SUPPORTING INFORMATION

Radical Reaction of Sodium Hypophosphite with Terminal Alkynes: Synthesis of 1,1-bis-H-Phosphinates

Sonia Gouault-Bironneau, Sylvine Deprèle, Amber Sutor, and Jean-Luc Montchamp*

Department of Chemistry, Box 298860, Texas Christian University, Fort Worth, Texas 76129

General Chemistry. ^{1}H NMR spectra were recorded on a 300 MHz spectrometer. Chemical shifts for ^{1}H NMR spectra are reported (in parts per million) relative to internal tetramethylsilane (Me_4Si, δ = 0.00 ppm) with $CDCl_3$ as solvent. ^{13}C NMR spectra were recorded at 75 MHz, or at 22.6 MHz. Chemical shifts for ^{13}C NMR spectra are reported (in parts per million) relative to $CDCl_3$ (δ = 77.0 ppm). ^{31}P NMR spectra were recorded at 121 MHz and/or at 36 MHz, and chemical shifts reported (in parts per million) relative to external 85% phosphoric acid (δ = 0.0 ppm). Mass spectrometry was provided by the the Mass Spectrometry Facility of the University of South Carolina. Elemental analyses were performed by Atlantic Microlab Inc. (Norcross, GA). Ethyl acetate/hexanes mixtures were used as the eluent for chromatographic purifications. TLC plates were visualized by immersion in anisaldehyde stain (by volume: 93% ethanol, 3.5% sulfuric acid, 1% acetic acid, and 2.5% anisaldehyde) followed by heating. Organic solutions of products were dried over anhydrous $MgSO_4$.

Reagents and Solvents. Sodium hypophosphite hydrate and other reagents were obtained from Aldrich and used as received. Reagent or HPLC grade solvents were used throughout this study and were not dried prior to use.

Radical reaction of sodium hypophosphite with alkynes: Representative procedure (Tables 1-2). To a solution of $NaH_2PO_2.H_2O$ (18.0 mmol) in a mixture of methanol (12.5 mL) and dioxane (2.5 mL) were added the alkyne (3 mmol) and triethylborane (1.0 M in hexane, 3 mL, 3 mmol). The solution was stirred for 4 hours at room-temperature in a flask open to air, and then filtered. The precipitate was washed several times with cold methanol and dried in vacuo over P_2O_5 to afford the the 1,1-bis-H-phosphinate sodium salt in good purity (typically >95%).

Disodium hexyl-1,1-bis-H-phosphinate (Table 1, Entry 9). Mp: > 250^{o}C (dec). ^{1}H NMR (D_2O) δ 6.85 (d, $^{1}J_{PH}$ = 524 Hz, 2 H), 1.62-1.28 (m, 5 H), 1.20-1.10 (bs, 4 H), 0.70 (t, $^{3}J_{HH}$ = 6.7 Hz, 3 H); ^{13}C NMR (H_2O, 90 MHz) δ 13.7, 21.6 (t, $^{3}J_{PC}$ = 2.7 Hz), 22.0, 29.0 (t, $^{2}J_{PC}$ = 6.6 Hz), 31.5, 44.4 (t, $^{1}J_{PC}$ = 78.5 Hz; ^{31}P NMR (D_2O) δ 26.4 (dm, $^{1}J_{PH}$ = 523 Hz). HRMS (FAB) calcd. for $C_6H_{14}Na_2O_4P_2$, (M-2Na^{+}+H^{+}) 213.0446, found 213.0446.

Disodium (3-hydroxy-propyl)-1,1-bis-H-phosphinate (Table 2, Entry 1). ^{1}H NMR (D_2O) δ 6.87 (d, $^{1}J_{PH}$ = 529.7 Hz, 2 H), 3.57 (t, $^{3}J_{HH}$ = 7.0 Hz, 2 H), 1.87-1.70 (m, 2 H), 1.63-1.46 (m, 1 H); ^{13}C NMR (D_2O) δ 23.9, 41.4 (t, $^{1}J_{PC}$ = 77.7 Hz), 61.2 (t, $^{2}J_{PC}$ = 7.8 Hz); ^{31}P NMR (D_2O) δ 25.6 (dm, $^{1}J_{PH}$ = 530 Hz). HRMS (FAB) calcd. for $C_3H_8Na_2O_5P_2$, (M-2Na^{+}+H^{+}) 186.9925, found 186.9924.

Disodium (5-hydroxy-pentyl)-1,1-bis-H-phosphinate (Table 2, Entry 2). ^{1}H NMR (D_2O) δ 6.84 (d, $^{1}J_{PH}$ = 523.6 Hz, 2 H), 3.42 (t, $^{3}J_{HH}$ = 6.0 Hz, 2 H), 1.7- 1.4 (m, 3 H), 1.48 (bs, 4 H); ^{13}C NMR (H_2O, 90 MHz) δ 20.8, 24.8, 31.0, 43.8 (t, $^{1}J_{PC}$ = 74.8 Hz); ^{31}P NMR (D_2O) δ 26.1 (dm, $^{1}J_{PH}$ = 525.5 Hz). Anal. calcd. for $C_5H_{12}Na_2O_5P_2$: C, 23.09; H, 4.65; O, 30.76. Found: C, 22.89; H, 4.79; O, 31.02.

Disodium (2-cyclohexyl-2-hydroxy-ethyl)-1,1-bis-H-phosphinate (Table2, Entry 3). ^{1}H NMR (D_2O) δ 6.86 (d, $^{1}J_{PH}$ = 531.8 Hz, 2 H), 1.90-1.68 (m, 3 H), 1.33 (bs, 10 H); ^{13}C NMR (D_2O) δ 22.0, 25.3, 31.7, 37.0, 39.4 (t, $^{1}J_{PC}$ = 77.2Hz), 71.5 (t, $^{2}J_{PC}$ = 5.8 Hz); ^{31}P NMR (D_2O) δ 24.9 (dm, $^{1}J_{PH}$ = 531.6 Hz). HRMS (FAB) calcd. for $C_8H_{16}Na_2O_5P_2$, (M-2Na$^+$+H$^+$) 255.0558, found 255.0551. Anal. calcd. for $C_8H_{16}Na_2O_5P_2 + H_2O$: C, 30.20; H, 5.70. Found: C, 30.27; H, 5.76.

Disodium (3-hydroxy-3-methyl-butyl)-1,1-bis-H-phosphinate (Table2, Entry 4). ^{1}H NMR (D_2O) δ 7.00 (d, $^{1}J_{PH}$ = 532.0 Hz, 2 H), 1.95-1.85 (m, 3 H), 1.2 (s, 6 H); ^{13}C NMR (D_2O) δ 28.4, 33.5, 40.6 (t, $^{1}J_{PC}$ = 76.9 Hz), 70.7 (t, $^{2}J_{PC}$ = 6.0 Hz); ^{31}P NMR (D_2O) δ 28.6 (dm, $^{1}J_{PH}$ = 532.0 Hz). HRMS (FAB) calcd. for $C_5H_{12}Na_2O_5P_2$, (M-2Na$^+$+H$^+$) 259.0241, found 259.0227.

Disodium (2-trimethylsilyl-ethyl)-1,1-bis-H-phosphinate (Table 2, Entry 5). ^{1}H NMR (D_2O) δ 6.91 (d, $^{1}J_{PH}$ = 524.8 Hz, 2 H), 1.67 (tt, $^{2}J_{PH}$ = 18.5 Hz, $^{3}J_{HH}$ = 6.2 Hz, 1 H), 0.77 (dt, $^{3}J_{HH}$ = 6.2 Hz, $^{3}J_{PH}$ = 17.5 Hz, 2 H), 0.00 (s, 9 H); ^{13}C NMR (H_2O, 90 MHz) δ -2.3, 5.7, 38.8 (t, $^{1}J_{PC}$ = 77.5 Hz); ^{31}P NMR (D_2O) δ 27.3 (dm, $^{1}J_{PH}$ = 524.2 Hz). HRMS (FAB) calcd. for $C_5H_{14}Na_2O_4P_2Si$, (M-Na$^+$+2H$^+$) 253.0191, found 253.0197. Anal. calcd. for $C_5H_{14}Na_2O_4P_2Si + H_2O$: C, 20.55; H, 5.52. Found: C, 20.75; H, 5.12.

Disodium decyl-1,1-bis-H-phosphinate (Table 2, Entry 6). ^{1}H NMR (D_2O) δ 7.00 (d, $^{1}J_{PH}$ = 524.0 Hz, 2 H), 1.70-1.60 (m, 2 H), 1.50-1.40 (m, 1 H), 1.30-1.20 (bs, 14 H); ^{13}C NMR (H_2O, 90 MHz) δ 12.9, 20.9, 21.5, 28.0 (bs), 30.7, 43.7 (t, $^{1}J_{PC}$ = 76.8 Hz); ^{31}P NMR (D_2O) δ 22.5 (dm, $^{1}J_{PH}$ = 524.0 Hz). HRMS (FAB) calcd. for $C_5H_{22}Na_2O_4P_2$ (M+H$^+$) 254.9540, found 254.9533.

Disodium (3,3-dimethyl-butyl)-1,1-bis-H-phosphinate (Table 2, Entry 7). ^{1}H NMR (D_2O) δ 6.83 (d, $^{1}J_{PH}$ = 527.1 Hz, 2 H), 1.66-1.34 (m, 3 H), 0.71 (bs, 9 H); ^{13}C NMR (H_2O, 90 MHz) δ 28.4, 29.7, 33.7, 40.1 (t, $^{1}J_{PC}$ = 78.5 Hz); ^{31}P NMR (D_2O) δ 27.1 (dm, $^{1}J_{PH}$ = 512.0 Hz**).** HRMS (FAB) calcd. for $C_6H_{14}Na_2O_4P_2$ (M-Na$^+$+2 H$^+$) 237.0422, found 237.0412.

Disodium (2-ethyl-ethyl-ester)-1,1-bis-H-phosphinate (Table 2, Entry 8). ^{1}H NMR (D_2O) δ 6.86 (d, $^{1}J_{PH}$ = 531.8 Hz, 2 H), 3.99 (q, $^{3}J_{HH}$ = 6.7 Hz, 2 H), 2.60-2.40 (m, 2 H), 2.19-1.86 (m, 1 H), 1.07 (t, $^{3}J_{HH}$ = 6.7 Hz, 3 H); ^{13}C NMR (H_2O, 90 MHz) δ 12.8, 26.6, 40.1 (t, $^{1}J_{PC}$ = 77.7 Hz), 61.5, 173.7; ^{31}P NMR (D_2O) δ 23.4 (dm, $^{1}J_{PH}$ = 532.8 Hz). HRMS (FAB) calcd. for $C_5H_{10}Na_2O_6P_2$ (M+Na$^+$+H$^+$) 296.9646, found 296.9646.

Disodium (3-methoxy-propyl)-1,1-bis-H-phosphinate (Table 2, Entry 9). ^{1}H NMR (D_2O) δ 6.84 (d, $^{1}J_{PH}$ = 529.1 Hz, 2 H), 3.44 (t, $^{3}J_{HH}$ = 6.5 Hz, 2 H), 3.18 (s, 3 H), 1.90-1.65 (m, 2 H), 1.60-1.35 (m, 1 H); ^{13}C NMR (H_2O, 90 MHz) δ 20.8, 40.3 (t, $^{1}J_{PC}$ = 76.2 Hz), 57.5, 71.0; ^{31}P NMR (D_2O) δ 25.3 (dm, $^{1}J_{PH}$ = 529.6 Hz). HRMS (FAB) calcd. for $C_4H_{10}Na_2O_5P_2$, (M+H$^+$) 246.9877, found 246.9867.

Disodium 5,5-bis-H-pentanoic acid phosphinate (Table 2, Entry 10). ^{1}H NMR (D_2O) δ 6.85 (d, $^{1}J_{PH}$ = 527.1 Hz, 2 H), 2.22 (t, $^{3}J_{HH}$ = 6.4 Hz, 2 H), 1.70-1.35 (m, 5 H); ^{13}C NMR (H_2O, 90 MHz) δ 20.2, 23.8 (t, $^{2}J_{PC}$ = 7.21 Hz), 33.7, 42.9 (t, $^{1}J_{PC}$ = 78.5 Hz), 178.4; ^{31}P NMR (D_2O) δ 25.8 (dm, $^{1}J_{PH}$ = 527.5 Hz). HRMS (FAB) calcd. for $C_5H_{10}Na_2O_6P_2$ ($M+H^+$) 274.9826, found 274.9836.

Disodium (4-phenyl-butyl)-1,1-bis-H-phosphinate (Table 2, Entry 11). ^{1}H NMR (D_2O) δ 7.16 (bs, 5 H), 6.84 (d, $^{1}J_{PH}$ = 524.8 Hz, 2 H), 2.50 (t, $^{3}J_{HH}$ = 7.0 Hz, 2 H), 1.70-1.4 (m, 5 H); ^{13}C NMR (D_2O) δ 21.3, 31.1, 35.4, 44.2 ((t, $^{1}J_{PC}$ = 77.7 Hz), 126.0, 128.7, 128.8, 142.9; ^{31}P NMR (D_2O) δ 26.0, (dm, $^{1}J_{PH}$ = 527.5 Hz). HRMS (FAB) calcd. for $C_{10}H_{14}Na_2O_4P_2$, ($M+H^+$) 307.0241, found 307.0227.

Disodium (3-aminopropyl)-1,1-bis-H-phosphinate hydrochloride (Table 2, Entry 12). ^{1}H NMR (D_2O) δ 6.87 (d, $^{1}J_{PH}$ = 533.2 Hz, 2 H), 3.02 (t, $^{3}J_{HH}$ = 7.03 Hz, 2 H), 2.10-1.80 (m, 2 H), 1.66-1.43 (m, 1 H); ^{31}P NMR (D_2O) δ 26.8 (dm, $^{1}J_{PH}$ = 534.0 Hz). HRMS (FAB) calcd. for $C_3H_9NNa_2O_4P_2$, ($M-2Na^+ + H^+$) 186.0085, found 186.0086.

Disodium [*N*,*N*-(dicarbobenzyloxy)-aminobutyl]-1,1-bis-H-phosphinate (Table 2, Entry 13). ^{1}H NMR (D_2O) δ 6.8 (d, $^{1}J_{PH}$ = 526.8 Hz, 2 H), 2.27 (bs, 10 H), 5.11 (s, 4 H), 3.58 (t, $^{3}J_{HH}$ = 6.7 Hz, 2 H), 1.66-1.43 (m, 5 H); ^{31}P NMR (D_2O) δ 25.5 (dm, $^{1}J_{PH}$ = 526.0 Hz). HRMS (FAB) calcd. for $C_{20}H_{23}NNa_2O_8P_2$, ($M-2Na^+ + H^+$) 468.0977, found 468.0964.

Disodium (3-oxiranylmethoxy-propyl)-1,1-bis-H-phosphinate (Table 2, Entry 14). ^{1}H NMR (D_2O) δ 6.88 (d, $^{1}J_{PH}$ = 529.4 Hz, 2 H), 3.77 (dd, J = 2.0 Hz, J = 11.4 Hz, 1 H), 3.57 (t, $^{3}J_{HH}$ = 7.3 Hz, 2 H), 3.28-3.14 (m, 2 H), 2.78 (t, J = 4.0 Hz, 1 H), 2.61 (dd, J = 2.9 Hz, J = 4.4 Hz, 1 H), 1.92-1.72 (m, 2 H), 1.64-1.46 (m, 1 H); ^{13}C NMR (D_2O) δ 21.3, 40.8 (t, $^{1}J_{PC}$ = 77.7 Hz), 45.2, 51.7, 70.3, 71.1; ^{31}P NMR (D_2O) δ 23.3 (dm, $^{1}J_{PH}$ = 529.2 Hz). HRMS (FAB) calcd. for $C_6H_{12}Na_2O_6P_2$, ($M-2Na^+ + H^+$) 243.0187, found 243.0187.

Disodium hexyl-1,1-bis-phosphonate (Eq. 3). Ozone was bubbled into a solution of disodium hexyl-1,1-bis-H-phosphinate (1.00 g, 3.88 mmol) in water (30 mL) at 0 ºC. The temperature of the solution was maintained between 0 ºC and 10 ºC during the reaction. After 6 h, the ice bath was removed and nitrogen was bubbled into the reaction mixture for 3 h to get rid of the excess of ozone. After concentration in vacuo, the residue was washed with cold methanol and dried over P_2O_5 to afford the disodium hexyl-1,1-bis-phosphonate (1.04 g, 92 %) as a white solid. ^{1}H NMR (D_2O) δ 1.87-1.51 (m, 3 H), 1.41-1.31 (m, 2 H), 1.20-1.05 (m, 4 H), 0.68 (t, $^{3}J_{HH}$ = 7.03 Hz, 3 H); ^{13}C NMR (H_2O, 90 MHz) δ 12.3, 20.6, 24.3, 27.7 (t, $^{2}J_{PC}$ = 6.6 Hz), 29.9, 36.6 (t, $^{1}J_{PC}$ = 117.7 Hz); ^{31}P NMR (D_2O) δ 22.1 (s). HRMS (FAB) calcd. for $C_6H_{14}Na_2O_6P_2$, ($M-2Na^+ + H^+$) 245.0344, found 245.0354.

Compound 1 (Scheme 1). To a solution of epiandrosterone (1.69g, 5.82 mmol) in CH_2Cl_2 (20 mL) at 0 ºC, was added pyridine (1.41 mL, 17.46 mmol). The solution was stirred at this temperature 5 min and propargyl chloroformate (0.58 mL, 5.99 mmol) was added dropwise. The resulting mixture was stirred overnight at room temperature and quenched by adding aqueous HCl (1 N). The solution was extracted with CH_2Cl_2 and the combined organic extracts were

washed successively with saturated aq $NaHCO_3$ (3 X) and brine (1 X). Drying and concentration in vacuo afforded compound **1** (1.94 g, 90 %) which did not require further purification. ^{1}H NMR ($CDCl_3$) δ 4.71 (d, $^4J_{HH}$ = 2.4 Hz, 2 H), 4.59 (sp, J_{HH} = 5.3 Hz, 1 H), 2.52 (t, $^4J_{HH}$ = 2.3 Hz, 1 H), 2.44 (dd, $^2J_{HH}$ = 19.0 Hz, $^3J_{HH}$ = 8.5 Hz, 1 H), 2.17-1.12 (m, 21 H), 0.86 (s, 3 H), 0.85 (s, 3 H); ^{13}C NMR ($CDCl_3$) . δ 12.4, 14.0, 20.7, 22.0, 27.5, 28.4, 31.0, 31.7, 34.0, 35.2, 35.8, 36.1, 36.8, 44.8, 48.0, 51.5, 54.4, 55.2, 75.7, 76.8, 78.4, 154.2, 221.5. HRMS (EI) calcd. for $C_{23}H_{32}O_4$, 372.2301, found 372.2294.

Compound 2 (Scheme 2). To a solution of $NaH_2PO_2.H_2O$ (1.02 g, 9.67 mmol) in a mixture of methanol (7 mL), acetone (4.5 mL) and ethyl acetate (4.5 mL) were added alkyne **1** (600 mg, 1.61 mmol) and triethylborane (1.0 M in hexane, 1.61 mL, 1.61 mmol). The solution was stirred for 4 hours at room-temperature in a flask open to air and then filtered. The precipitate was washed several times with cold methanol and dried in vacuo over P_2O_5 to afford the the 1,1-bis-H-phosphinate sodium salt **2** (165 mg). The filtrate was concentrated in vacuo and redissolved with the same mixture of methanol, acetone and ethyl acetate. Sodium hypophosphite hydrate (1.02 g, 9.67mmol) and triethylborane (1.0 M in hexane, 1.61 mL, 1.61 mmol) were added and the resulting mixture was stirred again 4 hours at room-temperature. As previously, the resulting precipitate was washed several times with cold methanol, and dried over P_2O_5 to afford sodium salt **2** (254 mg) as a white solid. The overall yield after both runs was 48 % (419 mg). ^{1}H NMR (D_2O) δ 6.88 (d, $^1J_{PH}$ = 528.9 Hz, 2 H), 4.41 (bs, 2 H), 4.14 (bs, 2 H), 2.33 (dd, $^2J_{HH}$ = 19.3 Hz, $^3J_{HH}$ = 9.6 Hz, 1 H), 2.1-0.50 (m, 23 H), 0.72 (s, 3 H.69 (s, 3 H); ^{13}C NMR (D_2O) δ 11.9, 13.7, 20.5, 21.0, 21.7, 27.2, 28.2, 30.8, 31.4, 33.7, 34.9, 35.4, 36.0, 36.5, 40.7 (t, $^1J_{PC}$ = 77.6 Hz), 44.4, 48.4, 51.1, 54.1, 67.3, 78.4; δ ^{31}P NMR (D_2O) δ 24.9 (dm, $^1J_{PH}$ = 529.5 Hz). HRMS (FAB) calcd. for $C_{23}H_{36}Na_2O_8P_2$, (M-2Na^++H^+) 503.1963, found 503.1975.

Compound 3 (Scheme 2). To a solution of **2** (200 mg, 0.37 mmol) in water (15 mL), at 0 ºC, was bubbled ozone. The temperature of the solution was maintained between 0 ºC and 10 ºC during the reaction. After 1.5 h, the ice bath was remove and nitrogen was bubbling into the reaction mixture during 3 h to get rid of the excess of ozone. After concentration under vacuum, the residue was washed with cold methanol and dried over P_2O_5 to afford the disodium 1,1-bis-phosphonate **3** (167 mg, 79 %) as a white solid. ^{1}H NMR (D_2O) δ 4.40 (bs, 2 H), 4.16 (t, $^3J_{HH}$ = 6.4 Hz, 2 H), 2.31 (dd, $^2J_{HH}$ = 19.6 Hz, $^3J_{HH}$ = 10.8 Hz, 1 H), 2.03-0.50 (m, 23 H), 0.70 (s, 3 H), 0.67 (s, 3 H); ^{31}P NMR (D_2O) δ 24.9 (dm, $^1J_{PH}$ = 529.5 Hz). HRMS (FAB) calcd. for $C_{23}H_{36}Na_2O_{10}P_2$, (M-2$Na^+$+$H^+$) 535.1862, found 535.1861.

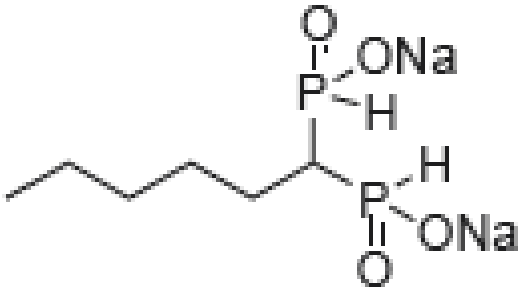
Diisopropyl hexyl-1,1-bis-H-phosphinate (Eq. 4). To a suspension of hexyl-1,1-bis-H-phosphinate (500 mg, 1.94 mmol) in toluene (25 ml) under nitrogen was added trimethylacetyl chloride (0.86 mL, 7.76 mmol) and isopropyl alcohol (1.19 mL, 15.52 mmol) at room temperature. The resulting solution was stirred for 24 h then concentrated in vacuo. The residue was partitioned between EtOAc and H_2O, the organic phase was separated and the aqueous layer was extracted with EtOAc. The combined organic extracts were washed with brine, dried over $MgSO_4$ and concentrated. Purification by chromatography over a silica gel (100 % EtOAc, v/v, EtOAc/MeOH 1:1) afforded the ester (359 mg, 62 %) as a colorless oil: ^{1}H NMR (D_2O) δ 7.3 (dm, 2 H), 4.70 (bs, 2 H), 2.30-1.40 (m, 9 H), 1.35 (bs, 12 H), 0.90 (bs, 3 H); ^{13}C NMR (D_2O,) δ 14.1, 22.3, 23,4, 24.3, 28.6 (t, $^3J_{PC}$ = 6.9 Hz), 31.7, 39.7 (t, $^1J_{PC}$ = 84.3 Hz), 50.4; ^{31}P NMR (D_2O) δ 29.76 (dm, $^1J_{PH}$ = 568.7 Hz), 29.78 (dm, $^1J_{PH}$ = 568.7 Hz), 30.29 (dm, $^1J_{PH}$ = 565.9 Hz),

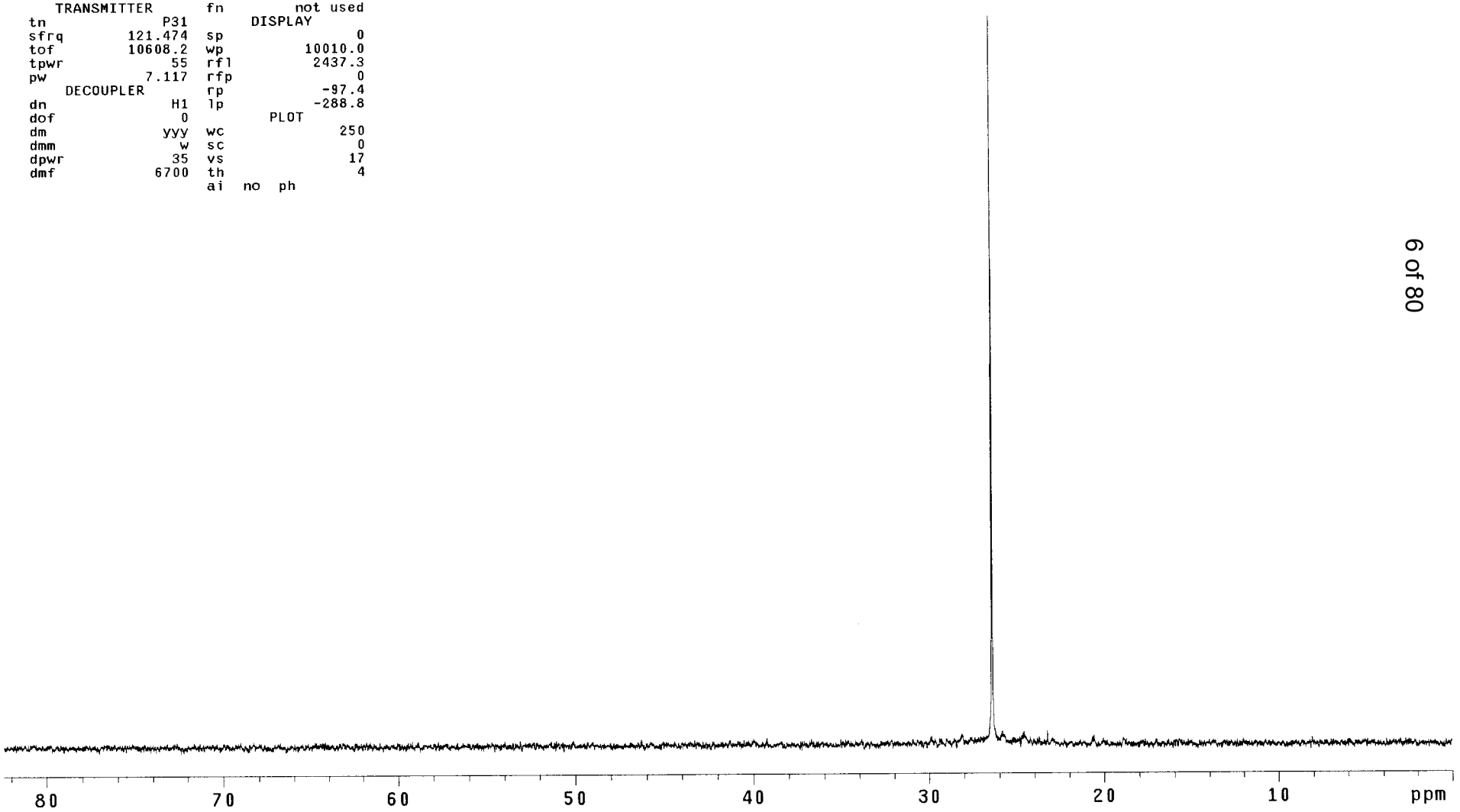
31.38 (dm, $^{1}J_{PH}$ = 569.1 Hz). HRMS (FAB) HRMS (FAB) calcd. for $C_{12}H_{28}O_4P_2$, ($M+H^+$) 299.1541, found 299.1536.

