Supporting Information

Mild Synthesis of Organophosphorus Compounds: Reaction of Phosphorus Containing Carbenoids with Organoboranes.

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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 $CDCl_3$ or in D_2O (δ = 4.75 ppm). ^{13}C NMR spectra were recorded at 75 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 chemical shifts reported (in parts per million) relative to external 85% phosphoric acid (δ = 0.0 ppm). ^{11}B NMR spectra were recorded at 29 MHz, and chemical shifts reported (in parts per million) relative to external $BF_3.Et_2O$ (δ = 0.0 ppm). Radial chromatography was carried out with chromatotron using 1, 2, or 4 mm layers of silica gel 60 PF_{254} containing gypsum. Silica gel (200-300 mesh, Natland International Corporation) was used for flash chromatography. 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. Mass spectrometry was provided by the Mass Spectrometry Facility of the University of South Carolina.

Reagents and Solvents. All reactions were conducted under nitrogen and all glassware was flamed before use. Bu_3B, (*sec*-Bu)$_3B$, Et_3B, B-benzyl-9-BBN, Alpine-Borane are commercially available. Other organoboranes reagents Cy_3B [1], (heptyl)$_3B$ [1], B–octyl-9-BBN [1] were prepared according to literature procedure. Butyllithium (1.6 M in hexanes) and $HBF_4.Et_2O$ were obtained from Aldrich and used as received. Tetrahydrofuran (THF) was distilled under N_2 from sodium benzophenone ketyl, and used immediately. Toluene, CH_2Cl_2 and Et_3N were freshly distilled from CaH_2.

General procedure for the reaction of halomethylphosphorus anions (4b-4g) with organoboranes.
A flame-dried, 50 mL, three-necked, round-bottomed flask was purged with nitrogen, charged with **4b-4g** (4.0 mmol, 1.0 equiv) and dry THF (20 mL). The solution was cooled below - 90 ^{o}C (liquid nitrogen/ethanol bath) and *n*-butyllitium (2.5 mL, 1.6 M solution in hexane, 4.0 mmol, 1.0 equiv) was added slowly *via* syringe followed by organoboranes (4.0 mmol, 1.0 equiv) in one portion. The reaction mixture was warmed slowly to rt and then quenched by addition of water. The resulting biphasic mixture was then stirred at reflux for 2 h. After cooling to rt, the layers were separated, the aqueous phase was extracted with EtOAc (3 X), the combined organic layers were dried with $MgSO_4$, and the solvents removed in vacuo. Purification of the crude product by chromatography on silica gel (EtOAc/hexanes) yielded the described compounds.

General procedure for the reaction of halomethylphosphorus anions (4i, 4j) with organoboranes.
A flame-dried, 50 mL, three-necked, round-bottomed flask was purged with nitrogen, charged with **4i** or **4j** (3.2 mmol, 1.0 equiv) and dry THF (15 mL). The solution was cooled below - 90 ^{o}C (liquid nitrogen/ethanol bath) and *n*-butyllithium (2.0 mL, 1.6 M solution in hexane, 3.2 mmol, 1.0 equiv) was added slowly *via* syringe followed by organoboranes (3.2 mmol, 1.0 equiv) in one portion. The reaction mixture was warmed slowly to rt and was quenched by addition of water. The resulting biphasic mixture was stirred for 30 min at rt. The layers were separated, the aqueous phase was extracted with

EtOAc (3 X), the combined organic layers were dried with $MgSO_4$, and solvents removed in vacuo. Purification of the crude product by chromatography on silica gel (EtOAc/hexanes) yielded the described compounds.

General procedure for the reaction of diphenyl (chloromethyl)phosphine-borane 4k with (Alkyl)$_3$B.
A flame-dried, 50 mL, three-necked, round-bottomed flask was purged with nitrogen, charged with diphenyl (chloromethyl)phosphine-borane **4k** (2.1 mmol, 521 mg, 1.0 equiv) and dry THF (15 mL). The solution was cooled below - 90 ^oC (liquid nitrogen/ethanol bath) and *sec*-butyllithium (1.5 mL, 1.4 M solution in cyclohexane, 2.1 mmol, 1.0 equiv) was added slowly *via* syringe followed by (Alkyl)$_3$B (2.1 mmol, 1.0 equiv) in one portion. The reaction mixture was warmed slowly to rt and was quenched by addition of water. The resulting biphasic mixture was stirred for 30 min at rt. The layers were separated, the aqueous phase was extracted with EtOAc (3 X), the combined organic layers were dried with $MgSO_4$, and solvents removed in vacuo. Purification of the crude product by chromatography on silica gel (EtOAc/hexanes) yielded the described compounds.

(Diazomethyl)phosphonate 4a[2] was prepared according to the literature.
^{1}H NMR ($CDCl_3$, 300 MHz) δ 3.79 (d, J = 12 Hz, 6 H); ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 23.6.

Diethyl (chloromethyl)phosphonate 2b.[3] Anhydrous Et_3N (38.45 g, 52.96 mL, 0.38 mol, 2.1 equiv) was added dropwise to the solution of (chloromethyl)phosphonic dichloride (30.00 g, 0.18 mol, 1.0 equiv) in dry THF (300 mL) at 0 ^oC. Absolute ethanol (17.49 g, 22.14 mL, 0.38 mol, 2.1 equiv) was added dropwise and the mixture stirred at rt, for 10 h. The precipitate was filtered and the filtrate evaporated. Purification of the crude product by distillation at 105-110 ^oC/10 torr gave diethyl (chloromethyl)phosphonate (28.20 g, 0.17 mol, 94 %).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 4.23 (quin, J = 7 Hz, 4 H), 3.57 (d, J = 10 Hz, 2 H), 1.38(t, J = 7 Hz, 6 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 63.4 (d, J_{POC} = 6 Hz), 33.4 (d, J_{PC} = 160 Hz), 16.4 (d, J_{POCC} = 6 Hz); ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 19.8.

Dibenzyl (chloromethyl)phosphonate 4c[4] was prepared according to the literature. Anhydrous Et_3N (10.30 mL, 73.9 mmol, 3.0 equiv) was added dropwise to the solution of chloromethylphosphonic dichloride (4.1 g, 24.5 mmol, 1.0 equiv) in dry THF (200 mL) at 0 ^oC. Benzyl alcohol (5.60 mL, 53.9 mmol, 2.2 equiv) was added dropwise and the mixture stirred at 0 ^oC for 1 h and then at rt, for 4 h. The precipitate was filtered, the filtrate evaporated. Purification of the crude product by chromatography on silica gel (EtOAc/hexanes 1:1, v/v) yielded dibenzyl (chloromethyl)phosphonate (6.05 g, 19.5 mmol, 80%).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.35 - 7.37 (m, 10 H), 4.05 - 5.18 (m, 4 H), 3.49 (d, J = 11, Hz, 2 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 135.8, 128.0, 127. 9, 69.1 (d, J_{POC} = 7 Hz), 33.9 (d, J_{PC} = 160 Hz); ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 20.8.

Tri(chloromethyl)phosphonate 4d[5] was prepared according to the literature.

^{1}H NMR ($CDCl_3$, 300 MHz) δ 4.37 - 4.52 (m, 4 H), 1.45 (t, J = 7 Hz, 6 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 92,1 (d, J_{PC} = 198 Hz), 67.1 (d, J_{POC} = 7 Hz), 16.4 (d, J_{POCC} = 6 Hz); ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 6.5.

Diethyl (1-chloroethyl)phosphonate 4e[6] **and diethyl 1-chloro-benzylphosphonate 4f**[6] were prepared according to literature from diethyl (hydroxyethyl)phosphonate and 1-hydroxy-benzylphosphonate respectively, triphenylphosphine and carbon tetrachloride.
Diethyl (hydroxyethyl)phosphonate[7] and 1-hydroxy-benzylphosphonate[7] were prepared according to the literature from diethyl phosphite and the appropriate aldehyde in the presence of Et_3N.
Diethyl (1-chloroethyl)phosphonate **4e**. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 4.17 - 4.29 (m, 4 H), 4.10 (dq, J_{HH} = 7 Hz, J_{HP} = 9 Hz, 1 H), 1.70 (dd, J_{HH} = 7 Hz, $J_{HP=}$ 16.6 Hz, 3 H), 1.37 (t, J = 7 Hz, 6 H); ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 22.2.
Diethyl 1-chloro-benzylphosphonate **4f.** ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.34 - 7.56 (m, 5 H), 4.90 (d, J = 14 Hz, 1 H), 3.85 - 4.24 (m, 4 H), 1.33 (t, J = 7 Hz, 3 H), 1.18 (t, J = 7 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 134.1, 129.0, 128.9 (d, J_{PCCC} = 6 Hz, 2 C),128.5 (2 C), 64.1 (d, J_{POC} = 7 Hz), 63.9 (d, J_{POC} = 7 Hz), 53.6 (d, J_{PC} = 160 Hz), 16.4 (d, J_{POCC} = 6 Hz), 16.2 (d, J_{POCC} = 6 Hz); ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 18.3.

Ethyl [bis(chloromethyl)]phosphinate 4g.[8] Anhydrous Et_3N (15.68 g, 21.60 mL, 0.155 mol, 1.2 equiv) was added dropwise to the solution of bis(chloromethyl)phosphinic chloride (23.20 g, 0.129 mol, 1.0 equiv) in dry THF (300 mL) at 0 ^{o}C. Absolute ethanol (7.14 g, 9.04 mL, 0.155 mol, 1.2 equiv) was added dropwise and the mixture stirred at rt, for 4 h. The precipitate was filtered and the filtrate evaporated. Purification of the crude product by distillation at 104-106 ^{o}C /1 torr gave ethyl [bis(chloromethyl)]phosphinate (20.83 g, 0.110 mol, 85 %).
^{1}H NMR ($CDCl_3$, 300 MHz): δ 4.16 - 4.26 (m, 2 H), 3.72 (d, J = 9 Hz, 4 H), 1.36 (t, J = 7 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 63.2 (d, J_{POC} = 7 Hz), 33.3 (d, J_{PC} = 106 Hz), 16.7 (d, J_{POCC} = 5 Hz); ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 40.7.

Bis(chloromethyl)phosphinic chloride[9] was prepared according to the literature. A mixture of H_3PO_2 (30.50 g, 0.32 mol, 50 % aqueous solution), 17.5 mL concentrated HCl and 14.5 g of paraformaldehyde was stirred at 40-45 ^{o}C until a clear solution was obtained and then refluxed for 30 h. Evaporation under reduced pressure gave crude bis(hydroxymethyl)phosphinic chloride, which was used in next step without purification.
To a 176.0 g of refluxing $SOCl_2$ was added slowly with stirring crude bis(hydroxymethyl)phosphinic chloride (30.00 g, 0.25 mol). After completion of the addition, refluxing was continued for 3 h until gas evolution ceased. Purification of the crude product by distillation b.p. 80-85 ^{o}C /0.1 torr gave bis(chloromethyl)phosphinic chloride (32.38 g, 0.18 mol, 72 %).

Diethyl (chloromethyl)phosphonothioate 4h[10] was prepared according to the literature from (chloromethyl)phosphonic dichloride and sodium ethoxide.

^{1}H NMR ($CDCl_3$, 300 MHz) δ 4.14 - 4.27 (m, 4 H), 3.70 (d, J = 7 Hz, 2 H), 1.36 (t, J = 7 Hz, 6 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 64.0 (d, J_{POC} = 6 Hz), 41.0 (d, J_{PC} = 124 Hz), 16.4 (d, J_{POCC} = 6 Hz); ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 85.5.

Diethoxy-(chloromethyl)phosphine-borane 4i. To a solution of dichloro-(chloromethyl)phosphine (66.1 mmol, 10.00 g, 1.0 equiv) in dry THF (150 mL), was added absolute ethanol (132.1 mmol, 6.10 g, 7.70 mL, 2.0 equiv) under nitrogen. The reaction mixture was then cooled to 0 °C and Et_3N (132.1 mmol, 18.4 mL, 2.0 equiv) was slowly added. After the solution has been stirred for 10 min at rt, it was again cooled to 0° C and borane-methyl sulfide (36.5 mL of a 2.0 M solution in THF, 73 mmol, 1.1 equiv) was added. The solution was allowed to warm up to rt and stirring was continued for 15 min. The precipitate was removed by filtration, the filtrate diluted with EtOAc and washed with water (1 X). The resulting organic layer was dried with $MgSO_4$, and the solvent removed in vacuo. Purification of the crude product by chromatography on silica gel (EtOAc/hexanes 1:99, v/v) yielded diethoxy-(chloromethyl)phosphine-borane **4i** (62.5 mmol, 11.5 g, 95 %).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 4.03 - 4.26 (m, 4 H), 3.55 (d, J = 3 Hz, 2 H), 1.35 (t, J = 7 Hz, 6 H), 0.50 (qd, J_{BH} = 96 Hz, J_{PBH} = 16 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 64.5 (d, J_{POC} = 4 Hz), 37.5 (d, J_{PC} = 55 Hz), 16.5 (d, J_{POCC} = 5 Hz); ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 137.5 (q, J_{PB} = 76 Hz); ^{11}B NMR ($CDCl_3$, 28.88 MHz) δ - 44.2 (dq, J_{BP} = 76 Hz, J_{BH} = 96 Hz); HRMS calcd. for $C_5H_{19}BClNO_2P$, ($[M + NH_4]^+$) 202.0937, found 202.0935.

Dichloro-(chloromethyl)phosphine[11] was prepared by the sulfur exchange reaction as reported in the literature.
The mixture of chloromethylphosphonothioic dichloride (17.50 g, 96.2 mmol, 1.0 equiv) and dichlorophenylphosphine (19.80 g, 110.6 mmol, 1.15 equiv) was heated at 175 ^{o}C under an atmosphere of nitrogen in the sealed tube for 3 h. It was then cooled and distilled. The fraction collected boiling at 67-132 ^{o}C at 100 torr was then carefully fractioned under atmospheric pressure (128-132 ^{o}C) to give dichloro-(chloromethyl)phosphine (10.24 g, 68.3 mmol, 71 %).
^{31}P NMR (36 MHz) δ 165.

(Chloromethyl)phosphonothioic dichloride[11] was prepared according to the literature.
A mixture of posphorus pentasulfide (8.05 g, 18.1 mmol, 0.12 equiv) and chloromethylphosphonic dichloride (25.00 g, 150.7 mmol, 1.0 equiv) was heated to reflux at 174-179 ^{o}C under nitrogen for 6 h, and then distilled at 50 ^{o}C/10 torr to give (chloromethyl)phosphonothioic dichloride (102.5 mmol, 18.64 g, 68 %).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 4.29 (d, J = 3 Hz, 2 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 41.0 (d, J_{PC} = 87 Hz); ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 75.1.

(Chloromethyl)phosphonic dichloride[12] was prepared according to the literature.
Dichloromethane (34.98 g, 0.4 mol, 25.7 mL, 1.0 equiv), phosphorus trichloride (54.93 g, 0.4 mol, 34.89 mL, 1.0 equiv) and aluminium chloride (53.34 g, 0.4 mmol, 1.0 equiv) were mixed and heated at 100 ^{o}C in the sealed tube for 24 h. After cooling to rt, the reaction mixture was dissolved in dichloromethane and the solution was cooled to about -

20 °C. Water (79.2 mL, 4.4 mol, 11.0 equiv) was then added in small portions with vigorous stirring. The solution was filtered, the solvent removed in vacuo and the residue distilled at 50 °C/ 0.5 torr to give (chloromethyl)phosphonic dichloride (33.28 g, 0.2 mol, 50 %).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 4.17 (d, J = 6 Hz, 2 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 45.5 (d, J_{PC} = 117 Hz); ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 39.2.

Diethoxy (1-chloroethyl)phosphine-borane 4j. A flame-dried, 50 mL, three-necked, round-bottomed flask was purged with nitrogen, charged with diethoxy (chloromethyl)phosphine-borane (4.0 mmol, 736 mg, 1.0 equiv) and dry THF (20 mL). The solution was cooled to - 78 °C and *n*-butyllithium (3.0 mL, 1.6 M solution in hexane, 4.8 mmol, 1.2 equiv) was added slowly *via* syringe. The reaction mixture was stirred at - 78 °C for 5 min, then iodomethane (4.8 mmol, 681 mg, 1.2 equiv) was added. The reaction was warmed slowly to rt and was quenched by addition of H_2O. The layers were separated and the aqueous phase was extracted with EtOAc (3 X). The combined organic layers were dried with $MgSO_4$, and solvents removed in vacuo. Purification of the crude product by chromatography on silica gel (EtOAc/hexanes 1:99, v/v) yielded diethoxy (1-chloroethyl)phosphine-borane **4j** (3.60 mmol, 713 mg, 90 %).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 4.10 - 4.24 (m, 4 H), 3.93 (q, J = 7 Hz, 1 H), 1.66 (d, J = 7 Hz, 3 H), 1.35 (t, J = 7 Hz, 6 H), 0.50 (qd, J_{BH} = 94 Hz, J_{PBH} = 16 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 64.6, 37.5 (d, J_{PCC} = 58 Hz), 18.3, 16.5; ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 141.4 (q, J_{PB} = 75 Hz); ^{11}B NMR ($CDCl_3$, 28.88 MHz) δ - 44.4 (dq , J_{BP} = 75Hz, J_{BH} = 94 Hz); HRMS calcd. for $C_6H_{21}BClNO_2P$, ($[M + NH_4]^+$) 216.1092, found 216.1089.

(Chloromethyl)diphenylphosphine[13] was prepared according to the literature.
(Chloromethyl)diphenylphosphine-borane 4k. A solution of KOH (53.5 mmol, 3.00 g, 5.0 equiv) in H_2O (2.5 mL) was mixed with a solution of *n*-Bu_4NCl (1.8 mmol, 500 mg, 0.17 equiv) in CH_2Cl_2 (30 mL) and toluene (5 mL). Diphenylphosphine (10.7 mmol, 2.00 g, 1.0 equiv) dissolved in CH_2Cl_2 (5 mL) was then added under nitrogen to the emulsion, under vigorous stirring, over 2 h. The reaction mixture was stirred for 14 h at rt, washed with H_2O, the organic layer separated and transferred to the round-bottomed flask. $BH_3.Me_2S$ (2.0 M solution in THF, 8.0 mL, 16 mmol, 1.5 equiv) was then added and the reaction mixture was stirred at rt, for 1 h. The solvent was removed in vacuo and the residue diluted with EtOAc then washed with water. The organic layer was dried with $MgSO_4$, and the solvent removed in vacuo. Purification of the crude product by chromatography on silica gel (EtOAc/hexanes 1:9, v/v) yielded (chloromethyl)diphenylphosphine-borane **4k** (9.6 mmol, 2.38 g, 90 %).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.40 - 7.70 (m, 10 H), 4.09 (d, J = 7 Hz, 3 H), 0.7 (q, J_{BH} = 100 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 133.1 (d, J_{PCCC} = 10 Hz, 4 C), 132.3 (d, J_{PCCCC} = 2.5 Hz, 2 C), 129.9 (d, J_{PC} = 55 Hz), 129.3 (d, J_{PCC} = 10 Hz, 4 C), 126.4 (d, J_{PC} = 57 Hz), 37.1 (d, J_{PC} = 32 Hz); ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 24.6 (d, J_{PB} = 60 Hz); ^{11}B NMR $CDCl_3$, 28.88 MHz) δ - 39.8 (dd , J_{BP} = 60 Hz, J_{BH} = 100 Hz); HRMS calcd. for $C_{13}H_{17}BClNP$, $[M + NH_4^+ - H_2]$ 264.0880, found 264.0888.

Dimethyl pentylphosphonate (3a-H, Scheme 2).[14] A flame-dried, 25 mL, three-necked, round-bottomed flask was purged with nitrogen, charged with dimethyl (diazomethyl)phosphonate **4a** (2.0 mmol, 300 mg, 1.0 equiv) and dry THF (10 mL). Bu_3B (2.0 mL, 1.0 M solution in diethyl ether, 2.0 mmol, 1.0 equiv) was then added in one portion. An exothermic reaction ensued and nitrogen evolved. The reaction was then stirred at rt for 1 h, and quenched by addition of water. The resulting biphasic mixture was heated at reflux for 2 h. After cooling to rt, the layers were separated, the aqueous phase was extracted with EtOAc (3 X), the combined organic layers were dried with $MgSO_4$, and the solvents removed in vacuo. Purification of the crude product by chromatography on silica gel (EtOAc/hexanes, 1:1, v/v) yielded dimethyl pentylphosphonate **3a-H** (1.72 mmol, 310 mg, 86 %).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 3.74 (d, J = 11 Hz, 6 H), 1.31 - 1.80 (m, 8 H), 0.89 (t, J = 7 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 52.5 (d, J_{POC} = 7 Hz), 33.0 (d, J_{PCCC} = 17 Hz), 24.9 (d, J_{PC} = 140 Hz), 22.4, 22.2 (d, J_{PCC} = 5 Hz), 14.0; ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 36.3.

Monodeuterated dimethyl pentylphosphonate (3a-D, Scheme 2). A flame-dried, 25 mL, three-necked, round-bottomed flask was purged with nitrogen, charged with dimethyl (diazomethyl)phosphonate **4b** (2.0 mmol, 300 mg, 1.0 equiv) and dry THF (10 mL). Bu_3B (2.0 mL, 1.0 M solution in diethyl ether, 2.0 mmol, 1.0 equiv) was then added in one portion. An exothermic reaction ensued and nitrogen evolved. The reaction was then stirred at rt for 1 h, then quenched by addition of D_2O. The resulting biphasic mixture was stirred for 12 h at rt. The layers were separated and the aqueous phase was extracted with EtOAc (3 X), the combined organic layers were dried with $MgSO_4$, and the solvents removed in vacuo. Purification of the crude product by chromatography on silica gel (EtOAc/hexanes 1:1, v/v) yielded dimethyl pentylphosphonate **3a-D** (1.6 mmol, 290 mg, 80%). Deuterium incorporation 98 %.
^{1}H NMR ($CDCl_3$, 300 MHz) δ 3.74 (d, J = 11 Hz, 6 H), 1.24 - 1.75 (m, 7 H), 0.89 (t, J = 7 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 52.5 (d, J_{POC} = 7 Hz), 33.0 (d, J_{PCCC} = 17 Hz), 24.9 (dt, J_{PC} = 140 Hz, J_{DC} = 19 Hz), 22.4, 22.1 (d, J_{PCC} = 5 Hz), 14.0; ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 36.4; HRMS (EI^+) calcd. for $C_7H_{17}DO_3P$, ($[M]^+$) 182.1056, found 182.1056.

Diethyl pentylphosphonate (3b-H, Scheme 3).[15] The title compound was prepared from diethyl (chloromethyl)phosphonate **4b** (4.0 mmol, 746 mg, 1.0 equiv) and Bu_3B (4.0 mL, 1.0 M solution in diethyl ether, 4.0 mmol, 1.0 equiv). Purification of the crude product by chromatography on silica gel (EtOAc/hexanes, 1:1, v/v) yielded diethyl pentylphosphonate **3b-H** (3.84 mmol, 800 mg, 96 %).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 4.03 - 4.16 (m, 4 H), 1.25 - 1.77 (m, 8 H), 1.32 (t, J = 7 Hz, 6 H), 0.90 (t, J = 7 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 61.6 (d, J_{POC} = 6 Hz), 33.0 (d, J_{PCCC} = 17 Hz), 25.5 (d, J_{PC} = 140 Hz), 22.3, 22.2 (d, J_{PCC} = 5 Hz), 16.6 (d, J_{POCC} = 6 Hz), 14.1; ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 33.8.

Monodeuterated diethyl pentylphosphonate (3b-D, Scheme 3). The title compound was prepared from diethyl (chloromethyl)phosphonate **4b** (4.0 mmol, 746 mg, 1.0 equiv) and Bu_3B (4.0 mL, 1.0 M solution in diethyl ether, 4.0 mmol, 1.0 equiv). The reaction

mixture was quenched by addition of D_2O at rt. The resulting biphasic mixture was stirred for 12 h at rt. Purification of the crude product by chromatography on silica gel (EtOAc/hexanes 1:1, v/v) yielded diethyl pentylphosphonate **3b-D** (3.56 mmol, 745 mg, 89 %). Deuterium incorporation 95 %.
^{1}H NMR ($CDCl_3$, 300 MHz) δ 4.02 - 4.17 (m, 4 H), 1.25 - 1.66 (m, 7 H), 1.35 (t, J = 7 Hz, 6 H), 0.91 (t, J = 7 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 61.6 (d, J_{POC} = 7 Hz), 32.9 (d, J_{PCCC} = 17 Hz), 25.9 (d, J_{PC} = 140 Hz), 22.4, 22.3 (d, J_{PCC} = 5 Hz), 16.7 (d, J_{POCC} = 6 Hz), 14.0; ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 33.8; HRMS (EI$^+$) calcd. for $C_9D_{21}DO_3P$, ([M]$^+$) 210.1369, found 210.1367.

Dibenzyl pentylphosphonate (Table 1, entry 1).[16] The title compound was prepared from dibenzyl (chloromethyl)phosphonate **4c** (4.0 mmol, 1.24 g, 1.0 equiv) and Bu_3B (4.0 mL, 1.0 M solution in diethyl ether, 4.0 mmol, 1.0 equiv). Purification of the crude product by chromatography on silica gel (EtOAc/hexanes 1:1, v/v) yielded dibenzyl pentylphosphonate (3.40 mmol, 1.13 g, 85 %).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.26 - 7.42 (m, 10 H), 4.93 - 5.14 (m, 4 H), 1.20 - 1.79, (m, 8 H), 0.85 (t, J = 7 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 136.4 (d, J_{PCC} = 6 Hz, 2 C), 128.6 (4 C), 128.4 (2 C),127.9 (4 C), 67.0 (d, J_{POC} = 7 Hz), 32.6 (d, J_{PCCC} = 15 Hz), 25.9 (d, J_{PC} = 140 Hz), 22.0, 13.8; ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 35.0.

Diethyl 1-chloropentylphosphonate (Table 1, entry 2).[17] A flame-dried, 50 mL, three-necked, round-bottomed flask was purged with nitrogen, charged with diethyl tri(chloromethyl)phosphonate **4d** (4.0 mmol, 1.01 g, 1.0 equiv) and dry THF (20 mL). The solution was cooled below - 100 ^{o}C (liquid nitrogen/ethanol bath) and *n*-butyllithium (2.5 mL, 1.6 M solution in hexane, 4.0 mmol, 1.0 equiv) was added slowly *via* syringe followed by Bu_3B (4.0 mL, 1.0 M solution in diethyl ether, 4.0 mmol, 1.0 equiv) in one portion. The reaction mixture was slowly warmed to - 50 ^{o}C and was quenched by addition of water. The resulting biphasic mixture was stirred at reflux for 2 h. The layers were separated, the aqueous phase was extracted with EtOAc (3 X), the combined organic layers were dried with $MgSO_4$, and solvents removed in vacuo. Purification of the crude product by chromatography on silica gel (EtOAc/hexanes 3:7, v/v) yielded diethyl 1-chloropentylphosphonate (2.8 mmol, 678 mg, 70 %), 90 % purity (10 % diethyl 1-butylpentylphosphinate).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 4.19 - 4.30 (m, 4 H), 3.85 (td, J = 11 Hz, J = 3 Hz, 1H), 1.28 - 2.11 (m, 6 H), 1.38 (t, J = 7 Hz, 6 H), 0.94 (t, J = 7 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 63.7 (d, J_{POC} = 7 Hz), 63.2 (d, J_{POC} = 7 Hz), 52.4 (d, J_{PC} = 160 Hz), 31.8 , 28.6 (d, J_{PCCC} = 12 Hz), 21.9, 16.6 (d, J_{POCC} = 5 Hz), 13.8; ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 21.8.

Diethyl 1-butylpentylphosphinate (Table 1, entry 3).[18] A flame-dried, 50 mL, three-necked, round-bottomed flask was purged with nitrogen, charged with diethyl (trichloromethyl)phosphonate **4d** (4.0 mmol, 1.01 g, 1.0 equiv) and dry THF (20 mL). The solution was cooled below - 100 ^{o}C (liquid nitrogen/ethanol bath) and *n*-butyllithium (2.5 mL, 1.6 M solution in hexane, 4.0 mmol, 1.0 equiv) was added slowly *via* syringe followed by Bu_3B (4.0 mL, 1.0 M solution in diethyl ether, 4.0 mmol, 1.0 equiv) in one portion. The reaction mixture was warmed slowly to - 50 ^{o}C and then was cooled down

to - 78 ^{o}C and *n*-butyllithium (2.5 mL, 1.6 M solution in hexane, 4.0 mmol, 1.0 equiv) was added slowly. After the addition the reaction mixture was quenched with water at rt The resulting biphasic mixture was stirred at reflux for 2 h. After cooling to rt, the layers were separated, the aqueous phase was extracted with EtOAc (3 X), the combined organic layers were dried with $MgSO_4$, and solvents removed in vacuo. Purification of the crude product by chromatography on silica gel (EtOAc/hexanes 1:1 v/v) yielded diethyl 1-butylpentylphosphinate (2.08 mmol, 549 mg, 52 %).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 4.03 - 4.14 (m, 4 H), 2.10 - 2. 25 (m, 1 H), 1.25 - 1.77 (m, 12 H), 1.30 (t, J = 7 Hz, 6 H), 0.90 (t, J = 7 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 61.2 (d, J_{POC} = 7 Hz), 35.9 (d, J_{PC} = 138 Hz), 29.8 (d, J_{PCCC} = 9 Hz), 28.0 (d, J_{PCC} = 4 Hz), 22.8, 16.5 (d, J_{POCC} = 6 Hz), 14.0; ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 36.2.

Diethyl 1-methyl-pentylphosphonate. (Table 1, entry 4).[10-19] The title compound was prepared from diethyl (1-chloroethyl)phosphonate **4e** (4.0 mmol, 800 mg, 1.0 equiv) and Bu_3B (4.0 mL, 1.0 M solution in diethyl ether, 4.0 mmol, 1.0 equiv). Purification of the crude product by chromatography on silica gel (EtOAc/hexanes 1:1, v/v) yielded diethyl 1-methyl-pentylphosphonate (2.52 mmol, 560 mg, 63 %).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 4.04 - 4.16 (m, 4 H), 1.36 - 1.79 (m, 7 H) 1.26 - 1.34 (t, J = 7 Hz, 3 H), 1.12 -1.20 (dd, J_{HH} = 7 Hz, $J_{HP=}$ 18 Hz, 3 H), 0.88 - 0.93 (t, J = 7 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 61.6 (t, J_{POC} = 6 Hz), 30.9 (d, J_{PC} = 140 Hz), 29.8 (2 C), 29.7, 29.6, 22.7, 16.7 (d, J_{POCC} = 6 Hz), 14.2, 13.3 (d, J_{PCC} = 5 Hz); ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 36.7.

Diethyl 1-phenyl-pentylphosphonate. (Table 1, entry 5).[19,20] The title compound was prepared from diethyl 1-chloro-benzylphosphonate **4f** (4.0 mmol, 1.05 g, 1.0 equiv) and Bu_3B (4.0 mL, 1.0 M solution in diethyl ether, 4.0 mmol, 1.0 equiv). Purification of the crude product by chromatography on silica gel (EtOAc/hexanes 1:1, v/v) yielded diethyl 1-phenyl-pentylphosphonate (2.4 mmol, 682 mg, 60 %).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.20 - 7.34 (m, 5 H), 3.66 - 4.13 (m, 4 H), 2.98 (ddd, J_{HH} = 4 Hz, J_{HH} = 11 Hz, J_{PH} = 22 Hz, 1 H), 1.90 - 2 .15, (m, 2 H), 1.28 (t, J = 7 Hz, 3 H), 1.14 -1.40 (m, 4 H), 1.09 (t, J = 7 Hz, 3 H), 0.83 (t, J = 7 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 136.3, 129.2 (d, J_{PCCC} = 7 Hz, 2 C),128.4 (2 C), 127.0, 62.5 (d, J_{POC} = 7 Hz), 61.7 (d, J_{POC} = 7 Hz), 44.6 (d, J_{PC} = 138 Hz), 29.8 (d, J_{PCCC} = 15 Hz), 28.5, 22.3, 16.4 (d, J_{POCC} = 6 Hz), 16.2 (d, J_{POCC} = 6 Hz), 13.8; ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 30.3.

Ethyl (chloromethyl)pentylphosphinate. (Table 1, entry 6). The title compound was prepared from ethyl [bis(chloromethyl)]phosphinate **4g** (4.0 mmol, 760 mg, 1.0 equiv) and Bu_3B (4.0 mL, 1.0 M solution in diethyl ether, 4.0 mmol, 1.0 equiv). Purification of the crude product by chromatography on silica gel (EtOAc/hexanes 1:1, v/v) yielded ethyl (chloromethyl)pentylphosphinate (3.12 mmol, 662 mg, 78 %).
^{1}H NMR ($CDCl_3$, 300 MHz): δ 4.03 - 4.26 (m, 2 H), 3.54 (d, J = 8 Hz, 2 H), 1.32 - 1.93 (m, 8 H), 1.30 (t, J = 7 Hz, 3 H), 0.91 (t, J = 7 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 61.4 (d, J_{POC} = 7 Hz), 34.7 (d, J_{PC} = 91 Hz), 33.0 (d, J_{PCCC} = 15 Hz), 25.6 (d, J_{PC} = 100 Hz), 22.3, 21.1 (d, J_{PCC} = 4 Hz), 16.4 (d, J_{POCC} = 6 Hz), 14.0; ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 50.4. HRMS calcd. for $C_8H_{18}ClO_2P$, 212.0733, found 212.0730.

Diethyl pentylphosphonothioate (Table 1, entry 7). A flame-dried, 50 mL, three-necked, round-bottomed flask was purged with nitrogen, charged with diethyl (chloromethyl)phosphonothioate **4h** (4.0 mmol, 808 mg, 1.0 equiv) and dry THF (20 mL). The solution was cooled to - 78 ^{o}C (liquid nitrogen/ethanol bath) and *n*-butyllitium (2.5 mL, 1.6 M solution in hexane, 4.0 mmol, 1.0 equiv) was added slowly *via* syringe. After the reaction mixture had been stirred for 10 min at this temperature, it was cooled to - 90^{o}C (liquid nitrogen/ethanol bath) and Bu_3B (4.0 mL, 1.0 M solution in diethyl ether, 4.0 mL, 1.0 equiv) was added in one portion. The reaction solution was warmed slowly to rt and was quenched by addition of water. The resulting biphasic mixture was stirred at rt for 2 h, the layers were separated, the aqueous phase was extracted with EtOAc (3 X), the combined organic layers were dried with $MgSO_4$, and solvents removed in vacuo. Purification of the crude product by chromatography on silica gel (EtOAc/hexanes 2:98, v/v) yielded diethyl pentylphosphonothioate (3.32 mmol, 774 mg, 83 %).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 3.99 - 4.23 (m, 4 H), 1.21 - 2.00 (m, 8 H), 1.30 (t, J = 7 Hz, 6 H), 0.90 (t, J = 7 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 62.5 (d, J_{POC} = 7 Hz), 34.8 (d, J_{PC} = 111 Hz), 32.6 (d, J_{PCCC} = 18 Hz), 22.7 (d, J_{PCC} = 4 Hz), 22.4 (d, J_{PCCCC} = 1 Hz), 16.4 (d, J_{POCC} = 7 Hz), 14.1. ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 100.9; HRMS calcd. for $C_9H_{21}O_2PS$, 224.1000, found 224.1000.

Diethoxy pentylphosphonite-borane (Table 1, Entry 8). The title compound was prepared from diethoxy (chloromethyl)phosphine-borane **4i** (3.2 mmol, 590 mg, 1.0 equiv) and Bu_3B (3.2 mL, 1.0 M solution in diethyl ether, 3.2 mmol, 1.0 equiv). Purification of the crude product by chromatography on silica gel (EtOAc/hexanes 0.5 : 99.5, v/v) yielded diethoxy pentylphosphonite-borane (2.94 mmol, 607 mg, 92%).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 3.95 - 4.16 (m, 4 H), 1.24 - 1.75 (m, 8 H), 1.31 (t, J = 7 Hz, 6 H), 0.91 (t, J = 7 Hz, 3 H), 0.50 (qd, J_{BH} = 94 Hz, J_{PBH} = 15 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 63.2 (d, J_{POC} = 5 Hz), 33.0 (d, J_{PCCC} = 14 Hz), 29.9 (d, J_{PC} = 56 Hz), 22.3, 21.4, 16.7 (d, J_{POCC} = 5 Hz), 14.0; ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 149.0 (q, J_{PB} = 81 Hz); ^{11}B NMR ($CDCl_3$, 28.88 MHz) δ - 43.6 (dq , J_{BP} = 81 Hz, J_{BH} = 94 Hz); HRMS calcd. for $C_9H_{28}BNO_2P$, $[M + NH_4]^+$ 224.1951, found 224.1944.

Monodeuterated Diethoxy pentylphosphonite-borane. The title compound was prepared from diethoxy (chloromethyl)phosphine-borane **4i** (3.2 mmol, 590 mg, 1.0 equiv) and Bu_3B (3.2 mL, 1.0 M solution in diethyl ether, 3.2 mmol, 1.0 equiv). The reaction mixture was quenched by addition of D_2O. The resulting biphasic mixture was stirred for 2 h at rt. Purification of the crude product by chromatography on silica gel (EtOAc/hexanes 0.5 : 99.5, v/v) yielded diethoxy pentylphosphonite-borane (2.94 mmol, 610 mg, 92%). Deuterium incorporation 95 %.
^{1}H NMR ($CDCl_3$, 300 MHz) δ 3.95 - 4.16 (m, 4 H), 1.21 - 1.75 (m, 7 H), 1.30 (t, J = 7 Hz, 6 H), 0.90 (t, J = 7 Hz, 3 H), 0.50 (q, J_{BH} = 92 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 63.2 (d, J_{POC} = 4 Hz), 33.0 (d, J_{PCCC} = 14 Hz), 29.7 (dt, J_{PC} = 52 Hz, J_{DC} = 19 Hz), 22.3, 21.3, 16.8 (d, J_{POCC} = 5 Hz), 14.0; ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 149.0 (q, J_{PB} = 80 Hz). ^{11}B NMR ($CDCl_3$, 28.88 MHz) δ - 43.7 (dq , J_{BP} = 80 Hz, J_{BH} = 92 Hz); HRMS calcd. for $C_9H_{27}DBNO_2P$, ($[M + NH_4]^+$) 225.2014, found 225.2010.

Diethoxy 1–methylpentylphosphonite-borane (Table 1, Entry 9). The title compound was prepared from diethoxy 1-chloroethylphosphine-borane **4j** (3.2 mmol, 634 mg, 1.0 equiv) and Bu_3B (3.2 mL, 1.0 M solution in diethyl ether, 3.2 mmol, 1.0 equiv). Purification of the crude product by chromatography on silica gel (EtOAc/hexanes 0.5 : 99.5, v/v) yielded diethoxy 1–methylpentylphosphonite-borane (2.75 mmol, 606 mg, 86 %).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 3.91 - 4.20 (m, 4H), 1.37 - 1.79 (m, 7 H), 1.31 (t, J = 7 Hz, 6 H), 1.12 (d, J = 7 Hz, 3 H), 0.91 (t, J = 7 Hz, 3 H), 0.50 (q, J_{BH} = 93 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 63.3 (d, J_{POC} = 5 Hz), 33.7 (d, J_{PC} = 57 Hz), 29.5 (d, J_{PCCC} = 12 Hz), 28.5 (d, J_{PCC} = 2 Hz) 22.5, 16.6 (d, J_{POCC} = 6 Hz), 13.9, 12.1; ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 151.1 (q, J_{PB} = 79 Hz); ^{11}B NMR ($CDCl_3$, 28.88 MHz) δ - 44.1 (dq , J_{BP} = 79 Hz, J_{BH} = 93 Hz).); HRMS calcd. for $C_{10}H_{30}BNO_2P$, ($[M + NH_4]^+$): 238.2107, found 238.2105.

Diphenyl pentylphosphine-borane (Table 1, entry 10). The title compound was prepared from diphenyl (chloromethyl)phosphine-borane **4k** (2.1 mmol, 521 mg, 1.0 equiv) and Bu_3B (2.1 mL, 1.0 M solution in diethyl ether, 2.1 mmol, 1.0 equiv). Purification of the crude product by chromatography on silica gel (EtOAc/hexanes 1:9, v/v) yielded diphenyl pentylphosphine-borane (1.30 mmol, 352 mg, 62 %).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.40 - 7.70 (m, 10 H), 2.14 - 2.23 (m, 2 H), 1.23 - 1.56 (m, 6 H), 0.85 (t, J = 7 Hz, 3 H), 0.50 (q, J_{BH} = 98 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 132.4 (d, J_{PCCC} = 10 Hz, 4 C), 131.3 (2 C), 129.9 (d, J_{PC} = 55 Hz, 2 C), 129.0 (d, J_{PCC} = 10 Hz, 4 C) , 33.5 (d, J_{PCCC} = 14 Hz), 25.8 (d, J_{PC} = 38 Hz) 22.9, 22.3, 14.1; ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 17.0 (m); ^{11}B NMR ($CDCl_3$, 28.88 MHz) δ – 40.4 (m); HRMS calcd. for $C_{17}H_{28}BNP$, $[M + NH_4 – H_2]$: 286.1896, found 286.1899.

Diethyl cyclohexylmethylphosphonate (Table 2, entry 1).[21] The title compound was prepared from diethyl (chloromethyl)phosphonate **4b** (4.0 mmol, 746 mg, 1.0 equiv) and Cy_3B (4.0 mL, 1.0 M solution in diethyl ether, 4.0 mmol, 1.0 equiv). Purification of the crude product by chromatography on silica gel (EtOAc/hexanes 1:1, v/v) yielded diethyl cyclohexylmethylphosphonate (3.32 mmol, 777 mg, 83 %).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 4.00 - 4.18 (m, 4 H), 1.89 (d, J = 13 Hz, 2 H), 1.33 (t, J = 7 Hz, 6 H), 1.26 - 1.89 (m, 11 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 61.2 (d, J_{POC} = 7 Hz), 34.5 (d, J_{PCCC} = 11 Hz, 2 C), 33.2 (d, J_{PC} = 138 Hz), 32.6 (d, J_{PCC} = 4 Hz), 26.1, 26.0 (d, J_{PCCCC} = 6 Hz, 2 C), 16.5 (d, J_{POCC} = 6 Hz); ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 33.0.

Diethyl 2-methyl-butylphosphonate (Table 2, entry 2). The title compound was prepared from diethyl (chloromethyl)phosphonate **4b** (4.0 mmol, 746 mg, 1.0 equiv) and (*sec*-Bu)$_3$B (4.0 mL, 1.0 M solution in diethyl ether, 4.0 mmol, 1.0 equiv). Purification of the crude product by chromatography on silica gel (EtOAc/hexanes 1:1, v/v) yielded diethyl 2-methyl-butylphosphonate (3.44 mmol, 716 mg, 86 %).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 4.06 - 4.14 (m, 4 H), 1.25 - 1.95 (m, 5 H), 1.34 (t, J = 7 Hz, 6 H), 1.05 (d, J = 7 Hz, 3 H) 0.89 (t, J = 7 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 62.3 (t, J_{POC} = 6 Hz), 30.7 (d, J_{PC} = 139 Hz), 31.2 (d, J_{PCCC} = 14 Hz), 29.7 (d, J_{PCC} = 4

Hz), 20.3 (d, J_{PCCC} = 7 Hz) 16.4 (d, J_{POCC} = 6 Hz), 10.9; ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 33.3; HRMS (EI^+) calcd. for $C_9H_{22}O_3P$, ($[M]^+$) 209.1307, found 209.1308.

Diethyl (2-phenylethyl)phosphonate (Table 2, entry 3).[22] The title compound was prepared from diethyl (chloromethyl)phosphonate **4b** (4.0 mmol, 746 mg, 1.0 equiv) and B-benzyl-9-BBN (8.0 mL, 0.5 M solution in THF, 4.0 mmol, 1.0 equiv). Purification of the crude product by chromatography on silica gel (EtOAc/hexanes 1:1, v/v) yielded diethyl (2-phenylethyl)phosphonate (2.76 mmol, 668 mg, 69 %).
^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.20 - 7.33 (m, 5 H), 4.05 - 4.16 (m, 4 H), 2.88 – 2.97 (m, 2 H), 2.60 (t, J = 17 Hz, 1 H), 2.06 (t, , J = 17 Hz, 1 H), 1.33 (t, J = 7 Hz, 6 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 141.1 (d, J_{PCCC} = 17 Hz), 128.8 (2 C), 128.3 (2 C), 126.6, 61.8 (d, J_{POC} = 7 Hz), 28.3 (d, J_{PCC} = 6 Hz), 27.8 (d, J_{PC} = 139 Hz), 16.7 (d, J_{POCC} = 6 Hz); ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 31.9.

Diethyl (cyclooctylmethyl)phosphonate (Table 2, entry 4). The title compound was prepared from diethyl (chloromethyl)phosphonate **4b** (4.0 mmol, 746 mg, 1.0 equiv) and Alpine-borane (8.0 mL, 0.5 M solution in THF, 4.0 mmol, 1.0 equiv). Purification of the crude product by chromatography on silica gel (EtOAc/hexanes 1:1, v/v) yielded diethyl (cyclooctylmethyl)phosphonate ((3.32 mmol, 870 mg, 83 %).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 4.00 - 4.18 (m, 4 H), 1.31 (t, J = 6 Hz, 6 H), 1.25– 2.05 (m, 17 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 61.2 (d, J_{POC} = 6 Hz), 33.5 (d, J_{PC} = 138 Hz), 33.0 (d, J_{PCCC} = 11 Hz, 2 C), 32.5, 27.3 (2 C), 26.0, 24.9 (2 C), 16.5 (d, J_{POCC} = 6 Hz); ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 33.3. HRMS calcd. for $C_{13}H_{27}O_3P$, 262.1698, found 209.1700.

Diethyl (1,2-diphenyl)ethylphosphonate (Table 2, entry 5).[23] The title compound was prepared from diethyl chloro-phenyl-methylphosphonate **4f** (4.0 mmol, 1.05 g, 1.0 equiv) and B-benzyl-9-BBN (8.0 mL, 1.0 M solution in THF, 4.0 mmol, 1.0 equiv). Purification of the crude product by chromatography on silica gel (EtOAc/hexanes 1:1, v/v) yielded diethyl (1,2-diphenyl)ethylphosphonate (2.36 mmol, 751 mg, 59 %).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 6.99 - 7.34 (m, 10 H), 3.65 - 4.12 (m, 4 H), 3.13 – 3.50 (m, 3 H), 1.28 (t, J = 7 Hz, 3 H), 1.09 (t, J = 7 Hz, 3 H). ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 139.3 (d, J_{PCCC} = 16 Hz), 135.7 (d, J_{PCC} = 6 Hz),129.7 (d, J_{PCCC} = 7 Hz, 2 C),128.8 (2 C), 128.6 (2 C), 128.4 (2 C), 127.4, 126.4, 63.0 (d, J_{POC} = 7 Hz), 62.1 (d, J_{POC} = 7 Hz), 46.8 (d, J_{PC} = 136 Hz), 36.5, 16.6 (d, J_{POCC} = 6 Hz), 16.4 (d, J_{POCC} = 6 Hz). ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 29.3.

Diethoxy 2–methylbutylphosphonite-borane (Table 2, entry 6). The title compound was prepared from diethoxy (chloromethyl)phosphine-borane **4i** (3.2 mmol, 590 mg, 1.0 equiv) and (*sec*-Bu)$_3$B (3.2 mL, 1.0 M solution in diethyl ether, 3.2 mmol, 1.0 equiv). Purification of the crude product by chromatography on silica gel (EtOAc/hexanes 0.5 : 99.5, v/v) yielded diethoxy 2–methylbutylphosphonite-borane (2.62 mmol, 541 mg, 82 %).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 3.95 - 4.20 (m, 4 H), 1.26 - 1.93 (m, 5 H), 1.30 (t, J = 7 Hz, 6 H), 1.01 (d, J = 7 Hz, 3 H) 0.89 (t, J = 7 Hz, 3 H), 0.50 (qd, J_{BH} = 94 Hz, J_{PBH} = 14 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 63.1 (d, J_{POC} = 4 Hz), 36.9 (d, J_{PC} = 54 Hz),

31.2 (d, J_{PCCC} = 10 Hz), 29.2, 20.8 (d, J_{PCCC} = 6 Hz), 16.7 (d, J_{POCC} = 6 Hz), 11.2; ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 149.5 (q, J_{PB} = 81 Hz); ^{11}B NMR ($CDCl_3$, 28.88 MHz) δ - 43.7 (dq , J_{BP} = 81 Hz, J_{BH} = 94 Hz); HRMS calcd. for $C_9H_{28}BNO_2P$, ($[M + NH_4]^+$) 224.1951, found 224.1952.

Diethoxy octylphosphonite-borane (Table 2, Entry 7). The title compound was prepared from diethoxy (chloromethyl)phosphine-borane **4i** (3.2 mmol, 590 mg, 1.0 equiv) and (heptyl)$_3$B (3.2 mL, 1.0 M solution in diethyl ether, 3.2 mmol, 1.0 equiv). Purification of the crude product by chromatography on silica gel (EtOAc/hexanes 0.5 : 99.5, v/v) yielded diethoxy octylphosphonite-borane (2.34 mmol, 580 mg, 73 %).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 3.94 - 4.16 (m, 4H), 1.23 - 1.76 (m, 14 H), 1.31 (t, J = 7 Hz, 6 H), 0.88 (t, J = 7 Hz, 3 H), 0.50 (q, J_{BH} = 94 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 63.0 (d, J_{POC} = 4 Hz), 31.8, 33.0 (d, J_{PCCC} = 15 Hz), 30.3 (d, J_{PC} = 56 Hz), 29.1 (2 C), 22.6, 21.6, 16.7 (d, J_{POCC} = 5 Hz); 14.1. ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 149.1 (q, $J_{PB=}$ 81 Hz); ^{11}B NMR ($CDCl_3$, 28.88 MHz) δ - 43.6 (dq , J_{BP} = 81 Hz, J_{BH} = 94 Hz); HRMS calcd. for $C_{12}H_{34}BNO_2P$, ($[M + NH_4]^+$) 266.2420, found 266.2413.

Diethoxy 2 – phenylethylphosphonite-borane (Table 2, Entry 8). The title compound was prepared from diethoxy (chloromethyl)phosphine-borane **4i** (3.2 mmol, 590 mg, 1.0 equiv) and B–benzyl-9-BBN (3.2 mL, 1.0 M solution in diethyl ether, 3.2 mmol, 1.0 equiv). Purification of the crude product by chromatography on silica gel (EtOAc/hexanes 0.5 : 99.5, v/v) yielded diethoxy 2 – phenylethylphosphonite-borane (2.27 mmol, 545 mg, 71 %).
^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.18 - 7.34 (m, 5 H), 3.96 - 4.23 (m, 4 H), 2.84 - 2.92 (m, 2 H), 2.02 - 2.12 (m, 2 H), 1.30 (t, J = 7 Hz, 6 H), 0.50 (q, J_{BH} = 92 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 141.2 (d, J = 14 Hz), 128.6 (2 C), 128.3 (2 C), 126.6, 63.4 (d, J_{POC} = 5 Hz), 31.9 (d, J_{PC} = 54 Hz), 28.0, 16.8 (d, J_{POCC} = 6 Hz); ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 147.6 (q, J_{PB} = 80 Hz); ^{11}B $CDCl_3$, 28.88 MHz) δ – 43.7 (dq, J_{BP} = 80 Hz, J_{BH} = 92 Hz); HRMS calcd. for $C_{12}H_{26}BNO_2P$, ($[M + NH_4]^+$): 258.1794, found 258.1796.

Diethoxy nonylphosphonite-borane (Table 2, entry 9). The title compound was prepared from diethoxy (chloromethyl)phosphine-borane **4i** (3.2 mmol, 590 mg, 1.0 equiv) and 9–octyl-BBN (3.2 mL, 1.0 M solution in diethyl ether, 3.2 mmol, 1.0 equiv). Purification of the crude product by chromatography on silica gel (EtOAc/hexanes 0.5:99.5, v/v) yielded diethoxy nonylphosphonite-borane (1.98 mmol, 520 mg, 62 %).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 3.94 - 4.18 (m, 4 H), 1.23 - 1.76 (m, 16 H), 1.30 (t, J = 7 Hz, 6 H), 0.89 (t, J = 7 Hz, 3 H), 0.50 (q, J_{BH} = 94 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 63.2 (d, J_{POC} = 5 Hz) 32.1, 30.9 (d, J_{PCCC} = 14 Hz), 30.4, 30.1 (d, J_{PC} = 38 Hz), 29.6, 29.5, 29.3, 22.9, 21.8, 16.8 (d, J_{POCC} = 6 Hz), 14.3; ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 149.0 (q, J_{PB} = 81 Hz); ^{11}B ($CDCl_3$, 28.88 MHz) δ - 43.6 (dq, J_{BP} = 81 Hz, J_{BH} = 94 Hz); HRMS calcd. for $C_{13}H_{36}BNO_2P$, ($[M + NH_4]^+$) 280.2577, found 280.2571.

Diphenyl propylphosphine-borane (Table 2, entry 10). The title compound was prepared from Diphenyl (chloromethyl)phosphine-borane **4k** (2.1 mmol, 521 mg, 1.0 equiv) and Et_3B (2.1 mL, 1.0 M solution in THF, 2.1 mmol, 1.0 equiv). Purification of

the crude product by chromatography on silica gel (EtOAc/hexanes 1:9, v/v) yielded diphenyl propylphosphine-borane (1.78 mmol, 432 mg, 85 %).
^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.40 - 7.70 (m, 10 H), 2.12 - 2.29 (m, 2 H), 1.48 - 1.63 (m, 2 H), 1.00 (t, J = 7 Hz, 3 H), 0.80 (q, J_{BH} = 98 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 132.4 (d, J_{PCCC} = 10 Hz, 4 C), 131.3 (2 C), 129.9 (d, J_{PC} = 55 Hz), 129.0 (d, J_{PCC} = 10 Hz, 4 C), 28.0 (d, J_{PC} = 37 Hz), 17.0, 16.1 (d, J_{PCCC} = 15 Hz); ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 16.6 (q, J_{PB} = 58 Hz); ^{11}B NMR ($CDCl_3$, 28.88 MHz) δ - 39.7 (dq , J_{BP} = 58 Hz, J_{BH} = 98 Hz), HRMS calcd. for $C_{15}H_{22}BNP$, [M + NH_4 – H_2]: 258.1583, found 258.1579.

General procedure for the conversion of phosphinite-boranes in to *H*–phosphinate esters.

To a 0.2 M solution of phosphinite-borane in dry CH_2Cl_2 at 0 °C, was added tetrafluoroboric acid diethyl ether complex (3.0 equiv). An exothermic reaction ensued and gas evolved. The reaction was then warmed to rt and stirred for additional 6 h. Subsequently, the mixture was cooled to 0 °C and saturated aqueous sodium bicarbonate solution was slowly added. The resulting biphasic mixture was stirred vigorously for 5 – 10 min and poured into separatory funnel. The organic layer was separated and aqueous layer was extracted with EtOAc (3 X). The combined organic layers were dried with $MgSO_4$, and concentrated in vacuo to give *H*-phosphinate.
Ethyl octyl-*H*-phosphinate (5, Scheme 5).[24] The title compound was prepared from diethoxy octylphosphinite-borane (1.6 mmol, 400 mg, 1.0 equiv) and tetrafluoroboric acid diethyl ether complex (4.8 mmol, 777 mg, 653 μl, 3.0 equiv) in 96 % yield (1.54 mmol, 317 mg).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.09 (d, J = 527 Hz, 1 H), 4.03 - 4.23 (m, 2 H), 1.27 - 1.80 (m, 14 H), 1.36 (t, J = 7 Hz, 3 H), 0.88 (t, J = 7 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 62.5 (d, J_{POC} = 7 Hz), 31.8, 30.4 (d, J_{PCCC} = 15 Hz), 29.1, 29.0, 28.6 (d, J_{PC} = 93 Hz), 22.6, 20.7, 16.2 (d, J_{POCC} = 6 Hz), 14.0; ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 40.7 (dm, J = 530 Hz).

Ethyl 2-methylbutyl-*H*-phosphinate (6, Scheme 5) . The title compound was prepared from diethoxy 2-methylbutylphosphinite-borane (1.6 mmol, 330 mg, 1.0 equiv) and tetrafluoroboric acid diethyl ether complex (4.8 mmol, 777 mg, 653 μl, 3.0 equiv) in 96 % yield (1.54 mmol, 253 mg).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.20 (d, J = 527 Hz, 1 H), 4.01 - 4.27 (m, 2 H), 1.25 - 1.96 (m, 5 H), 1.38 (t, J = 7 Hz, 3 H), 1.10 (d, J = 15 Hz, 3 H) 0.92 (t, J = 7 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 62.5 (d, J_{POC} = 4 Hz), 35.6 (d, J_{PC} = 93 Hz), 30.9 (d J_{PCCC} = 13 Hz), 29.0, 20.5 (d, J_{PCC} = 7 Hz), 16.4 (d, J_{POC} = 6 Hz), 11.1; ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 39.5, 39.2 (dm, J = 527 Hz). HRMS (EI^+) calcd. for $C_7H_{18}O_2P$, ($[M]^+$) 165.1044, found 165.1043.

Ethyl pentyl-*H*-phosphinate (7, Scheme 5). The title compound was prepared from diethoxy pentylphosphinite-borane (1.0 mmol, 207 mg, 1.0 equiv) and tetrafluoroboric

acid diethyl ether complex (1.0 mmol, 486 mg, 408 μl, 3.0 equiv) in 95 % yield (0.95 mmol, 157 mg), 95 % of deuterated compound.
^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.09 (d, J = 524 Hz, 1 H), 4.01 - 4.26 (m, 2 H), 1.26 - 1.83 (m, 7 H), 1.38 (t, J = 7 Hz, 3 H), 0.91 (t, J = 7 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 62.4(d, J_{POC} = 5 Hz), 32.6 (d, J_{PCCC} = 16 Hz), 28.3 (dt, J_{PC} = 94 Hz, J_{DC} = 19 Hz), 22.2, 20.4, 16.3 (d, J_{POCC} = 6 Hz), 13.8; ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 40.3 (dm, J = 527 Hz); HRMS (EI^+) calcd. for $C_7H_{17}DO_2P$ ($[M]^+$) 166.1107, found 166.1109.

Ethyl 2-phenylethyl-*H*-phosphinate (8, Scheme 5). The title compound was prepared from diethoxy 2 - phenylethylphosphinite-borane (1.2 mmol, 288 mg, 1.0 equiv.) and tetrafluoroboric acid diethyl ether complex (3.6 mmol, 583 mg, 490 μl, 3.0 equiv.) in 98 % yield (1.18 mmol, 233 mg).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.20 - 7.31 (m, 5 H), 7.11 (d, J = 532 Hz, 1 H), 4.02 - 4.26 (m, 2 H), 2.87 – 2.98 (m, 2 H), 2.05 - 2.16 (m, 2 H), 1.38 (t, J = 7 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 140.1 (d, J_{PCCC} = 15 Hz), 128.7 (2 C), 128.1 (2 C), 126.5, 62.6 (d, J_{POC} = 6 Hz), 31.3 (d, J_{PC} = 93 Hz), 26.9, 16.2 (d, J_{POCC} = 6 Hz); ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 38.2 (d, J = 534 Hz), HRMS calcd. for $C_{10}H_{15}O_2P$, 198.0810, found 198.0810.

Ethyl allyl-octylphosphinate (9, Scheme 5). Method B. A flame-dried, 50 mL, three-necked, round-bottomed flask charged with diethoxy octylphosphinite-borane (1.6 mmol, 400 mg, 1.0 equiv) *N*-methylpiperazine (12.8 mmol, 1.28 g, 1.4 mL, 8.0 equiv) and dry toluene (10 mL). The solution was heated at reflux for 24 h. After cooling to rt, the mixture was concentrated in high vacuo to remove access of *N*-methylpiperazine. The residue was diluted with dry toluene (20 mL) and allyl bromide (3.2 mmol, 387 mg, 270 μl, 2.0 equiv) was added. The reaction mixture was heated under reflux for 3 h. After cooling to rt, the mixture was washed with H_2O, the aqueous phase was extracted with EtOAc (3 X) and the combined organic fractions were dried with $MgSO_4$, concentrated in vacuo. Purification of the crude product by chromatography on silica gel (EtOAc) yielded ethyl allyl-octylphosphinate **9** (0.94 mmol, 231 mg, 59 %).
Method C. A flame-dried, seal tube was purged with nitrogen, charged with diethoxy octylphosphonite-borane (1.6 mmol, 400 mg, 1.0 equiv) and diethylamine (40 mL). The solution was heated at 55^{o}C for 3 days. After cooling to rt, the mixture was concentrated in high vacuo. The residue was diluted with dry toluene (20 mL) and transferred *via* cannula under nitrogen to the flame-dried, 50 mL, three-necked, round-bottomed flask. Allyl bromide (8.0 mmol, 968 mg, 678 μl, 5.0 equiv) was than added and the mixture was heated under reflux for 3 h. After cooling to rt, the reaction mixture was washed with H_2O, the aqueous phase was extracted with EtOAc (3 X) and the combined organic fractions were dried with $MgSO_4$, concentrated in vacuo. Purification of the crude product by chromatography on silica gel (EtOAc) yielded ethyl allyl-octylphosphinate **9** (1.04 mmol, 256 mg, 65 %).
^{1}H NMR ($CDCl_3$, 300 MHz) δ 5.74 - 5.90 (m, 1 H), 5.16 - 5.25 (m, 2 H), 4.02 - 4.15 (m, 2 H), 2.57 (dd, J = 8 Hz, J = 10 Hz, 2 H), 1.27 - 1.77 (m, 14 H), 1.32 (t, J = 7 Hz, 6 H), 0.88 (t, J = 7 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 128.3 (d, J_{PCC} = 9 Hz), 120.0 (d, J_{PCCC} = 12 Hz), 60.5 (d, J_{POC} = 7 Hz), 34.6 (d, J_{PC} = 85 Hz), 32.0, 31.0 (d, J_{PCCC} = 15 Hz), 29.2, 27.6 (d, J_{PC} = 93 Hz), 22.8, 21.8 (d, J_{PCC} = 4 Hz), 16.8 (d, J_{POCC} = 6 Hz), 14.3; ^{31}P

NMR ($CDCl_3$, 121.47 MHz) δ 54.9; HRMS calcd. for $C_{13}H_{27}O_2P$; 246.1749, found 246.1750.

Procedure for the synthesis of α-iodo phosphonates.

Diethyl 1-iodopentylphosphonate (3b-I, Scheme 6). A flame-dried, 50 mL, three-necked, round-bottomed flask was purged with nitrogen, charged with diethyl (chloromethyl)phosphonate (4.0 mmol, 746 mg, 1.0 equiv) and dry THF (20 mL). The solution was cooled below - 90 ^oC (liquid nitrogen/ethanol bath) and *n*-butyllitium (2.5 mL, 1.6 M solution in hexane, 4.0 mmol, 1.0 equiv) was added slowly *via* syringe followed by Bu_3B (4.0 mL, 1.0 M solution in diethyl ether, 4.0 mmol, 1.0 equiv) in one portion. The reaction mixture was warmed slowly to rt and I_2 (4.8 mmol, 1.2 g, 1.2 equiv) was added in one portion. The resulting mixture was stirred at reflux for 2 h, after cooling to rt, was diluted with EtOAc washed with aqueous solution of sodium thiosulfate (1 X) and water (1 X). Organic layer was dried with $MgSO_4$, and solvents removed in vacuo. Purification of the crude product by chromatography on silica gel (EtOAc/hexanes 3:7, v/v) yielded diethyl pentylphosphonate (3.05 mmol, 1.02 g, 76 %).
^1H NMR ($CDCl_3$, 300 MHz) δ 4.14 - 4.25 (m, 4 H), 3.69 - 3.79 (m, 1 H), 1.31 - 2.04 (m, 7 H), 1.35 (t, J = 7 Hz, 6 H), 0.92 (t, J = 7 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75.45 MHz) δ 63.7 (q, J_{POC} = 7 Hz), 32.9 (d, J_{PCC} = 2 Hz), 32.3 (d, J_{PCCC} = 13 Hz), 21.7, 18.2 (d, J_{PC} = 154 Hz), 16.6 (d, J_{POCC} = 6 Hz), 14.0; ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 23.2; HRMS (EI^+) calcd. for $C_9H_{20}IO_3P$, ($[M]^+$) 334.0195, found 334.0201.

