warmed to 50 °C. Trimethylacetyl chloride (3.96 mL, 32.2 mmol) was then added and the resulting reaction mixture was aged overnight at 50 °C. After cooling to rt, the reaction was quenched by addition of 2 M hydrochloric acid (100 mL) and then extracted with ^{i}PrOAc (3 × 100 mL). The combined extracts were washed with water and then concentrated to dryness. Recrystallization of the residue obtained from *tert*-butyl methyl ether and hexane afforded *N*-Piv aminopyridine **17** (4.37 g, 67%): mp 87.5–88.5 °C (lit.[1] 86–89 °C); ^{1}H NMR (250 MHz, CD_2Cl_2) δ 8.15 (dd, *J* = 8.2, 0.7 Hz, 1 H), 7.99 (br s, 1 H), 7.66 (dd, *J* = 8.2, 7.8 Hz, 1 H), 7.05 (dd, *J* = 7.7, 0.8 Hz, 1 H), 1.29 (s, 9 H); ^{13}C NMR (101 MHz, CD_2Cl_2) δ 177.5, 152.4, 149.4, 141.5, 119.9, 112.4, 40.4, 27.8. Anal Calcd for $C_{10}H_{13}ClN_2O$: C, 56.47; H, 6.16; N, 13.17. Found: C, 56.50; H, 6.18; N, 13.17.

***tert*-Butyl 2-[(1*E*)-3-ethoxy-3-oxoprop-1-en-1-yl]-5,6,7,8-tetrahydro-9*H*-pyrido[2,3-*b*]azepine-9-carboxylate (21).** A stirred solution of *N*-Boc pyridoazepine **20** (3.10 g, 11.0 mmol), ethyl acrylate (2.40 mL, 22.2 mmol), triethylamine (3.06 mL, 22.0 mmol), tetrabutylammonium bromide (3.50 g, 10.9 mmol), palladium acetate (0.25 g, 1.1 mmol) and bis(diphenylphosphino)ferrocene (0.61 g, 1.1 mmol) in DMF (30 mL) was degassed and then put under an atmosphere of nitrogen. The resulting reaction mixture was heated to 115 °C for 15 h and then allowed to cool to rt. Once cooled, the mixture was diluted with EtOAc (60 mL) and washed sequentially with 10% aqueous citric acid (60 mL), 10% aqueous Na_2CO_3 (30 mL) and saturated brine (30 mL). The resulting organic layer was concentrated in vacuo and the residue obtained was purified by flash column chromatography (1:4 EtOAc/hexane) to afford ester **21** (3.08 g, 81%): mp 105.5–106.5 °C; ^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.60 (d, *J* = 15.6 Hz,

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(1) Turner, J. A. *J. Org. Chem.* **1983**, *48*, 3401–3408.

1 H), 7.58 (d, J = 7.6 Hz, 1 H), 7.26 (d, J = 7.6 Hz, 1 H), 6.82 (d, J = 15.6 Hz, 1 H), 4.23 (q, J = 7.1 Hz, 2 H), 4.0–3.1 (br, 2 H), 2.73 (dd, J = 6.4, 5.0 Hz, 2 H), 1.86–1.79 (m, 2 H), 1.74–1.61 (m, 2 H), 1.39 (s, 9 H), 1.31 (t, J = 7.1 Hz, 3 H); ^{13}C NMR (101 MHz, CD_2Cl_2) δ 167.2, 156.5, 154.4, 150.9, 143.3, 139.7, 136.5, 123.2, 122.5, 80.5, 61.1, 47.3, 34.1, 29.9, 28.6, 26.6, 14.7. Anal Calcd for $C_{19}H_{26}N_2O_4$: C, 65.87; H, 7.56; N, 8.09. Found: C, 65.91; H, 7.63; N, 8.15.

(3*R*)-5-Methoxy-3-(2-methoxypyrimidin-5-yl)-5-oxopentanoic acid (*ent*-29). Toluene (100 mL) was added to a round-bottom flask containing solid anhydride **25** (5.00 g, 22.5 mmol) and quinine (7.16 g, 22.1 mmol) under a nitrogen atmosphere. The slurry obtained was cooled, with stirring, to –40 °C and methanol (4.50 mL, 111 mmol) was then added. The resulting reaction mixture was stirred at this temperature for 17 h and then warmed to 40 °C. The slurry that had now formed was aged at 40 °C for 2 h and then at rt for 20 h. Following addition of toluene (100 mL), the batch was again stirred at rt overnight and then filtered to afford the quinine salt of *ent*-**29** (8.49 g, 65%, 87% ee) as an off-white solid. This salt was treated with 1 M hydrochloric acid (35 mL) and the resulting mixture was stirred at rt for 30 min, during which acid-ester crystallized from solution. The solid was then collected by filtration, washing the wet-cake with water (3 × 5 mL). Drying under vacuum at 50 °C provided the title compound (2.38 g, 42%) as a beige solid. The enantiomeric excess of this product was determined as 98.3% by chiral stationary phase HPLC (250 × 4.6 mm Chirobiotic T column; UV detection at 274 nm; isocratic elution with 80:20 aqueous Et_3N (0.036 M)–AcOH (0.044 M) / methanol for 20 min; 0.5 mL/min; 25 °C. Retention times: (*R*)-acid ester *ent*-**29** = 10.5 min; (*S*)-acid ester **29** = 11.4 min): mp 143.5–146.0 °C; $[\alpha]^{20}_D$ -12.1 (*c* 2.0 in MeOH); ^{1}H NMR and ^{13}C NMR data were identical to those reported in the Experimental Section for acid-ester **29**; Anal Calcd for $C_{11}H_{14}N_2O_5$: C, 51.97; H, 5.55; N, 11.02. Found: C, 51.95; H, 5.56; N, 10.77.

(3*S*)-6-(Diisopropoxyphosphoryl)-3-(2-methoxypyrimidin-5-yl)-5-oxohexanoic acid (30). To a stirred solution of diisopropyl methylphosphonate (2.83 g, 15.7 mmol) in THF (30 mL) was added butyllithium (6.29 mL, 2.5 M solution in hexanes, 15.7 mmol) at –78 °C. The resulting reaction mixture was stirred for 1 h at –78 °C and a solution of magnesium bromide diethyl etherate (4.46 g, 17.3

mmol) in diethyl ether (120 mL) was then added. Once the addition was complete, the cooling bath was removed and the cloudy reaction mixture was stirred for 30 min. The resulting batch was then cooled back to –78 °C and a solution of acid-ester *ent*-**29** (1.00 g, 3.94 mmol) in THF (12 mL) was added. Once the addition was complete, the reaction was allowed to gradually warm to rt overnight. The reaction mixture was then quenched by addition of water (50 mL) and the organics were removed in vacuo. The resulting aqueous mixture was acidified with 2 M hydrochloric acid (40 mL) and then extracted into DCM (80 mL). The organic layer was then treated with 1 M aqueous NaOH (40 mL) and the two layers were separated. The resulting aqueous phase was washed three times with DCM (3 × 30 mL) and then acidified to pH 1 with conc. hydrochloric acid. The mixture obtained was extracted three times with DCM (3 × 50 mL) and the combined extracts were concentrated in vacuo to afford phosphonate **30** (1.22 g, 77%) as a pale yellow oil: ^{1}H NMR (250 MHz, CD_2Cl_2) δ 9.4–8.2 (br, 1 H), 8.45 (s, 2 H), 4.72–4.47 (m, 2 H), 3.93 (s, 3 H), 3.60 (app quintet, *J* = 7.0 Hz, 1 H), 3.14–2.88 (m, 4 H), 2.72 (dd, *J* = 16.3, 6.7 Hz, 1 H), 2.58 (dd, *J* = 16.3, 8.0 Hz, 1 H), 1.28–1.17 (m, 12 H); ^{13}C NMR (63 MHz, CD_2Cl_2) δ 200.0 (d, *J* = 6 Hz), 173.9, 164.9, 159.2, 130.2, 72.5 (d, *J* = 7 Hz), 72.4 (d, *J* = 7 Hz), 55.3, 49.0, 44.2 (d, *J* = 128 Hz), 40.1, 31.8, 24.2–23.9 (m).

Methyl (3*S*)-6-(diisopropoxyphosphoryl)-3-(2-methoxypyrimidin-5-yl)-5-oxohexanoate (31). Sulfuric acid (0.2 mL, 3.76 mmol) was added to a stirred solution of phosphonate **30** (822 mg, 2.04 mmol) in methanol (15 mL) and the resulting reaction mixture was then heated to reflux for 15 h. After cooling to rt, the reaction was quenched by addition of 10% aqueous $KHCO_3$ (10 mL) and then diluted with half-saturated brine (9 mL). The aqueous mixture obtained was extracted three times with EtOAc (2 × 10 mL, then 20 mL) and the combined extracts were concentrated in vacuo to afford phosphonate **31** (819 mg, 96%) as a yellow oil: ^{1}H NMR (250 MHz, CD_2Cl_2) δ 8.40 (s, 2 H), 4.70–4.46 (m, 2 H), 3.93 (s, 3 H), 3.68–3.53 (m, 1 H), 3.58 (s, 3 H), 3.18–2.84 (m, 4 H), 2.71 (dd, *J* = 16.1, 6.5 Hz, 1 H), 2.57 (dd, *J* = 16.1, 8.6 Hz, 1 H), 1.30–1.18 (m, 12 H); ^{13}C NMR (63 MHz, CD_2Cl_2) δ 200.0 (d, *J* = 6 Hz), 172.0, 165.3, 159.1, 129.8, 71.9 (d, *J* = 7 Hz), 55.2, 52.1, 49.0, 44.6 (d, *J* = 127 Hz), 40.1, 31.9, 24.3–23.9 (m).

Methyl (3*S*)-3-(2-methoxypyrimidin-5-yl)-5-oxo-6-(triphenylphosphoranylidene)hexanoate (34). *Via isobutoxy anhydride **32**:* Isobutyl chloroformate (1.12 mL, 8.63 mmol) was added to a stirred solution of acid-ester **29** (2.00 g, 7.87 mmol) and triethylamine (1.31 mL, 9.42 mmol) in DCM (20 mL) at –40 °C. The resulting reaction mixture was allowed to warm to 0 °C, aged at this temperature for 1 h and 10% aqueous $KHCO_3$ (10 mL) was then added. The biphasic mixture was separated and the aqueous layer was then re-extracted with DCM (10 mL). The combined organic layers were washed with saturated brine (10 mL), dried (Na_2SO_4) and then concentrated in vacuo to afford anhydride **32** which was used without further purification.

Butyllithium (10.3 mL, 1.6 M in hexanes, 16.5 mmol) was slowly added to a stirred suspension of methyltriphenylphosphonium bromide (6.18 g, 17.3 mmol) in THF (45 mL) at –70 °C. Once the addition was complete, the batch was allowed to warm to 0 °C, aged at this temperature for 30 min and then re-cooled to –78 °C. A solution of mixed anhydride **32**, prepared above, in THF (15 mL) was then added over 5 min. The resulting batch was aged for 2 h at –78 °C and then quenched by addition of 5% aqueous NH_4Cl (30 mL). Once the quench was complete, the mixture was warmed to rt and then extracted twice with EtOAc (2 × 30 mL). The combined extracts were washed sequentially with 5% aqueous NaCl (20 mL) and saturated brine (20 mL), dried (Na_2SO_4) and then concentrated in vacuo. The partially solidified residue was triturated with 95:5 *tert*-butyl methyl ether/hexane and the resulting slurry was then aged at rt for 6 h before filtering. The solid collected was dried under vacuum to afford phosphorane **34** (2.64 g, 65%) as a cream colored solid. ^{1}H NMR and ^{13}C NMR data were identical to those reported in the Experimental Section.