sb-I-86pp in D2O

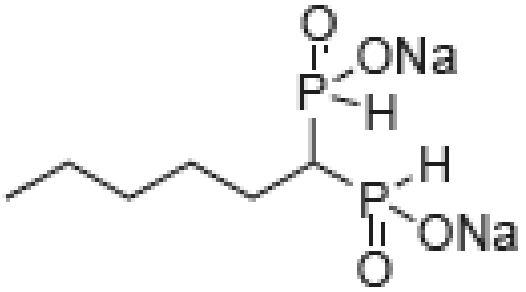
exp1 s2pul

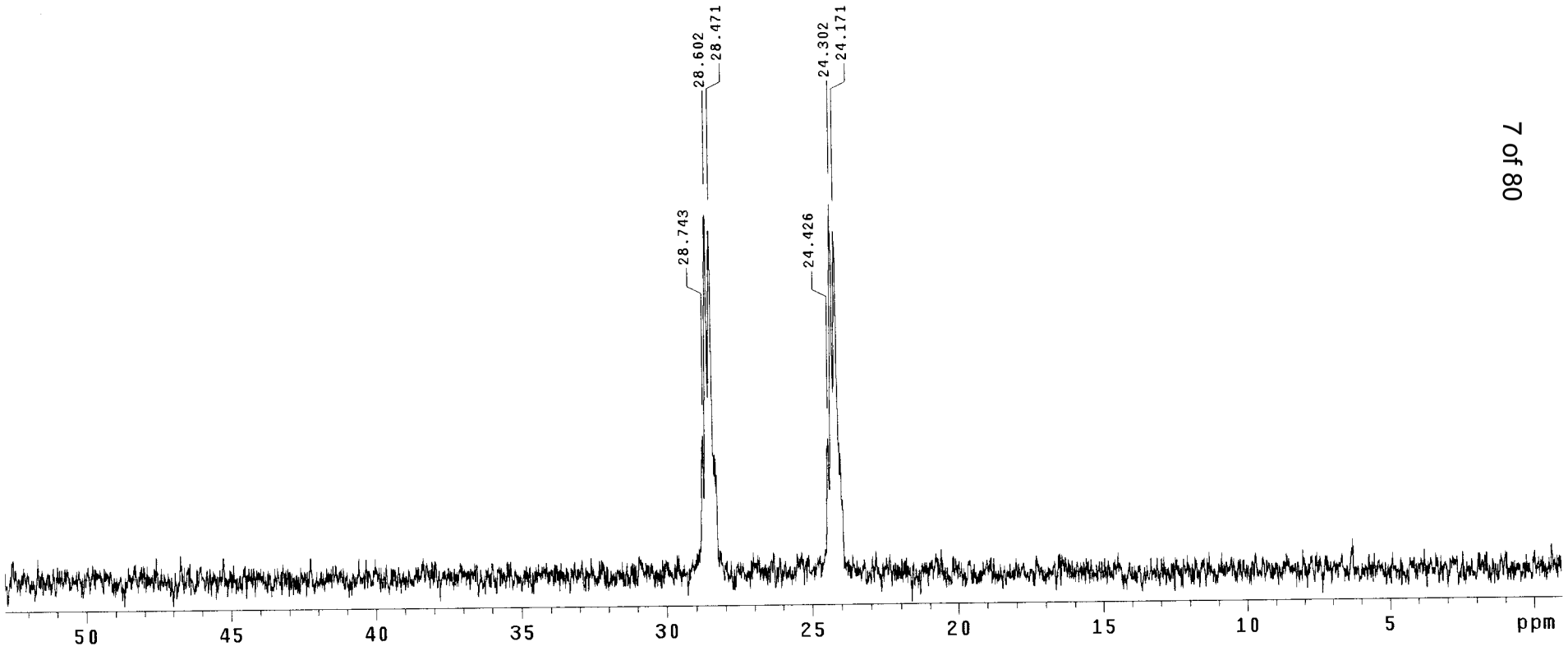
SAMPLE		SPECIAL	
date	Feb 11 2005	temp	not used
solvent	D2O	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	**FLAGS**	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	32	**PROCESSING**	
ct	32	lb	2.00
TRANSMITTER		fn	not used
tn	P31	**DISPLAY**	
sfrq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rfp	0
DECOUPLER		rp	-97.4
dn	H1	lp	-288.8
dof	0	**PLOT**	
dm	yyy	wc	250
dmm	w	sc	0
dpwr	35	vs	17
dmf	6700	th	4
		ai no ph	



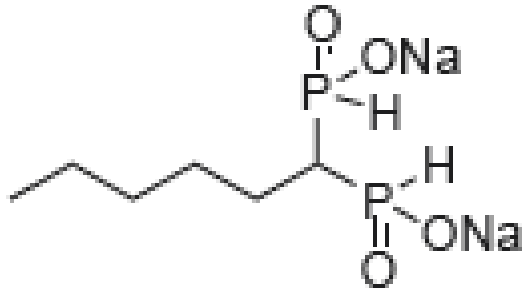


INDEX	FREQUENCY	PPM	HEIGHT
1	3491.182	28.743	22.3
2	3474.047	28.602	57.8
3	3458.135	28.471	55.2
4	2966.916	24.426	21.6
5	2951.821	24.302	59.3
6	2935.909	24.171	54.9



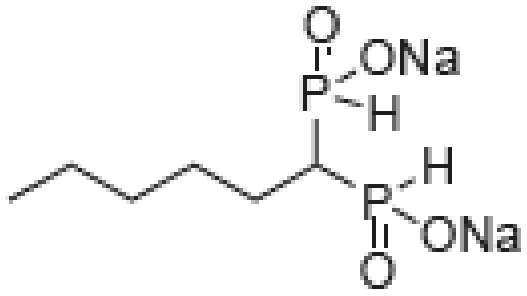


SB-I-81 PP in D_2O

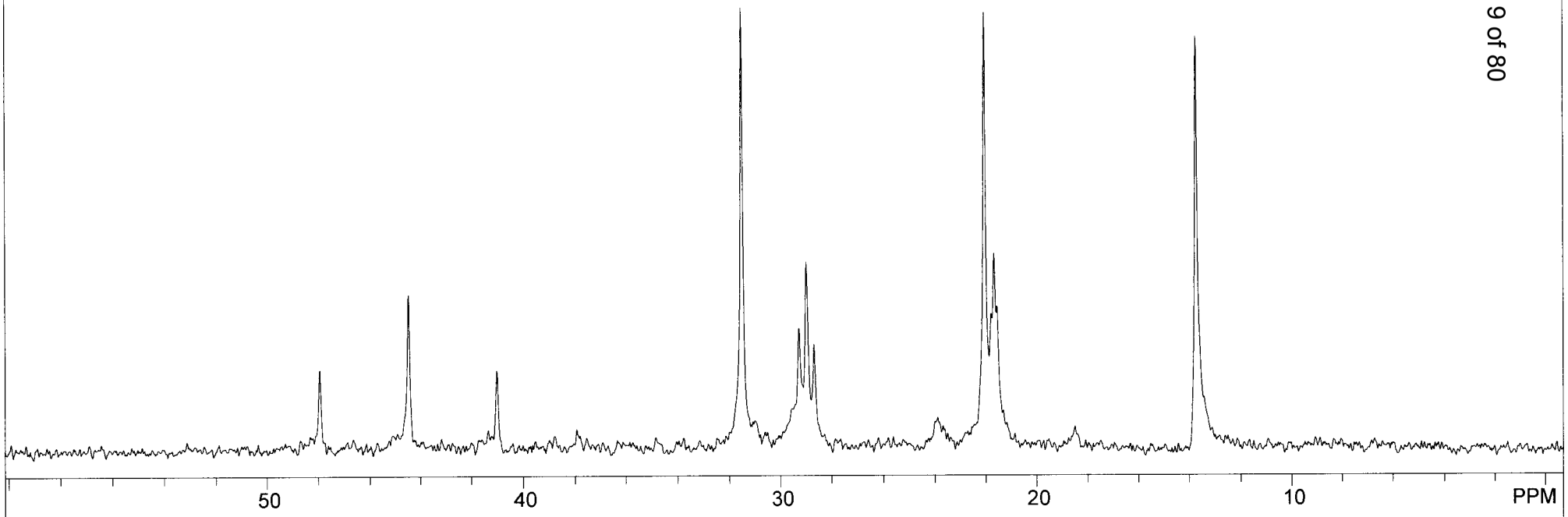


8 7 6 5 4 3 2 1 -0 ppm

PEAKLIST						
LINE	POINT	HZ	PPM	INTENSITY	REL_INT	LABEL
1	10828	1084.65	47.918	19858536	18.429	1
2	11001	1006.11	44.448	38126628	35.382	2
3	11173	927.99	40.997	19767290	18.344	3
4	11646	713.25	31.510	109187504	101.327	4
5	11758	662.24	29.257	29154802	27.056	5
6	11773	655.62	28.964	46309264	42.975	6
7	11788	648.89	28.667	26189134	24.304	7
8	12119	498.51	22.024	107336736	99.609	8
9	12138	489.70	21.634	47337060	43.929	9
10	12532	310.78	13.730	100141848	92.932	10



(in water).



9 of 80

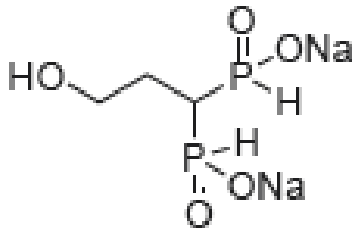
sb-I-144 13C

USER: -- DATE: 06/29/05 (18:26)

F1: 22.635	SW1: 7441		OF1: 2282.0		PTS1d: 16384
EX: bapr.ppg	PW: 18.2 usec	PD: 2.8 sec	NA: 1000	LB: 1.0	WinNuts - my_bapr so

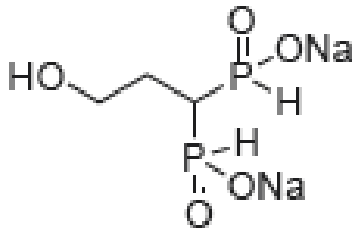
exp1 s2pul

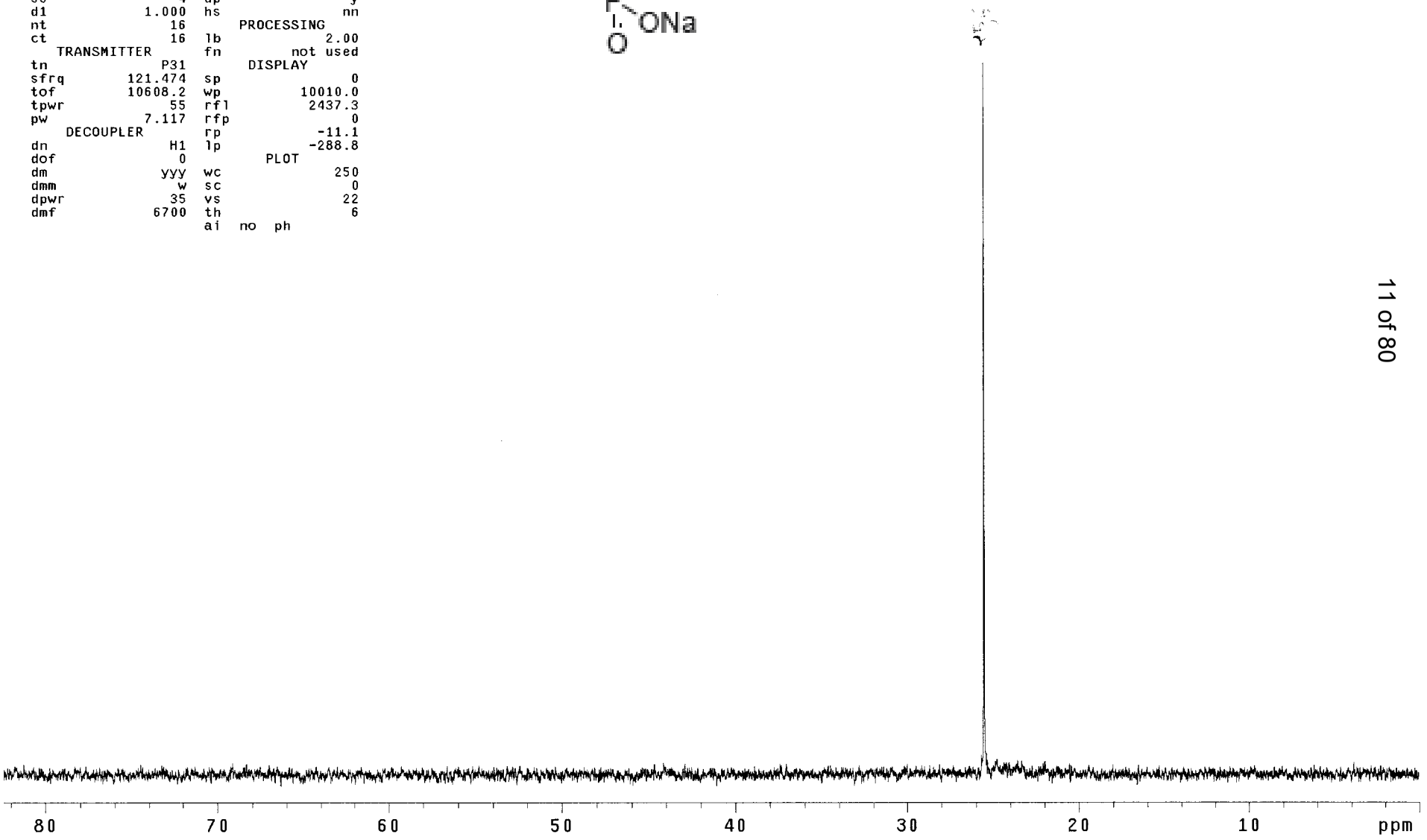
SAMPLE		SPECIAL	
date	Dec 1 2004	temp	not used
solvent	D2O	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	4803.1	pw90	17.200
at	1.994	alfa	20.000
np	19158	**FLAGS**	
fb	not used	il	n
bs	16	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	16	**PROCESSING**	
ct	16	fn	not used
TRANSMITTER		**DISPLAY**	
tn	H1	sp	-600.9
sfrq	300.054	wp	4802.8
tof	300.9	rfl	601.2
tpwr	55	rfp	0
pw	8.600	rp	-107.2
DECOUPLER		lp	-68.5
dn	C13	**PLOT**	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	211
dpwr	45	th	5
dmf	13100	ai cdc ph	



exp1 s2pul

SAMPLE		SPECIAL	
date	Dec 1 2004	temp	not used
solvent	D2O	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	FLAGS	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	16	PROCESSING	
ct	16	lb	2.00
TRANSMITTER		fn	not used
tn	P31	DISPLAY	
sfrq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rfp	0
DECOUPLER		rp	-11.1
dn	H1	lp	-288.8
dof	0	PLOT	
dm	yyy	wc	250
dmm	w	sc	0
dpwr	35	vs	22
dmf	6700	th	6
		ai no ph	

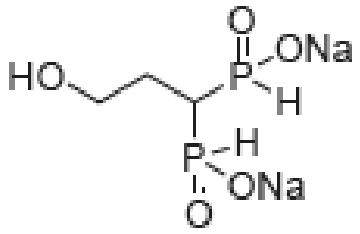


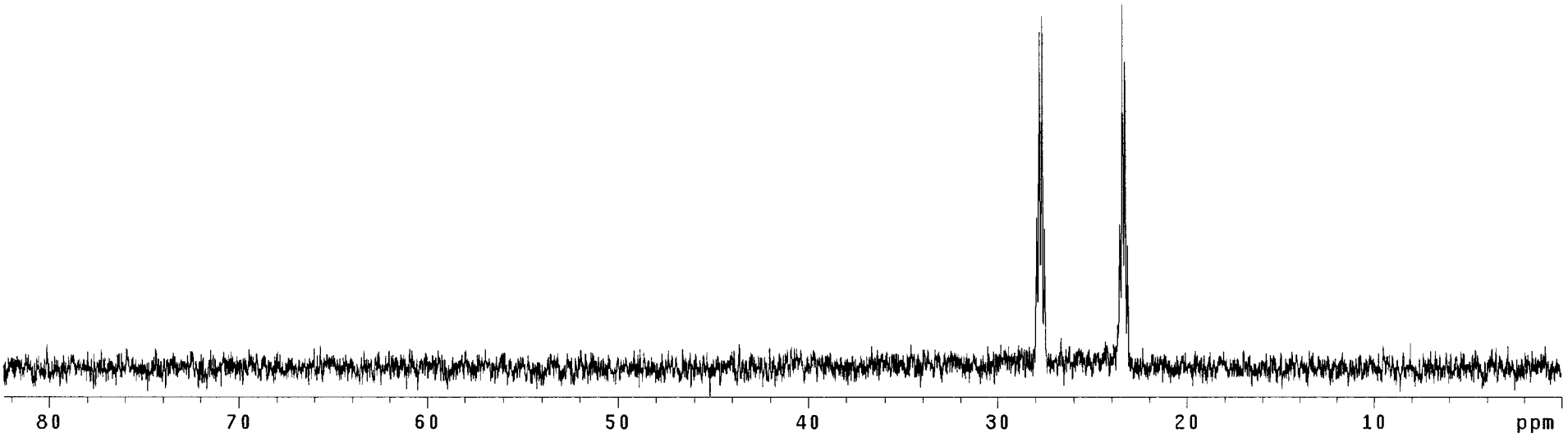


Sb-I-05 ^{31}P

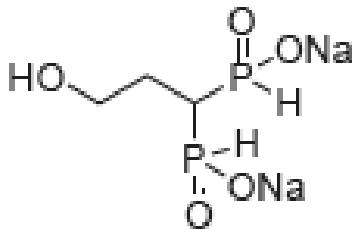
exp1 s2pul

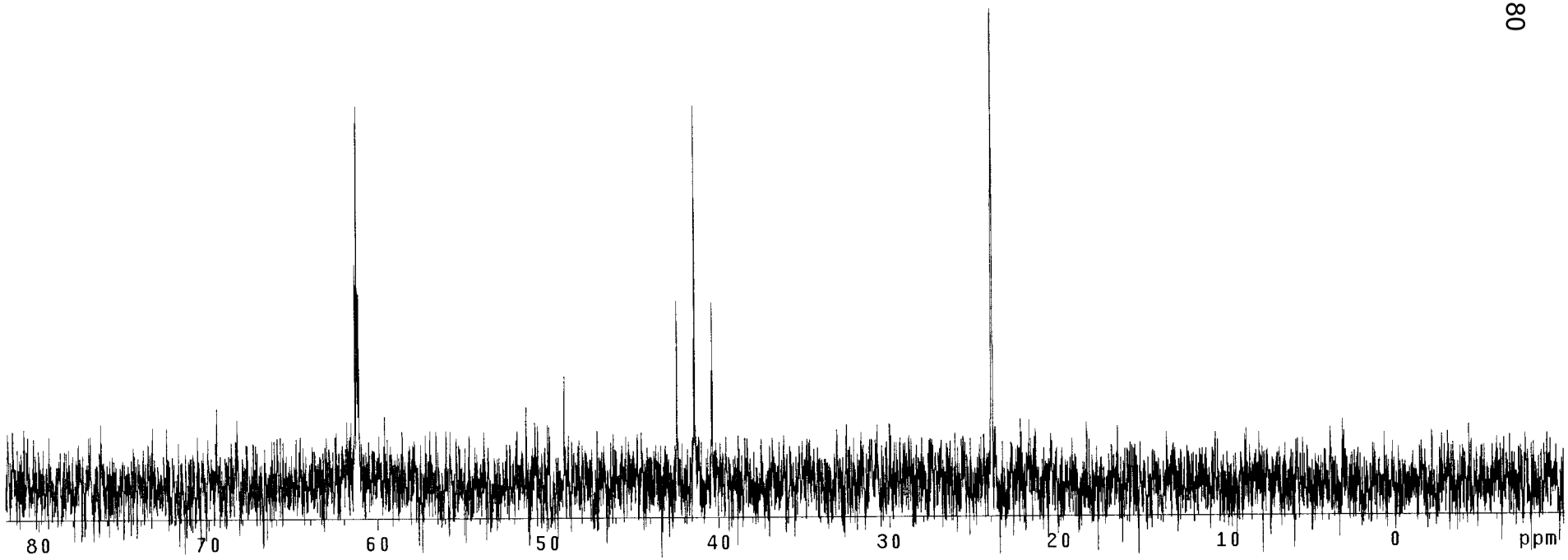
SAMPLE		SPECIAL	
date	Dec 1 2004	temp	not used
solvent	D2O	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	FLAGS	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	64	PROCESSING	
ct	64	lb	2.00
TRANSMITTER		fn	not used
tn	P31	DISPLAY	
sfrq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rfp	0
DECOUPLER		rp	-50.5
dn	H1	lp	-500.5
dof	0	PLOT	
dm	ynn	wc	250
dmm	w	sc	0
dpwr	35	vs	83
dmf	6700	th	12
		ai no ph	





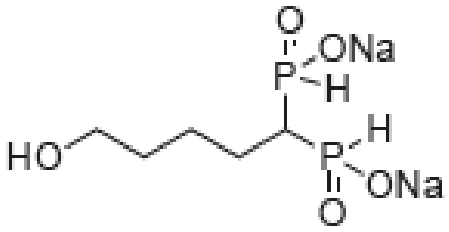
INDEX	FREQUENCY	PPM	HEIGHT
1	4626.526	61.321	35.7
2	4618.753	61.218	61.1
3	4610.980	61.115	30.9
4	3200.837	42.424	29.7
5	3123.104	41.394	61.0
6	3045.370	40.364	29.4
7	1806.242	23.940	76.4

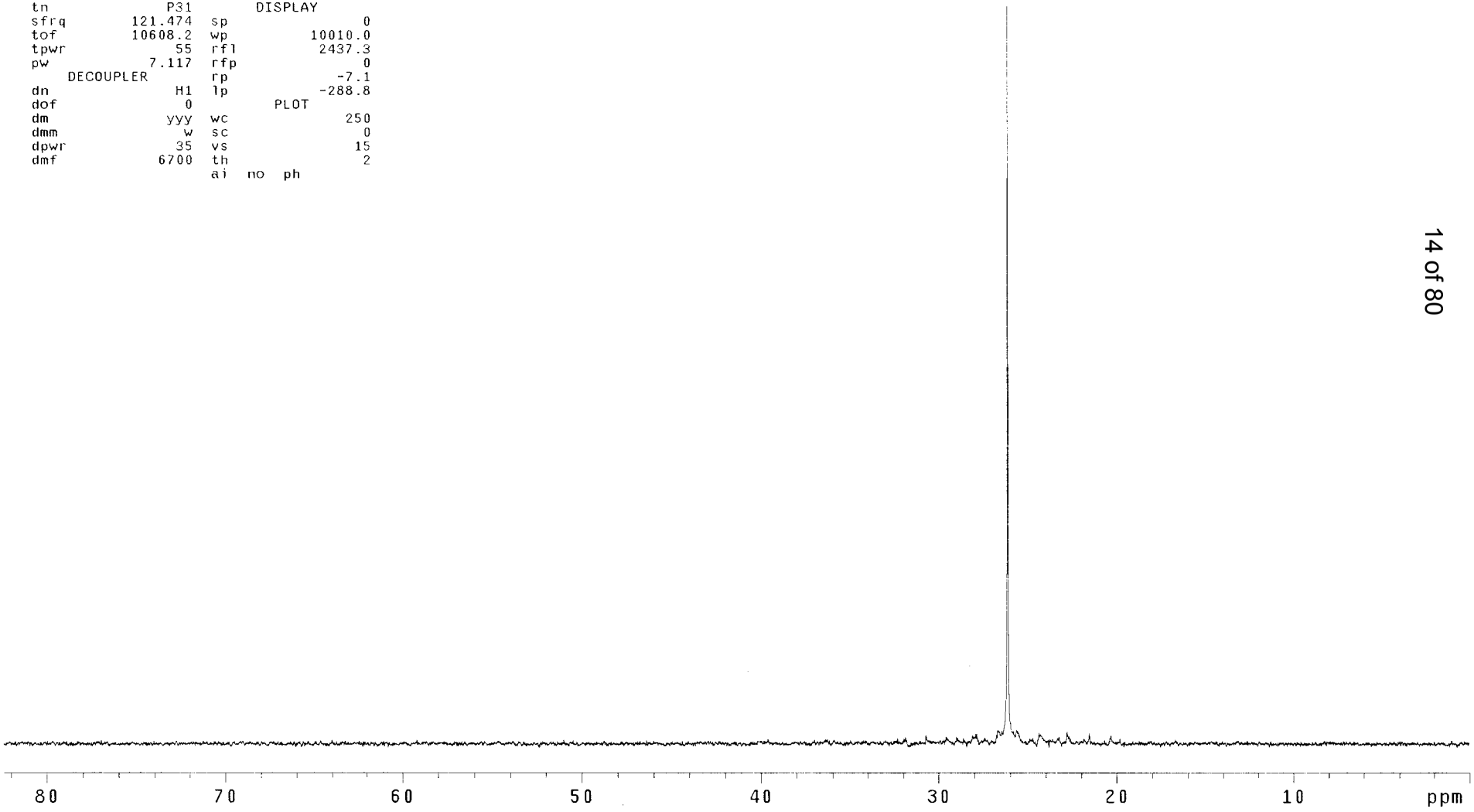




exp1 s2pul

SAMPLE		SPECIAL	
date	Apr 25 2005	temp	not used
solvent	D2O	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	**FLAGS**	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	16	**PROCESSING**	
ct	16	lb	2.00
TRANSMITTER		fn	not used
tn	P31	**DISPLAY**	
sfrq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rfp	0
DECOUPLER		rp	-7.1
dn	H1	lp	-288.8
dof	0	**PLOT**	
dm	yyy	wc	250
dmm	w	sc	0
dpwr	35	vs	15
dmf	6700	th	2
		ai no ph	

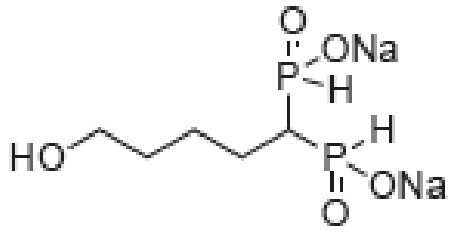




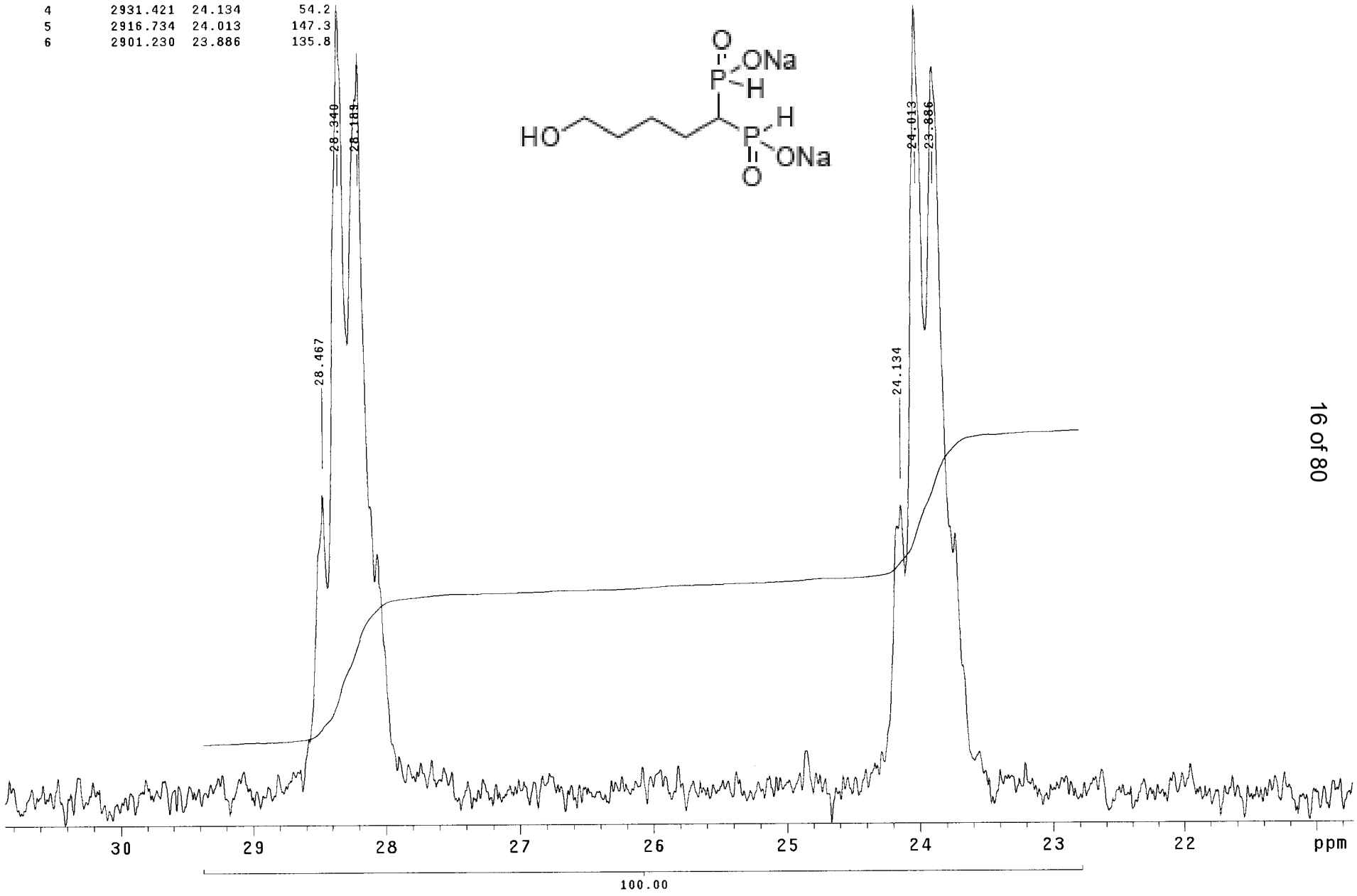
sb-I-139 pp in D2O

exp1 s2pul

SAMPLE		SPECIAL	
date	Mar 21 2005	temp	not used
solvent	D2O	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	4803.1	pw90	17.200
at	1.994	alfa	20.000
np	19158	FLAGS	
fb	not used	il	n
bs	16	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	16	PROCESSING	
ct	16	fn	not used
TRANSMITTER		DISPLAY	
tn	H1	sp	-600.9
sfrq	300.054	wp	4802.8
tof	300.9	rfl	601.2
tpwr	55	rfp	0
pw	8.600	rp	-110.2
DECOUPLER		lp	-63.5
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	952
dpwr	45	th	25
dmf	13100	ai cdc ph	