Diethyl 1–iodo–2-methylbutylphosphonate (Scheme 6). A flame-dried, 50 mL, three-necked, round-bottomed flask was purged with nitrogen, charged with diethyl (chloromethyl)phosphonate (4.0 mmol, 746 mg, 1.0 equiv) and dry THF (20 mL). The solution was cooled below – 90^oC (liquid nitrogen/ethanol bath) and *n*-butyllitium (2.5 mL, 1.6 M solution in hexane, 4.0 mmol, 1.0 equiv) was added slowly *via* syringe followed by (*sec*-Bu)$_3$B (4.0 mL, 1.0 M solution in diethyl ether, 4.0 mmol, 1.0 equiv) in one portion. The reaction mixture was warmed slowly to rt and I_2 (4.8 mmol, 1.2 g, 1.2 equiv) was added in one portion. The resulting mixture was stirred at reflux for 2 h, after cooling to rt, was diluted with EtOAc washed with aqueous. solution of sodium thiosulfate (1 X) and water (1 X). Organic layer was dried with $MgSO_4$, and solvents removed in vacuo. Purification of the crude product by chromatography on silica gel (EtOAc/hexane 3:7 v/v) yielded diethyl pentylphosphonate (2.88 mmol, 962 mg, 72%), mixture of diastereoisomers (50/50)
^1H NMR ($CDCl_3$, 300 MHz) δ 4.15 - 4.26 (m, 4 H), 3.95 (2dd, J = 13 Hz, J = 2 Hz, 1 H), 1.70 - 1.91 (m, 1 H), 1.36 (t, J = 7 Hz, 6 H), 1.15 - 1.26 (m, 2 H), 1.02 (t, J = 6 Hz, 3 H) 0.91 (2t, J = 7 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 100.57 MHz) δ 63.7 (dd, J_{POC} = 7 Hz, J = 3 Hz,), 63.3 (q, J_{POC} = 7 Hz), 36.4, 35.2, 30.7 (d, J_{PCCC} = 16 Hz), 29.4 (d, J_{PC} = 150 Hz), 28.7 (d, J_{PC} = 150 Hz), 28.1, 19.7 (d, J_{PCCC} = 15 Hz), 18.9, 16.4 (d, J_{POCC} = 6 Hz), 11.6, 11.3; ^{31}P NMR ($CDCl_3$, 121.47 MHz) δ 22.5, 22.2; HRMS (EI^+) calcd. for $C_9H_{20}IO_3P$, ($[M]^+$) 334.0195, found 334.0197.

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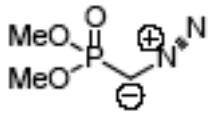

4a

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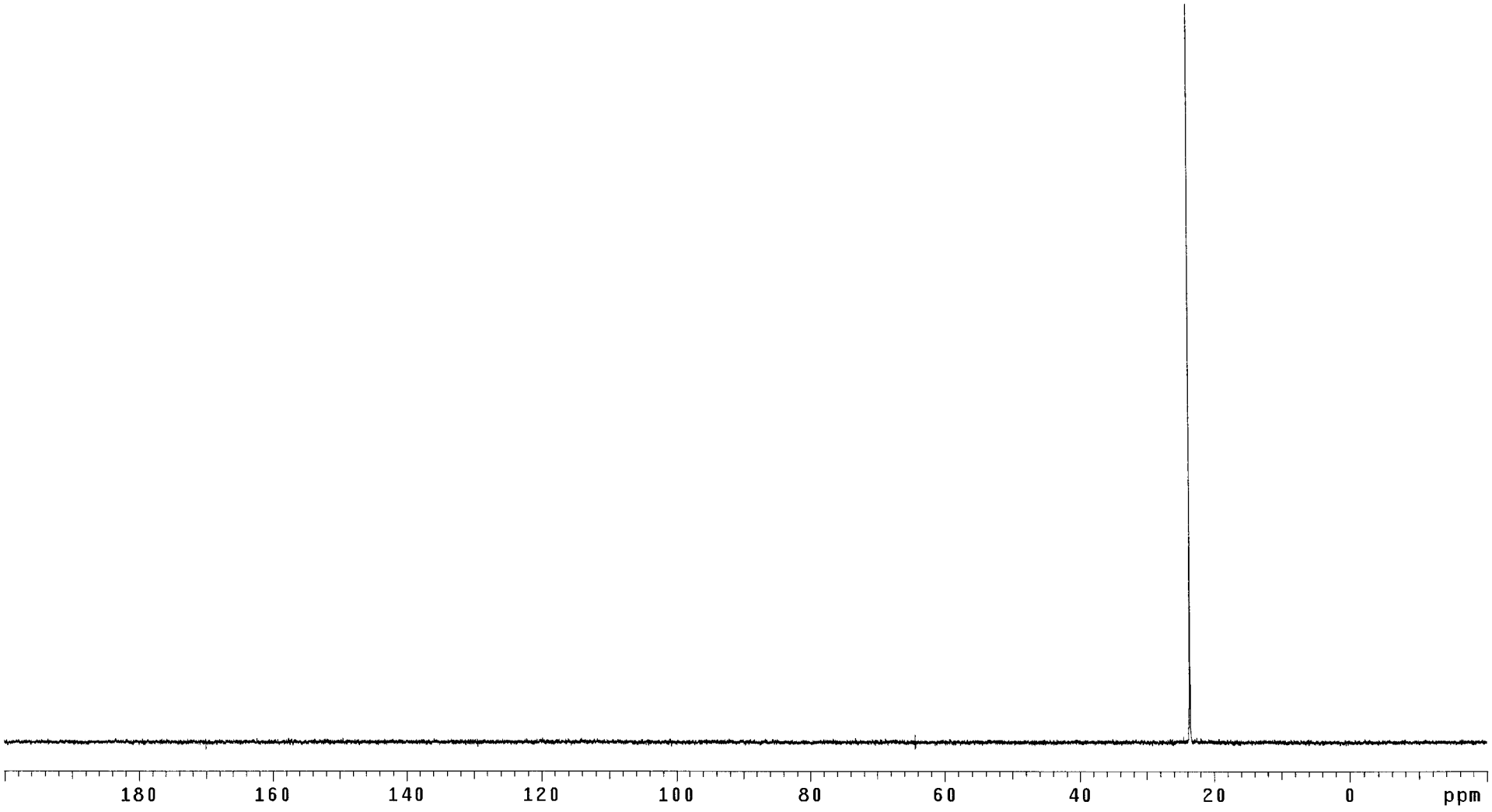

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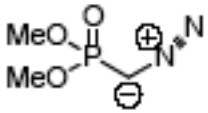

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6	1149.173	3.830	5.3
7	1148.587	3.828	5.5
8	1147.707	3.825	5.7
9	1147.121	3.823	6.8
10	1145.655	3.818	10.4
11	1145.069	3.816	14.2
12	1143.310	3.810	142.3
13	1141.844	3.805	5.6
14	1141.258	3.804	4.5
15	1139.792	3.799	21.0
16	1138.326	3.794	3.6
17	1137.740	3.792	3.7
18	1136.860	3.789	3.7
19	1136.274	3.787	4.4
20	1135.688	3.785	4.8
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22	1134.222	3.780	7.1
23	1132.756	3.775	14.4
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26	1128.652	3.762	17.6
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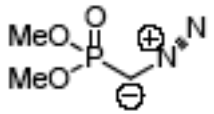

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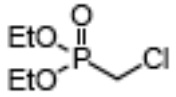

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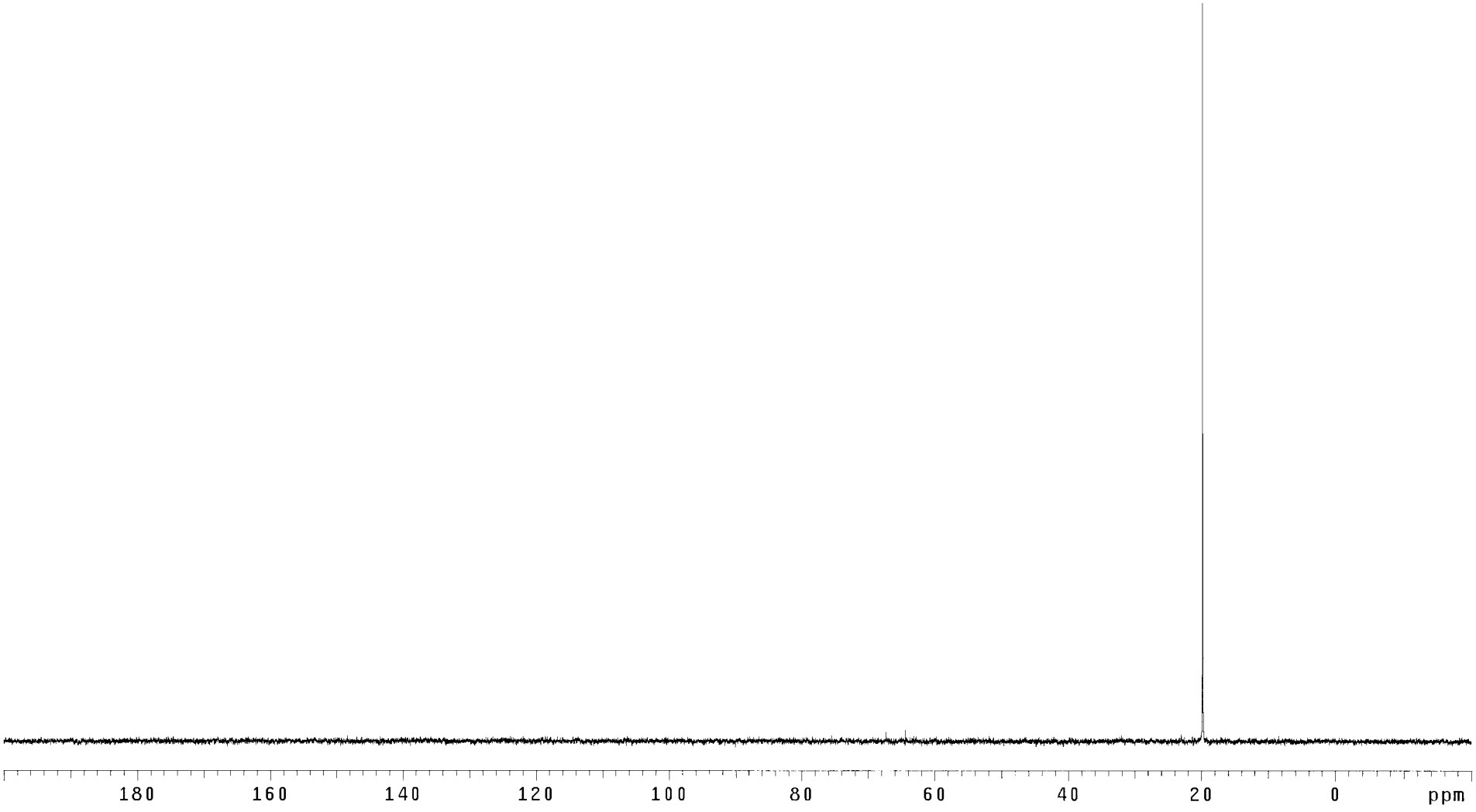

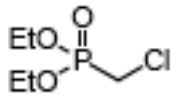

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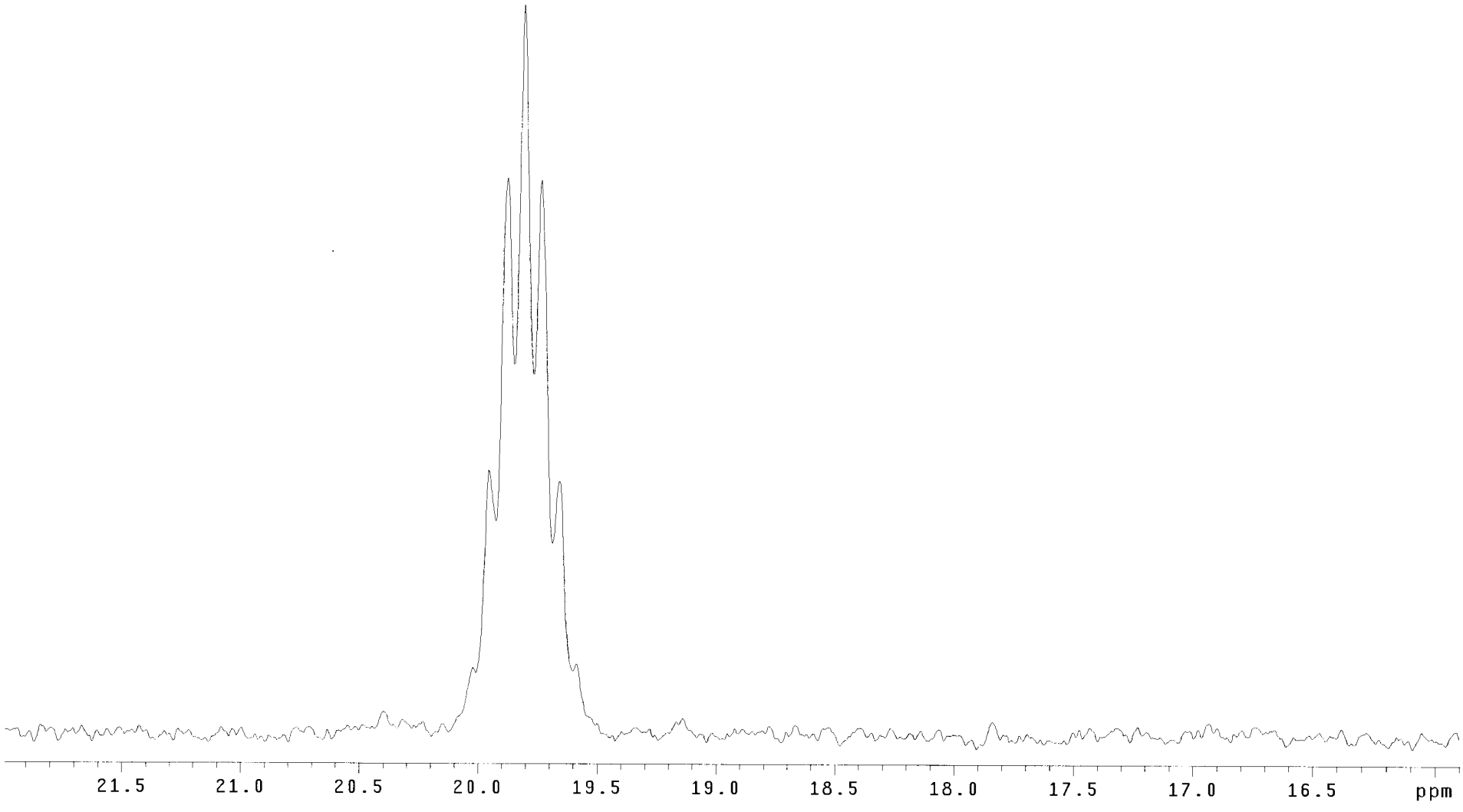

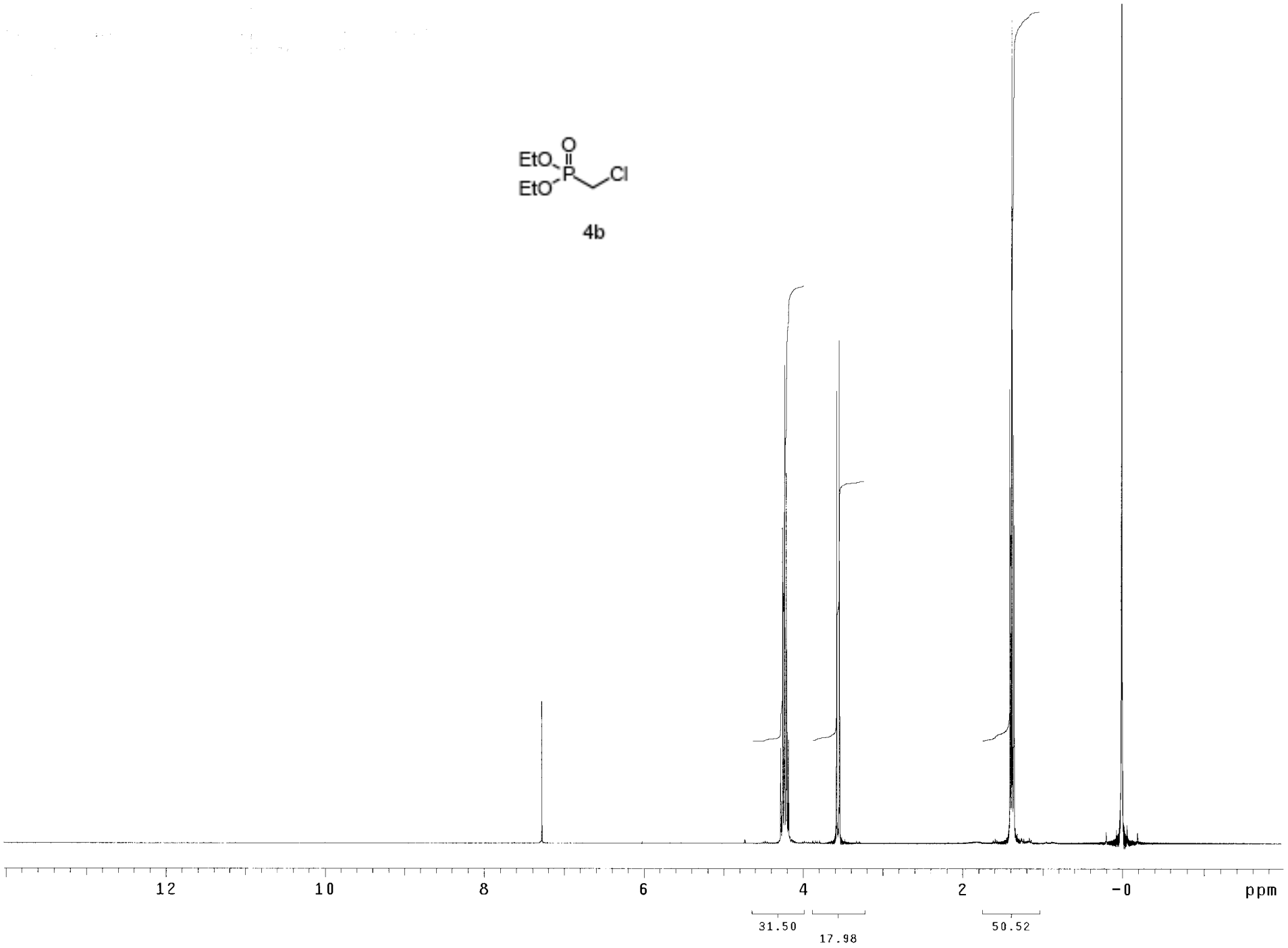
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Cl
EtO
4b
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10
8
6
4
2
-0
ppm
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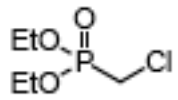
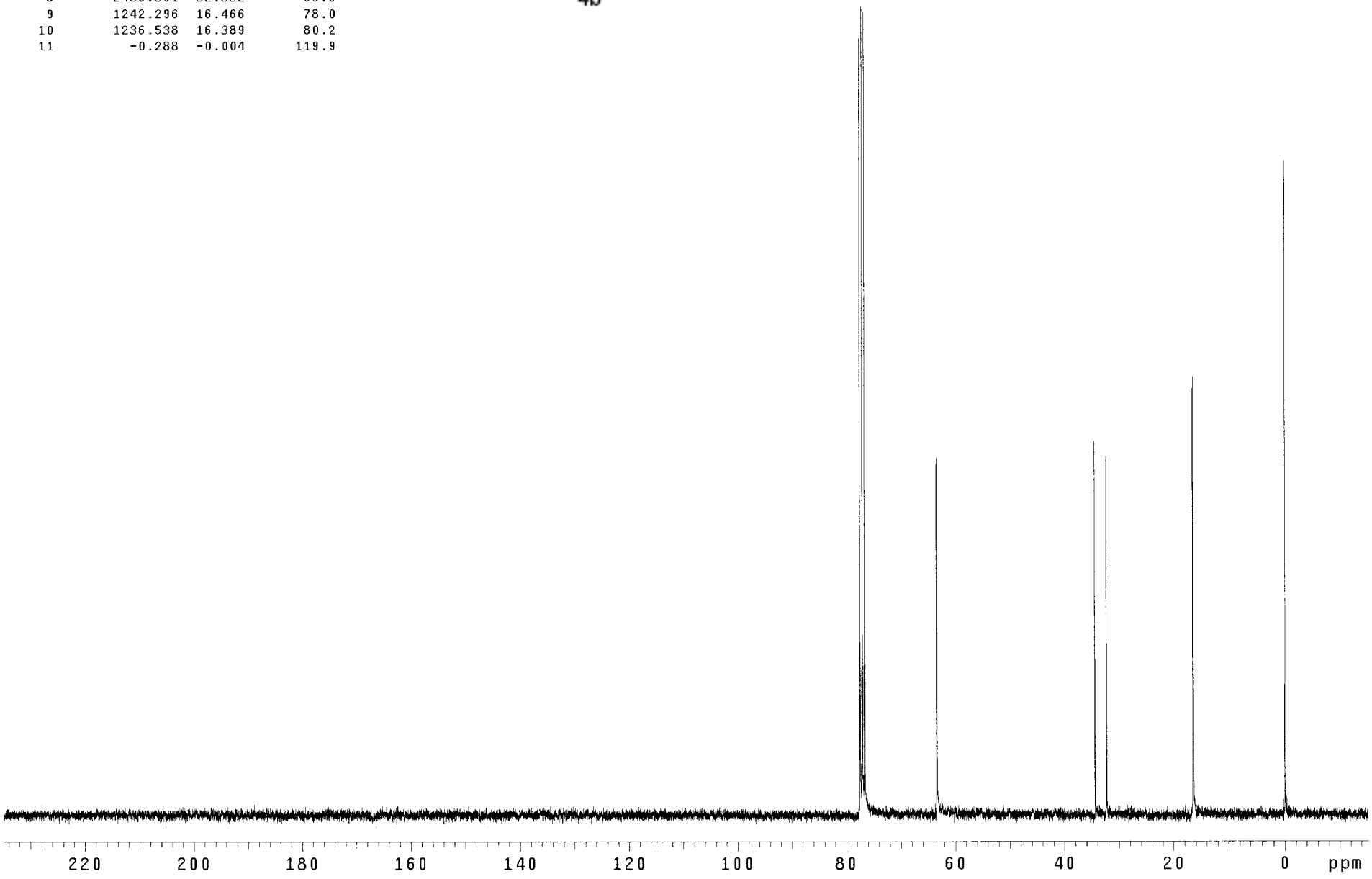
17.98
50.52

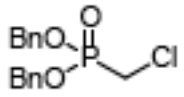
INDEX	FREQUENCY	PPM	HEIGHT
1	5844.692	77.467	142.4
2	5827.994	77.245	9.1
3	5812.735	77.043	148.2
4	5780.778	76.619	147.3
5	4787.805	63.458	64.1
6	4781.471	63.374	65.3
7	2599.176	34.450	68.3
8	2439.391	32.332	65.6
9	1242.296	16.466	78.0
10	1236.538	16.389	80.2
11	-0.288	-0.004	119.9



4b

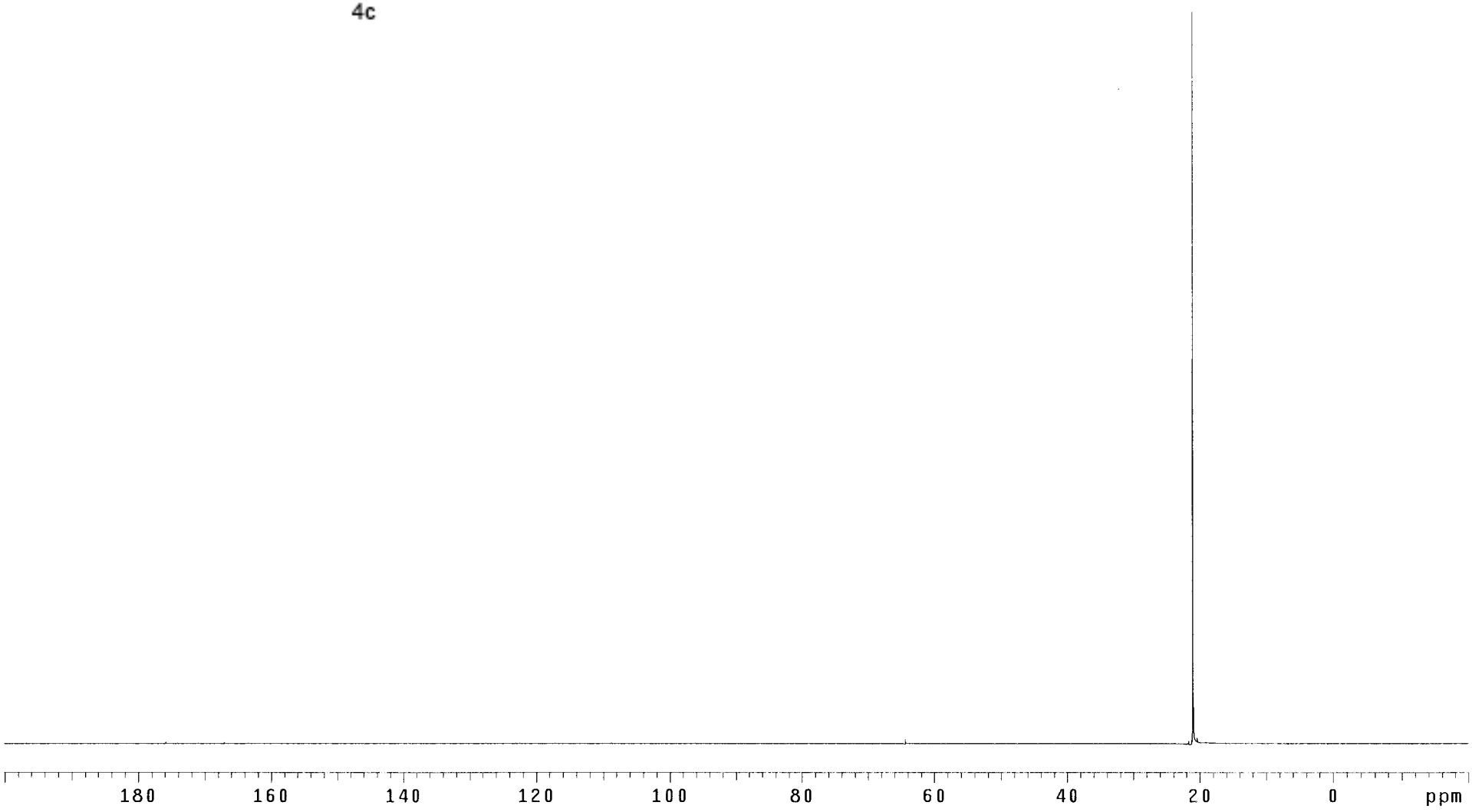


INDEX	FREQUENCY	PPM	HEIGHT
1	2557.264	21.054	126.0

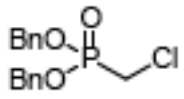


$^{31}P/^{1}H$ decoupled

4c



INDEX	FREQUENCY	PPM	HEIGHT
1	2585.008	21.282	10.7
2	2576.032	21.208	42.9
3	2566.240	21.128	93.7
4	2557.264	21.054	126.0
5	2547.881	20.977	94.1
6	2538.089	20.896	40.5
7	2529.113	20.822	9.7



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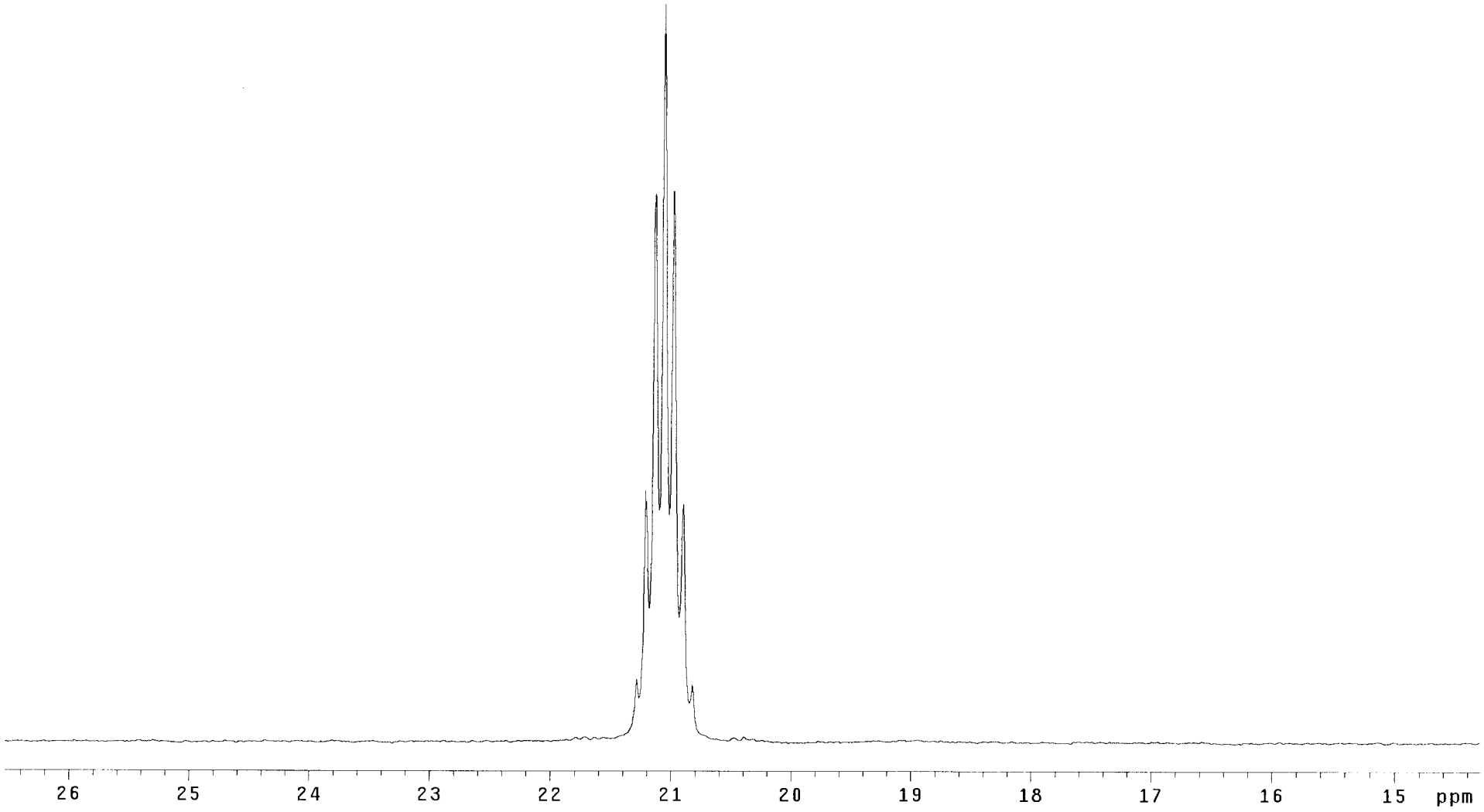
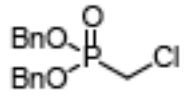
$^{31}P/^{1}H$ coupled

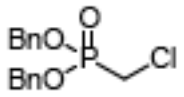


INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	2228.868	7.428	3.4	40	20.521	0.068	10.5
2	2229.747	7.431	3.5	41	9.967	0.033	3.8
3	2210.106	7.366	123.1	42	9.381	0.031	3.5
4	2209.226	7.363	132.7	43	8.502	0.028	3.5
5	2188.705	7.294	3.1	44	7.915	0.026	4.1
6	2179.618	7.264	11.7	45	7.036	0.023	4.7
7	2178.738	7.261	7.9	46	6.449	0.021	6.0
8	1554.022	5.179	3.8	47	5.863	0.020	6.3
9	1553.142	5.176	4.1	48	4.984	0.017	9.0
10	1545.227	5.150	4.5	49	4.104	0.014	18.2
11	1542.296	5.140	16.5	50	2.932	0.010	24.1
12	1541.416	5.137	17.6	51	0.586	0.002	357.2
13	1536.433	5.121	16.4	52	0.000	0.000	342.4
14	1535.553	5.118	18.7	53	-4.397	-0.015	7.2
15	1533.501	5.111	20.1	54	-4.984	-0.017	6.1
16	1532.622	5.108	18.9	55	-19.348	-0.064	3.3
17	1527.931	5.092	16.2	56	-19.935	-0.066	4.2
18	1527.052	5.089	17.4	57	-20.814	-0.069	8.1
19	1524.999	5.082	7.9				
20	1516.205	5.053	3.3				
21	1515.325	5.050	3.7				
22	1240.931	4.136	4.8				
23	1240.051	4.133	5.0				
24	1233.895	4.112	5.1				
25	1233.016	4.109	4.9				
26	1052.725	3.508	23.5				
27	1051.845	3.506	24.9				
28	1042.171	3.473	23.2				
29	1041.291	3.470	24.6				
30	615.042	2.050	23.0				
31	614.163	2.047	20.1				
32	484.587	1.615	9.7				
33	386.087	1.287	6.6				
34	384.914	1.283	6.1				
35	378.758	1.262	12.8				
36	377.879	1.259	12.8				
37	371.722	1.239	6.7				
38	370.843	1.236	5.8				
39	21.400	0.071	5.2				



4c

INDEX	FREQUENCY	PPM	HEIGHT
1	10255.848	135.933	14.4
2	10250.090	135.857	13.7
3	9733.019	129.003	75.3
4	9728.700	128.946	126.0
5	9704.804	128.629	17.7
6	9687.530	128.400	121.9
7	9618.722	127.488	6.8
8	9593.674	127.156	18.3
9	5216.129	69.136	34.4
10	5209.507	69.048	34.8
11	2637.682	34.960	15.6
12	2477.896	32.842	15.6



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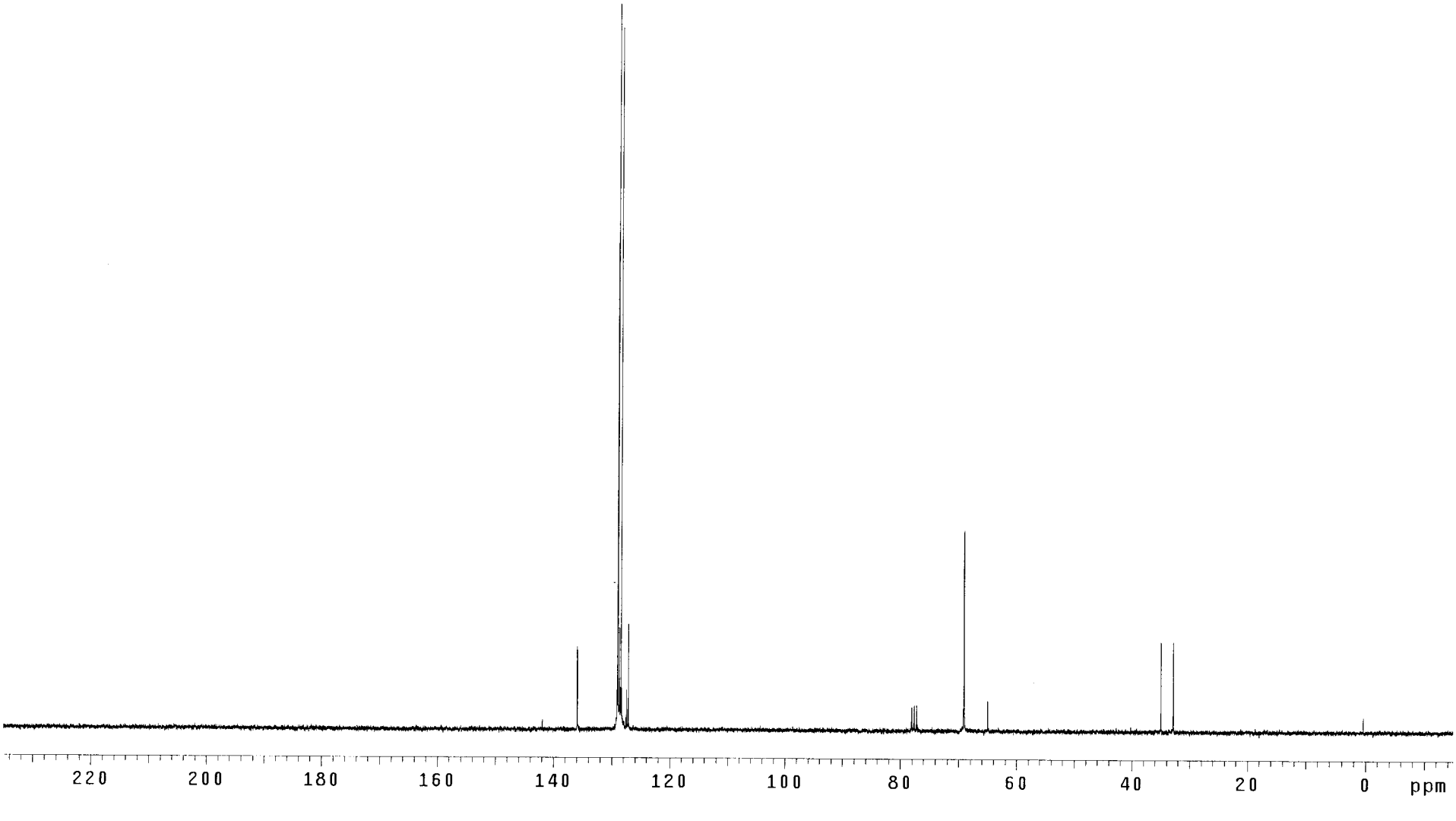
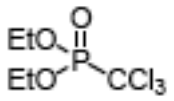
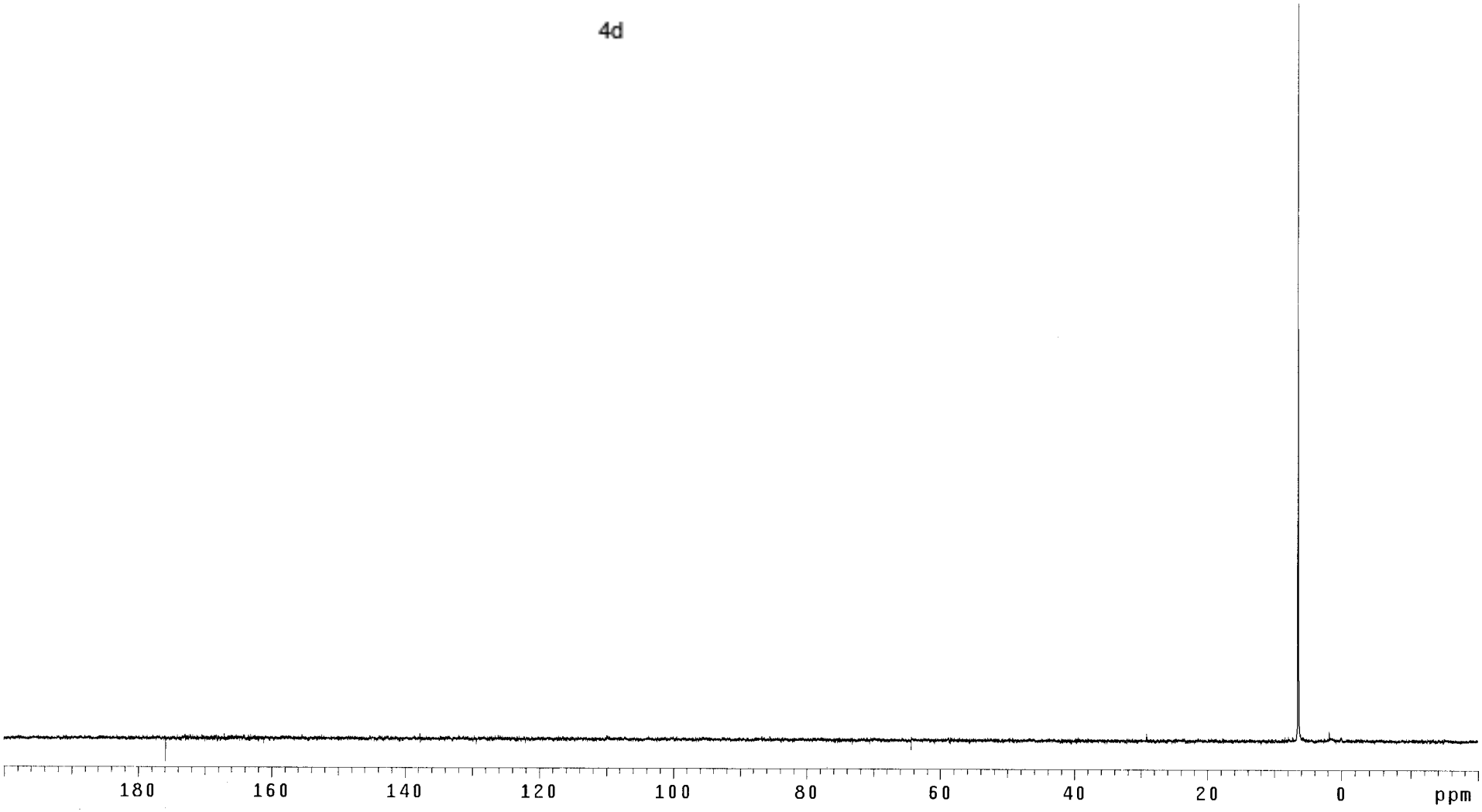


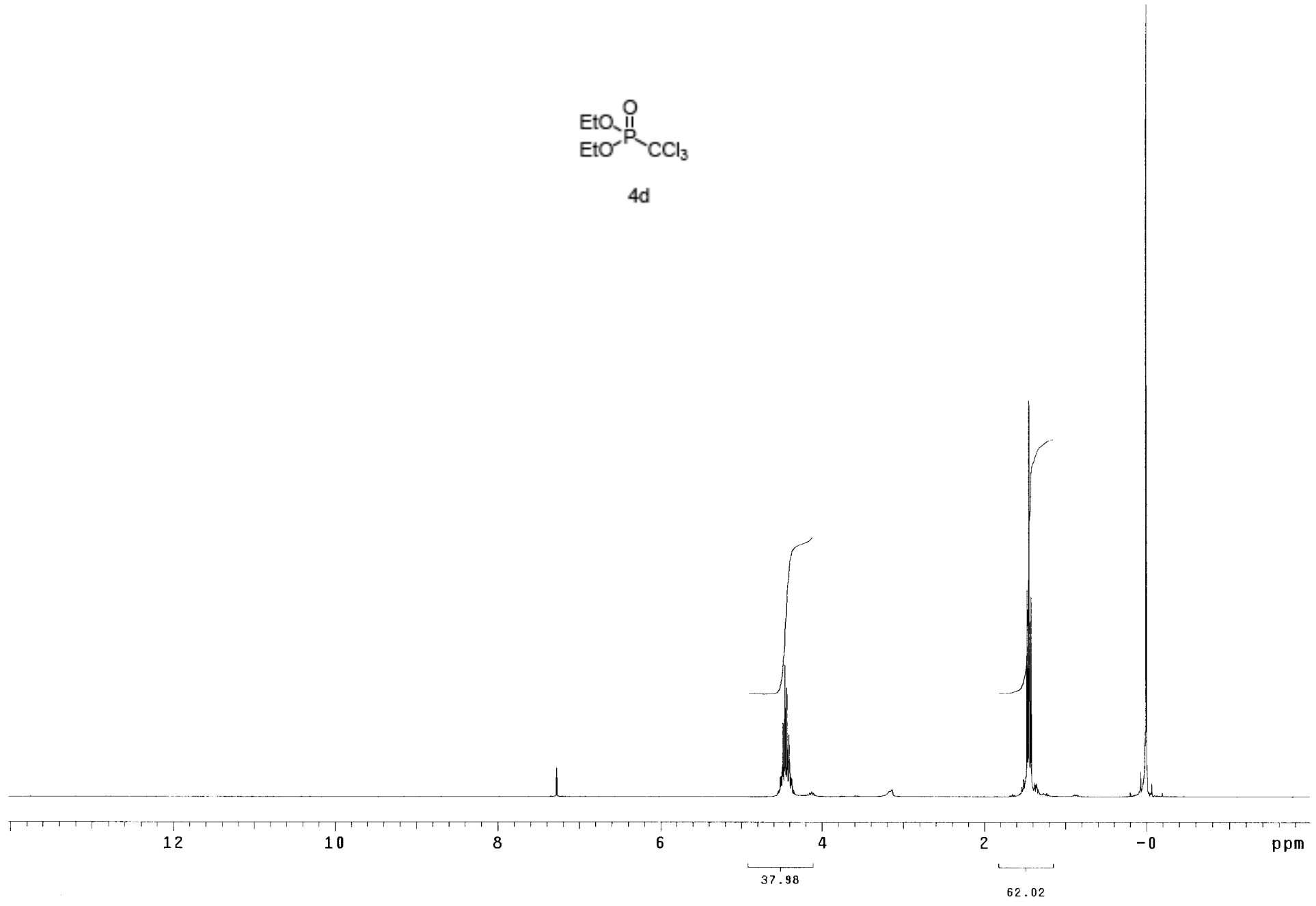
INDEX	FREQUENCY	PPM	HEIGHT
1	788.632	6.493	126.0



4d

$^{31}P/^{1}H$ decoupled




EtO
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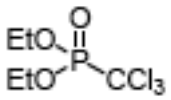
EtO
CCl3
4d
12
10
8
6
4
2
-0
ppm
37.98
62.02

INDEX	FREQUENCY	PPM	HEIGHT
1	7105.413	94.176	6.3
2	6796.495	90.082	5.8
3	6598.707	87.460	4.9
4	5868.012	77.776	14.0
5	5835.767	77.348	14.8
6	5803.522	76.921	14.5
7	5065.918	67.145	105.4
8	5059.008	67.053	102.7
9	1240.568	16.443	126.0
10	1234.810	16.366	118.9
11	0.000	0.000	7.6



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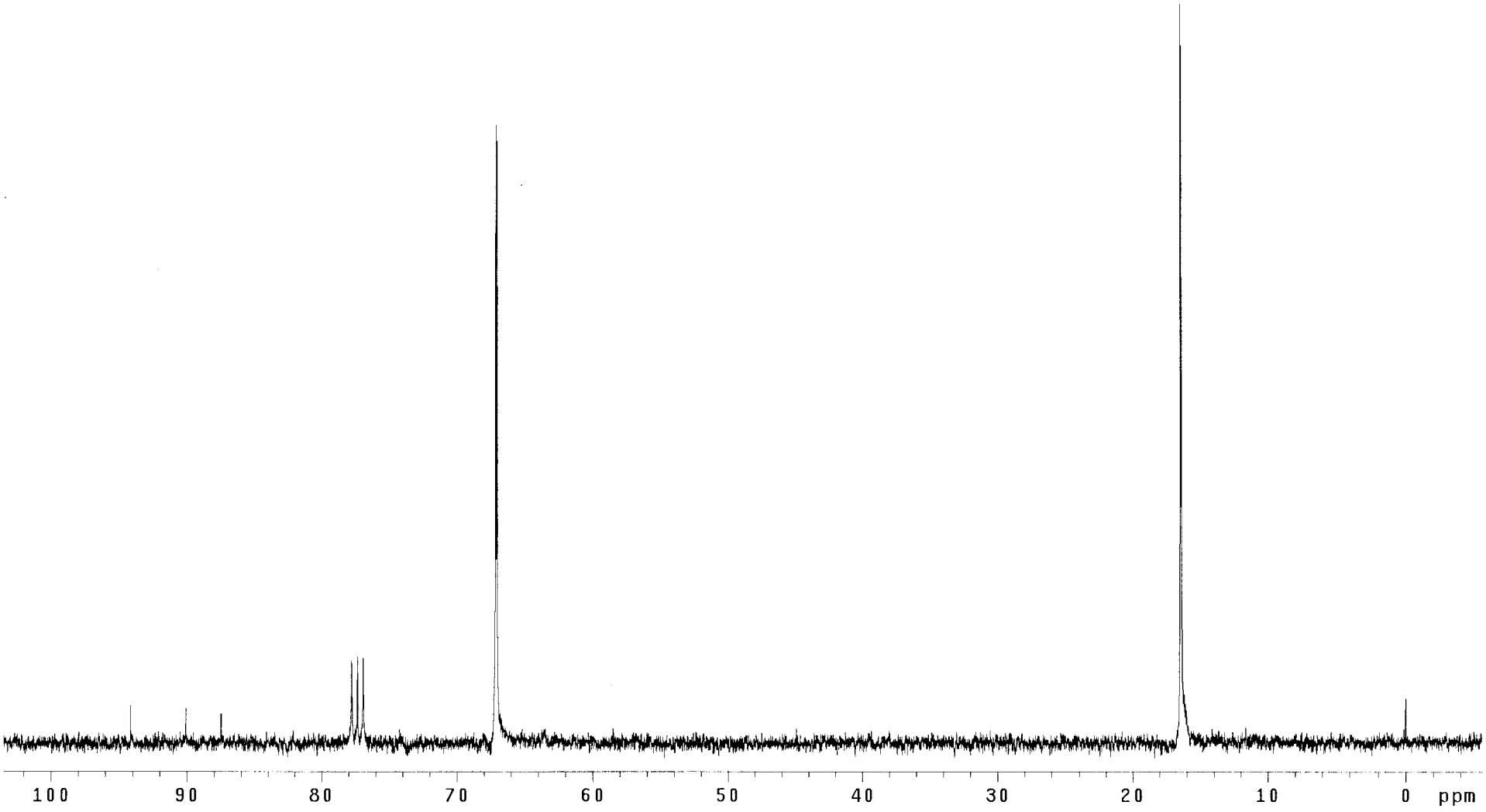
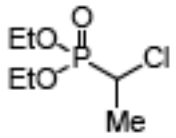
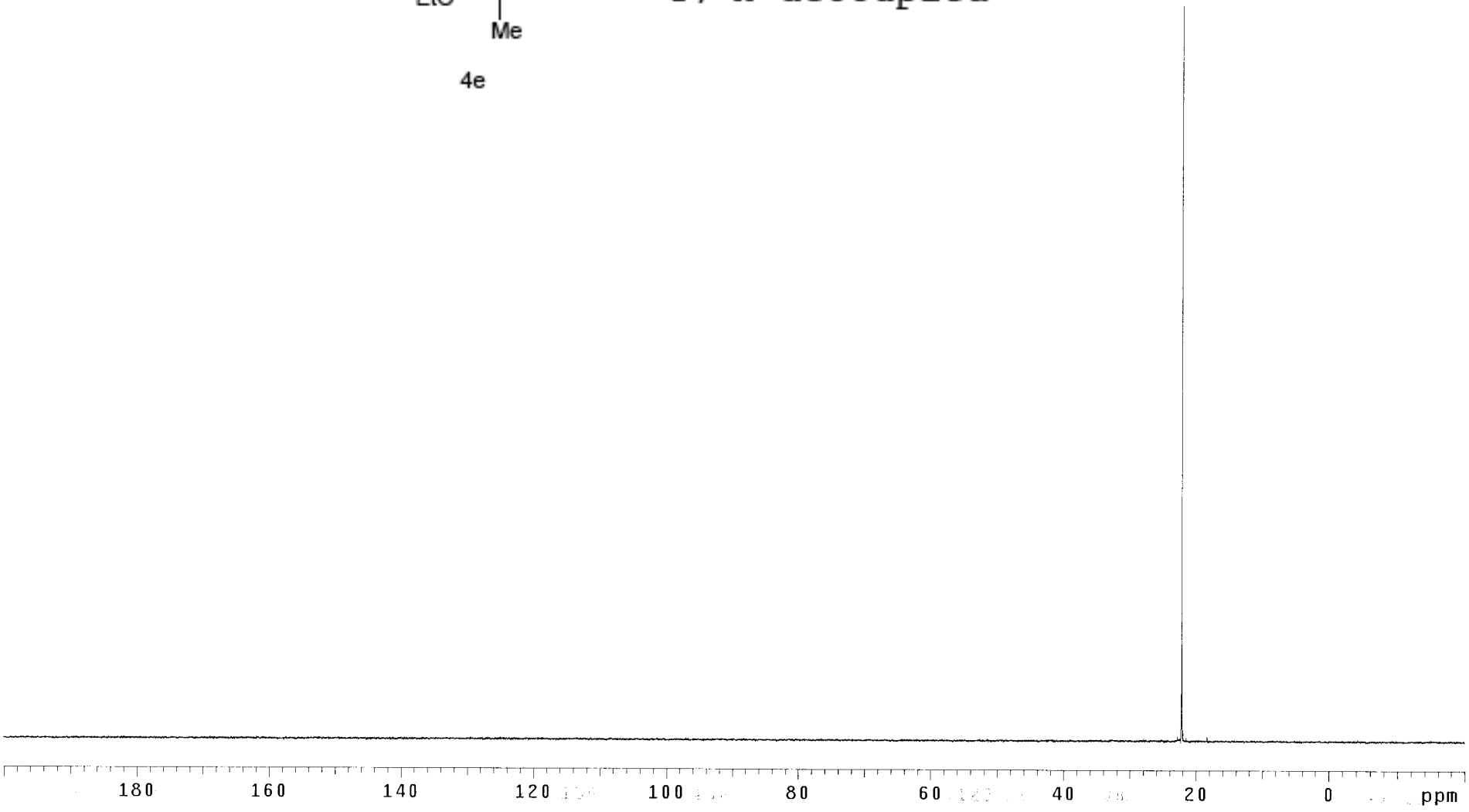


INDEX	FREQUENCY	PPM	HEIGHT
1	2697.613	22.209	126.0

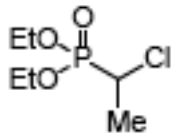


4e

$^{31}P/^{1}H$ decoupled

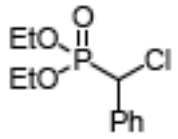


INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	2186.653	7.288	3.9	40	403.676	1.345	78.7
2	2186.067	7.286	5.5	41	397.520	1.325	2.4
3	1287.250	4.290	5.7	42	390.484	1.301	2.5
4	1286.663	4.288	7.9	43	0.586	0.002	18.5
5	1281.093	4.270	10.5	44	0.000	0.000	30.7
6	1280.214	4.267	19.1				
7	1279.628	4.265	26.8				
8	1274.058	4.246	32.0				
9	1273.178	4.243	29.7				
10	1272.299	4.240	38.7				
11	1271.712	4.238	25.7				
12	1267.022	4.223	38.6				
13	1266.142	4.220	34.4				
14	1265.263	4.217	27.5				
15	1264.383	4.214	25.0				
16	1259.693	4.198	23.1				
17	1259.107	4.196	25.8				
18	1258.227	4.193	9.2				
19	1257.348	4.190	9.6				
20	1252.657	4.175	6.3				
21	1252.071	4.173	8.2				
22	1209.563	4.031	3.5				
23	1202.234	4.007	11.3				
24	1199.889	3.999	3.9				
25	1194.905	3.982	11.0				
26	1192.560	3.975	11.6				
27	1187.870	3.959	4.1				
28	1185.231	3.950	11.2				
29	1178.195	3.927	3.5				
30	523.577	1.745	40.1				
31	522.991	1.743	55.9				
32	515.662	1.719	54.3				
33	506.281	1.687	52.3				
34	503.643	1.679	2.6				
35	502.177	1.674	2.2				
36	499.832	1.666	39.2				
37	499.245	1.664	51.8				
38	417.748	1.392	83.9				
39	410.712	1.369	162.0				



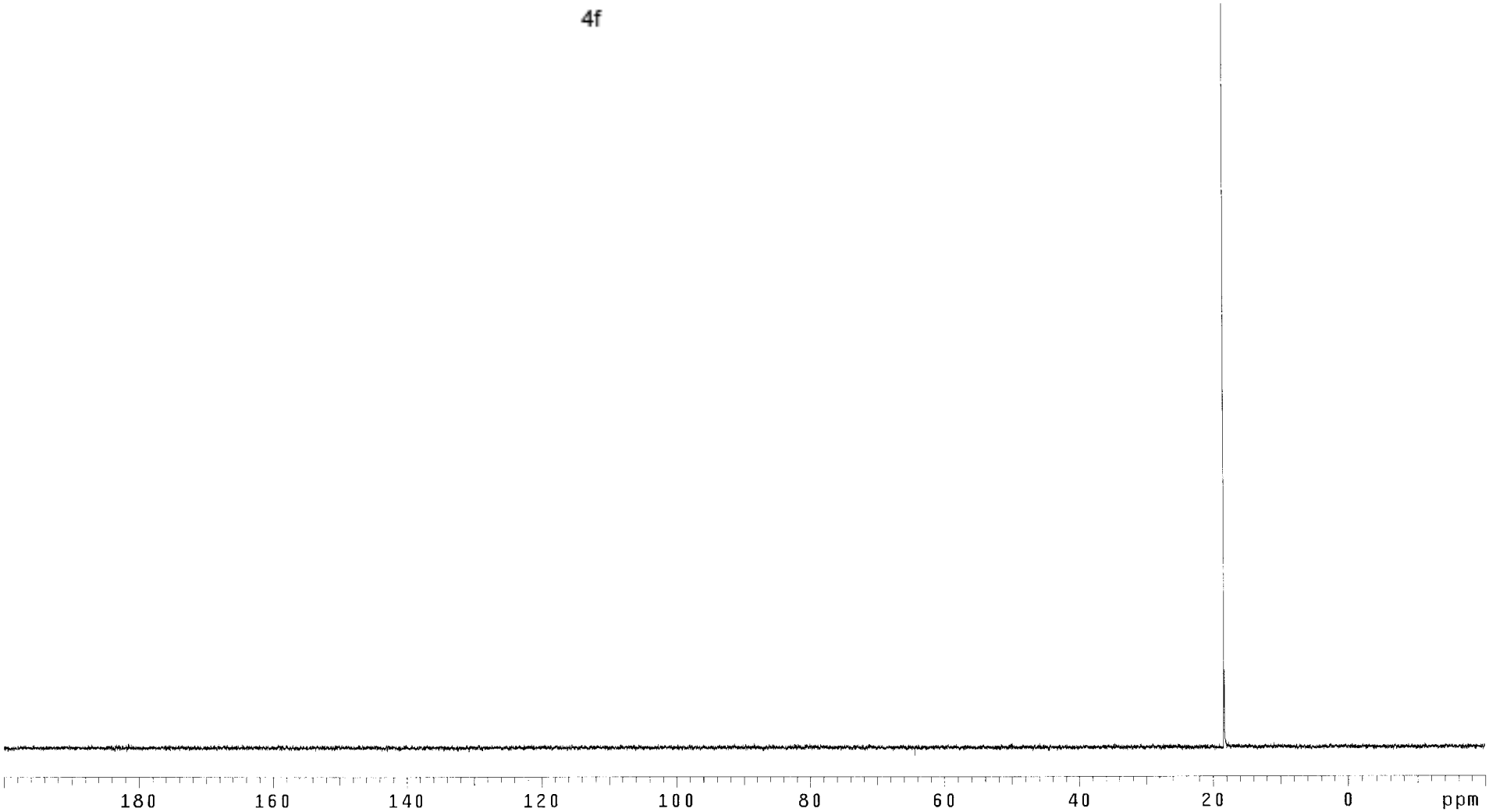
4e

INDEX	FREQUENCY	PPM	HEIGHT
1	2228.425	18.347	126.0

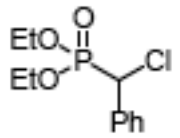


4f

$^{31}P/^{1}H$ decoupled

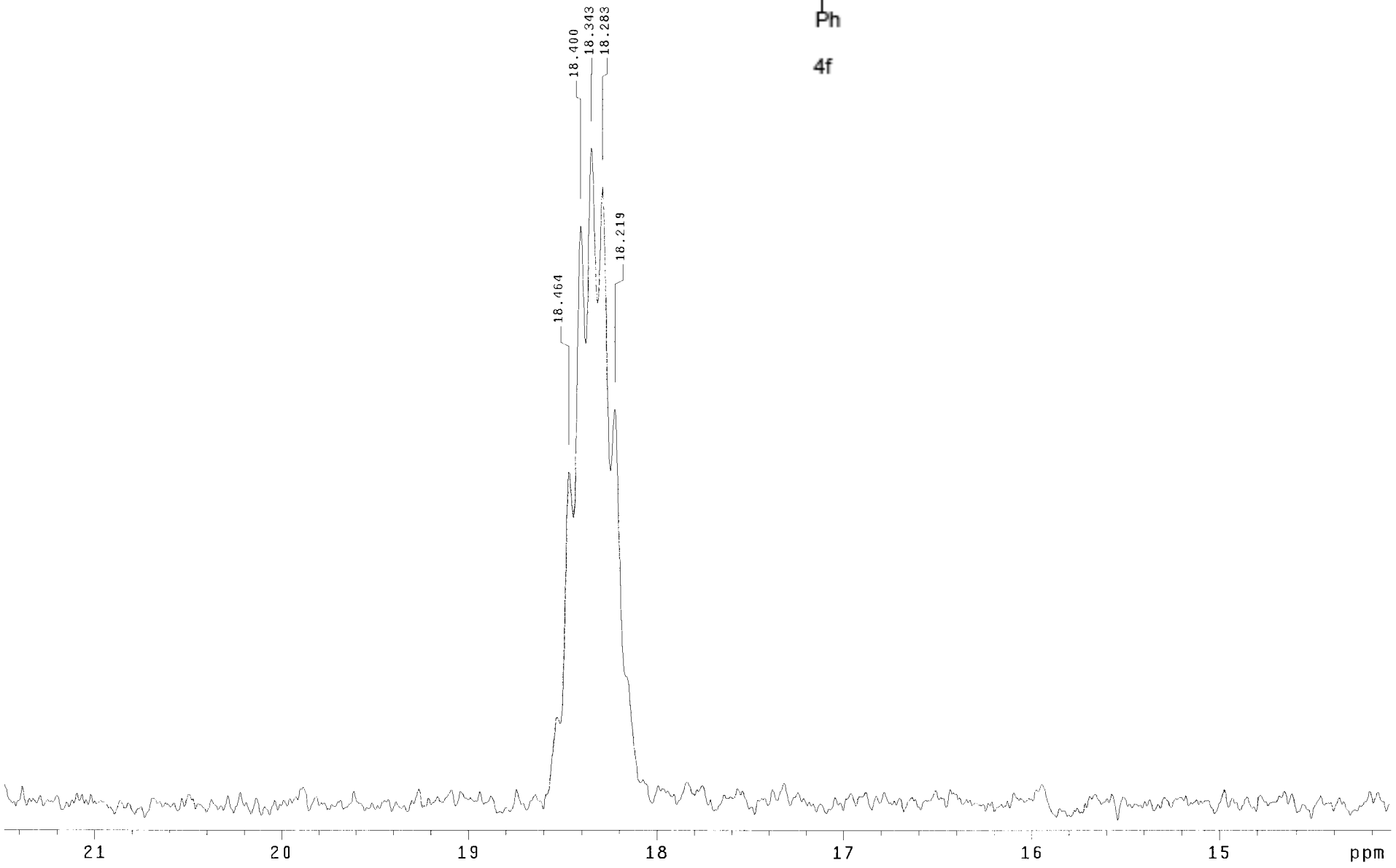


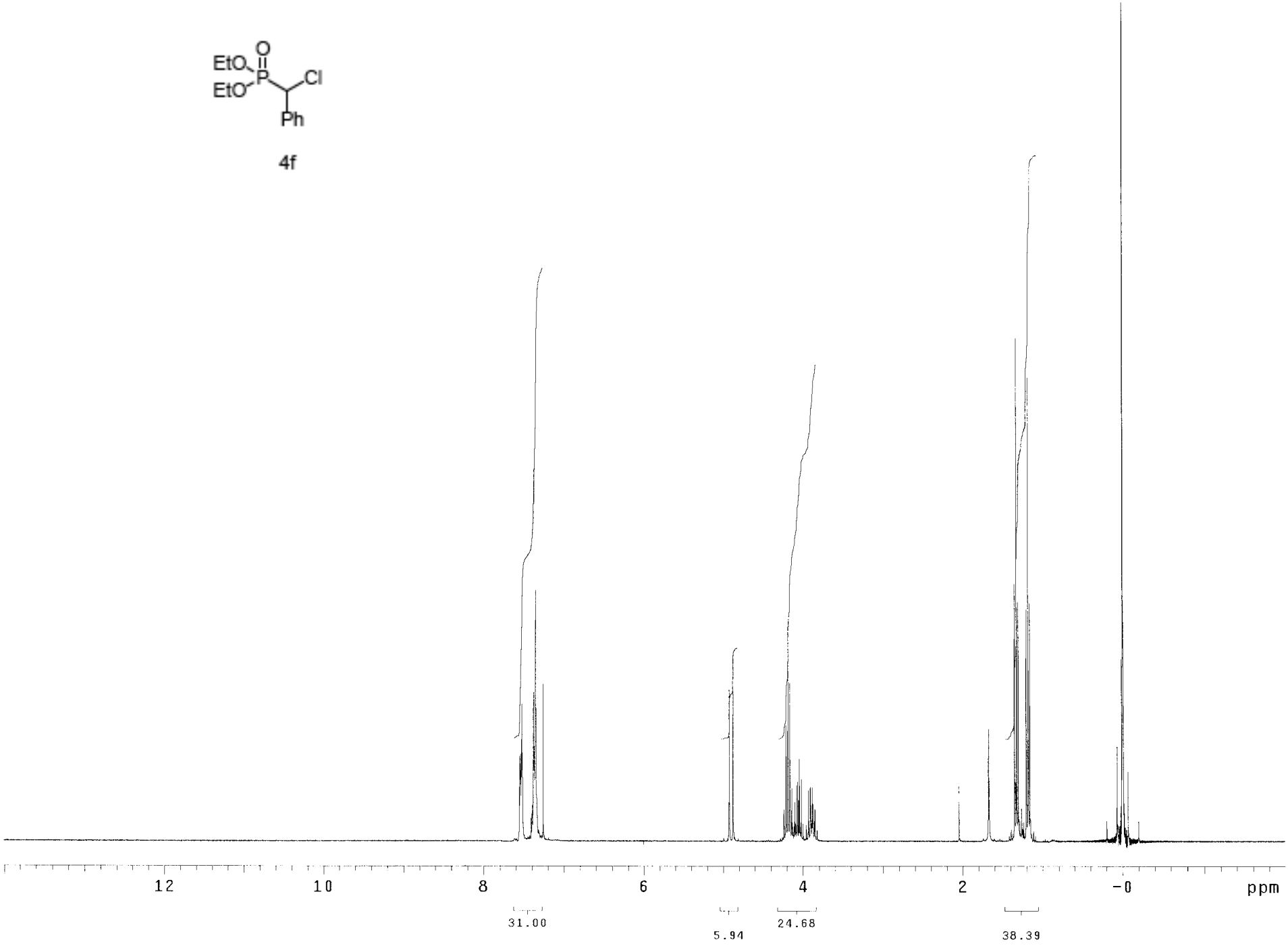
INDEX	FREQUENCY	PPM	HEIGHT
1	2242.705	18.464	59.8
2	2234.953	18.400	104.6
3	2228.017	18.343	118.6
4	2220.673	18.283	111.6
5	2212.922	18.219	71.2