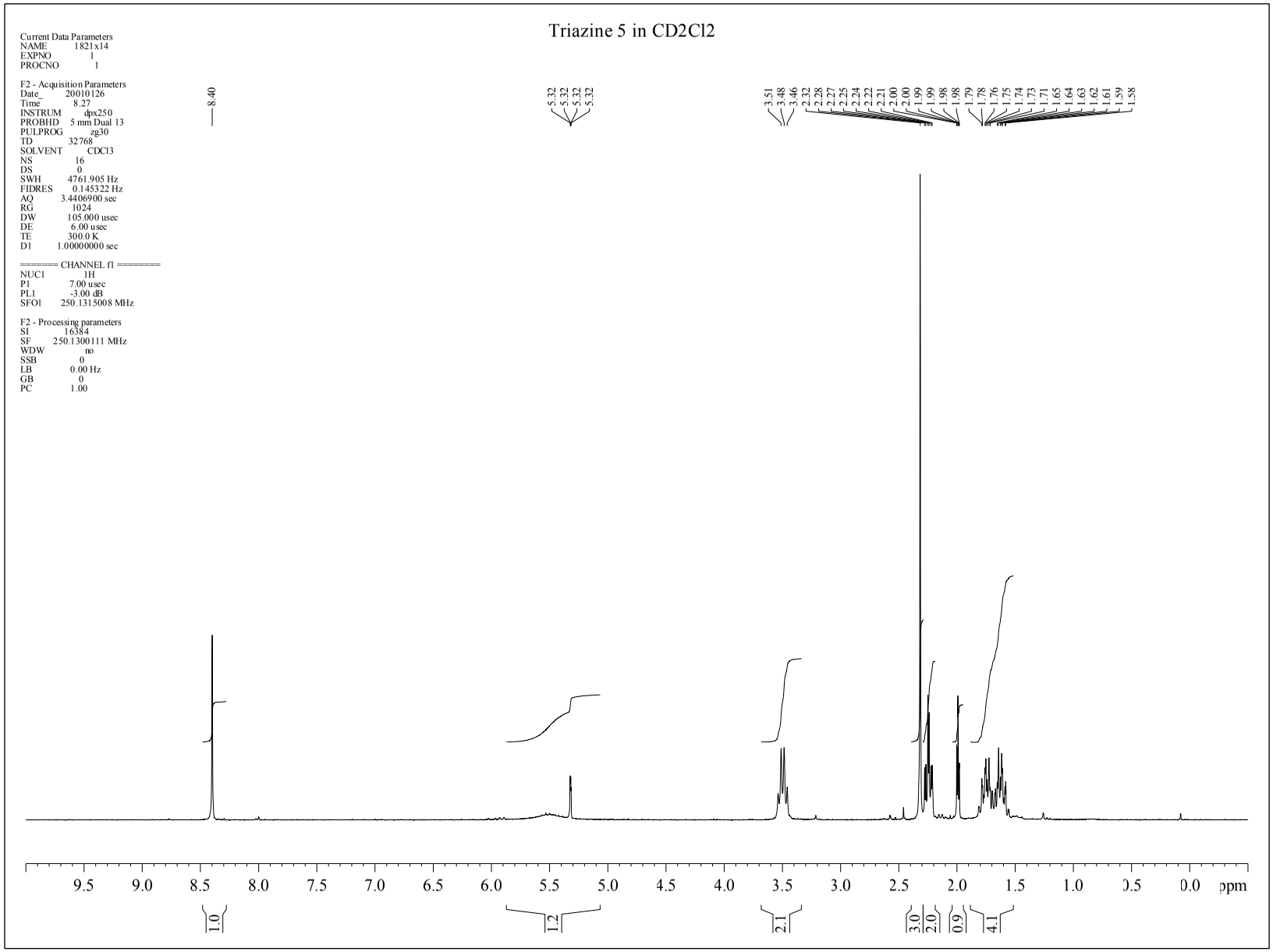
Methyl (3*S*)-3-(2-methoxypyrimidin-5-yl)-5-oxo-6-(triphenylphosphoranylidene)hexanoate (34). *Via* tert-*butyl mixed anhydride **33** – removal of $Et_3N{\cdot}HCl$ by filtration:* Triethylamine (5.76 mL, 41.4 mmol) was added, over a period of 20 min, to a stirred slurry of acid-ester **29** (10.0 g, 39.4 mmol) and trimethylacetyl chloride (4.92 mL, 39.9 mmol) in THF (75 mL) at 0 °C. The resulting reaction mixture was aged at 0 °C for 20 min and then filtered, washing through with THF (40 mL). The filtrate and

wash were combined, and this crude solution of mixed anhydride **33** was then used directly as described below.

Butyllithium (34.8 mL, 2.5 M in hexanes, 87.0 mmol) was slowly added to a stirred suspension of methyltriphenylphosphonium bromide (32.4 g, 90.7 mmol) in THF (227 mL) at –60 °C. Once the addition was complete, the batch was allowed to warm to 0 °C, aged at this temperature for 2 h and then re-cooled to –78 °C. The solution of mixed anhydride **33**, prepared above, was then slowly added over 25 min, keeping the internal temperature below –70 °C. The resulting batch was aged for 40 min at –78 °C and then quenched into a cooled (0 °C) aqueous solution of potassium dihydrogenphosphate (2.68 g KH_2PO_4 in 150 mL of water). Once the quench was complete, the mixture was extracted twice with ^{i}PrOAc (2 × 225 mL) and the combined extracts were washed with saturated aqueous NaCl (2 × 100 mL). The organic layer was then concentrated under reduced pressure to a volume of 100 mL, at which point product began to crystallize from solution. The resulting slurry was stirred at rt overnight, cooled to 0 °C and the solid was collected by filtration. Drying under vacuum afforded phosphorane **34** (14.7 g, 73%) as a cream colored solid. ^{1}H NMR and ^{13}C NMR data were identical to those reported in the Experimental Section.

***tert*-Butyl 2-[(7*S*)-9-methoxy-7-(2-methoxypyrimidin-5-yl)-5,9-dioxonon-3-en-1-yl]-5,6,7,8-tetrahydro-9*H*-pyrido[2,3-*b*]azepine-9-carboxylate (35).** *Via Horner-Wadsworth-Emmons reaction:* KHMDS (1.94 mL, 0.5 M solution in toluene, 0.97 mmol) was slowly added to a stirred solution of phosphonate **31** (405 mg, 0.97 mmol) in THF (3 mL) at –78 °C. The resulting reaction mixture was aged at this temperature for 20 min and then allowed to warm to 0 °C. After stirring at 0 °C for 1 h, a solution of aldehyde **24** (297 mg, 0.98 mmol) in THF (2 mL) was added and the batch was aged for a further 3 h at this temperature. The cooling bath was then removed and the reaction was stirred at rt overnight (23 h). Water was added to the resulting reaction mixture and the two layers were separated. The aqueous layer was then extracted with EtOAc and the combined organic layers were concentrated to dryness. The residue obtained was purified by flash column chromatography (100:0 to 97:3 EtOAc/MeOH gradient elution) to afford enone **35** (275 mg, 53%) as a colorless oil: ^{1}H NMR (250