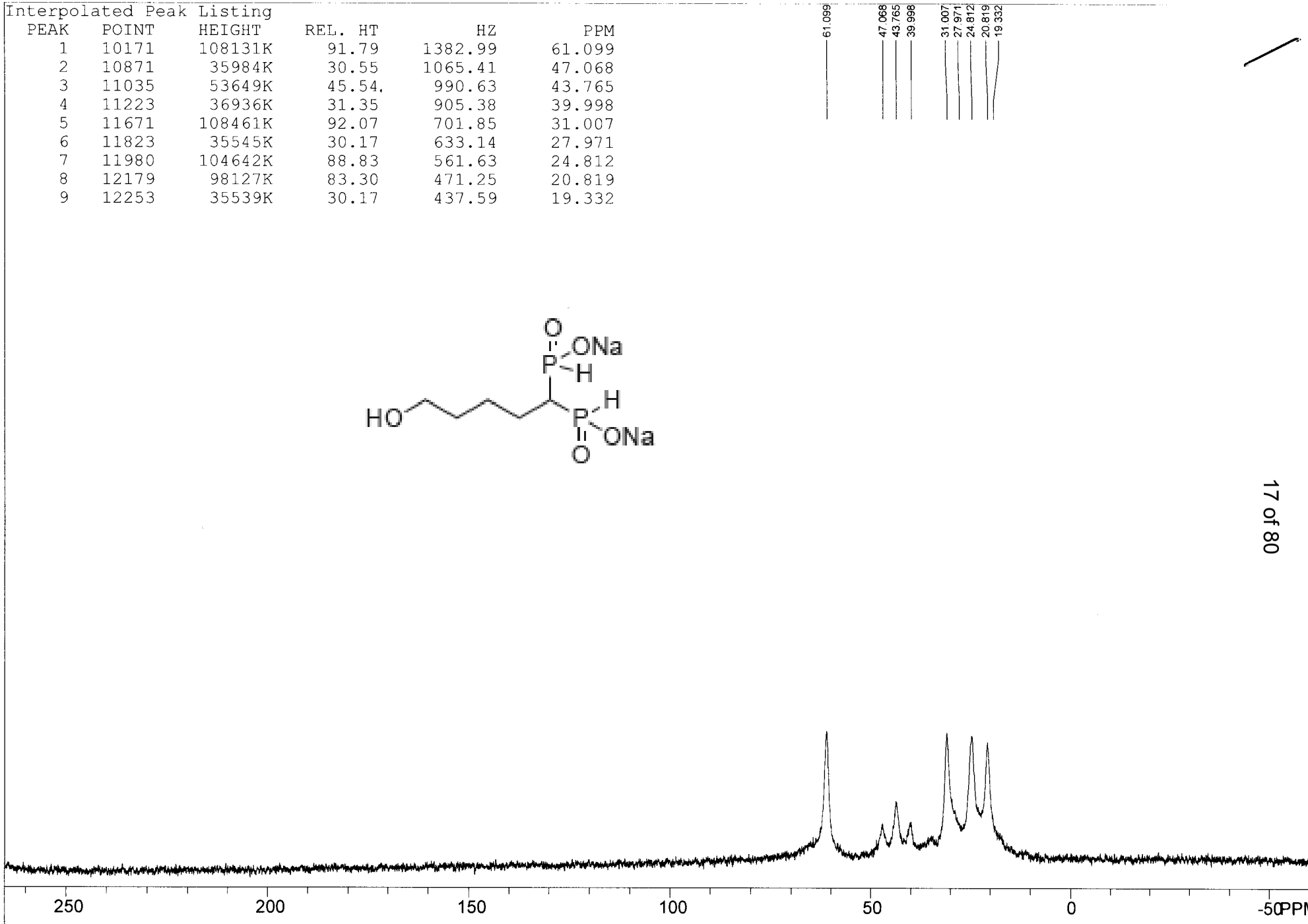


INDEX	FREQUENCY	PPM	HEIGHT
1	3457.727	28.467	56.5
2	3442.223	28.340	147.9
3	3423.864	28.189	138.8
4	2931.421	24.134	54.2
5	2916.734	24.013	147.3
6	2901.230	23.886	135.8



Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	10171	108131K	91.79	1382.99	61.099
2	10871	35984K	30.55	1065.41	47.068
3	11035	53649K	45.54.	990.63	43.765
4	11223	36936K	31.35	905.38	39.998
5	11671	108461K	92.07	701.85	31.007
6	11823	35545K	30.17	633.14	27.971
7	11980	104642K	88.83	561.63	24.812
8	12179	98127K	83.30	471.25	20.819
9	12253	35539K	30.17	437.59	19.332



sb-I-139 C13

USER: -- DATE: 07/02/05 (12:58)

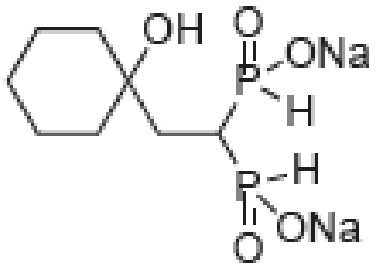
F1: 22.635	SW1: 7441		OF1: 2282.0		PTS1d: 16384
EX: bapr.ppg	PW: 18.2 usec	PD: 2.8 sec	NA: 2000	LB: 1.0	WinNuts - my_bapr so 139 b

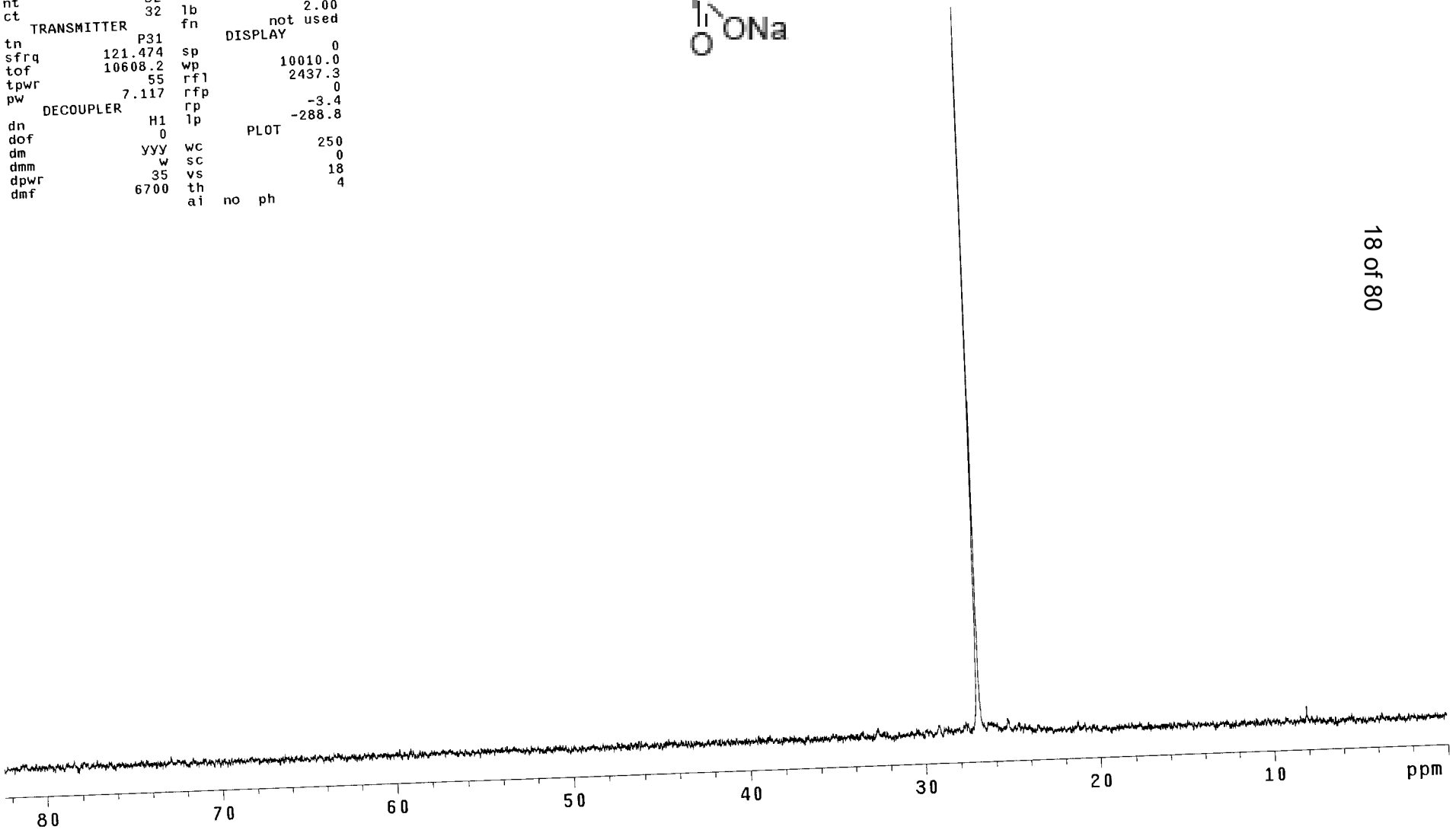
INDEX	FREQUENCY	PPM	HEI
1	3274.540	26.959	

sb-I-138 pp

exp1 s2pul

SAMPLE		SPECIAL	
date	Mar 21 2005	temp	not used
solvent	D2O	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	FLAGS	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	32	PROCESSING	
ct	32	lb	2.00
TRANSMITTER		fn	not used
tn	P31	DISPLAY	
sfrq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rfp	0
DECOUPLER		rp	-3.4
dn	H1	lp	-288.8
dof	0	PLOT	
dm	yyy	wc	250
dmm	w	sc	0
dpwr	35	vs	18
dmf	6700	th	4
		ai	no ph

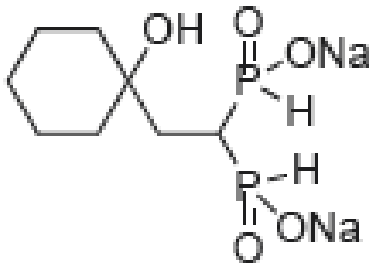


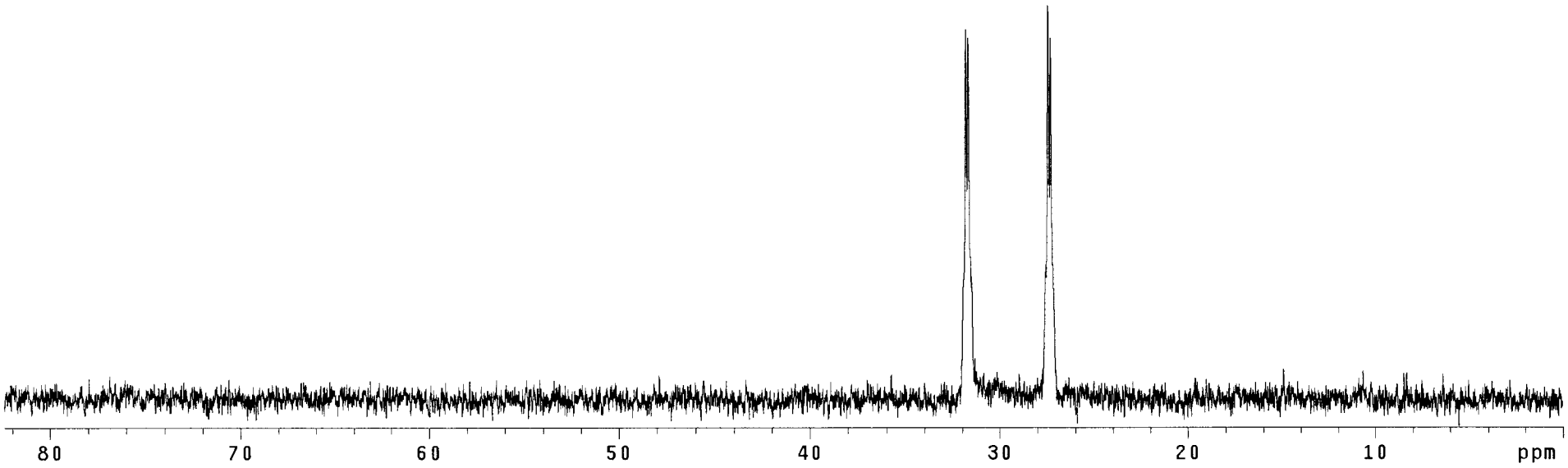


exp1 s2pul

SAMPLE		SPECIAL	
date	Mar 21 2005	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	**FLAGS**	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	32	**PROCESSING**	
ct	32	lb	2.00
TRANSMITTER		fn	not used
tn	P31	**DISPLAY**	
sfrq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rfp	0
DECOUPLER		rp	71.6
dn	H1	lp	-621.5
dof	0	**PLOT**	
dm	ynn	wc	250
dmm	w	sc	0
dpwr	35	vs	60
dmf	6700	th	13
		ai no ph	

INDEX	FREQUENCY	PPM	HEI
1	3861.605	31.793	
2	3846.102	31.665	
3	3330.404	27.419	
4	3314.084	27.285	

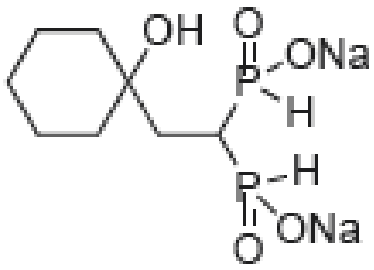




sb-I-138 pp in D2O

exp1 s2pul

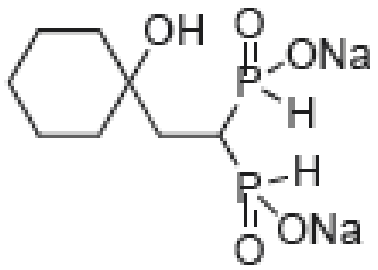
SAMPLE		SPECIAL	
date	Mar 21 2005	temp	not used
solvent	D2O	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	4803.1	pw90	17.200
at	1.994	alfa	20.000
np	19158	FLAGS	
fb	not used	il	n
bs	16	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	16	PROCESSING	
ct	16	fn	not used
TRANSMITTER		DISPLAY	
tn	H1	sp	-600.9
sfrq	300.054	wp	4802.8
tof	300.9	rfl	601.2
tpwr	55	rfp	0
pw	8.600	rp	-89.4
DECOUPLER		lp	-118.9
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	642
dpwr	45	th	35
dmf	13100	ai cdc ph	

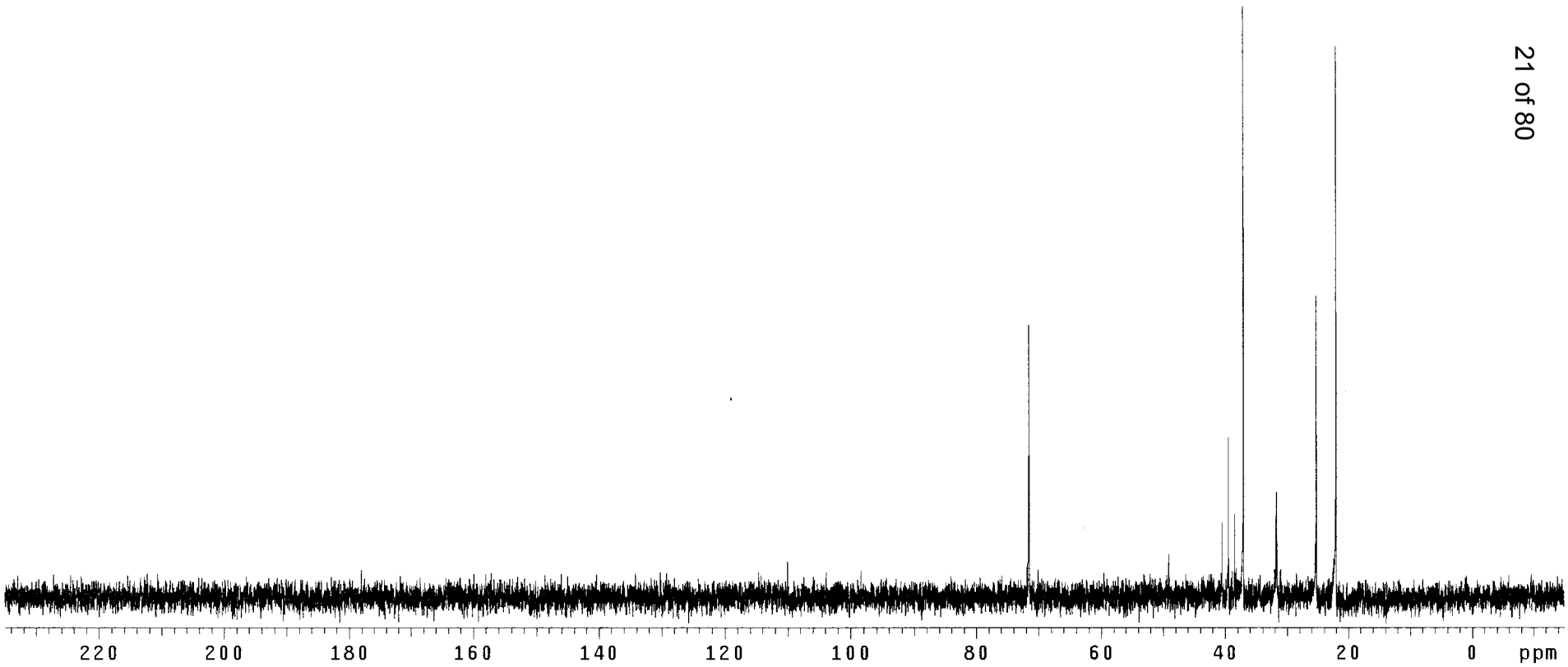


sb-I-09 13C in D2O

exp1 s2pul

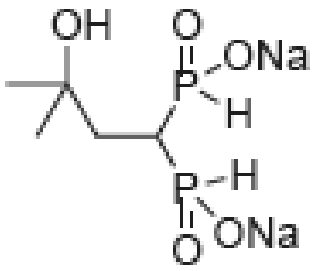
SAMPLE		SPECIAL	
date	Dec 28 2004	temp	not used
solvent	D2O	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	18867.9	pw90	18.500
at	1.815	alfa	20.000
np	68492	FLAGS	
fb	10400	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	1000	PROCESSING	
ct	1000	lb	1.00
TRANSMITTER		fn	not used
tn	C13	DISPLAY	
sfrq	75.456	sp	-1134.4
tof	737.2	wp	18867.6
tpwr	58	rfl	1134.7
pw	9.250	rfp	0
DECOUPLER		rp	167.1
dn	H1	lp	-333.6
dof	0	PLOT	
dm	yyy	wc	250
dmm	w	sc	0
dpwr	35	vs	280
dmf	6700	th	17
		ai no ph	

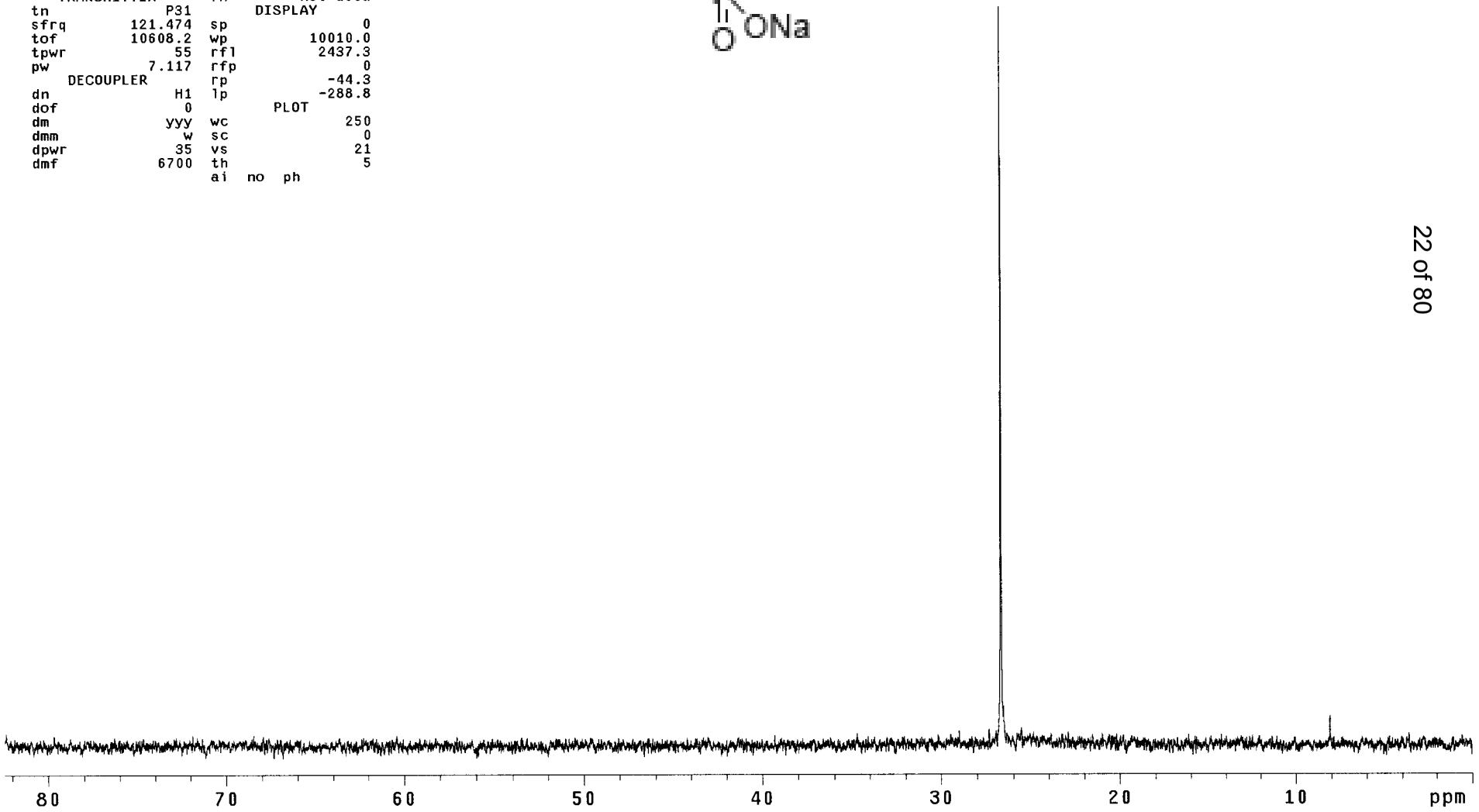




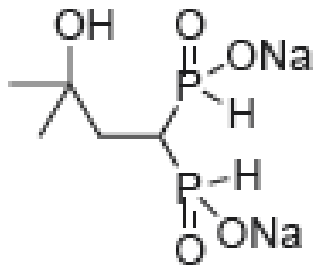
exp1 s2pul

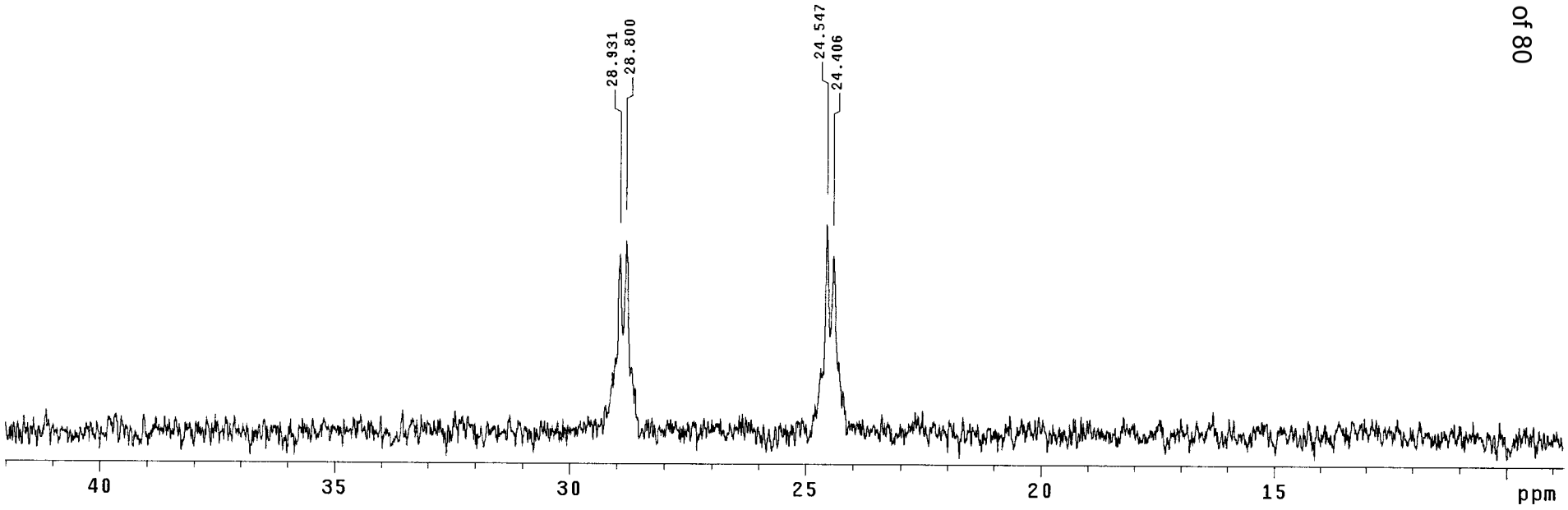
SAMPLE		SPECIAL	
date	Jul 5 2005	temp	not used
solvent	D2O	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	FLAGS	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	16	PROCESSING	
ct	16	lb	2.00
TRANSMITTER		fn	not used
tn	P31	DISPLAY	
sfrq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rfp	0
DECOUPLER		rp	-44.3
dn	H1	lp	-288.8
dof	0	PLOT	
dm	yyy	wc	250
dmm	w	sc	0
dpwr	35	vs	21
dmf	6700	th	5
		ai no ph	





INDEX	FREQUENCY	PPM	HEIGHT
1	3514.030	28.931	28.8
2	3498.118	28.800	31.0
3	2981.604	24.547	33.6
4	2964.468	24.406	28.6

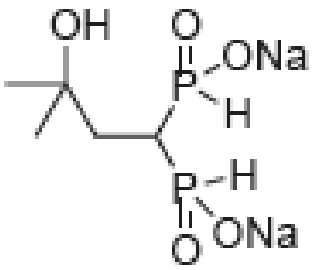




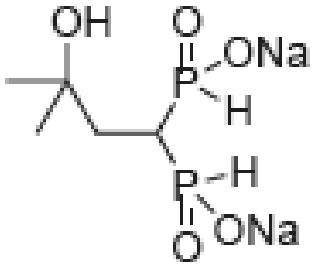
sb-II-243 pp

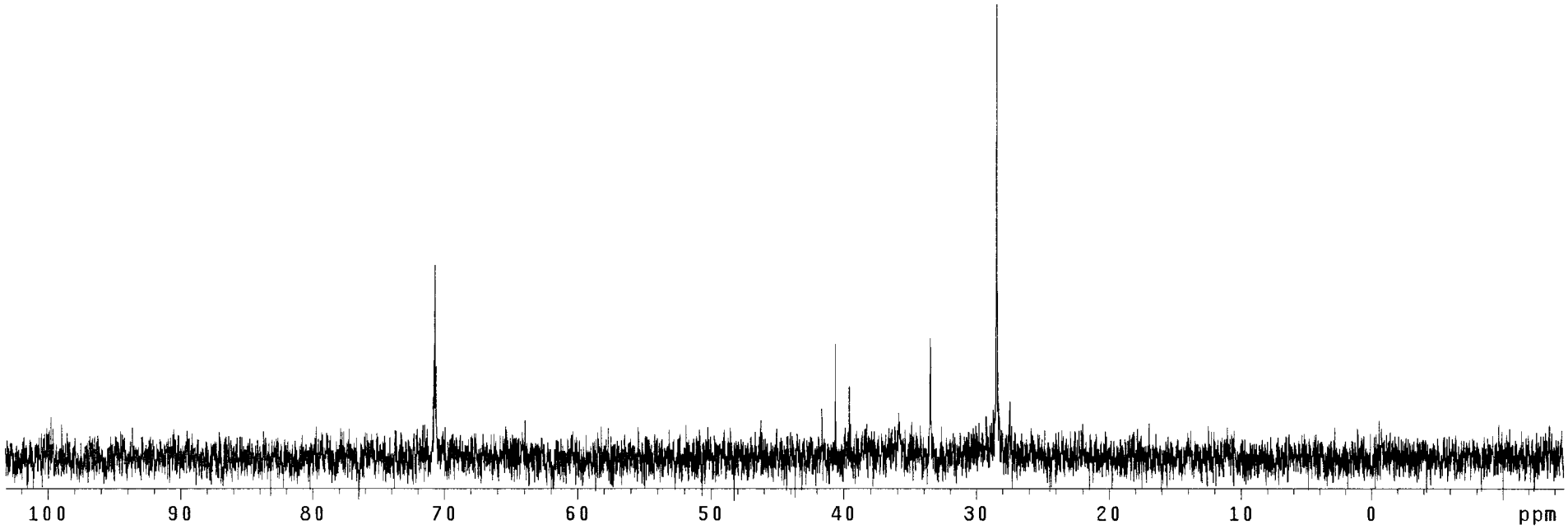
exp1 s2pul

SAMPLE		SPECIAL	
date	Jul 5 2005	temp	not used
solvent	D2O	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	4803.1	pw90	17.200
at	1.994	alfa	20.000
np	19158	**FLAGS**	
fb	not used	il	n
bs	16	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	16	**PROCESSING**	
ct	16	fn	not used
TRANSMITTER		**DISPLAY**	
tn	H1	sp	-60.0
sfrq	300.054	wp	3060.5
tof	300.9	rfl	601.2
tpwr	55	rfp	0
pw	8.600	rp	-109.4
DECOUPLER		lp	-75.6
dn	C13	**PLOT**	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	817
dpwr	45	th	27
dmf	13100	ai cdc ph	



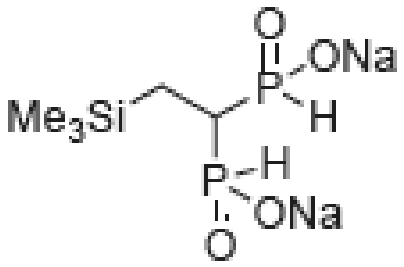
INDEX	FREQUENCY	PPM	HEIGHT
1	5339.083	70.765	17.7
2	5333.037	70.685	30.8
3	5327.279	70.609	14.6
4	3060.917	40.570	18.1
5	2525.996	33.480	19.1
6	2144.238	28.420	72.7