4f

$^{31}P/^{1}H$ coupled

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EtO
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4
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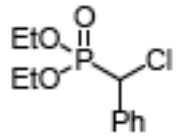
24.68
38.39

INDEX	FREQUENCY	PPM	HEIGHT
1	10123.487	134.179	19.3
2	10120.032	134.133	18.7
3	9735.107	129.031	67.0
4	9732.516	128.997	71.6
5	9729.637	128.958	120.4
6	9723.591	128.878	117.9
7	9701.135	128.581	126.0
8	9699.408	128.558	124.3
9	5851.602	77.558	31.9
10	5819.645	77.135	32.8
11	5787.687	76.711	32.5
12	4837.900	64.122	32.1
13	4830.990	64.031	32.3
14	4825.808	63.962	34.2
15	4818.898	63.871	32.5
16	4127.071	54.701	51.3
17	3967.285	52.583	51.8
18	1241.432	16.454	37.5
19	1235.674	16.378	36.7
20	1227.037	16.263	37.0
21	1221.279	16.187	35.8



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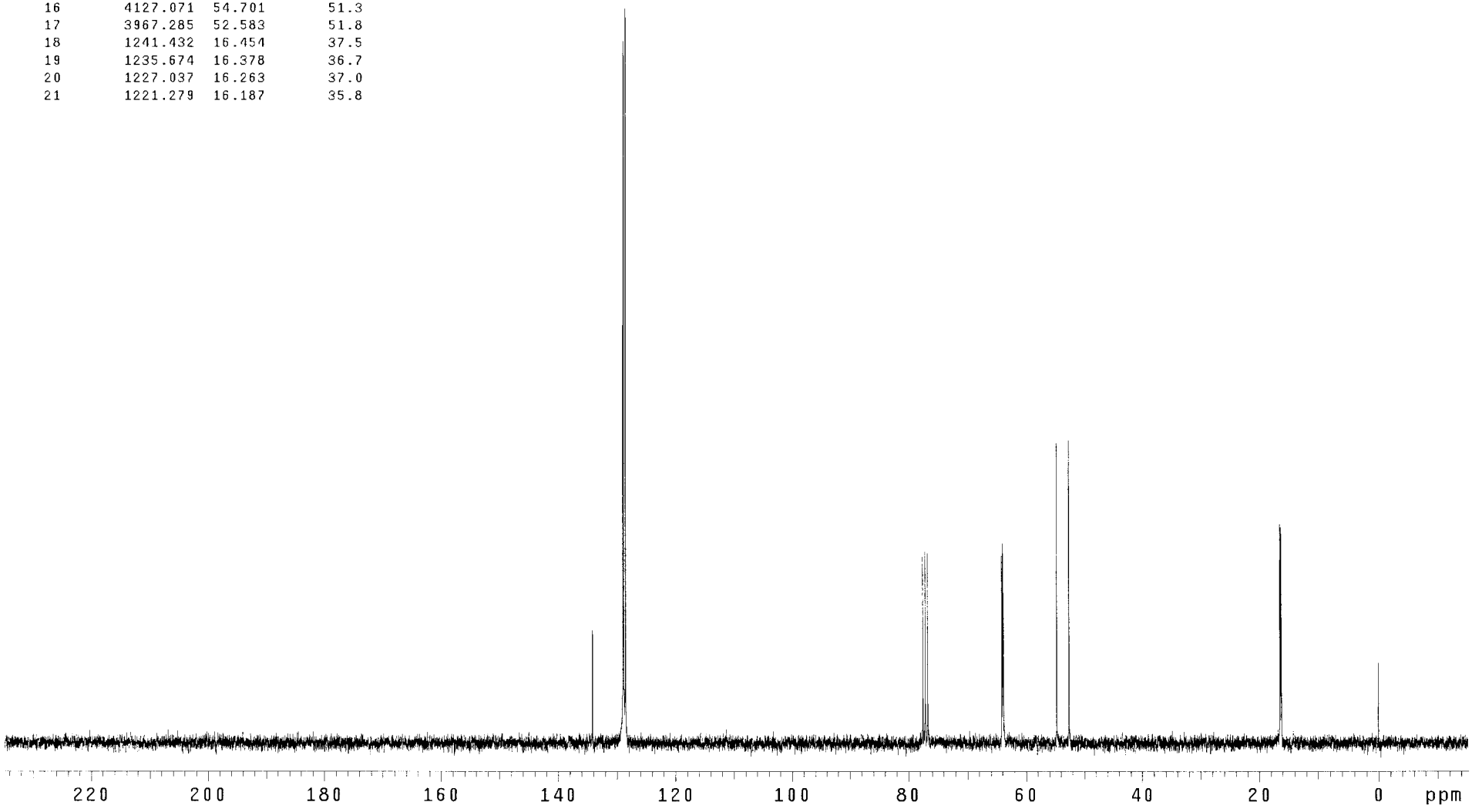

INDEX	FREQUENCY	PPM	HEIGHT
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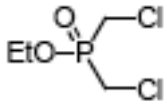
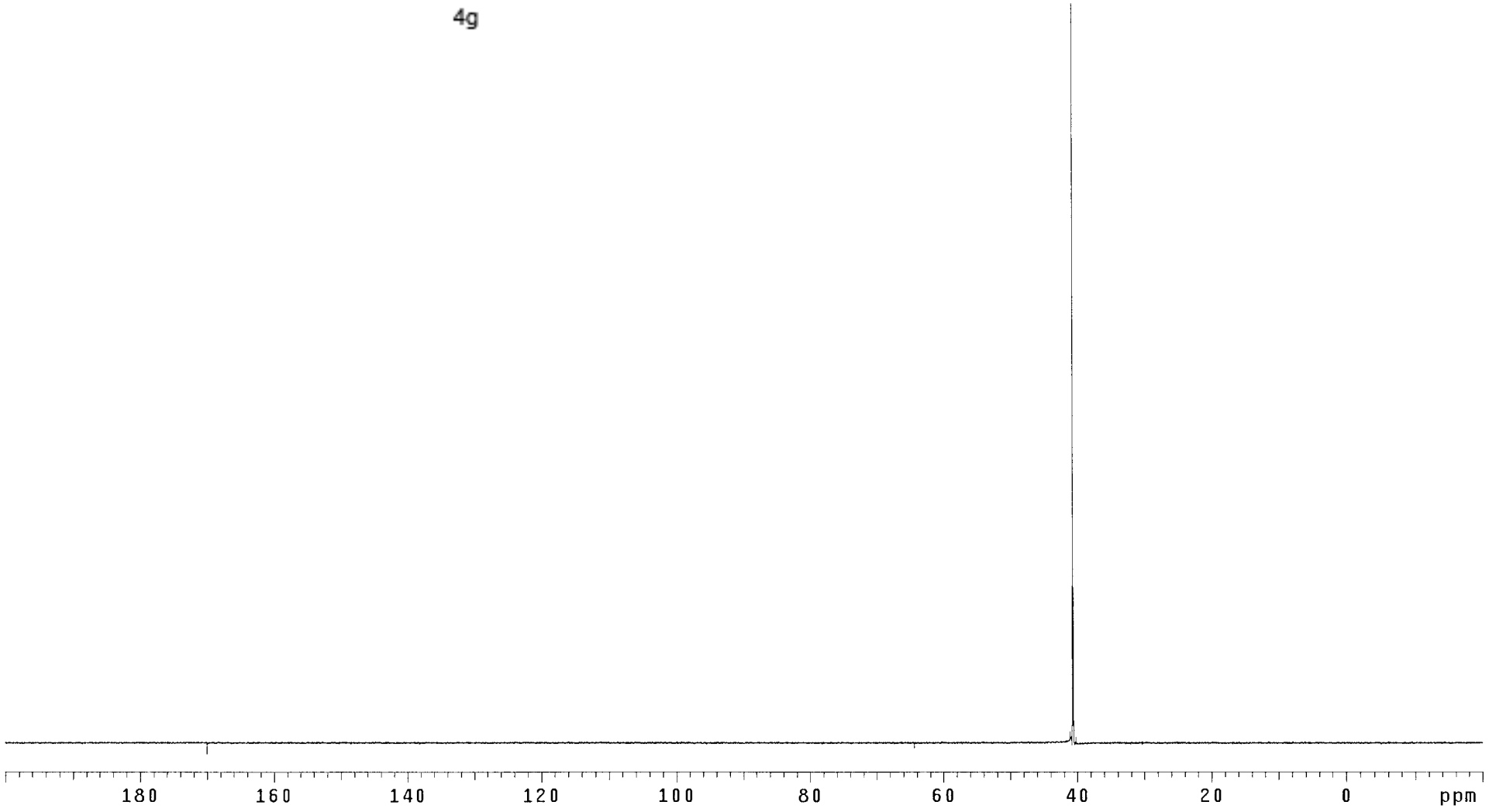
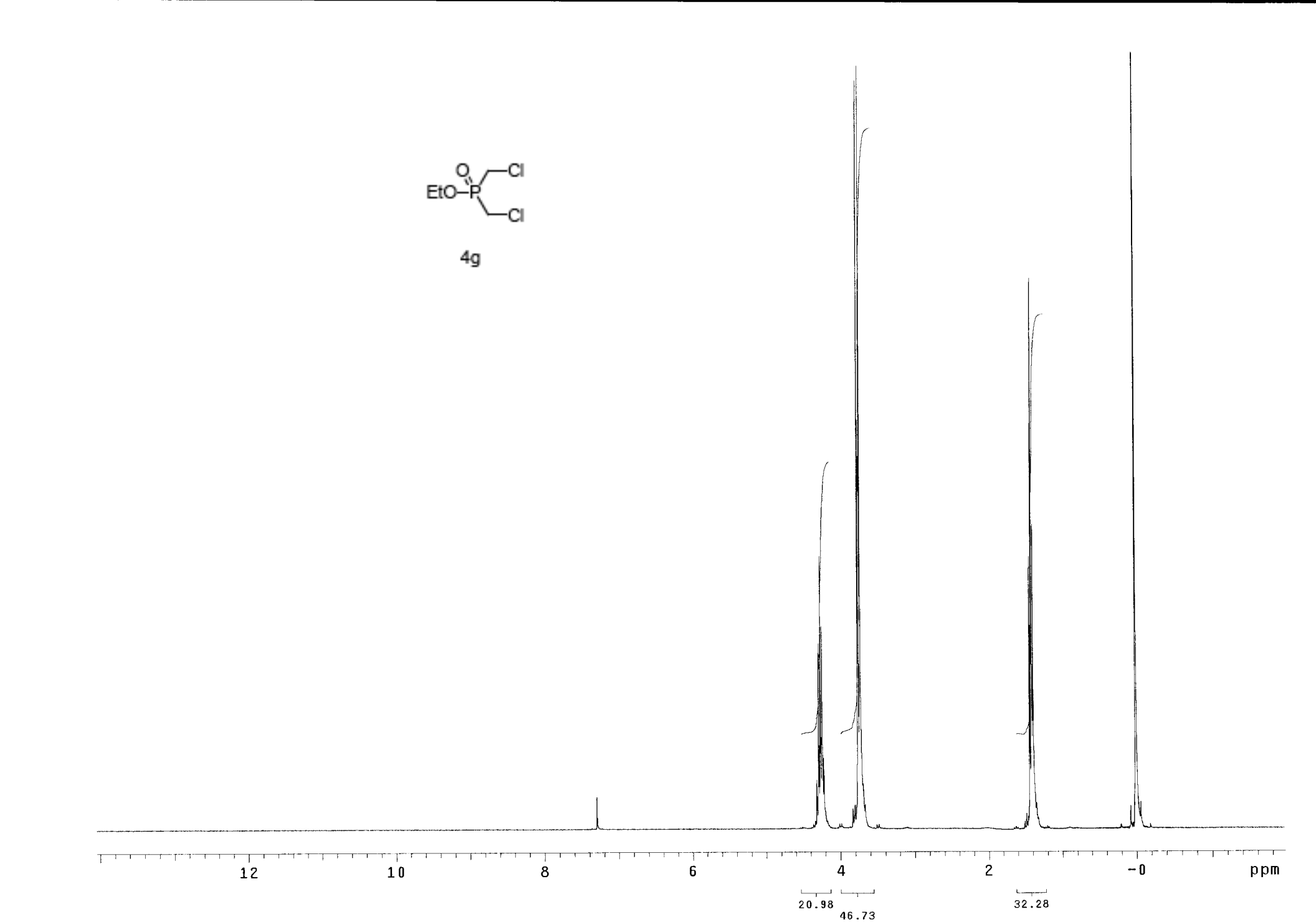


$^{31}P/^{1}H$ decoupled

4g

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Cl
4g
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6
4
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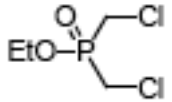
32.28

INDEX	FREQUENCY	PPM	HEIGHT
1	5867.939	77.775	21.4
2	5835.694	77.347	23.0
3	5803.736	76.924	23.4
4	4769.882	63.221	28.2
5	4762.972	63.129	30.6
6	2531.158	33.548	125.9
7	2424.634	32.137	126.0
8	1261.512	16.720	36.3
9	1256.041	16.648	34.1



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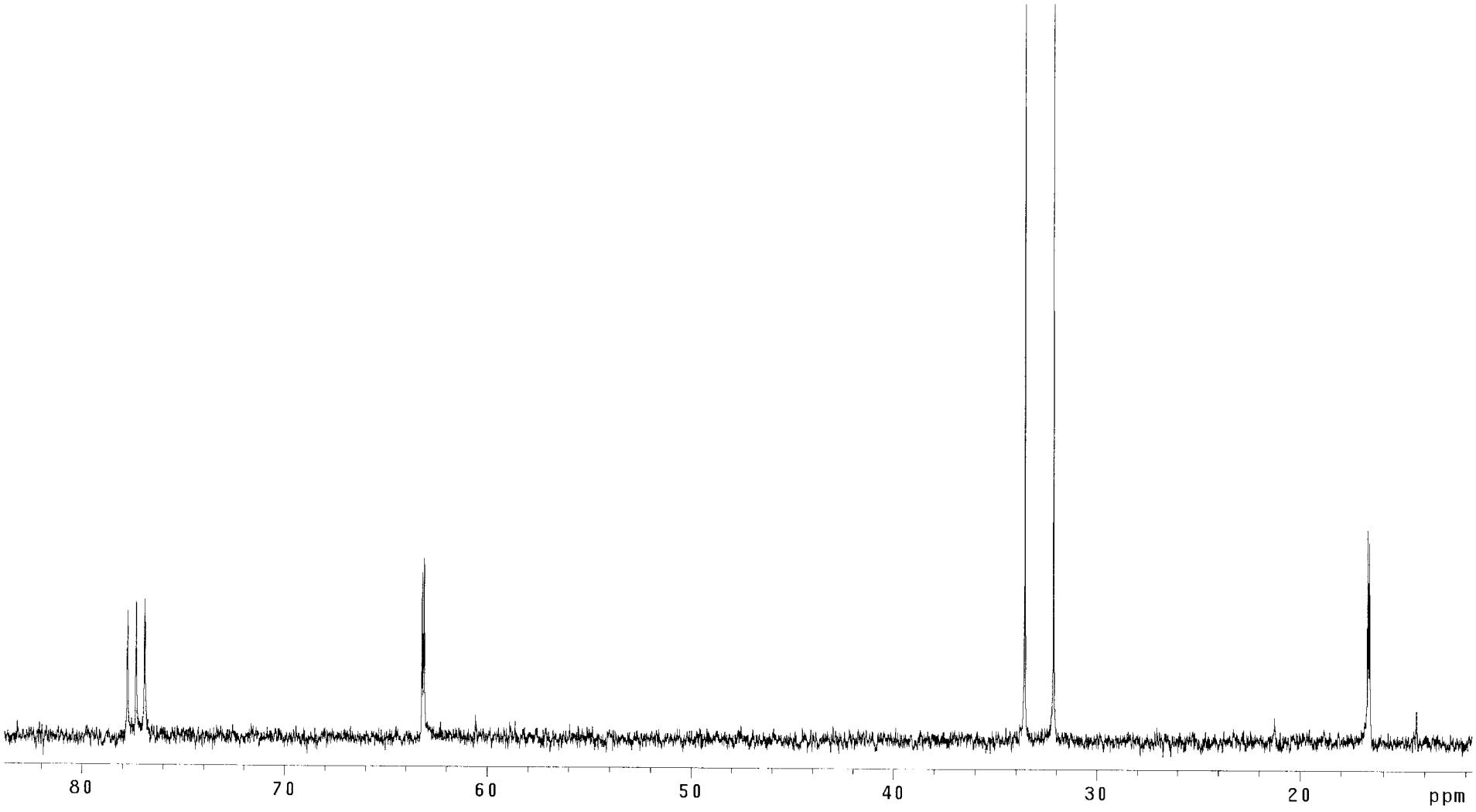
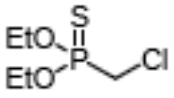
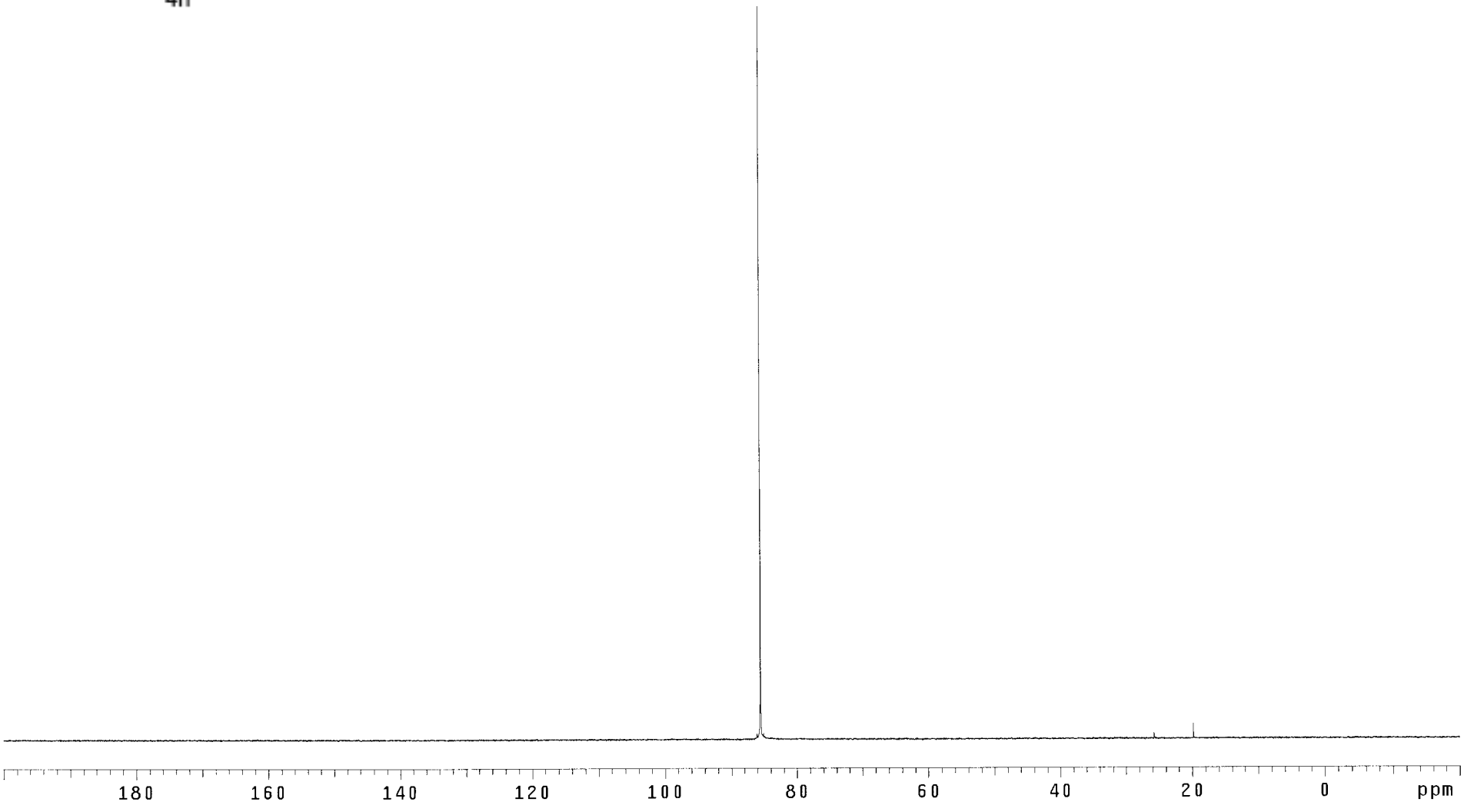


INDEX	FREQUENCY	PPM	HEIGHT
1	10394.325	85.576	126.0

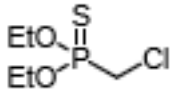


4h

$^{31}P/^{1}H$ decoupled

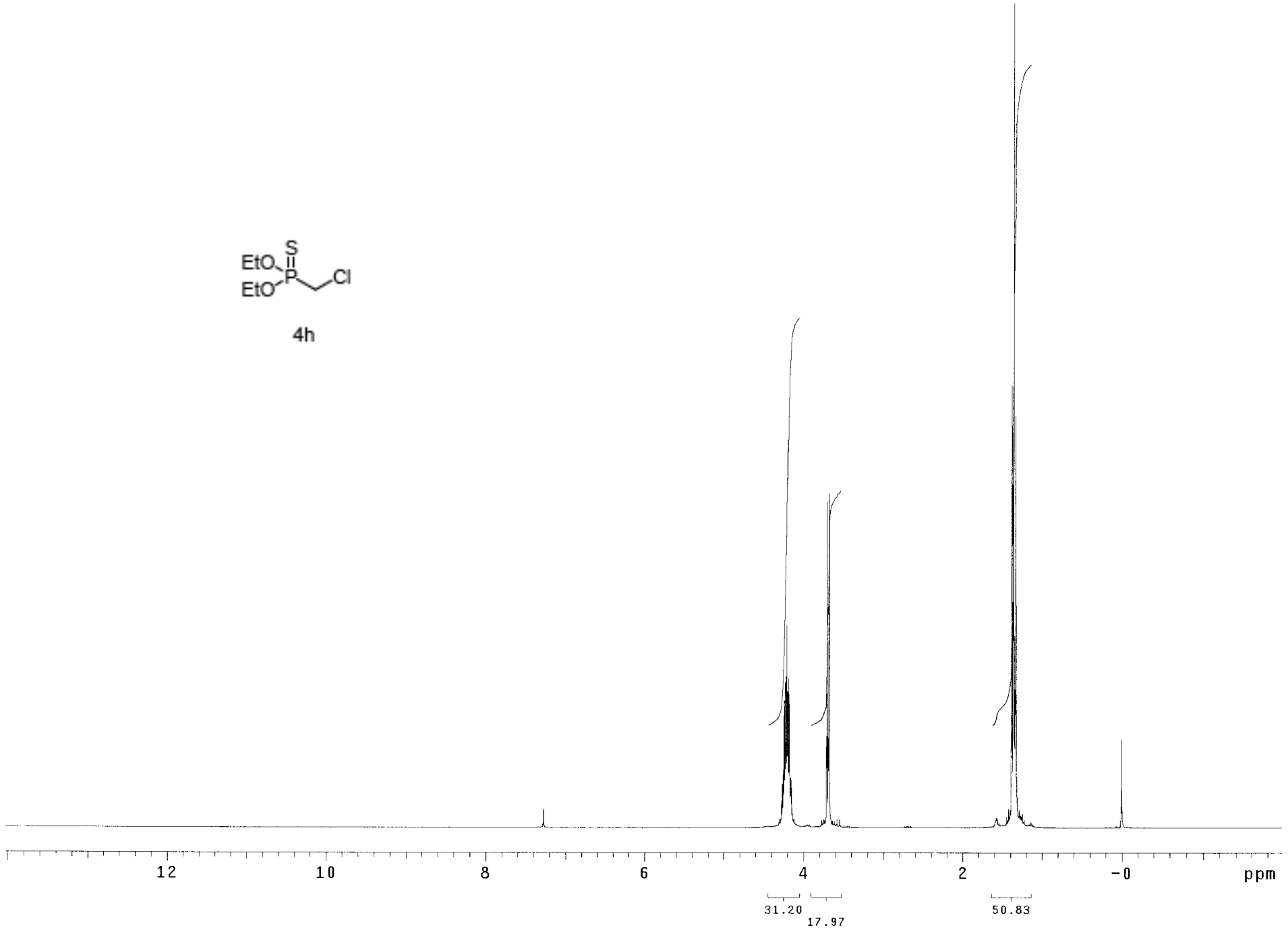


INDEX	FREQUENCY	PPM	HEIGHT
1	10422.477	85.808	11.7
2	10412.277	85.724	44.8
3	10404.117	85.657	88.7
4	10394.325	85.576	126.0
5	10384.534	85.496	97.8
6	10376.782	85.432	51.8
7	10366.582	85.348	17.3

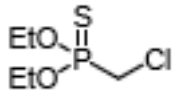


4h

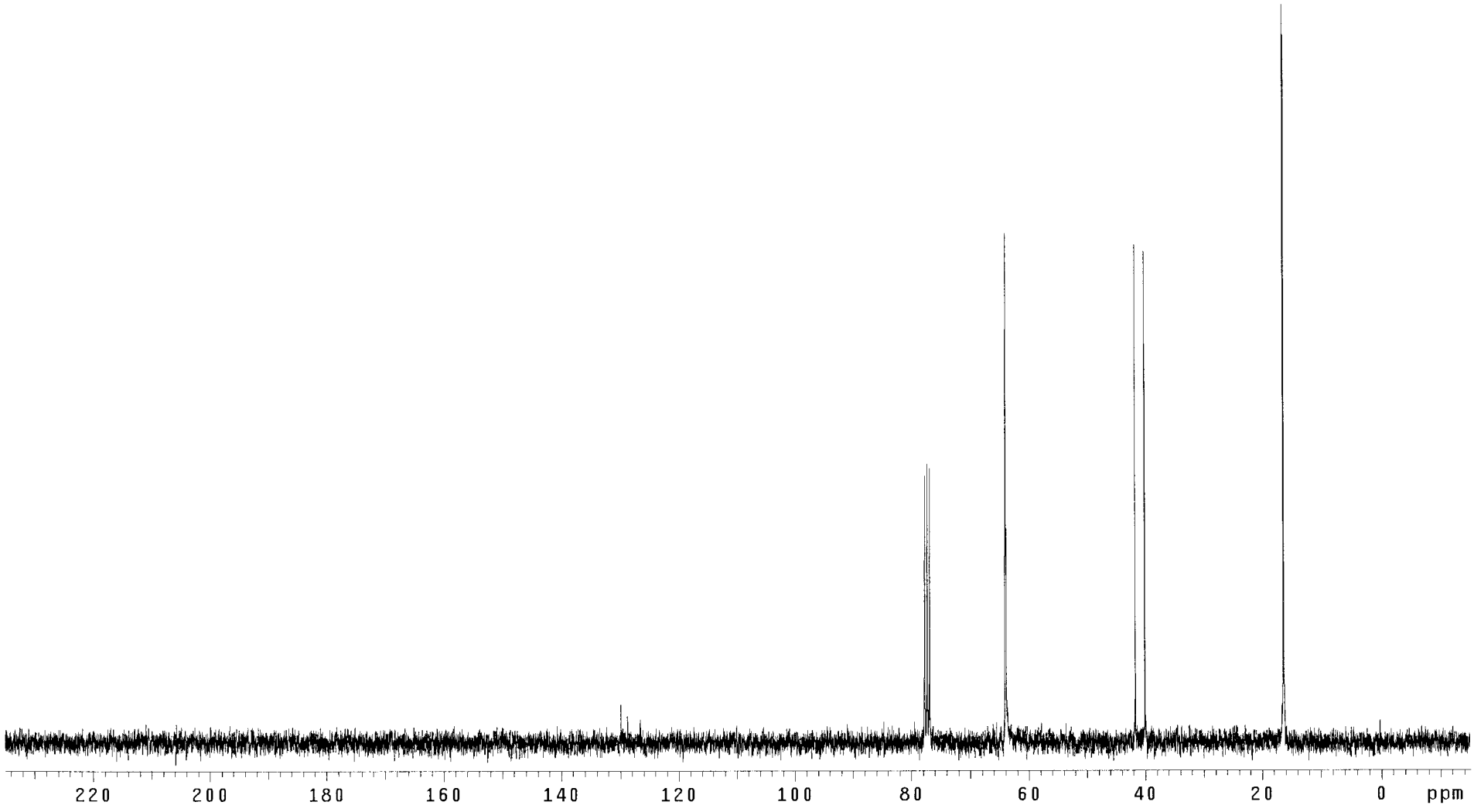
$^{31}P/^{1}H$ coupled


EtO
EtO
S
P
Cl
4h
12
10
8
6
4
2
-0
ppm
31.20
17.97
50.83

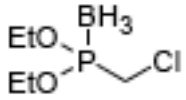
INDEX	FREQUENCY	PPM	HEIGHT
1	5862.756	77.706	45.5
2	5830.799	77.282	47.4
3	5798.842	76.859	46.6
4	4832.932	64.057	70.4
5	4826.886	63.976	87.0
6	3149.283	41.741	84.9
7	3025.197	40.097	83.8
8	1239.343	16.426	126.0
9	1232.434	16.335	120.8



4h

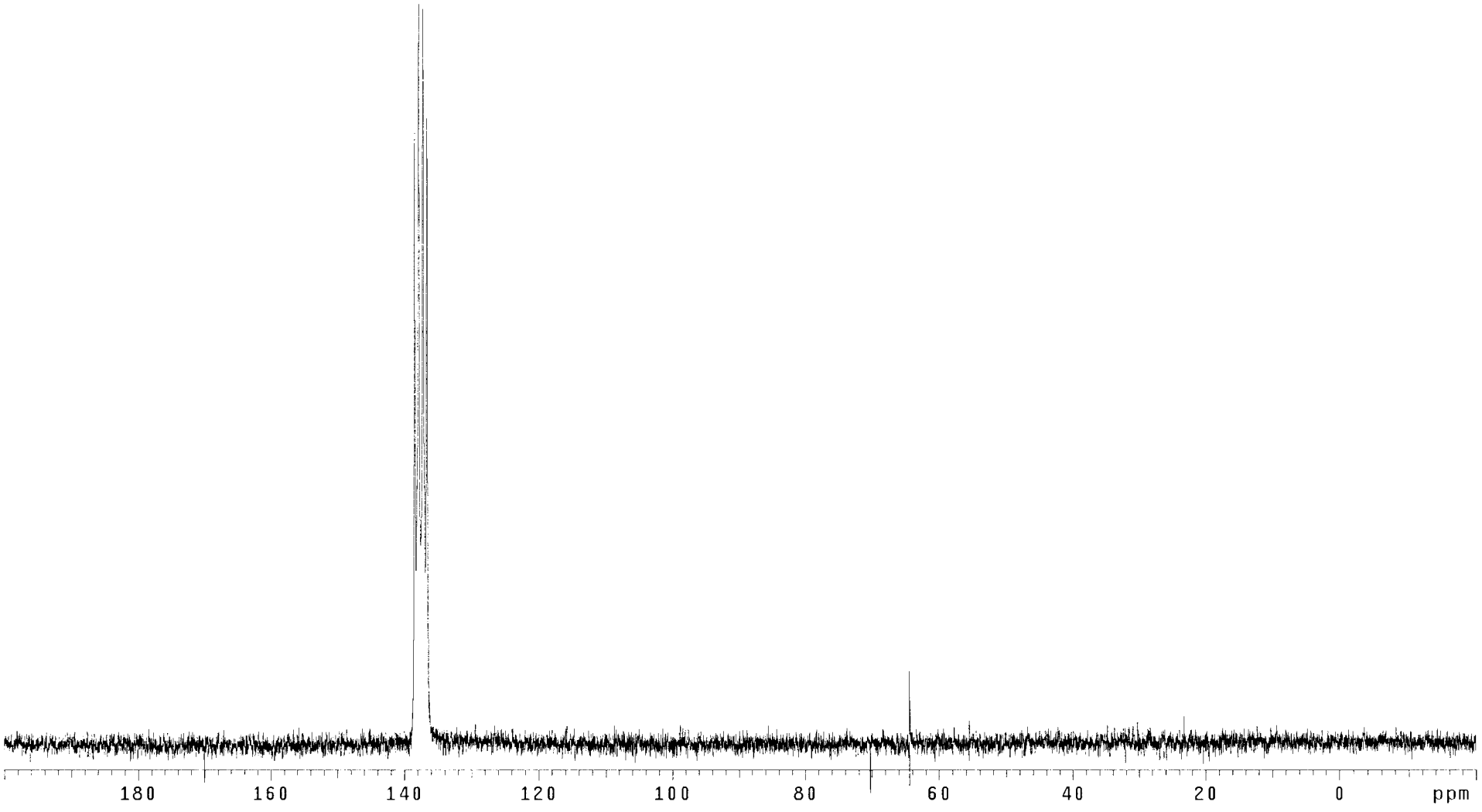


INDEX	FREQUENCY	PPM	HEIGHT
1	16818.928	138.470	104.5
2	16746.306	137.872	126.0
3	16670.012	137.244	125.1
4	16596.166	136.636	106.6

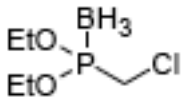


4i

$^{31}P/^{1}H$ decoupled

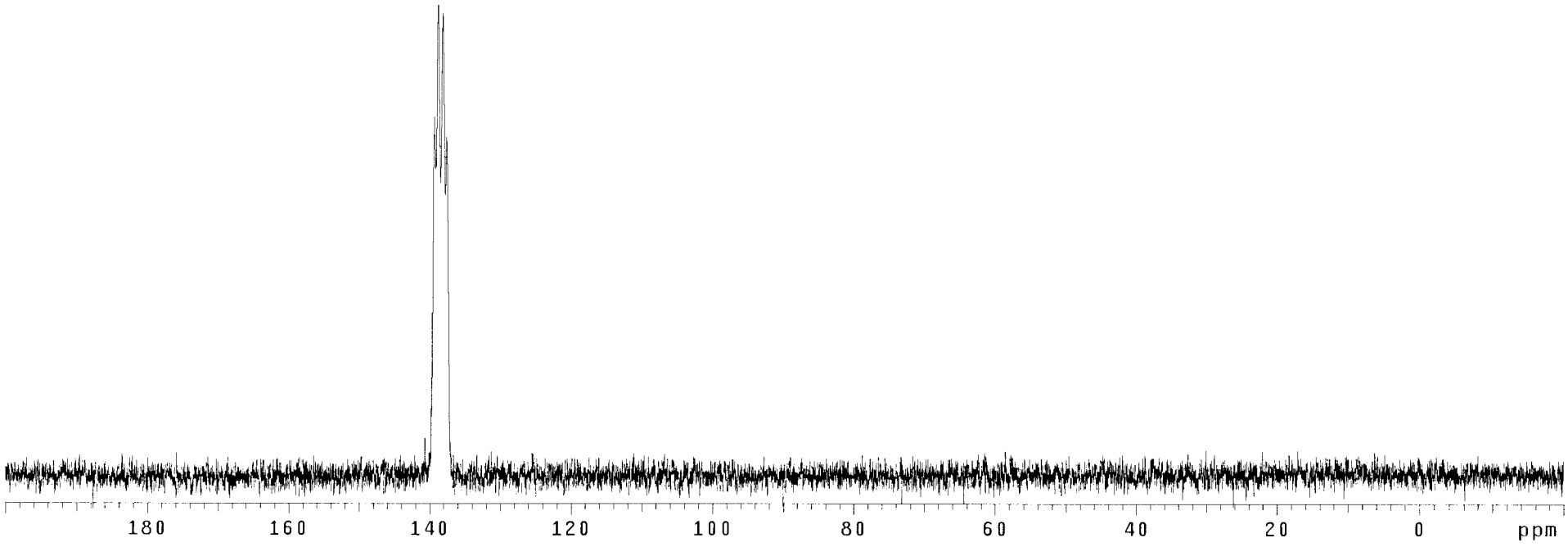


INDEX	FREQUENCY	PPM	HEIGHT
1	16927.045	139.360	57.4
2	16856.463	138.779	75.5
3	16778.129	138.134	74.2
4	16718.971	137.647	54.1



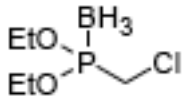
4i

$^{31}P/^{1}H$ coupled



Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	1278	32697	103.67	-1239.96	-42.932
2	1293	29255	92.76	-1316.32	-45.576



4i

$^{11}B/^{1}H$ decoupled

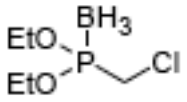
USER: -- DATE: 12/03/07 (09:52)

F1: 28.882 | SW1: 10000 | OF1: 0.0 | PTS1d: 2048

I:X: c:\cft\global\zg.ppg | PW: 15.5 usec | PD: 0.1 sec | NA: 64 | LB: 0.0

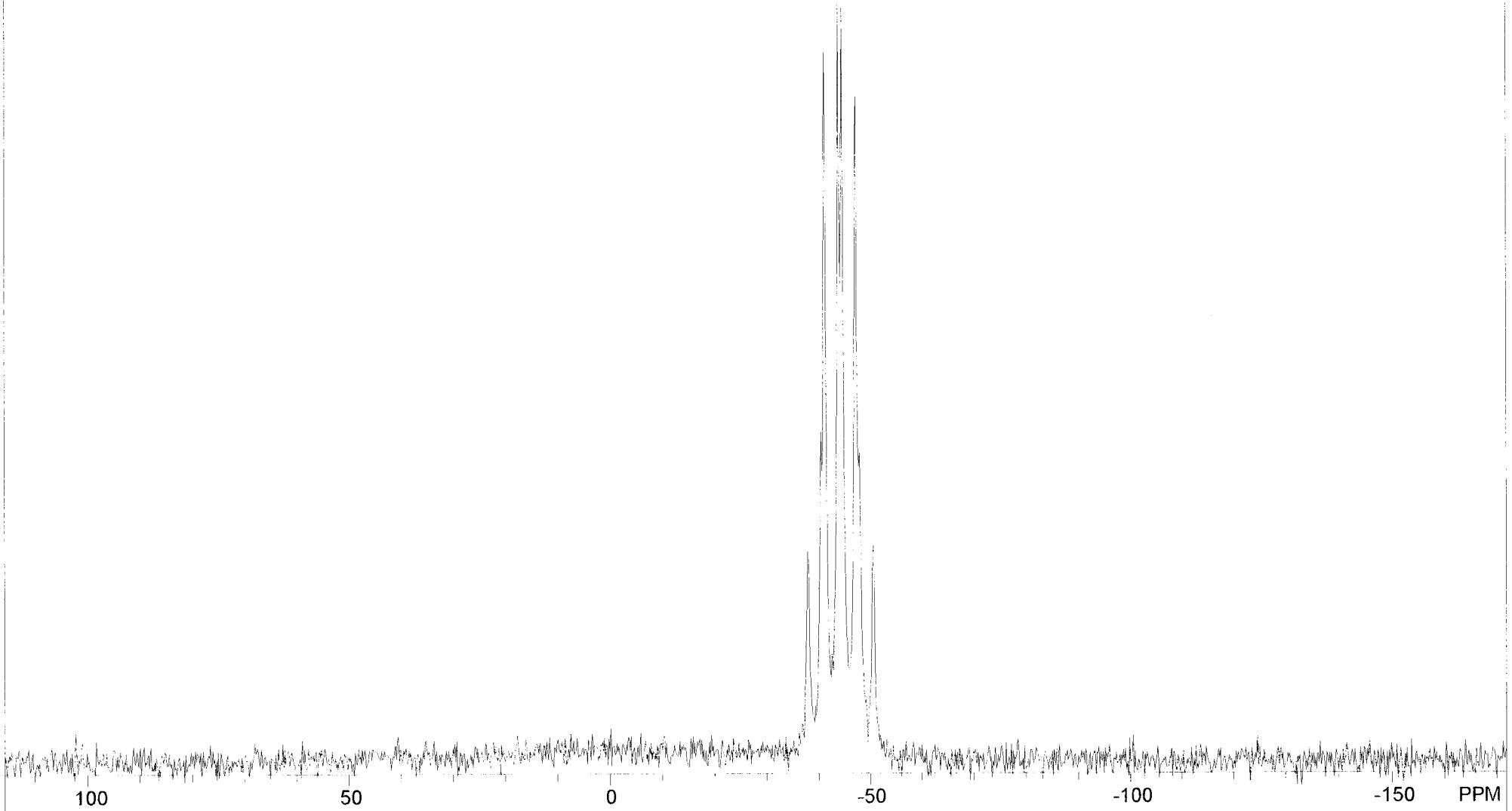
Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	1248	3799	26.35	-1096.29	-37.958
2	1264	6292	43.64	-1172.87	-40.609
3	1268	13839	95.97	-1192.83	-41.300
4	1284	14644	101.55	-1269.73	-43.963
5	1288	14232	98.70	-1288.48	-44.612
6	1304	12569	87.17	-1369.44	-47.415
7	1324	3889	26.97	-1467.68	-50.817



4i

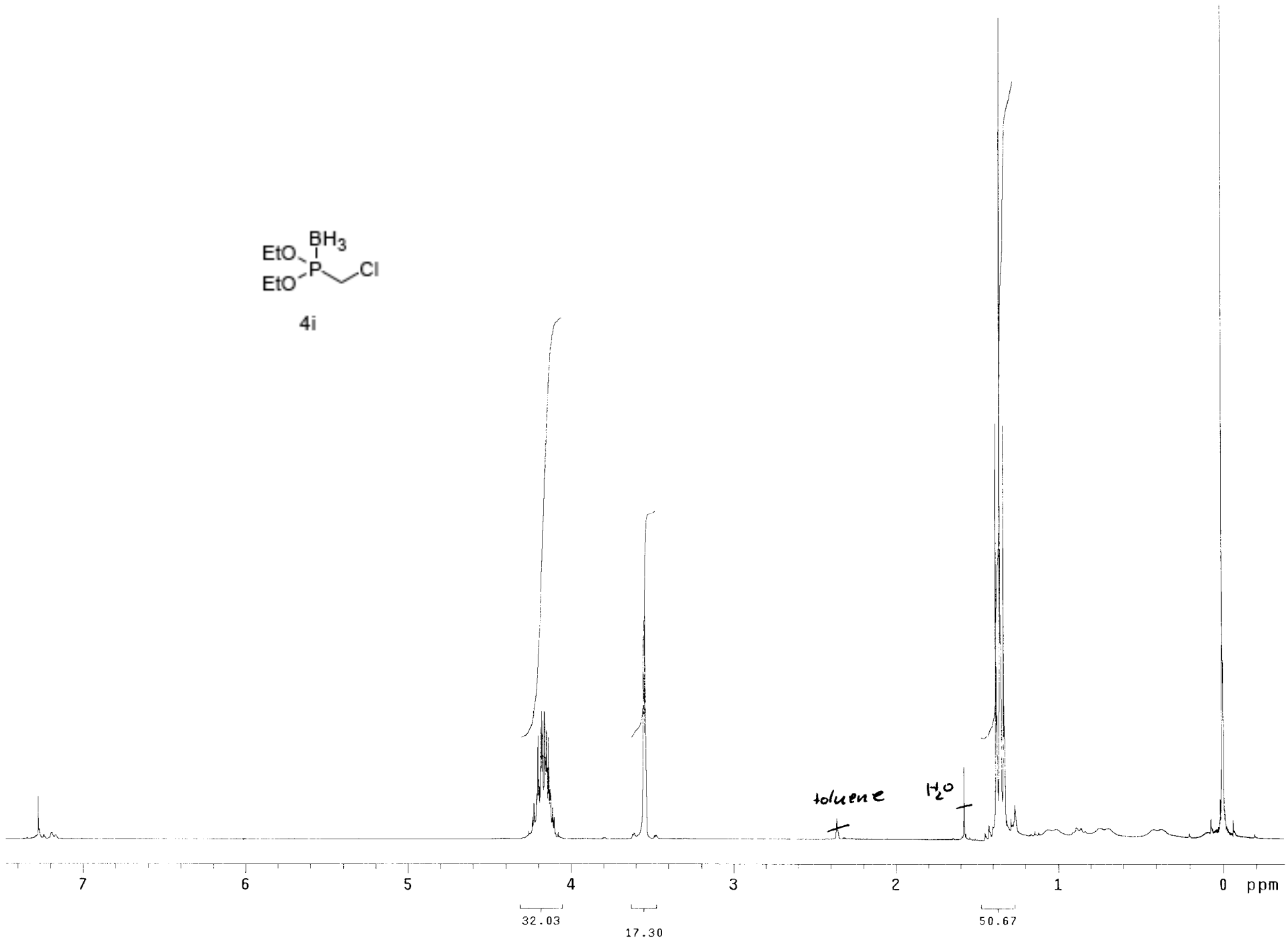
$^{11}B/^{1}H$ coupled



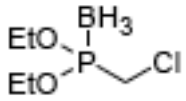
USER: -- DATE: 12/03/07 (09:57)

F1: 28.882 | SW1: 10000 | OF1: 0.0 | PTS1d: 2048

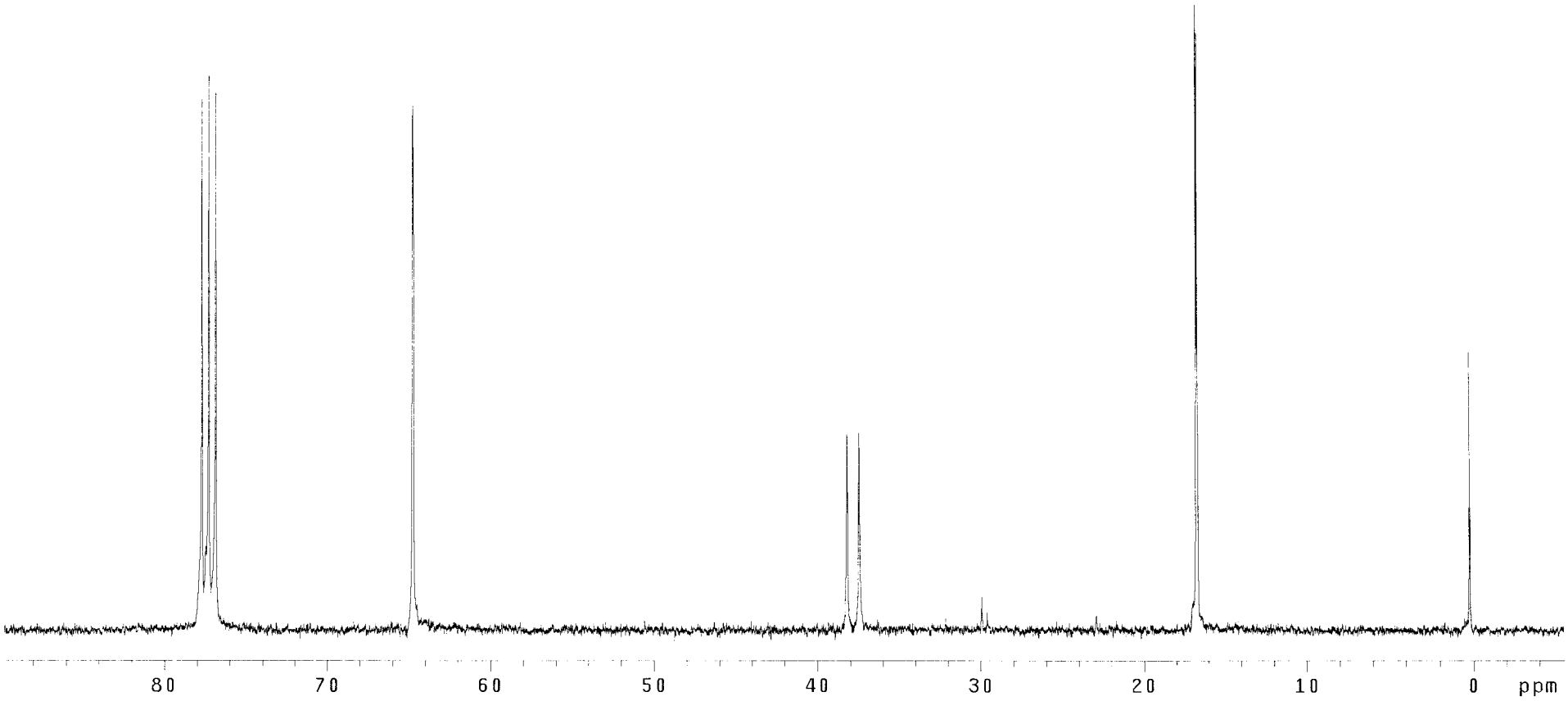
EX: c:\cft\global\zg.ppg | PW: 15.5 usec | PD: 0.1 sec | NA: 64 | LB: 0.0


EtO
BH3
P
Cl
EtO
4i
toluene
H2O
7
6
5
4
3
2
1
0
ppm
32.03
17.30
50.67

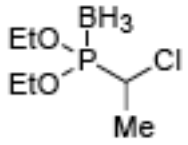
INDEX	FREQUENCY	PPM	HEIGHT
1	5861.605	77.691	85.8
2	5829.648	77.267	89.5
3	5797.691	76.844	86.9
4	4887.058	64.774	84.6
5	4883.315	64.724	77.8
6	2880.383	38.177	31.7
7	2825.106	37.444	31.9
8	1264.103	16.755	102.0
9	1258.633	16.682	96.5
10	16.337	0.217	44.8



4i

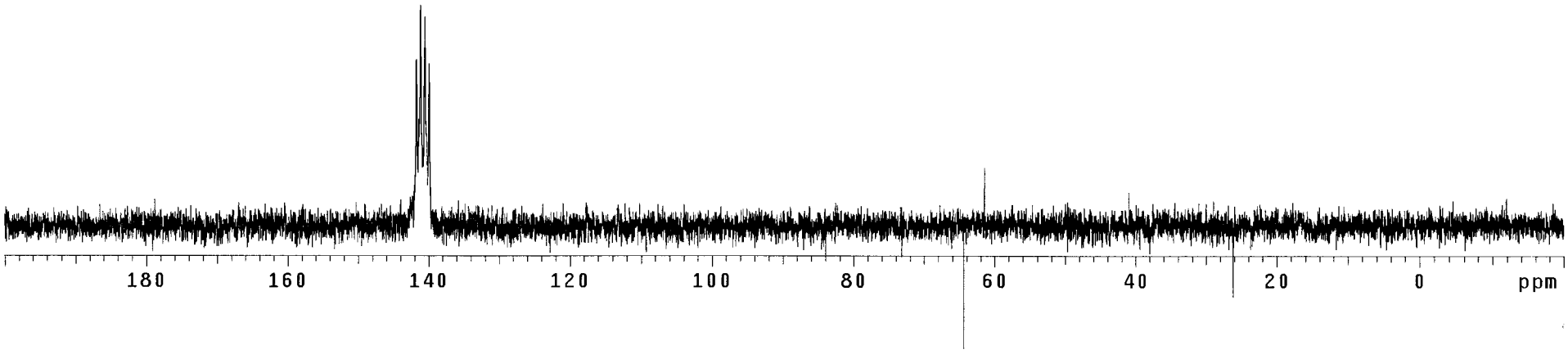


INDEX	FREQUENCY	PPM	HEIGHT
1	17219.166	141.765	26.9
2	17145.320	141.157	35.6
3	17069.842	140.536	33.8
4	16998.035	139.944	26.2
5	7819.915	64.381	-24.0
6	3181.488	26.193	-11.6

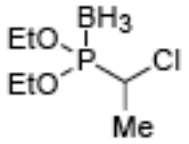


4j

$^{31}P/^{1}H$ decoupled

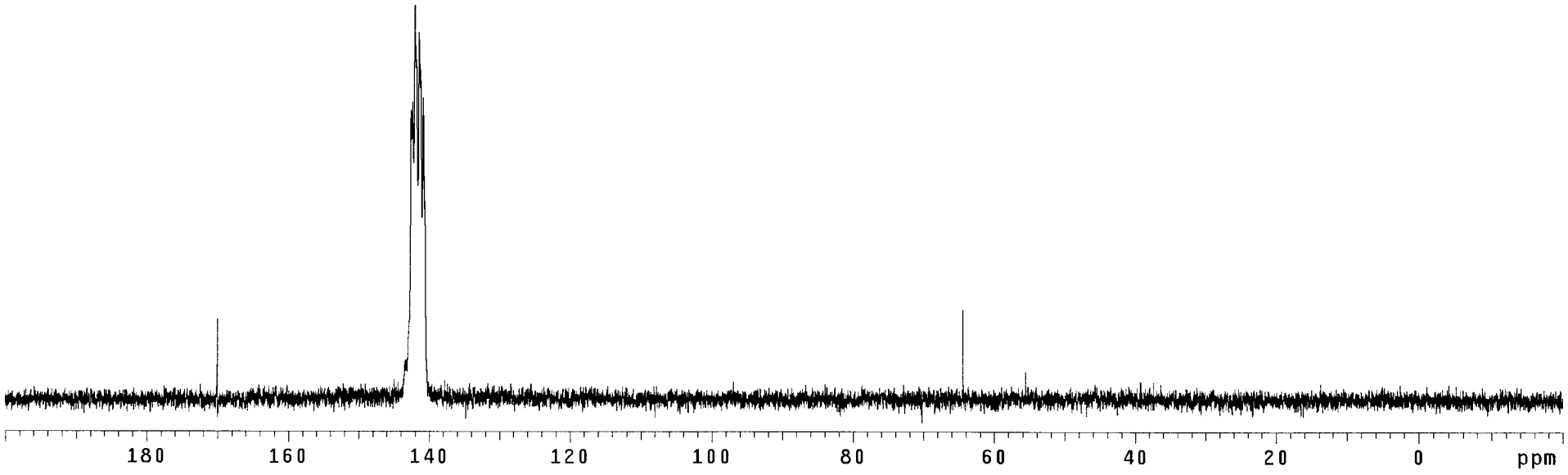


INDEX	FREQUENCY	PPM	HEIGHT
1	20654.025	170.044	13.3
2	17317.083	142.571	47.0
3	17288.932	142.339	48.3
4	17246.093	141.987	63.8
5	17175.103	141.402	59.4
6	17106.153	140.835	49.0
7	7819.507	64.378	14.8



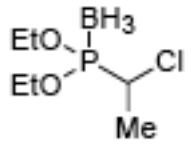
4j

$^{31}P/^{1}H$ coupled



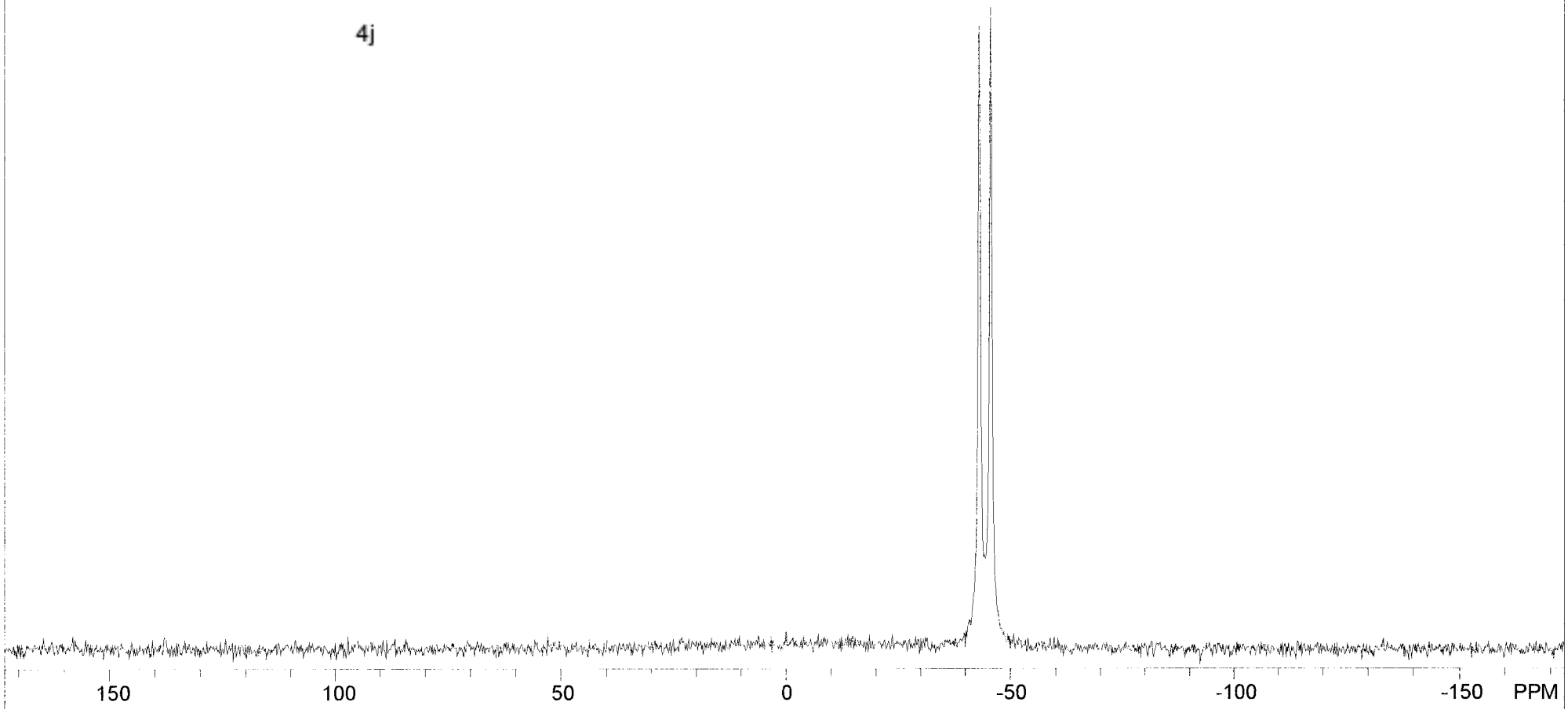
Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	1279	5481K	98.08	-1243.62	-43.059
2	1294	5817K	104.08	-1318.98	-45.668



4j

$^{11}B/^{1}H$ decoupled



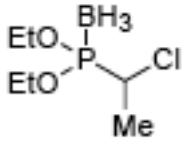
USER: -- DATE: 01/04/08 (12:10)

F1: 28.882 | SW1: 10000 | OF1: 0.0 | PTS1d: 2048

EX: c:\eft\global\zg.ppg | PW: 15.5 usec | PD: 0.1 sec | NA: 128 | LB: 0.0

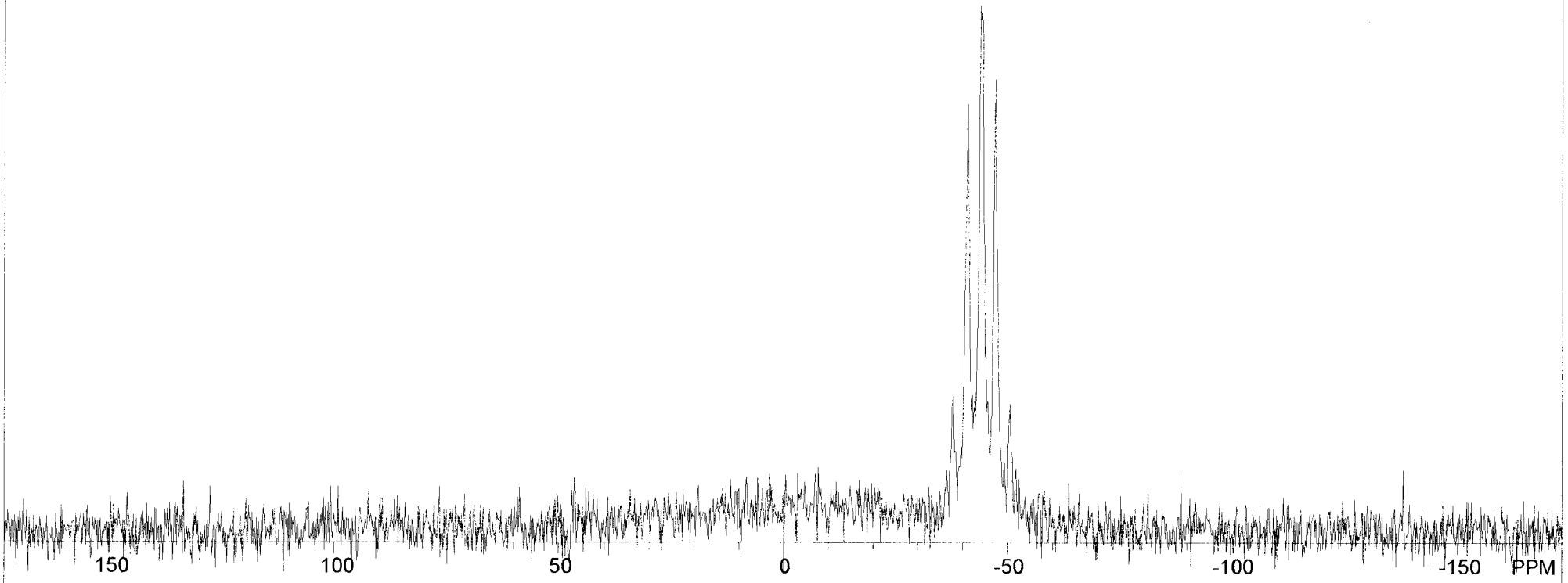
Interpolated Peak Listing

#	POINT	HEIGHT	REL. HT	HZ	PPM
1	1248	240370	22.85	-1094.92	-37.910
2	1267	803148	76.35	-1186.11	-41.068
3	1284	971077	92.32	-1271.44	-44.022
4	1303	832301	79.13	-1361.11	-47.127
5	1315	121847	11.58	-1419.31	-49.142
6	1323	232877	22.14	-1461.62	-50.607