MHz, CD_2Cl_2) δ 8.38 (s, 2 H), 7.46 (d, *J* = 7.6 Hz, 1 H), 6.94 (d, *J* = 7.6 Hz, 1 H), 6.86 (dt, *J* = 15.9, 6.7 Hz, 1 H), 6.05 (dt, *J* = 15.9, 1.5 Hz, 1 H), 3.93 (s, 3 H), 3.8–3.2 (br, 2H), 3.71–3.55 (m, 1 H), 3.57 (s, 3 H), 3.02–2.80 (m, 4 H), 2.78–2.53 (m, 6 H), 1.85–1.50 (m, 4 H), 1.38 (s, 9H); ^{13}C NMR (63 MHz, CD_2Cl_2) δ 197.6, 172.0, 165.3, 159.0, 157.9, 155.6, 154.2, 147.6, 139.5, 132.6, 130.9, 130.1, 121.7, 80.1, 55.2, 52.1, 47.3, 45.4, 40.2, 36.3, 33.6, 32.7, 32.4, 30.0, 28.6, 26.6.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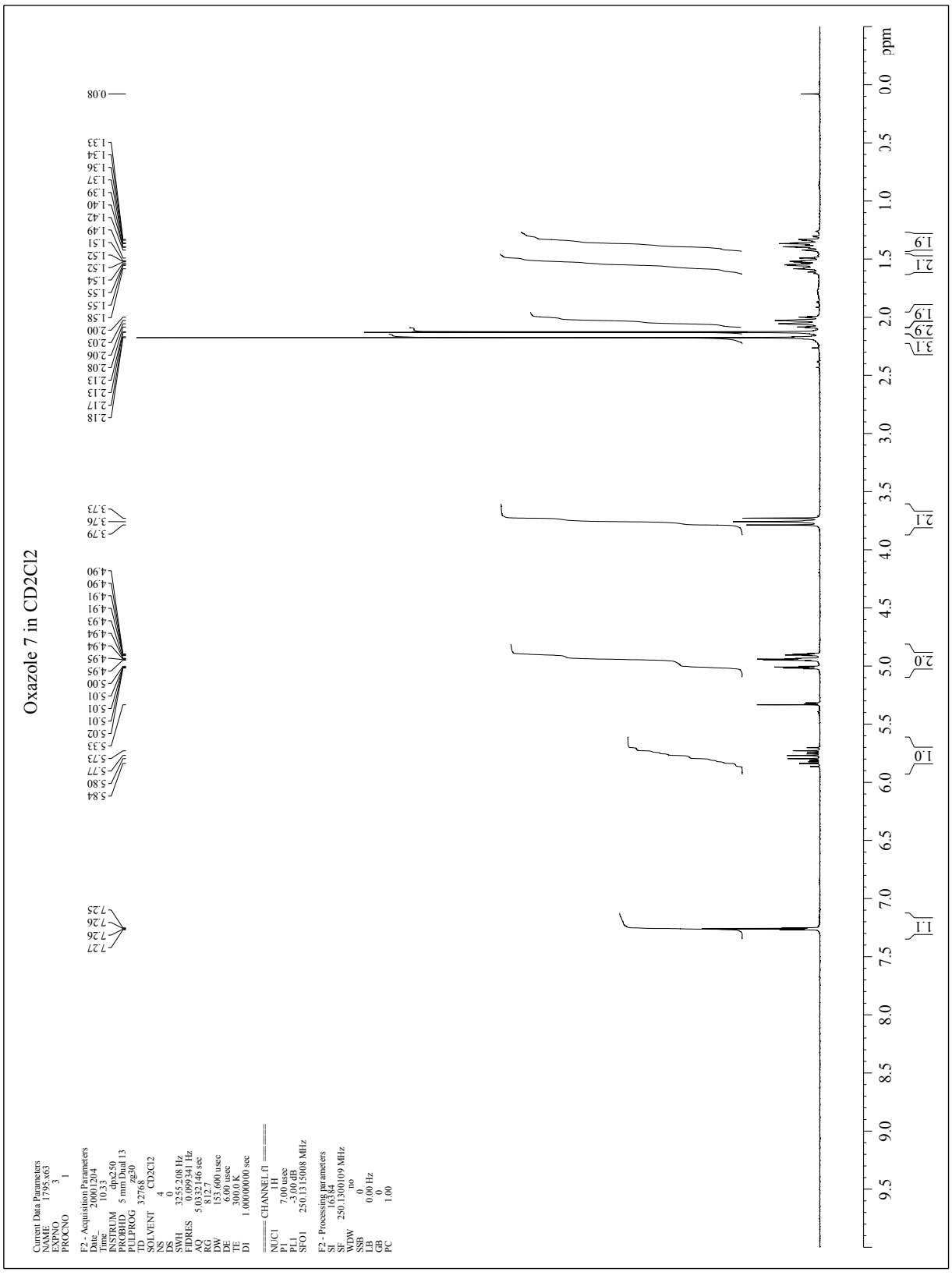
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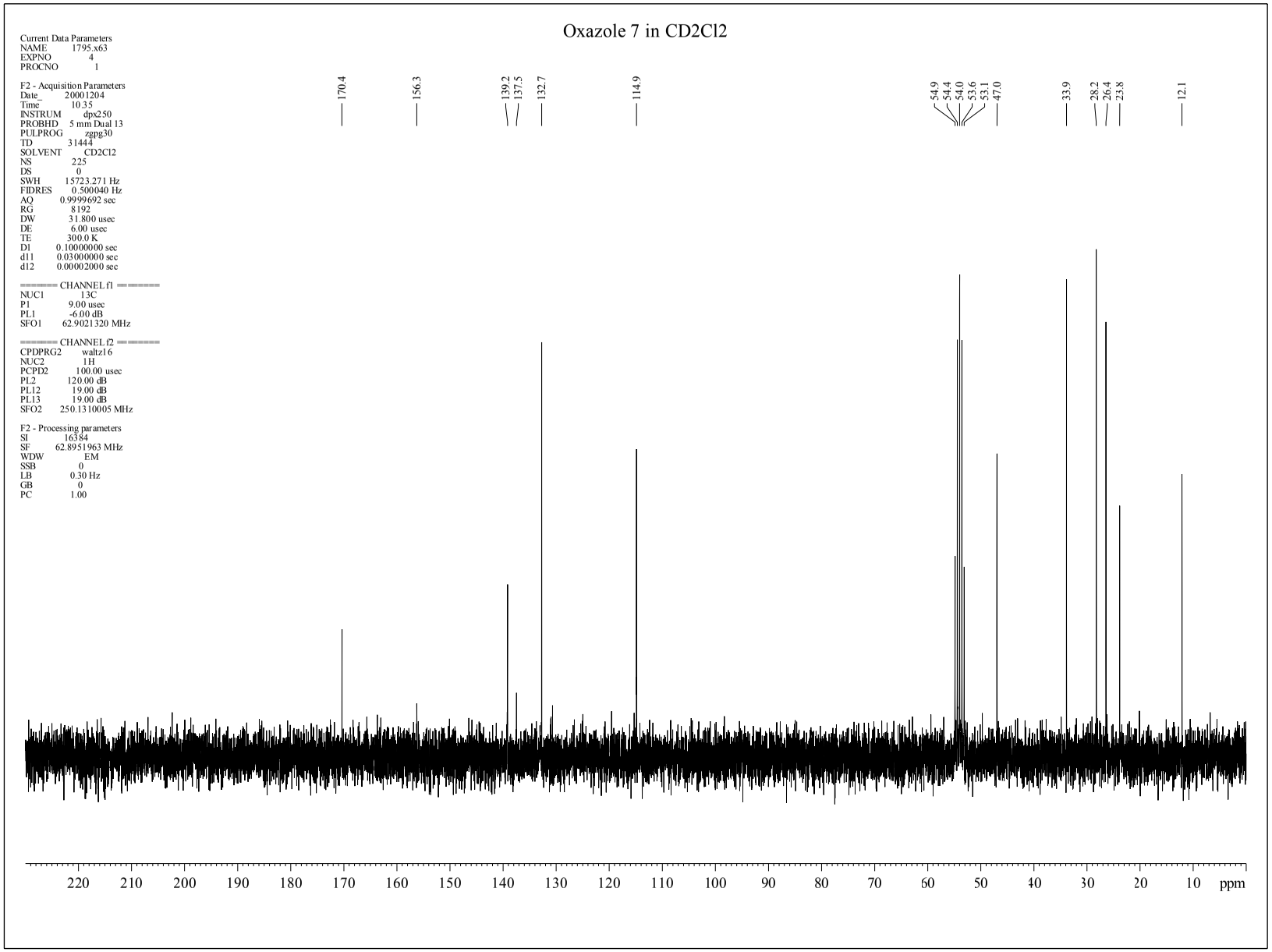
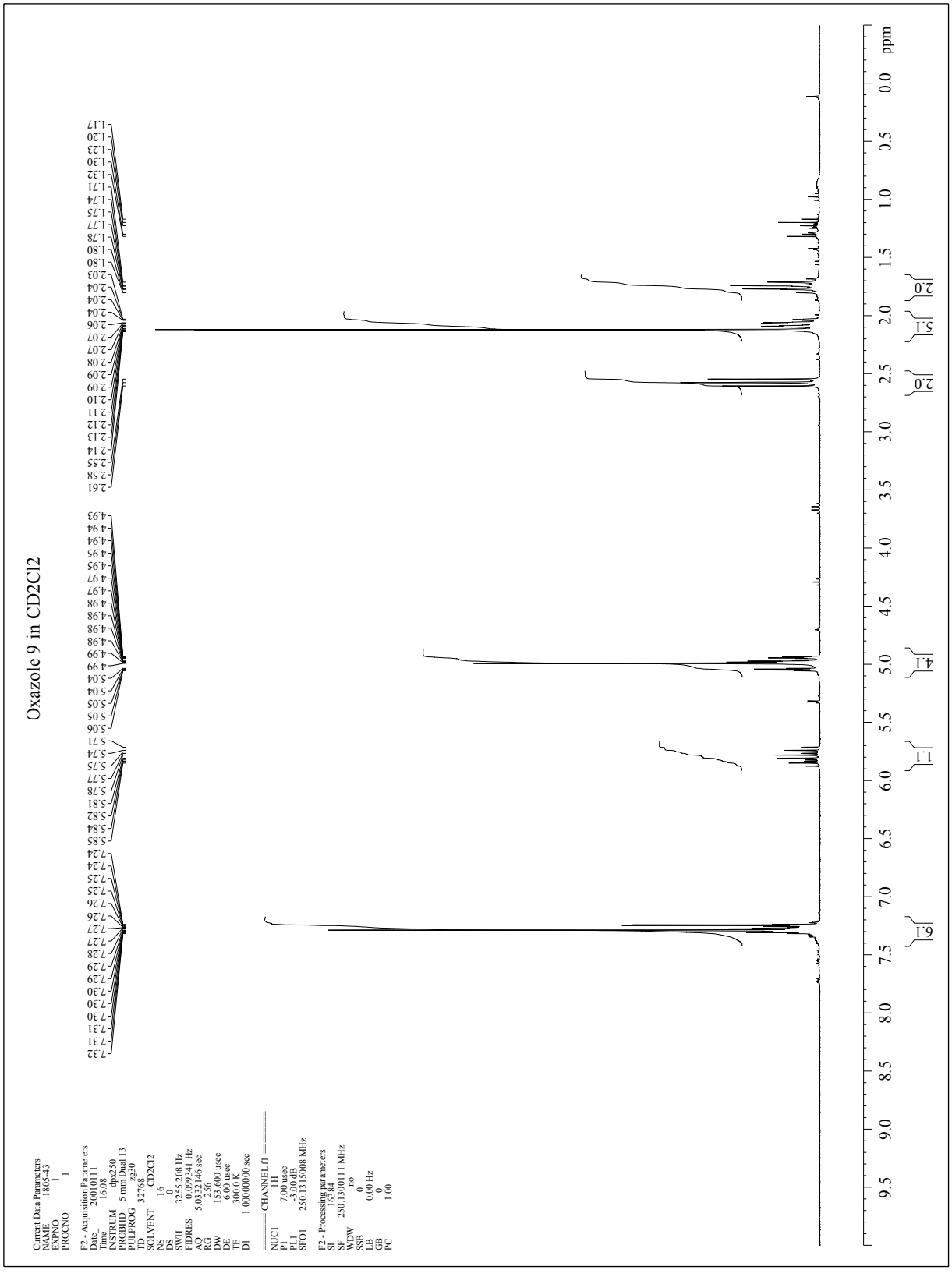
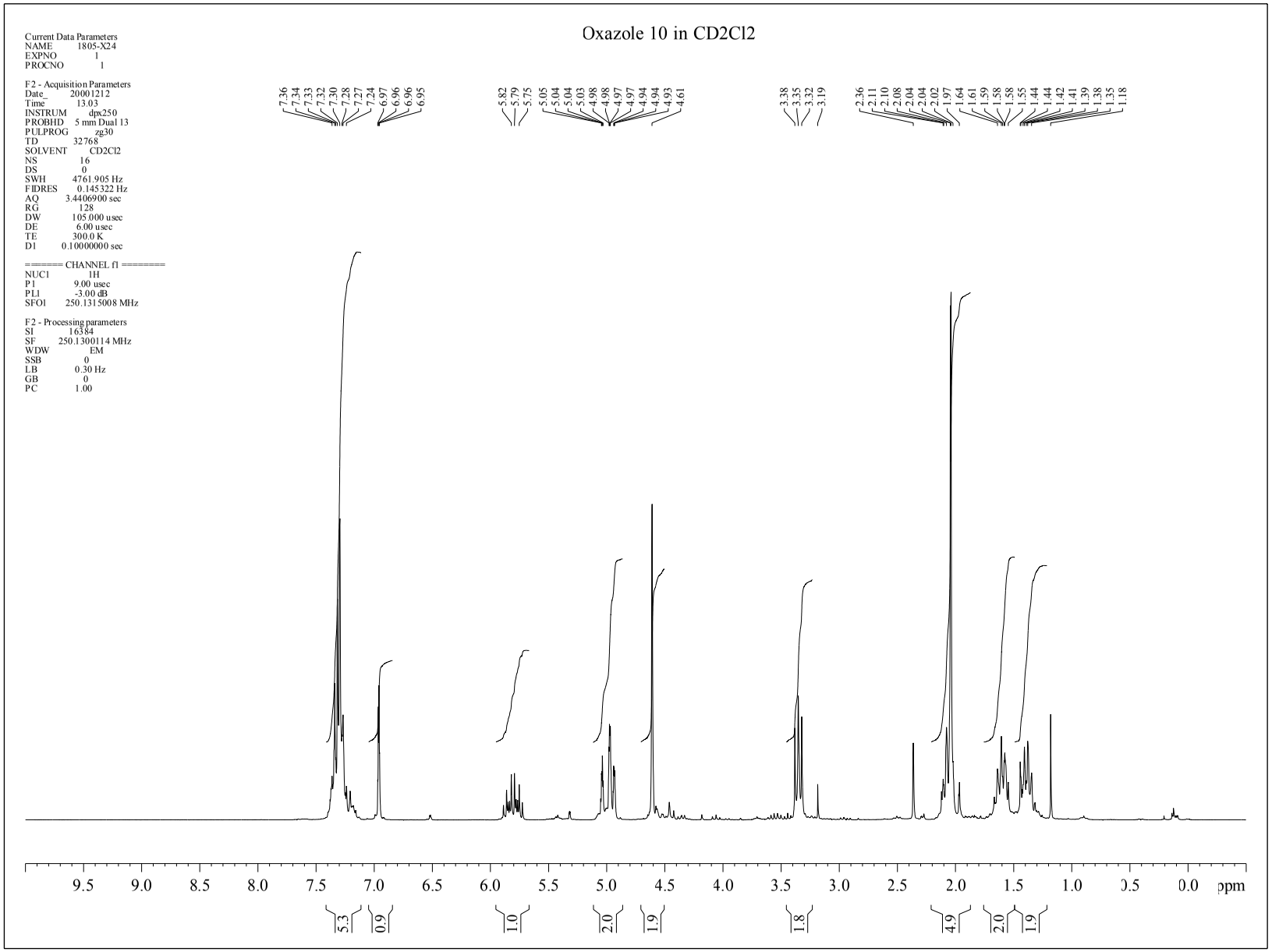
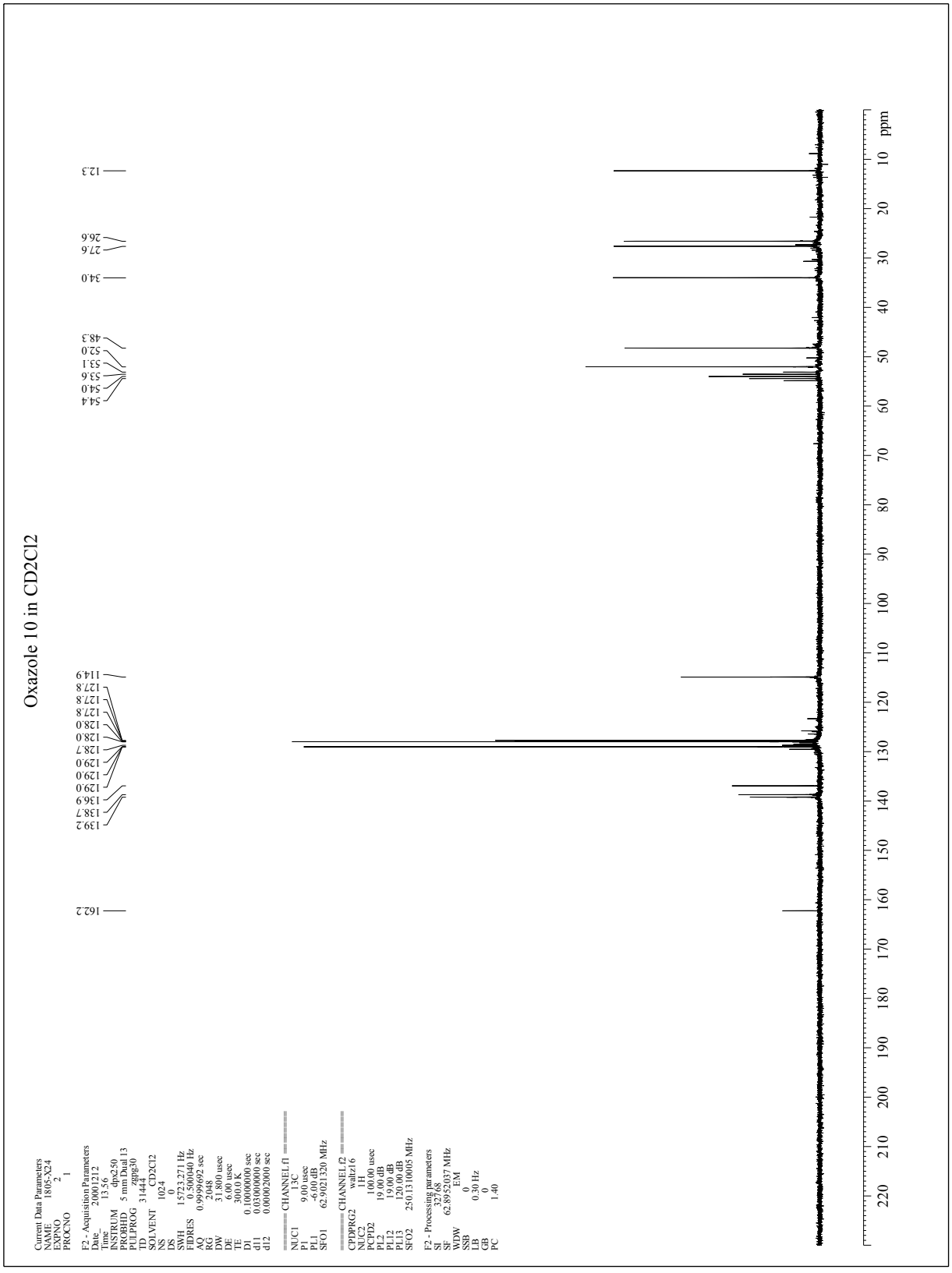
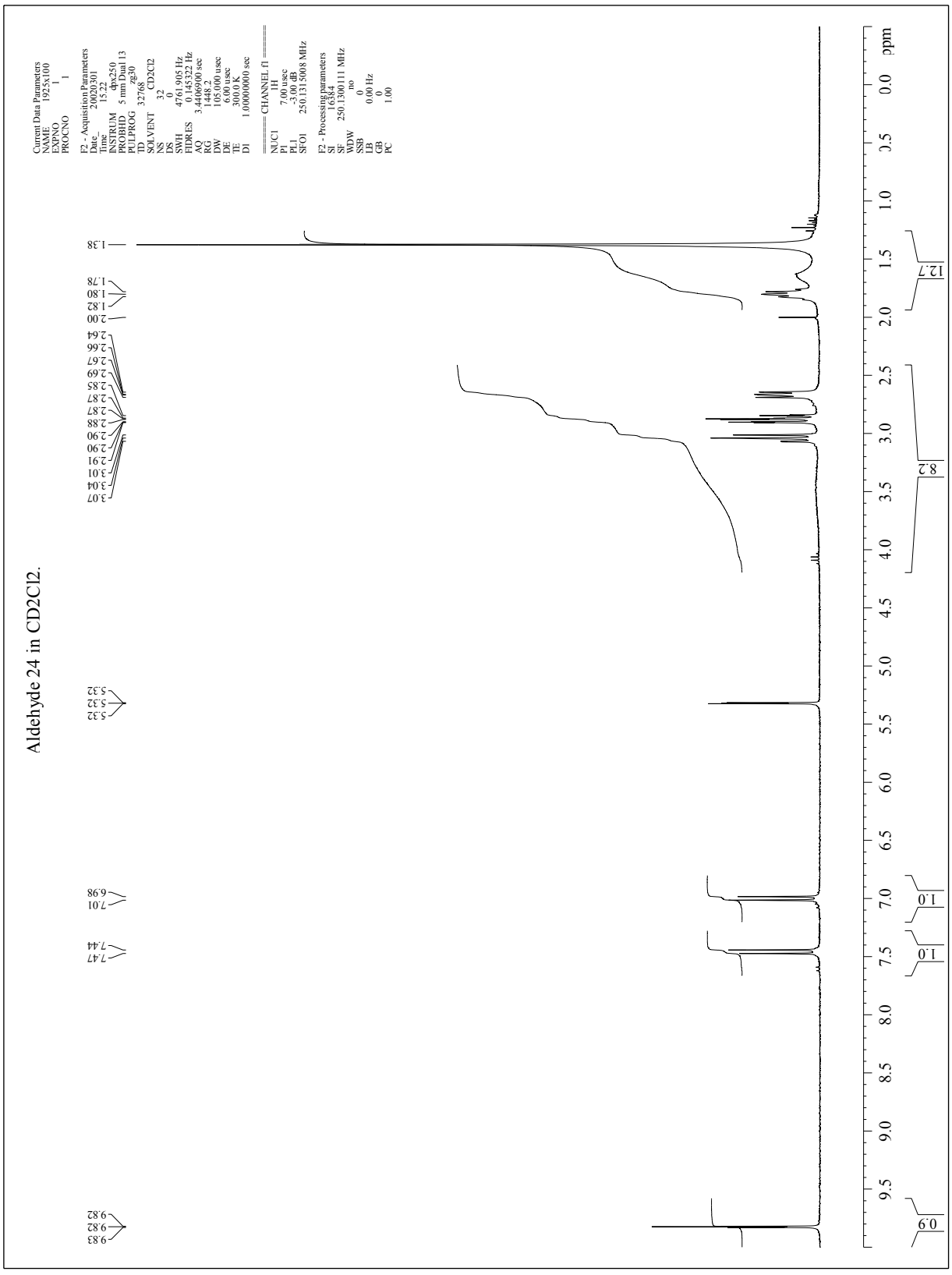
2.00
1.58
1.55
1.55
1.54
1.52
1.52
1.51
1.49
1.42
1.40
1.39
1.37
1.36
1.34
1.33
0.08
1.1
1.0
2.0
2.1
3.1
2.9
1.9
2.1
1.9
ppm