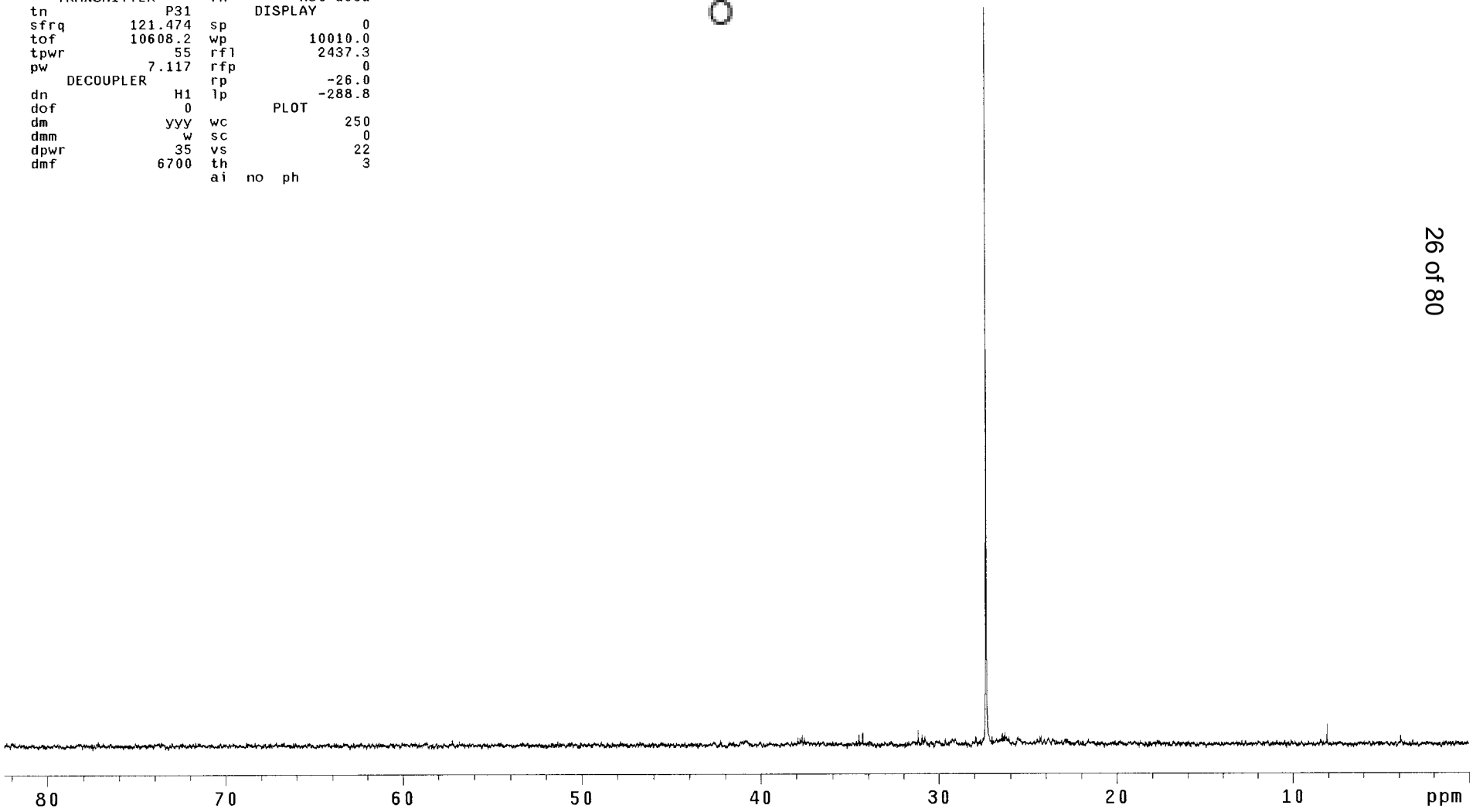


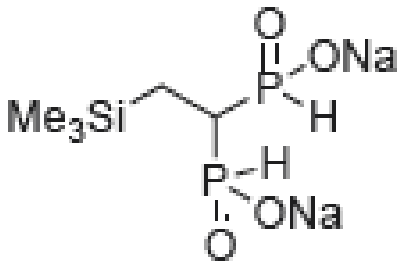


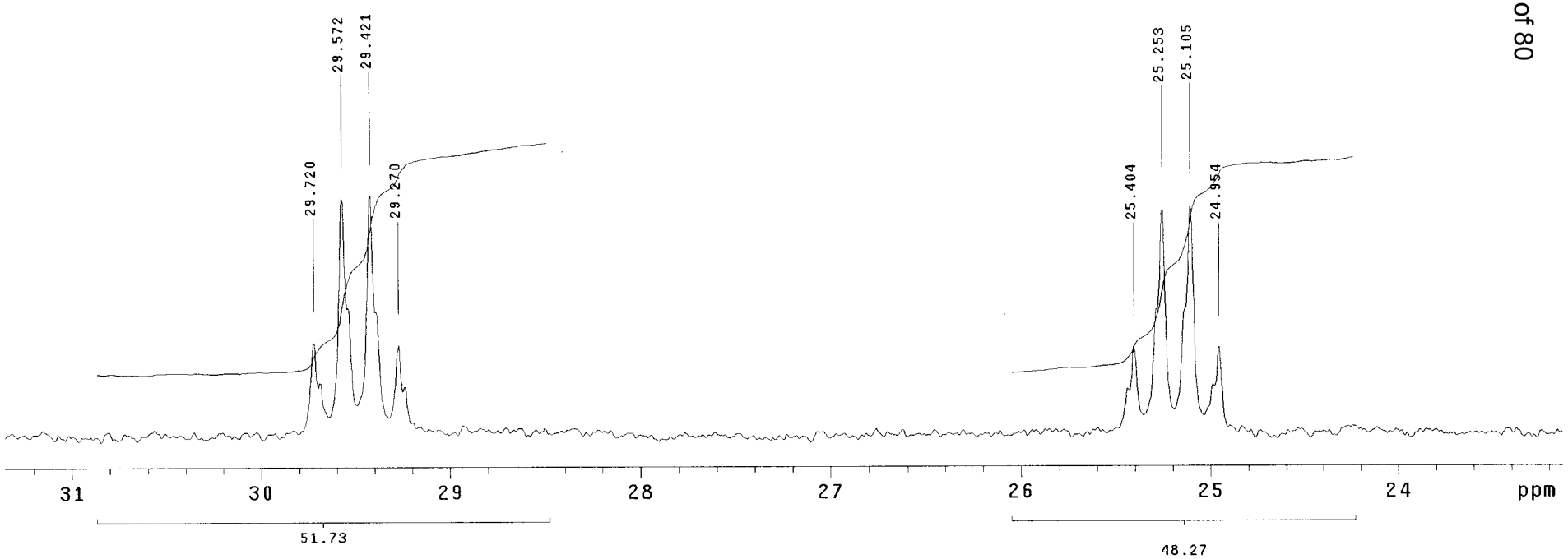
exp1 s2pul

SAMPLE		SPECIAL	
date	Dec 10 2004	temp	not used
solvent	D2O	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	**FLAGS**	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	32	**PROCESSING**	
ct	32	lb	2.00
TRANSMITTER		fn	not used
tn	P31	**DISPLAY**	
sfrq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rfp	0
DECOUPLER		rp	-26.0
dn	H1	lp	-288.8
dof	0	**PLOT**	
dm	yyy	wc	250
dmm	w	sc	0
dpwr	35	vs	22
dmf	6700	th	3
		ai no ph	

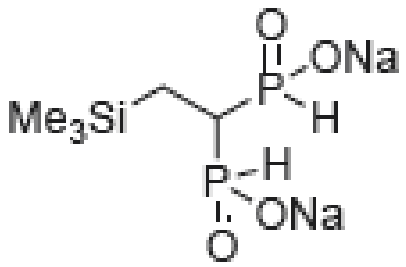





O
ONa
P
H
Me3Si
P
H
ONa
O


29.720
29.572
29.421
29.270
25.404
25.253
25.105
24.954
31
30
29
28
27
26
25
24
ppm
51.73
48.27

ppm
0
1
2
3
4
5
6
7
8
9
Me3Si
P
H
ONa
O
P
H
ONa
O



41.396
38.037
34.577
4.942
-3.040

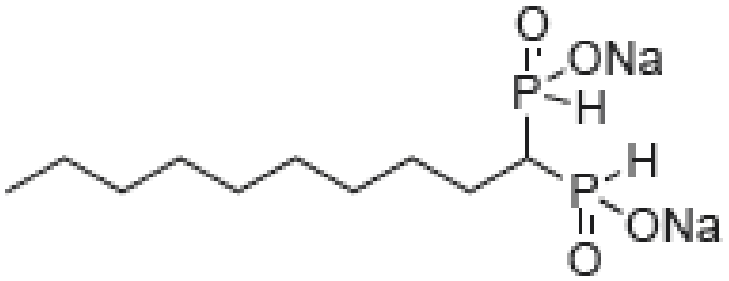
80 60 40 20 0 PPM

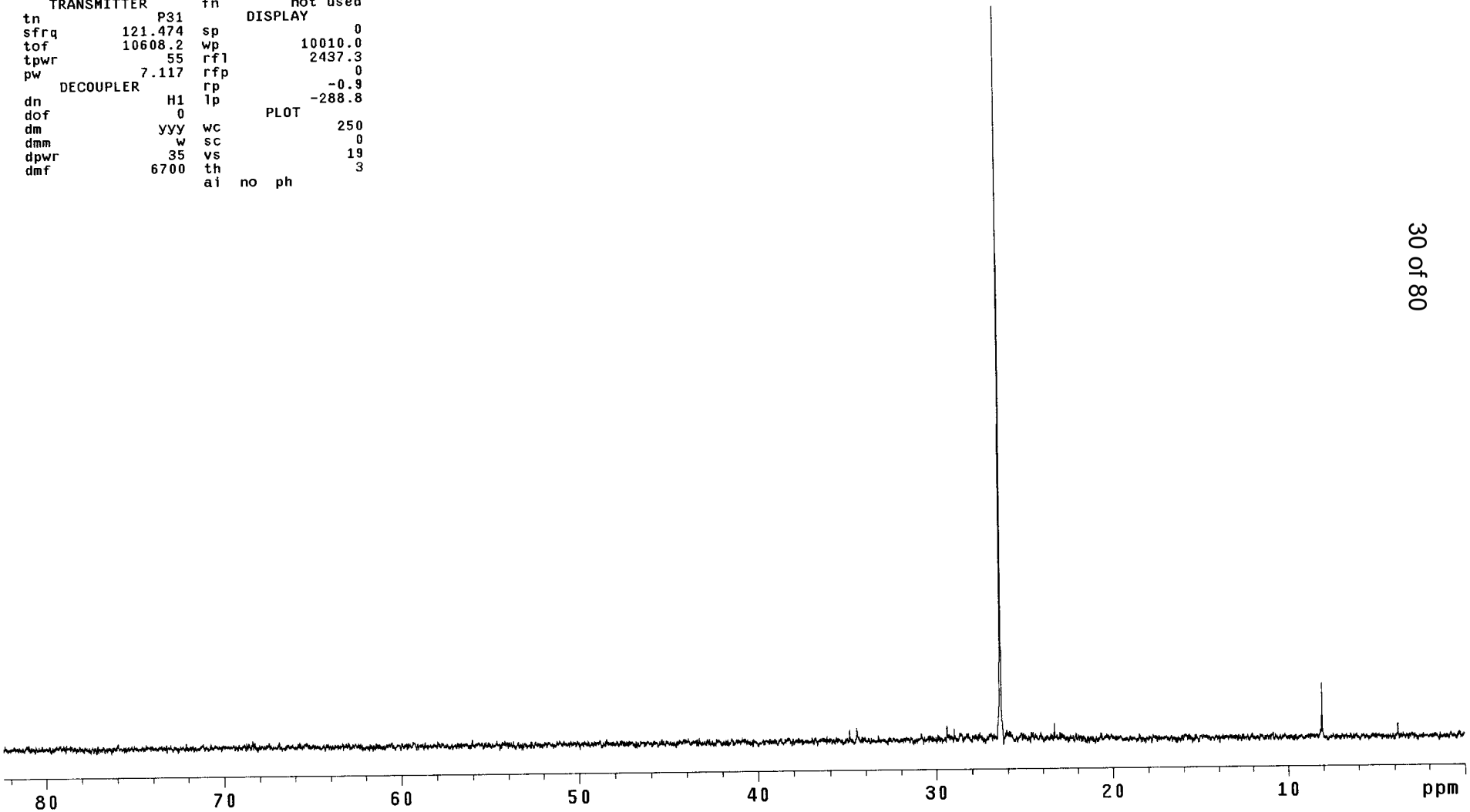
sb-II-237 in H_2O USER: -- DATE: 08/29/05 (20:18)

F1: 22.635	SW1: 7441		OF1: 2282.0		PTS1d: 16384
EX: bapr.ppg	PW: 18.2 usec	PD: 2.8 sec	NA: 1000	LB: 1.0	WinNuts - my_bapr_sbII237

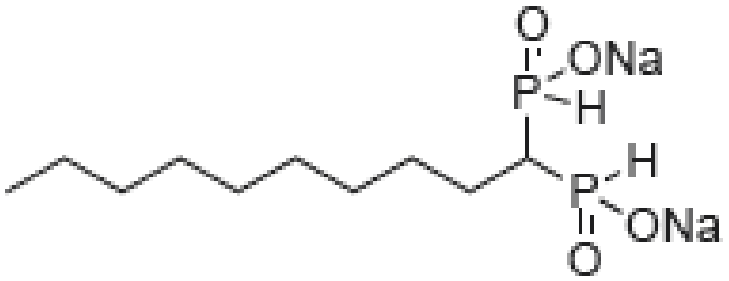
exp1 s2pul

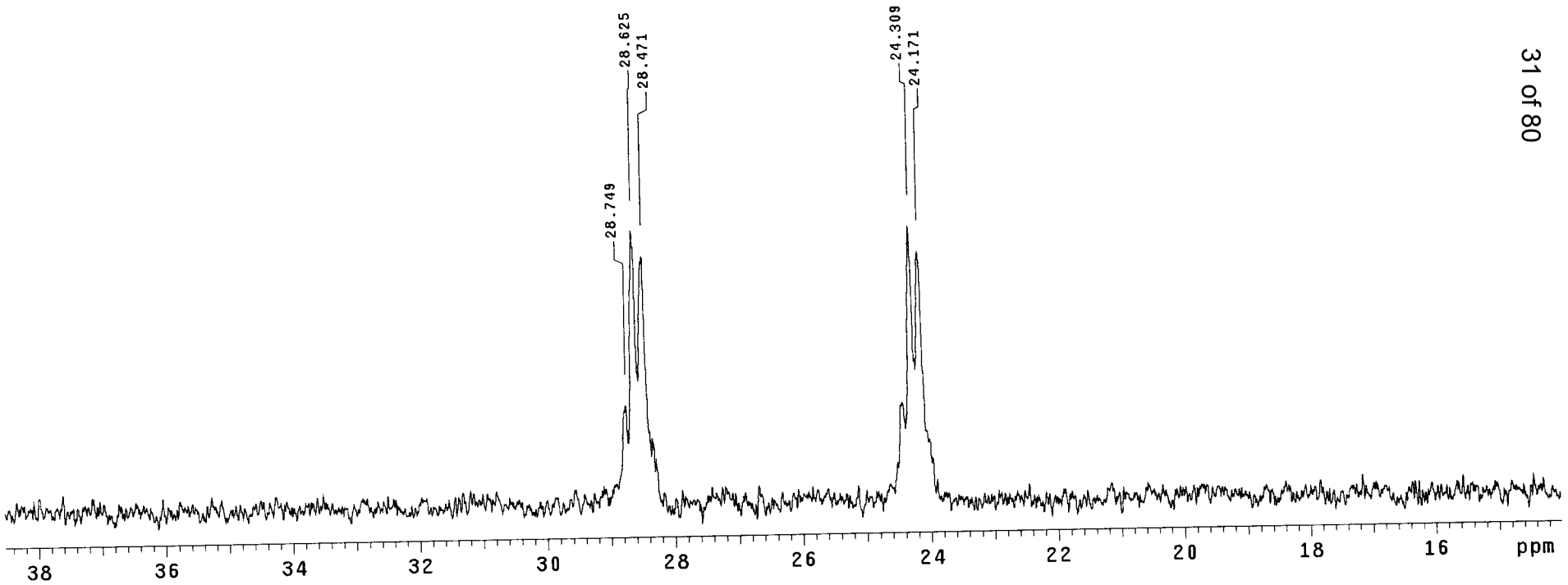
SAMPLE		SPECIAL	
date	Jun 30 2005	temp	not used
solvent	D2O	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	**FLAGS**	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	16	**PROCESSING**	
ct	16	lb	2.00
TRANSMITTER		fn	not used
tn	P31	**DISPLAY**	
sfrq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rfp	0
DECOUPLER		rp	-0.9
dn	H1	lp	-288.8
dof	0	**PLOT**	
dm	yyy	wc	250
dmm	w	sc	0
dpwr	35	vs	19
dmf	6700	th	3
		ai	no ph

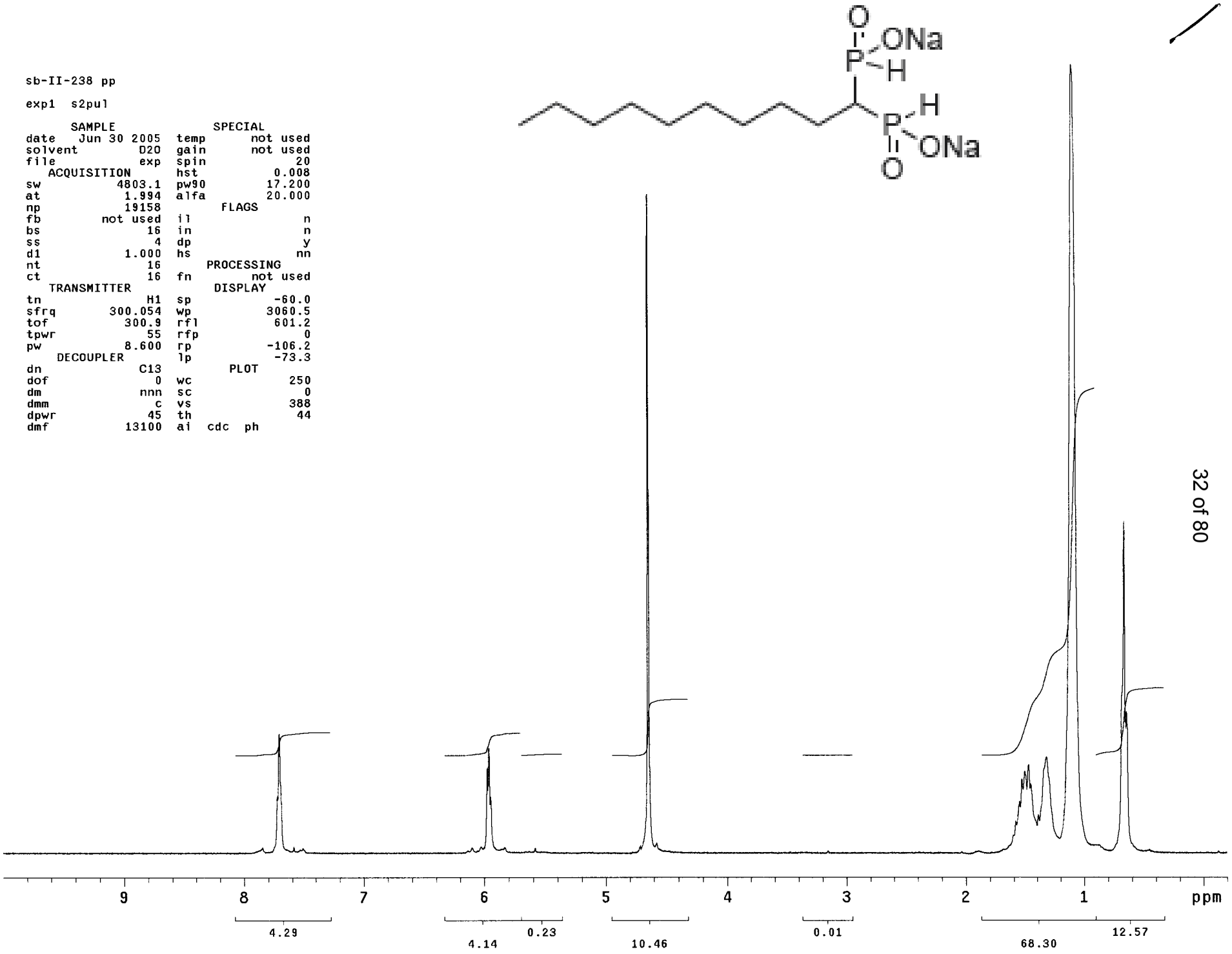




INDEX	FREQUENCY	PPM	HEIGHT
1	3491.998	28.749	16.3
2	3476.903	28.625	44.4
3	3458.135	28.471	40.3
4	2952.637	24.309	44.4
5	2935.909	24.171	40.3

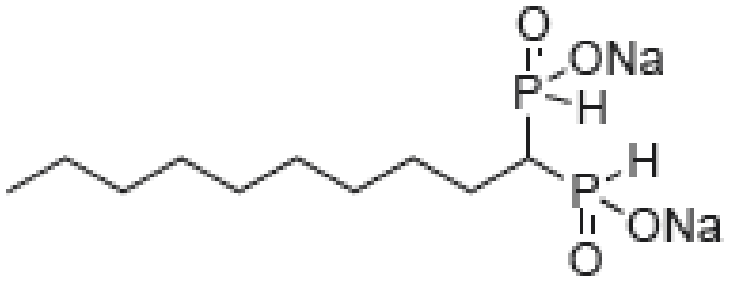





sb-II-238 pp
exp1 s2pul
SAMPLE
date Jun 30 2005
solvent D2O
file exp
ACQUISITION
sw 4803.1
at 1.994
np 19158
fb not used
bs 16
ss 4
d1 1.000
nt 16
ct 16
TRANSMITTER
tn H1
sfrq 300.054
tof 300.9
tpwr 55
pw 8.600
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 45
dmf 13100
SPECIAL
temp not used
gain not used
spin 20
hst 0.008
pw90 17.200
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -60.0
wp 3060.5
rfl 601.2
rfp 0
rp -106.2
lp -73.3
PLOT
wc 250
sc 0
vs 388
th 44
ai cdc ph
O
ONa
P
H
H
P
O
ONa
32 of 80
9
8
7
6
5
4
3
2
1
ppm
4.29
4.14
0.23
10.46
0.01
68.30
12.57

Interpolated Peak Listing

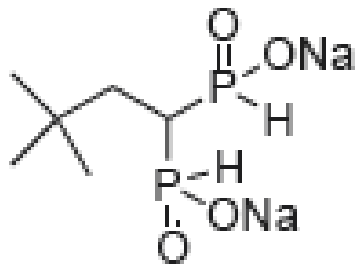
PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	11039	9834K	17.75	988.90	43.688
2	11688	27546K	49.71	694.06	30.663
3	11819	51903K	93.67	634.84	28.046
4	12145	25041K	45.19	486.87	21.509
5	12172	13257K	23.93	474.38	20.957
6	12572	22467K	40.55	292.69	12.930



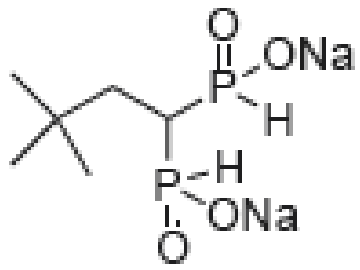
sb-II_238 C13 USER: -- DATE: 07/07/05 (17:04)

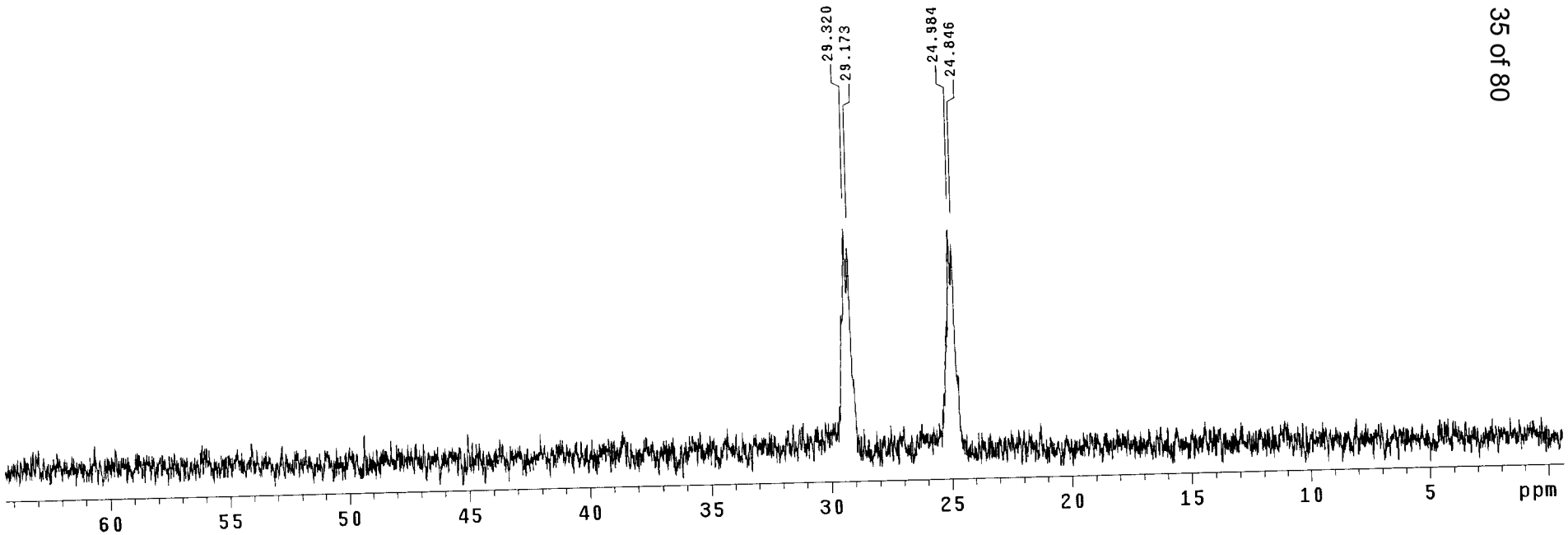
F1: 22.635		SW1: 7441		OF1: 2282.0		PTS1d: 16384	
EX: bapr.ppg		PW: 18.2 usec	PD: 2.8 sec	NA: 2000	LB: 1.0	WinNuts - my_bapr-sb-II-238	

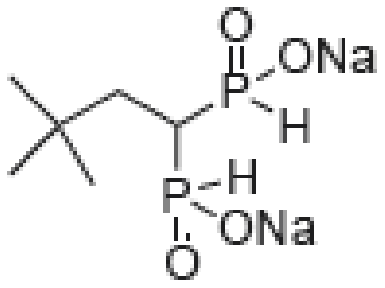
INDEX	FREQUENCY	PPM	HEIGHT
1	3288.004	27.070	126.0



INDEX	FREQUENCY	PPM	HEIGHT
1	3561.356	29.320	35.6
2	3543.405	29.173	32.5
3	3034.642	24.984	35.1
4	3017.915	24.846	32.8





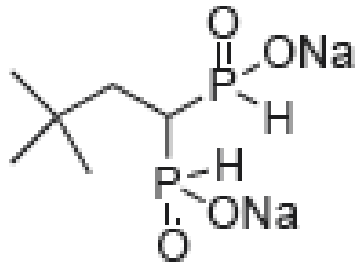


9 8 7 6 5 4 3 2 1 ppm

7.07 6.79 19.10 67.08 -0.04

Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	11216	21577K	12.53	908.40	40.132
2	11537	43880K	25.48	762.76	33.698
3	11734	31524K	18.30	673.53	29.756
4	11801	169589K	98.47	642.75	28.396



80 60 40 20 PPM

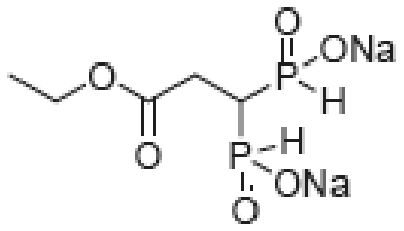
sb-II-240 in H2O USER: -- DATE: 07/26/05 (21:18)