4j

$^{11}B/^{1}H$ coupled



USER: -- DATE: 01/04/08 (12:16)

F1: 28.882	SW1: 10000		OF1: 0.0		PTS1d: 2048
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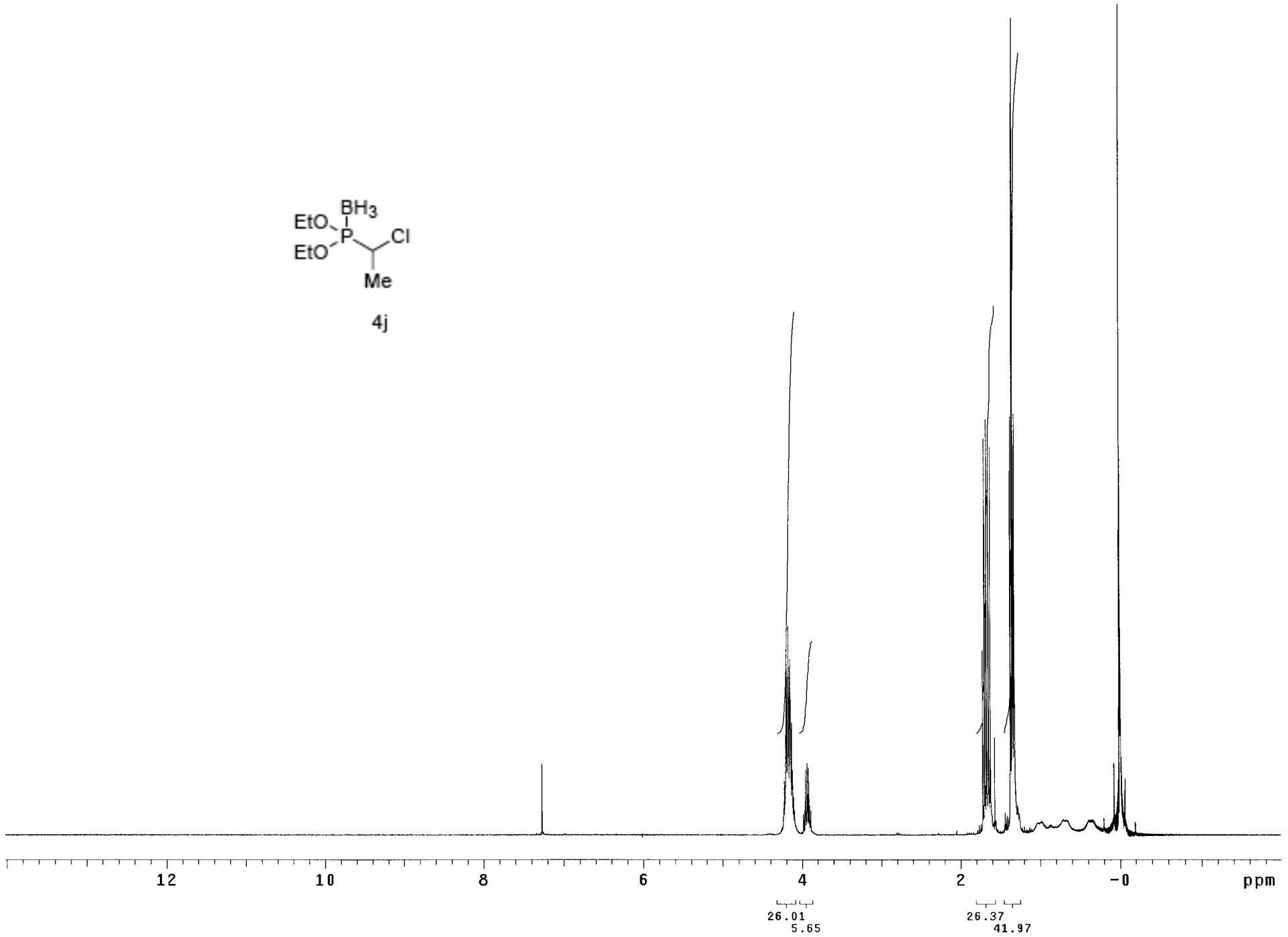
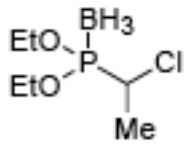
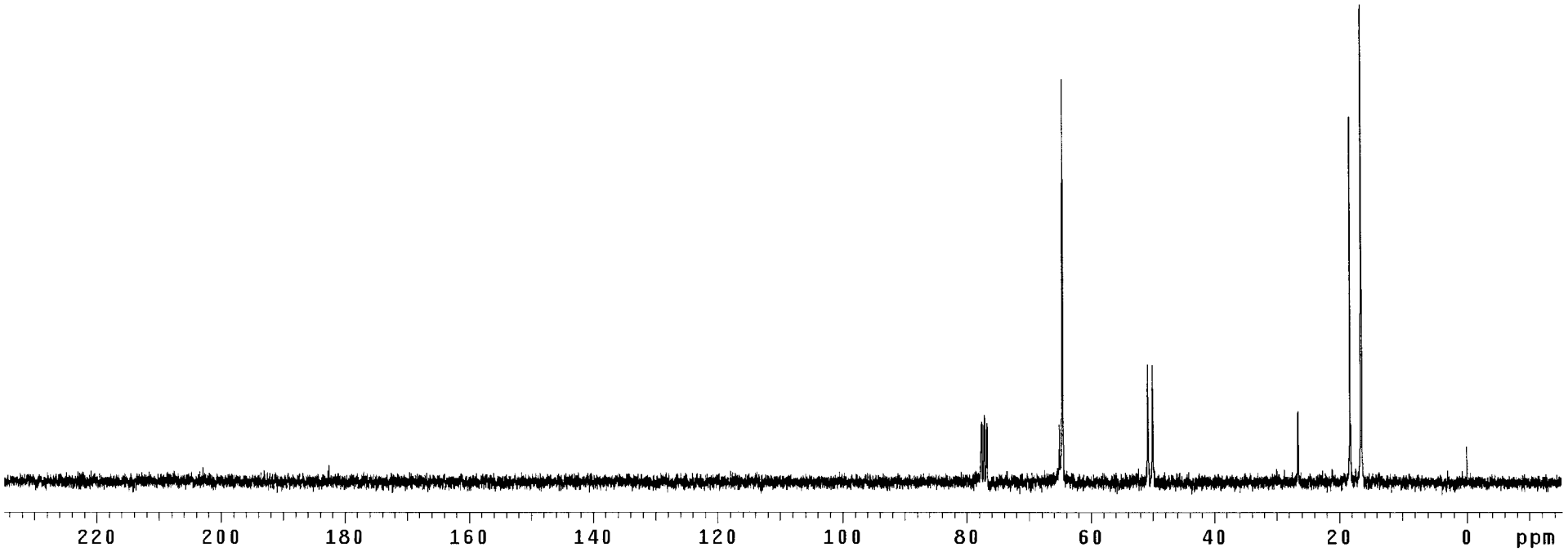

BH3
EtO
EtO
P
Cl
Me
4j
12
10
8
6
4
2
-0
ppm
26.01
5.65
26.37
41.97

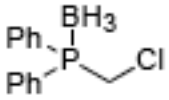
INDEX	FREQUENCY	PPM	HEIGHT
1	5857.072	77.631	9.8
2	5823.099	77.180	10.9
3	5791.718	76.764	9.5
4	4911.603	65.099	9.2
5	4875.615	64.622	65.1
6	3831.683	50.786	19.1
7	3773.527	50.015	18.9
8	2005.523	26.582	11.5
9	1378.185	18.267	59.1
10	1243.159	16.477	77.3
11	0.000	0.000	5.6



4j

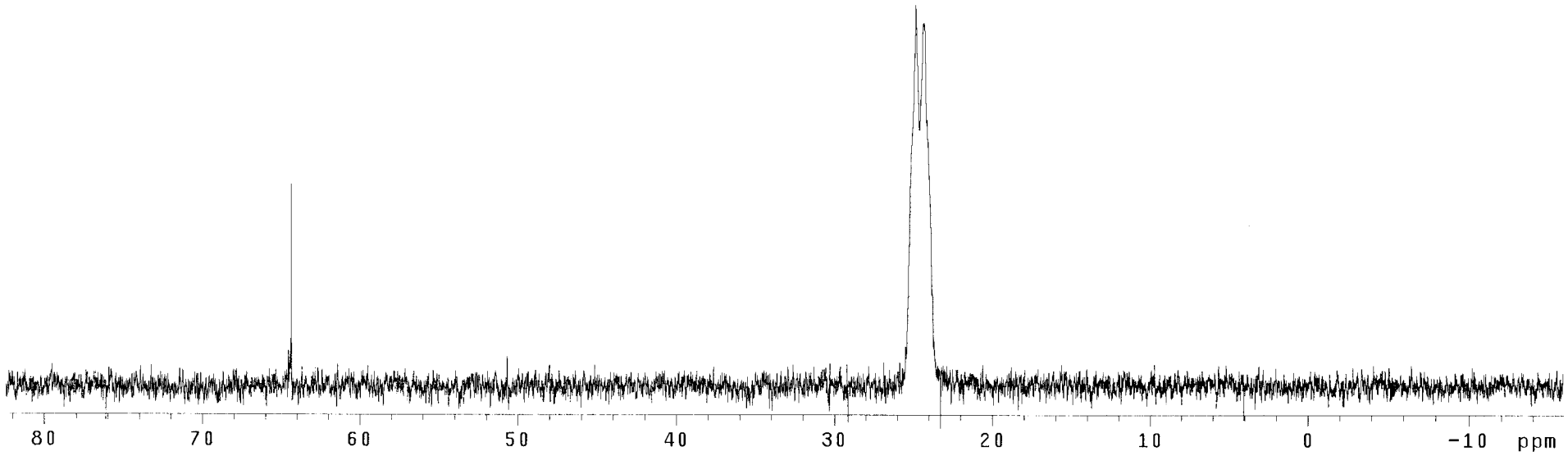


INDEX	FREQUENCY	PPM	HEIGHT
1	7819.915	64.381	32.2
2	3014.212	24.816	61.0
3	2953.014	24.312	58.1

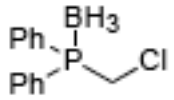


4k

$^{31}P/^{1}H$ decoupled



INDEX	FREQUENCY	PPM	HEIGHT
1	7819.915	64.381	26.0
2	3014.620	24.819	17.6

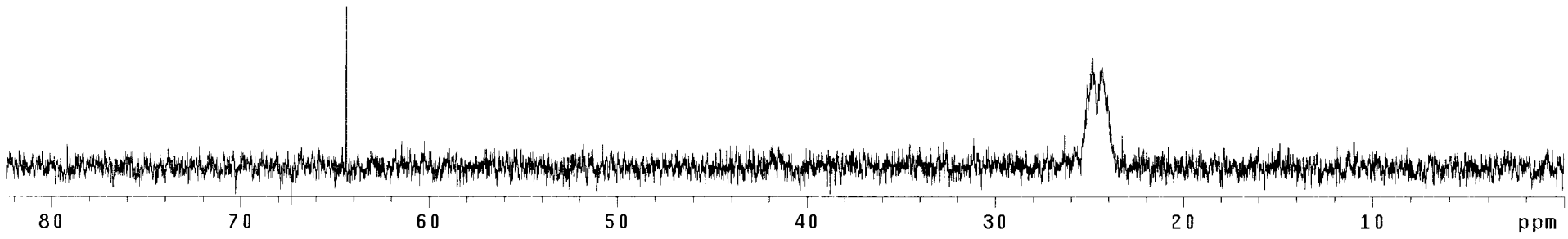


4k

$^{31}P/^{1}H$ coupled

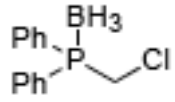
exp1 s2pul

SAMPLE		SPECIAL	
date	Nov 13 2007	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	128	**PROCESSING**	
ct	128	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	-169.6
dn	H1	lp	-451.8
dof	0	**PLOT**	
dm	yyy	wc	250
dmm	w	sc	0
dpwr	35	vs	200
dmf	6700	th	17
		ai	no ph



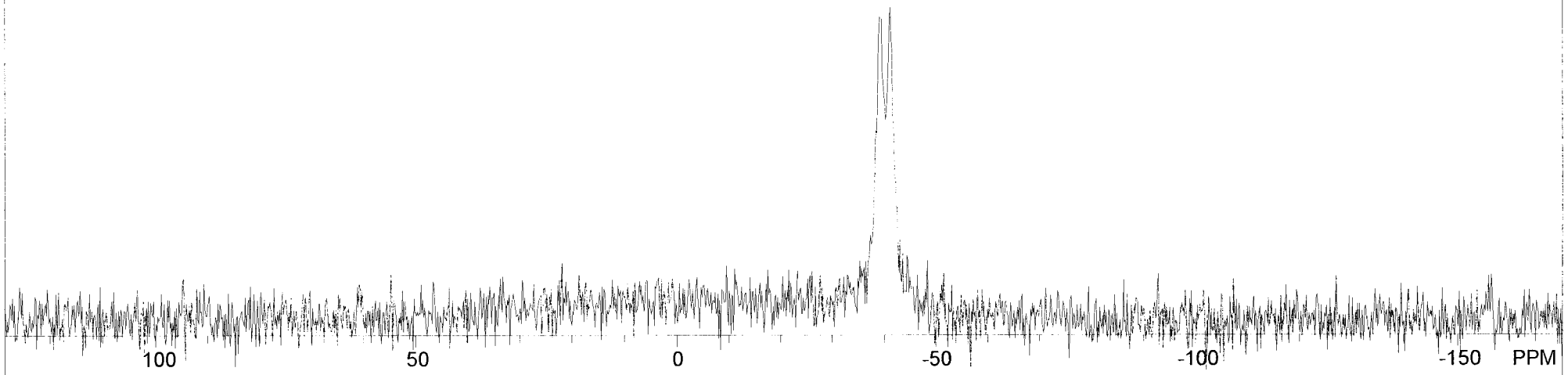
Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	1258	5728	78.90	-1145.29	-39.654
2	1268	6175	85.05	-1189.90	-41.199
3	1279	1970	27.14	-1244.24	-43.080



4k

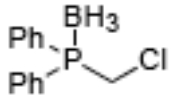
$^{11}B/^{1}H$ decoupled



USER: -- DATE: 12/03/07 (12:28)

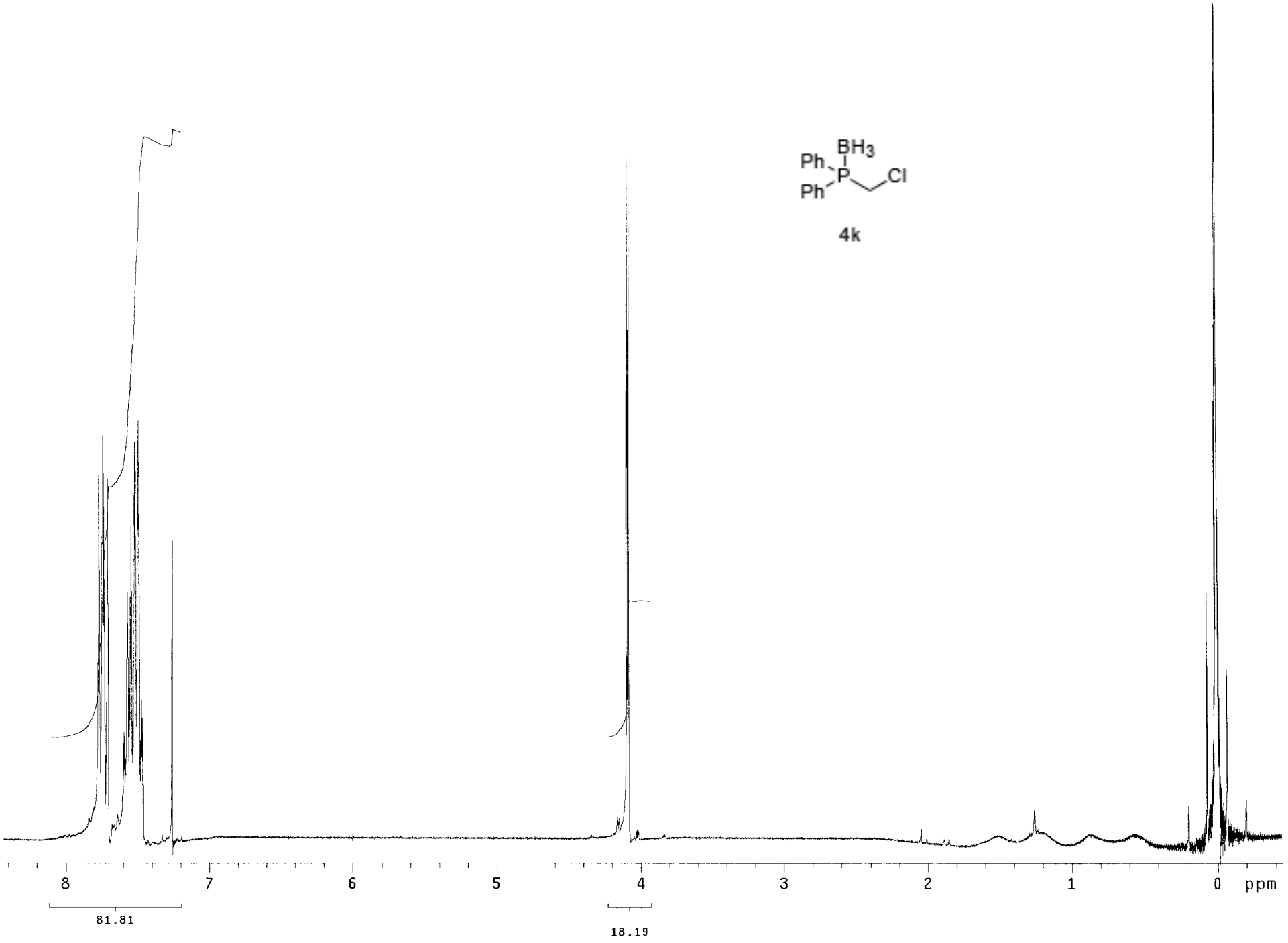
F1: 28.882 | SW1: 10000 | OF1: 0.0 | PTS1d: 2048

FX: c:\eft\global\zg.ppg | PW: 15.5 usec | PD: 0.1 sec | NA: 128 | LB: 0.0

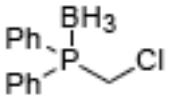


4k

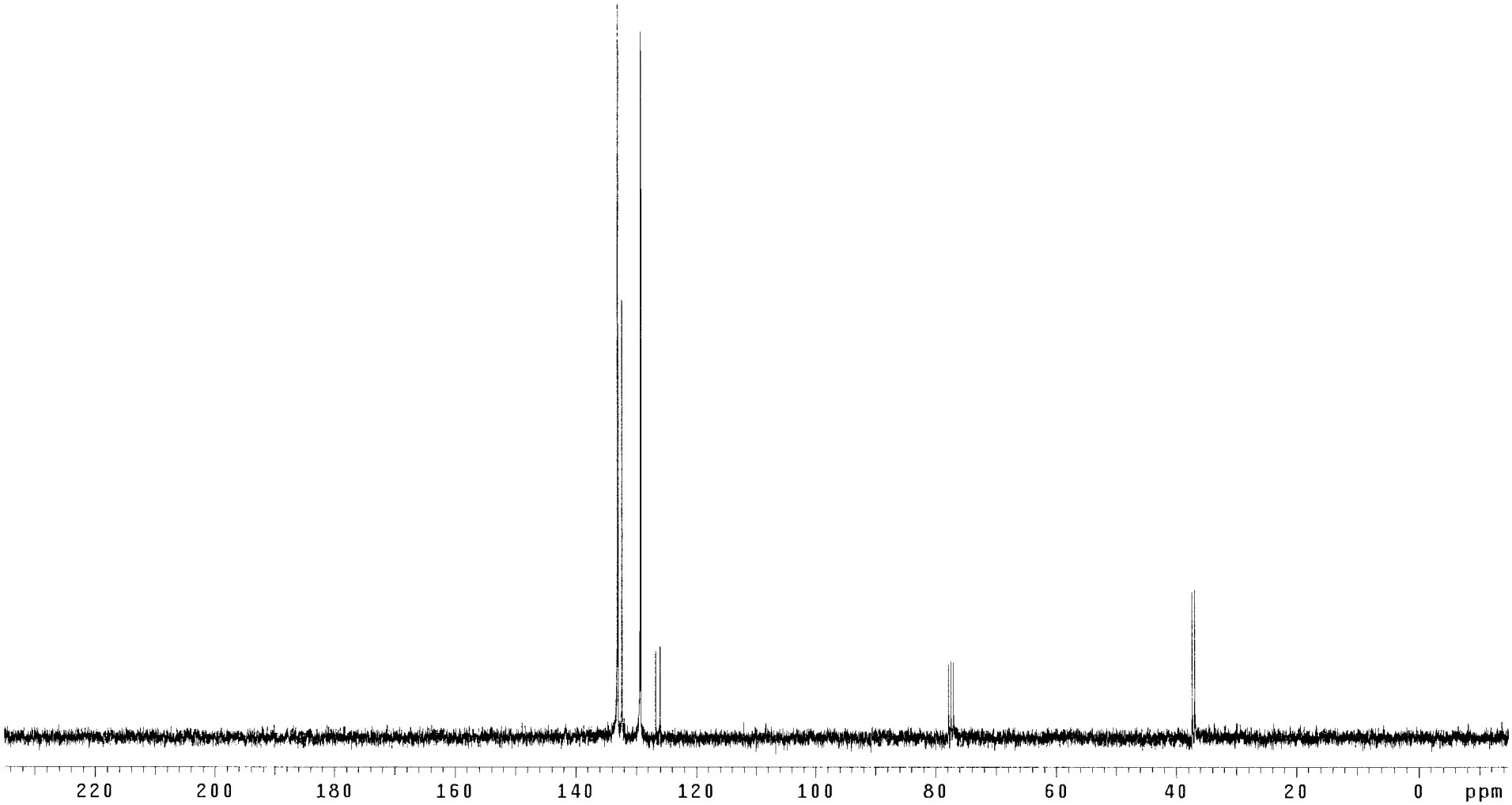
$^{11}B/^{1}H$ coupled


BH3
Ph
P
Cl
Ph
4k
8
7
6
5
4
3
2
1
0
ppm
81.81
18.19

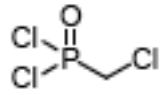
INDEX	FREQUENCY	PPM	HEIGHT
1	10046.544	133.159	122.9
2	10037.043	133.033	115.4
3	9986.372	132.361	73.2
4	9983.781	132.327	72.9
5	9758.930	129.347	118.2
6	9748.853	129.213	114.9
7	9566.036	126.790	14.5
8	9509.031	126.034	15.3
9	5875.136	77.870	12.3
10	5843.179	77.447	12.7
11	5810.934	77.019	12.6
12	2817.620	37.345	24.5
13	2785.951	36.926	24.8



4k

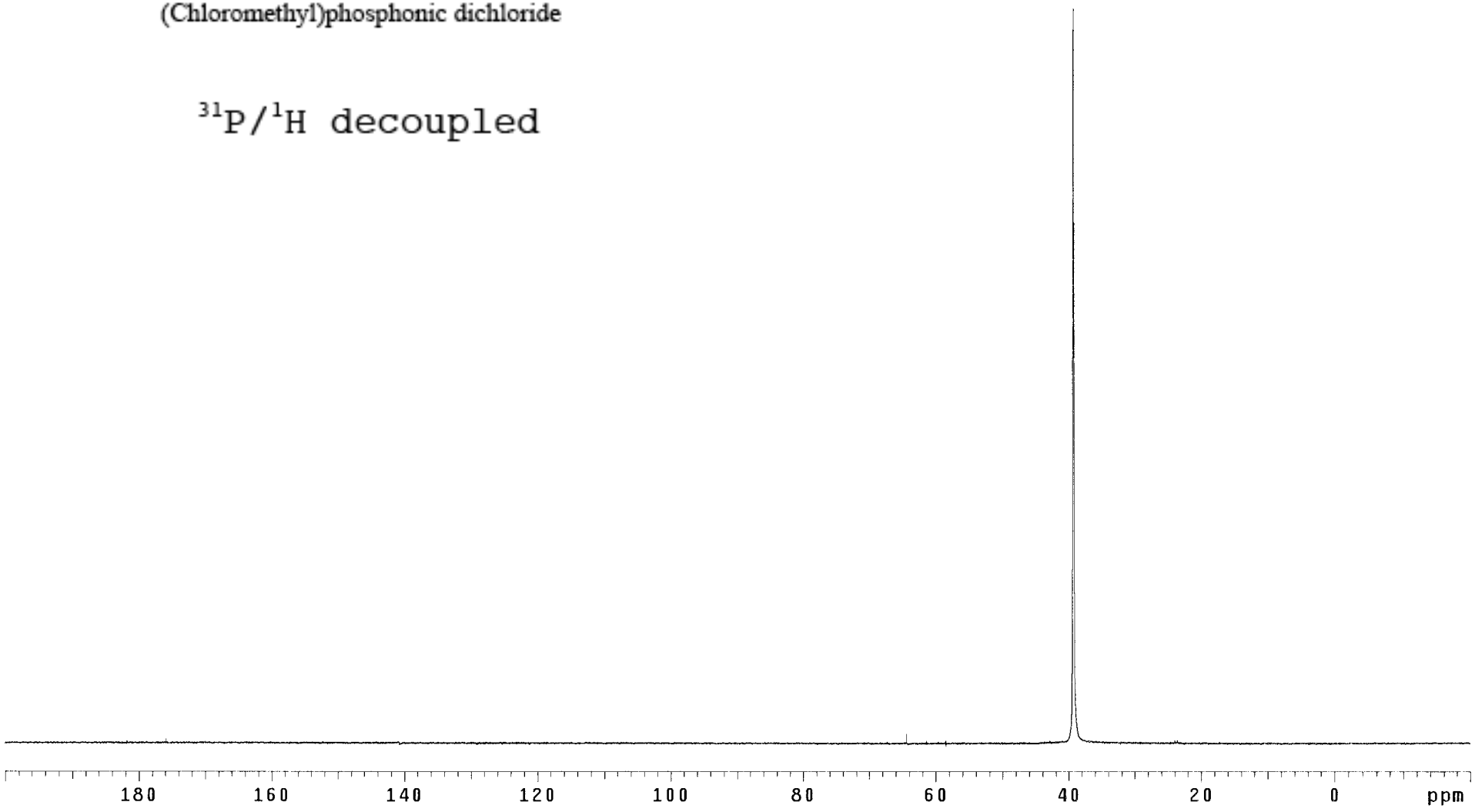


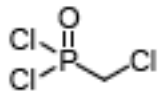
INDEX	FREQUENCY	PPM	HEIGHT
1	4768.565	39.259	126.0



(Chloromethyl)phosphonic dichloride

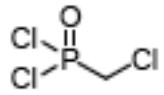
$^{31}P/^{1}H$ decoupled



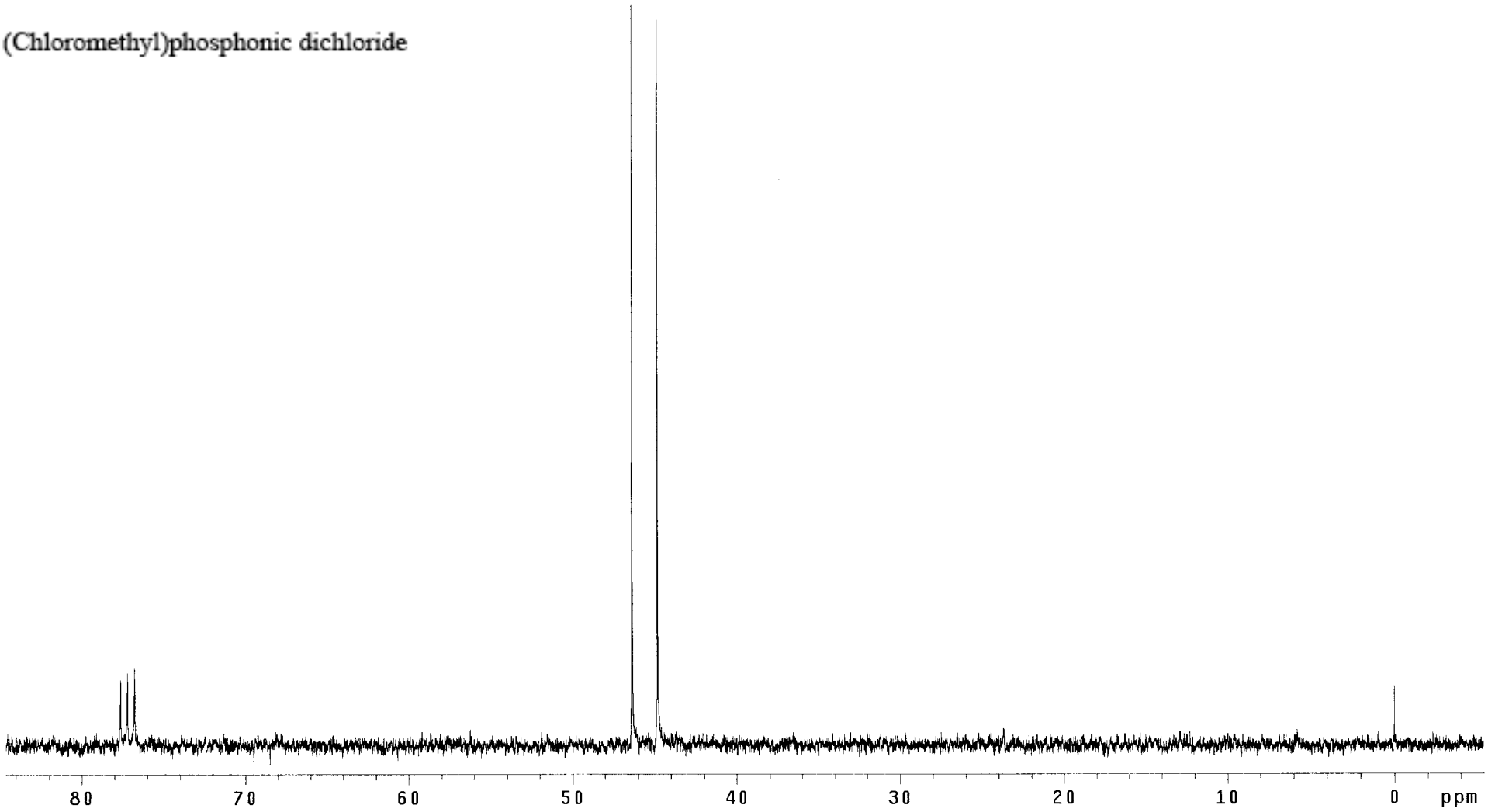


(Chloromethyl)phosphonic dichloride

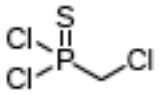
INDEX	FREQUENCY	PPM	HEIGHT
1	3499.157	46.378	126.2
2	3382.269	44.829	123.5



(Chloromethyl)phosphonic dichloride

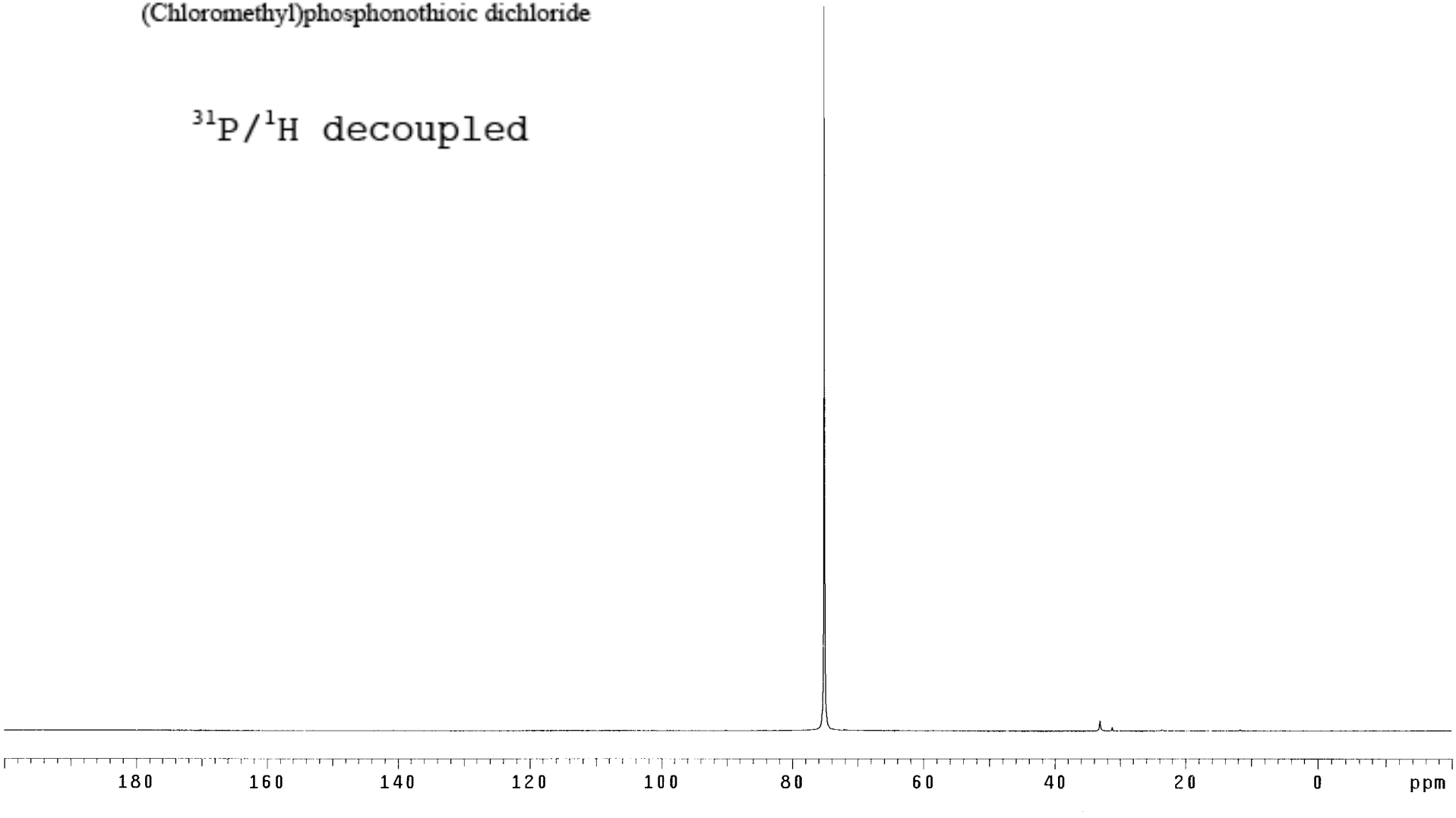


INDEX	FREQUENCY	PPM	HEIGHT
1	9125.072	75.126	126.0

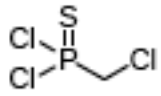


(Chloromethyl)phosphonothioic dichloride

$^{31}P/^{1}H$ decoupled

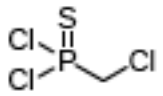


INDEX	FREQUENCY	PPM	HEIGHT
1	1288.715	4.295	116.3
2	1288.129	4.293	82.8
3	1287.543	4.291	75.7
4	1285.784	4.285	111.1
5	1285.198	4.283	75.0
6	1284.611	4.281	66.4
7	1.173	0.004	180.7
8	0.586	0.002	118.6
9	-0.000	-0.000	96.0

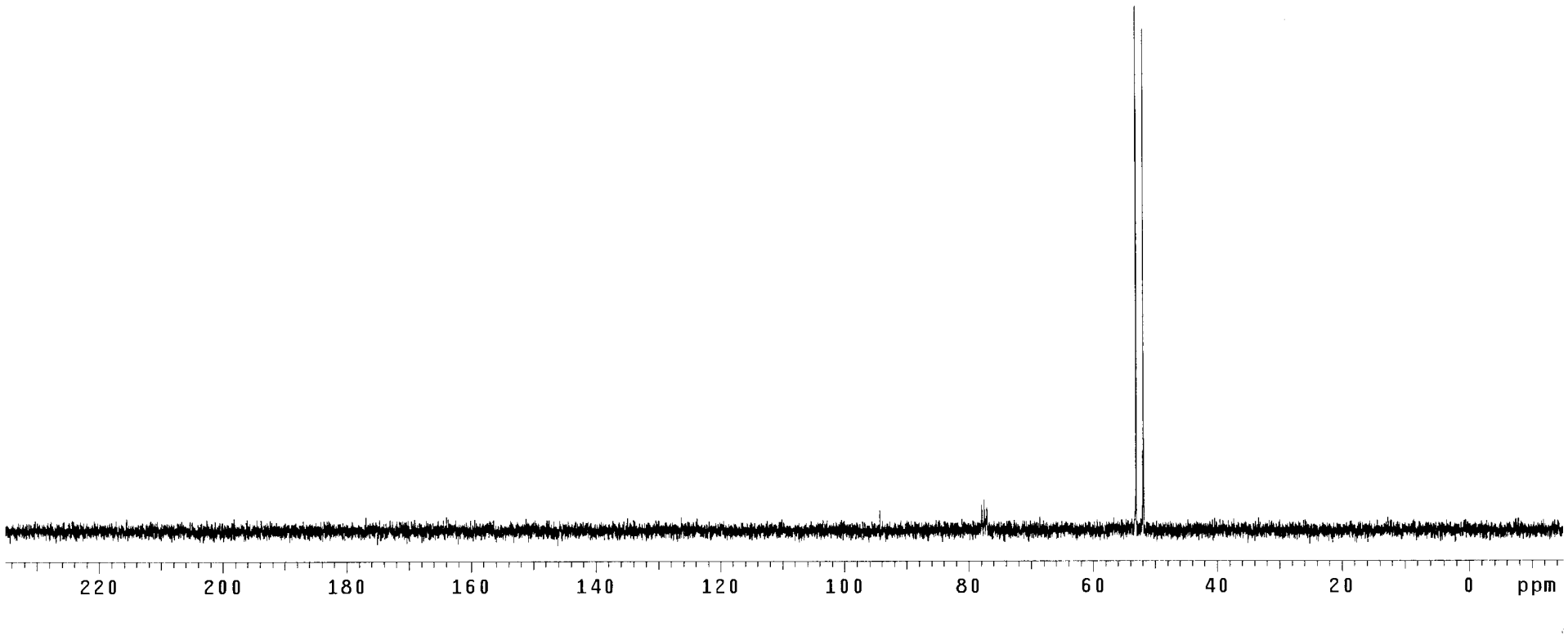


(Chloromethyl)phosphonothioic dichloride

INDEX	FREQUENCY	PPM	HEIGHT
1	4003.487	53.063	84.6
2	3916.541	51.911	80.9

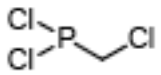


(Chloromethyl)phosphonothioic dichloride



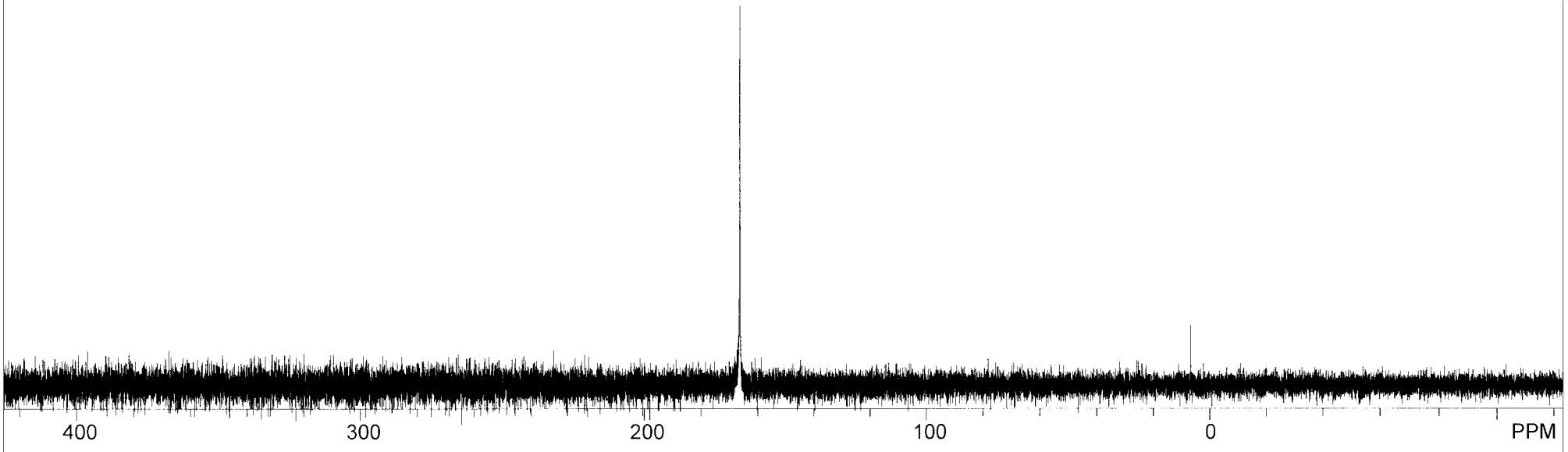
Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	15487	15663K	90.65	6057.21	166.222



Dichloro-(chloromethyl)phosphine

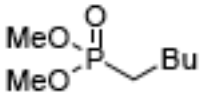
$^{31}P/^{1}H$ decoupled



USER: -- DATE: 05/25/06 (16:58)

F1: 36.441	SW1: 20000		OF1: 5510.0		PTS1d: 32768
EX: c:\eft\global\zg.ppg	PW: 12.4 usec	PD: 2.0 sec	NA: 64	LB: 0.0	

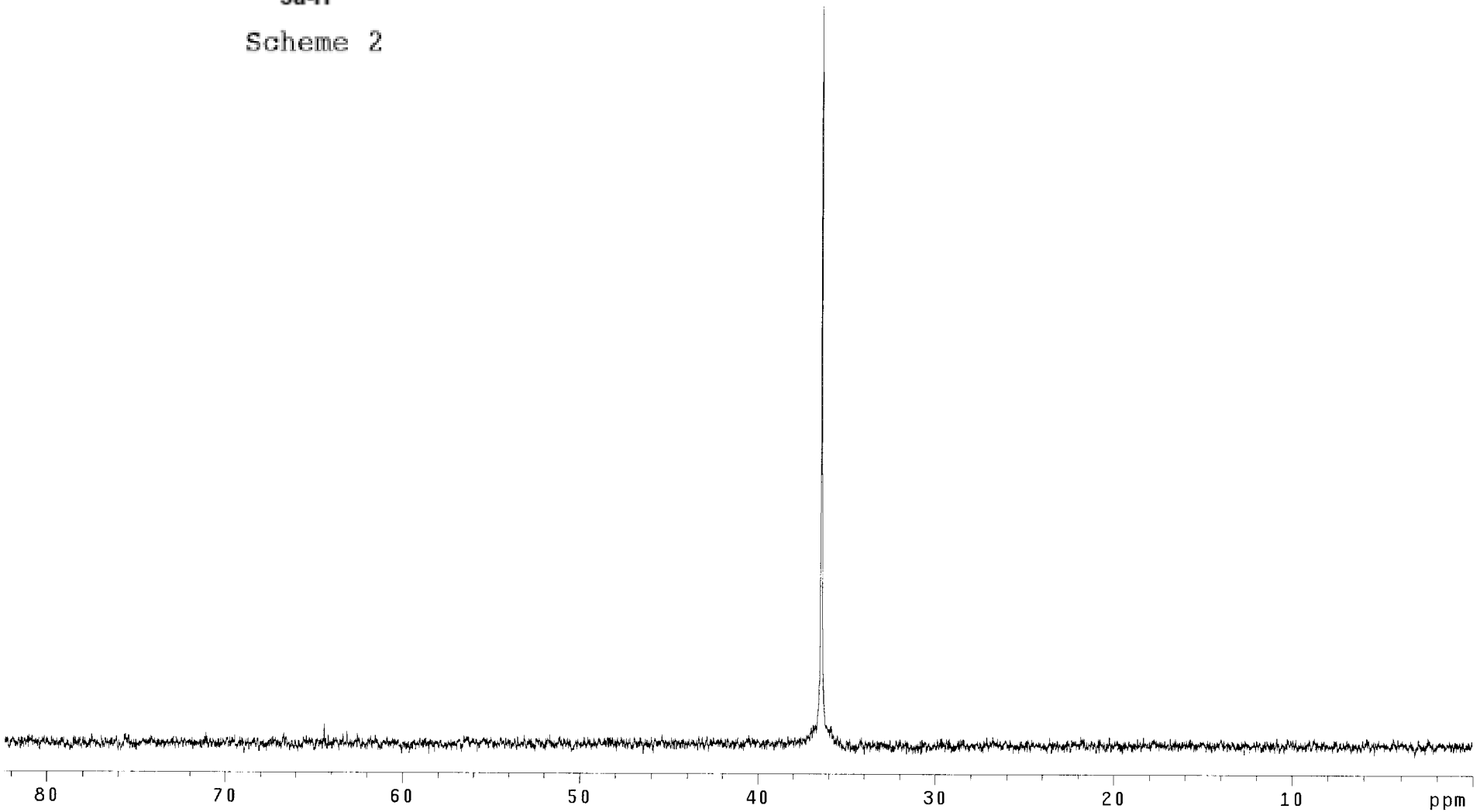
INDEX	FREQUENCY	PPM	HEIGHT
1	4425.038	36.431	126.0



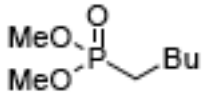
3a-H

$^{31}P/^{1}H$ decoupled

Scheme 2



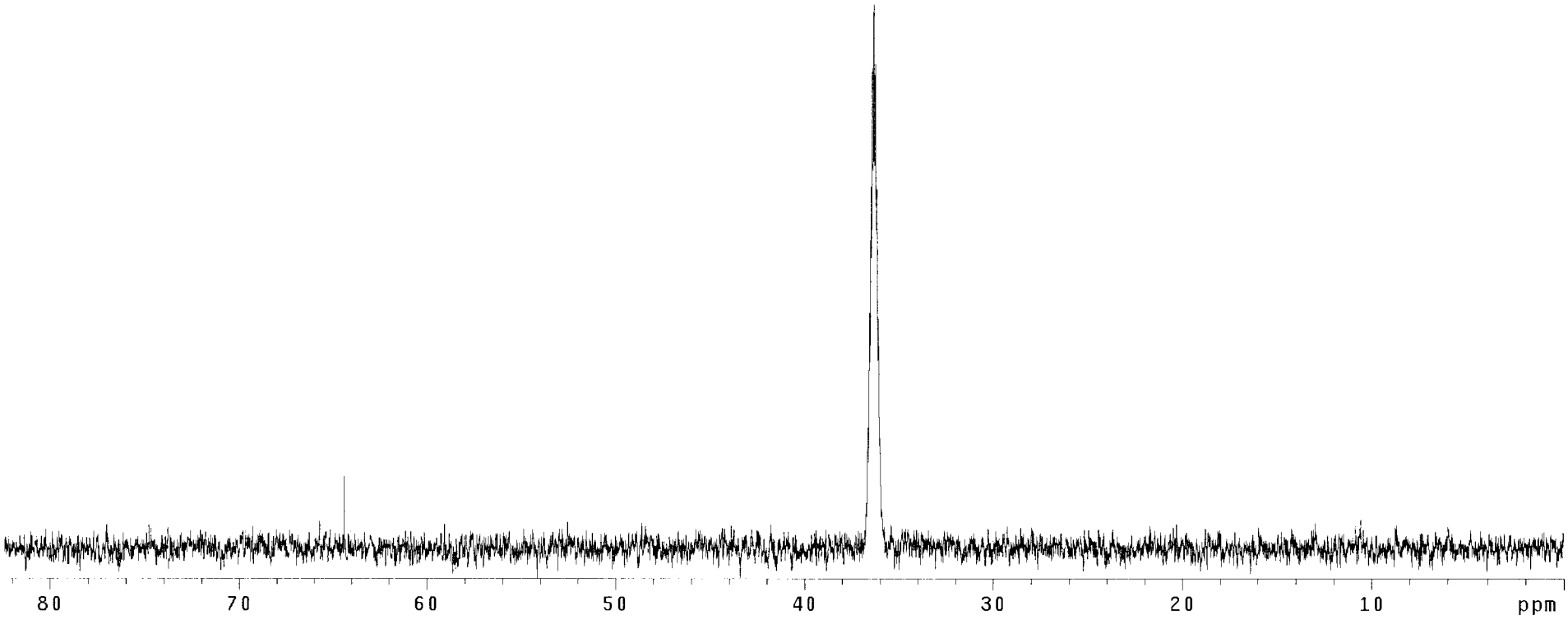
INDEX	FREQUENCY	PPM	HEIGHT
1	4440.950	36.562	37.7
2	4429.934	36.472	57.9
3	4422.590	36.411	77.0
4	4412.390	36.327	87.2
5	4401.783	36.240	77.7
6	4394.847	36.183	57.5
7	4384.239	36.095	36.6

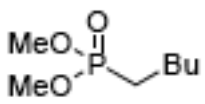


3a-H

$^{31}P/^{1}H$ coupled

Scheme 2

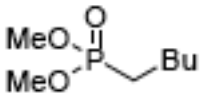




3a-H

Scheme 2

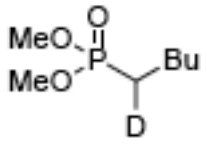
INDEX	FREQUENCY	PPM	HEIGHT
1	5860.741	77.679	153.6
2	5828.784	77.256	157.4
3	5796.827	76.832	154.0
4	3961.454	52.506	39.4
5	3954.832	52.418	39.7
6	2495.746	33.079	44.7
7	2478.760	32.854	46.0
8	1945.854	25.791	47.1
9	1805.646	23.932	46.4
10	1688.470	22.379	53.5
11	1678.681	22.250	48.1
12	1673.499	22.181	48.2
13	1059.405	14.042	65.7
14	15.761	0.209	97.1



3a-H

Scheme 2

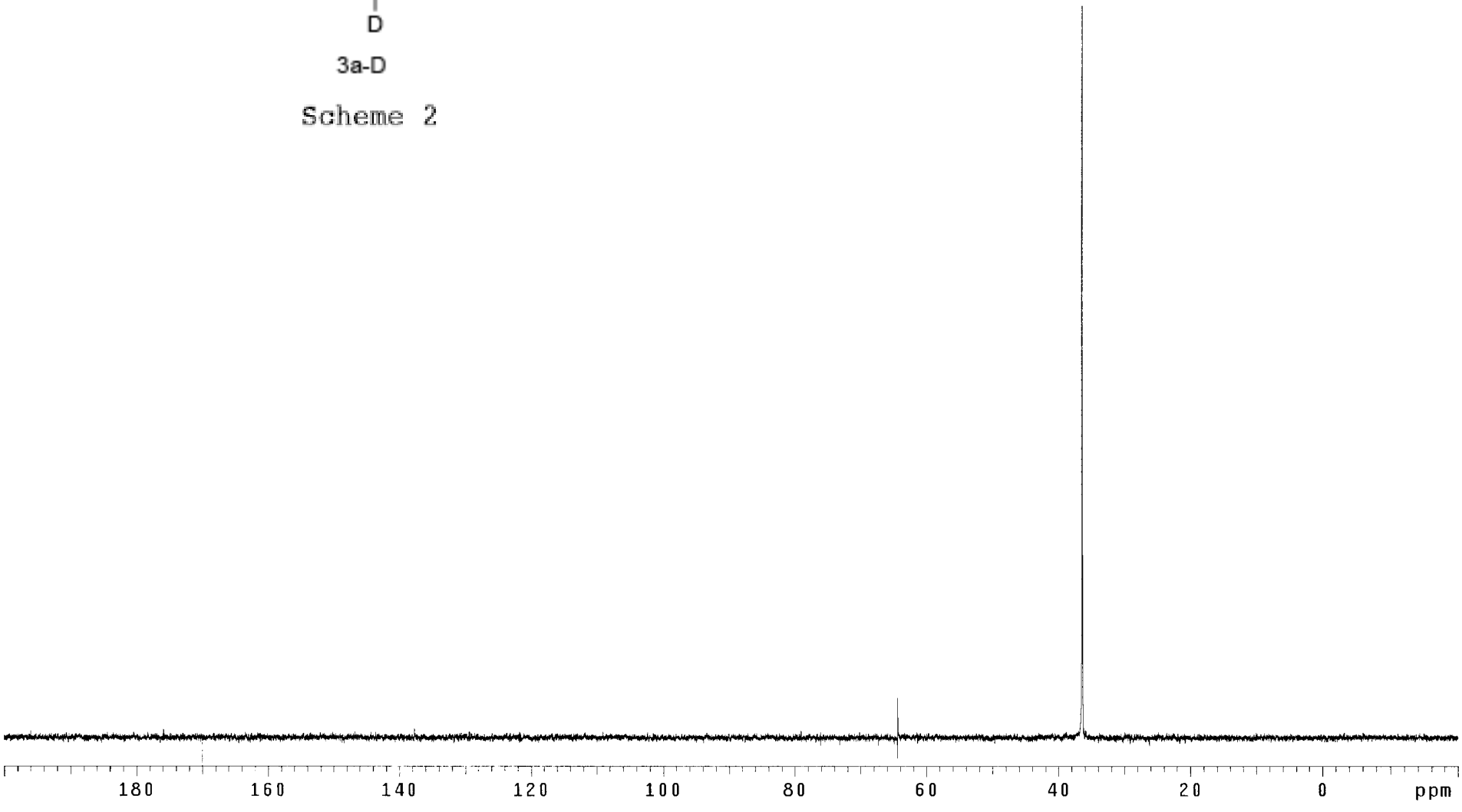
INDEX	FREQUENCY	PPM	HEIGHT
1	20653.209	170.037	-6.4
2	7819.099	64.374	6.7
3	4424.630	36.428	126.0



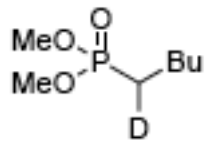
3a-D

Scheme 2

$^{31}P/^{1}H$ decoupled



INDEX	FREQUENCY	PPM	HEIGHT
1	4425.854	36.438	126.0



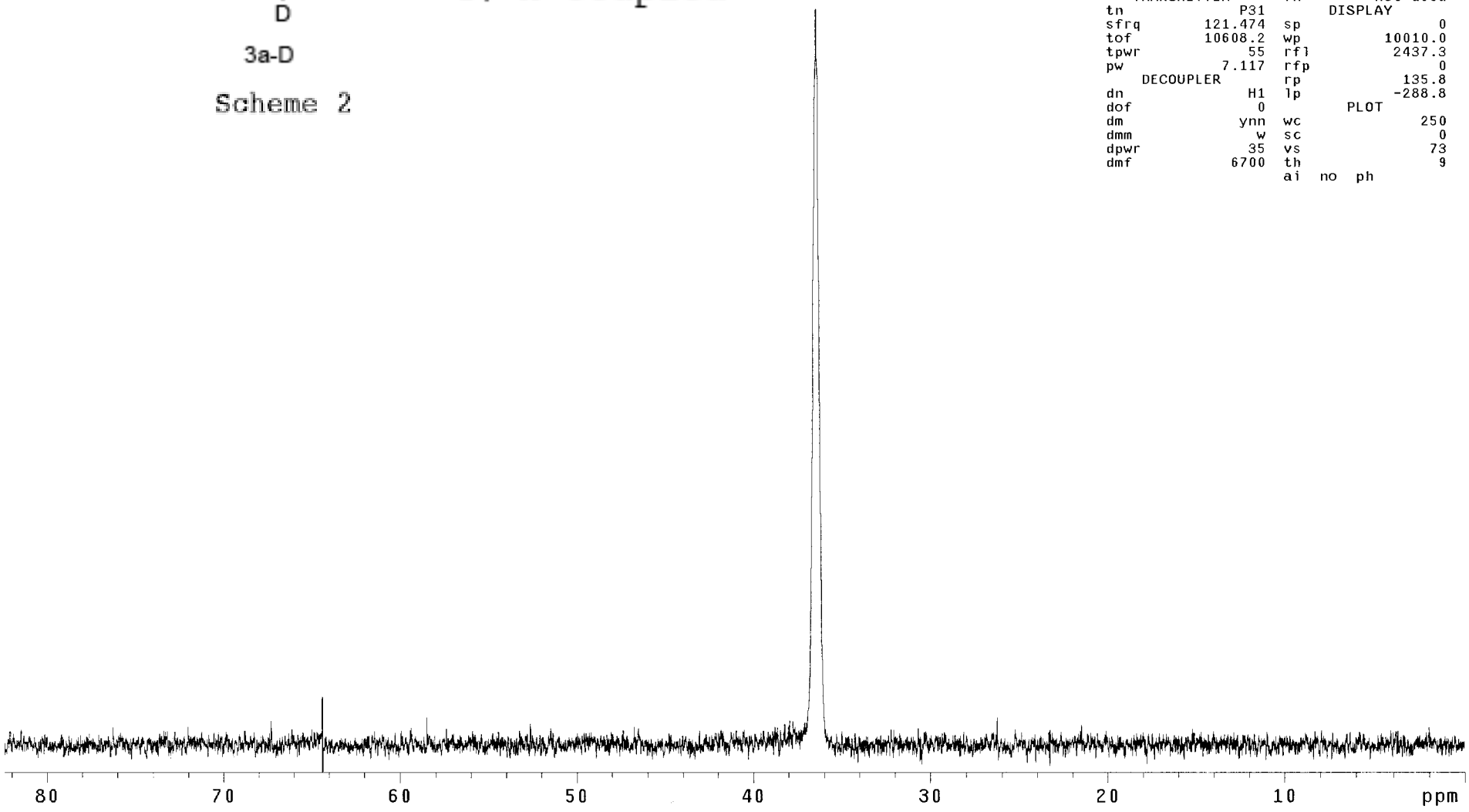
3a-D

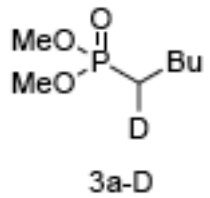
Scheme 2

$^{31}P/^{1}H$ coupled

exp1 s2pul

SAMPLE		SPECIAL	
date	Feb 1 2006	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	135.8
dn	H1	lp	-288.8
dof	0	PLOT	
dm	ynn	wc	250
dmm	w	sc	0
dpwr	35	vs	73
dmf	6700	th	9
		ai no ph	

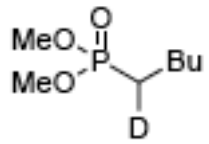




3a-D

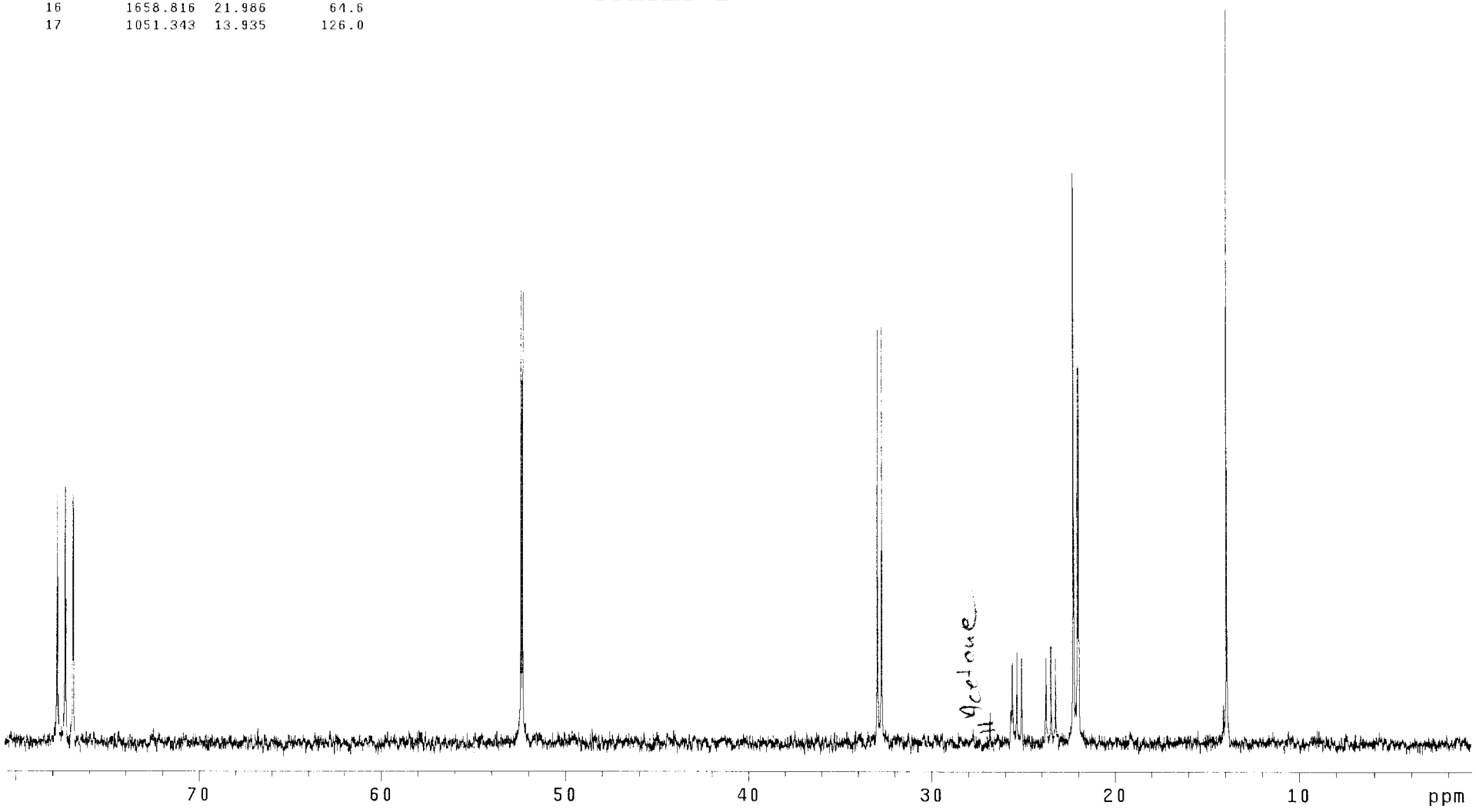
Scheme 2

INDEX	FREQUENCY	PPM	HEIGHT
1	5866.499	77.756	42.5
2	5834.542	77.332	43.8
3	5802.297	76.905	42.3
4	3953.392	52.399	77.7
5	3946.771	52.311	77.3
6	2485.670	32.946	70.8
7	2468.971	32.724	71.5
8	1932.610	25.615	13.7
9	1913.321	25.360	15.7
10	1893.744	25.100	14.6
11	1792.402	23.757	14.6
12	1773.113	23.501	16.7
13	1753.823	23.245	14.6
14	1681.560	22.288	97.9
15	1664.286	22.059	67.4
16	1658.816	21.986	64.6
17	1051.343	13.935	126.0