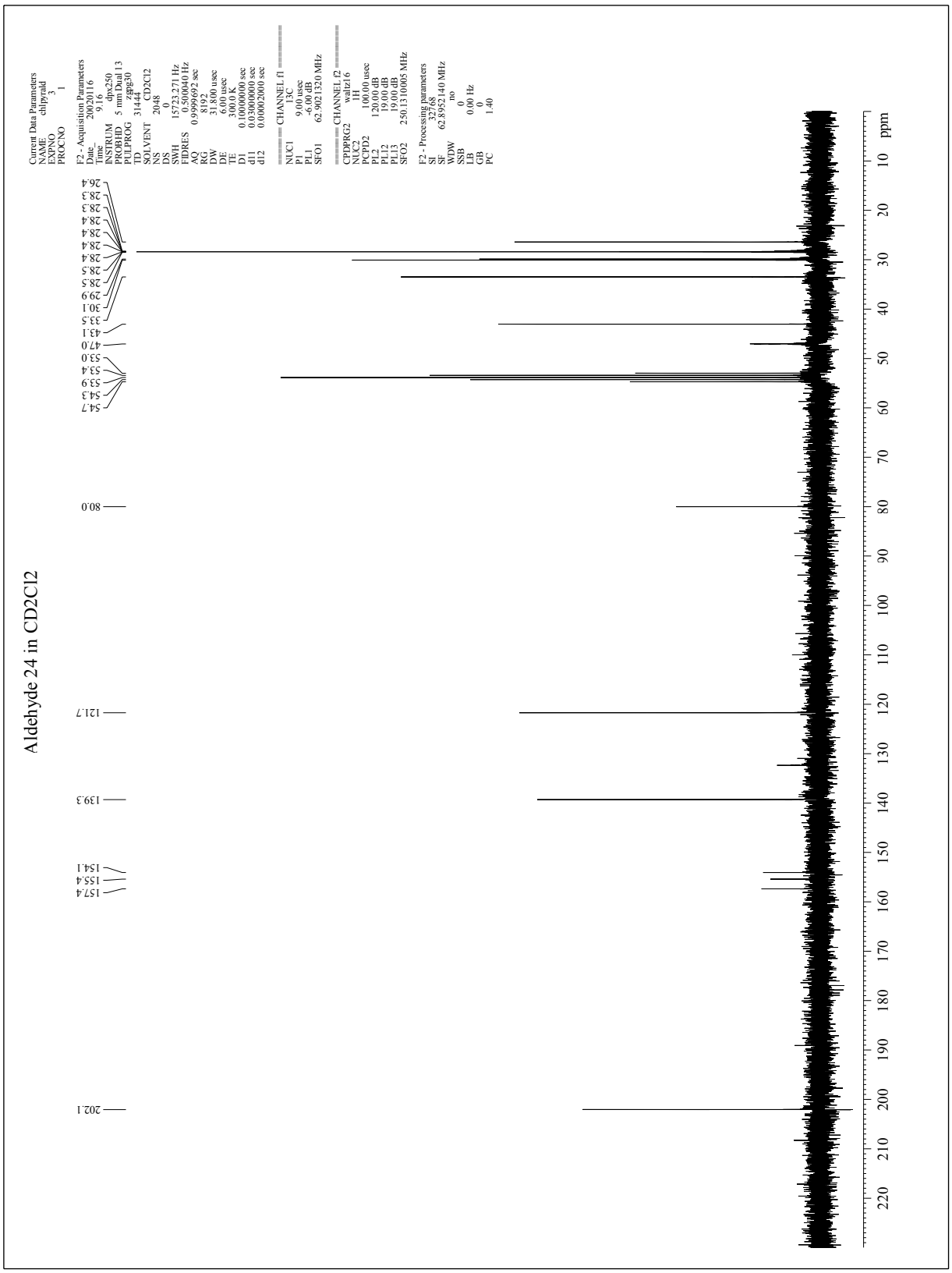

Oxazole 7 in CD2Cl2
Current Data Parameters
NAME 1795.x63
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20001204
Time 10.35
INSTRUM dpx250
PROBHD 5 mm Dual 13
PULPROG zgpg30
TD 31444
SOLVENT CD2Cl2
NS 225
DS 0
SWH 15723.271 Hz
FIDRES 0.500040 Hz
AQ 0.9999692 sec
RG 8192
DW 31.800 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======= CHANNEL f1 =======
NUC1 13C
P1 9.00 usec
PL1 -6.00 dB
SFO1 62.9021320 MHz
======= CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 19.00 dB
PL13 19.00 dB
SFO2 250.1310005 MHz
F2 - Processing parameters
SI 16384
SF 62.8951963 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
170.4
156.3
139.2
137.5
132.7
114.9
54.9
54.4
54.0
53.6
53.1
47.0
33.9
28.2
26.4
23.8
12.1
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

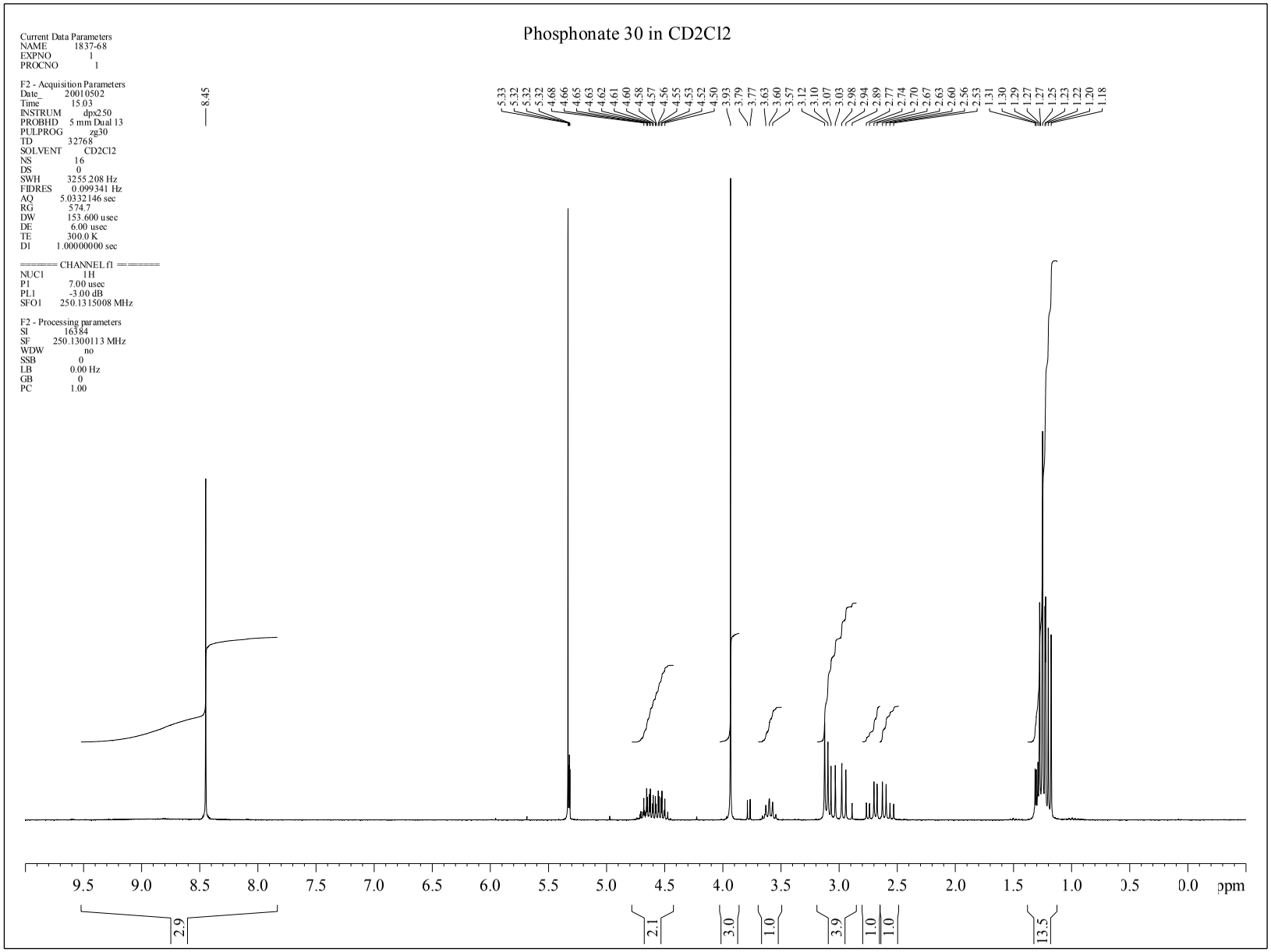

Oxazole 9 in CD2Cl2
Current Data Parameters
NAME 1805-43
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20010111
Time 16.08
INSTRUM spect250
PROBHD 5 mm Dual 13
PULPROG zg30
TD 32768
SOLVENT CD2Cl2
NS 16
DS 2
SWH 325.208 Hz
FIDRES 0.009.941 Hz
AQ 5.032146 sec
RG 256
DW 153.600 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 7.00 usec
PL1 -3.00 dB
SFO1 250.1315008 MHz
F2 - Processing parameters
SI 65536
SF 250.1300111 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
6.1
1.1
4.1
2.0
5.1
2.0
ppm

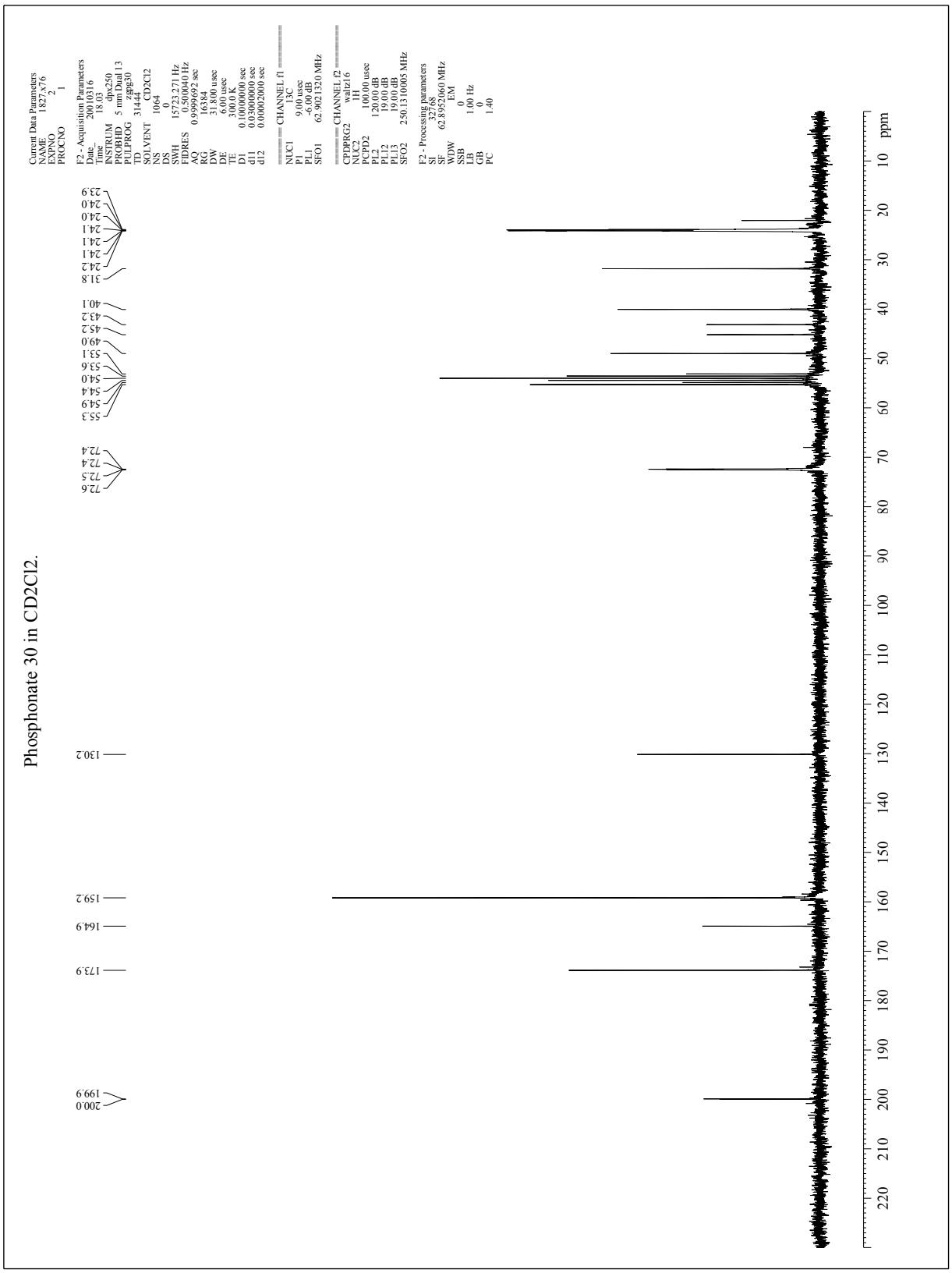

Oxazole 10 in CD2Cl2
Current Data Parameters
NAME 1805-X24
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20001212
Time 13.03
INSTRUM dpx250
PROBHD 5 mm Dual 13
PULPROG zg30
TD 32768
SOLVENT CD2Cl2
NS 16
DS 0
SWH 4761.905 Hz
FIDRES 0.145322 Hz
AQ 3.4406900 sec
RG 128
DW 105.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
====== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 -3.00 dB
SFO1 250.1315008 MHz
F2 - Processing parameters
SI 16384
SF 250.1300114 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.36
7.34
7.33
7.32
7.30
7.28
7.27
7.24
6.97
6.96
6.96
6.95
5.82
5.79
5.75
5.05
5.04
5.04
5.03
4.98
4.98
4.97
4.97
4.94
4.94
4.93
4.61
3.38
3.35
3.32
3.19
2.36
2.11
2.10
2.08
2.04
2.04
2.02
1.97
1.64
1.61
1.59
1.58
1.58
1.55
1.44
1.44
1.42
1.41
1.39
1.38
1.35
1.18
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
5.3
0.9
1.0
2.0
1.9
1.8
4.9
2.0
1.9