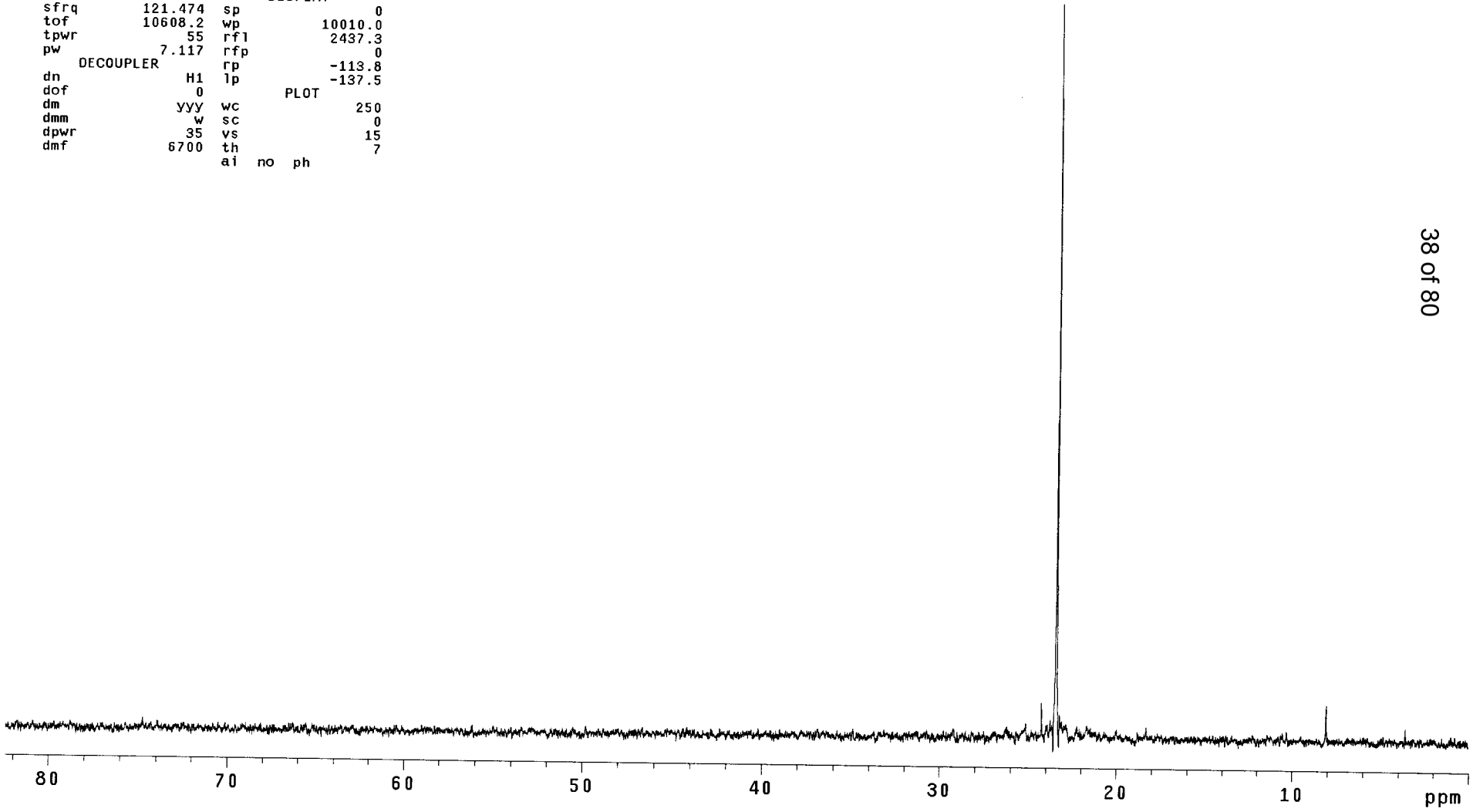
F1: 22.635	SW1: 7441		OF1: 2282.0		PTS1d: 16384
EX: bapr.ppg	PW: 18.2 usec	PD: 2.8 sec	NA: 1500	LB: 1.0	WinNuts - my_bapr_sb-II-240 in H2O

sb-II-241 pp

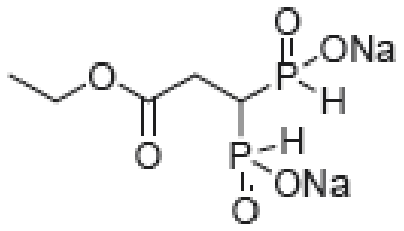
exp1 s2pul

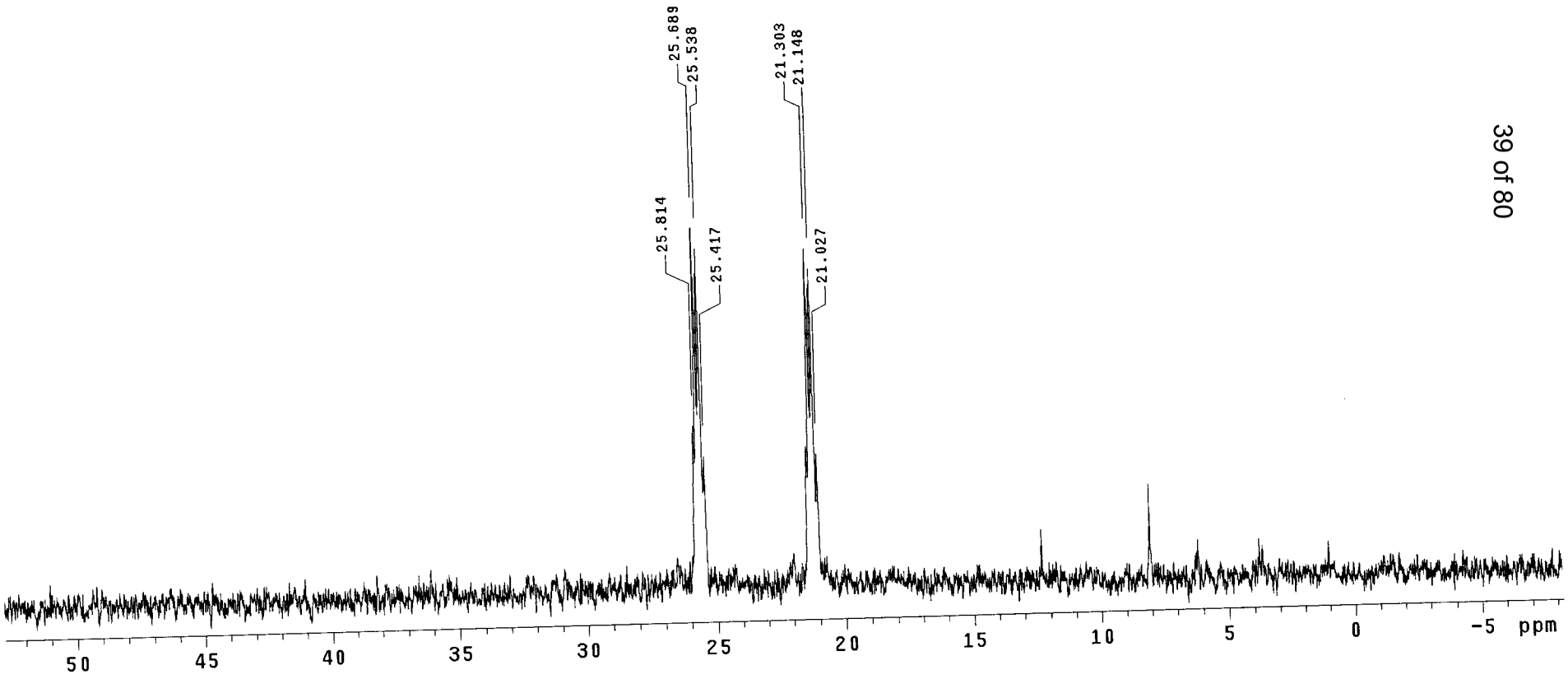
SAMPLE		SPECIAL	
date	Jul 5 2005	temp	not used
solvent	D2O	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	FLAGS	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	16	PROCESSING	
ct	16	lb	2.00
TRANSMITTER		fn	not used
tn	P31	DISPLAY	
sfrq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rfp	0
DECOUPLER		rp	-113.8
dn	H1	lp	-137.5
dof	0	PLOT	
dm	yyy	wc	250
dmm	w	sc	0
dpwr	35	vs	15
dmf	6700	th	7
		ai no ph	





INDEX	FREQUENCY	PPM	HEIGHT
1	3135.416	25.814	26.6
2	3120.320	25.689	58.5
3	3101.961	25.538	55.0
4	3087.273	25.417	21.6
5	2587.487	21.303	54.6
6	2568.719	21.148	51.4
7	2554.031	21.027	21.5

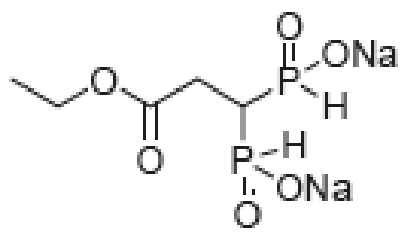


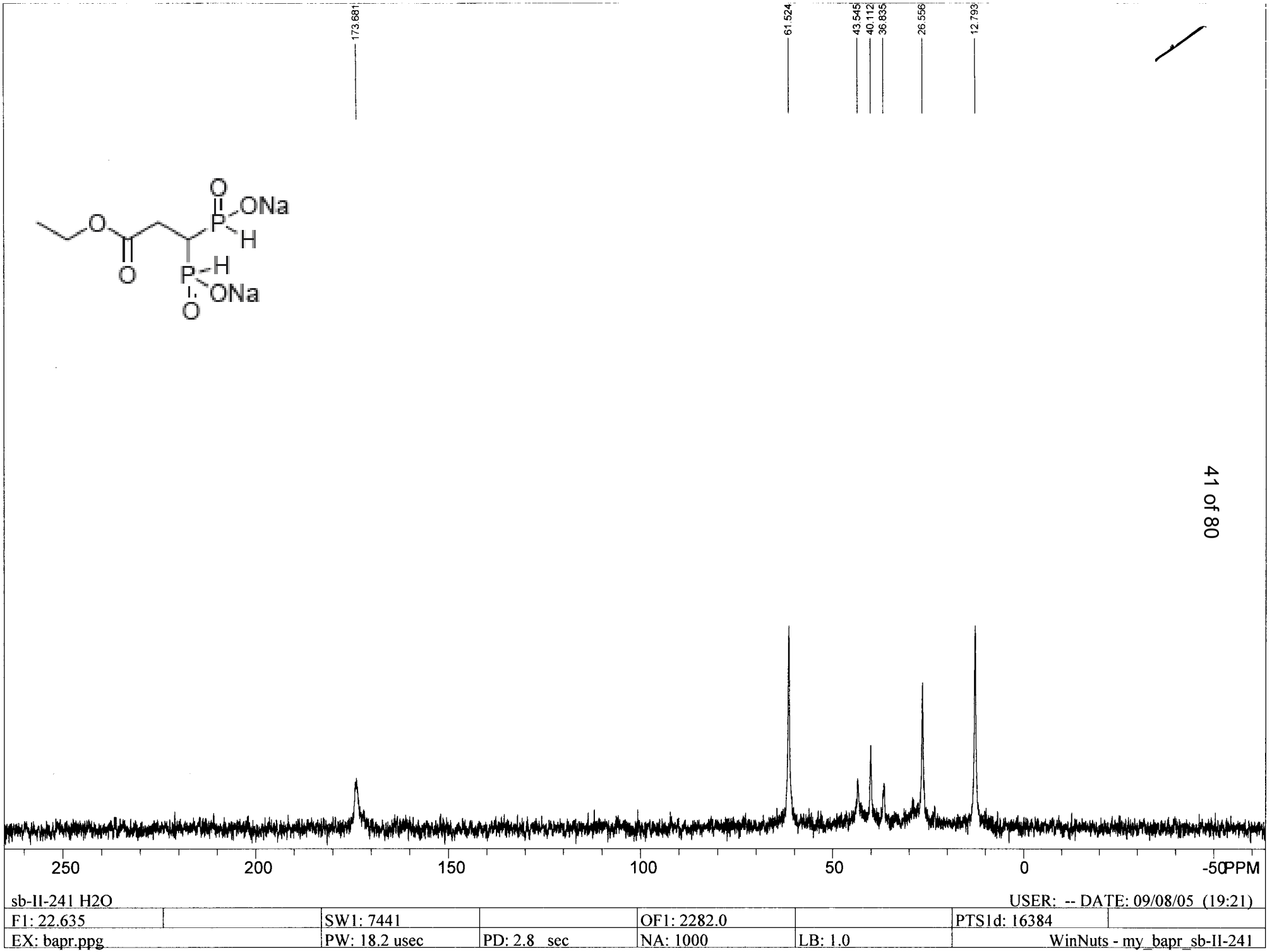


sb-II-241 pp

exp1 s2pul

SAMPLE		SPECIAL	
date	Jul 5 2005	temp	not used
solvent	D2O	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	4803.1	pw90	17.200
at	1.994	alfa	20.000
np	19158	**FLAGS**	
fb	not used	il	n
bs	16	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	16	**PROCESSING**	
ct	16	fn	not used
TRANSMITTER		**DISPLAY**	
tn	H1	sp	-60.0
sfrq	300.054	wp	3060.5
tof	300.9	rfl	601.2
tpwr	55	rfp	0
pw	8.600	rp	-106.9
DECOUPLER		lp	-83.9
dn	C13	**PLOT**	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	521
dpwr	45	th	38
dmf	13100	ai cdc ph	





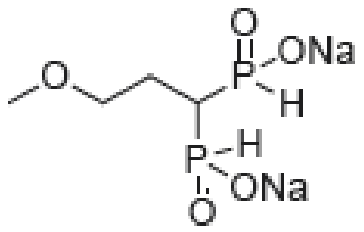
41 of 80

sb-II-241 H2O

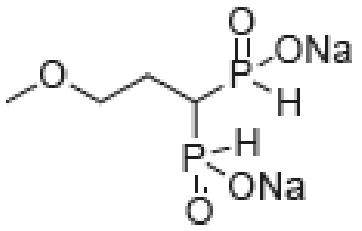
USER: -- DATE: 09/08/05 (19:21)

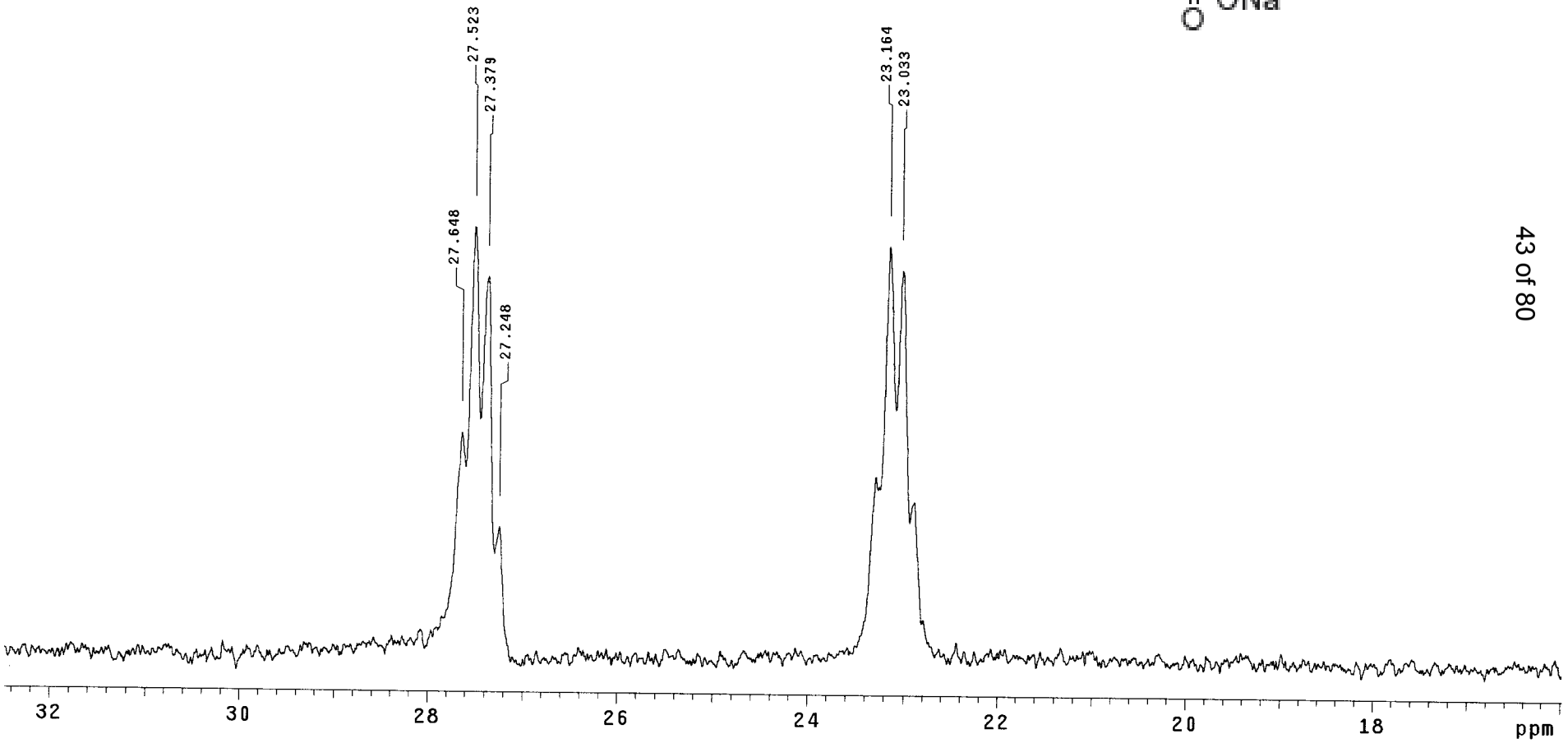
F1: 22.635		SW1: 7441		OF1: 2282.0		PTS1d: 16384	
EX: bapr.ppg		PW: 18.2 usec	PD: 2.8 sec	NA: 1000	LB: 1.0	WinNuts - my_bapr_sb-II-241	

INDEX	FREQUENCY	PPM	HEIGHT
1	3069.730	25.273	126.0



INDEX	FREQUENCY	PPM	HEIGHT
1	3358.178	27.648	36.8
2	3343.082	27.523	69.9
3	3325.539	27.379	61.9
4	3309.627	27.248	21.6
5	2813.512	23.164	67.3
6	2797.601	23.033	63.4

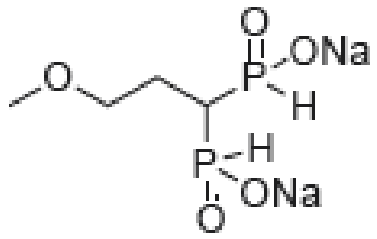




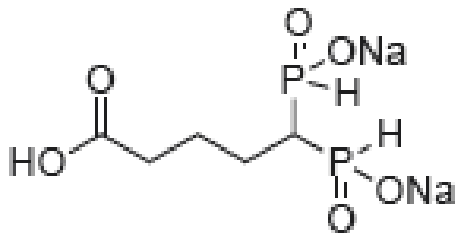
sb-II-242 pp

exp1 s2pul

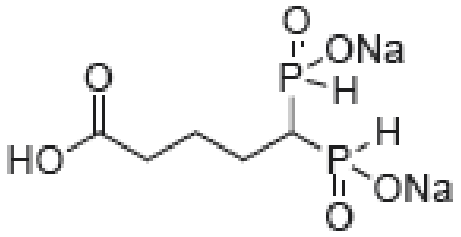
SAMPLE		SPECIAL	
date	Jul 5 2005	temp	not used
solvent	D2O	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	4803.1	pw90	17.200
at	1.994	alfa	20.000
np	19158	FLAGS	
fb	not used	il	n
bs	16	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	16	PROCESSING	
ct	16	fn	not used
TRANSMITTER		DISPLAY	
tn	H1	sp	-600.9
sfrq	300.054	wp	4802.8
tof	300.9	rfl	601.2
tpwr	55	rfp	0
pw	8.600	rp	-106.9
DECOUPLER		lp	-68.9
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	374
dpwr	45	th	54
dmf	13100	ai cdc ph	

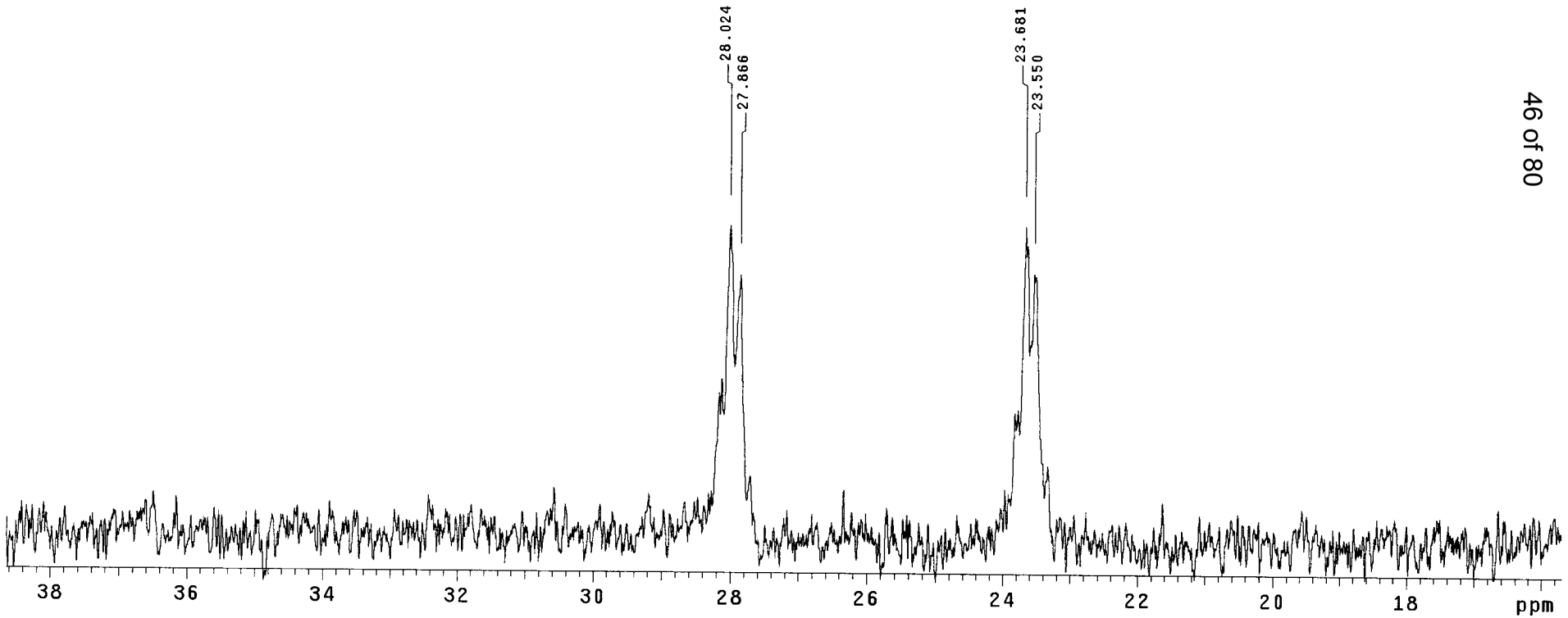


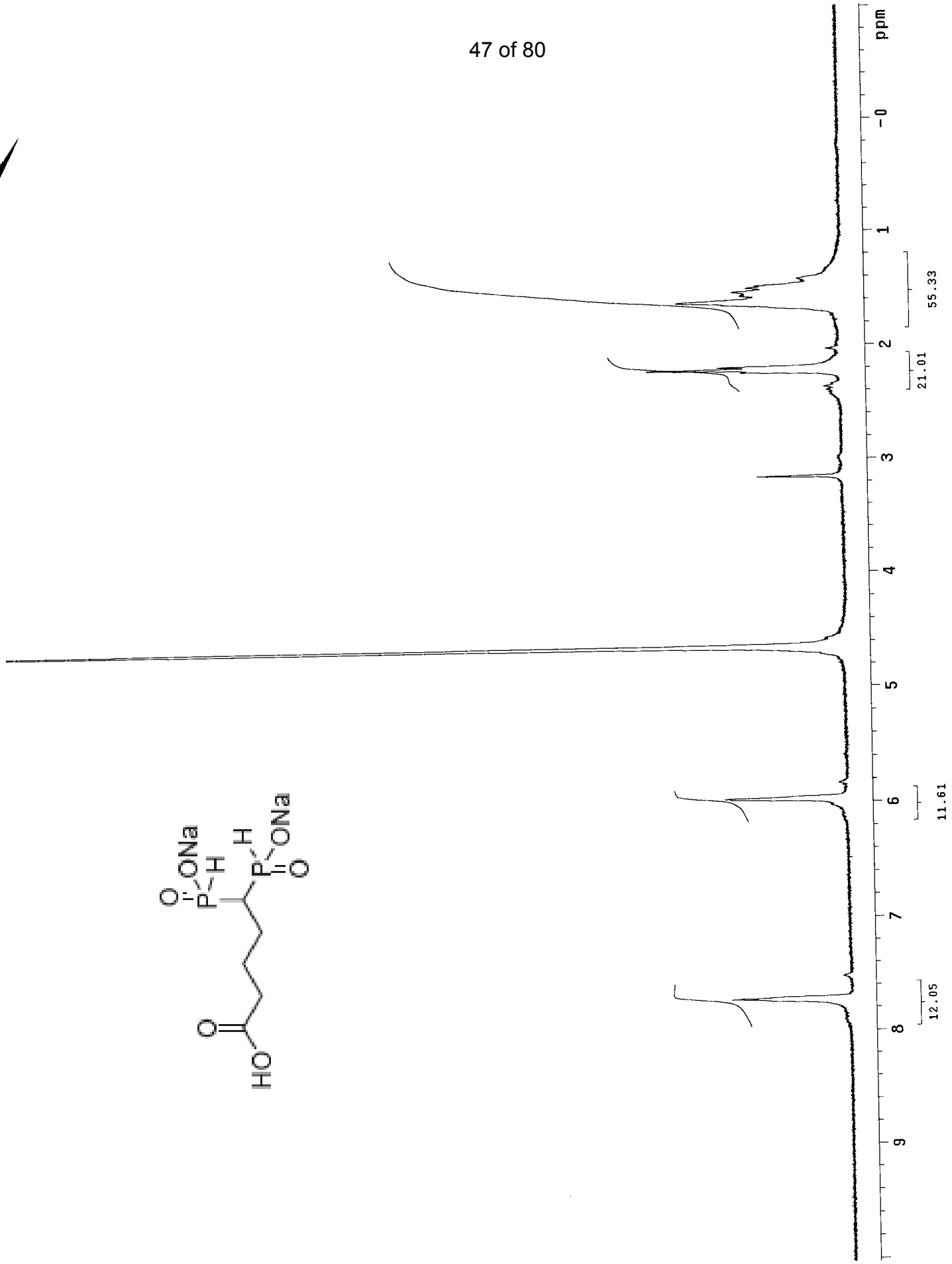
INDEX	FREQUENCY	PPM	HEIGHT
1	3130.112	25.770	126.0

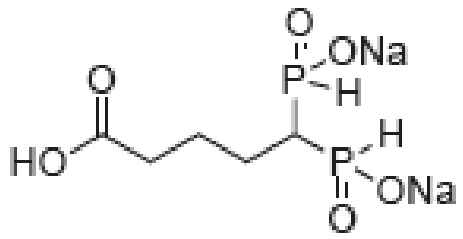


INDEX	FREQUENCY	PPM	HEIGHT
1	3403.873	28.024	51.0
2	3384.697	27.866	43.1
3	2876.343	23.681	51.1
4	2860.431	23.550	43.4






ppm
-0
1
2
3
4
5
6
7
8
9
55.33
21.01
11.61
12.05
O
ONa
P
H
O
P
H
ONa
O
HO



178.394

46.413
42.945
39.495
33.674
20.203

48 of 80

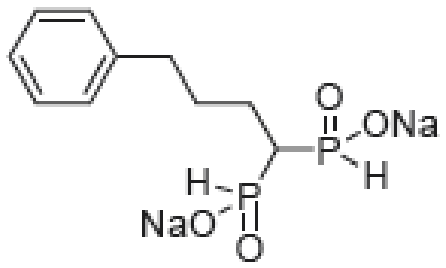
250 200 150 100 50 0 -50PPM

sb-II-249 (H_2O) Sodium

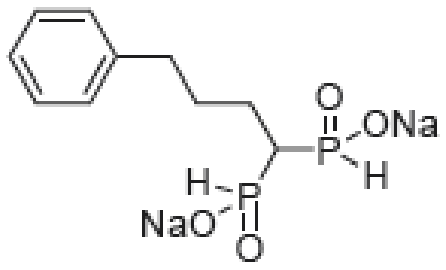
USER: -- DATE: 08/18/05 (20:02)

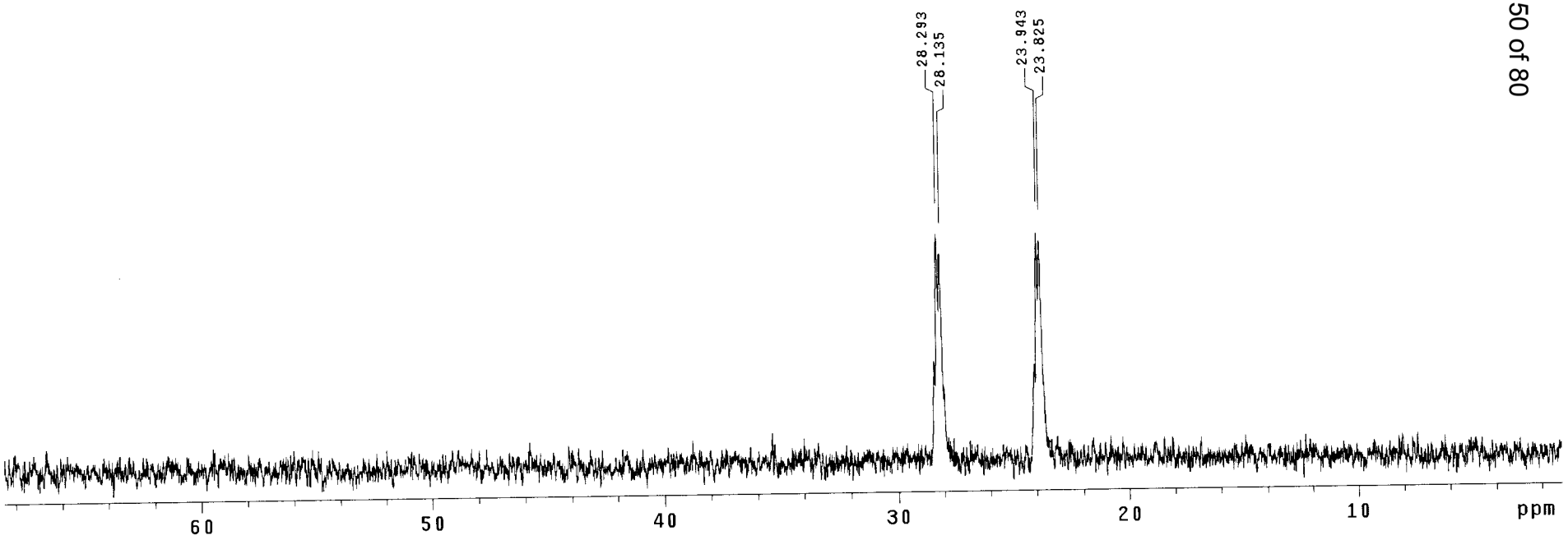
F1: 22.635	SW1: 7441		OF1: 2282.0		PTS1d: 16384
EX: bapr.ppg	PW: 18.2 usec	PD: 2.8 sec	NA: 1000	LB: 1.0	WinNuts - my_bapr_sbII-249