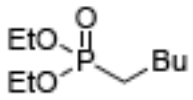


3a-D

Scheme 2



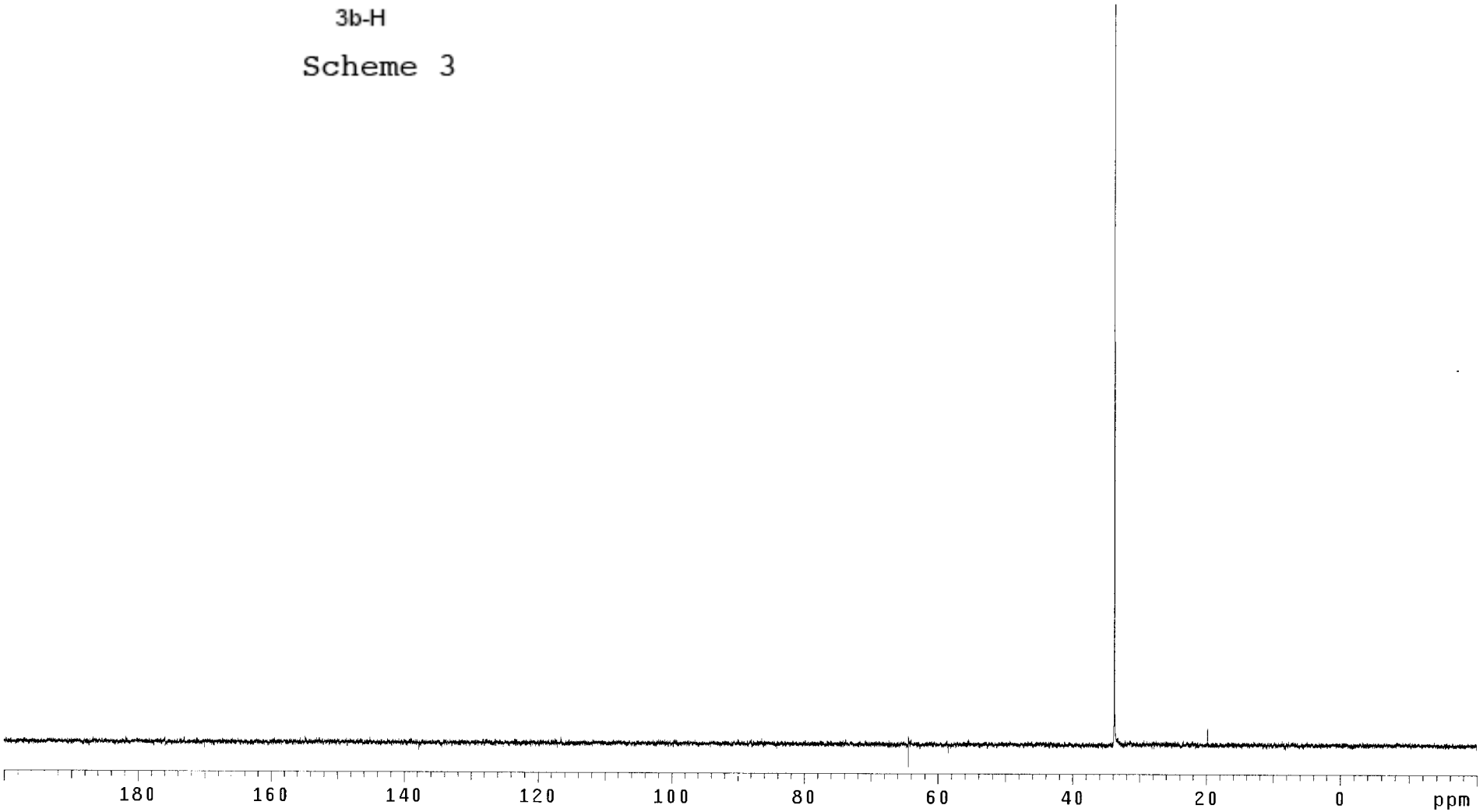
INDEX	FREQUENCY	PPM	HEIGHT
1	4101.095	33.764	126.0



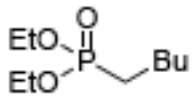
3b-H

Scheme 3

$^{31}P/^{1}H$ decoupled



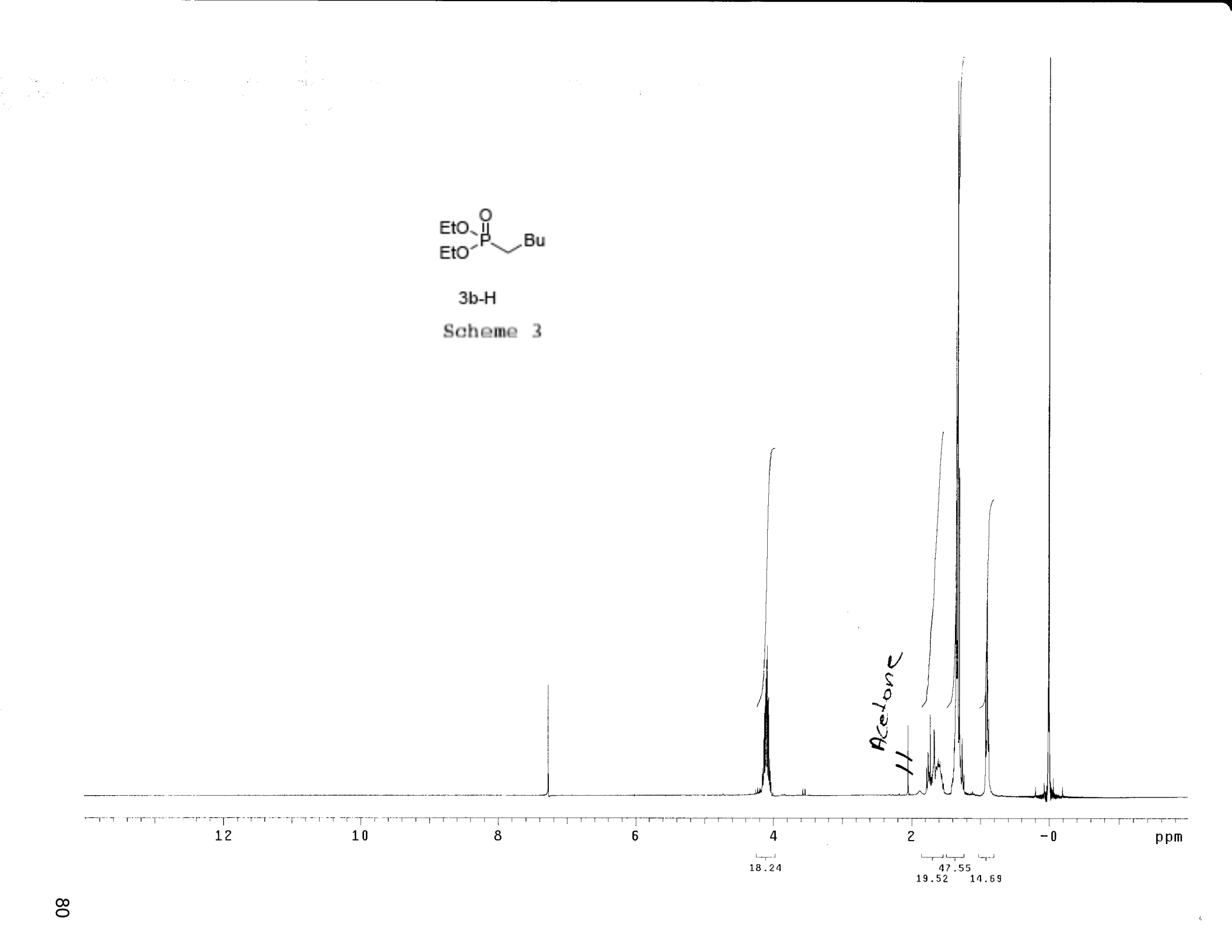
INDEX	FREQUENCY	PPM	HEIGHT
1	4127.614	33.983	61.9
2	4119.454	33.915	111.6
3	4110.479	33.841	149.7
4	4102.319	33.774	168.5
5	4093.751	33.704	149.7
6	4085.183	33.633	107.2
7	4077.023	33.566	59.3



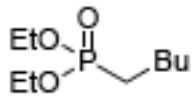
$^{31}P/^{1}H$ coupled

3b-H

Scheme 3


EtO
O
P
Bu
EtO
3b-H
Scheme 3
Acetone
12
10
8
6
4
2
-0
ppm
18.24
47.55
19.52
14.69

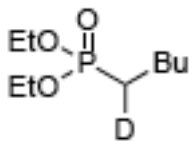
INDEX	FREQUENCY	PPM	HEIGHT
1	5860.453	77.676	150.7
2	5828.496	77.252	155.6
3	5796.539	76.828	152.9
4	4649.251	61.622	42.0
5	4642.917	61.538	42.2
6	2497.761	33.106	38.9
7	2480.775	32.881	39.5
8	2022.436	26.806	45.0
9	1882.228	24.947	43.7
10	1687.894	22.372	47.9
11	1685.879	22.345	46.9
12	1680.697	22.276	42.6
13	1263.239	16.743	46.6
14	1257.193	16.663	48.2
15	1060.844	14.061	53.5
16	15.761	0.209	88.3



3b-H

Scheme 3

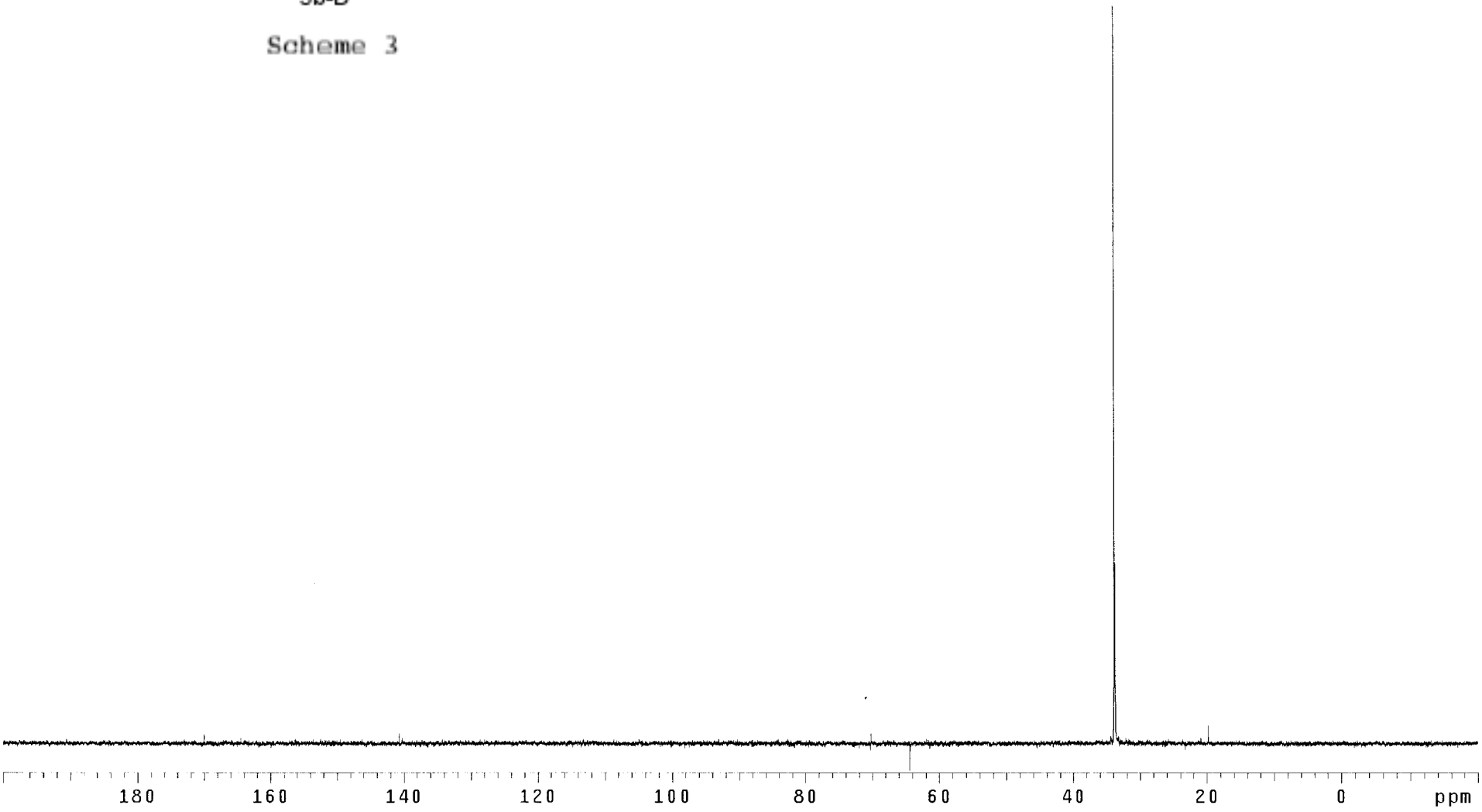
INDEX	FREQUENCY	PPM	HEIGHT
1	4109.663	33.835	126.0



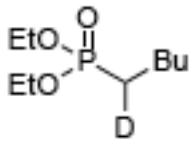
3b-D

Scheme 3

$^{31}P/^{1}H$ decoupled



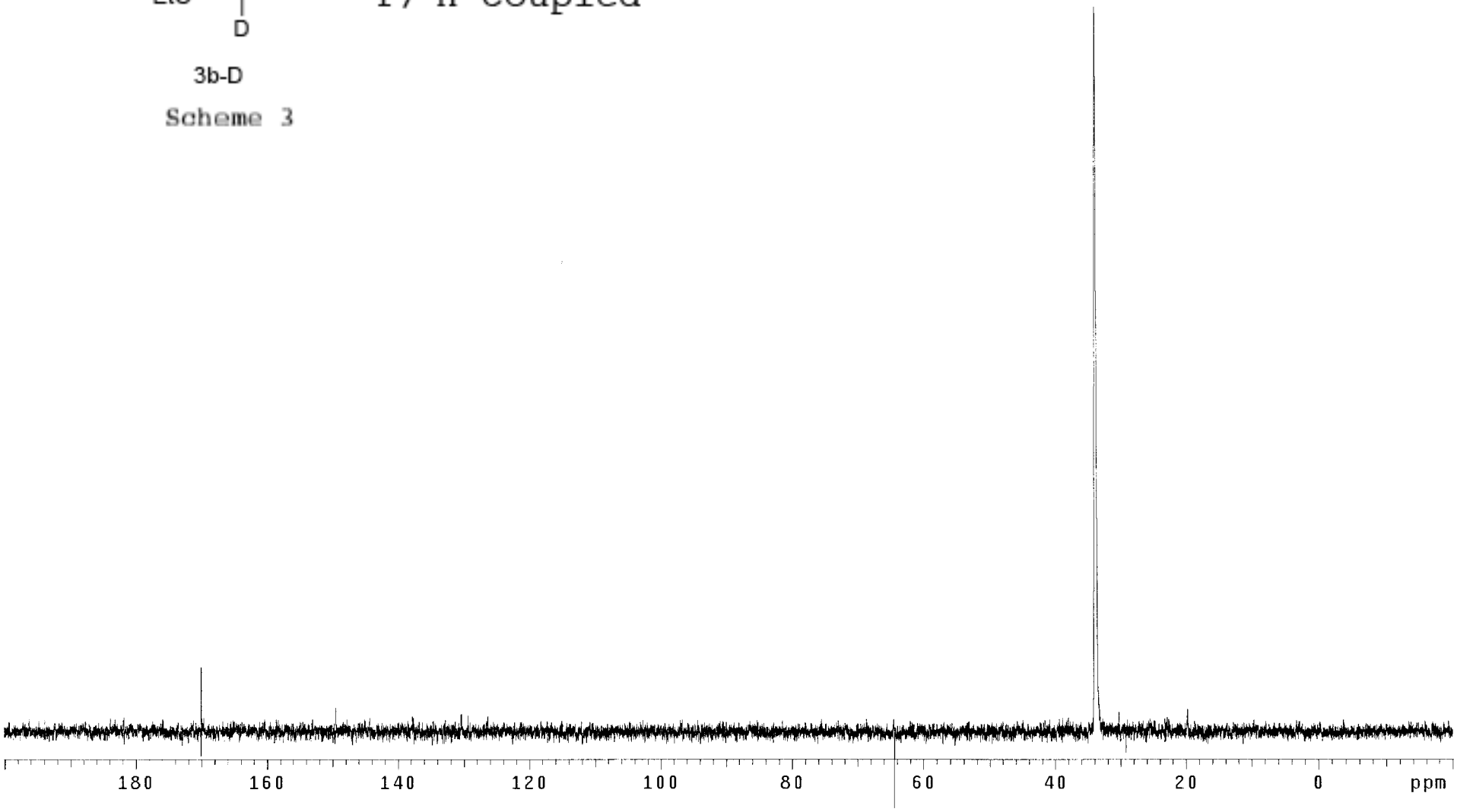
INDEX	FREQUENCY	PPM	HEIGHT
1	4110.071	33.838	126.0
2	4101.911	33.771	122.6

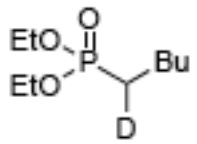


3b-D

Scheme 3

$^{31}P/^{1}H$ coupled

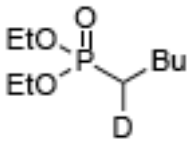




3b-D

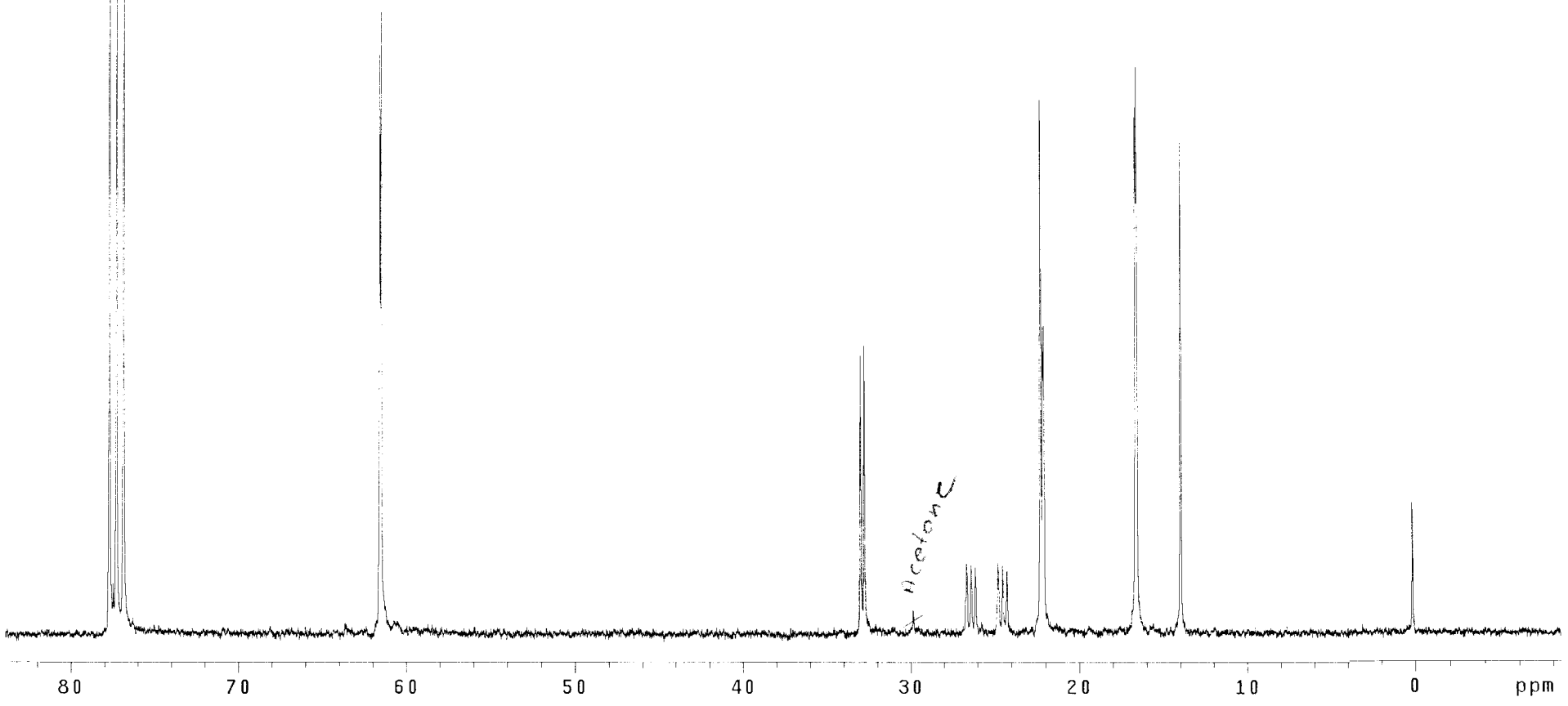
Scheme 3

INDEX	FREQUENCY	PPM	HEIGHT
1	5863.908	77.721	178.6
2	5846.922	77.496	7.8
3	5831.663	77.294	181.8
4	5799.706	76.870	179.5
5	4646.948	61.591	93.4
6	4640.326	61.504	100.2
7	2490.276	33.007	44.6
8	2473.290	32.781	46.2
9	2011.496	26.661	11.2
10	1991.342	26.394	10.8
11	1972.629	26.146	10.4
12	1871.287	24.802	11.3
13	1851.710	24.543	11.1
14	1832.133	24.283	9.8
15	1683.863	22.318	86.0
16	1673.499	22.181	49.3
17	1668.605	22.116	49.4
18	1258.057	16.675	84.8
19	1252.299	16.598	91.4
20	1055.662	13.992	79.9
21	11.155	0.148	20.8



3b-D

Scheme 3



INDEX	FREQUENCY	PPM	HEIGHT
1	4249.195	34.984	126.0

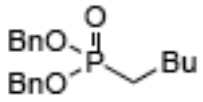
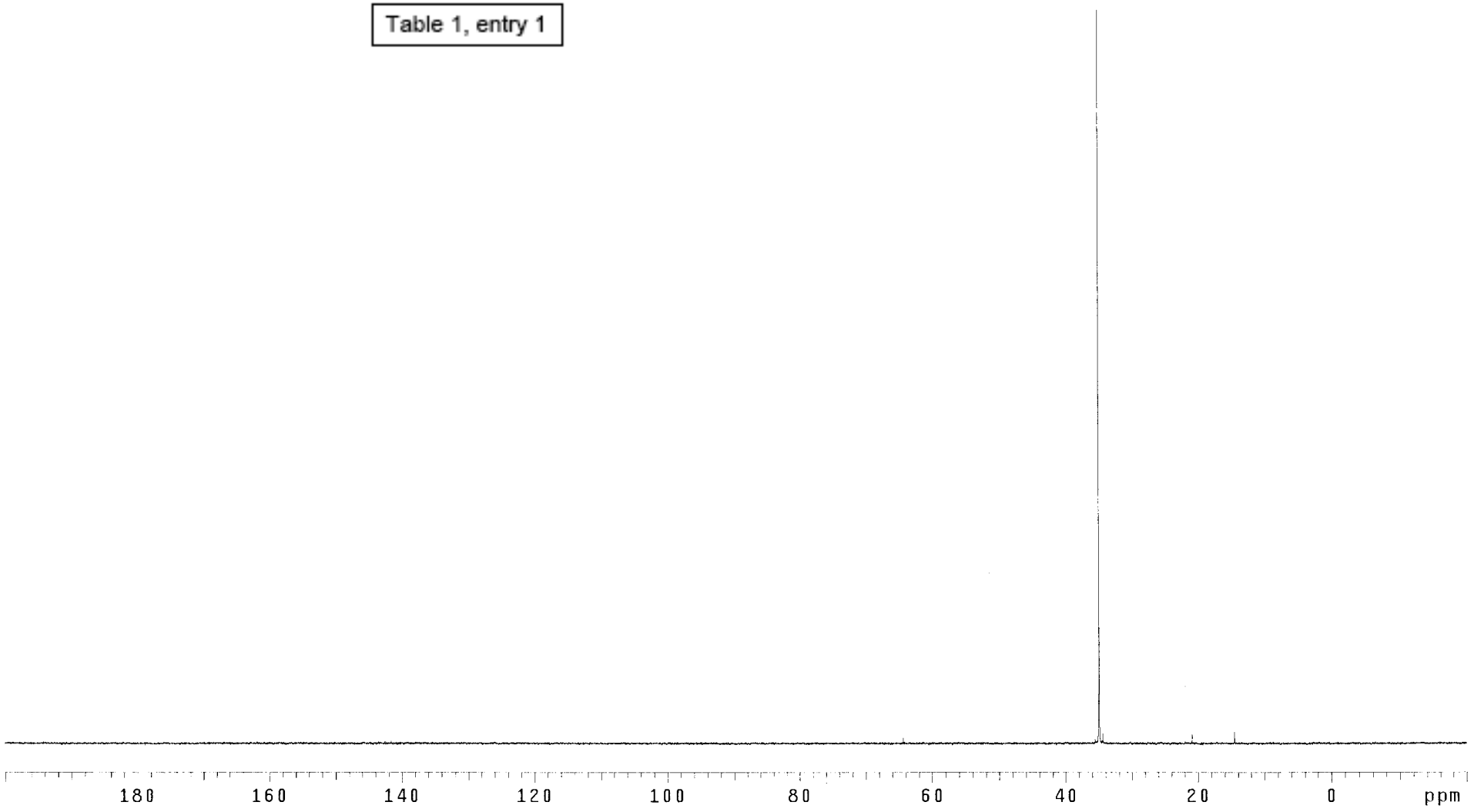
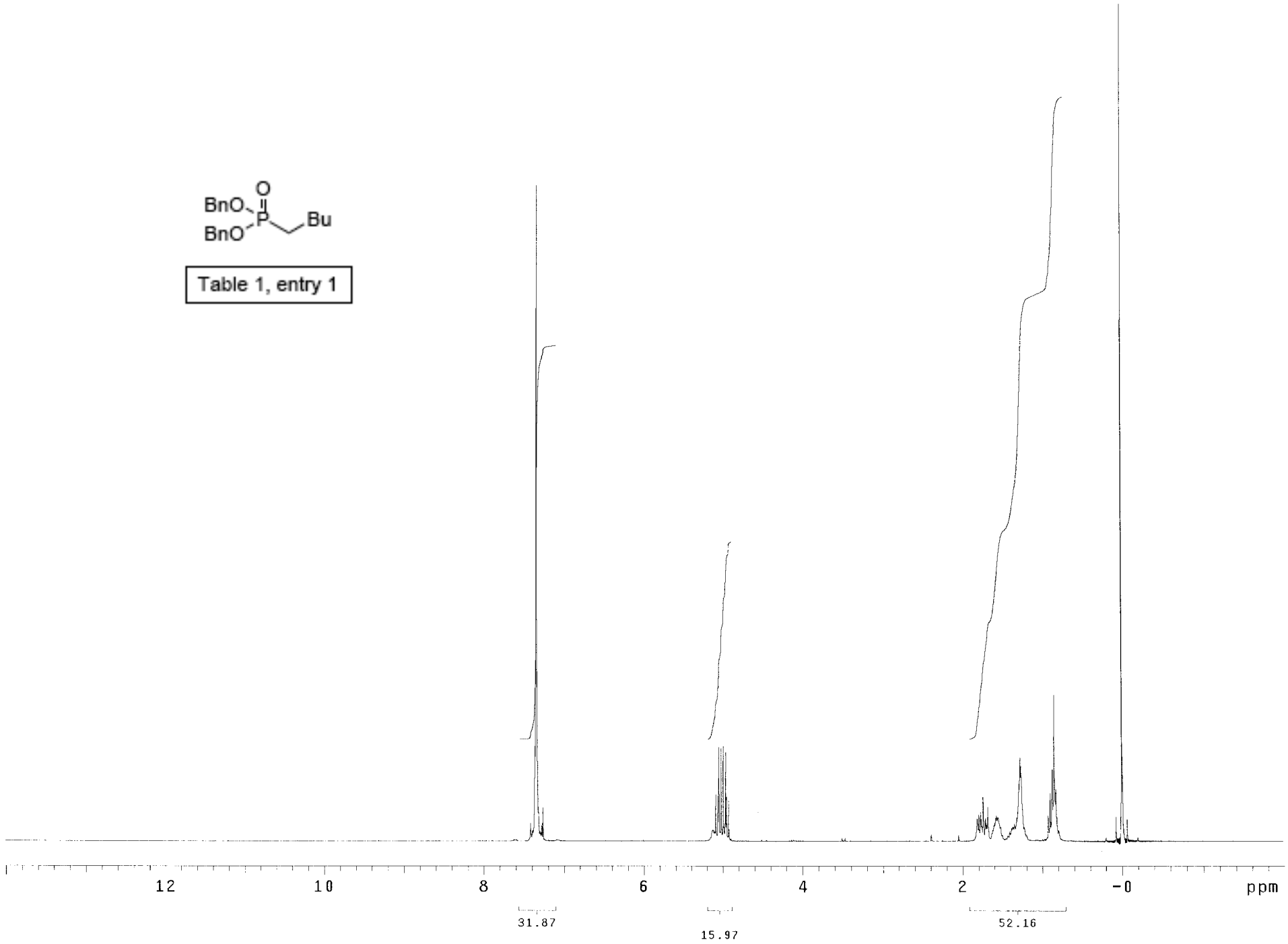


Table 1, entry 1

$^{31}P/^{1}H$ decoupled




BnO
O
P
Bu
BnO
Table 1, entry 1
12
10
8
6
4
2
-0
ppm
31.87
15.97
52.16

INDEX	FREQUENCY	PPM	HEIGHT
1	10295.652	136.460	12.5
2	10289.606	136.380	12.4
3	9699.983	128.565	141.9
4	9684.437	128.359	86.4
5	9648.737	127.886	142.4
6	5850.738	77.547	15.0
7	5818.781	77.123	15.9
8	5786.536	76.696	15.8
9	5064.766	67.129	44.6
10	5058.145	67.042	45.3
11	2469.621	32.733	23.5
12	2452.347	32.504	23.1
13	2023.661	26.822	22.4
14	2013.296	26.685	10.6
15	1884.029	24.971	22.3
16	1663.784	22.052	28.6
17	1654.571	21.930	22.6
18	1649.389	21.861	22.9
19	1056.311	14.001	9.3
20	1040.477	13.791	37.5

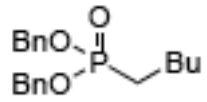


Table 1, entry 1

INDEX	FREQUENCY	PPM	HEIGHT
1	4400.967	36.233	13.6
2	2646.206	21.786	126.0

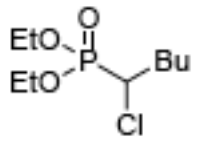
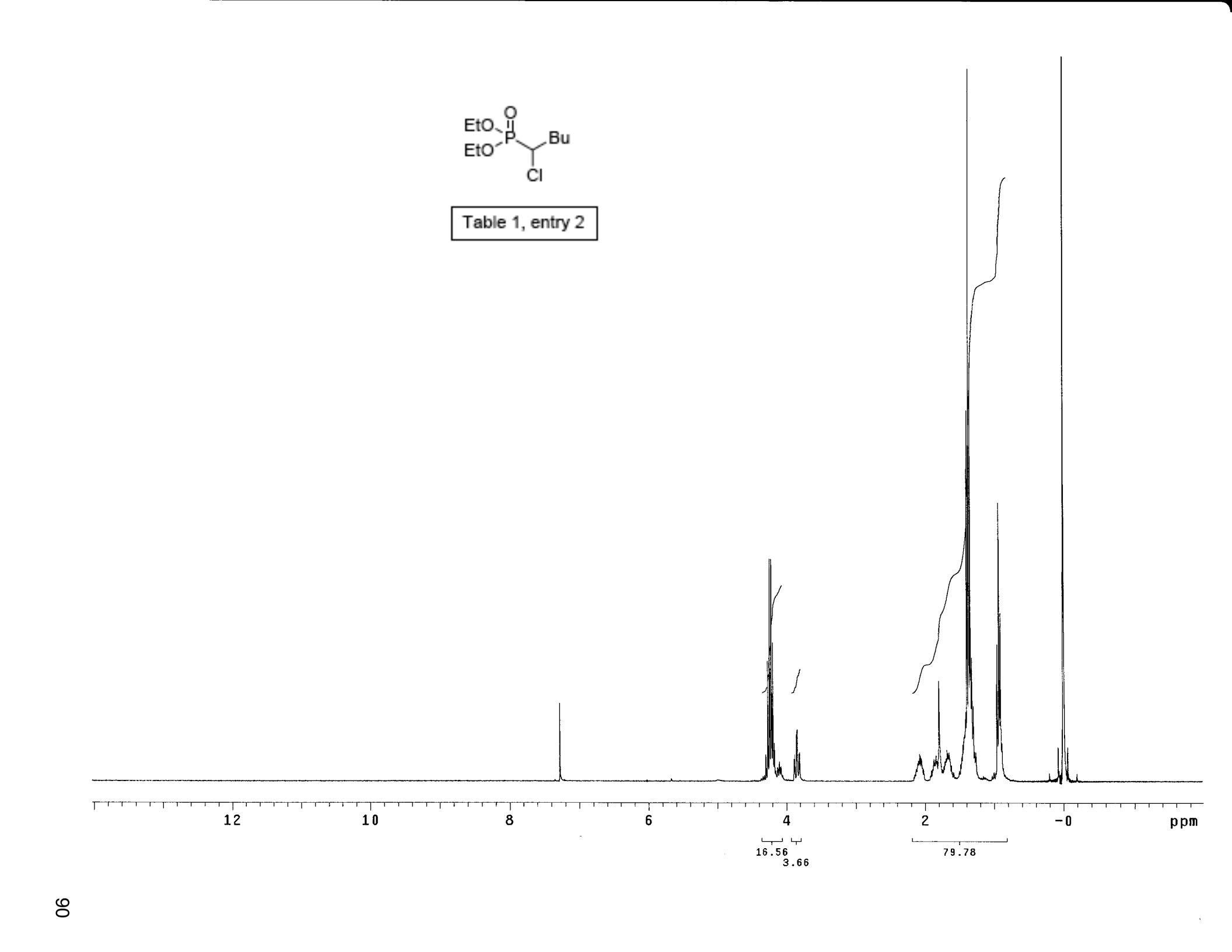


Table 1, entry 2

$^{31}P/^{1}H$ decoupled


EtO
O
P
Bu
EtO
Cl
Table 1, entry 2
12
10
8
6
4
2
-0
ppm
16.56
3.66
79.78

INDEX	FREQUENCY	PPM	HEIGHT
1	5856.208	77.619	27.4
2	5824.251	77.196	28.3
3	5792.006	76.768	27.5
4	4808.822	63.737	34.6
5	4801.912	63.645	34.3
6	4776.577	63.310	33.0
7	4769.667	63.218	33.8
8	4631.762	61.390	7.4
9	4624.565	61.295	7.5
10	4036.957	53.507	46.0
11	3877.172	51.389	45.0
12	2401.100	31.825	72.6
13	2254.270	29.879	11.0
14	2245.057	29.756	12.1
15	2162.717	28.665	51.2
16	2150.338	28.501	51.8
17	2108.592	27.948	11.2
18	2105.137	27.902	10.2
19	1720.788	22.808	19.1
20	1649.677	21.865	84.9
21	1247.478	16.534	44.1
22	1244.311	16.492	55.2
23	1242.008	16.462	52.9
24	1238.841	16.420	44.7
25	1053.432	13.962	19.7
26	1044.219	13.840	87.7

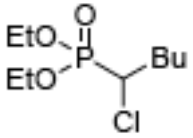
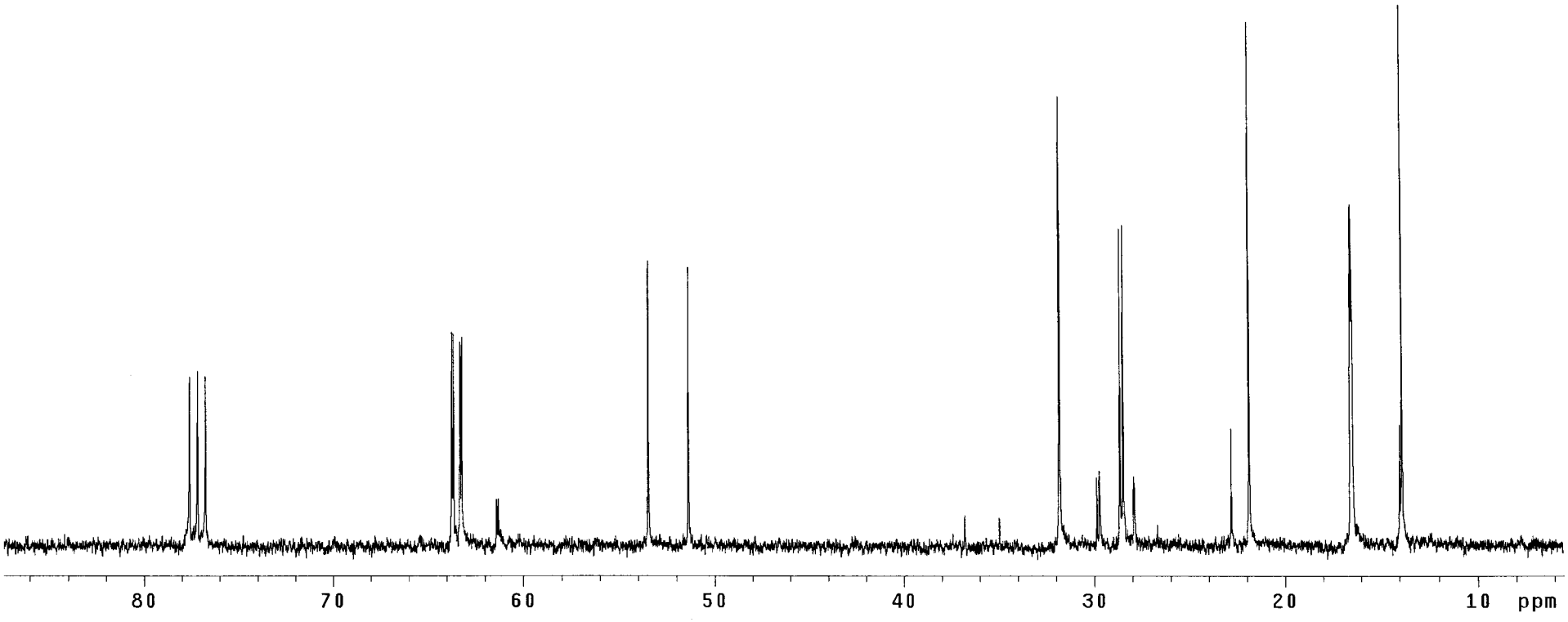
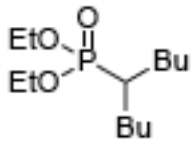


Table 1, entry 2

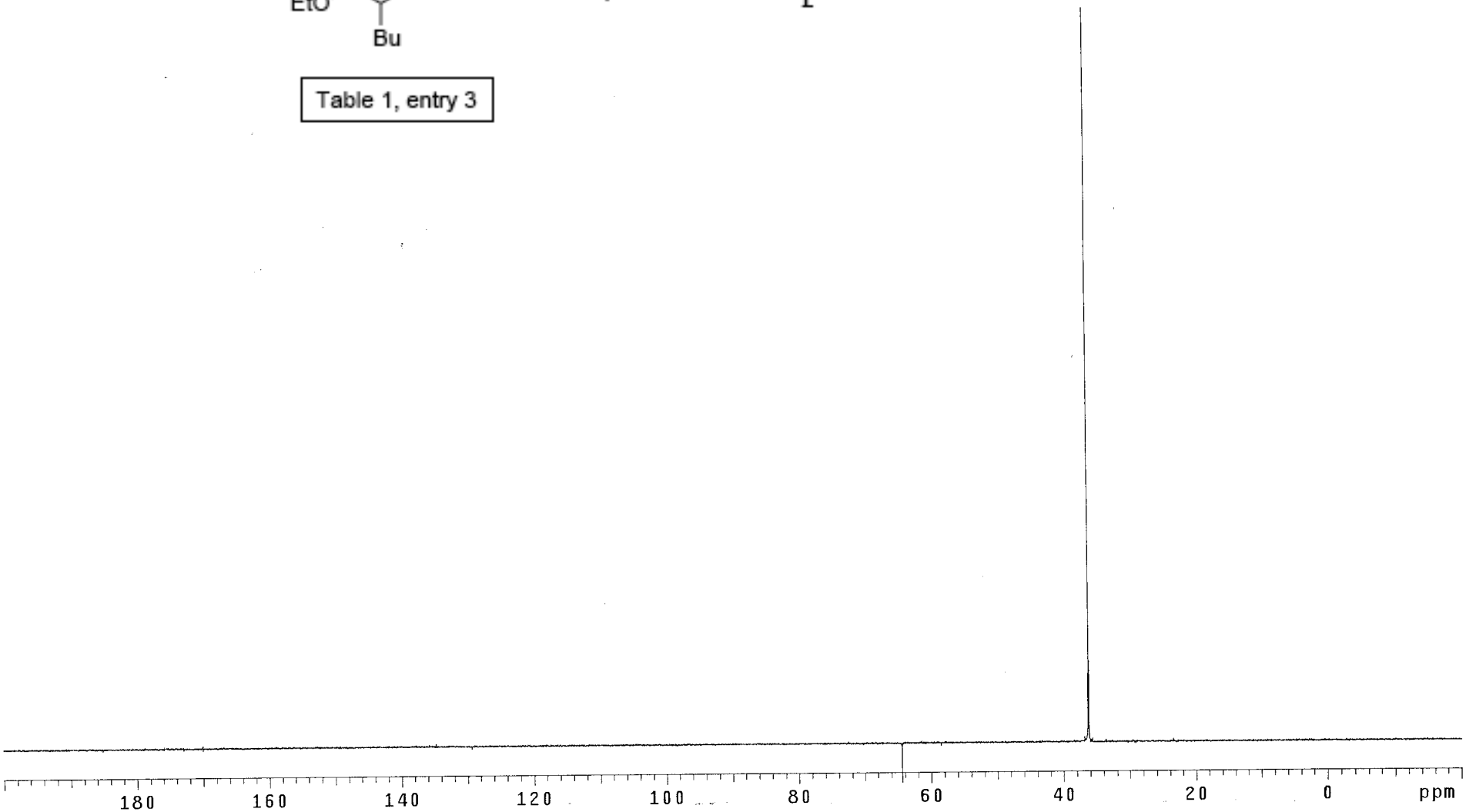


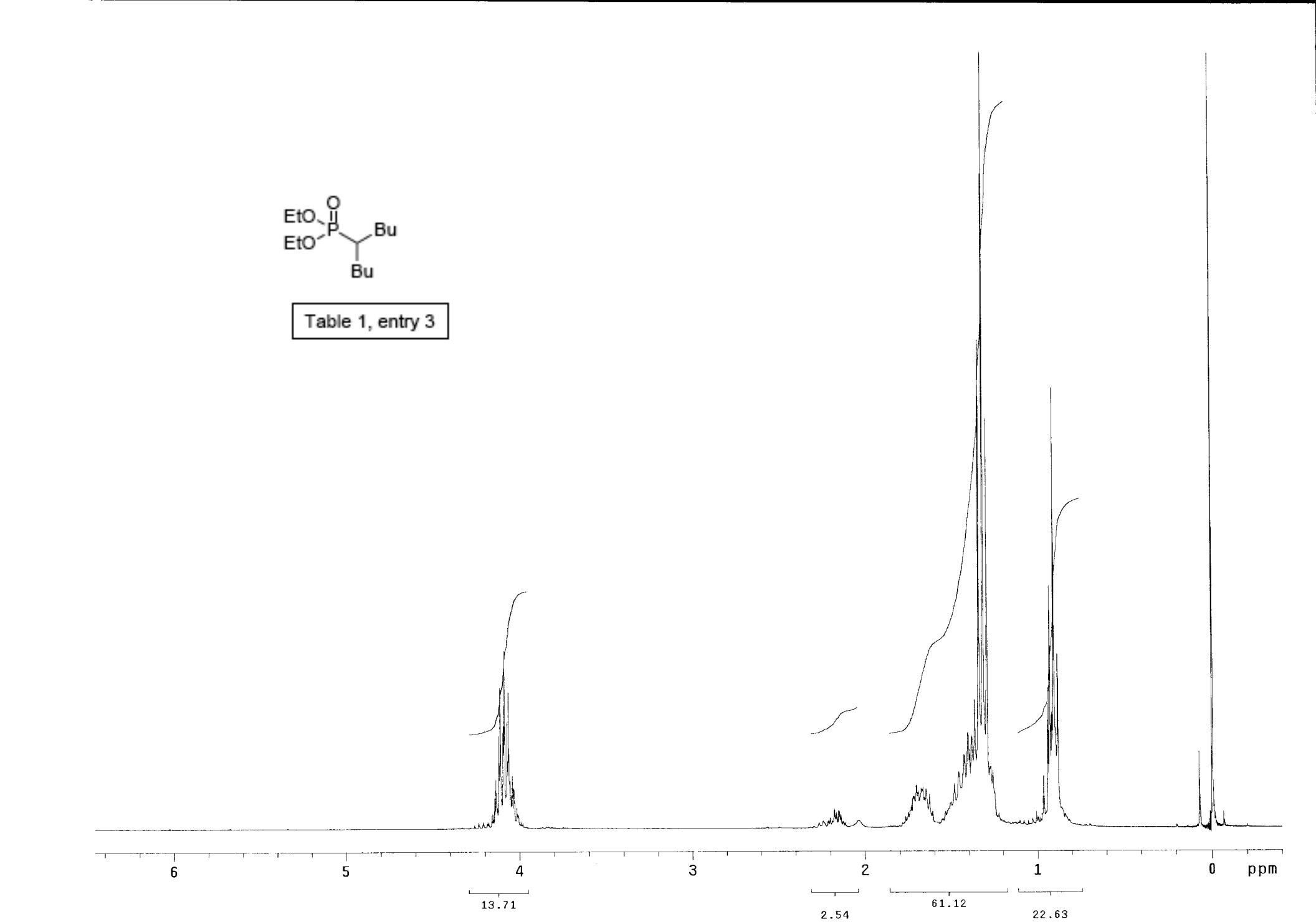
INDEX	FREQUENCY	PPM	HEIGHT
1	4403.415	36.253	126.0



$^{31}P/^{1}H$ decoupled

Table 1, entry 3




EtO
EtO
O
P
Bu
Bu
Table 1, entry 3
6
5
4
3
2
1
0
ppm
13.71
2.54
61.12
22.63

INDEX	FREQUENCY	PPM	HEIGHT
1	5848.435	77.516	39.9
2	5816.190	77.089	41.0
3	5784.233	76.665	41.1
4	4628.595	61.348	35.7
5	4621.686	61.257	32.3
6	2781.706	36.869	24.4
7	2643.801	35.041	23.8
8	2255.998	29.901	52.4
9	2246.785	29.779	53.1
10	2111.759	27.990	53.0
11	2108.016	27.940	53.4
12	1721.076	22.811	90.5
13	1250.069	16.569	36.0
14	1244.311	16.492	38.3
15	1052.569	13.951	84.3

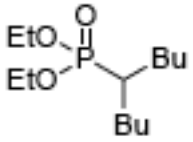
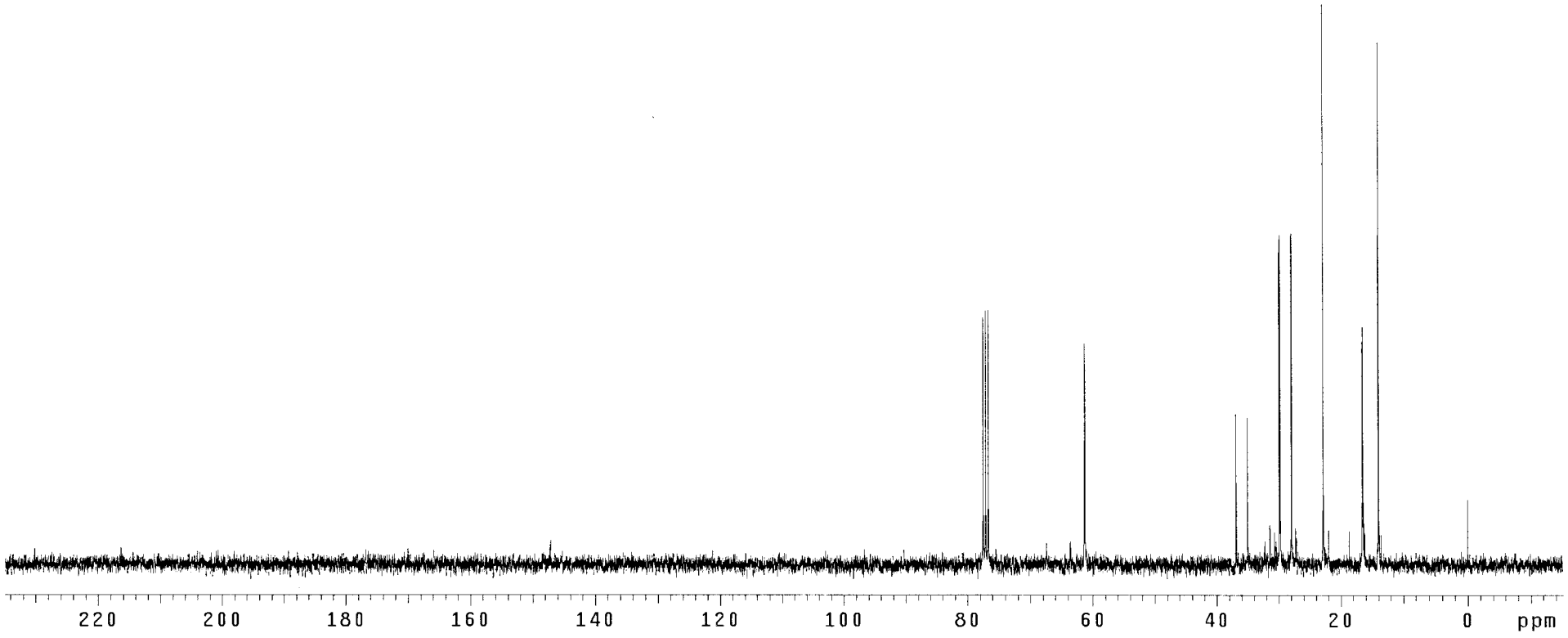
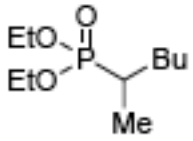


Table 1, entry 3

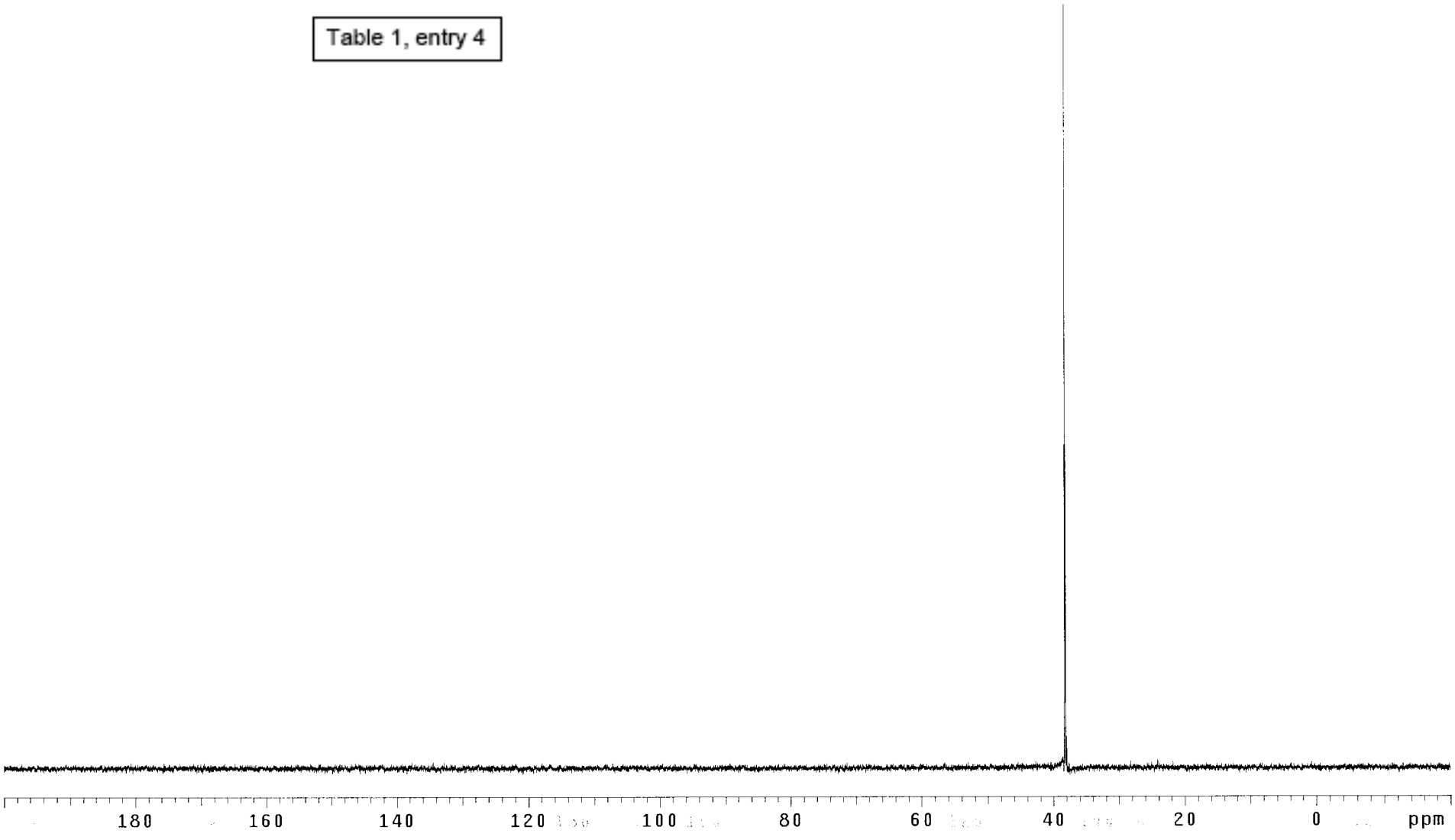


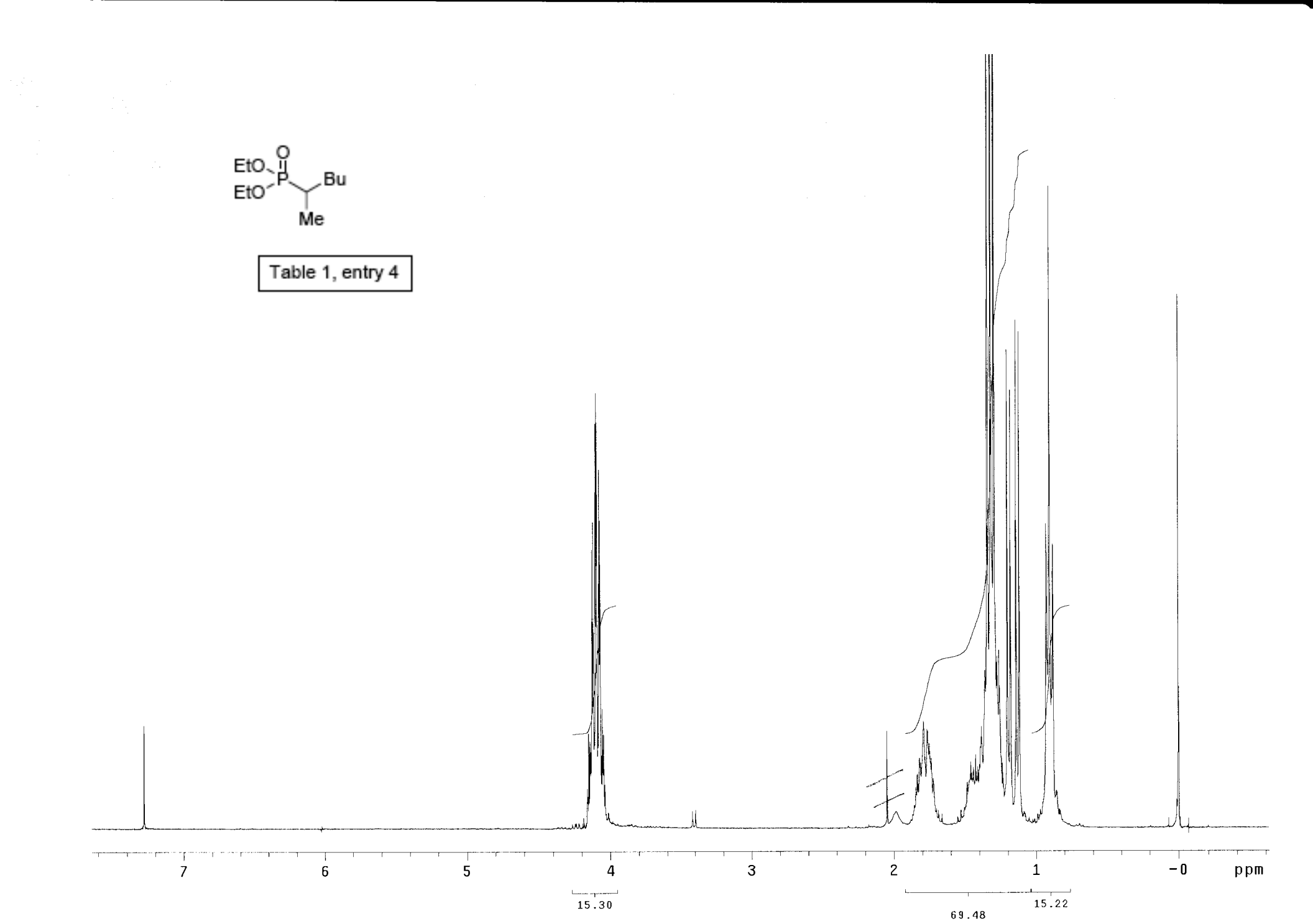
INDEX	FREQUENCY	PPM	HEIGHT
1	4640.048	38.201	132.1
2	4619.241	38.030	5.3



$^{31}P/^{1}H$ decoupled

Table 1, entry 4




EtO
EtO
O
P
Bu
Me
Table 1, entry 4
7
6
5
4
3
2
1
-0
ppm
15.30
69.48
15.22

INDEX	FREQUENCY	PPM	HEIGHT
1	5860.741	77.679	141.2
2	5828.784	77.256	146.4
3	5796.827	76.832	144.4
4	4652.706	61.668	68.4
5	4646.084	61.580	71.8
6	2403.330	31.854	80.7
7	2262.834	29.992	79.6
8	2250.166	29.824	168.4
9	2246.423	29.775	102.8
10	2236.347	29.641	88.4
11	1714.669	22.727	130.8
12	1264.966	16.766	106.3
13	1259.208	16.690	111.7
14	1068.330	14.160	125.0
15	1007.007	13.347	77.0
16	1001.824	13.278	82.4

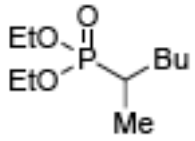
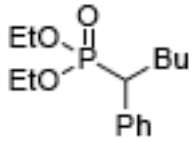


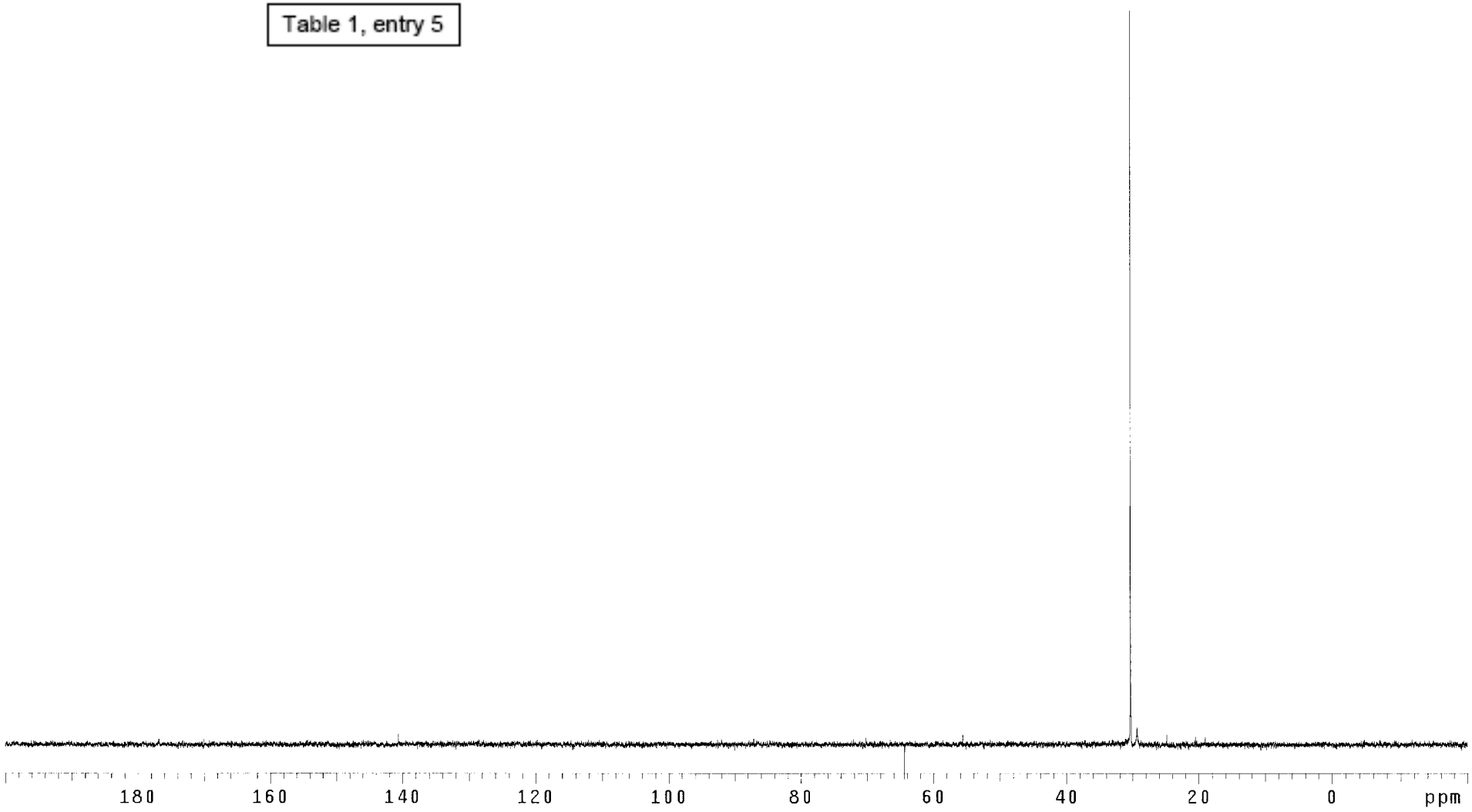
Table 1, entry 4

INDEX	FREQUENCY	PPM	HEIGHT
1	7819.507	64.378	-6.0
2	3680.458	30.301	126.0



$^{31}P/^{1}H$ decoupled

Table 1, entry 5



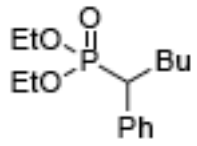


Table 1, entry 5

INDEX	FREQUENCY	PPM	HEIGHT
1	10285.575	136.327	14.8
2	10278.954	136.239	17.2
3	9754.397	129.287	78.0
4	9747.487	129.195	83.1
5	9688.755	128.416	126.0
6	9583.383	127.020	52.6
7	9581.368	126.993	53.8
8	9529.834	126.310	10.0
9	9519.181	126.169	11.5
10	9430.507	124.994	9.9
11	7099.080	94.092	7.1
12	5851.602	77.558	25.5
13	5819.069	77.127	26.9
14	5787.400	76.707	29.3
15	4715.830	62.504	33.9
16	4708.920	62.413	36.6
17	4656.234	61.715	28.9
18	4649.036	61.619	31.6
19	3435.531	45.535	38.7
20	3298.777	43.723	38.7
21	2494.668	33.065	7.3
22	2252.543	29.856	37.7
23	2237.284	29.653	41.7
24	2213.964	29.344	57.4
25	2153.217	28.539	9.4
26	1814.068	24.044	8.8
27	1680.194	22.270	71.4
28	1240.568	16.443	32.5
29	1234.235	16.359	36.5
30	1227.037	16.263	37.9
31	1221.279	16.187	34.3
32	1056.599	14.004	9.1
33	1040.477	13.791	66.3
34	0.000	0.000	10.0