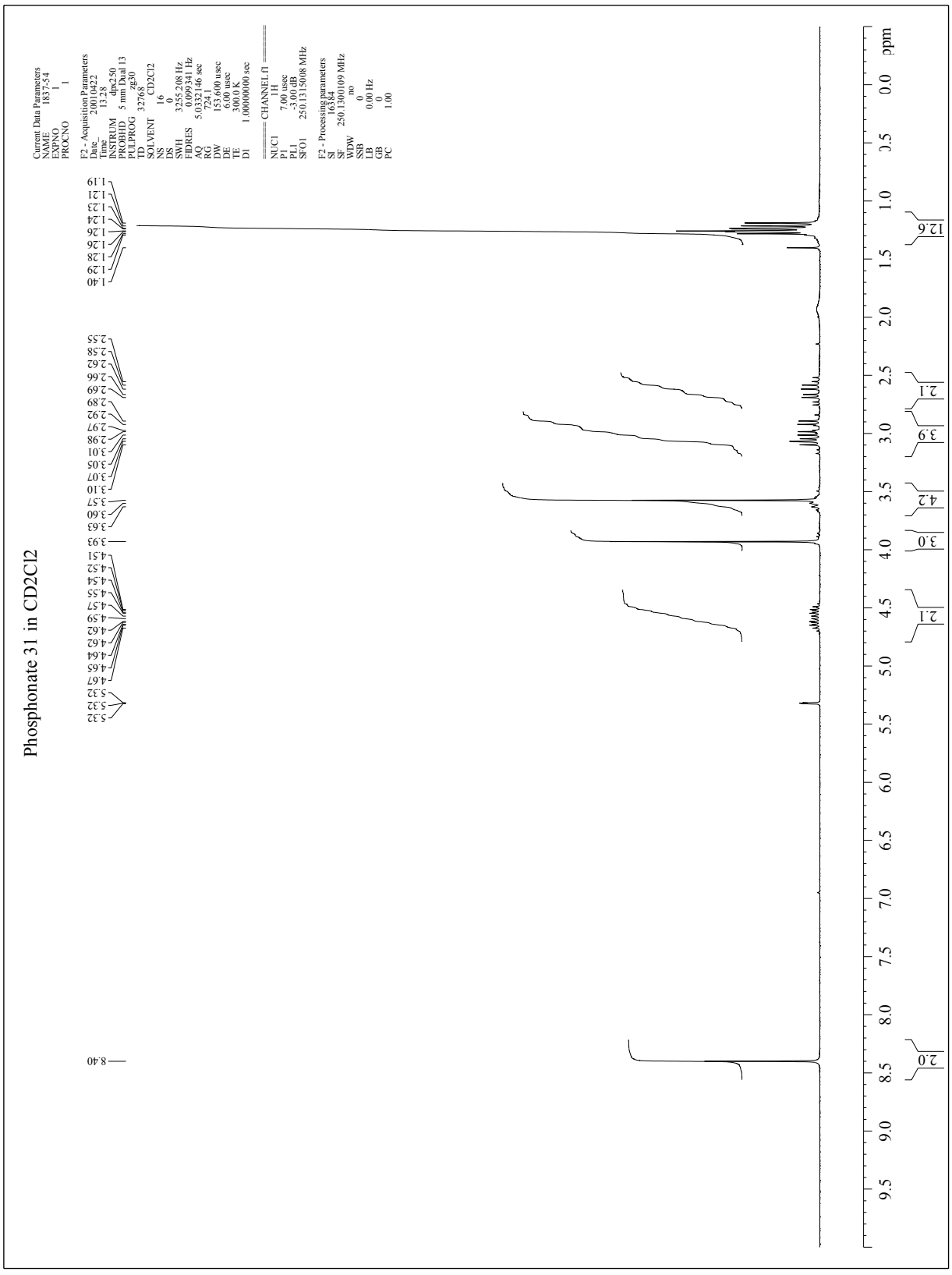

Oxazole 10 in CD2Cl2
Current Data Parameters
NAME 1805-X24
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20001212
Time 13.56
INSTRUM spect
PROBHD 5 mm Dual 13
PULPROG zgpg30
TD 31444
SOLVENT CD2Cl2
NS 1024
DS 0
SWH 15723.271 Hz
FIDRES 0.479060 Hz
AQ 0.999692 sec
RG 2048
DW 31.800 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 -6.00 dB
SFO1 62.9021320 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 19.00 dB
PL12 19.00 dB
PL13 120.00 dB
SFO2 250.1310005 MHz
F2 - Processing parameters
SI 32768
SF 62.8952037 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
162.2
139.2
138.7
136.9
129.0
129.0
129.0
128.7
128.0
128.0
127.8
127.8
127.8
114.9
54.4
54.0
53.6
53.1
52.0
48.3
34.0
27.6
26.6
12.3
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

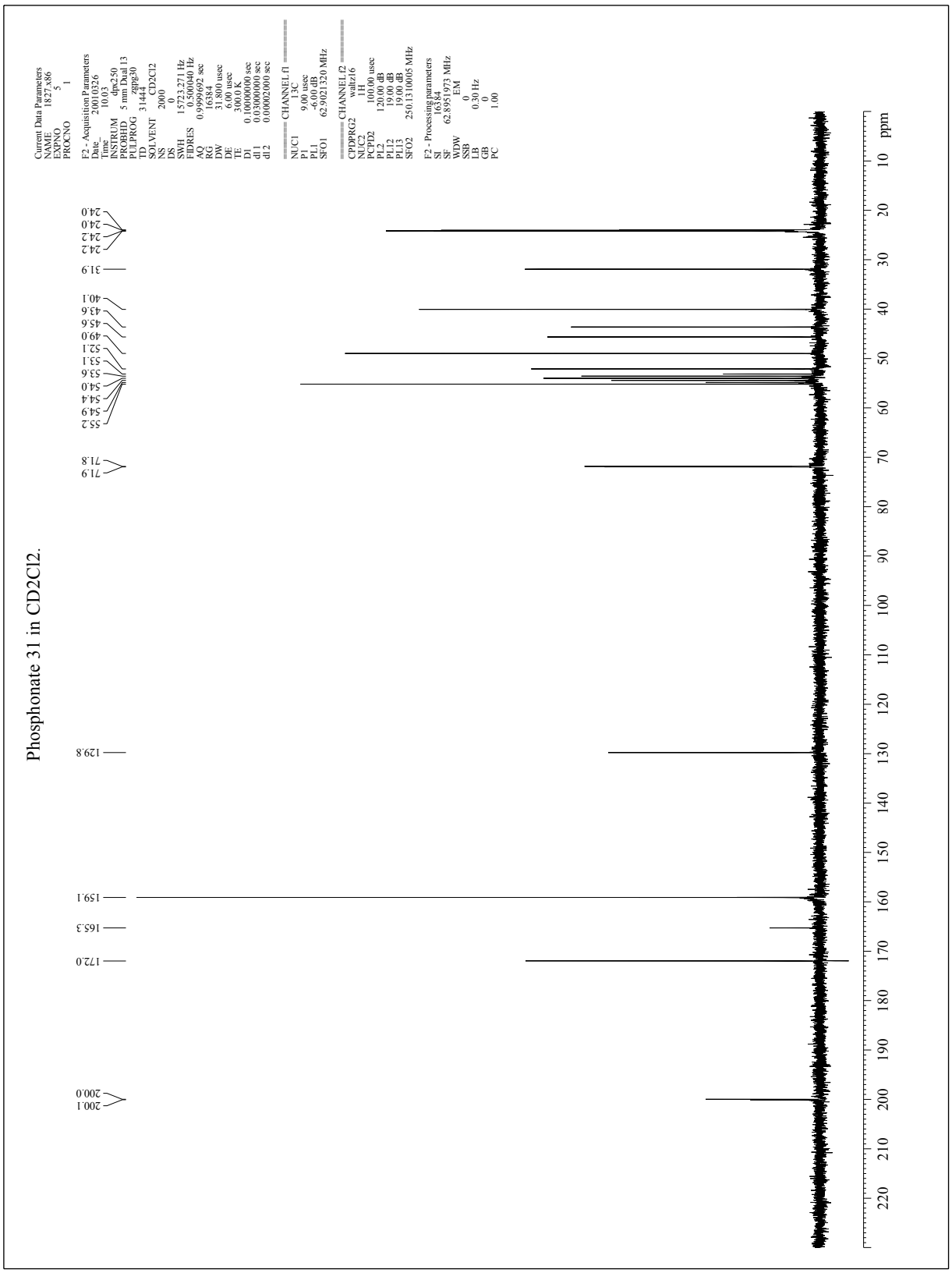

Aldehyde 24 in CD2Cl2.
Current Data Parameters
NAME 192-5x100
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20020301
Time 15.22
INSTRUM dpx250
PROBHD 5 mm Dual 13
PULPROG zg30
TD 32768
SOLVENT CD2Cl2
NS 32
DS 0
SWH 4761.905 Hz
FIDRES 0.145322 Hz
AQ 3.44106900 sec
RG 1448.2
DW 105.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
CHANNEL f1
NUC1 1H
P1 7.00 usec
PL1 -3.00 dB
SFO1 250.1315008 MHz
F2 - Processing parameters
SI 16384
SF 250.1300111 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
9.83
9.82
9.82
7.47
7.44
7.01
6.98
5.33
5.32
5.32
3.07
3.04
3.01
2.91
2.90
2.90
2.88
2.87
2.87
2.85
2.69
2.67
2.66
2.64
2.00
1.82
1.80
1.78
1.38
0.9
1.0
1.0
8.2
12.7
ppm