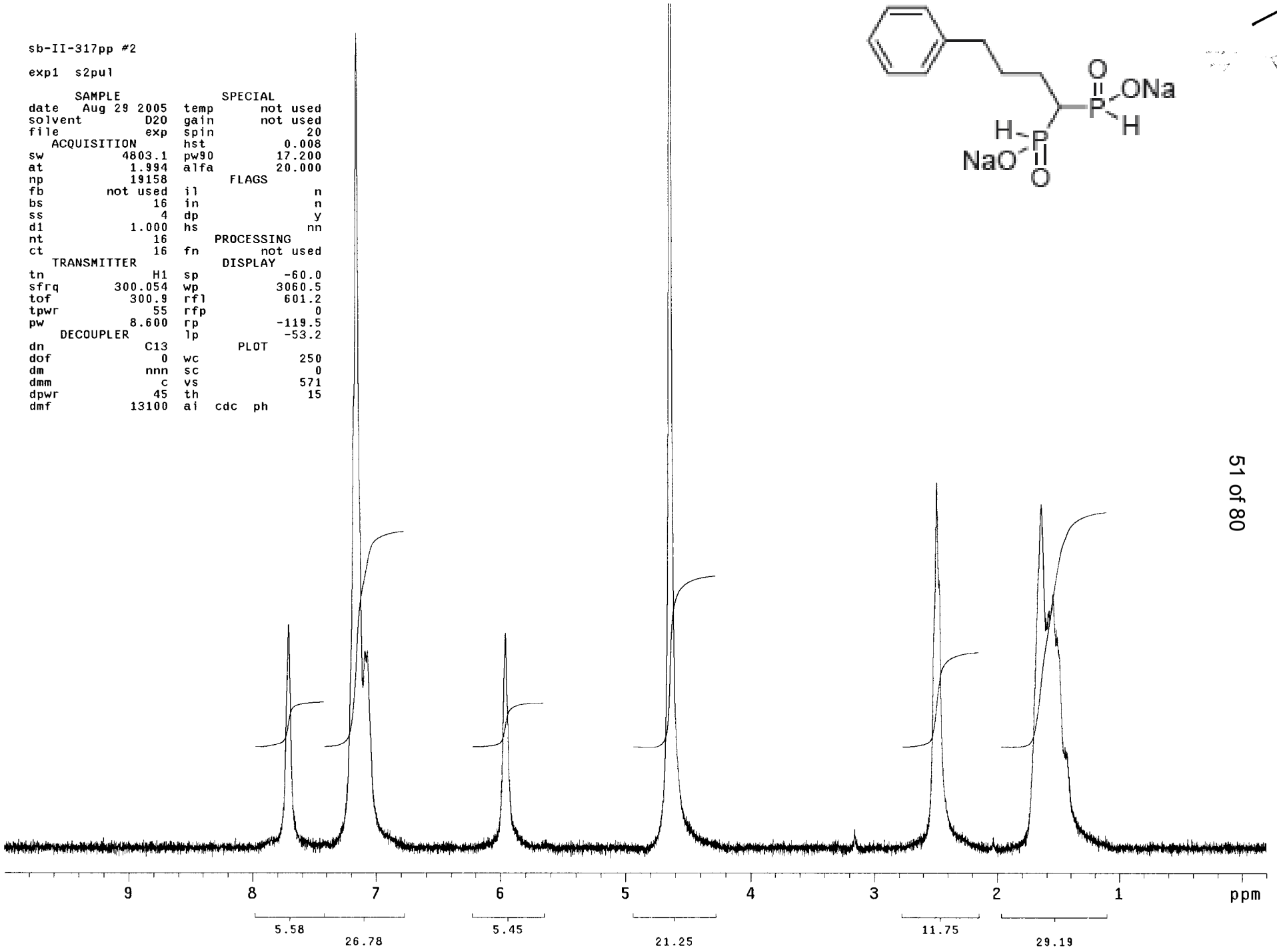
INDEX	FREQUENCY	PPM	HEIGHT
1	3164.383	26.052	126.0

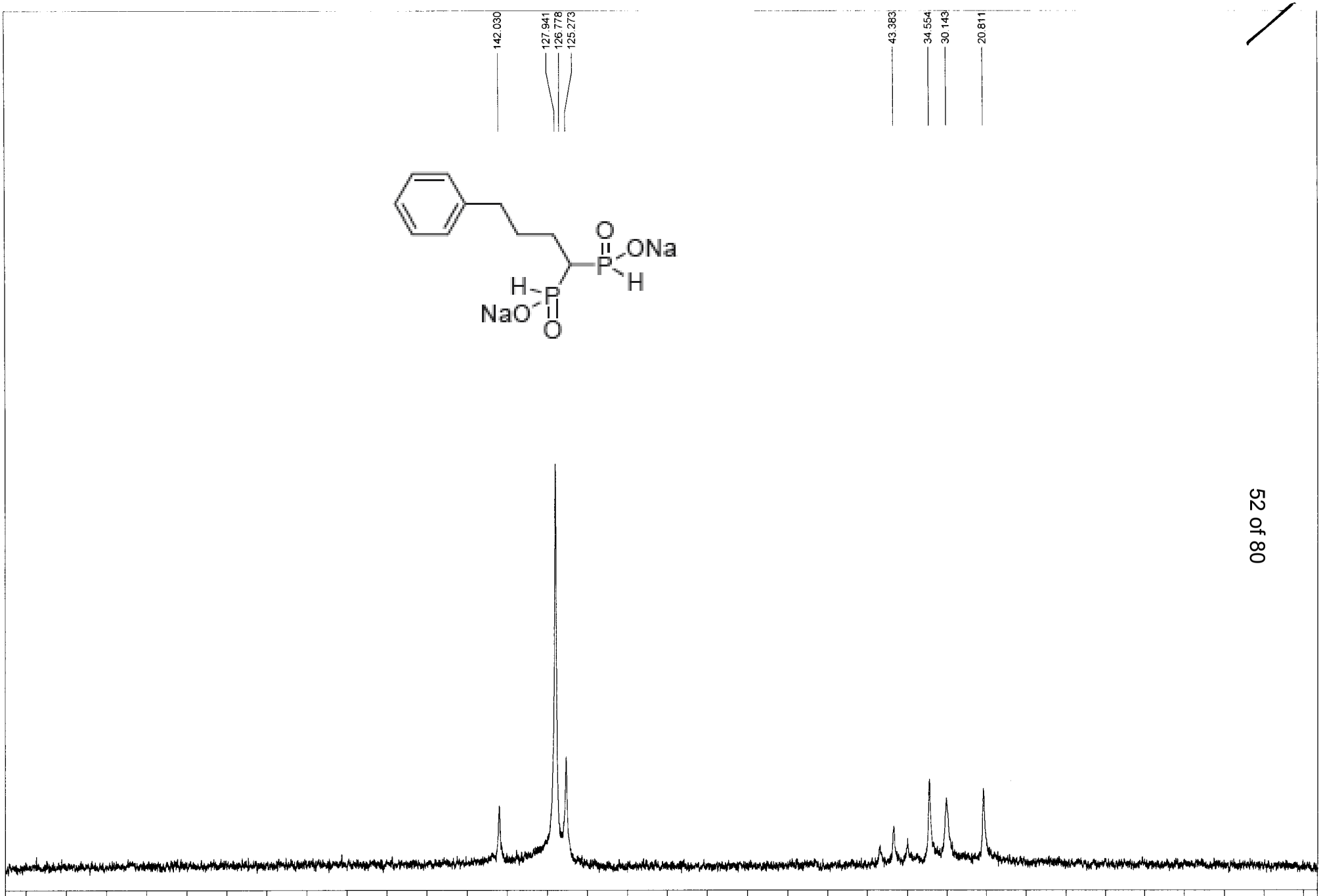


INDEX	FREQUENCY	PPM	HEIGHT
1	3436.512	28.293	36.4
2	3417.336	28.135	33.2
3	2908.166	23.943	36.4
4	2893.886	23.825	35.2





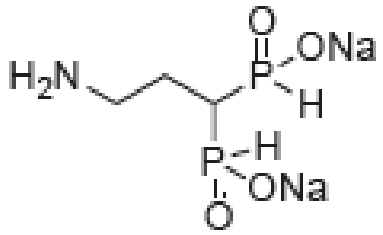

sb-II-317pp #2
exp1 s2pul
SAMPLE
date Aug 29 2005
solvent D2O
file exp
ACQUISITION
sw 4803.1
at 1.994
np 19158
fb not used
bs 16
ss 4
d1 1.000
nt 16
ct 16
TRANSMITTER
tn H1
sfrq 300.054
tof 300.9
tpwr 55
pw 8.600
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 45
dmf 13100
SPECIAL
temp not used
gain not used
spin 20
hst 0.008
pw90 17.200
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -60.0
wp 3060.5
rfl 601.2
rfp 0
rp -119.5
lp -53.2
PLOT
wc 250
sc 0
vs 571
th 15
ai cdc ph
ONa
NaO
9
8
7
6
5
4
3
2
1
ppm
5.58
26.78
5.45
21.25
11.75
29.19



sb-I-101 13C					USER: -- DATE: 07/06/05 (19:02)
F1: 22.635	SW1: 7441		OF1: 2282.0		PTS1d: 16384
EX: bapr.ppg	PW: 18.2 usec	PD: 2.8 sec	NA: 2000	LB: 1.0	WinNuts - my_bapr_sb101

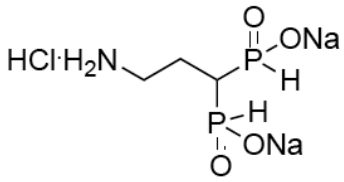
SB-I-102 pp

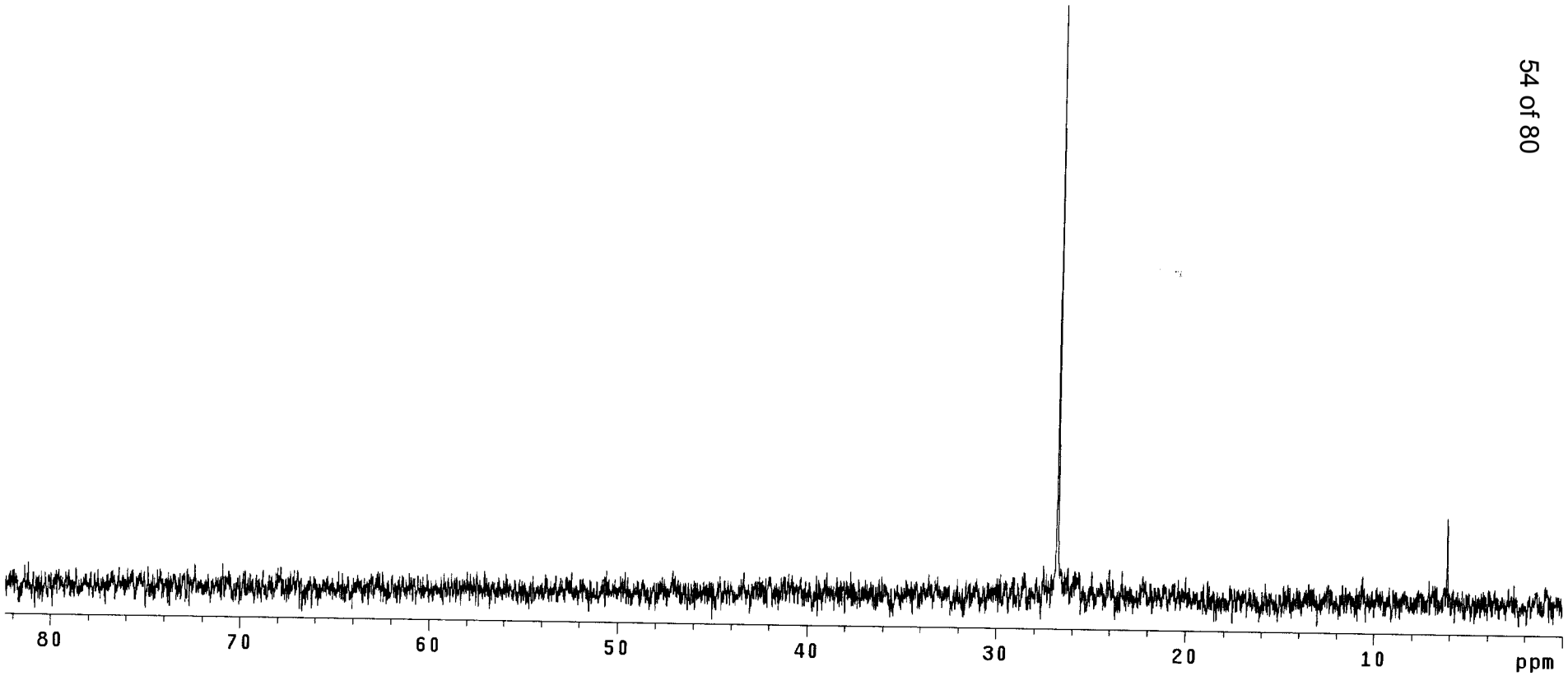
INDEX	FREQUENCY	PPM	HEIGHT
1	2330.347	7.766	15.5
2	1797.975	5.992	16.6
3	1394.299	4.647	219.3
4	904.434	3.014	15.7
5	897.692	2.992	24.5
6	890.363	2.967	16.2
7	571.702	1.905	10.0
8	564.373	1.881	12.7
9	557.630	1.858	12.8
10	550.301	1.834	10.7
11	490.497	1.635	4.5
12	471.735	1.572	6.8
13	452.973	1.510	4.6



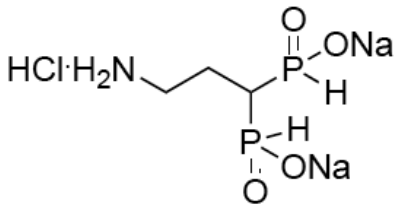
exp1 s2pul

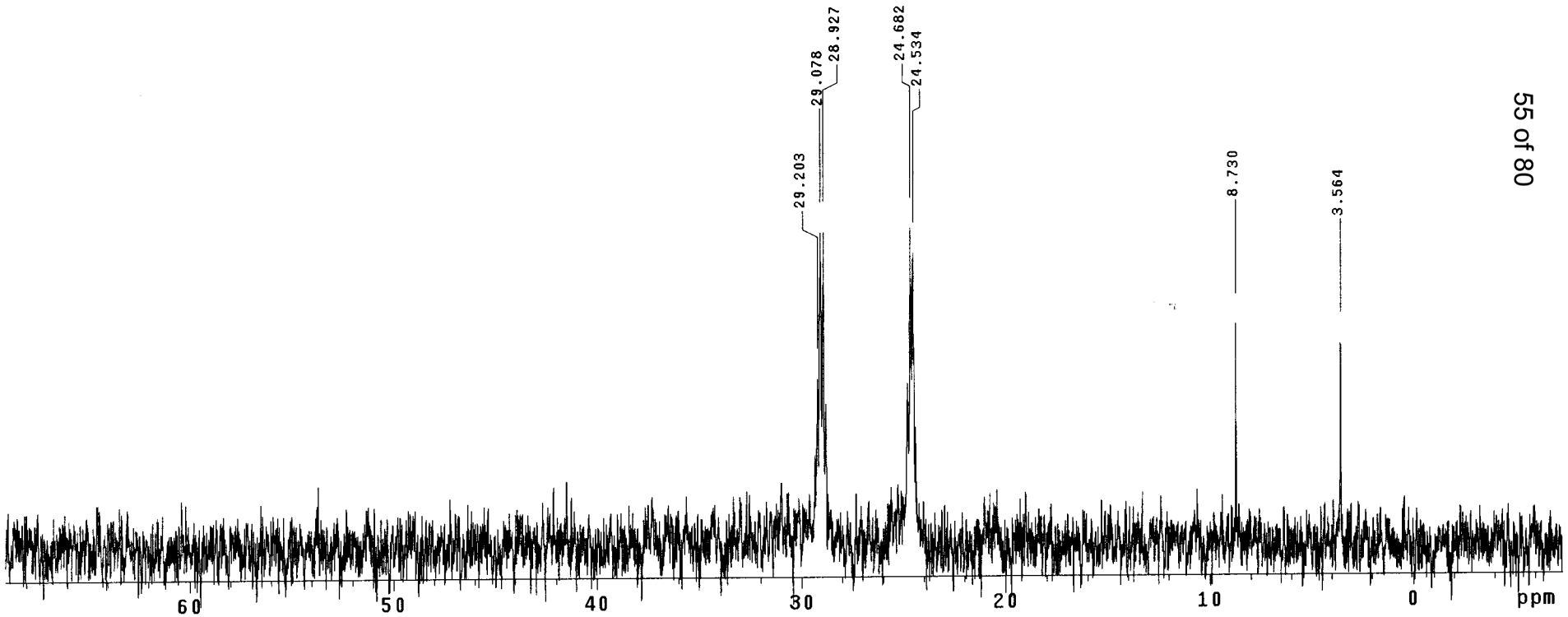
SAMPLE		SPECIAL	
date	Sep 26 2005	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	**FLAGS**	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	16	**PROCESSING**	
ct	16	lb	2.00
TRANSMITTER		fn	not used
tn	P31	**DISPLAY**	
sfrq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rfp	0
DECOUPLER		rp	-102.7
dn	H1	lp	-288.8
dof	0	**PLOT**	
dm	yyy	wc	250
dmm	w	sc	0
dpwr	35	vs	43
dmf	6700	th	17
		ai no ph	

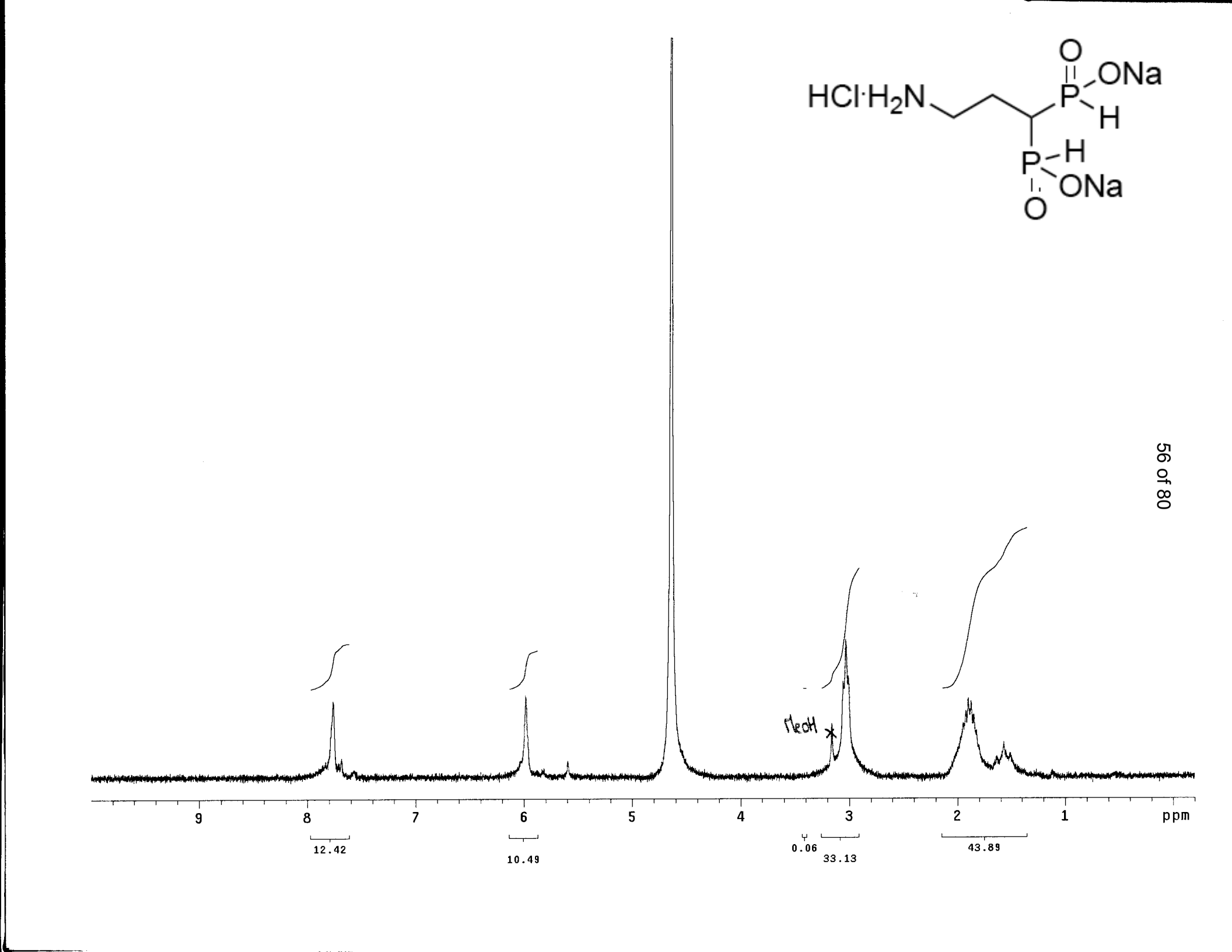




INDEX	FREQUENCY	PPM	HEIGHT
1	3547.046	29.203	26.8
2	3531.950	29.078	50.4
3	3513.591	28.927	50.5
4	2997.893	24.682	51.0
5	2979.941	24.534	47.0
6	1060.353	8.730	35.3
7	432.866	3.564	32.1



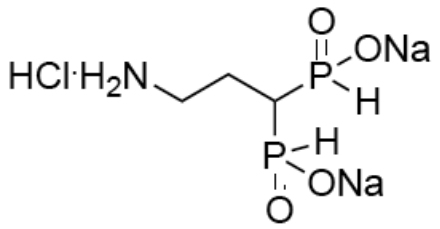



HCl·H2N
O
ONa
P
H
P
H
ONa
O
MeOH
9
8
7
6
5
4
3
2
1
ppm
12.42
10.49
0.06
33.13
43.89

sb-II-333

exp1 s2pul

SAMPLE		SPECIAL	
date	Sep 26 2005	temp	not used
solvent	D20	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	4803.1	pw90	17.200
at	1.994	alfa	20.000
np	19158	FLAGS	
fb	not used	il	n
bs	16	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	16	PROCESSING	
ct	16	fn	not used
TRANSMITTER		DISPLAY	
tn	H1	sp	-60.0
sfrq	300.054	wp	3060.5
tof	300.9	rfl	601.2
tpwr	55	rfp	0
pw	8.600	rp	-101.5
DECOUPLER		lp	-90.6
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	947
dpwr	45	th	4
dmf	13100	ai cdc ph	



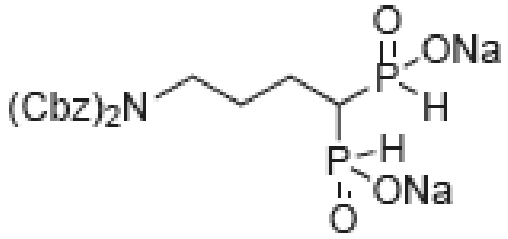
9 8 7 6 5 4 3 2 1 ppm

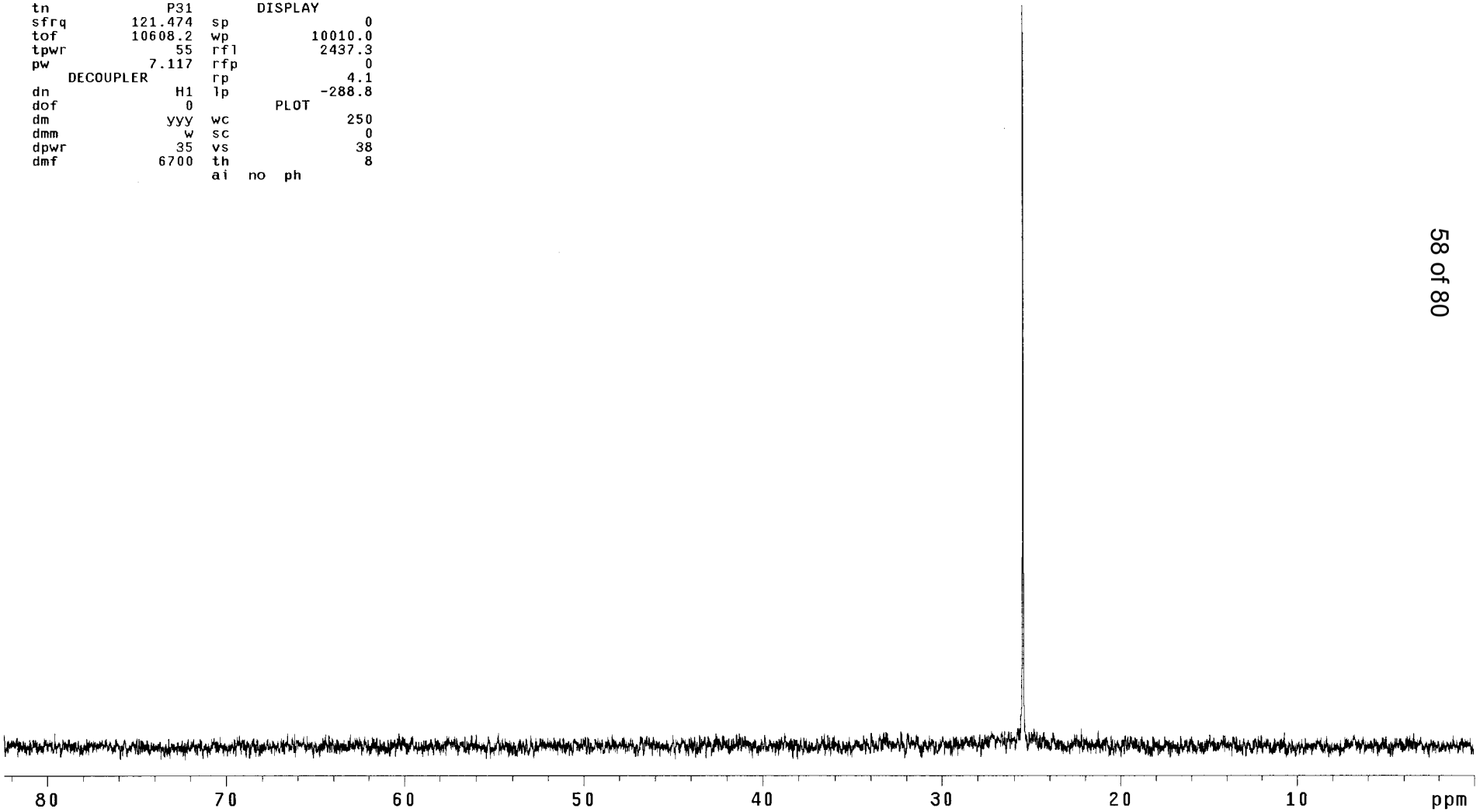
6.47 6.01 0.84 47.15 18.14 16.88 4.51

sb-I-79pp

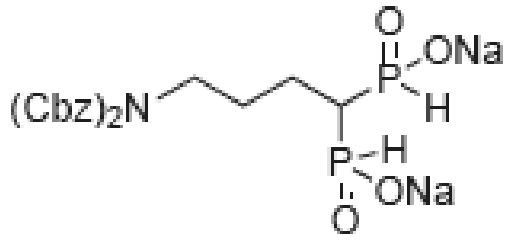
exp1 s2pul

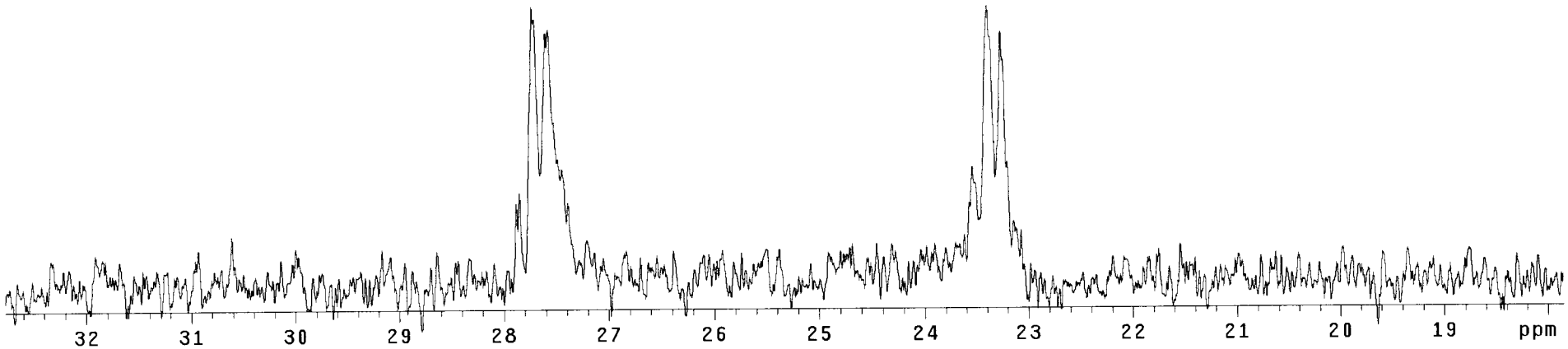
SAMPLE		SPECIAL	
date	Feb 9 2005	temp	not used
solvent	D2O	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	FLAGS	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	32	PROCESSING	
ct	32	lb	2.00
TRANSMITTER		fn	not used
tn	P31	DISPLAY	
sfrq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rfp	0
DECOUPLER		rp	4.1
dn	H1	lp	-288.8
dof	0	PLOT	
dm	yyy	wc	250
dmm	w	sc	0
dpwr	35	vs	38
dmf	6700	th	8
		ai no ph	



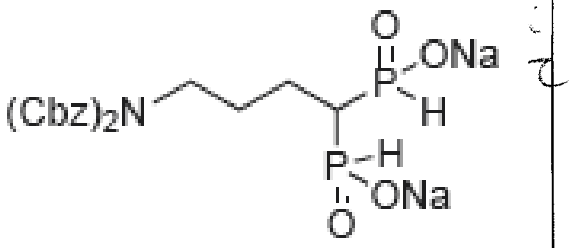


INDEX	FREQUENCY	PPM	HEIGHT
1	3382.249	27.846	13.7
2	3367.562	27.725	43.6
3	3348.794	27.570	39.9
4	2841.256	23.392	43.4
5	2825.344	23.261	39.4