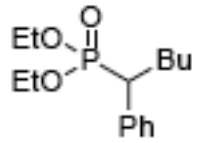
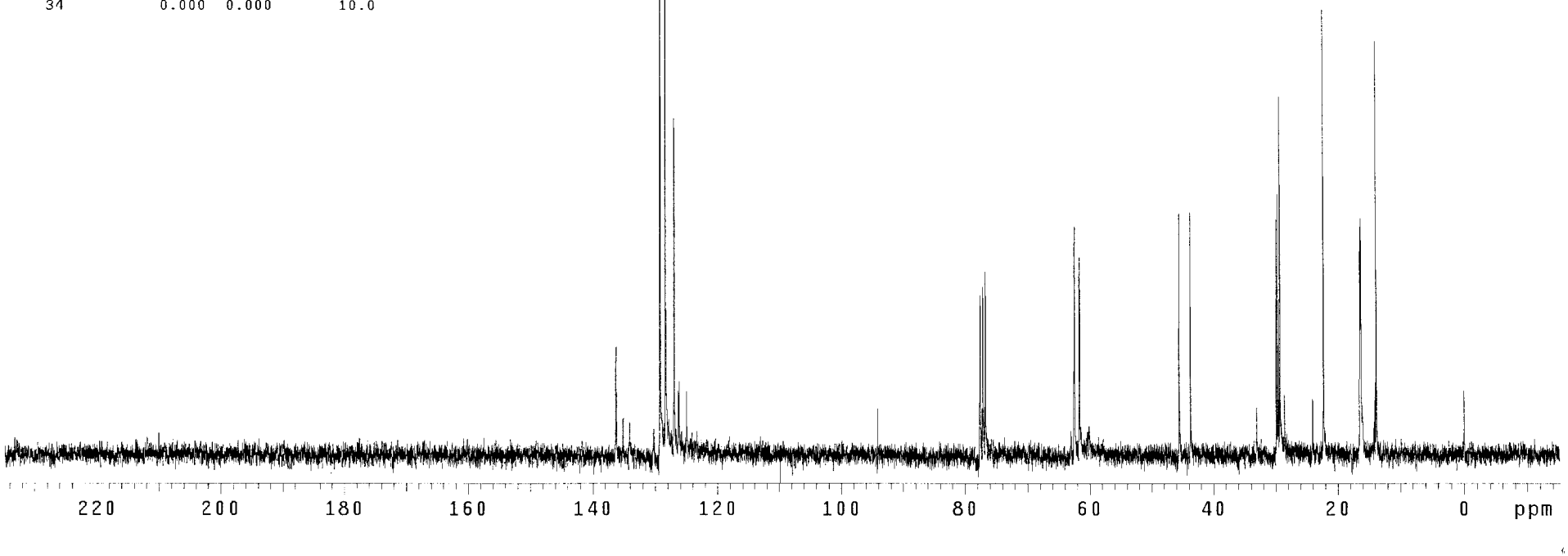


Table 1, entry 5

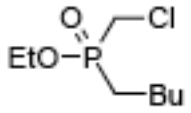
```
std

exp1  s2pul

      SAMPLE                SPECIAL
date   Jun 27 2007   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             1000     PROCESSING
ct              320  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          1149.0
pw            9.250  rfp                0
    DECOUPLER       rp             82.8
dn               H1  lp           -266.9
dof               0        PLOT
dm              yyy  wc              250
dmm               w  sc                0
dpwr             35  vs              121
dmf            6700  th                6
                     ai   no   ph
```

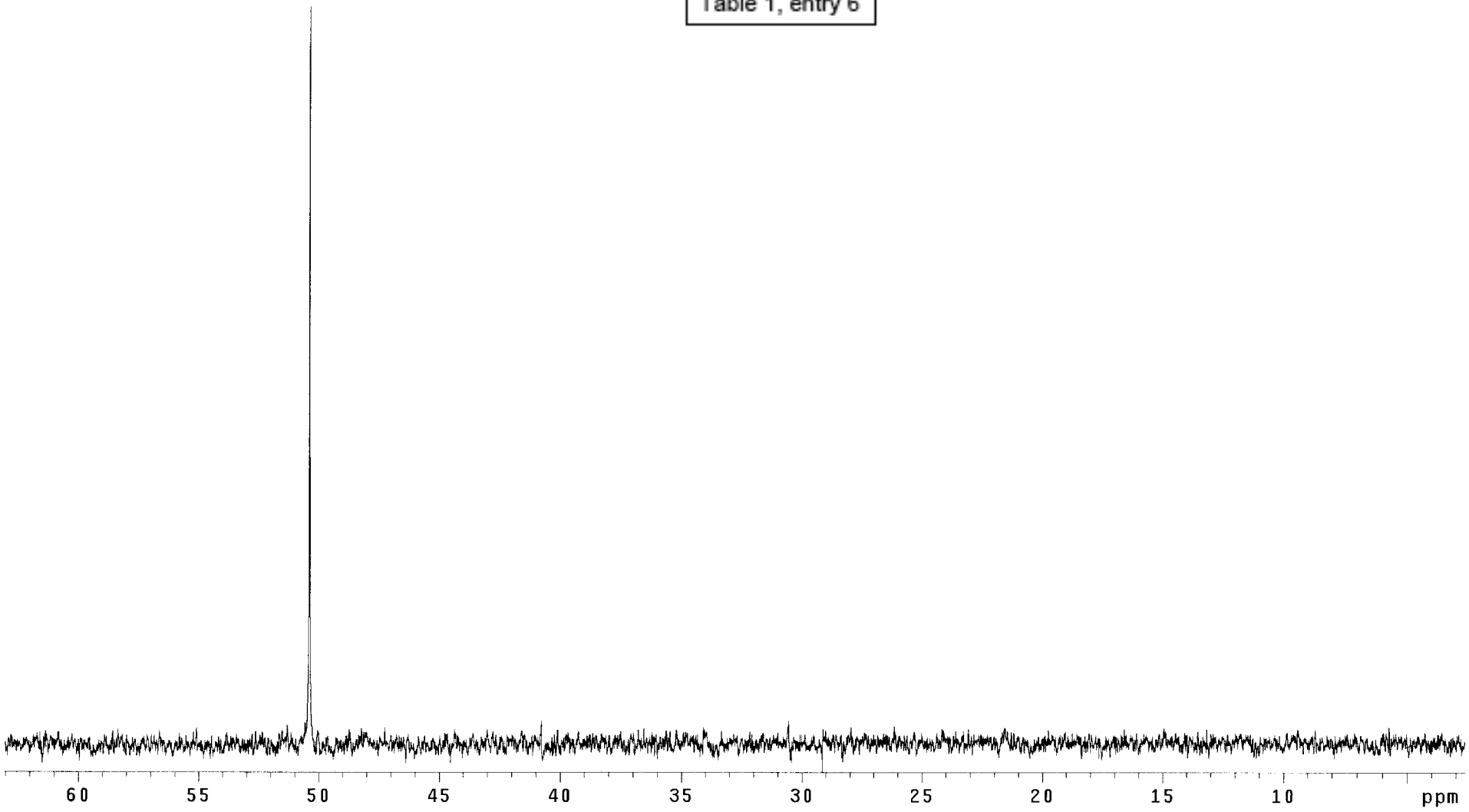


INDEX	FREQUENCY	PPM	HEIGHT
1	6121.456	50.398	126.8

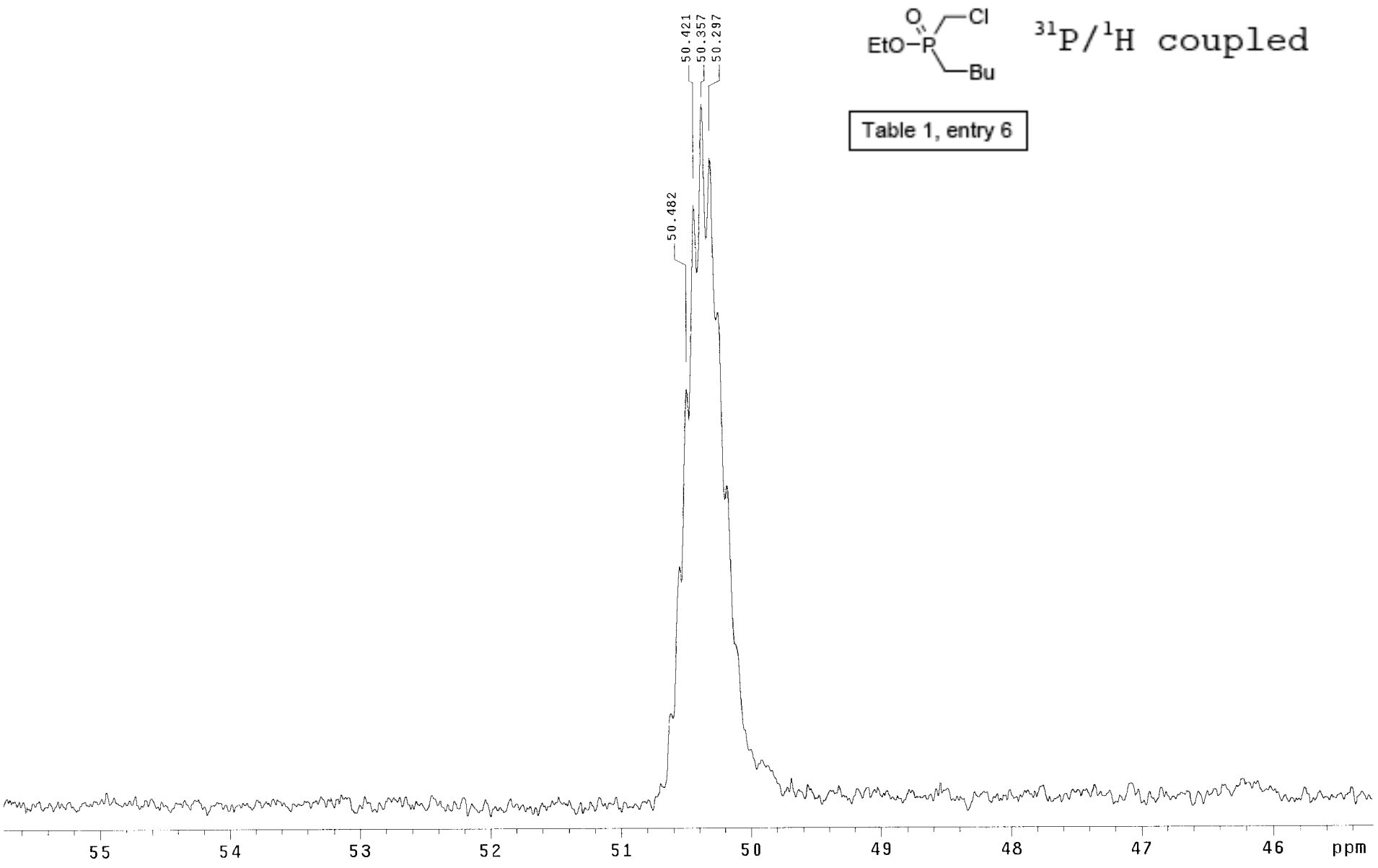


$^{31}P/^{1}H$ decoupled

Table 1, entry 6



INDEX	FREQUENCY	PPM	HEIGHT
1	6131.656	50.482	75.5
2	6124.312	50.421	109.3
3	6116.561	50.357	127.8
4	6109.217	50.297	117.9



$^{31}P/^{1}H$ coupled

Table 1, entry 6

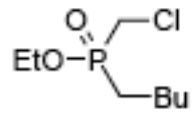


Table 1, entry 6

INDEX	FREQUENCY	PPM	HEIGHT
1	5864.772	77.733	67.6
2	5832.527	77.305	70.9
3	5800.570	76.882	68.9
4	4638.023	61.473	50.9
5	4631.113	61.382	50.1
6	2662.729	35.292	66.1
7	2572.040	34.090	64.3
8	2498.337	33.113	60.1
9	2482.791	32.907	58.0
10	1985.296	26.313	56.4
11	1885.395	24.989	57.7
12	1682.712	22.303	91.3
13	1590.007	21.074	70.2
14	1585.977	21.021	75.6
15	1264.966	16.766	54.9
16	1259.208	16.690	47.9
17	1055.662	13.992	93.7

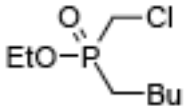
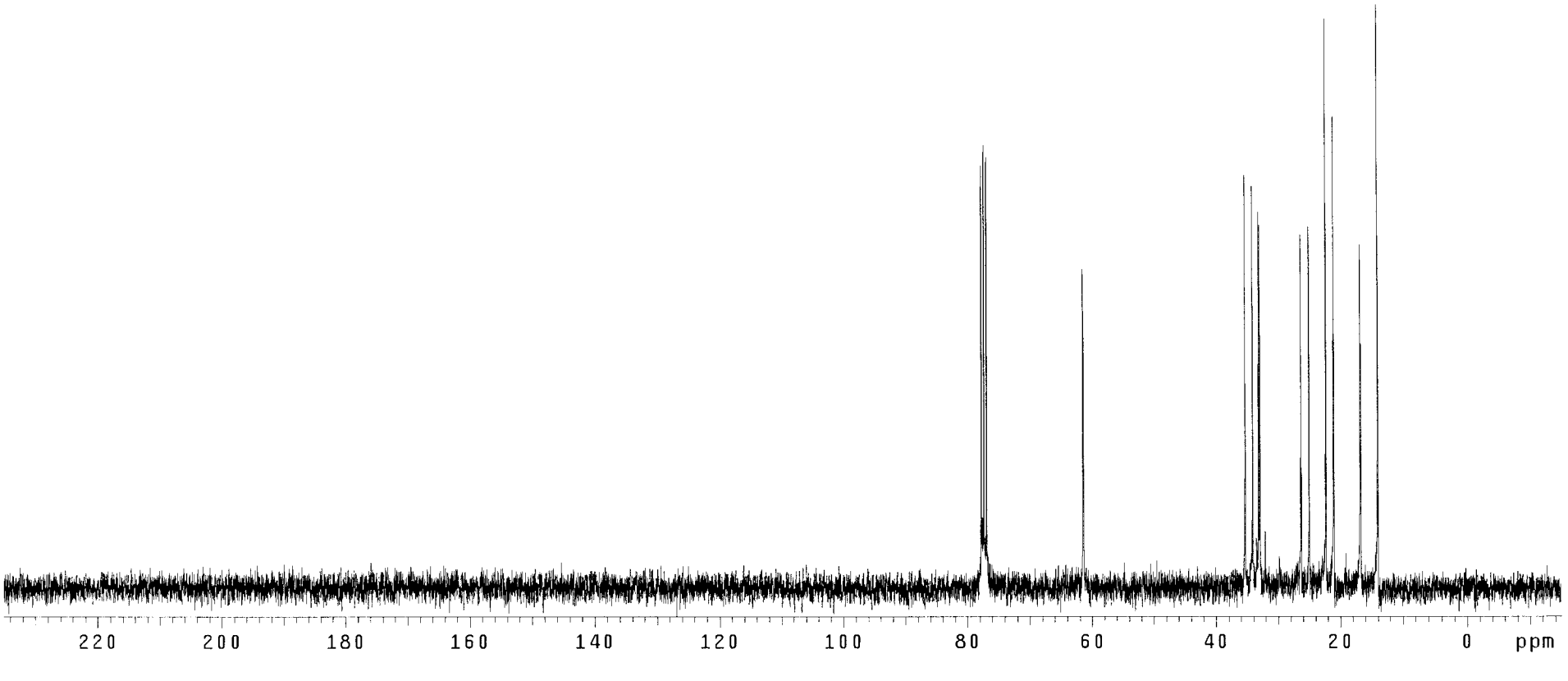


Table 1, entry 6



INDEX	FREQUENCY	PPM	HEIGHT
1	12261.691	100.950	126.0

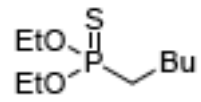


Table 1, entry 7

INDEX	FREQUENCY	PPM	HEIGHT
1	12298.410	101.253	11.9
2	12288.210	101.169	27.2
3	12277.195	101.078	43.6
4	12272.299	101.038	55.2
5	12261.691	100.950	68.1
6	12256.795	100.910	48.7
7	12251.491	100.866	53.5
8	12246.187	100.823	40.3
9	12235.988	100.739	24.4
10	12225.380	100.651	10.8

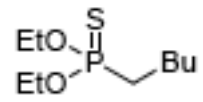
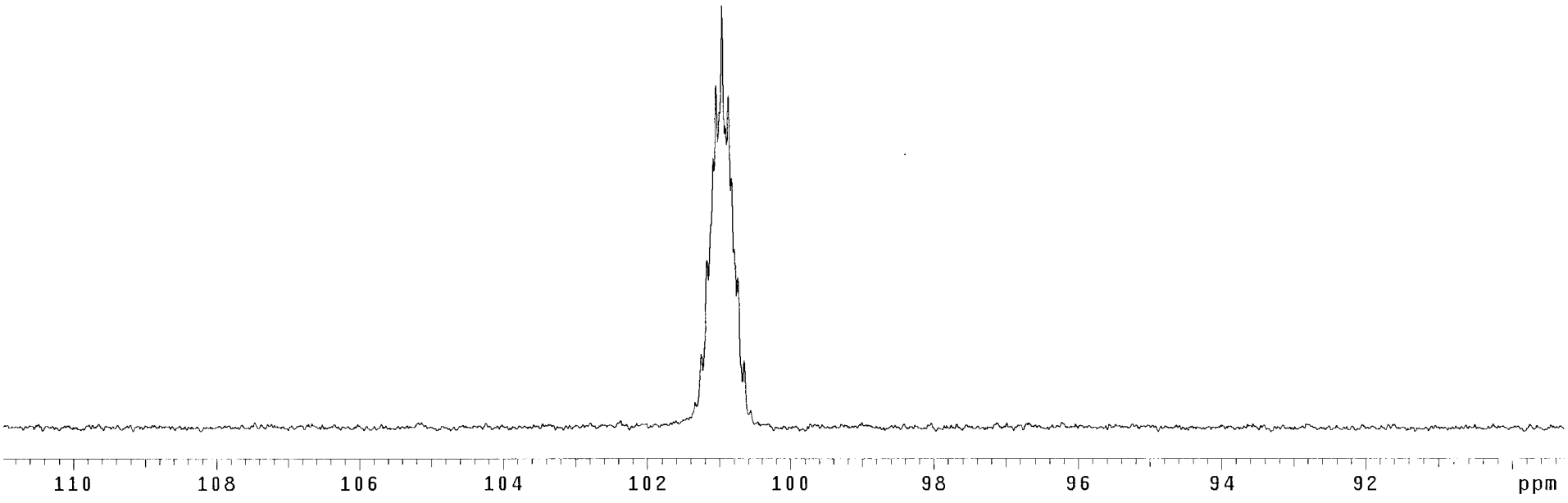
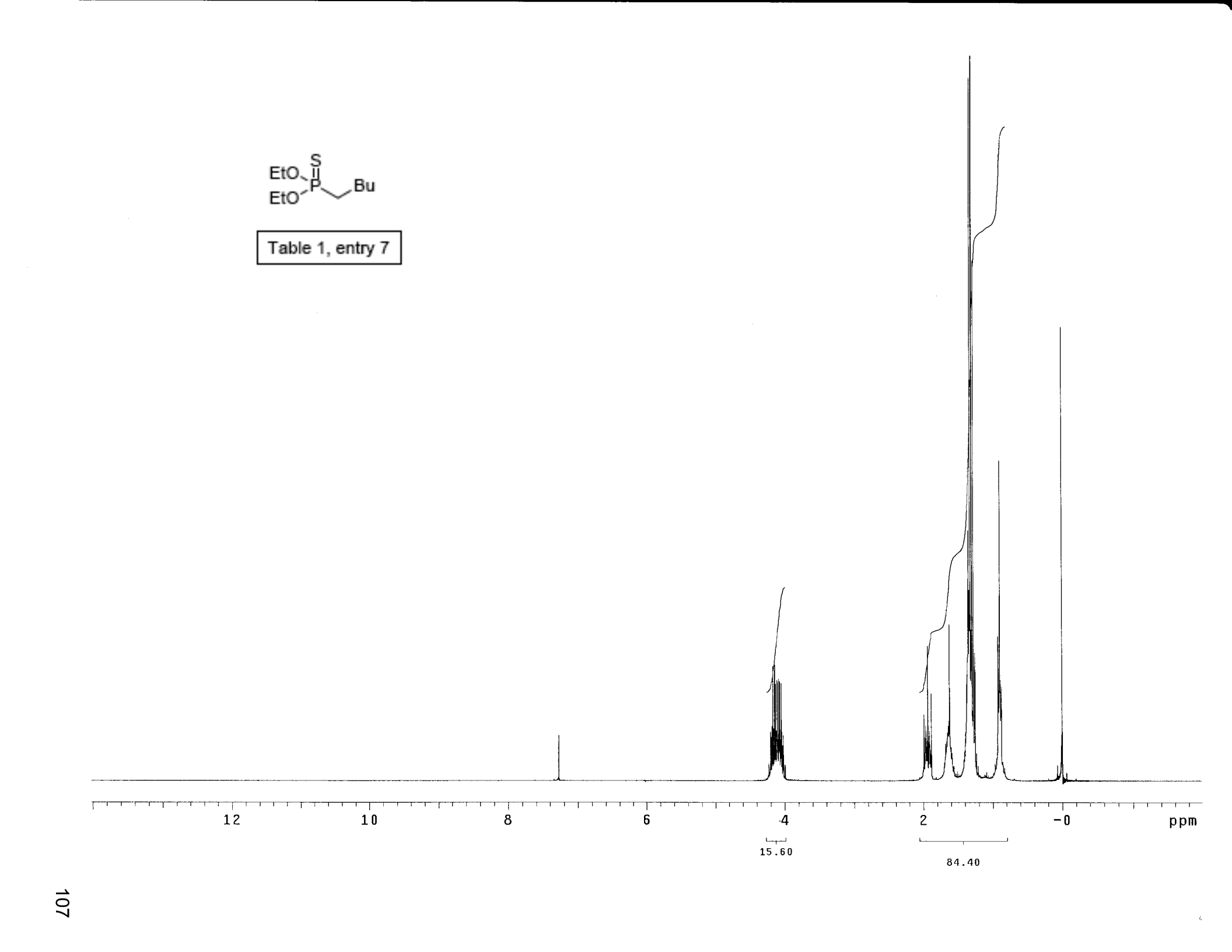


Table 1, entry 7




EtO
EtO
S
P
Bu
Table 1, entry 7
12
10
8
6
4
2
0
ppm
15.60
84.40

INDEX	FREQUENCY	PPM	HEIGHT
1	5861.605	77.691	46.9
2	5829.648	77.267	47.8
3	5797.691	76.844	46.8
4	4716.620	62.515	45.0
5	4709.710	62.423	45.0
6	2678.564	35.502	41.6
7	2567.434	34.029	41.4
8	2472.714	32.774	40.1
9	2454.288	32.530	41.6
10	2257.651	29.923	16.1
11	1713.229	22.707	40.3
12	1709.199	22.654	41.5
13	1691.061	22.414	46.4
14	1690.197	22.402	46.9
15	1244.525	16.495	48.1
16	1237.616	16.404	52.9
17	1062.572	14.084	63.8

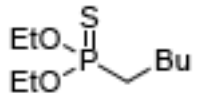
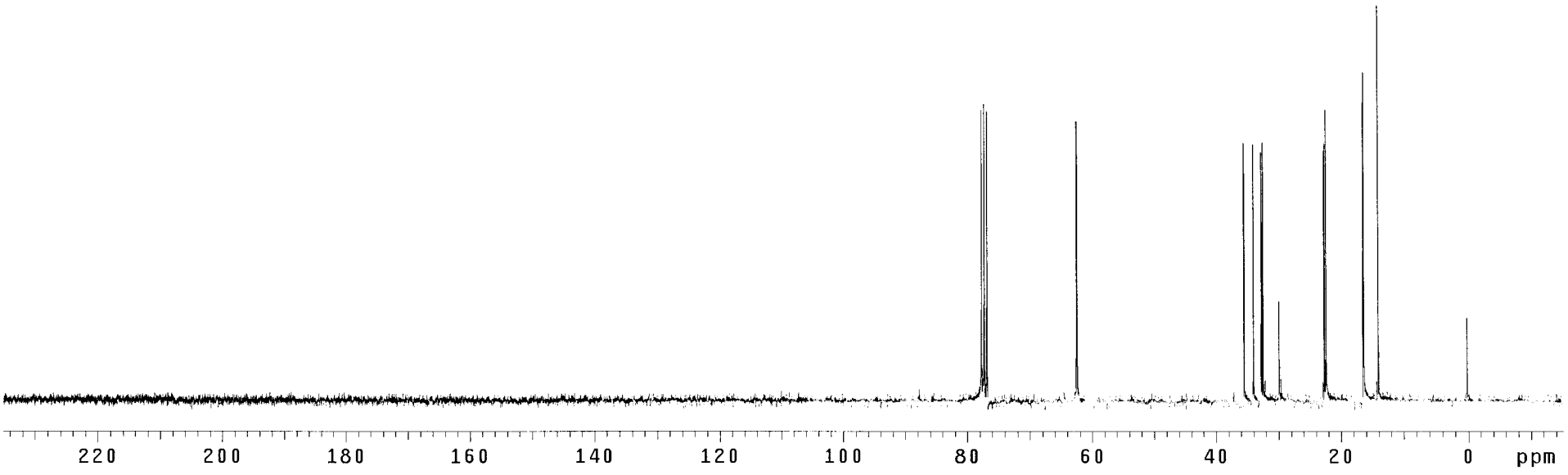
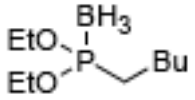


Table 1, entry 7

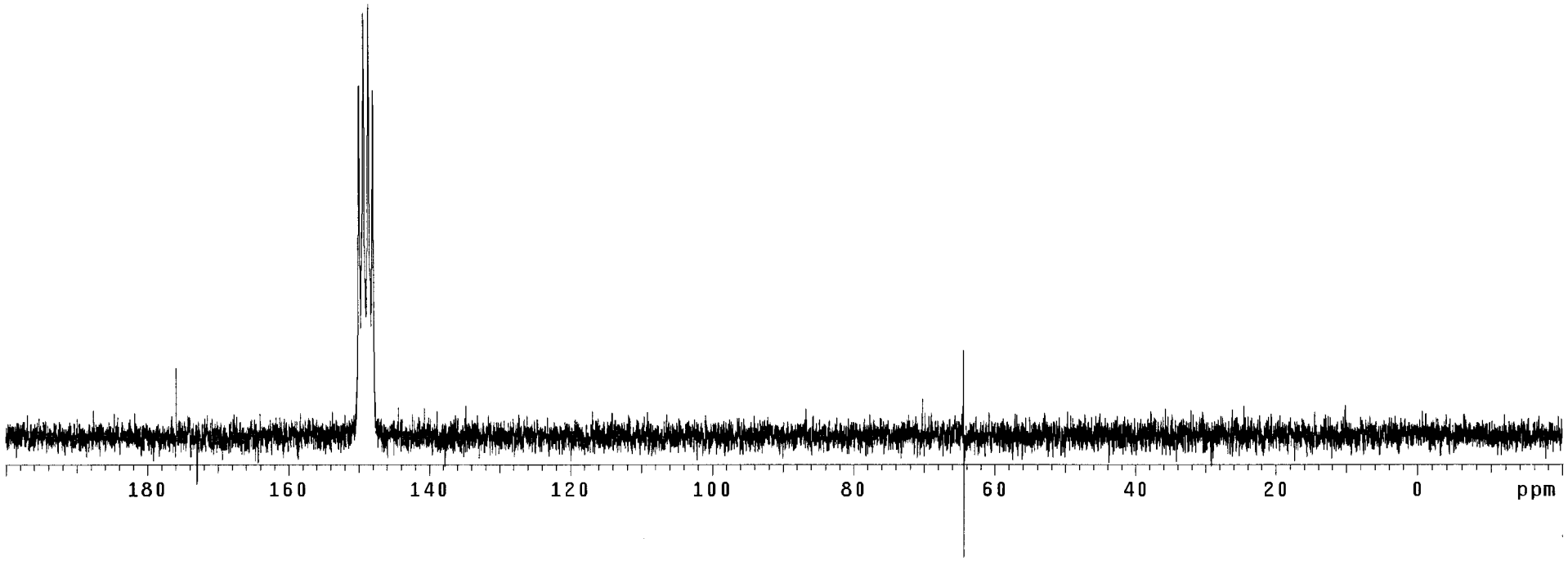


INDEX	FREQUENCY	PPM	HEIGHT
1	18215.067	149.964	56.4
2	18137.549	149.326	68.2
3	18052.687	148.627	69.6
4	17974.761	147.986	55.7
5	7821.139	64.391	13.6
6	7818.691	64.371	-21.2

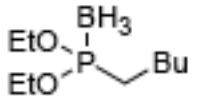


$^{31}P/^{1}H$ decoupled

Table 1, entry 8

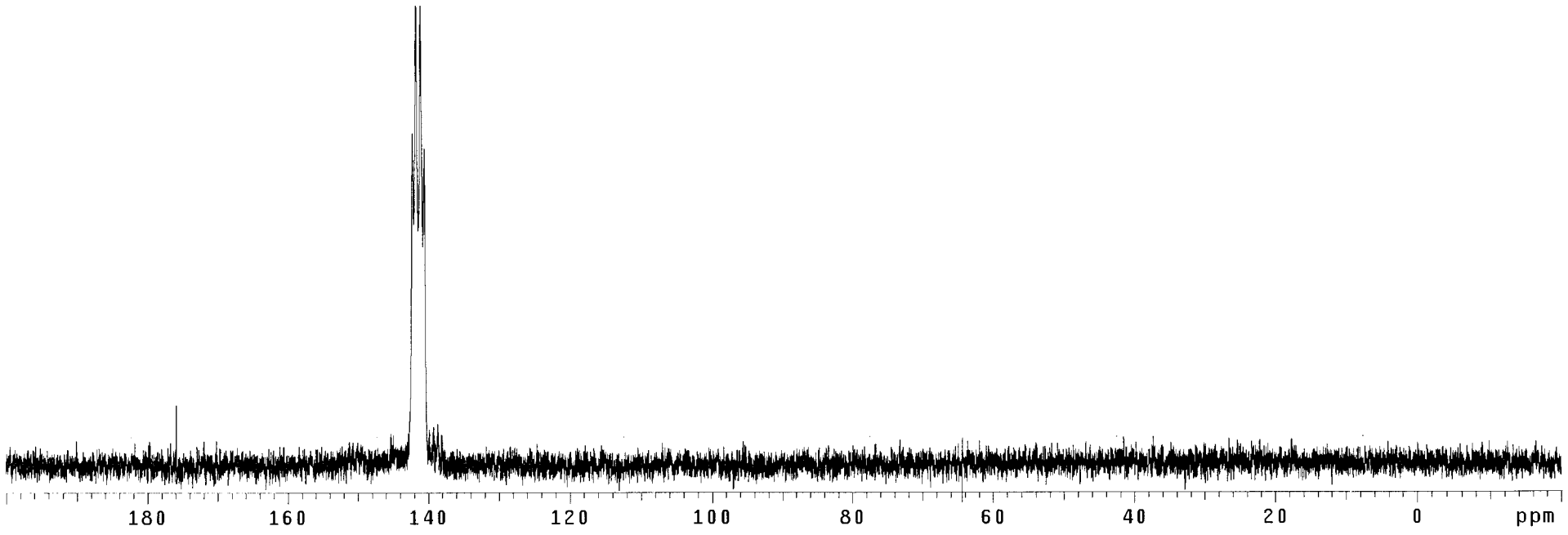


INDEX	FREQUENCY	PPM	HEIGHT
1	17273.836	142.215	53.1
2	17211.006	141.698	73.9
3	17134.304	141.066	73.9
4	17071.474	140.549	50.8



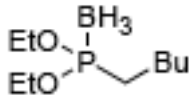
$^{31}P/^{1}H$ coupled

Table 1, entry 8



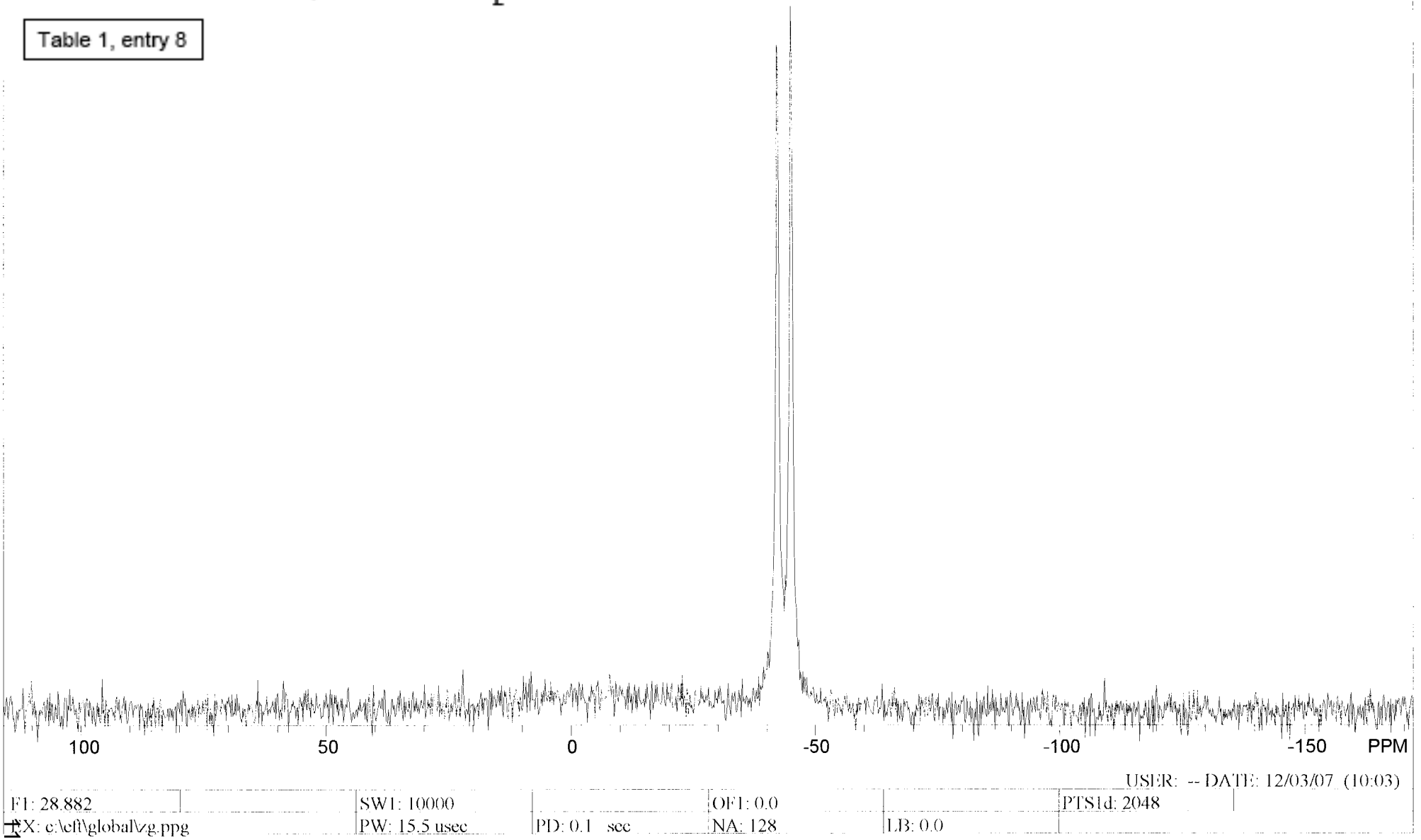
Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	1273	19972	90.70	-1218.60	-42.193
2	1290	22082	100.28	-1300.26	-45.020



$^{11}B/^{1}H$ decoupled

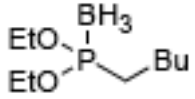
Table 1, entry 8



F1

Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	1245	2978	23.49	-1080.60	-37.415
2	1264	10059	79.34	-1172.82	-40.607
3	1283	11927	94.08	-1267.29	-43.879
4	1300	9931	78.33	-1349.33	-46.719
5	1320	3336	26.31	-1446.26	-50.075



$^{11}B/^{1}H$ coupled

Table 1, entry 8

USER: -- DATE: 12/03/07 (10:07)

F1: 28.882 | SW1: 10000 | OF1: 0.0 | PTS1d: 2048

EX: c:\cft\global\zg.ppg | PW: 15.5 usec | PD: 0.1 sec | NA: 128 | LB: 0.0

H2

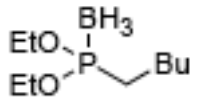


Table 1, entry 8

INDEX	FREQUENCY	PPM	HEIGHT
1	5859.302	77.660	35.5
2	5827.344	77.237	36.1
3	5795.099	76.809	35.9
4	4769.306	63.213	43.2
5	4764.699	63.152	45.6
6	2498.913	33.121	42.4
7	2484.806	32.934	42.5
8	2288.169	30.328	22.0
9	2232.316	29.588	22.4
10	1684.439	22.326	60.0
11	1615.343	21.410	81.1
12	1266.694	16.789	55.3
13	1261.224	16.716	59.4
14	1057.101	14.011	65.3
15	12.306	0.163	22.1

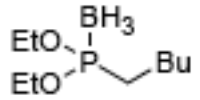
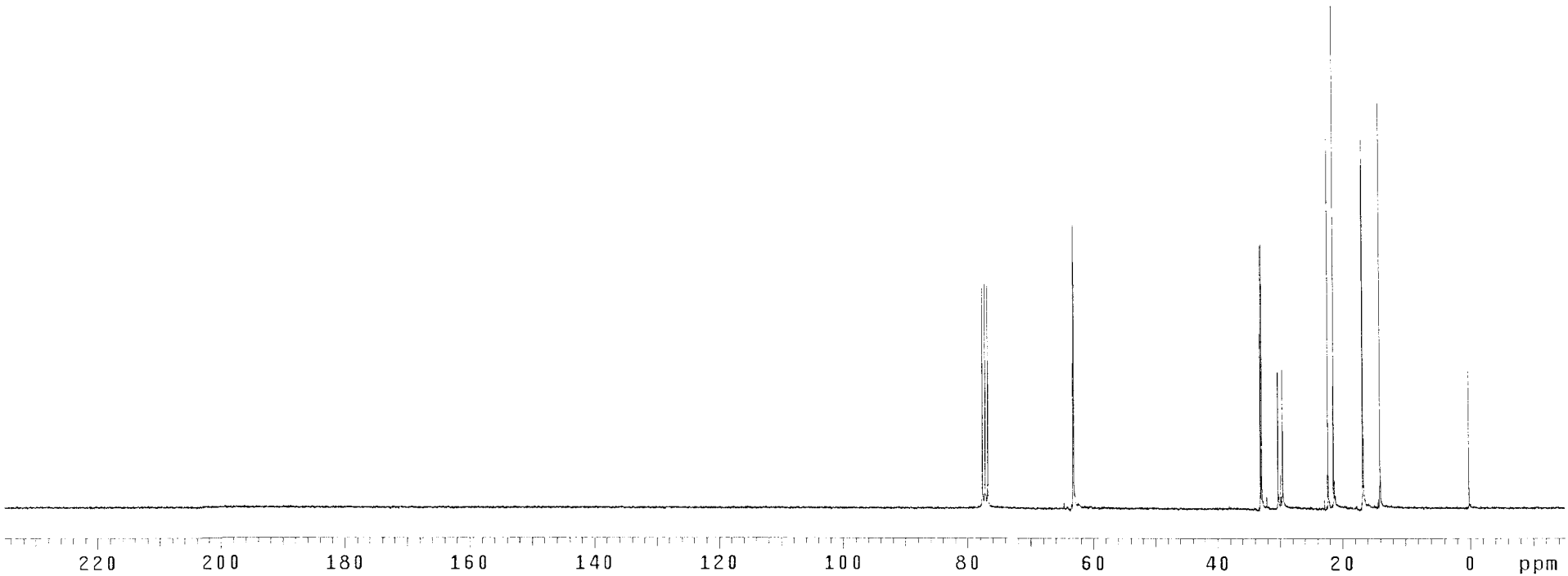
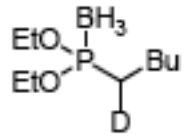


Table 1, entry 8

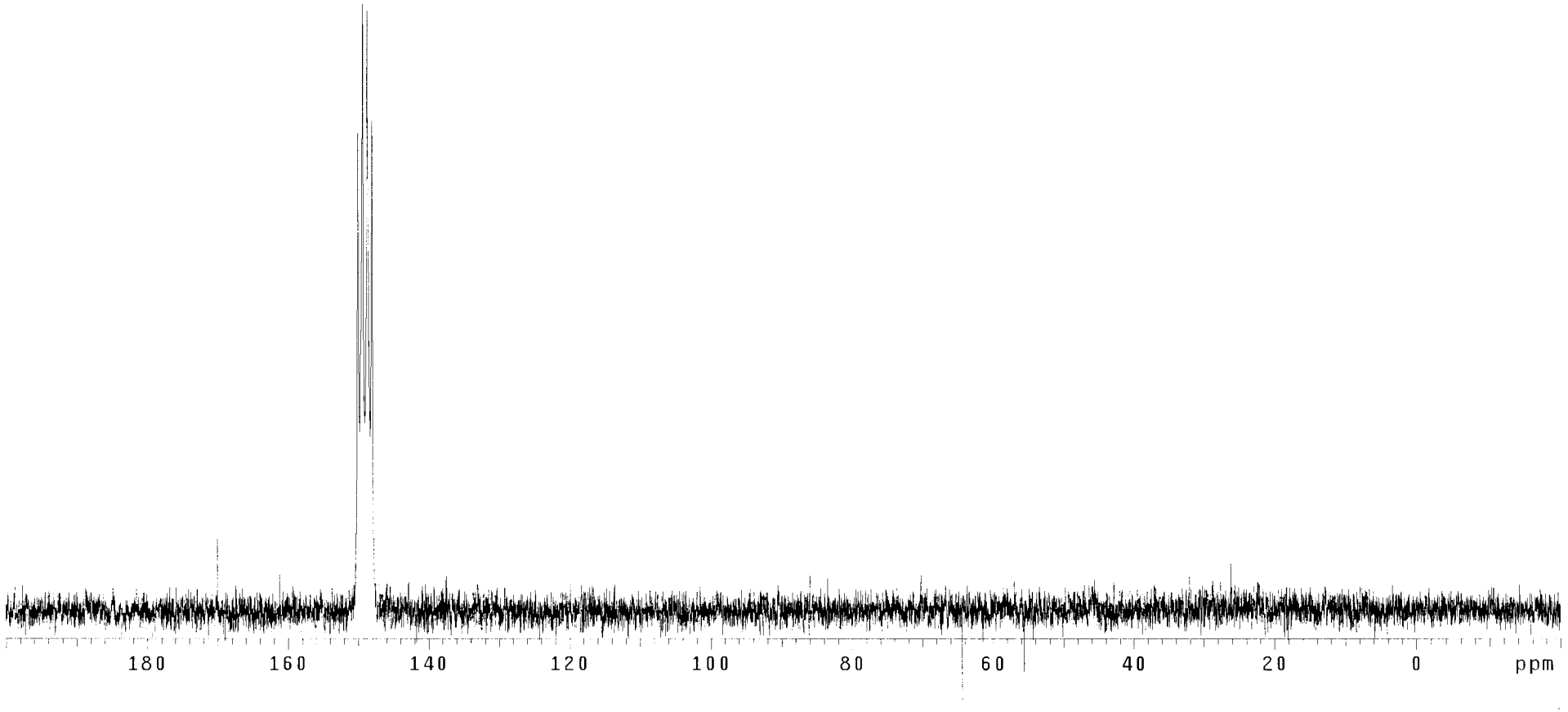


INDEX	FREQUENCY	PPM	HEIGHT
1	18223.634	150.035	76.9
2	18145.709	149.393	97.8
3	18062.071	148.705	97.0
4	17983.329	148.056	78.9
5	7819.915	64.381	-14.9



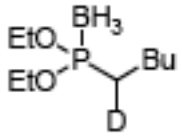
$^{31}P/^{1}H$ decoupled

Diethoxy pentylphosphonite-borane Scheme 4



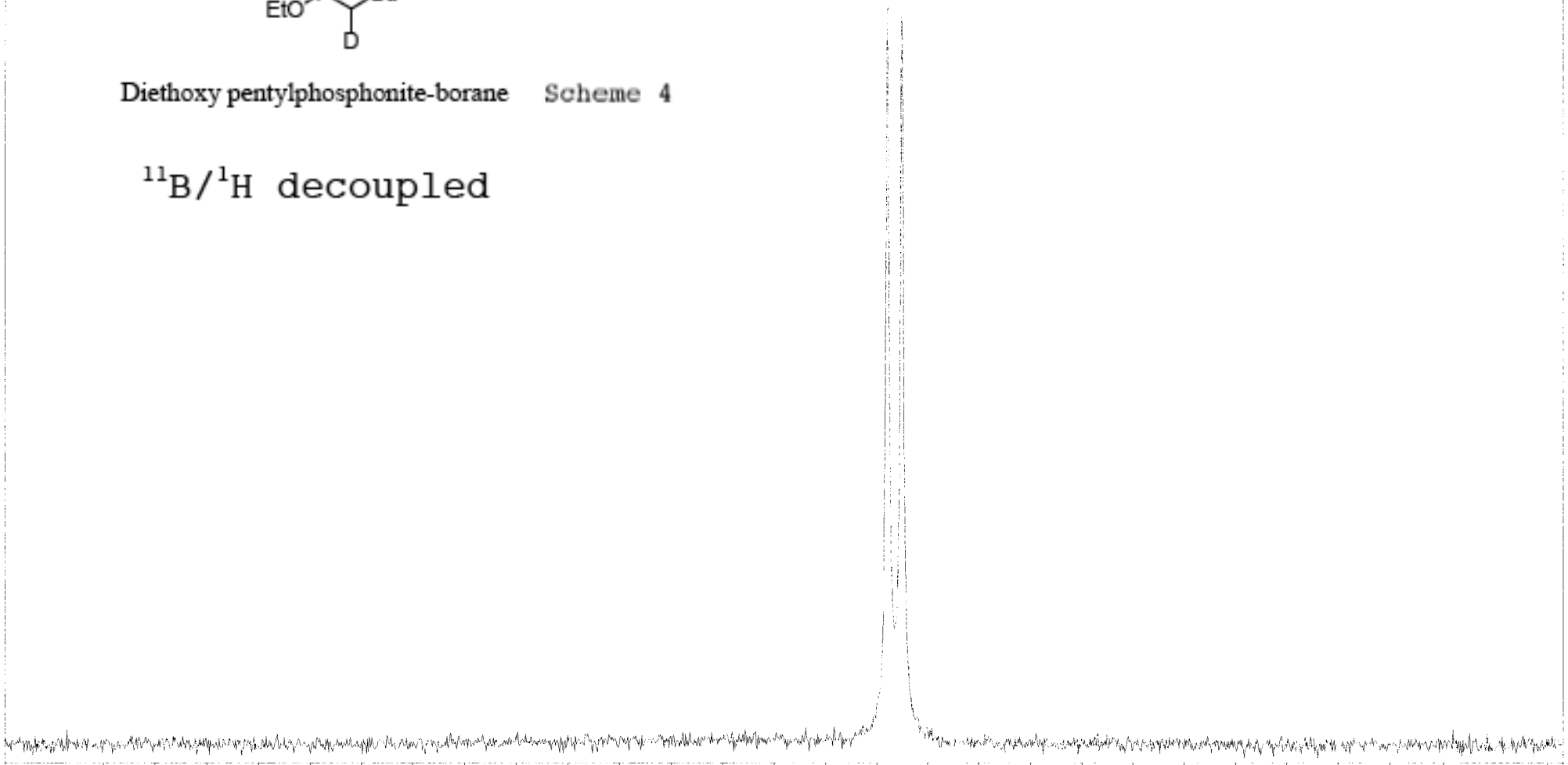
Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	1274	54699	103.28	-1221.55	-42.295
2	1290	52373	98.88	-1301.15	-45.051



Diethoxy pentylphosphonite-borane Scheme 4

$^{11}B/^{1}H$ decoupled



USER: -- DATE: 12/04/07 (19:25)

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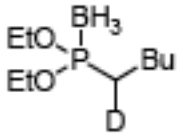
EX: c:\eft\global\zg.ppg	PW: 15.5 usec	PD: 0.1 sec	NA: 128	LB: 0.0	

Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	1245	7062	23.79	-1080.98	-37.428
2	1264	23915	80.57	-1172.65	-40.602
3	1281	28990	97.67	-1257.21	-43.529
4	1300	23836	80.30	-1349.01	-46.708
5	1319	6405	21.58	-1443.55	-49.981



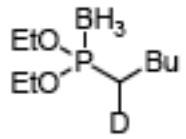
Diethoxy pentylphosphonite-borane Scheme 4

$^{11}B/^{1}H$ coupled

USER: -- DATE: 12/04/07 (19:22)

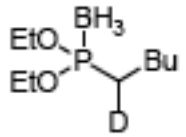
F1: 28.882 | SW1: 10000 | OF1: 0.0 | PTS1d: 2048

EX: c:\cft\global\zg.ppg | PW: 15.5 usec | PD: 0.1 sec | NA: 128 | LB: 0.0

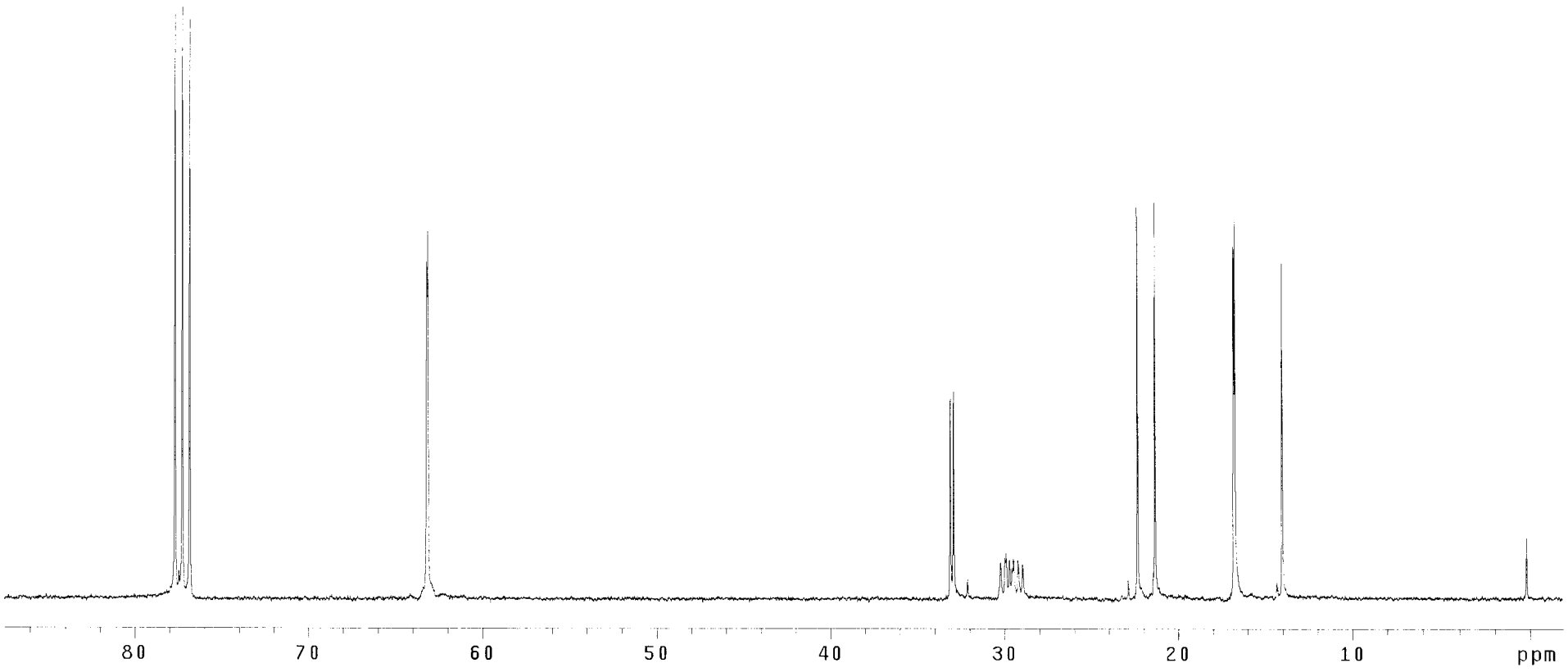


Diethoxy pentylphosphonite-borane Scheme 4

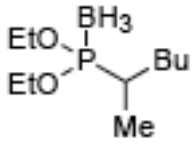
INDEX	FREQUENCY	PPM	HEIGHT
1	5862.756	77.706	94.1
2	5830.799	77.282	95.5
3	5798.842	76.859	93.3
4	4769.882	63.221	54.1
5	4765.563	63.164	59.0
6	2498.049	33.110	32.3
7	2484.230	32.926	33.3
8	2280.972	30.232	5.7
9	2256.500	29.908	7.3
10	2241.529	29.710	6.1
11	2225.119	29.492	6.3
12	2205.541	29.233	6.1
13	2186.252	28.977	5.7
14	1686.455	22.353	63.0
15	1610.161	21.341	63.8
16	1268.133	16.808	56.7
17	1262.663	16.736	60.7
18	1058.829	14.034	54.0
19	14.034	0.186	9.8



Diethoxy pentylphosphonite-borane **Scheme 4**

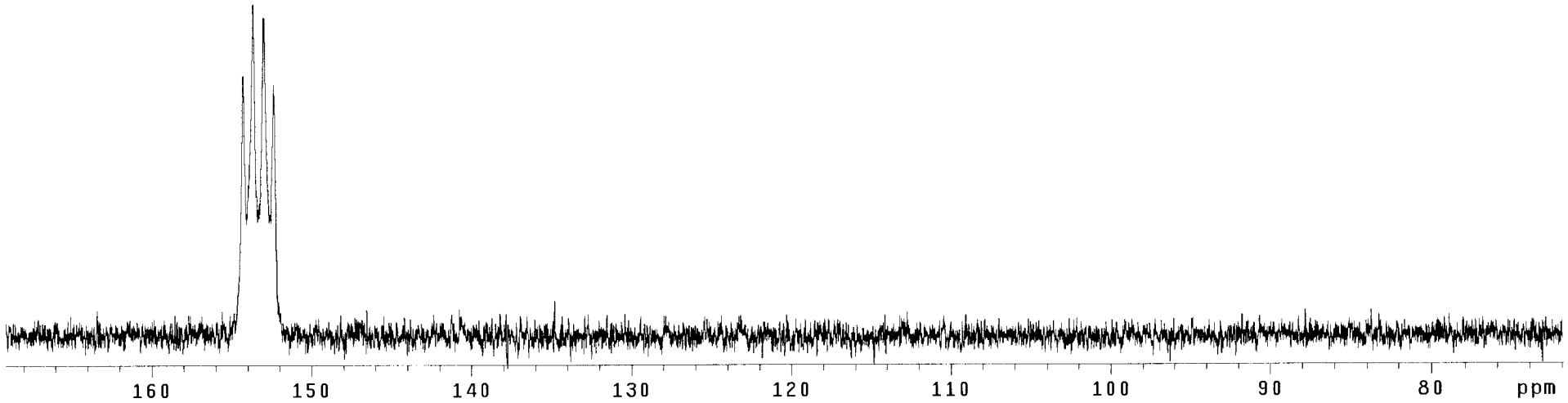


INDEX	FREQUENCY	PPM	HEIGHT
1	18739.333	154.280	41.8
2	18663.039	153.652	53.3
3	18583.481	152.997	51.2
4	18505.147	152.352	40.3

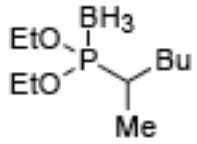


$^{31}P/^{1}H$ decoupled

Table 1, entry 9

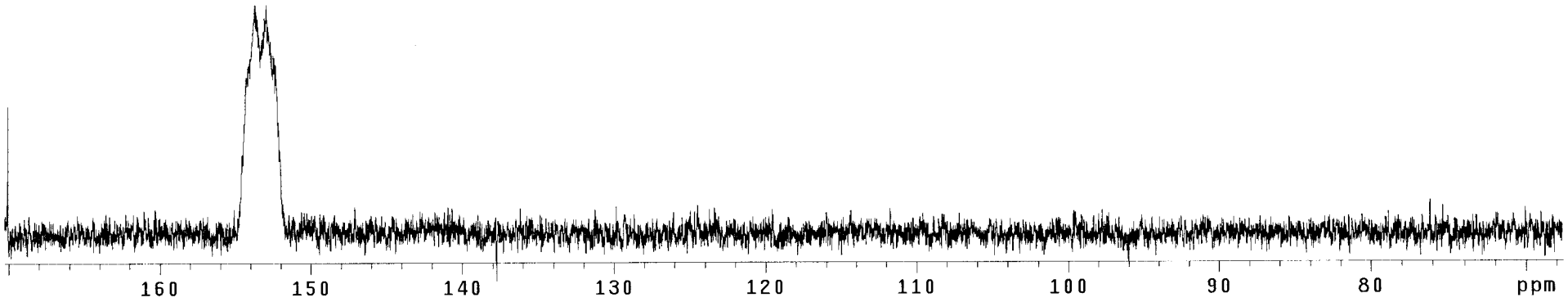


INDEX	FREQUENCY	PPM	HEIGHT
1	20654.025	170.044	20.4
2	18667.935	153.693	36.8
3	18578.177	152.954	36.7



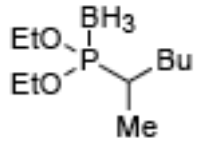
$^{31}P/^{1}H$ coupled

Table 1, entry 9



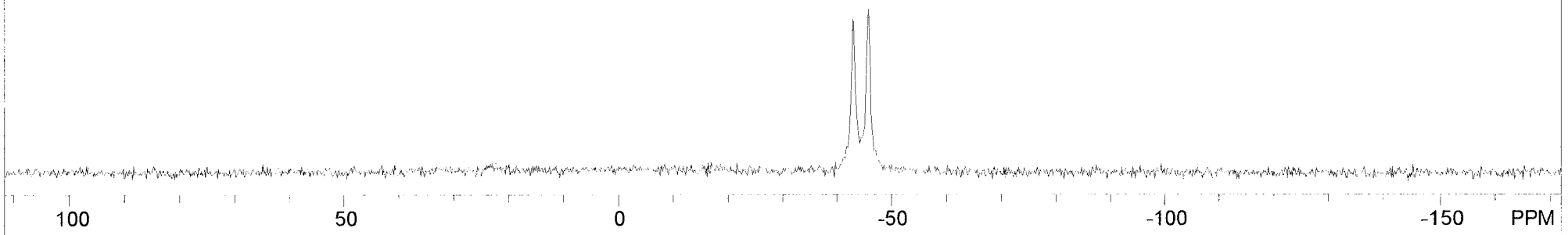
Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	1278	15436	89.52	-1242.87	-43.033
2	1295	16339	94.76	-1322.03	-45.774



$^{11}B/^{1}H$ decoupled

Table 1, entry 9



USER: --DATE: 12/19/07 (16:02)

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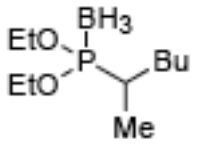
X: c:\cft\global\zg.ppg	PW: 15.5 usec	PD: 0.1 sec	NA: 128	LB: 0.0	

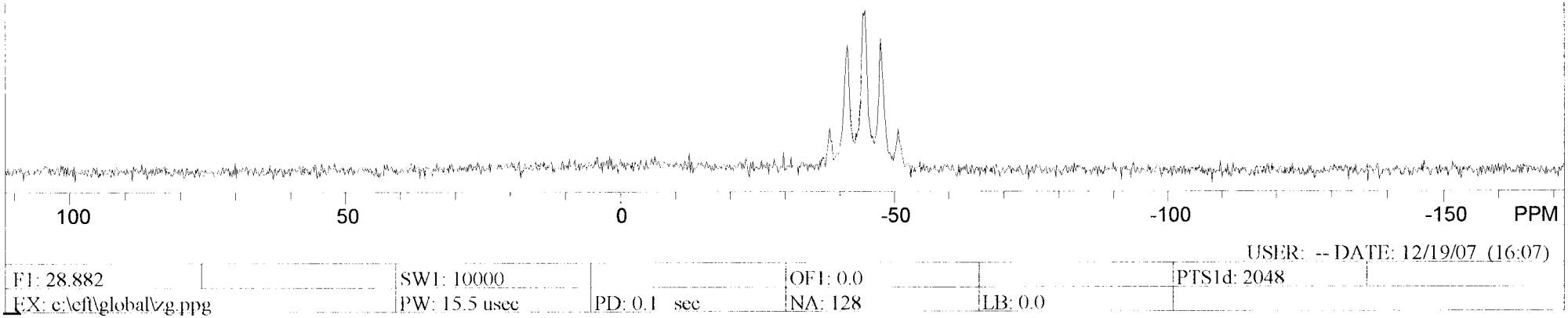
Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	1250	2519	24.75	-1103.73	-38.215
2	1269	7440	73.09	-1196.86	-41.440
3	1288	9495	93.29	-1290.99	-44.699
4	1305	7741	76.05	-1371.58	-47.489
5	1324	2307	22.66	-1463.32	-50.666



$^{11}B/^{1}H$ coupled

Table 1, entry 9



USER: -- DATE: 12/19/07 (16:07)

F1: 28.882 | SW1: 10000 | OF1: 0.0 | PTS1d: 2048

EX: c:\cft\global\zg.ppg | PW: 15.5 usec | PD: 0.1 sec | NA: 128 | LB: 0.0

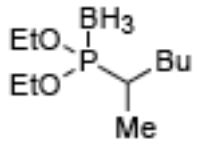


Table 1, entry 9

INDEX	FREQUENCY	PPM	HEIGHT
1	5845.268	77.474	74.6
2	5813.311	77.051	77.5
3	5781.354	76.627	77.4
4	4778.016	63.329	32.1
5	4773.122	63.264	63.2
6	4768.516	63.203	41.7
7	2570.674	34.072	33.6
8	2513.382	33.313	34.3
9	2233.541	29.604	62.7
10	2221.737	29.447	60.2
11	2152.929	28.535	75.6
12	2150.913	28.509	74.7
13	1696.029	22.479	101.5
14	1254.388	16.626	83.0
15	1248.917	16.553	80.4
16	1052.281	13.947	83.8
17	910.633	12.070	68.4
18	0.000	0.000	26.6

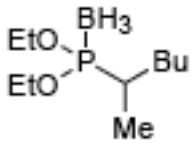
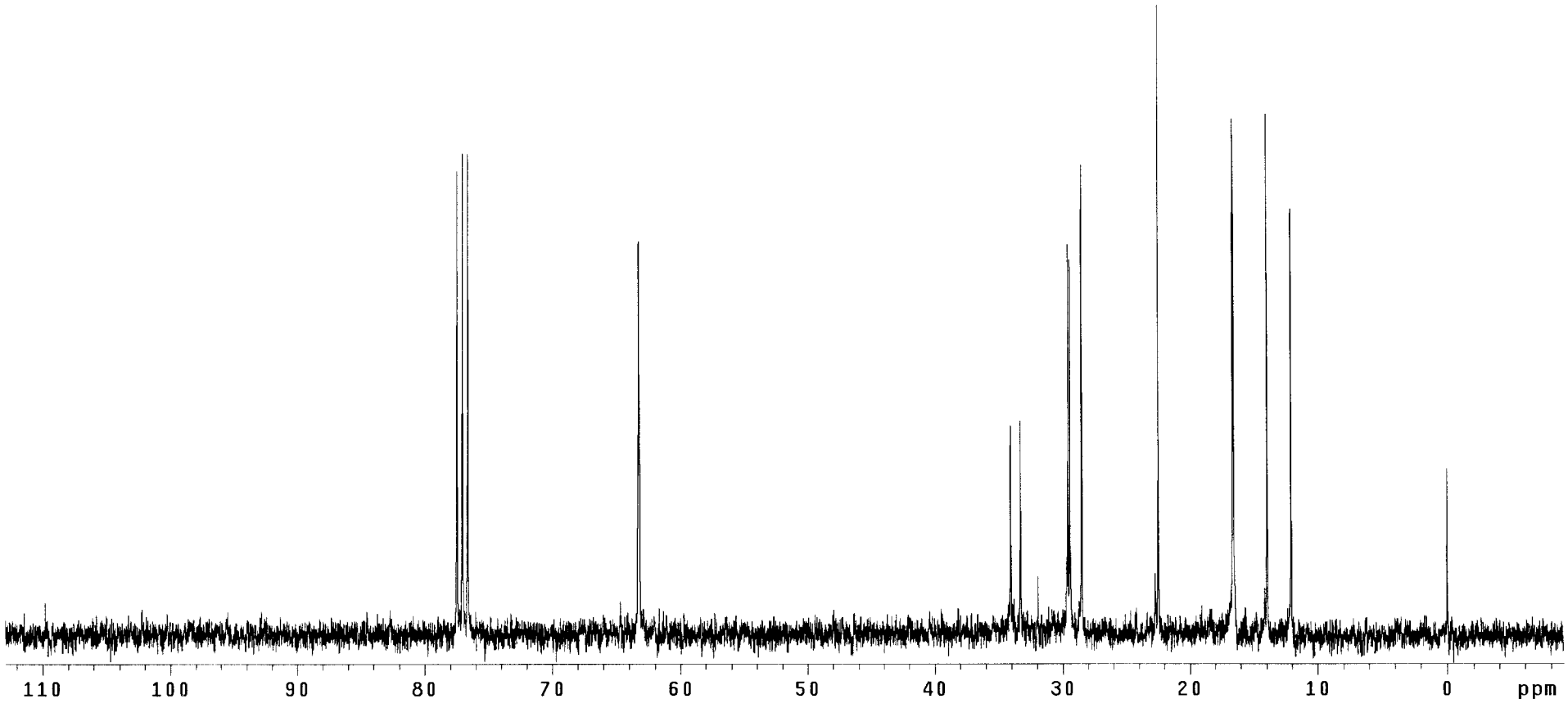
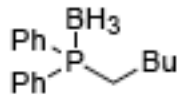