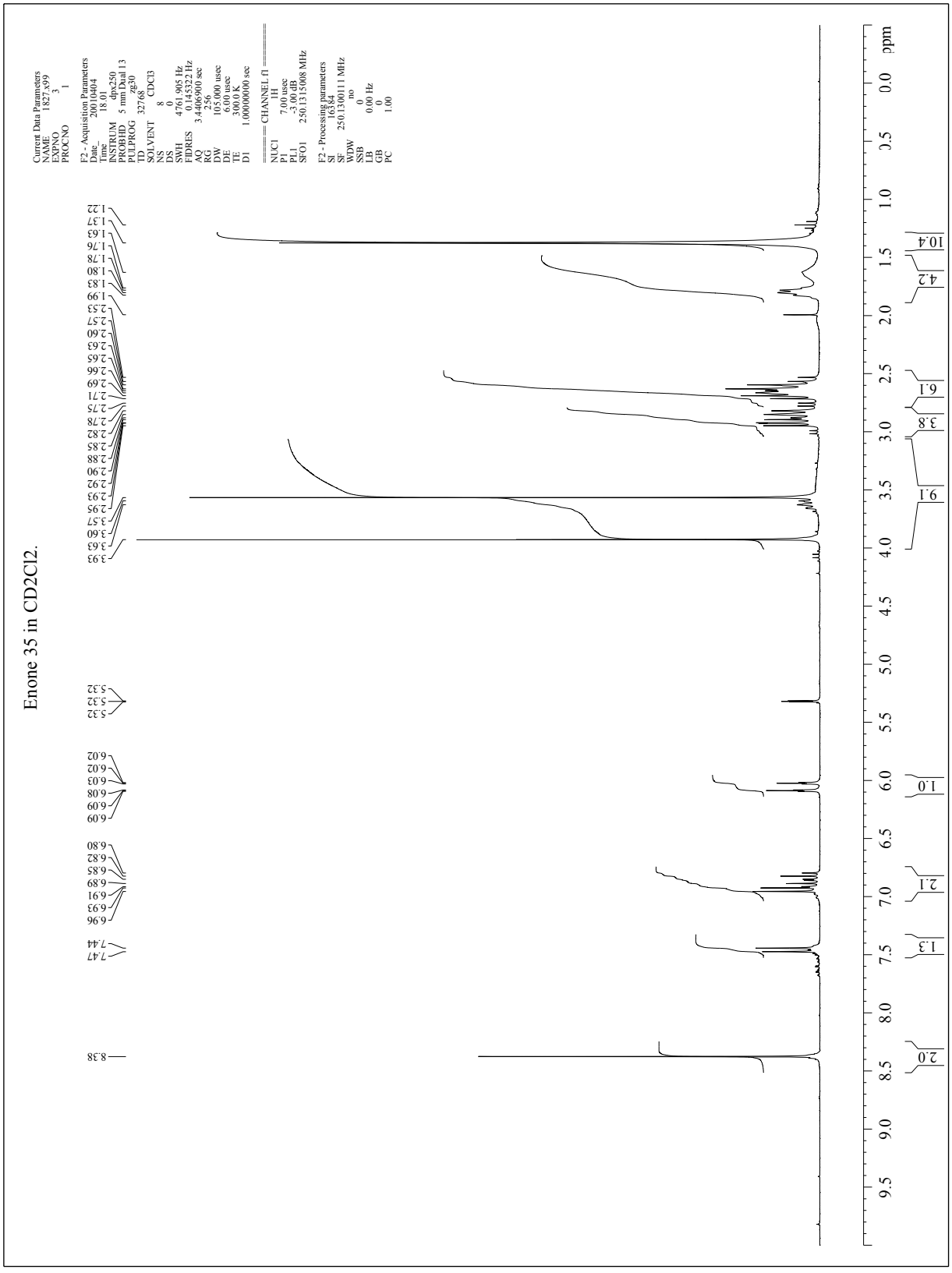

Aldehyde 24 in CD2Cl2
Current Data Parameters
NAME chlyrald
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20020116
Time 9.16
INSTRUM dpx250
PROBHD 5 mm Dual 13
PULPROG zgpg30
TD 31444
SOLVENT CD2Cl2
NS 2048
DS 0
SWH 15723.271 Hz
FIDRES 0.500040 Hz
AQ 0.9999692 sec
RG 8192
DW 31.800 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 -6.00 dB
SFO1 62.902320 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL12 120.00 dB
PL12 19.00 dB
PL13 19.00 dB
SFO2 250.13100005 MHz
F2 - Processing parameters
SI 32768
SF 62.8952140 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
202.1
157.4
155.4
154.1
139.3
121.7
80.0
54.7
54.3
53.9
53.4
53.0
47.0
43.1
33.5
30.1
29.9
28.5
28.5
28.4
28.4
28.4
28.4
28.4
28.3
28.3
26.4
ppm

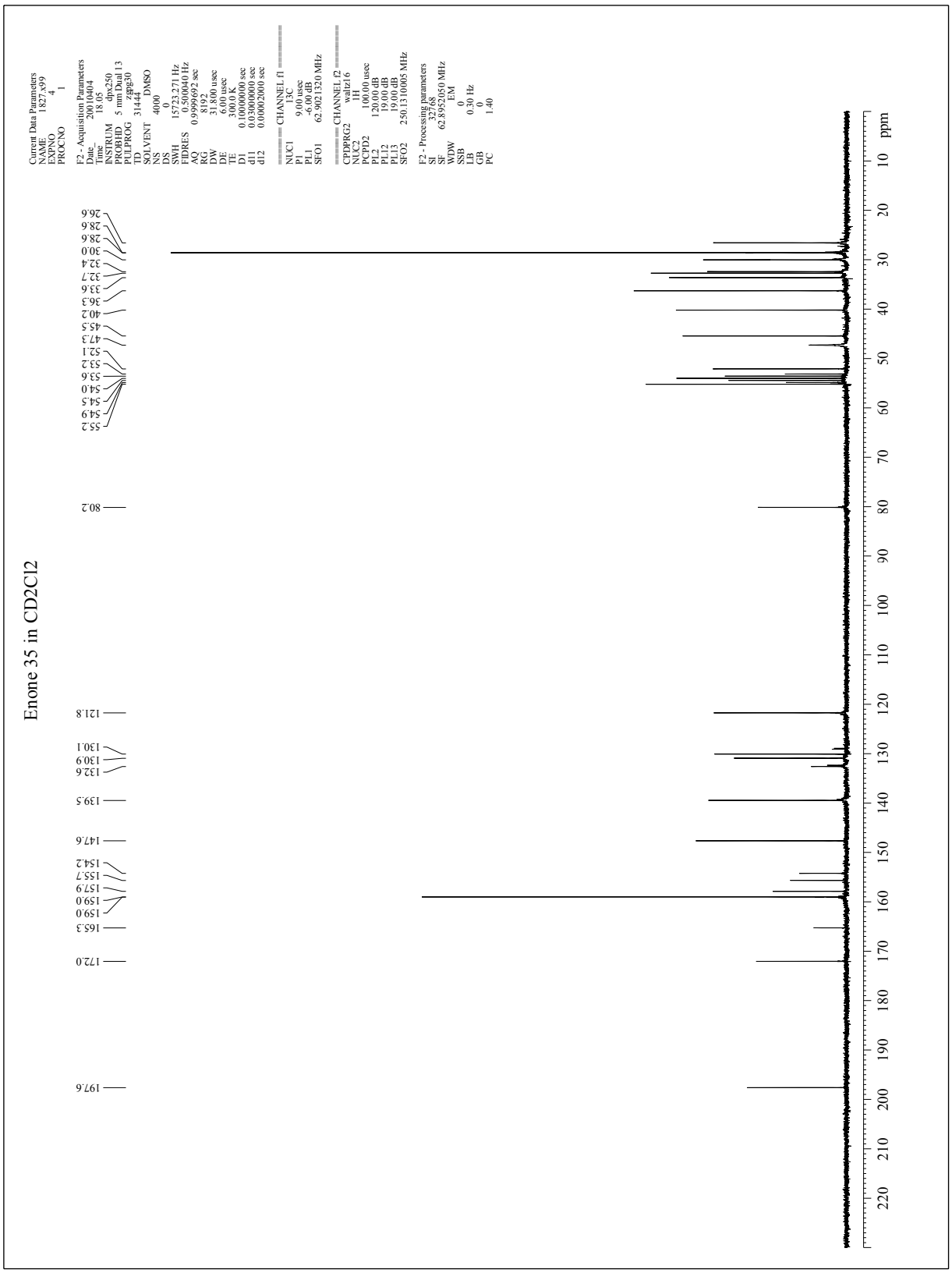

Phosphonate 30 in CD2Cl2
Current Data Parameters
NAME 1837-68
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20010502
Time 15.03
INSTRUM dpx250
PROBHD 5 mm Dual 13
PULPROG zg30
TD 32768
SOLVENT CD2Cl2
NS 16
DS 0
SWH 3255.208 Hz
FIDRES 0.099341 Hz
AQ 5.0332146 sec
RG 574.7
DW 153.600 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
====== CHANNEL f1 ======
NUC1 1H
P1 7.00 usec
PL1 -3.00 dB
SFO1 250.1315008 MHz
F2 - Processing parameters
SI 16384
SF 250.1300113 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8.45
5.33
5.32
5.32
5.32
4.68
4.66
4.65
4.63
4.62
4.61
4.60
4.58
4.57
4.56
4.55
4.53
4.52
4.50
3.93
3.79
3.77
3.63
3.60
3.57
3.12
3.10
3.07
3.03
2.98
2.94
2.89
2.77
2.74
2.70
2.67
2.63
2.60
2.56
2.53
1.31
1.30
1.29
1.27
1.27
1.25
1.23
1.22
1.20
1.18
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.9
2.1
3.0
1.0
3.9
1.0
1.0
13.5