SB-I-79 pp

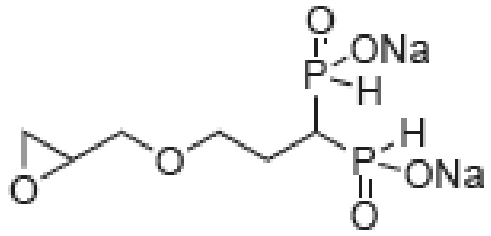


9 8 7 6 5 4 3 2 1 -0 ppm

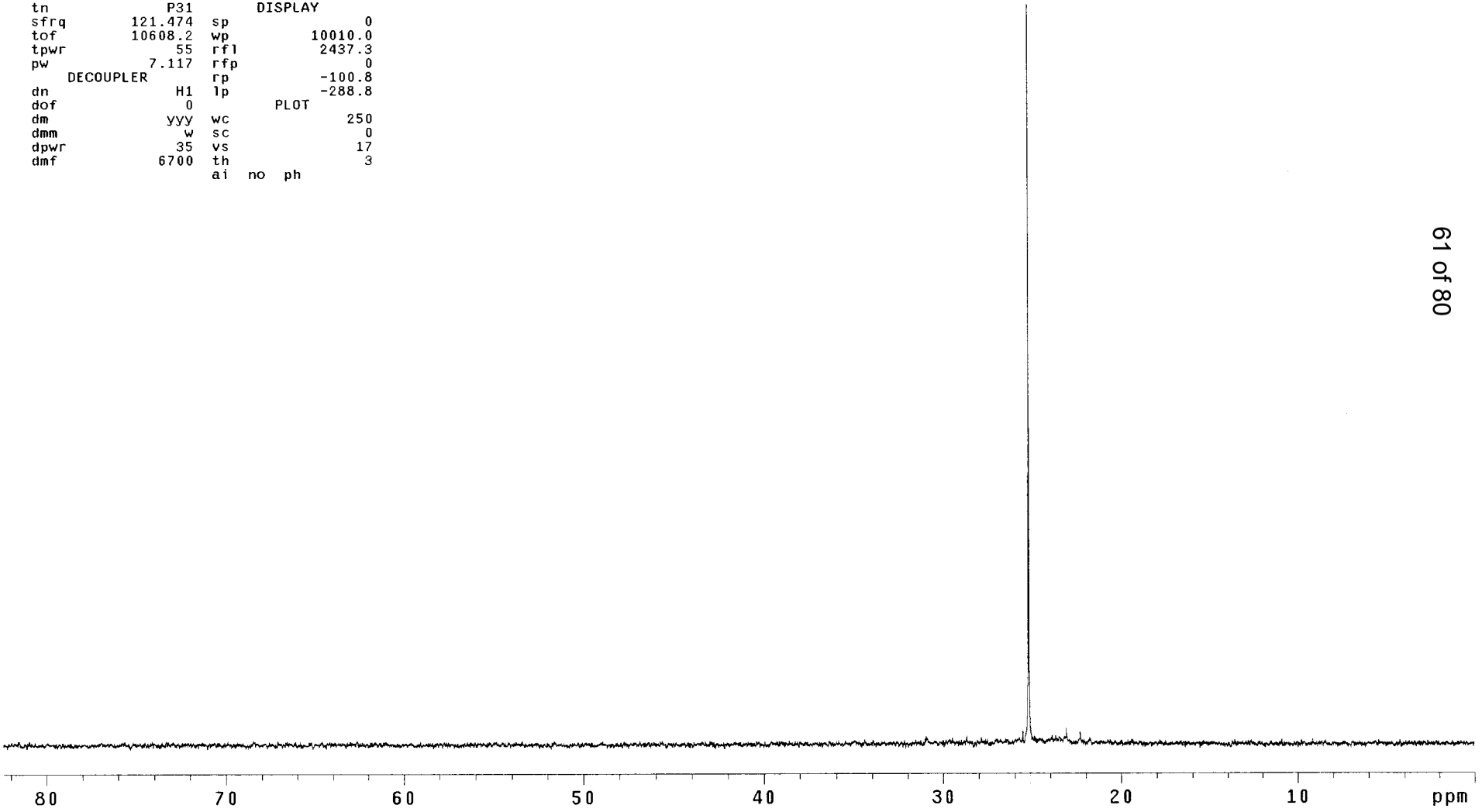
SB-I-04 31P

exp1 s2pul

SAMPLE		SPECIAL	
date	Dec 1 2004	temp	not used
solvent	D2O	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	FLAGS	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	64	PROCESSING	
ct	64	lb	2.00
TRANSMITTER		fn	not used
tn	P31	DISPLAY	
sfrq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rfp	0
DECOUPLER		rp	-100.8
dn	H1	lp	-288.8
dof	0	PLOT	
dm	yyy	wc	250
dmm	w	sc	0
dpwr	35	vs	17
dmf	6700	th	3
		ai no ph	



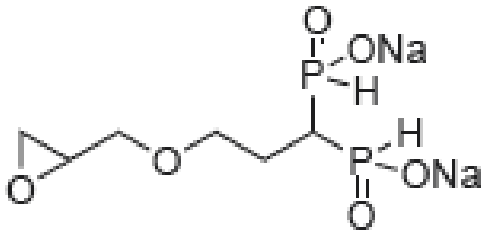
8.46

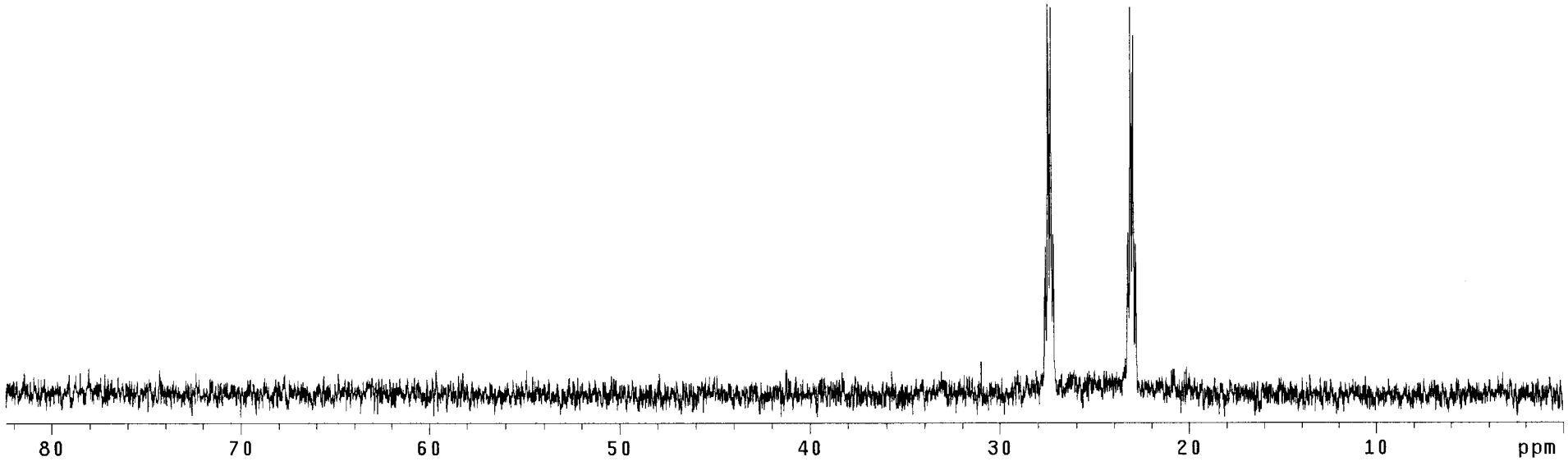


SB-I-04 31P

exp1 s2pul

SAMPLE		SPECIAL	
date	Dec 1 2004	temp	not used
solvent	D2O	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	**FLAGS**	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	64	**PROCESSING**	
ct	64	lb	2.00
TRANSMITTER		fn	not used
tn	P31	**DISPLAY**	
sfrq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rfp	0
DECOUPLER		rp	-27.4
dn	H1	lp	-589.0
dof	0	**PLOT**	
dm	ynn	wc	250
dmm	w	sc	0
dpwr	35	vs	83
dmf	6700	th	11
		ai no ph	

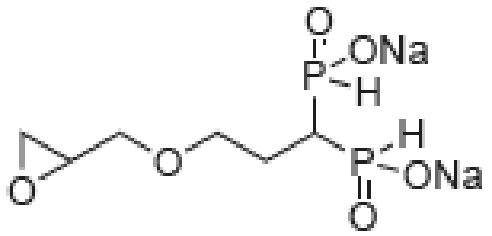




sb-I-04

exp1 s2pul

SAMPLE		SPECIAL	
date	Dec 1 2004	temp	not used
solvent	D2O	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	4803.1	pw90	17.200
at	1.994	alfa	20.000
np	19158	FLAGS	
fb	not used	il	n
bs	16	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	16	PROCESSING	
ct	16	fn	not used
TRANSMITTER		DISPLAY	
tn	H1	sp	-600.9
sfrq	300.054	wp	4802.8
tof	300.9	rfl	601.2
tpwr	55	rfp	0
pw	8.600	rp	-116.7
DECOUPLER		lp	-42.9
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	509
dpwr	45	th	7
dmf	13100	ai cdc ph	



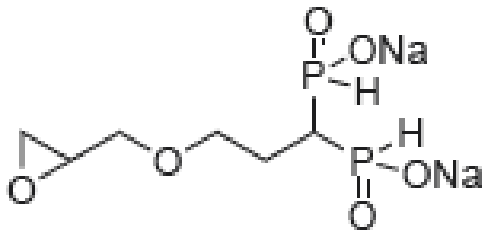
12 10 8 6 4 2 -0 ppm

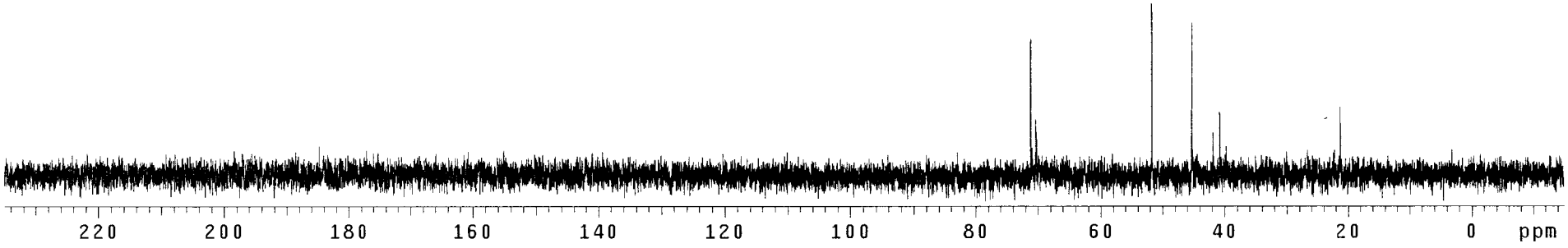
6.84 6.55 16.32 21.94 13.86 13.50 0.36 20.07 0.57

sb-I-04

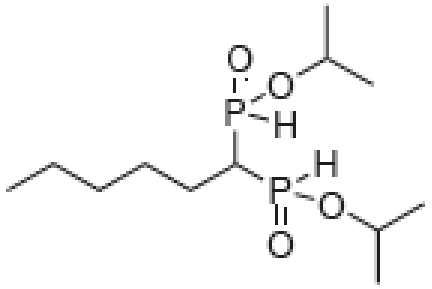
exp1 s2pul

SAMPLE		SPECIAL	
date	Dec 22 2004	temp	not used
solvent	D2O	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	18867.9	pw90	18.500
at	1.815	alfa	20.000
np	68492	FLAGS	
fb	10400	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	2000	PROCESSING	
ct	2000	lb	1.00
TRANSMITTER		fn	not used
tn	C13	DISPLAY	
sfrq	75.456	sp	-1134.4
tof	737.2	wp	18867.6
tpwr	58	rfl	1134.7
pw	9.250	rfp	0
DECOUPLER		rp	-175.0
dn	H1	lp	-456.3
dof	0	PLOT	
dm	yyy	wc	250
dmm	w	sc	0
dpwr	35	vs	372
dmf	6700	th	12
		ai no ph	



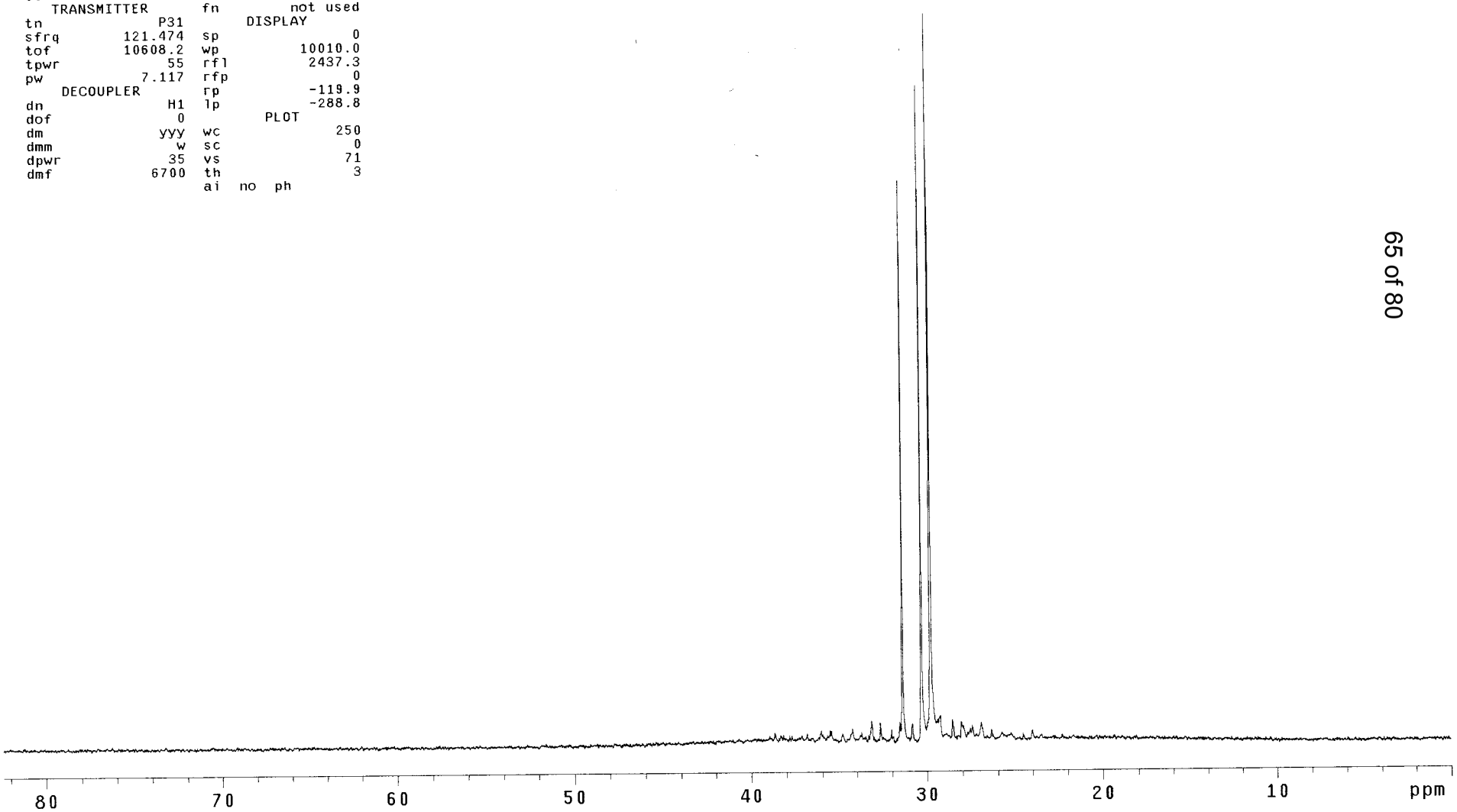


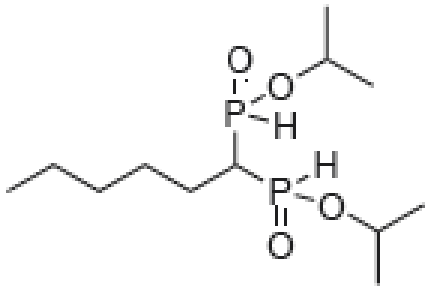
SB-I-161

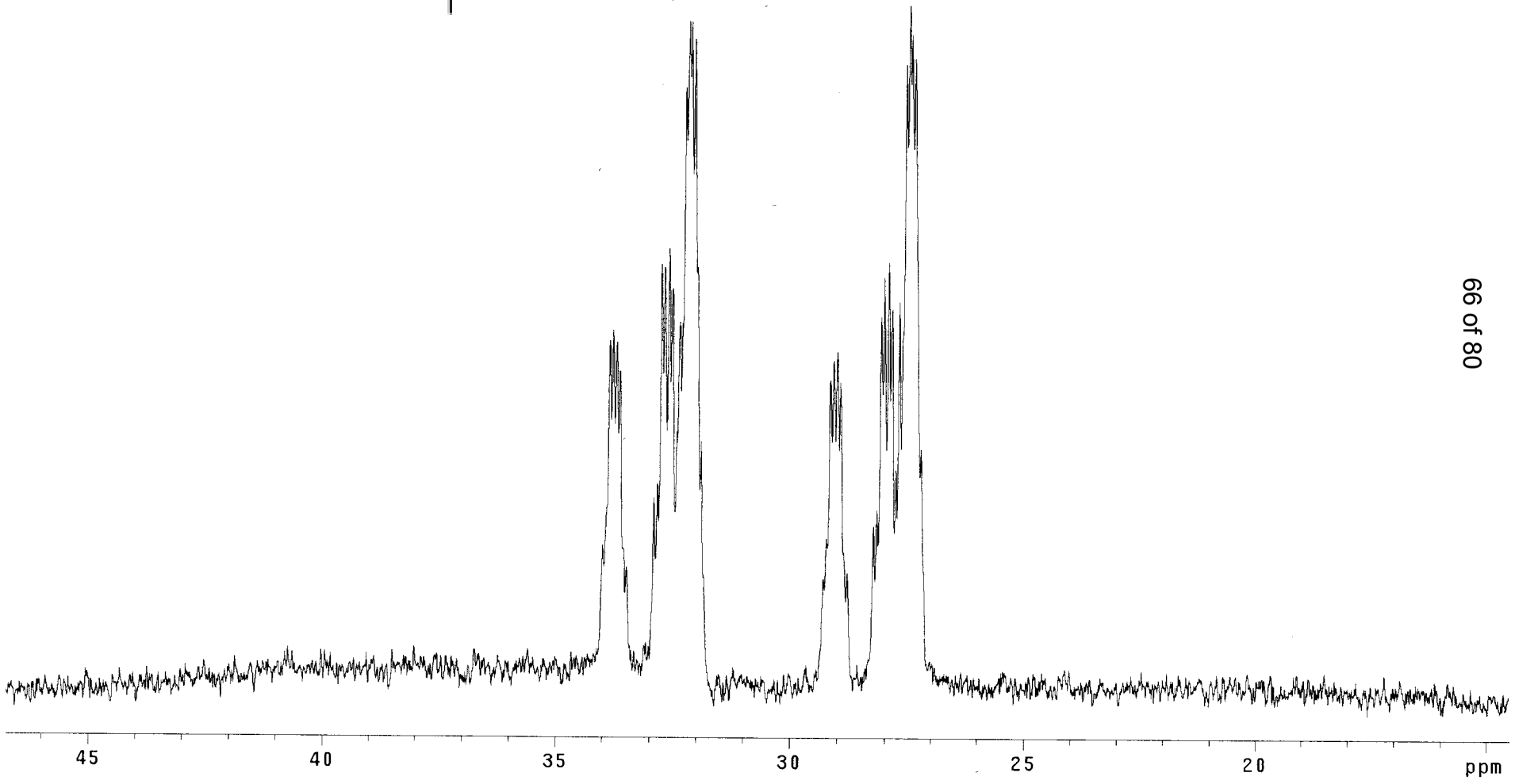


exp1 s2pul

SAMPLE		SPECIAL	
date	Apr 15 2005	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	FLAGS	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	16	PROCESSING	
ct	16	lb	2.00
TRANSMITTER		fn	not used
tn	P31	DISPLAY	
sfrq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rfp	0
DECOUPLER		rp	-119.9
dn	H1	lp	-288.8
dof	0	PLOT	
dm	yyy	wc	250
dmm	w	sc	0
dpwr	35	vs	71
dmf	6700	th	3
		ai no ph	



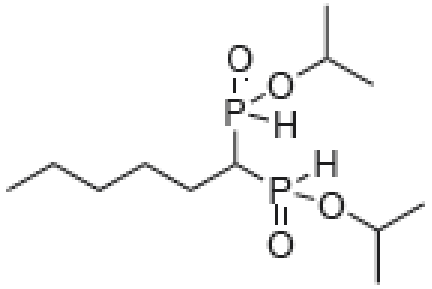



45
40
35
30
25
20
ppm

sb-I-161 chromato purge

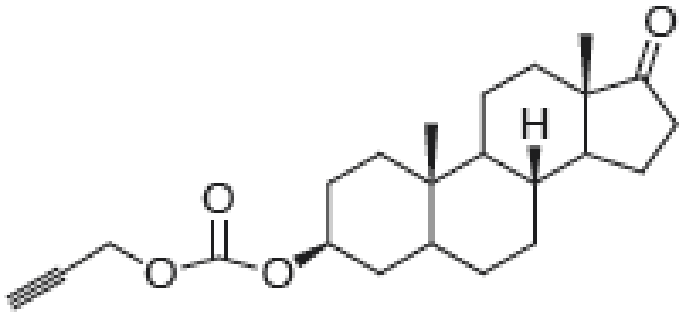
exp1 s2pul

SAMPLE		SPECIAL	
date	Apr 15 2005	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	4803.1	pw90	17.200
at	1.994	alfa	20.000
np	19158	FLAGS	
fb	not used	il	n
bs	16	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	16	PROCESSING	
ct	16	fn	not used
TRANSMITTER		DISPLAY	
tn	H1	sp	-581.0
sfrq	300.053	wp	4802.8
tof	300.9	rfl	581.3
tpwr	55	rfp	0
pw	8.600	rp	-127.4
DECOUPLER		lp	-33.6
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	687
dpwr	45	th	2
dmf	13100	ai cdc ph	



exp1 s2pul

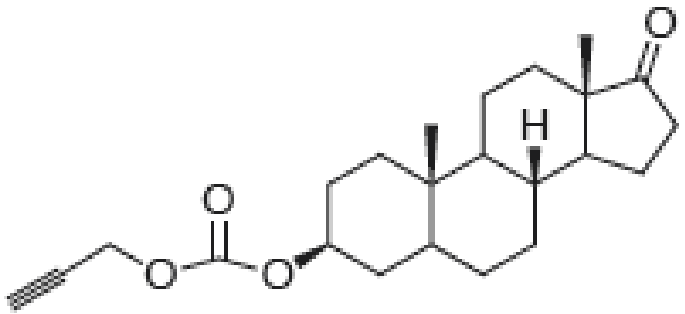
SAMPLE		SPECIAL	
date	Aug 31 2005	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	4803.1	pw90	17.200
at	1.994	alfa	20.000
np	19158	**FLAGS**	
fb	not used	il	n
bs	16	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	16	**PROCESSING**	
ct	16	fn	not used
TRANSMITTER		**DISPLAY**	
tn	H1	sp	-60.0
sfrq	300.053	wp	3060.5
tof	300.9	rfl	598.9
tpwr	55	rfp	0
pw	8.600	rp	-107.2
DECOUPLER		lp	-73.0
dn	C13	**PLOT**	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	173
dpwr	45	th	3
dmf	13100	ai cdc ph	



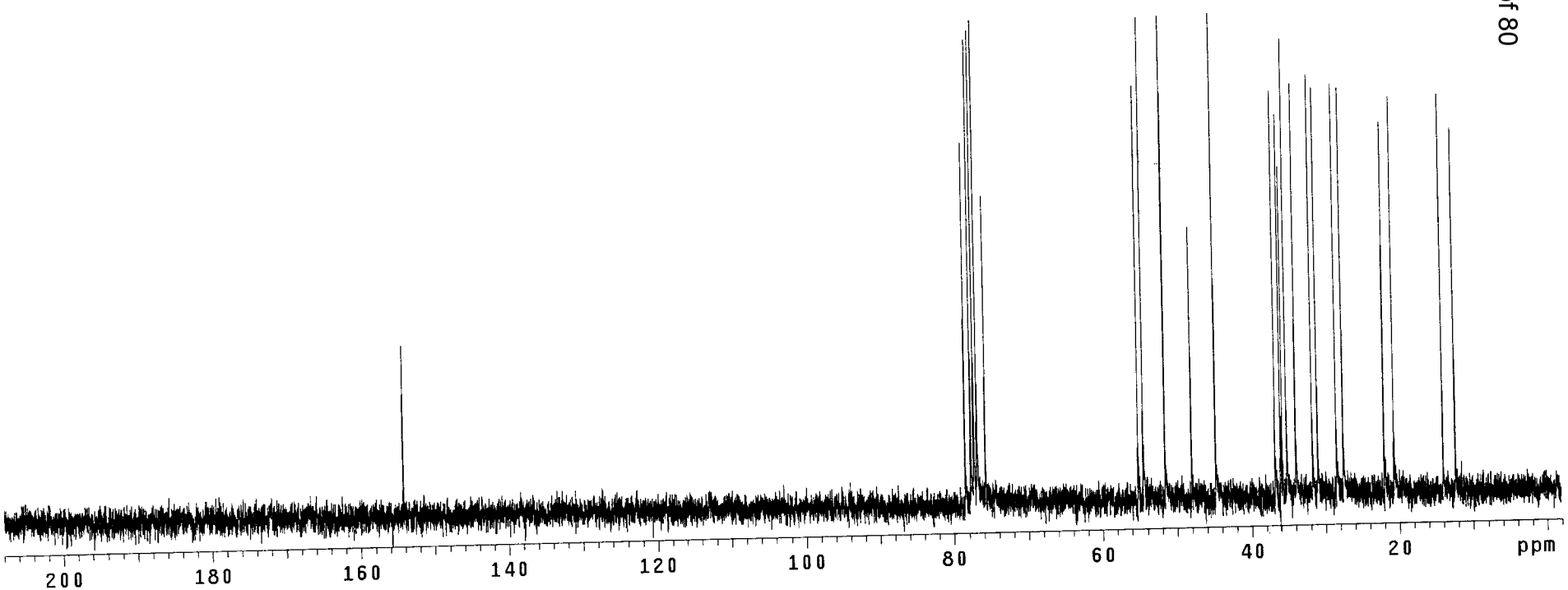
1

exp1 s2pul

SAMPLE		SPECIAL	
date	Oct 17 2005	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	18867.9	pw90	18.500
at	1.815	alfa	20.000
np	68492	**FLAGS**	
fb	10400	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	512	**PROCESSING**	
ct	512	lb	1.00
TRANSMITTER		fn	not used
tn	C13	**DISPLAY**	
sfrq	75.456	sp	-150.9
tof	737.2	wp	15844.1
tpwr	58	rfl	1134.7
pw	9.250	rfp	0
DECOUPLER		rp	46.3
dn	H1	lp	-203.8
dof	0	**PLOT**	
dm	yyy	wc	250
dmm	w	sc	0
dpwr	35	vs	213
dmf	6700	th	13
		ai	no ph



1



INDEX	FREQUENCY	PPM	HEIGHT
1	3052.536	25.131	126.0

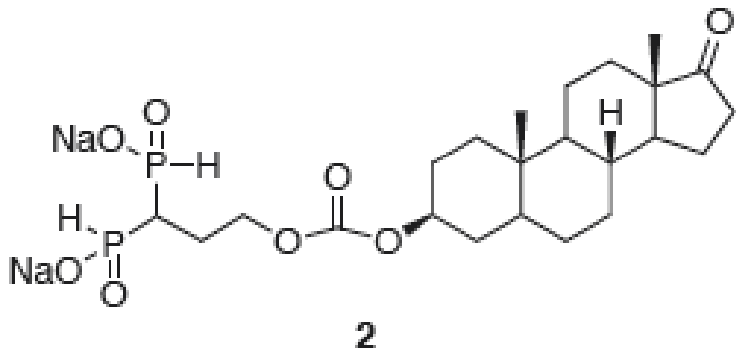
SD IV-157 decoupled

Archive directory: /export/home
Sample directory:
File: PHOSPHORUS

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercuryplus300"

Relax. delay 1.000 sec
Pulse 35.0 degrees
Acq. time 1.597 sec
Width 4863.8 Hz
16 repetitions
OBSERVE P31, 121.4670933 MHz
DECOUPLE H1, 300.0632664 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 16384
Total time 0 min, 58 sec