Table 1, entry 9

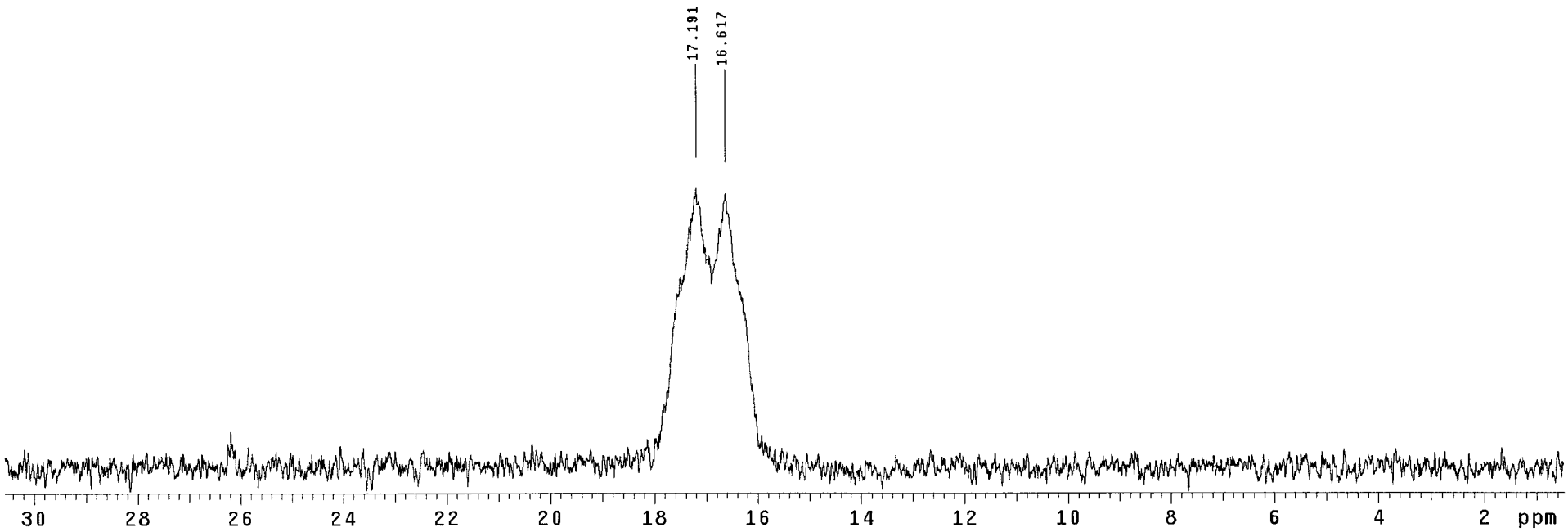


INDEX	FREQUENCY	PPM	HEIGHT
1	2088.077	17.191	44.4
2	2018.311	16.617	43.5



$^{31}P/^{1}H$ decoupled

Table 1, entry 10



INDEX	FREQUENCY	PPM	HEIGHT
1	7819.507	64.378	34.7
2	2100.317	17.292	35.9
3	2028.511	16.701	33.1

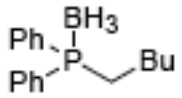
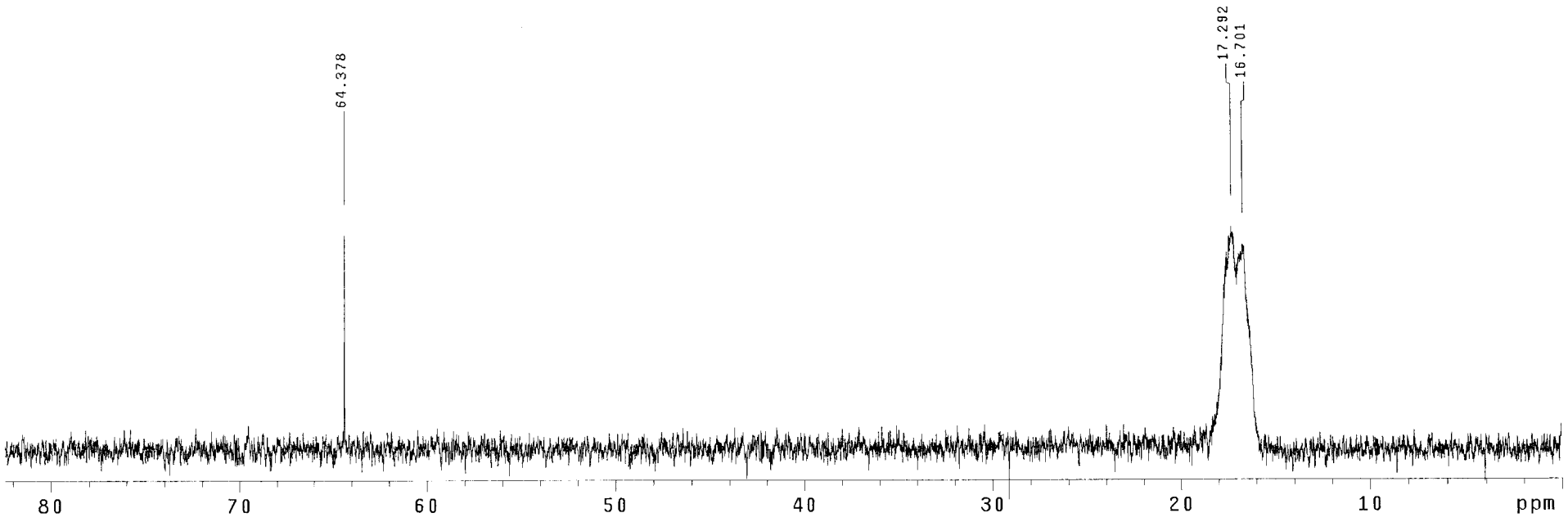
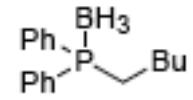


Table 1, entry 10

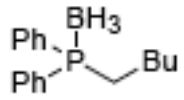
$^{31}P/^{1}H$ coupled





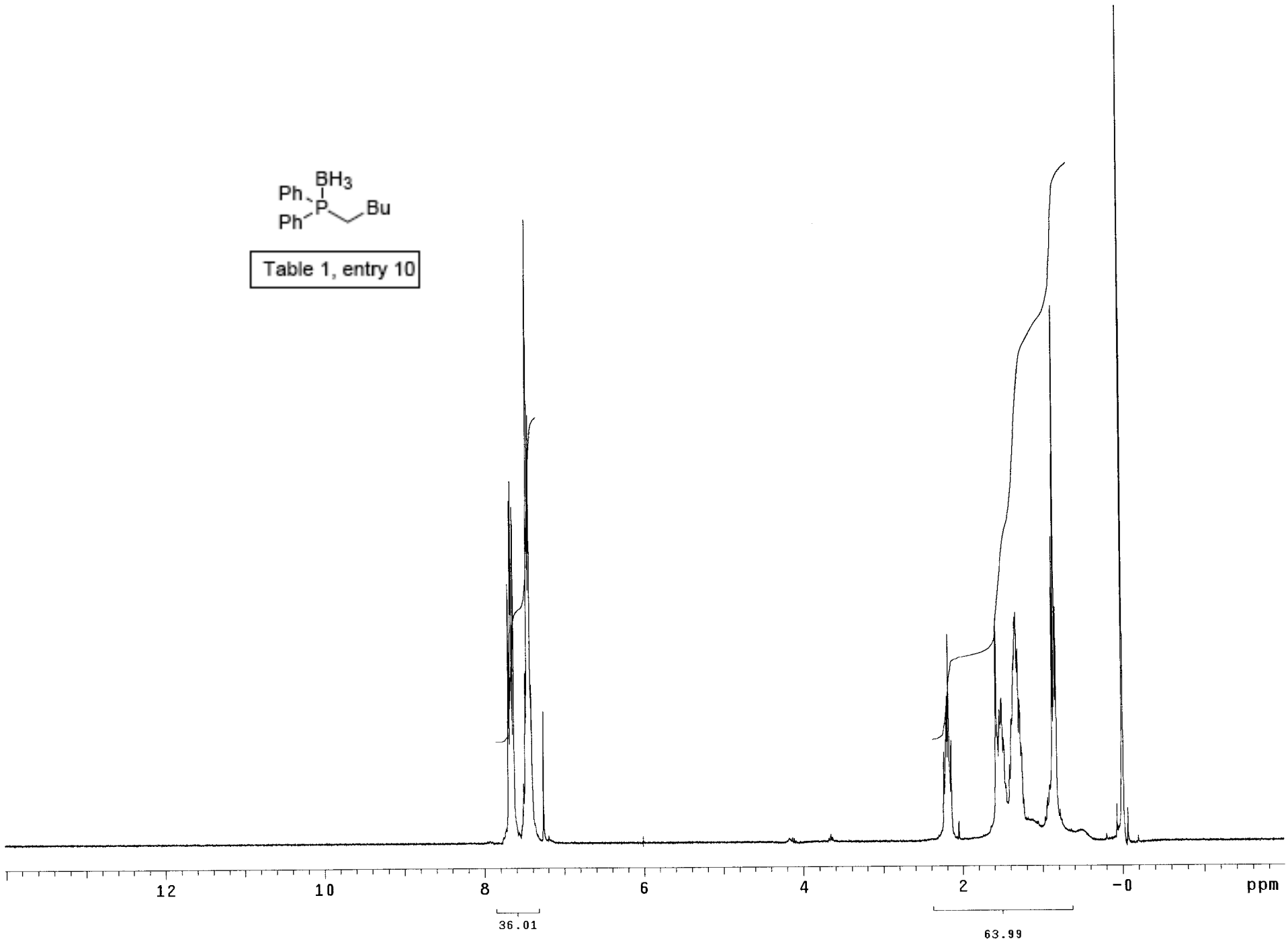
$^{11}B/^{1}H$ decoupled

Table 1, entry 10



$^{11}B/^{1}H$ coupled

Table 1, entry 10


BH3
Ph
P
Ph
Bu
Table 1, entry 10
12
10
8
6
4
2
-0
ppm
36.01
63.99

INDEX	FREQUENCY	PPM	HEIGHT
1	9991.266	132.426	126.0
2	9982.341	132.308	117.4
3	9908.351	131.327	98.5
4	9831.769	130.312	14.7
5	9776.780	129.583	18.0
6	9739.352	129.087	120.2
7	9729.564	128.957	113.2
8	5865.059	77.737	39.2
9	5833.102	77.313	41.6
10	5801.145	76.889	41.6
11	2534.901	33.598	36.2
12	2521.081	33.415	36.5
13	1966.295	26.062	31.5
14	1929.444	25.573	32.6
15	1725.897	22.875	67.5
16	1684.151	22.322	61.9
17	1063.147	14.091	58.8

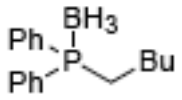
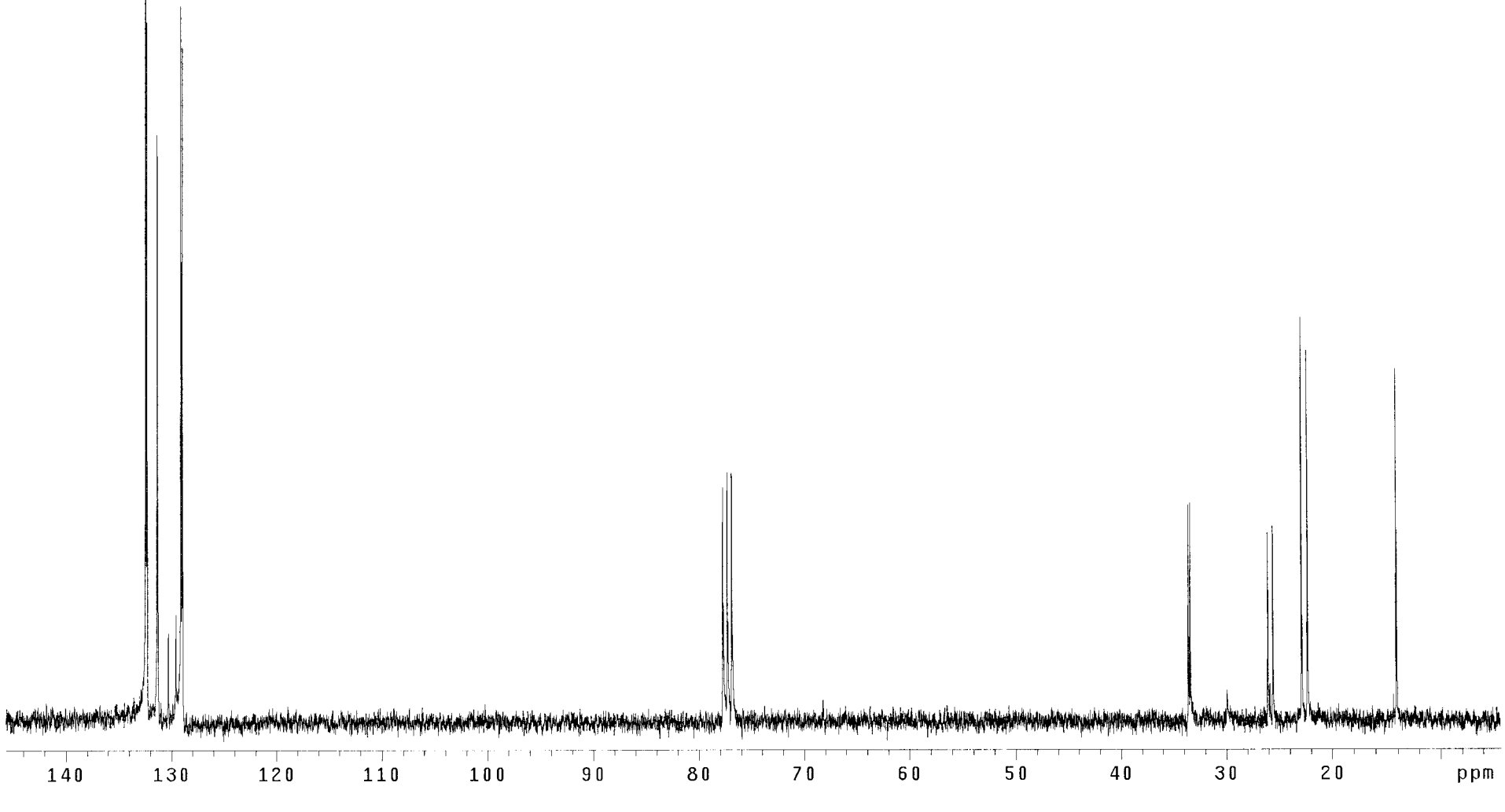
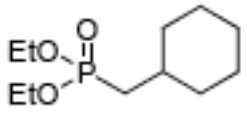


Table 1, entry 10

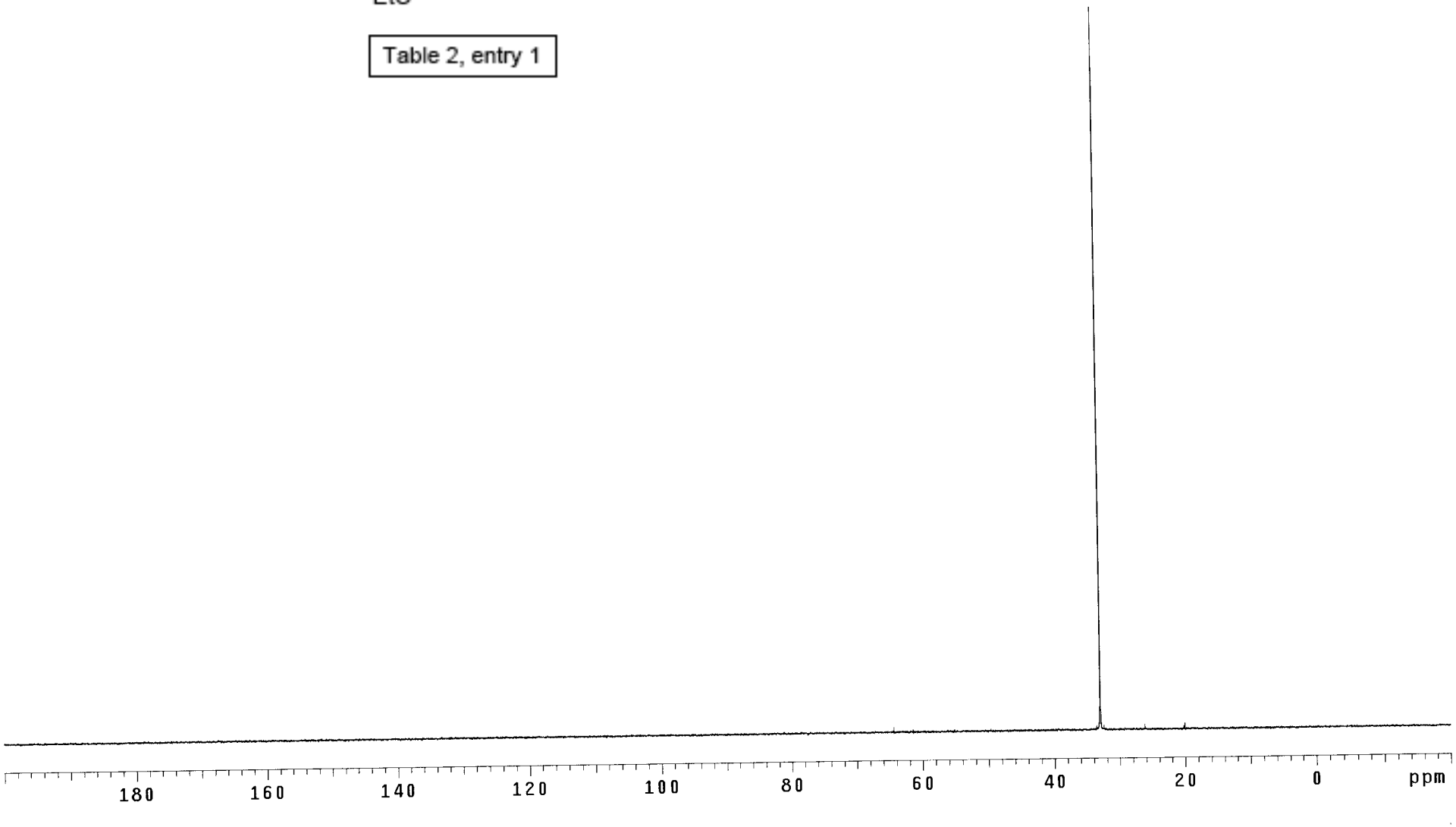


INDEX	FREQUENCY	PPM	HEIGHT
1	4009.297	33.008	126.0

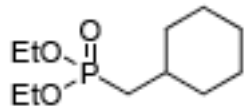


$^{31}P/^{1}H$ decoupled

Table 2, entry 1

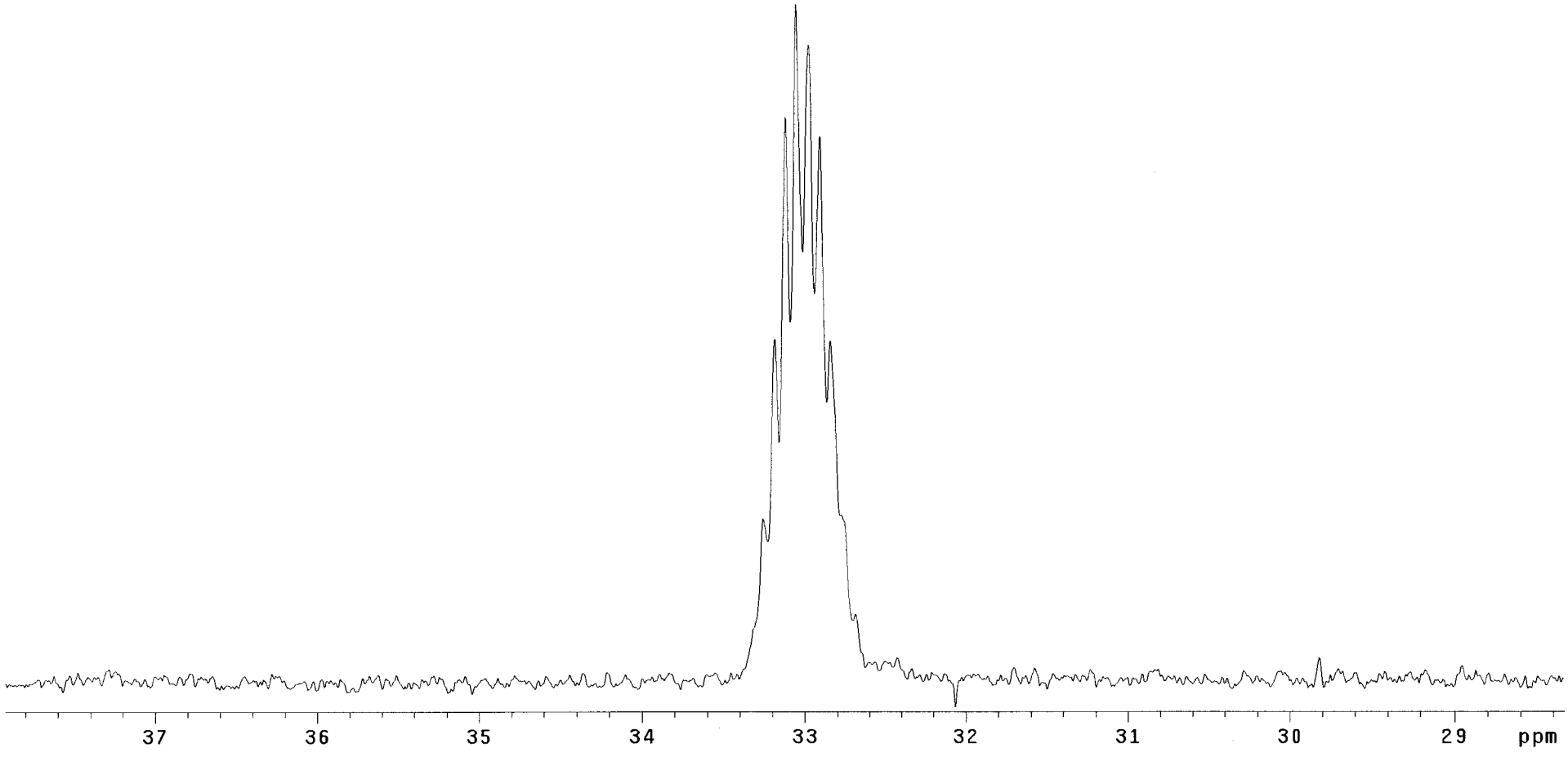


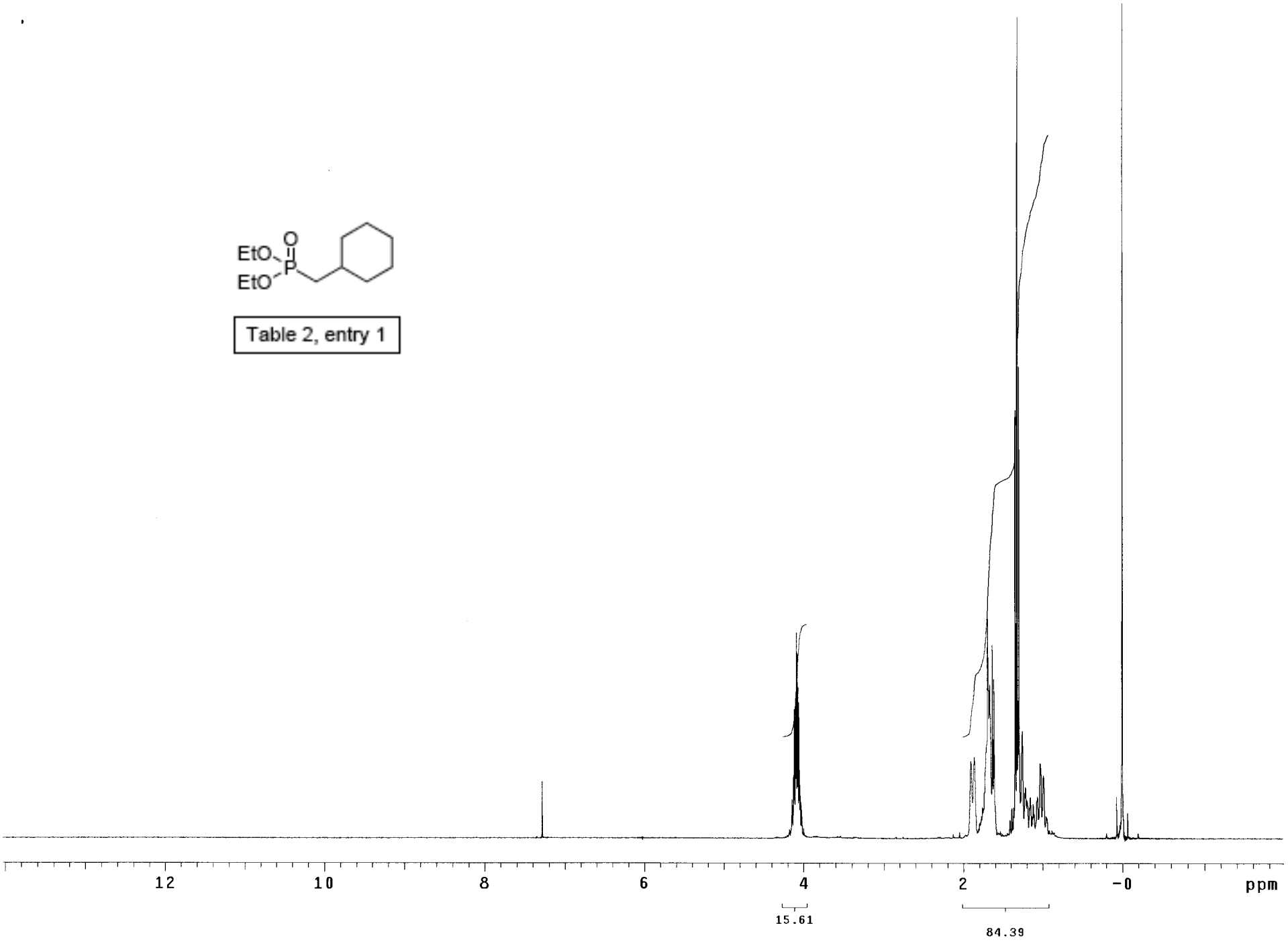
INDEX	FREQUENCY	PPM	HEIGHT
1	4039.080	33.254	26.3
2	4030.105	33.180	55.2
3	4022.353	33.116	91.2
4	4014.193	33.049	109.6
5	4004.809	32.971	103.0
6	3996.242	32.901	88.1
7	3988.490	32.837	55.0



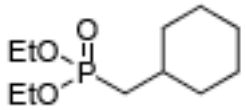
$^{31}P/^{1}H$ coupled

Table 2, entry 1




EtO
EtO
O
P
Table 2, entry 1
12
10
8
6
4
2
-0
ppm
15.61
84.39

INDEX	FREQUENCY	PPM	HEIGHT
1	5845.844	77.482	91.4
2	5813.887	77.058	92.7
3	5781.929	76.635	91.1
4	4623.413	61.280	57.8
5	4616.791	61.192	58.3
6	2612.708	34.629	86.5
7	2602.055	34.488	87.7
8	2578.735	34.179	36.8
9	2464.150	32.660	36.7
10	2459.832	32.603	36.0
11	2440.543	32.347	35.3
12	1968.384	26.089	111.1
13	1962.626	26.013	103.5
14	1246.902	16.527	61.1
15	1240.568	16.443	60.4
16	-0.000	-0.000	37.0



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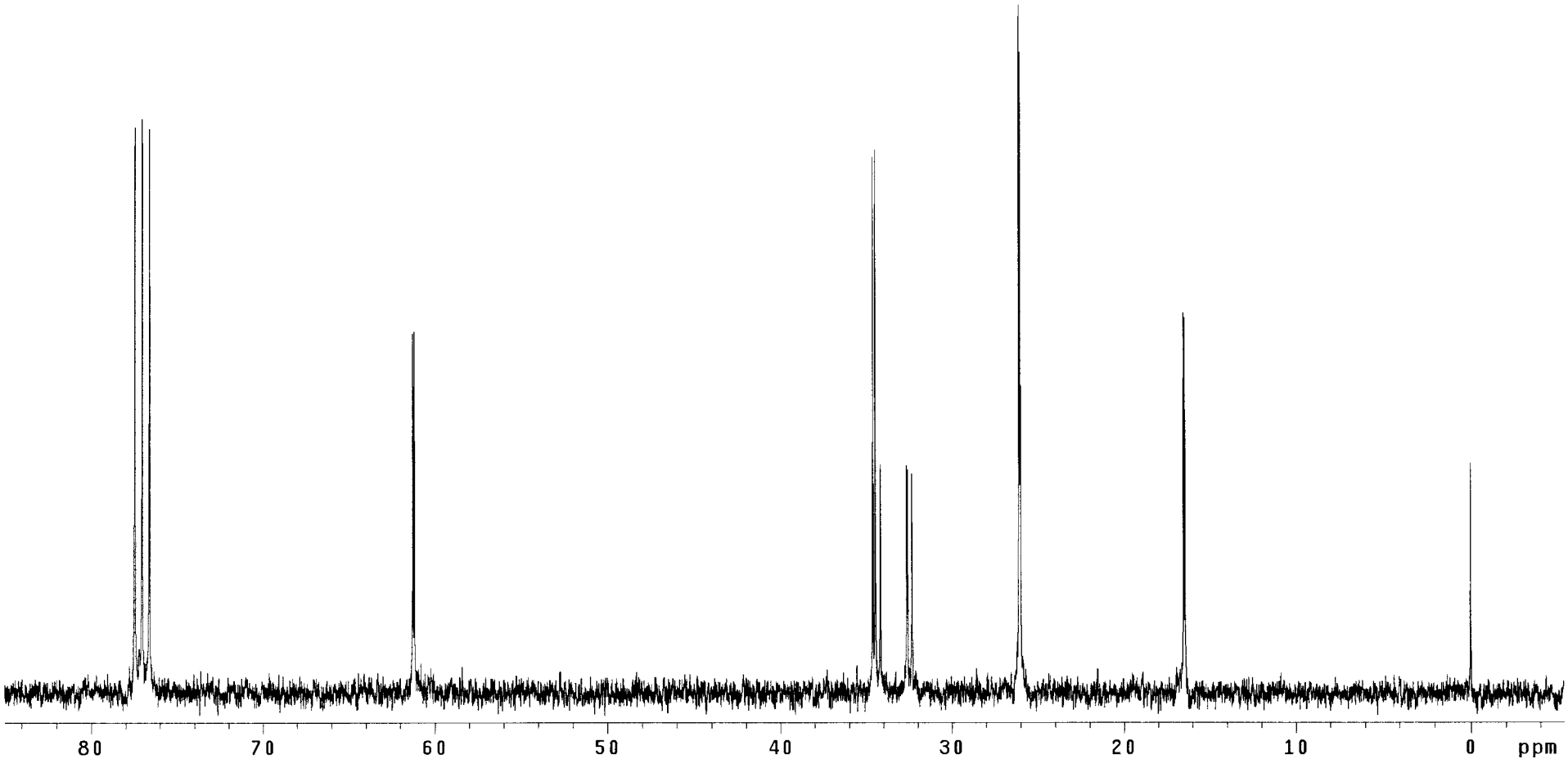


INDEX	FREQUENCY	PPM	HEIGHT
1	4049.688	33.341	126.0

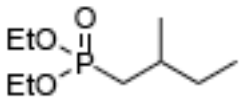
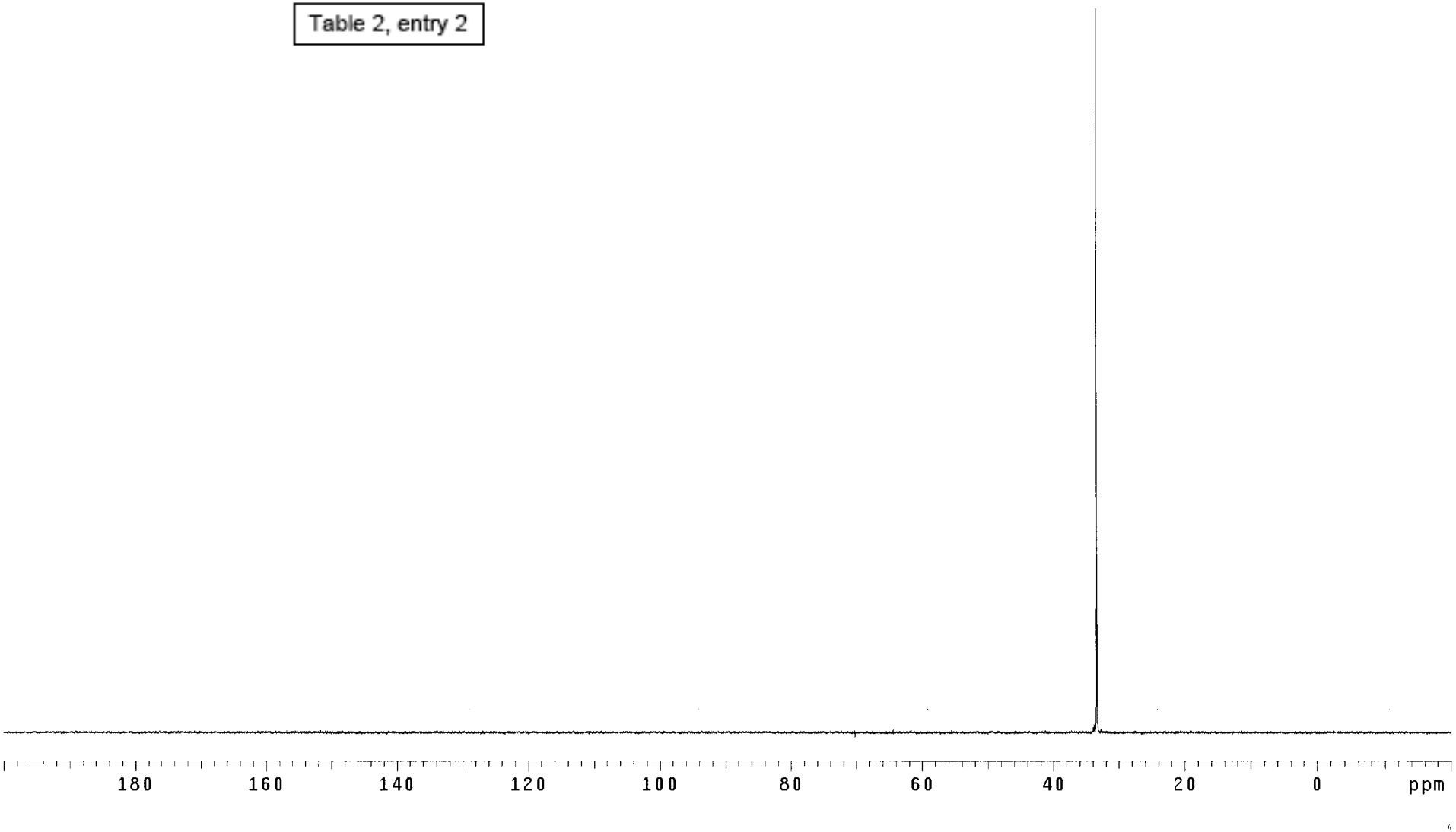
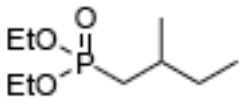


Table 2, entry 2



INDEX	FREQUENCY	PPM	HEIGHT
1	4063.152	33.452	103.0
2	4055.400	33.388	126.0
3	4045.200	33.304	118.1
4	4037.041	33.237	98.2



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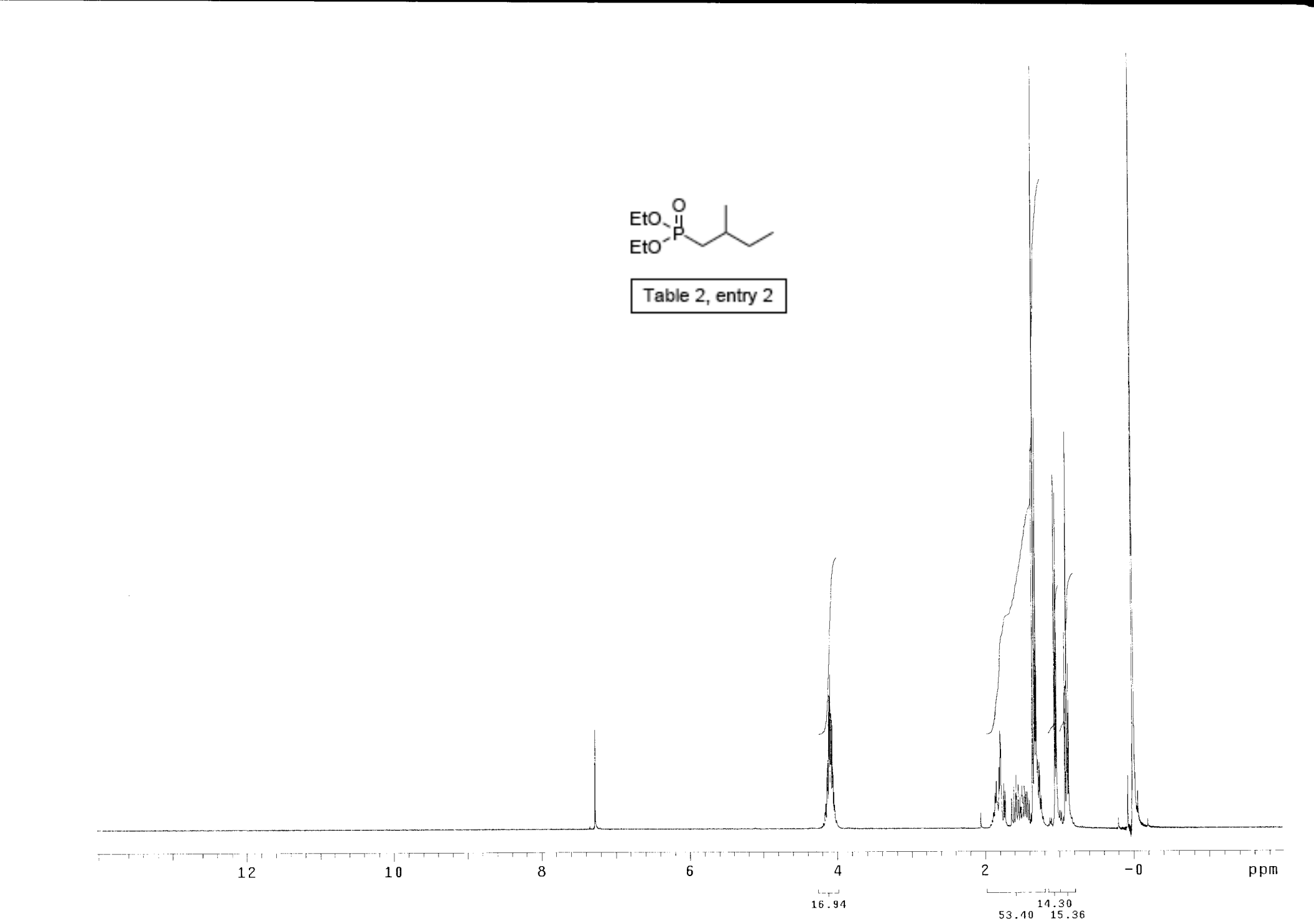
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P
EtO
Table 2, entry 2
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4
2
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ppm
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14.30
53.40
15.36

INDEX	FREQUENCY	PPM	HEIGHT
1	5881.470	77.954	12.1
2	5848.937	77.523	11.5
3	5816.692	77.095	11.5
4	4621.036	61.248	66.4
5	4614.990	61.168	103.1
6	4609.520	61.095	78.4
7	2503.519	33.182	85.8
8	2364.751	31.343	85.2
9	2326.748	30.839	80.6
10	2312.353	30.648	83.5
11	2242.968	29.729	71.2
12	2238.650	29.671	83.6
13	1533.867	20.330	79.0
14	1526.381	20.231	86.0
15	1237.328	16.400	114.1
16	1230.994	16.316	126.0
17	826.204	10.951	113.1

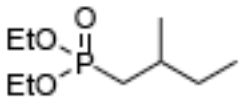
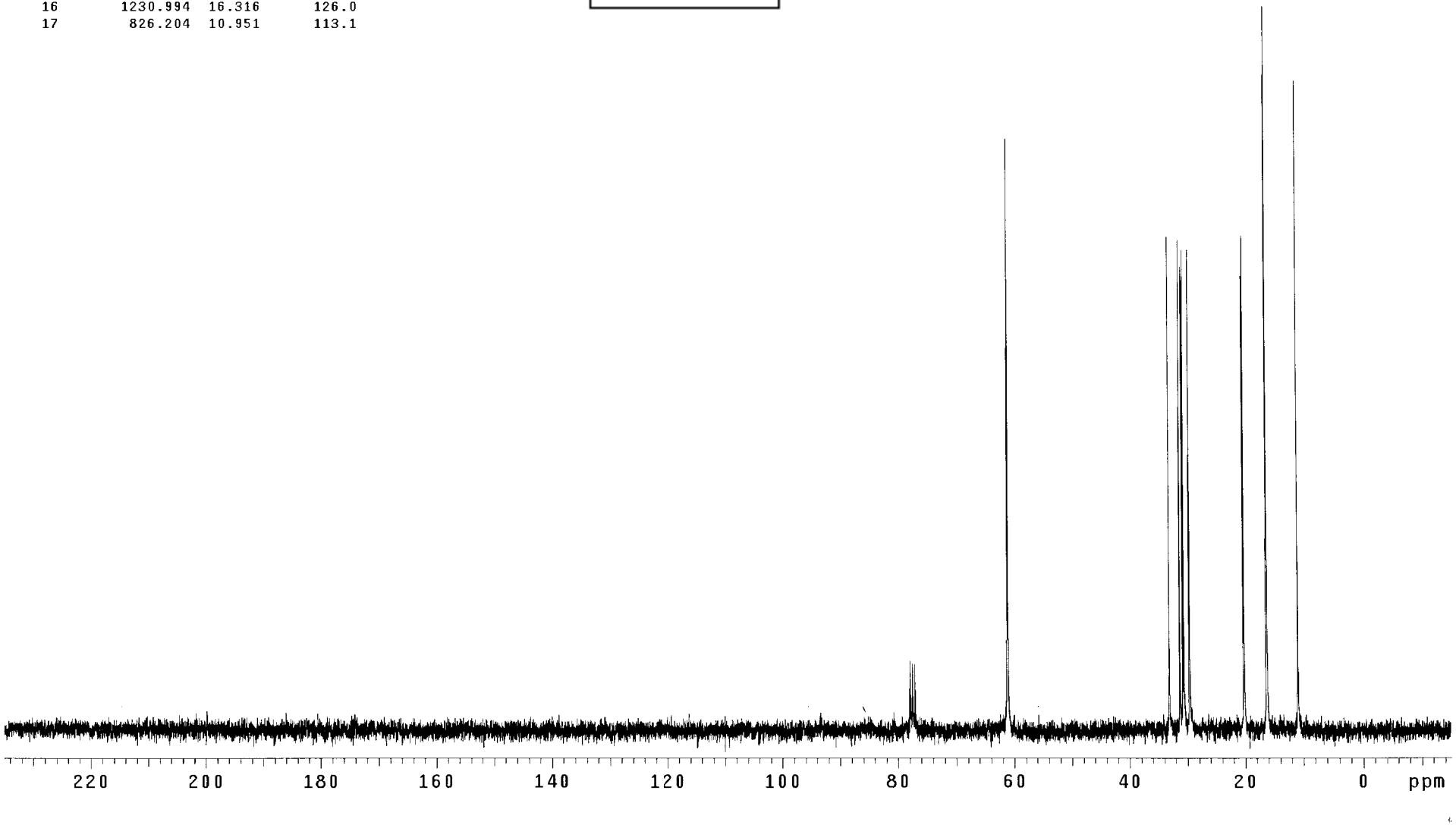
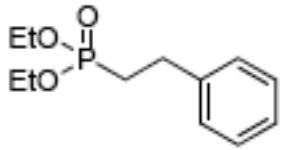


Table 2, entry 2



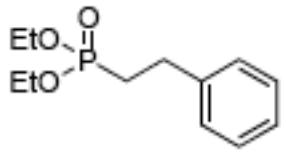
INDEX	FREQUENCY	PPM	HEIGHT
1	7820.323	64.385	-6.5
2	3877.925	31.927	147.8



$^{31}P/^{1}H$ decoupled

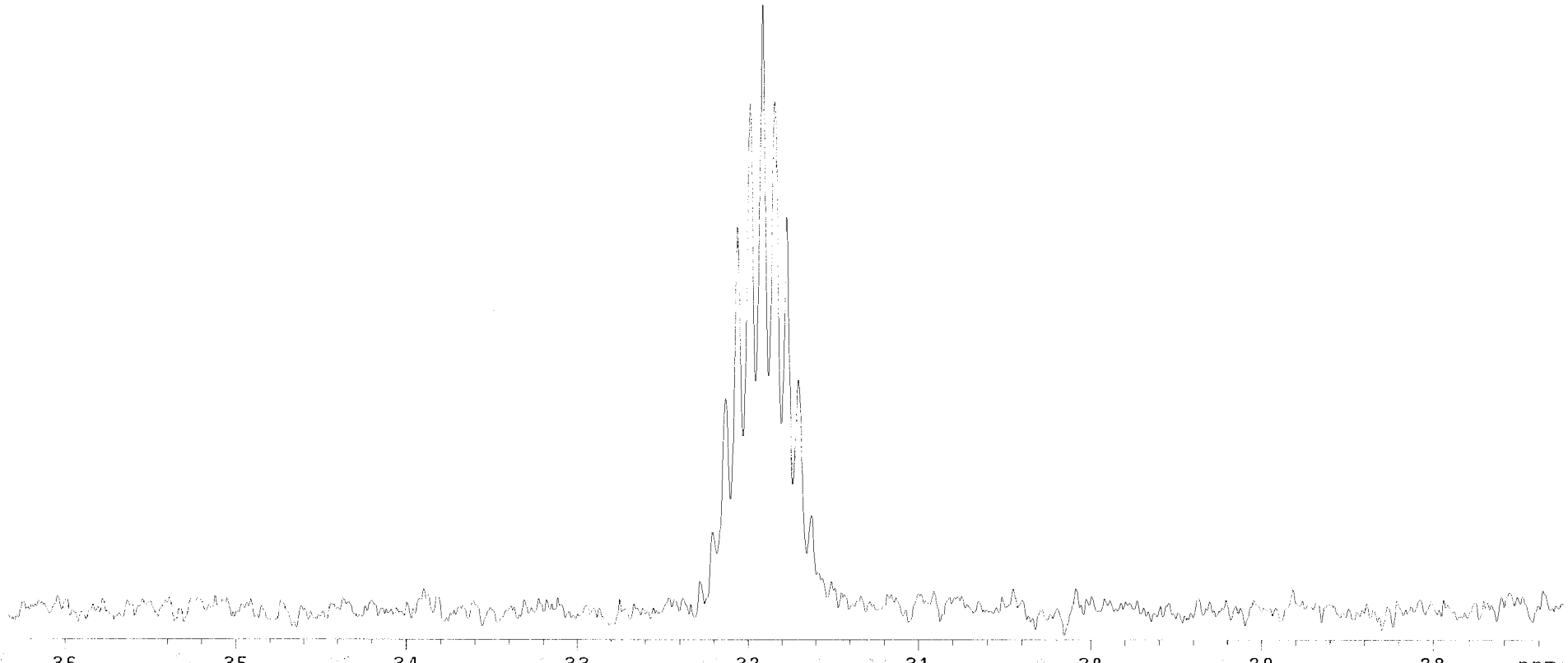
Table 2, entry 3

INDEX	FREQUENCY	PPM	HEIGHT
1	3902.812	32.132	33.8
2	3894.652	32.065	61.3
3	3885.269	31.987	81.2
4	3876.701	31.917	97.2
5	3868.133	31.846	81.6
6	3859.157	31.772	63.0
7	3850.590	31.702	36.8



$^{31}P/^{1}H$ coupled

Table 2, entry 3



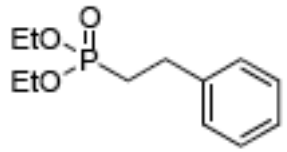
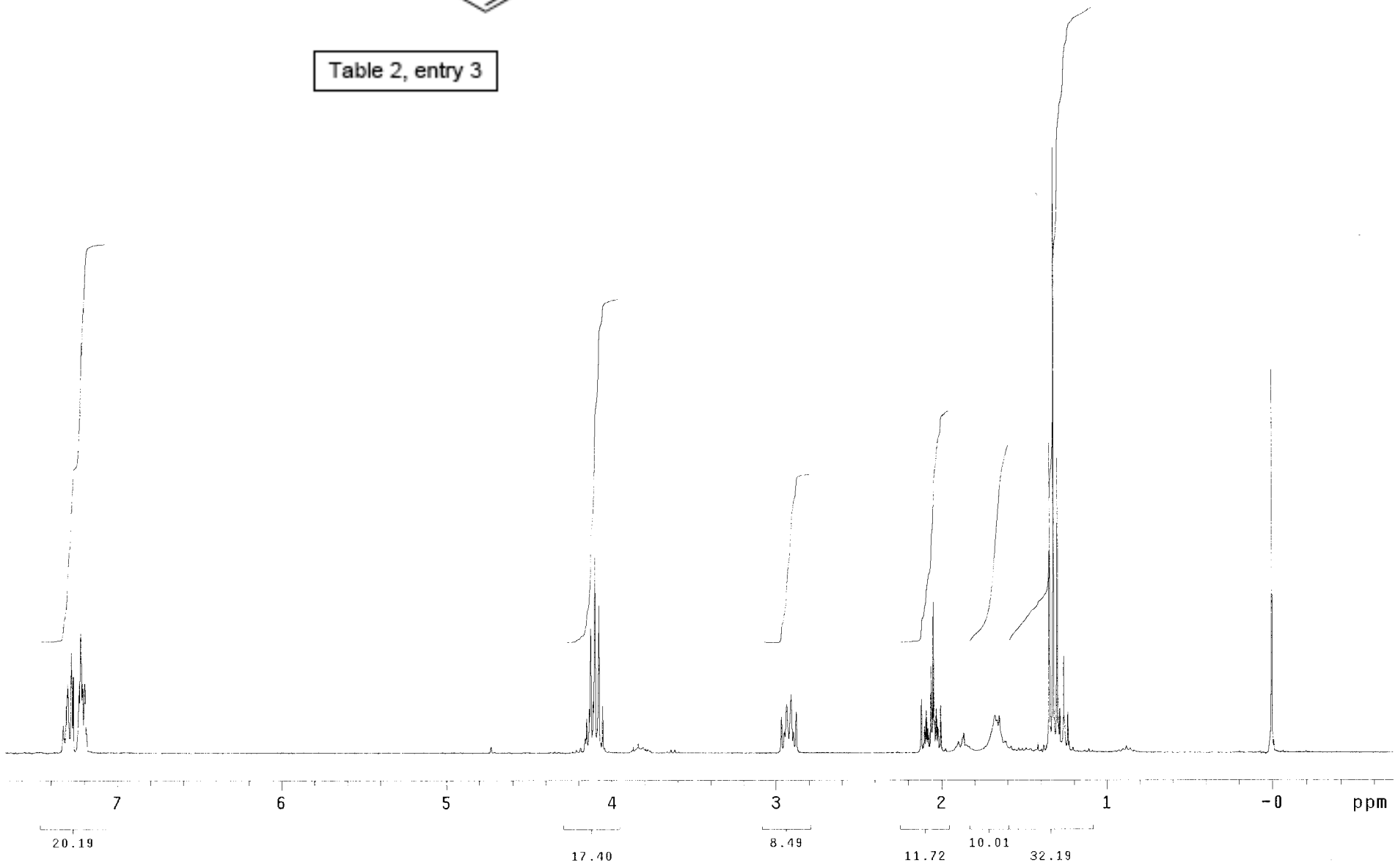


Table 2, entry 3



INDEX	FREQUENCY	PPM	HEIGHT
1	10658.910	141.275	11.0
2	10641.348	141.042	11.7
3	9716.608	128.786	126.0
4	9677.166	128.263	125.3
5	9548.474	126.557	70.2
6	5866.499	77.756	29.7
7	5834.542	77.332	31.2
8	5802.585	76.909	29.3
9	4668.252	61.874	41.6
10	4661.630	61.786	43.1
11	2173.872	28.813	37.7
12	2169.554	28.756	34.2
13	2164.947	28.695	33.8
14	2025.603	26.848	36.2
15	1261.224	16.716	45.8
16	1255.466	16.640	45.1

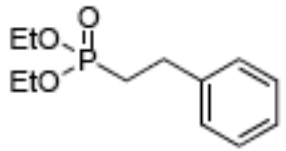


Table 2, entry 3

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std

exp1  s2pul

      SAMPLE                SPECIAL
date   Jun 14 2006    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              192    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             17.0
dn               H1    lp           -251.7
dof               0          PLOT
dm              yyy    wc              250
dmm               w    sc                0
dpwr             35    vs               74
dmf            6700    th                9
                       ai  no  ph
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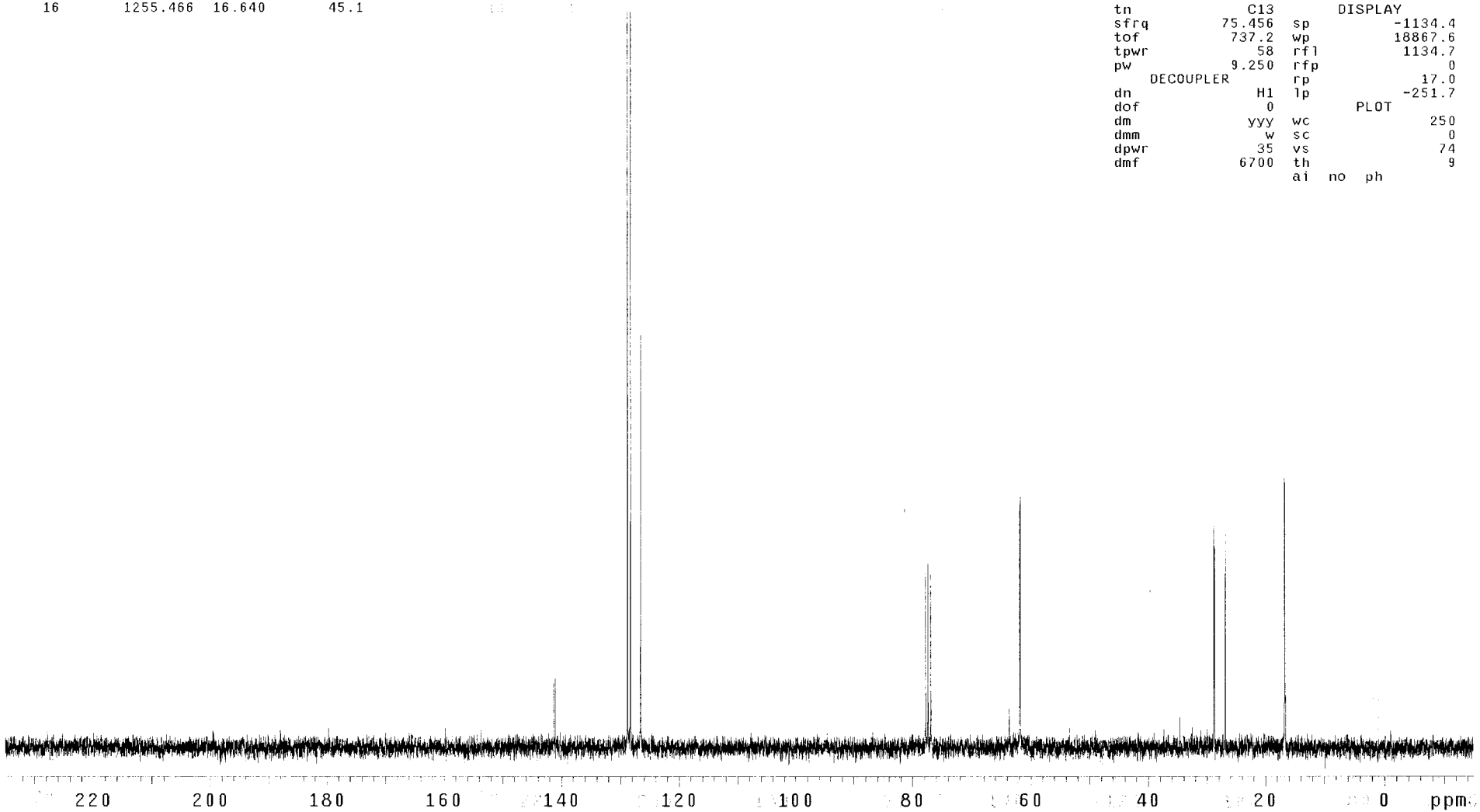
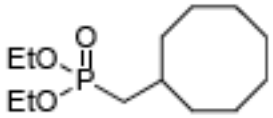
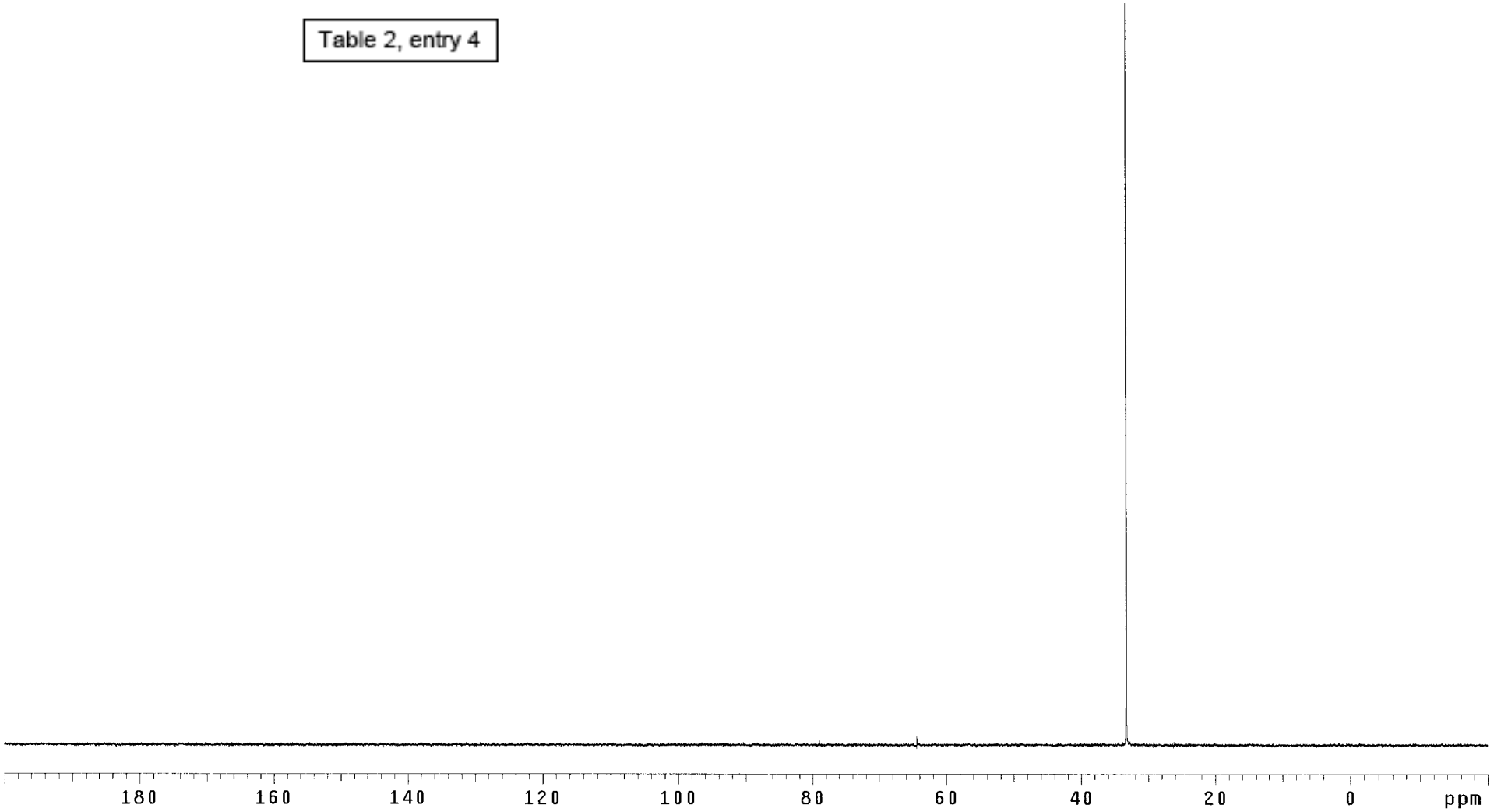