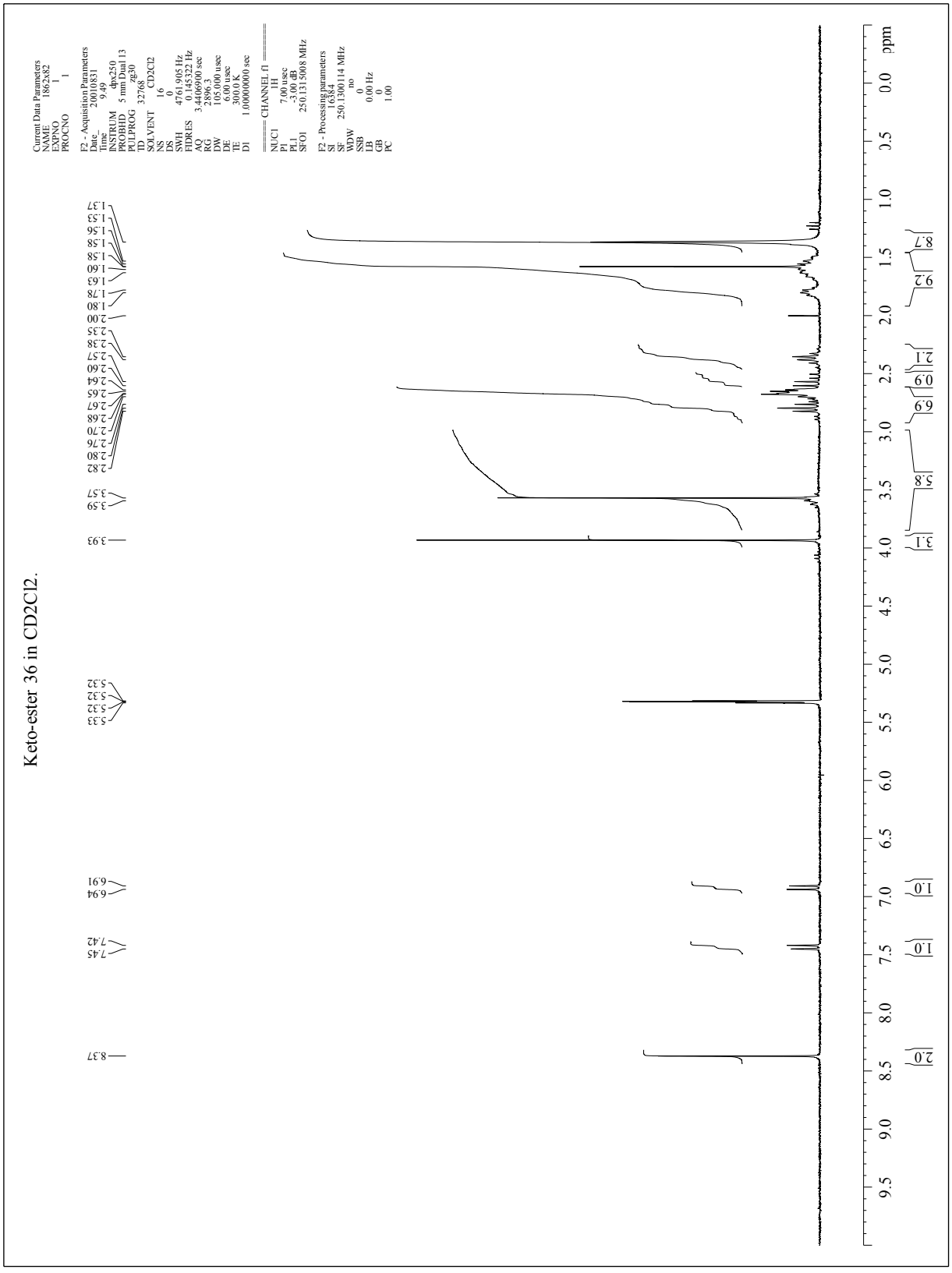

Phosphonate 30 in CD2Cl2.
Current Data Parameters
NAME 1827.x76
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20010316
Time 18.03
INSTRUM dpx250
PROBHD 5 mm Dual 13
PULPROG zgpg30
TD 31444
SOLVENT CD2Cl2
NS 1064
DS 0
SWH 15723.271 Hz
FIDRES 0.500040 Hz
AQ 0.9999692 sec
RG 16384
DW 31.800 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 9.00 usec
PL1 -6.00 dB
SFO1 62.9021320 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 19.00 dB
PL13 19.00 dB
SFO2 250.1310005 MHz
F2 - Processing parameters
SI 32768
SF 62.8952060 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200.0
199.9
173.9
164.9
159.2
130.2
72.6
72.5
72.4
72.4
55.3
54.9
54.4
54.0
53.6
53.1
49.0
45.2
43.2
40.1
31.8
24.2
24.1
24.1
24.1
24.0
24.0
23.9
ppm

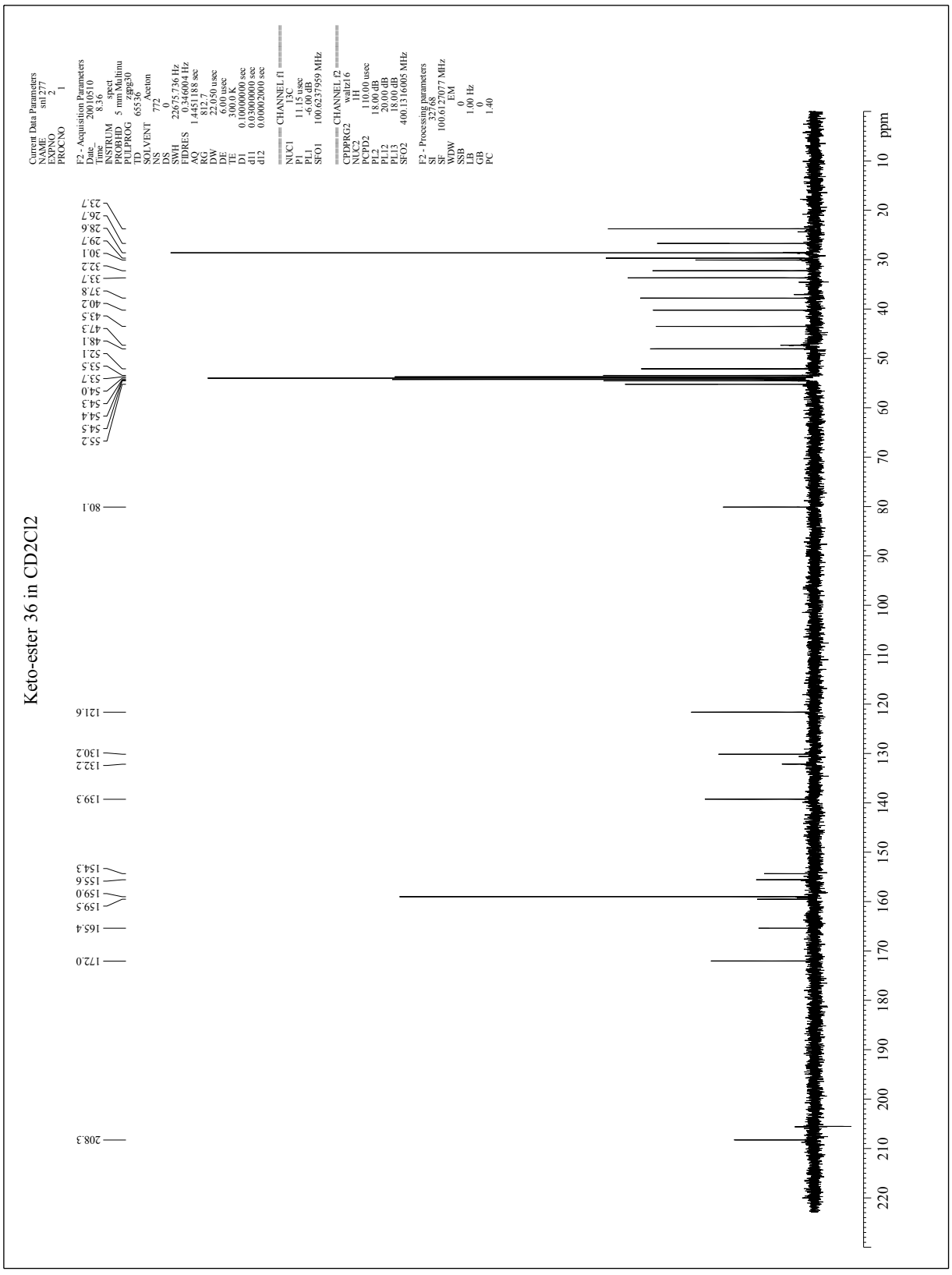

Phosphonate 31 in CD2Cl2
Current Data Parameters
NAME 1837-54
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20011022
Time 13.26
INSTRUM spect 250
PROBHD 5 mm Dual 13
PULPROG zg30
TD 32768
SOLVENT CD2Cl2
NS 16
DS 0
SWH 3255.208 Hz
FIDRES 0.099341 Hz
AQ 5.0332146 sec
RG 724.1
DW 153.600 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
CHANNEL f1
NUC1 1H
P1 7.00 usec
PL1 -3.00 dB
SFO1 250.1315008 MHz
F2 - Processing parameters
SI 16384
SF 250.1300109 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8.40
5.32
5.32
4.67
4.65
4.64
4.62
4.62
4.59
4.57
4.55
4.54
4.52
4.51
3.93
3.63
3.60
3.57
3.10
3.07
3.05
3.01
2.98
2.97
2.92
2.89
2.69
2.66
2.62
2.58
2.55
1.40
1.29
1.28
1.26
1.24
1.23
1.21
1.19
2.0
2.1
3.0
4.2
3.9
2.1
12.6
ppm


Phosphonate 31 in CD2Cl2.
Current Data Parameters
NAME 182-X86
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20010326
Time 10.03
INSTRUM dpx250
PROBHD 5 mm Dual 13
PULPROG zgpg30
TD 31444
SOLVENT CD2Cl2
NS 2000
DS 0
SWH 15723.271 Hz
FIDRES 0.499969 Hz
AQ 0.9999692 sec
RG 16384
DW 31.800 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 9.00 usec
PL1 -6.00 dB
SFO1 62.9021320 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 19.00 dB
PL13 19.00 dB
SFO2 250.1310005 MHz
F2 - Processing parameters
SI 16384
SF 62.8951973 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
200.1
200.0
172.0
165.3
159.1
129.8
71.9
71.8
55.2
54.9
54.4
54.0
53.6
53.1
52.1
49.0
45.6
43.6
40.1
31.9
24.2
24.2
24.0
24.0
ppm


Enone 35 in CD2Cl2.
Current Data Parameters
NAME 1827.x99
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20010404
Time 18.40
INSTRUM dpx250
PROBHD 5 mm Dual 13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 4761.905 Hz
FIDRES 0.145322 Hz
AQ 3.4406900 sec
RG 256
DW 105.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 7.00 usec
PL1 -3.00 dB
SFO1 250.1315008 MHz
F2 - Processing parameters
SI 16384
SF 250.1300111 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8.38
7.47
7.44
6.96
6.93
6.91
6.89
6.85
6.82
6.80
6.09
6.08
6.03
6.02
6.02
6.02
5.32
5.32
5.32
3.93
3.63
3.60
3.57
2.95
2.93
2.92
2.90
2.88
2.85
2.82
2.78
2.75
2.71
2.69
2.66
2.65
2.63
2.60
2.57
2.53
1.99
1.83
1.80
1.78
1.76
1.63
1.37
1.22
2.0
1.3
2.1
1.0
9.1
3.8
6.1
4.2
10.4
ppm


Enone 35 in CD2Cl2
Current Data Parameters
NAME 1827.x99
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20010404
Time 18.05
INSTRUM dpx250
PROBHD 5 mm Dual 13
PULPROG zgpg30
TD 31444
SOLVENT DMSO
NS 4000
DS 0
SWH 15723.271 Hz
FIDRES 0.500040 Hz
AQ 0.999692 sec
RG 8192
DW 31.800 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 -6.00 dB
SFO1 62.902320 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 19.00 dB
PL13 19.00 dB
SFO2 250.13100005 MHz
F2 - Processing parameters
SI 32768
SF 62.8952050 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
197.6
172.0
165.3
159.0
159.0
157.9
155.7
154.2
147.6
139.5
132.6
130.9
130.1
121.8
80.2
55.2
54.9
54.5
54.0
53.6
53.2
52.1
47.3
40.2
36.3
33.6
32.7
32.4
30.0
28.6
28.6
26.6
ppm


Keto-ester 36 in CD2Cl2.
Current Data Parameters
NAME 1862c82
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20010831
Time 9.49
INSTRUM dpx250
PROBHD 5 mm Dual 13
PULPROG zg30
TD 32768
SOLVENT CD2Cl2
NS 16
DS 0
SWH 4761.905 Hz
FIDRES 0.145322 Hz
AQ 3.44106900 sec
RG 2896.3
DW 105.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 7.00 usec
PL1 -3.00 dB
SFO1 250.1315008 MHz
F2 - Processing parameters
SI 16384
SF 250.1300114 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8.37
7.45
7.42
6.94
6.91
5.33
5.32
5.32
5.32
3.93
3.59
3.57
2.82
2.80
2.76
2.70
2.68
2.67
2.65
2.64
2.60
2.57
2.38
2.35
2.00
1.80
1.78
1.63
1.60
1.58
1.58
1.56
1.53
1.37
2.0
1.0
1.0
1.0
3.1
5.8
6.9
2.1
0.9
9.2
8.7
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm


Keto-ester 36 in CD2Cl2
Current Data Parameters
NAME snl277
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20010510
Time 8.36
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT Aceton
NS 772
DS 0
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 812.7
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 11.15 usec
PL1 -6.00 dB
SFO1 100.6237959 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 110.00 usec
PL2 18.00 dB
PL12 20.00 dB
PL13 18.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127077 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
208.3
172.0
165.4
159.5
159.0
155.6
154.3
139.3
132.2
130.2
121.6
80.1
55.2
54.5
54.4
54.3
54.0
53.7
53.5
52.1
48.1
47.3
43.5
40.2
37.8
33.7
32.2
30.1
29.7
28.6
26.7
23.7
ppm