2

INDEX	FREQUENCY	PPM	HEIGHT
1	3328.059	27.399	30.9
2	3309.651	27.247	25.5
3	2797.202	23.028	27.4
4	2779.388	22.882	24.0

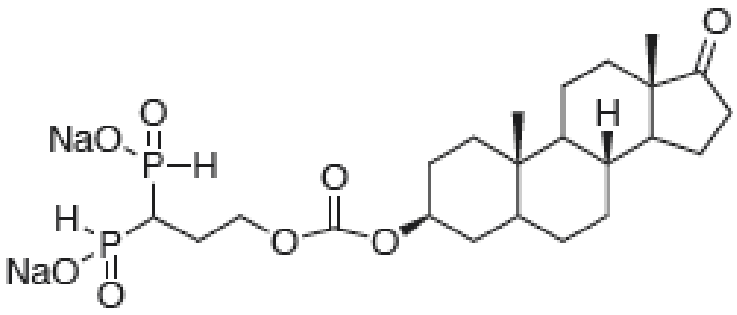
SD IV-157 coupled

Archive directory: /export/home
Sample directory:
File: PHOSPHORUS

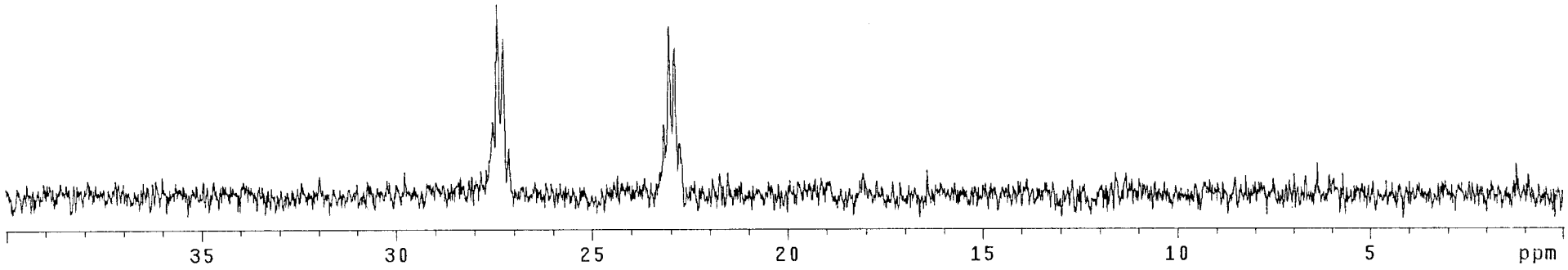
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercuryplus300"

Relax. delay 1.000 sec
Pulse 35.0 degrees
Acq. time 1.597 sec
Width 4863.8 Hz
16 repetitions
OBSERVE P31, 121.4670933 MHz
DECOUPLE H1, 300.0632664 MHz
Power 35 dB
off during acquisition
on during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 16384
Total time 0 min, 58 sec



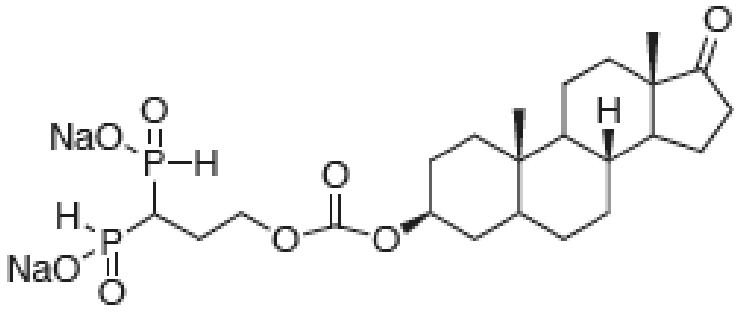
2



SD IV-157

Archive directory: /export/home/TCUuser/vnmrsys/data
Sample directory:
File: PROTON

Pulse Sequence: s2pul

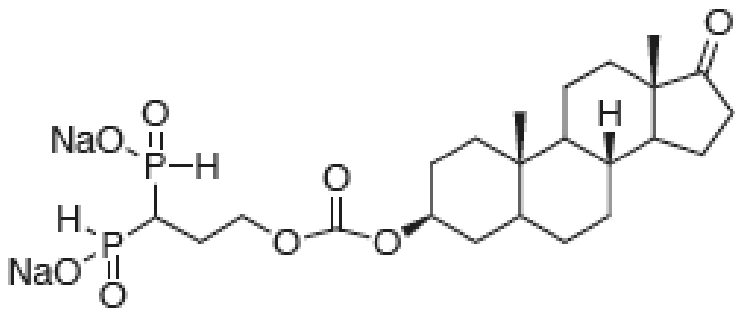


2

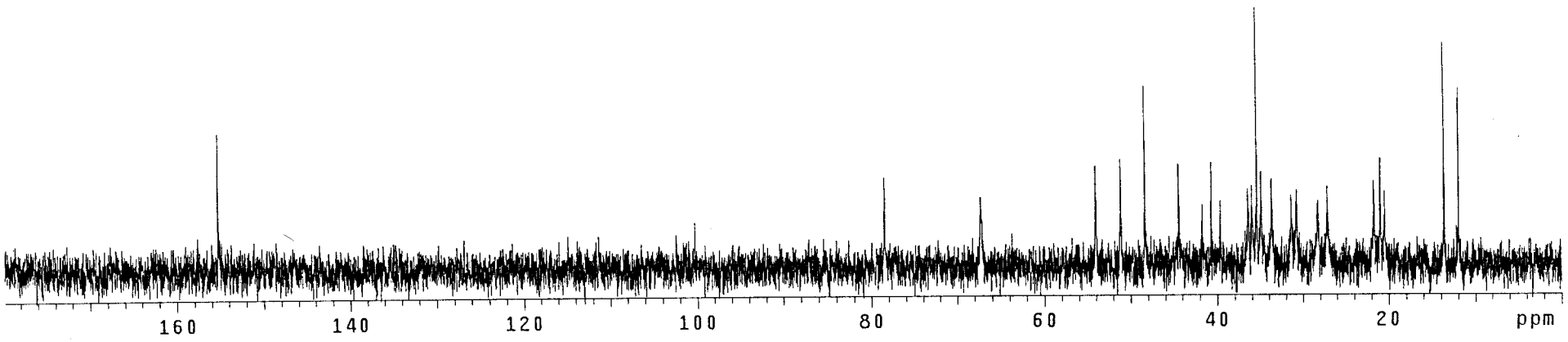
8 7 6 5 4 3 2 1 ppm

1.98 1.94 18.83 2.49 4.41 1.80 68.55

INDEX	FREQUENCY	PPM	HEIGHT
1	11707.561	155.168	21.7
2	5918.580	78.443	14.1
3	5075.469	67.269	10.8
4	4080.913	54.087	15.8
5	3860.473	51.165	16.9
6	3649.481	48.369	28.5
7	3353.748	44.449	16.1
8	3149.053	41.737	9.5
9	3071.470	40.708	16.2
10	2993.314	39.672	10.2
11	2754.266	36.504	12.0
12	2715.331	35.988	12.5
13	2674.106	35.442	40.9
14	2635.457	34.929	14.8
15	2542.987	33.704	13.6
16	2368.925	31.397	10.9
17	2321.974	30.775	11.8
18	2130.449	28.236	10.2
19	2049.717	27.166	12.4
20	1641.760	21.759	13.2
21	1585.934	21.019	16.8
22	1546.713	20.500	11.6
23	1031.399	13.670	35.2
24	904.288	11.985	28.0

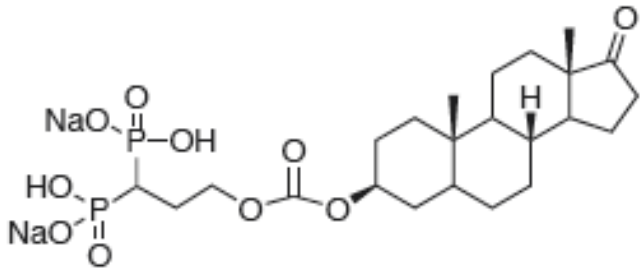


2

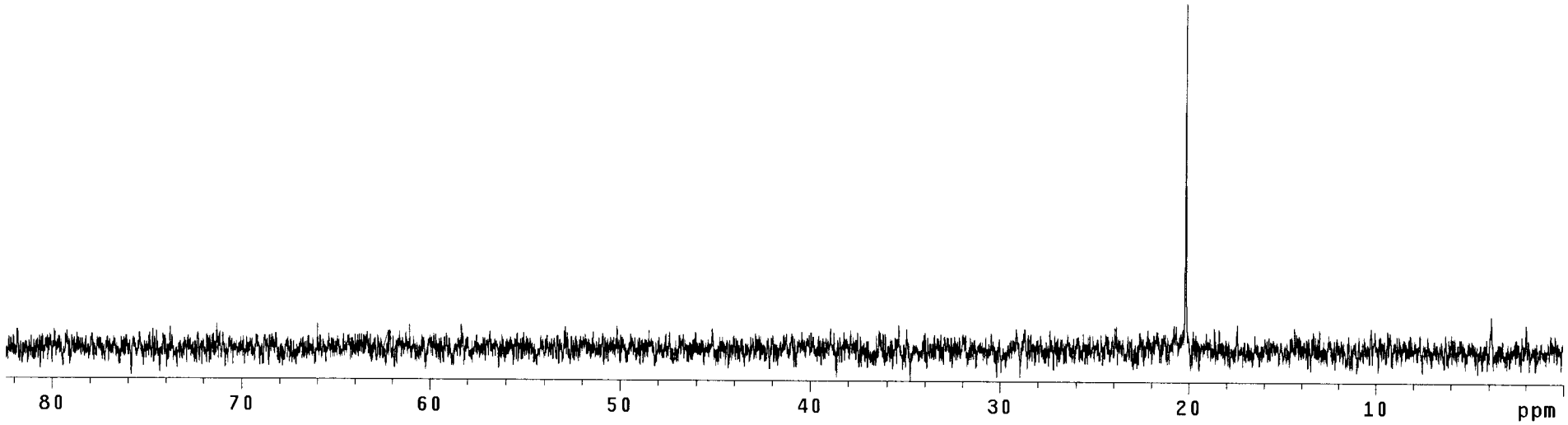


exp1 s2pul

SAMPLE		SPECIAL	
date	Sep 2 2005	temp	not used
solvent	D2O	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	**FLAGS**	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	32	**PROCESSING**	
ct	32	lb	2.00
TRANSMITTER		fn	not used
tn	P31	**DISPLAY**	
sfrq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rfp	0
DECOUPLER		rp	-97.9
dn	H1	lp	-288.8
dof	0	**PLOT**	
dm	yyy	wc	250
dmm	w	sc	0
dpwr	35	vs	61
dmf	6700	th	12
		ai no ph	

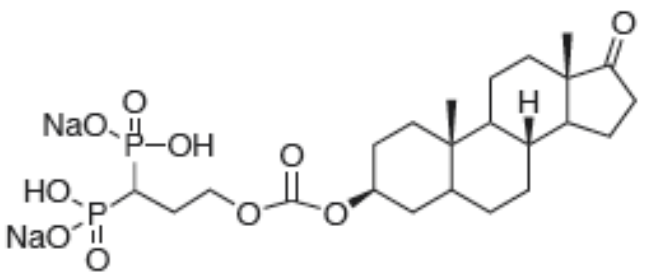


3

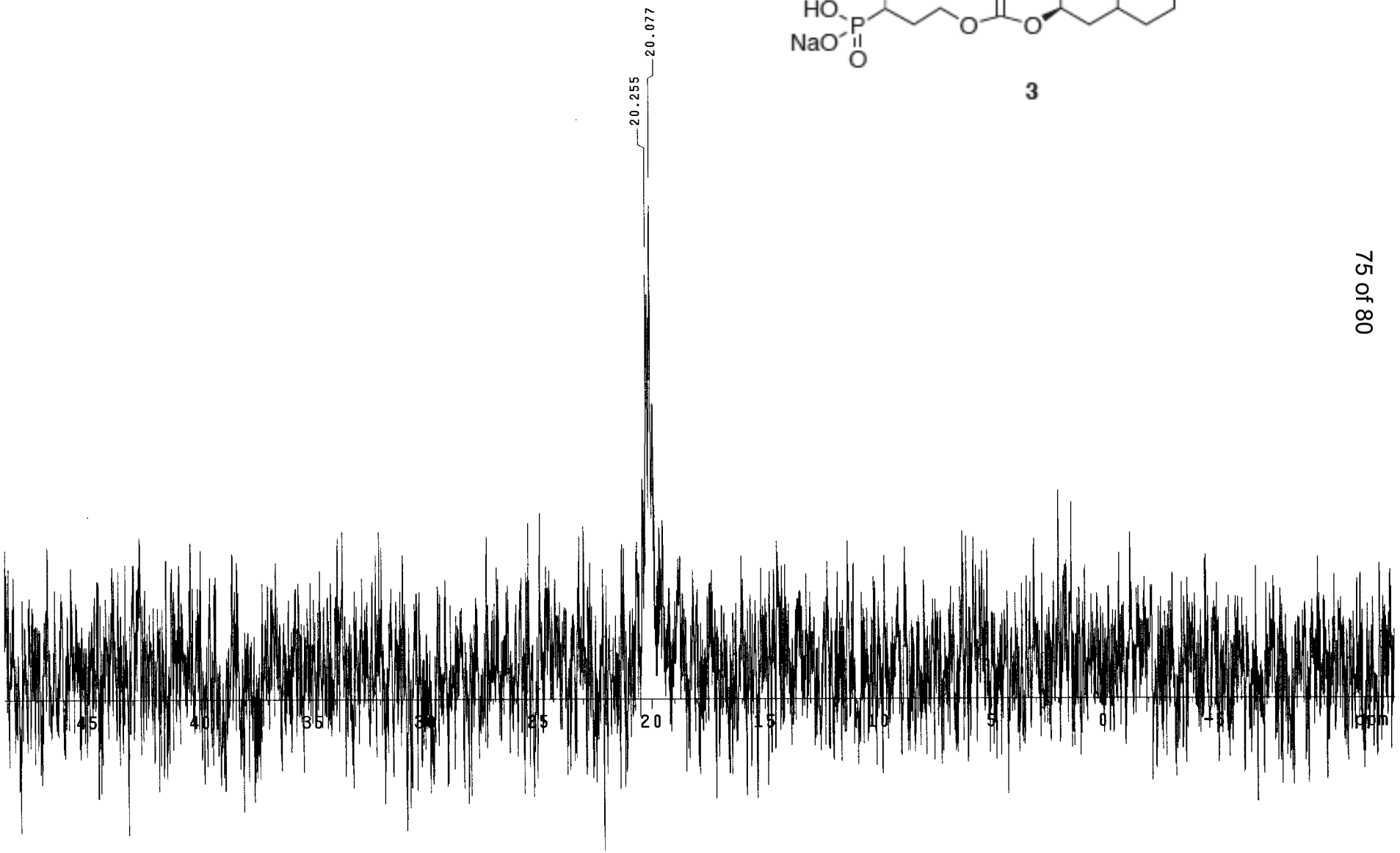


coupled

INDEX	FREQUENCY	PPM	HEIGHT
1	2460.194	20.255	71.5
2	2438.571	20.077	84.0



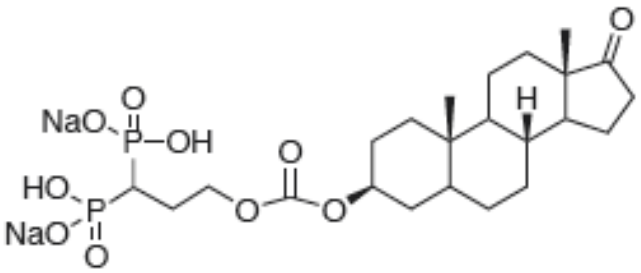
3



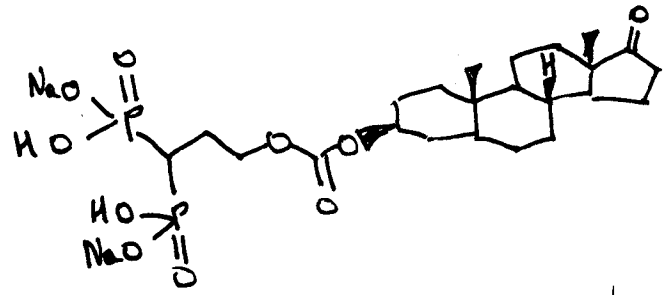
sb-II-320

exp1 s2pul

SAMPLE		SPECIAL	
date	Sep 2 2005	temp	not used
solvent	D2O	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	4803.1	pw90	17.200
at	1.994	alfa	20.000
np	19158	**FLAGS**	
fb	not used	il	n
bs	16	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	16	**PROCESSING**	
ct	16	fn	not used
TRANSMITTER		**DISPLAY**	
tn	H1	sp	-60.0
sfrq	300.054	wp	3060.5
tof	300.9	rfl	601.2
tpwr	55	rfp	0
pw	8.600	rp	-108.2
DECOUPLER		lp	-65.3
dn	C13	**PLOT**	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	1161
dpwr	45	th	5
dmf	13100	ai cdc ph	



3



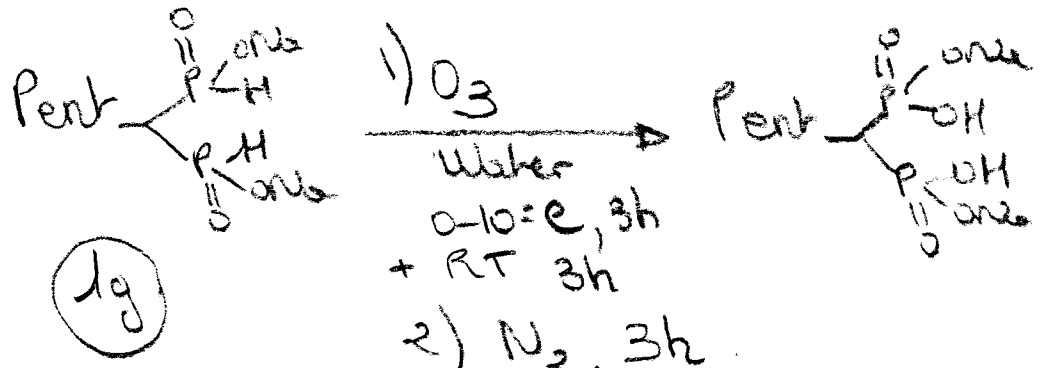
CRUDE

Decoupled

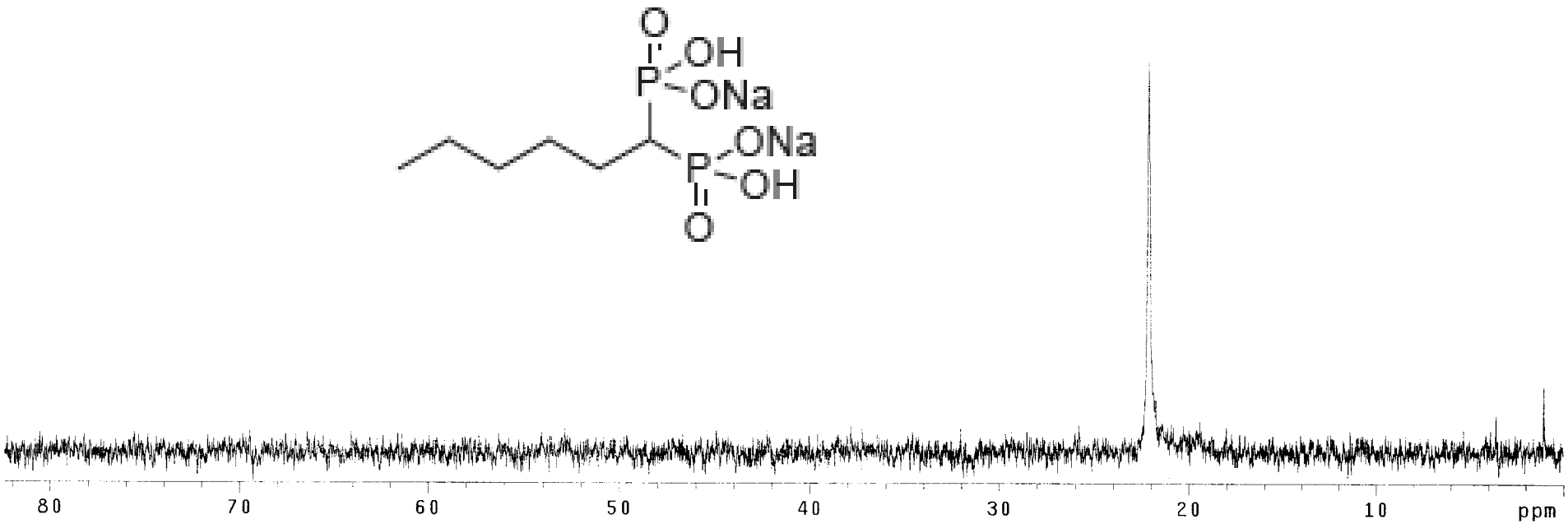
sb-II-311

exp1 s2pul

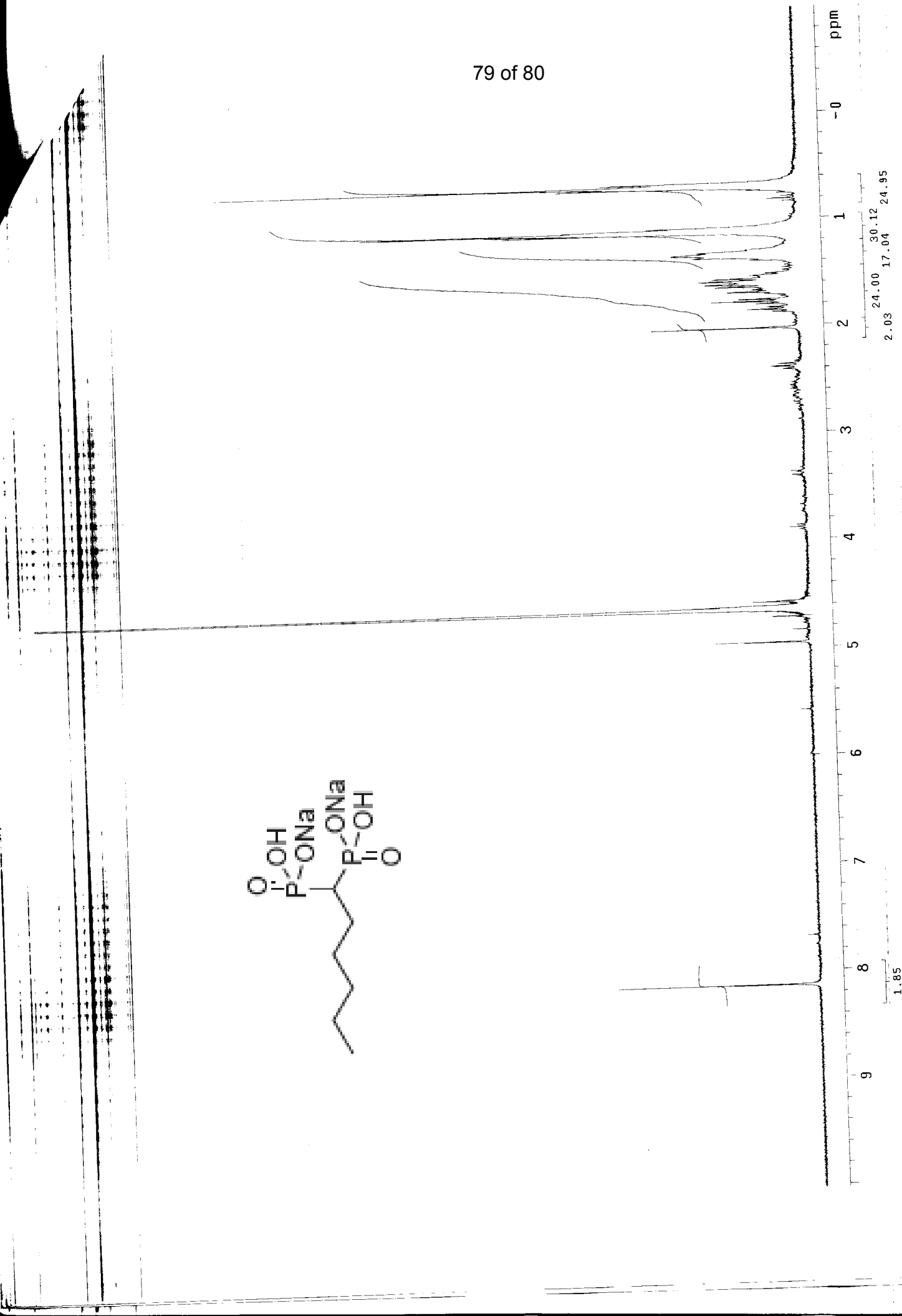
SAMPLE		SPECIAL	
date	Aug 23 2005	temp	not used
solvent	D2O	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	FLAGS	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	64	PROCESSING	
ct	64	lb	2.00
TRANSMITTER		fn	not used
tn	P31	DISPLAY	
sfrq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rfp	0
DECOUPLER		rp	-7.2
dn	H1	lp	-288.8
dof	0	PLOT	
dm	yyy	wc	250
dmm	w	sc	0
dpwr	35	vs	86
dmf	6700	th	11
		ai no ph	



1g

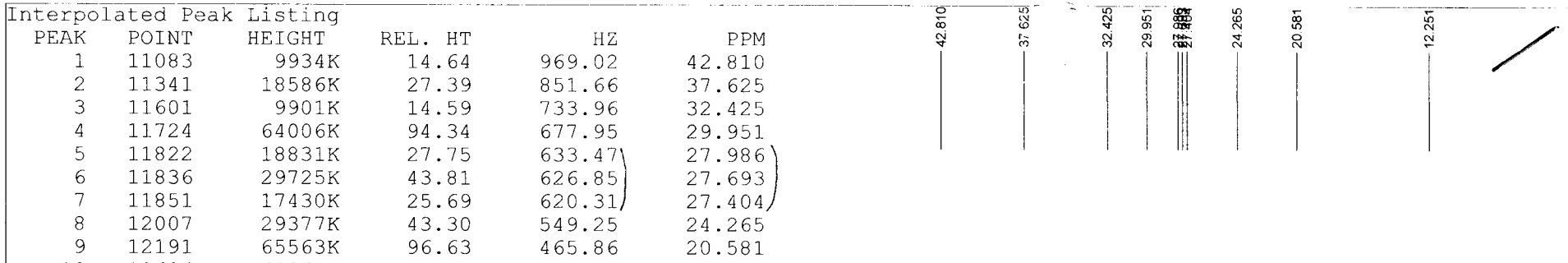


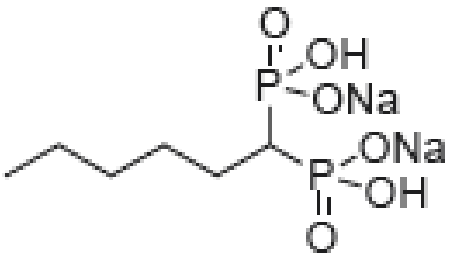
EX: c:\eft\global\zg.ppg
F1: 36.441
SW1: 20000
PW: 12.4 usec
PD: 2.0 sec
OF1: 5510.0
NA: 64
LB: 0.0
PTS1d: 32768
USER: + DATE: 08/19/05 (15:23)
40
30
20
100.00
19.400
O
OH
P
ONa
O
P
ONa
OH

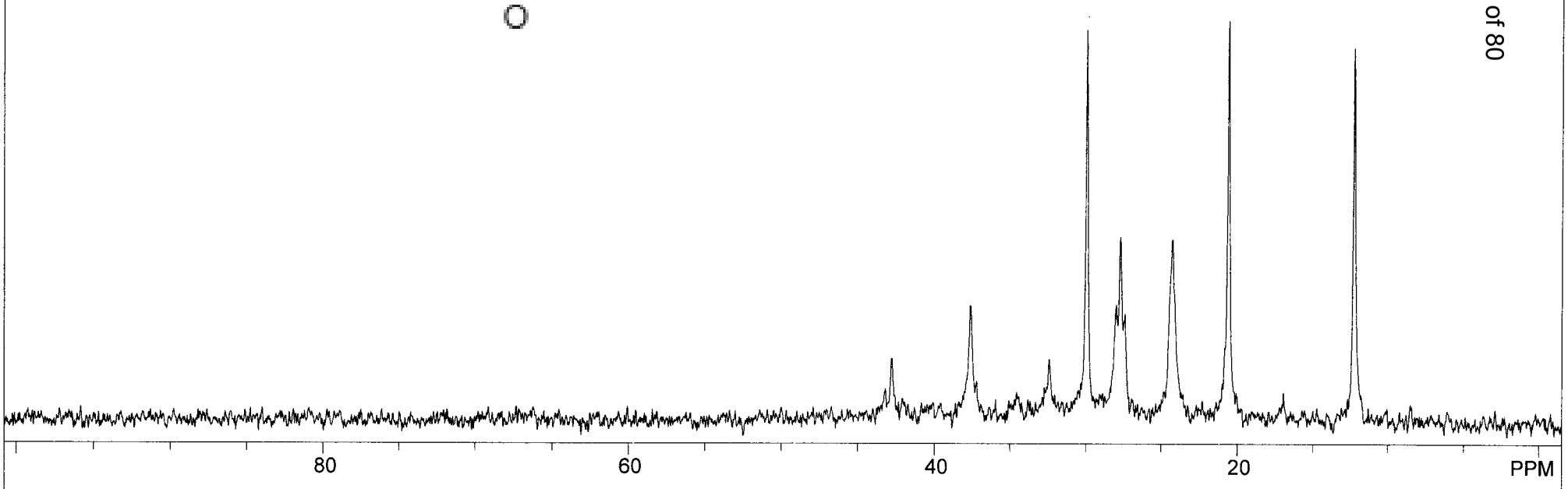

O
OH
P
ONa
ONa
P
OH
O
ppm
0
1
2
3
4
5
6
7
8
9
24.00
2.03
17.04
30.12
24.95
1.85

Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	11083	9934K	14.64	969.02	42.810
2	11341	18586K	27.39	851.66	37.625
3	11601	9901K	14.59	733.96	32.425
4	11724	64006K	94.34	677.95	29.951
5	11822	18831K	27.75	633.47	27.986
6	11836	29725K	43.81	626.85	27.693
7	11851	17430K	25.69	620.31	27.404
8	12007	29377K	43.30	549.25	24.265
9	12191	65563K	96.63	465.86	20.581
10	12606	61061K	90.00	277.31	12.251







sb-II-311 hexyl phosphonate

USER: -- DATE: 08/24/05 (20:14)

F1: 22.635	SW1: 7441		OF1: 2282.0		PTS1d: 16384
EX: bapr.ppg	PW: 18.2 usec	PD: 2.8 sec	NA: 1000	LB: 1.0	WinNuts - my_bapr_sb-II-311