INDEX	FREQUENCY	PPM	HEIGHT
1	4046.016	33.311	126.0

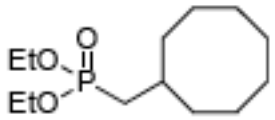


$^{31}P/^{1}H$ decoupled

Table 2, entry 4

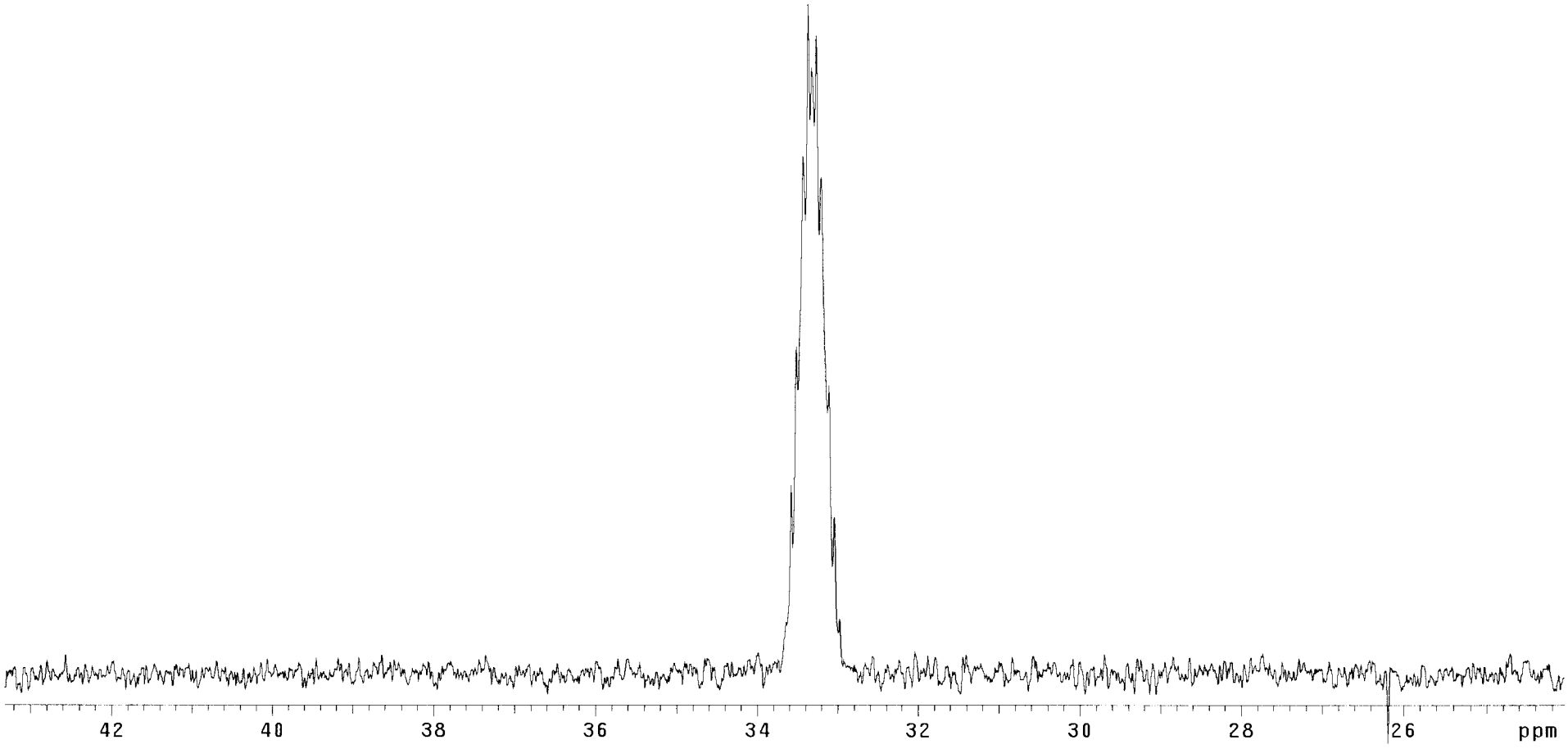


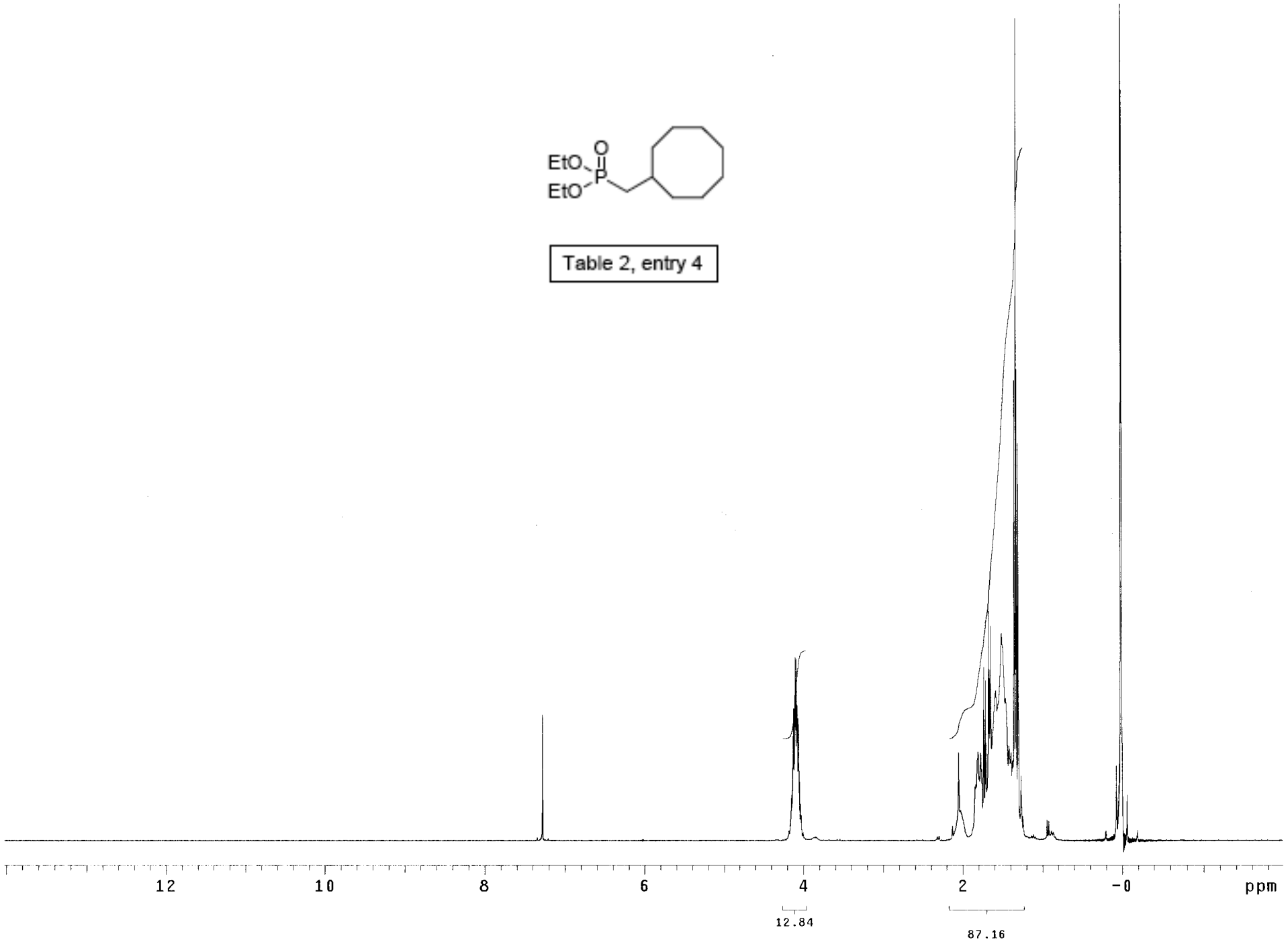
INDEX	FREQUENCY	PPM	HEIGHT
1	4078.655	33.579	30.5
2	4070.496	33.512	52.8
3	4059.888	33.425	83.5
4	4052.136	33.361	108.2
5	4046.832	33.317	98.0
6	4039.488	33.257	103.1
7	4032.145	33.197	80.2
8	4013.377	33.042	25.5



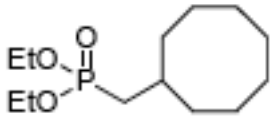
$^{31}P/^{1}H$ coupled

Table 2, entry 4




EtO
O
P
EtO
Table 2, entry 4
12
10
8
6
4
2
-0
ppm
12.84
87.16

INDEX	FREQUENCY	PPM	HEIGHT
1	5846.419	77.489	36.4
2	5814.174	77.062	34.5
3	5782.217	76.639	33.2
4	5770.989	76.490	-5.9
5	4622.261	61.264	50.2
6	4615.928	61.180	48.9
7	2599.464	34.454	31.9
8	2498.123	33.111	72.6
9	2487.471	32.969	72.0
10	2461.847	32.630	36.4
11	2450.331	32.477	38.9
12	2102.546	27.868	5.2
13	2060.224	27.307	123.7
14	1959.171	25.967	70.8
15	1877.695	24.887	124.6
16	1246.326	16.519	48.6
17	1240.568	16.443	52.6
18	-0.288	-0.004	24.2



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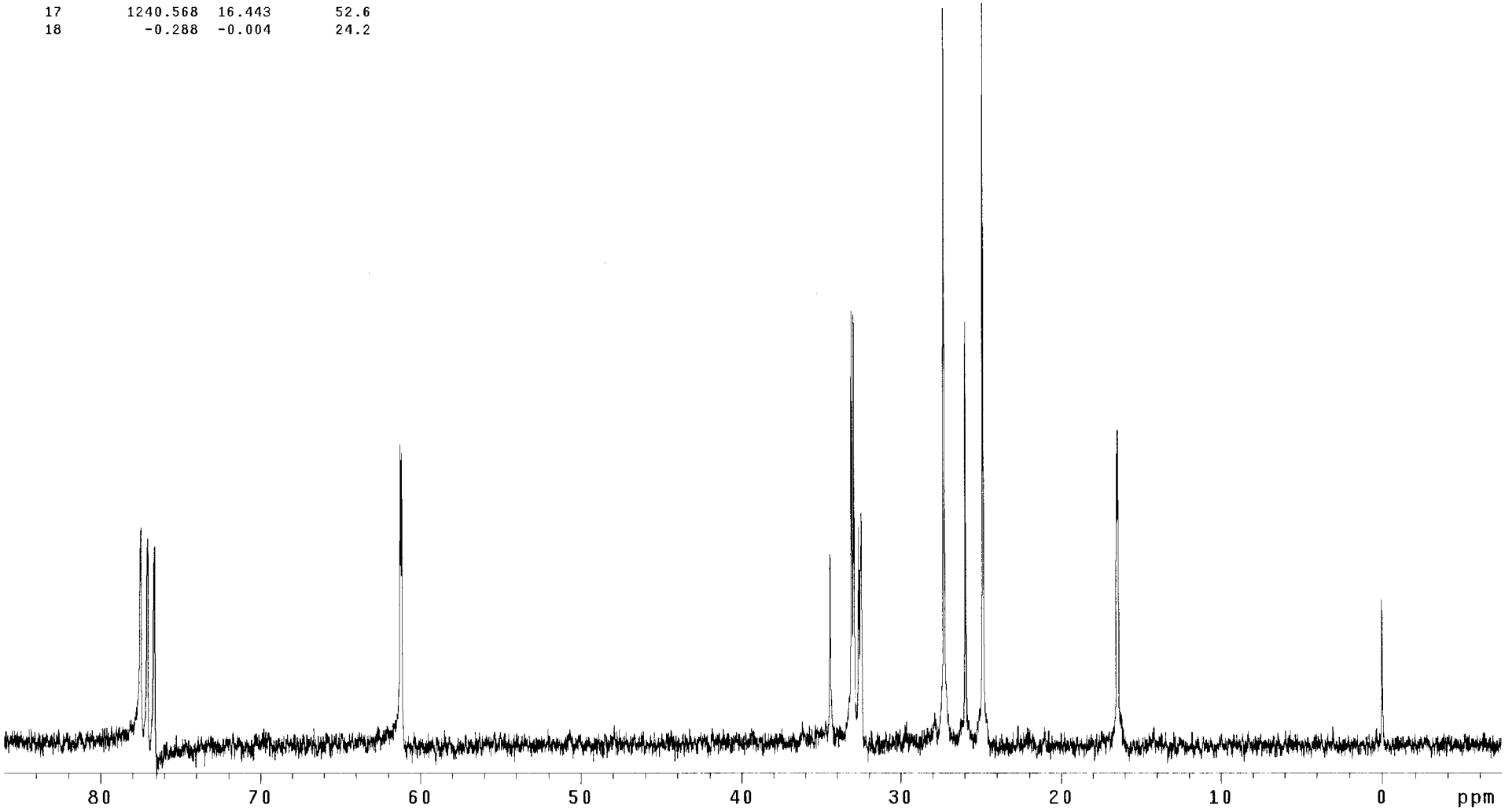


INDEX	FREQUENCY	PPM	HEIGHT
1	3555.614	29.273	125.2

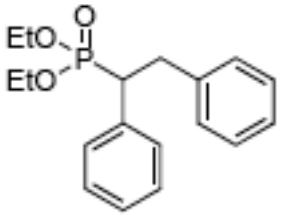
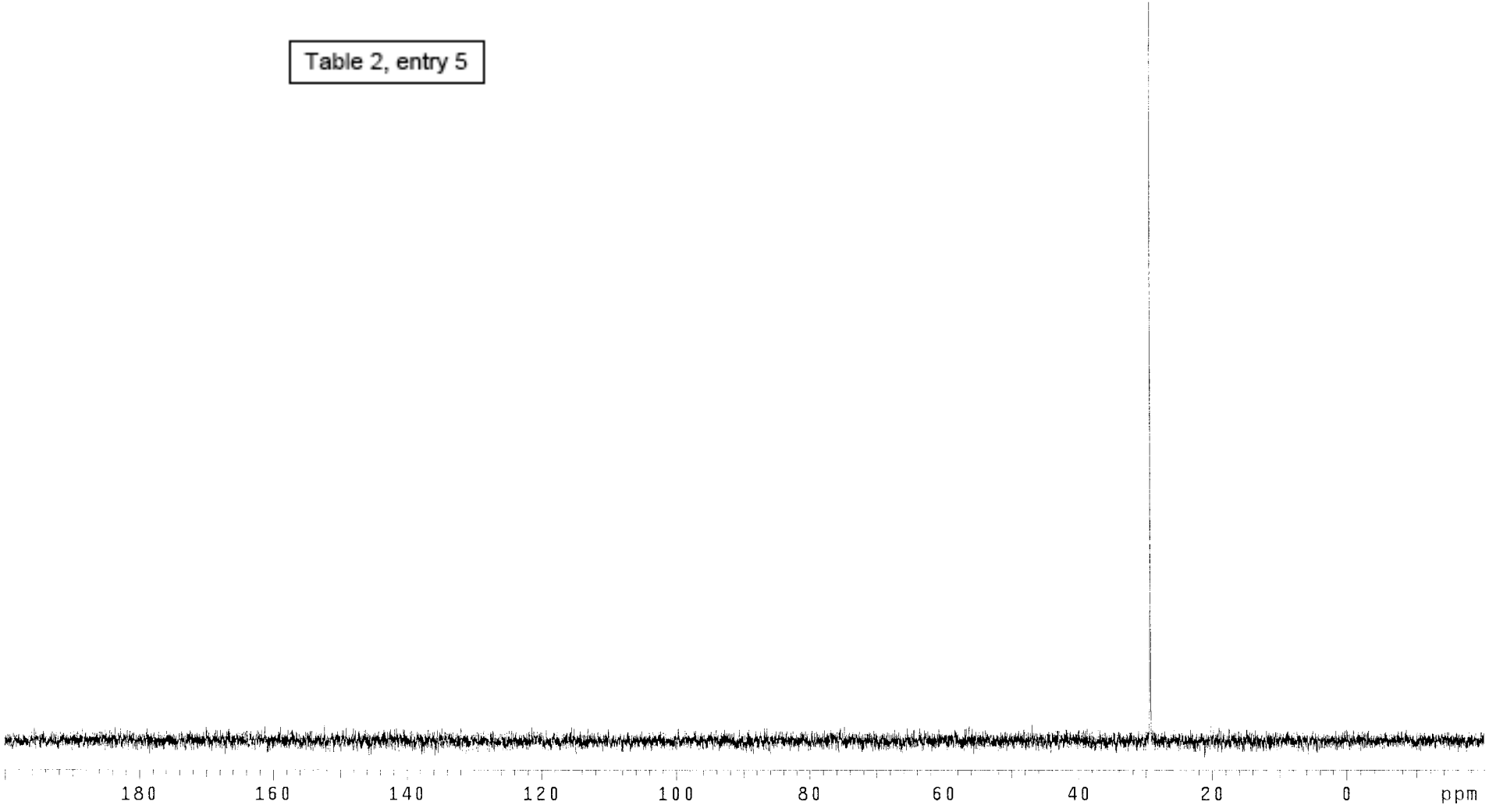
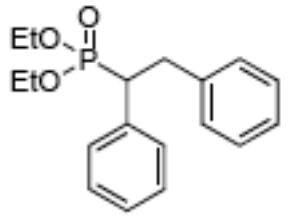


Table 2, entry 5



INDEX	FREQUENCY	PPM	HEIGHT
1	3557.653	29.290	71.6



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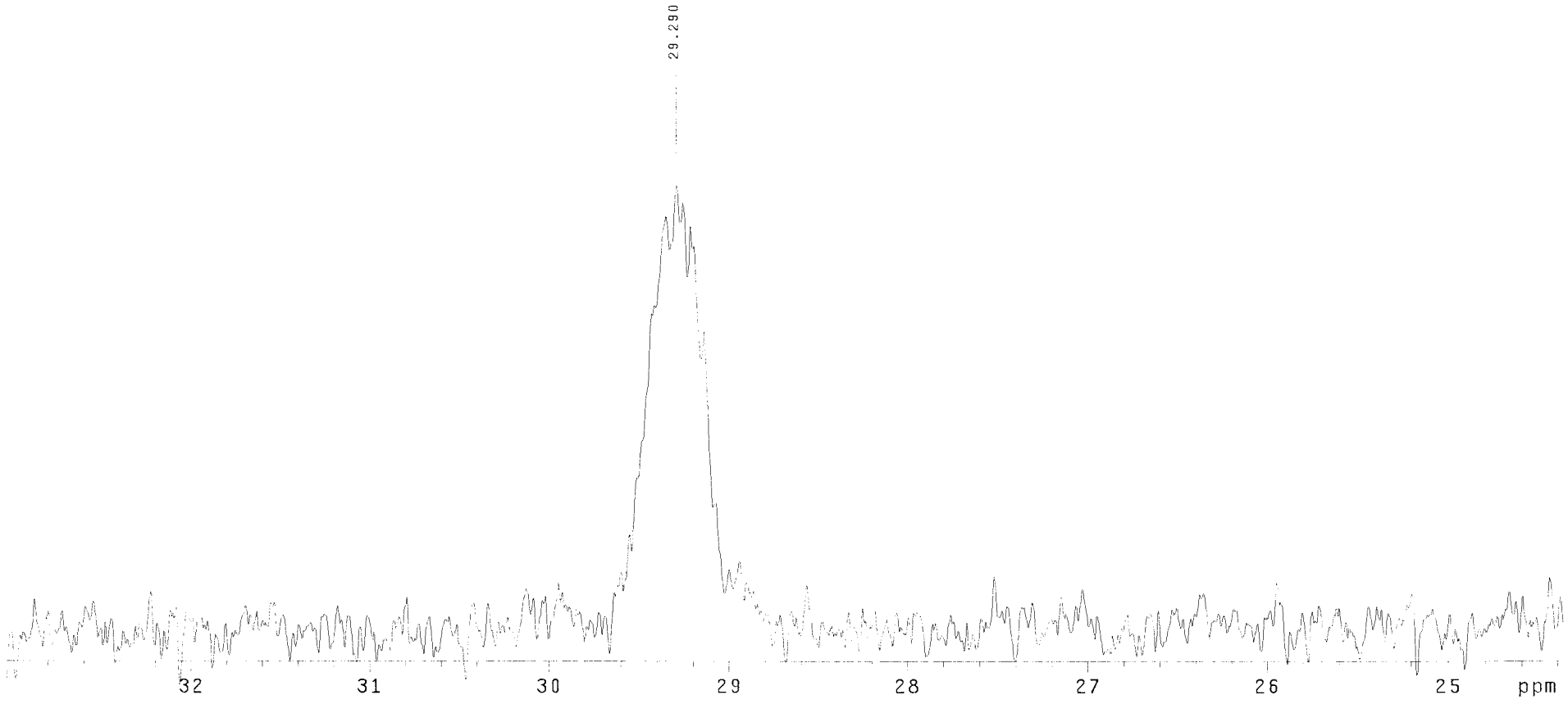

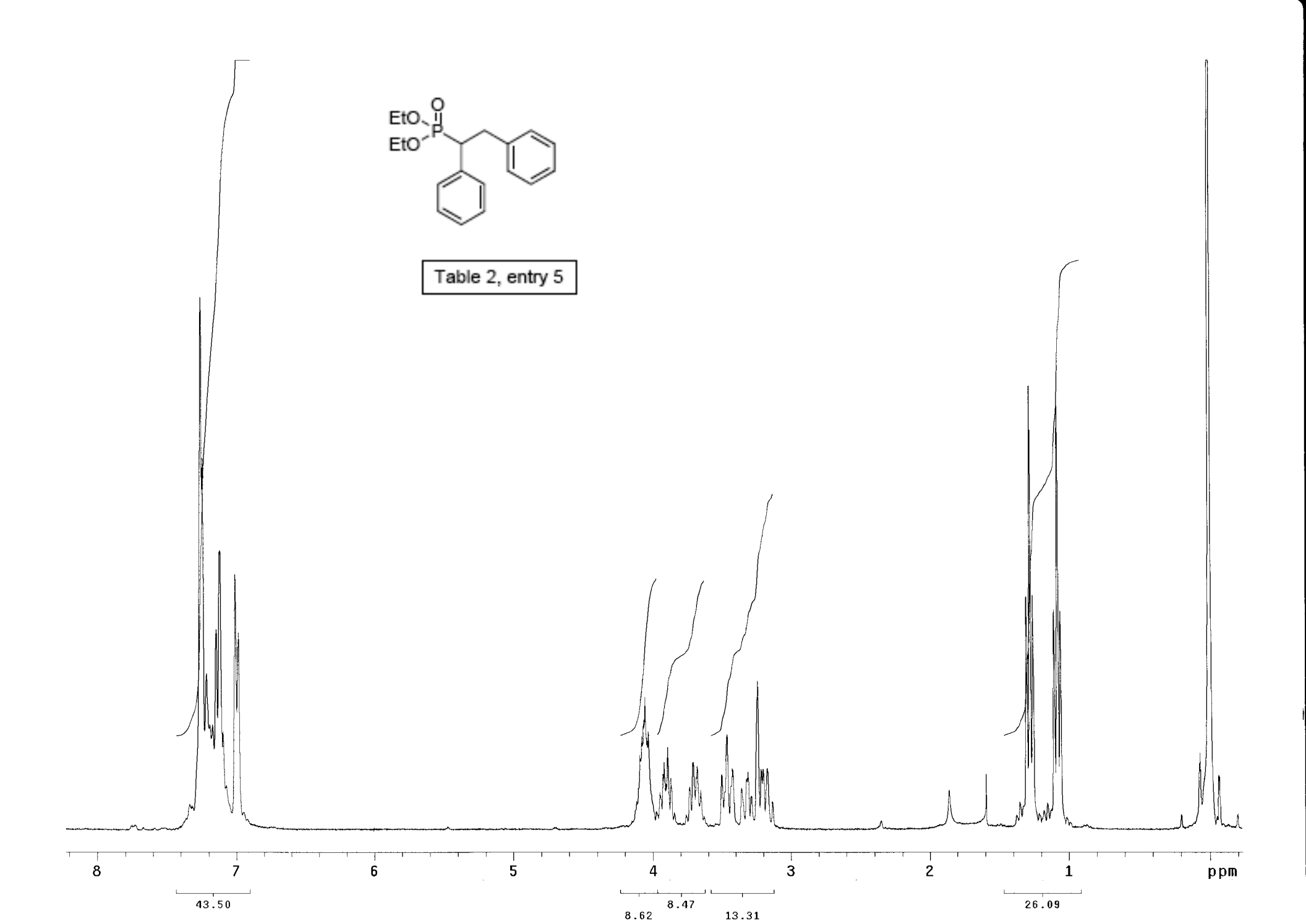
EtO
EtO
O
P
Table 2, entry 5
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ppm
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8.47
13.31
26.09

INDEX	FREQUENCY	PPM	HEIGHT
1	10516.399	139.386	15.3
2	10500.277	139.173	14.2
3	10242.892	135.761	15.5
4	10236.559	135.677	13.3
5	9788.296	129.736	70.4
6	9781.386	129.644	71.7
7	9736.185	129.045	139.8
8	9703.077	128.606	81.7
9	9700.774	128.576	85.4
10	9684.939	128.366	138.0
11	9611.812	127.397	35.9
12	9608.933	127.359	38.7
13	9537.245	126.408	68.5
14	5873.984	77.855	14.1
15	5842.027	77.431	17.0
16	5809.782	77.004	16.1
17	4754.911	63.022	23.1
18	4748.001	62.931	26.6
19	4687.254	62.126	22.3
20	4679.768	62.026	27.1
21	3599.561	47.709	35.2
22	3463.096	45.901	33.6
23	2756.297	36.532	32.8
24	2753.706	36.498	42.4
25	1256.041	16.648	22.5
26	1249.996	16.568	26.7
27	1243.374	16.480	27.0
28	1237.616	16.404	29.3

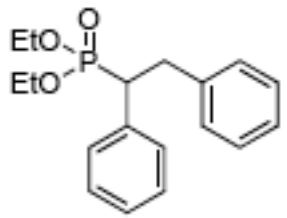
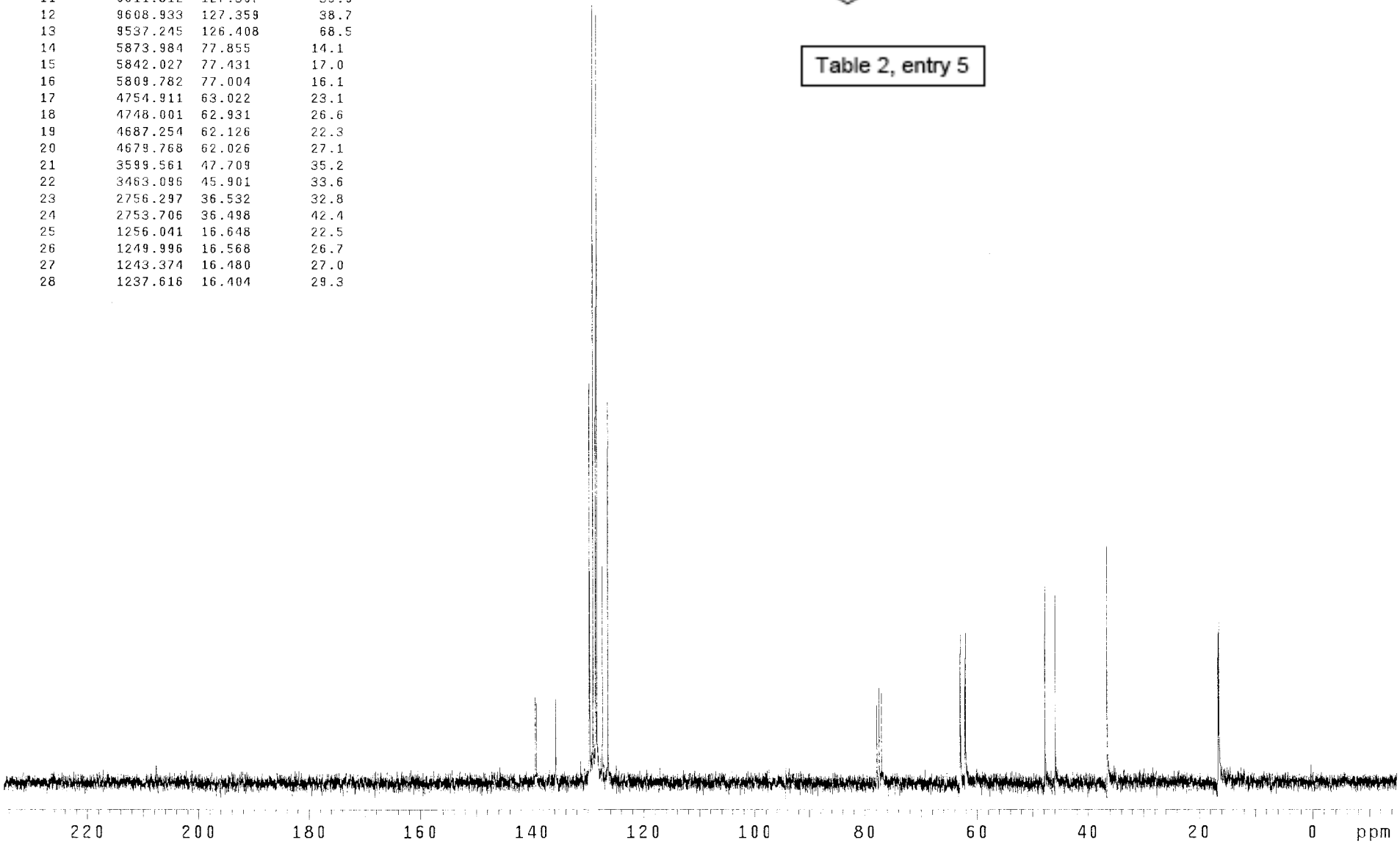
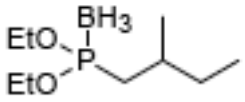


Table 2, entry 5

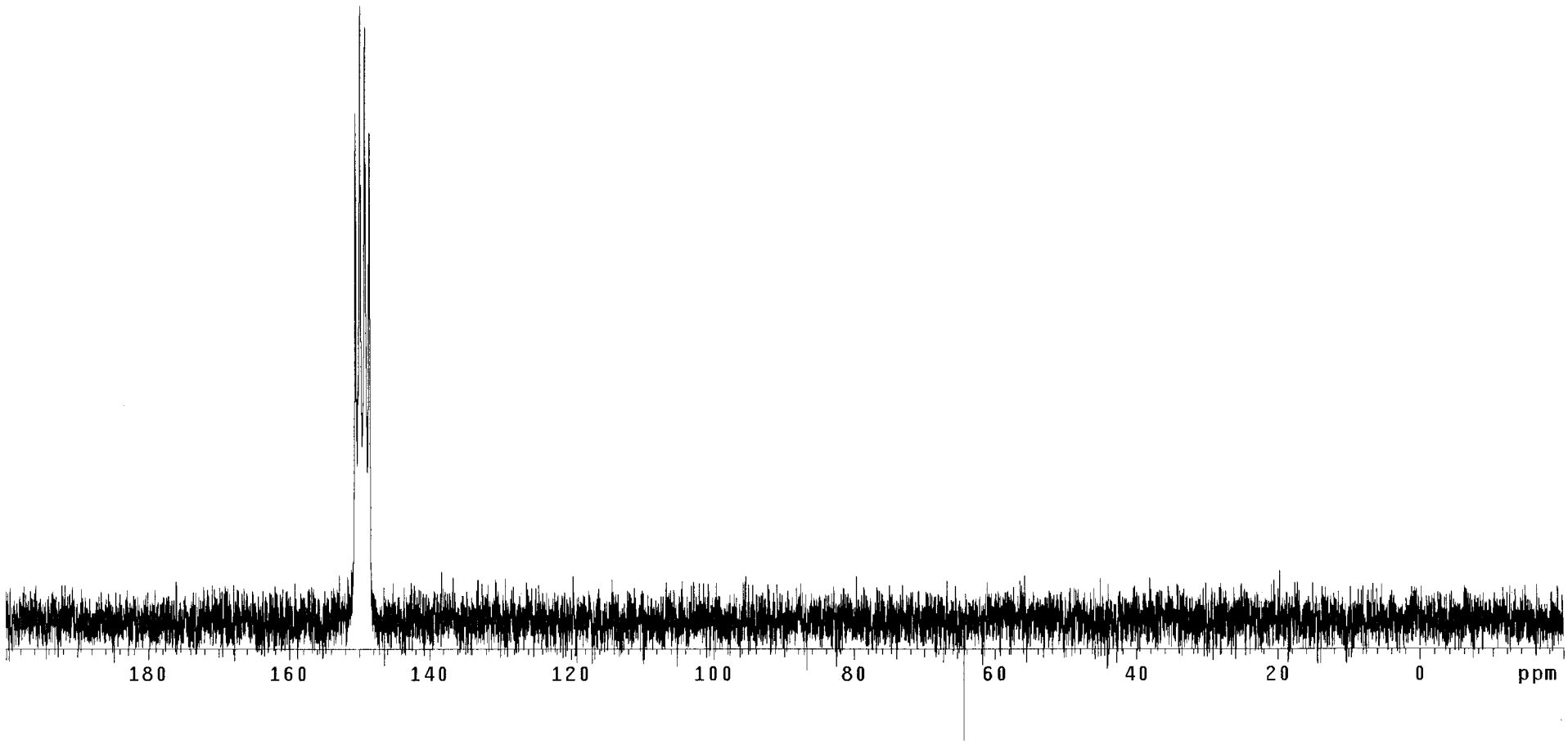


INDEX	FREQUENCY	PPM	HEIGHT
1	18285.649	150.545	81.6
2	18207.315	149.900	99.0
3	18122.861	149.205	95.5
4	18044.527	148.560	78.4
5	7819.915	64.381	-19.6



$^{31}P/^{1}H$ decoupled

Table 2, entry 6



INDEX	FREQUENCY	PPM	HEIGHT
1	18209.355	149.917	51.8
2	18127.757	149.245	50.2
3	18061.255	148.698	38.1

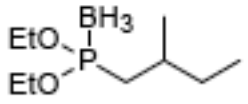
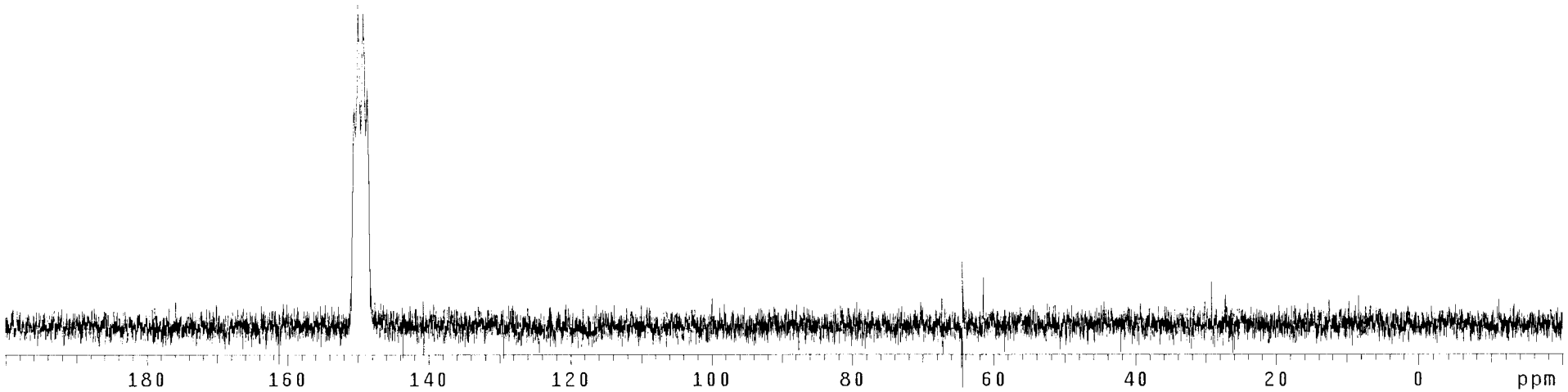
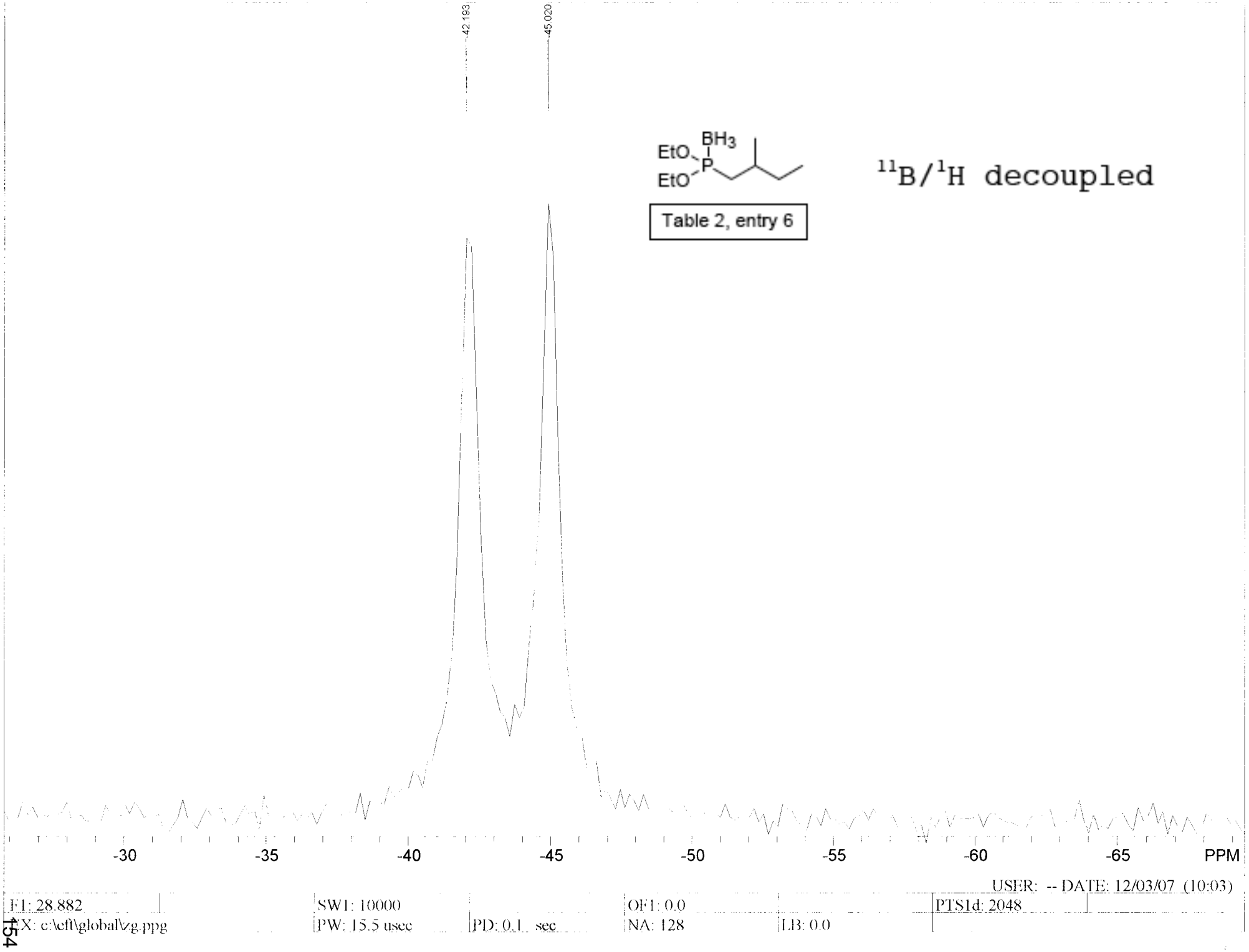


Table 2, entry 6

$^{31}P/^{1}H$ coupled




42.193
45.020
EtO
BH3
P
EtO
Table 2, entry 6
11B/1H decoupled
-30
-35
-40
-45
-50
-55
-60
-65
PPM
USER: -- DATE: 12/03/07 (10:03)
F1: 28.882
SW1: 10000
OF1: 0.0
PTS1d: 2048
EX: c:\cft\global\zg.ppg
PW: 15.5 usec
PD: 0.1 sec
NA: 128
LB: 0.0

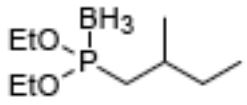


Table 2, entry 6

$^{11}B/^{1}H$ coupled

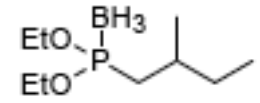


Table 2, entry 6

INDEX	FREQUENCY	PPM	HEIGHT
1	5867.363	77.767	19.2
2	5835.118	77.340	19.6
3	5803.161	76.916	17.4
4	4759.517	63.084	91.7
5	4755.486	63.030	93.4
6	2814.165	37.299	52.4
7	2760.328	36.586	52.2
8	2359.857	31.278	80.6
9	2349.204	31.137	82.5
10	2204.965	29.225	115.2
11	1571.870	20.834	82.7
12	1566.112	20.758	86.2
13	1264.391	16.758	117.0
14	1258.633	16.682	126.0
15	842.327	11.164	119.1

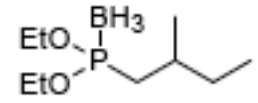
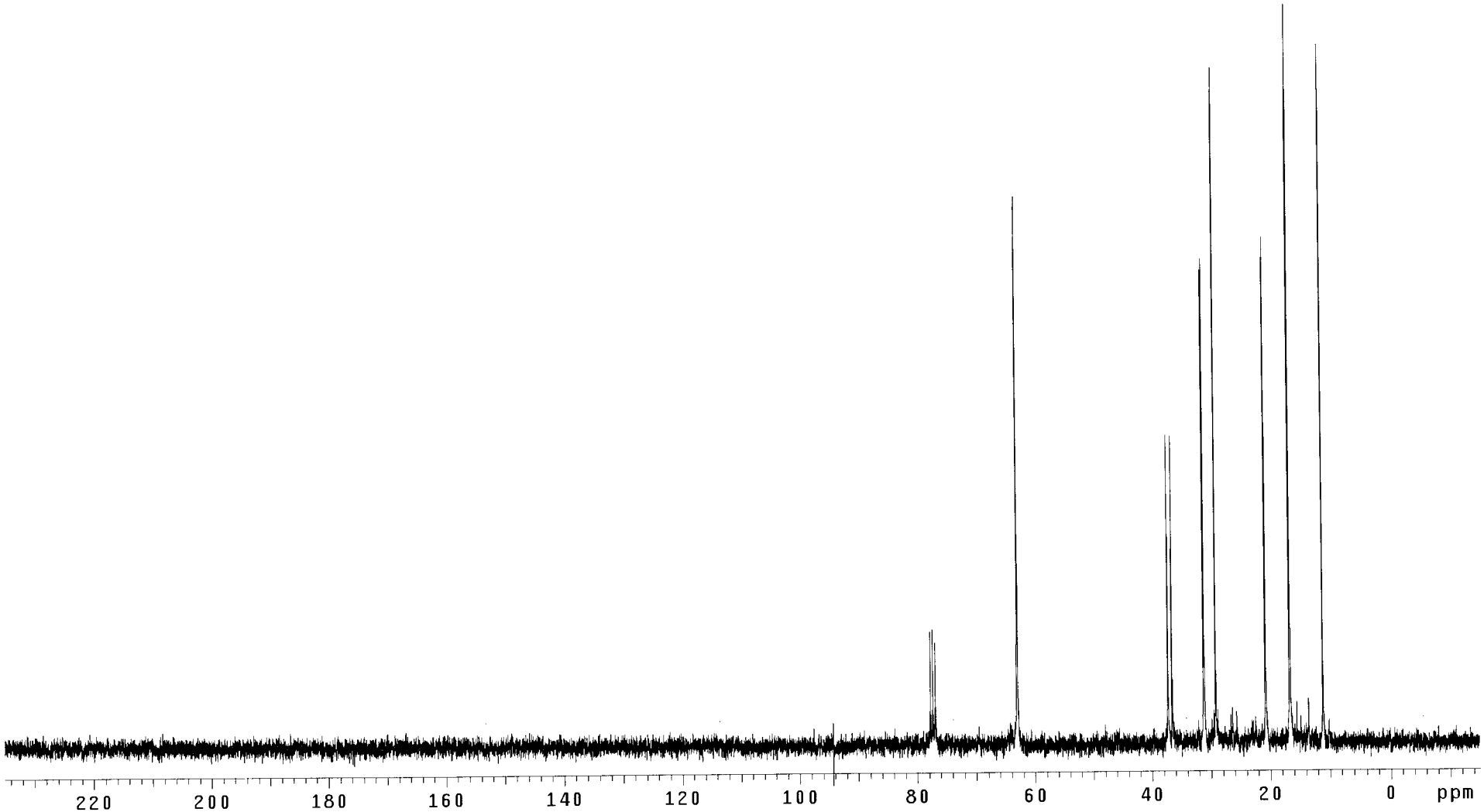
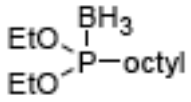


Table 2, entry 6

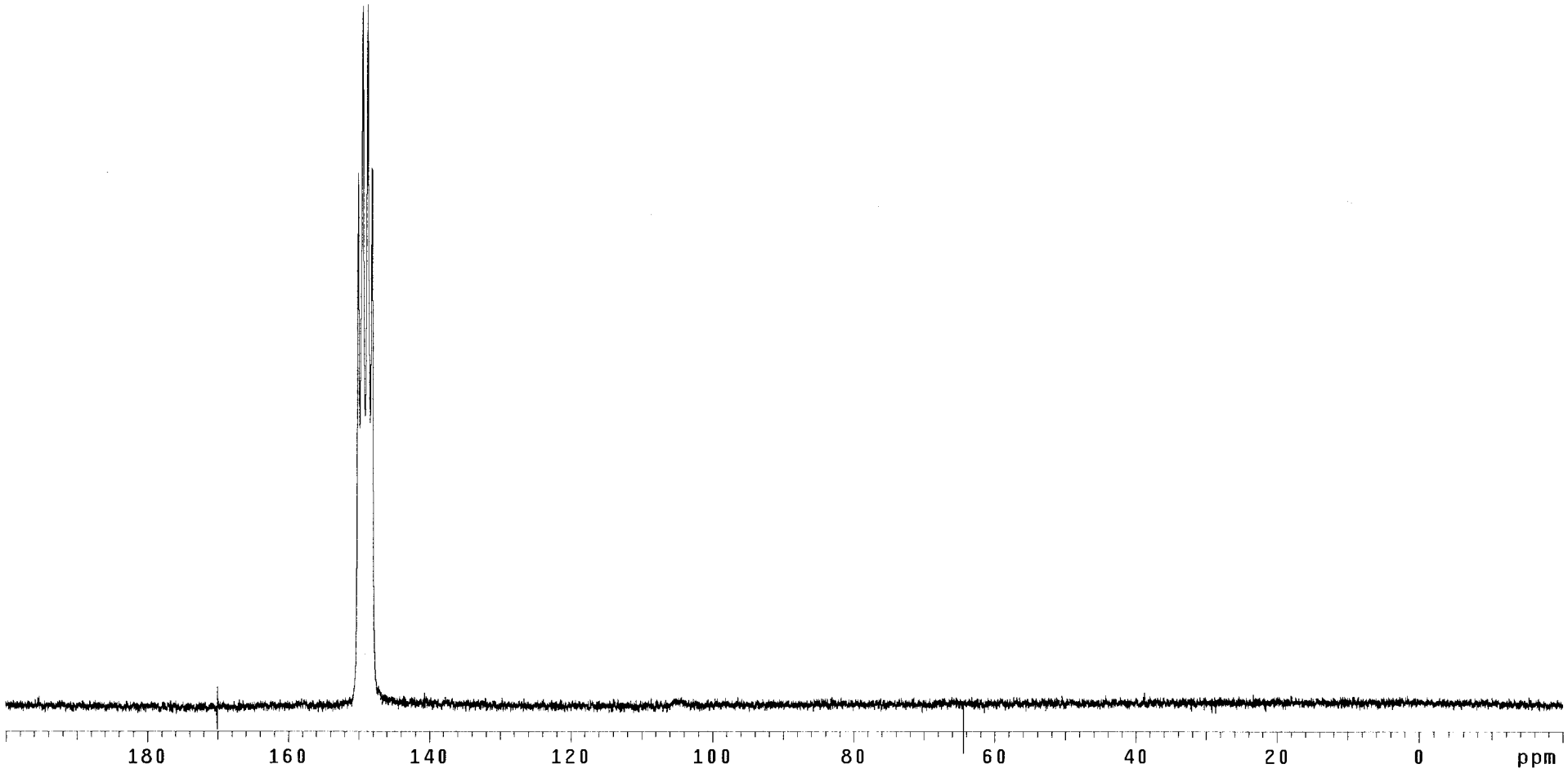


INDEX	FREQUENCY	PPM	HEIGHT
1	18228.938	150.078	85.3
2	18151.828	149.444	112.3
3	18067.783	148.752	112.6
4	17990.673	148.117	86.1



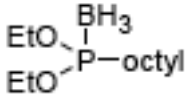
$^{31}P/^{1}H$ decoupled

Table 2, entry 7



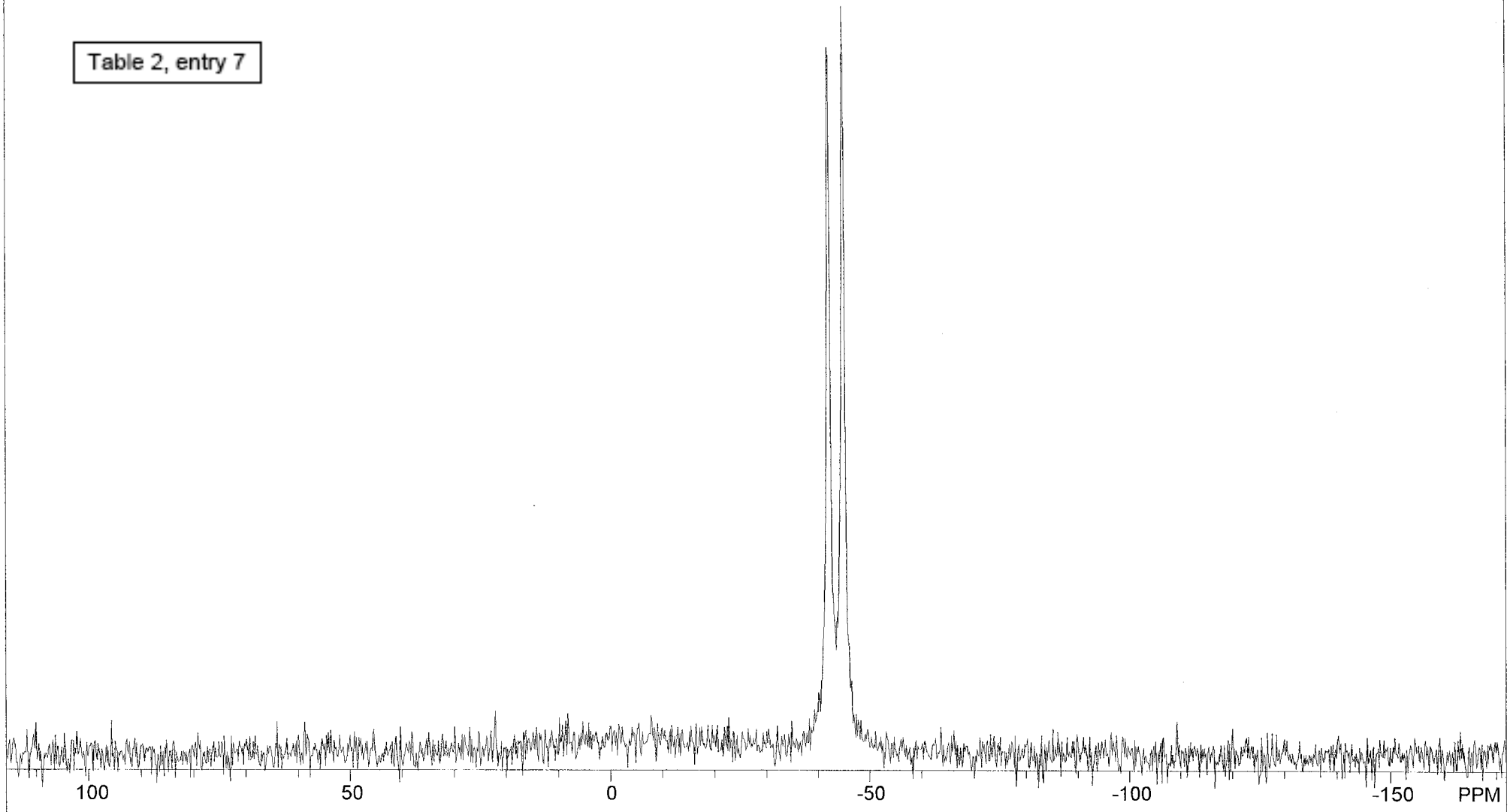
Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	1273	19972	90.70	-1218.60	-42.193
2	1290	22082	100.28	-1300.26	-45.020



$^{11}B/^{1}H$ decoupled

Table 2, entry 7

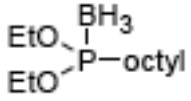


USER: -- DATE: 12/03/07 (10:03)

F1: 28.882		SW1: 10000		OF1: 0.0		PTS1d: 2048	
F2: c:\eft\global\zg.ppg		PW: 15.5 usec	PD: 0.1 sec	NA: 128		LB: 0.0	

Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	1245	2978	23.49	-1080.60	-37.415
2	1264	10059	79.34	-1172.82	-40.607
3	1283	11927	94.08	-1267.29	-43.879
4	1300	9931	78.33	-1349.33	-46.719
5	1320	3336	26.31	-1446.26	-50.075



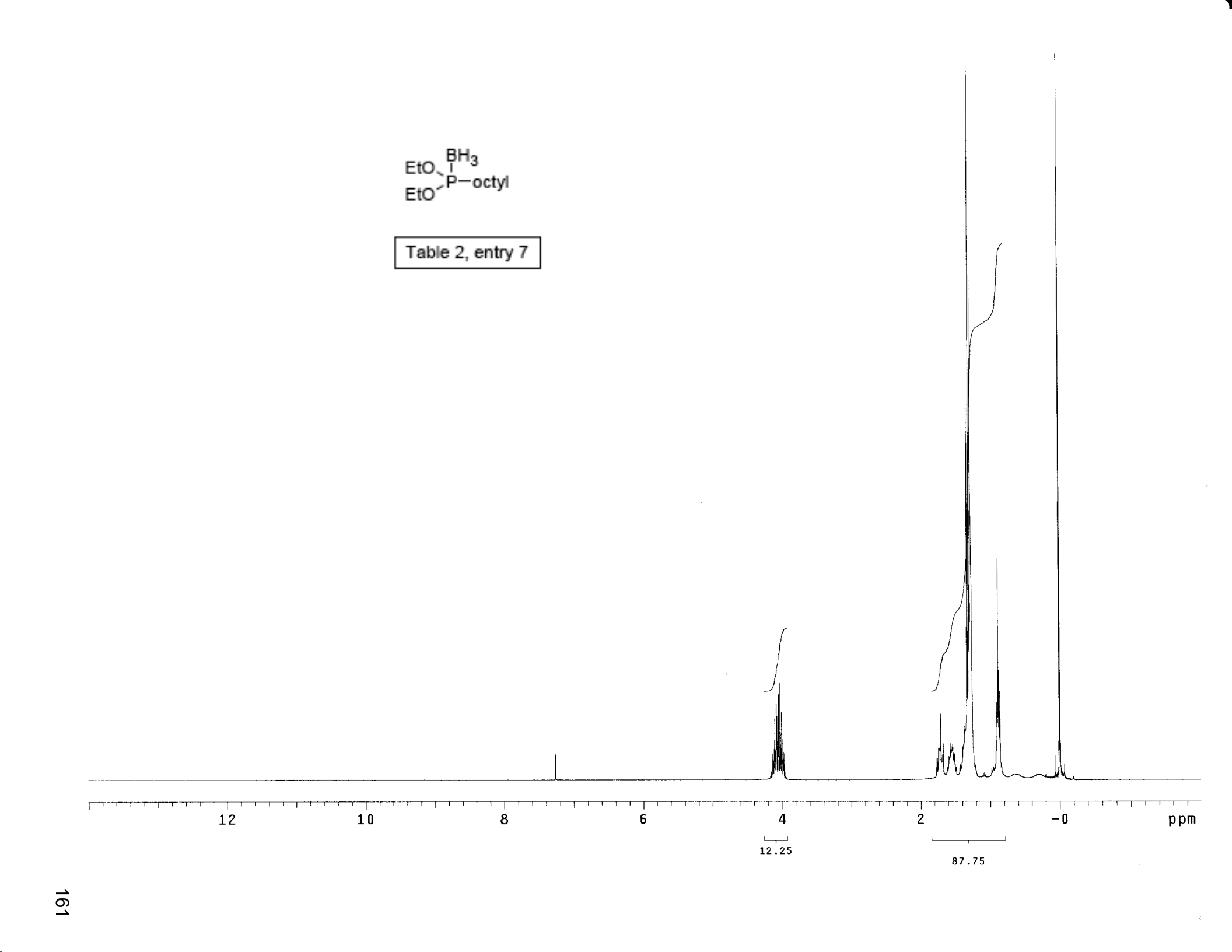
$^{11}B/^{1}H$ coupled

Table 2, entry 7

USER: -- DATE: 12/03/07 (10:07)

F1: 28.882		SW1: 10000		OF1: 0.0		PTS1d: 2048	
EX: c:\left\global\zg.ppg		PW: 15.5 usec	PD: 0.1 sec	NA: 128	LB: 0.0		

190


EtO
BH3
P—octyl
EtO
Table 2, entry 7
12
10
8
6
4
2
-0
ppm
12.25
87.75

INDEX	FREQUENCY	PPM	HEIGHT
1	5862.180	77.698	18.3
2	5830.223	77.275	17.8
3	5797.978	76.847	16.8
4	4769.882	63.221	41.5
5	4766.427	63.175	43.0
6	2415.709	32.018	81.8
7	2340.279	31.018	39.8
8	2326.172	30.832	40.4
9	2292.200	30.381	26.7
10	2236.059	29.637	27.8
11	2208.708	29.275	157.3
12	1722.730	22.833	68.0
13	1642.406	21.769	84.6
14	1268.133	16.808	51.1
15	1262.375	16.732	53.9
16	1076.967	14.274	52.5

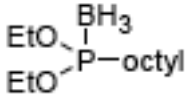
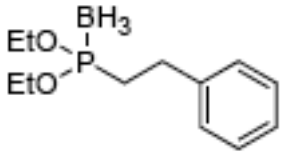


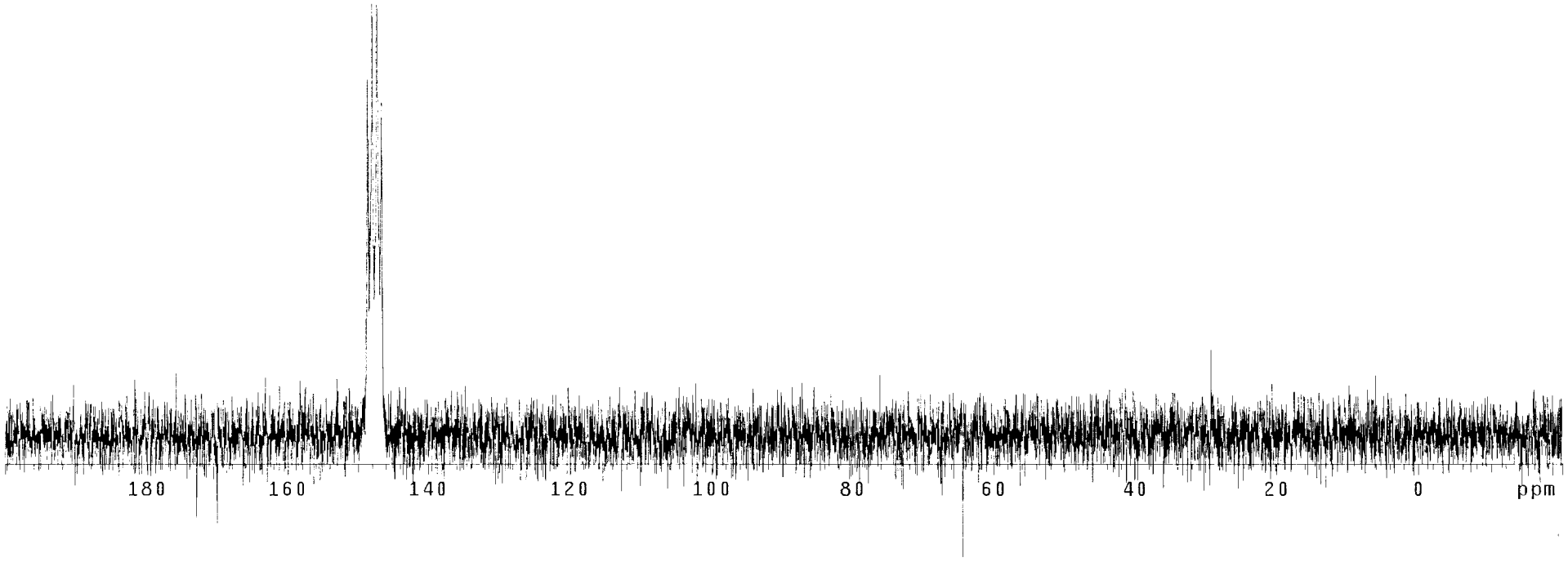
Table 2, entry 7

INDEX	FREQUENCY	PPM	HEIGHT
1	18053.911	148.637	57.4
2	17974.761	147.986	69.5
3	17896.019	147.337	69.2
4	17814.829	146.669	53.4
5	7819.507	64.378	-35.1



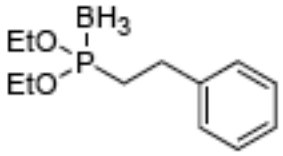
$^{31}P/^{1}H$ decoupled

Table 2, entry 8



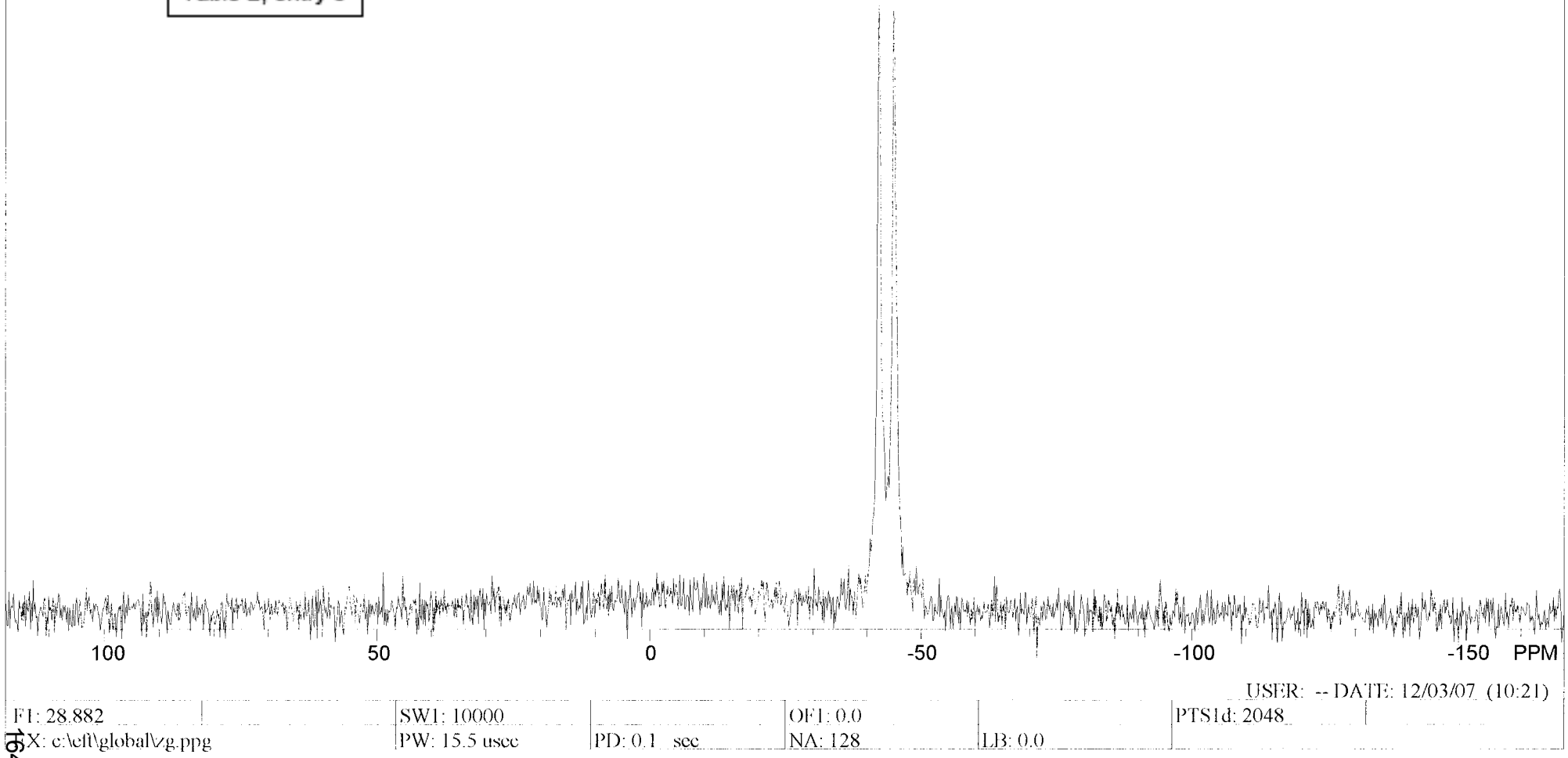
Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	1274	15788	96.07	-1221.49	-42.293
2	1290	15058	91.63	-1301.70	-45.070



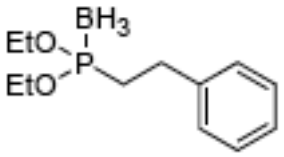
$^{11}B/^{1}H$ decoupled

Table 2, entry 8



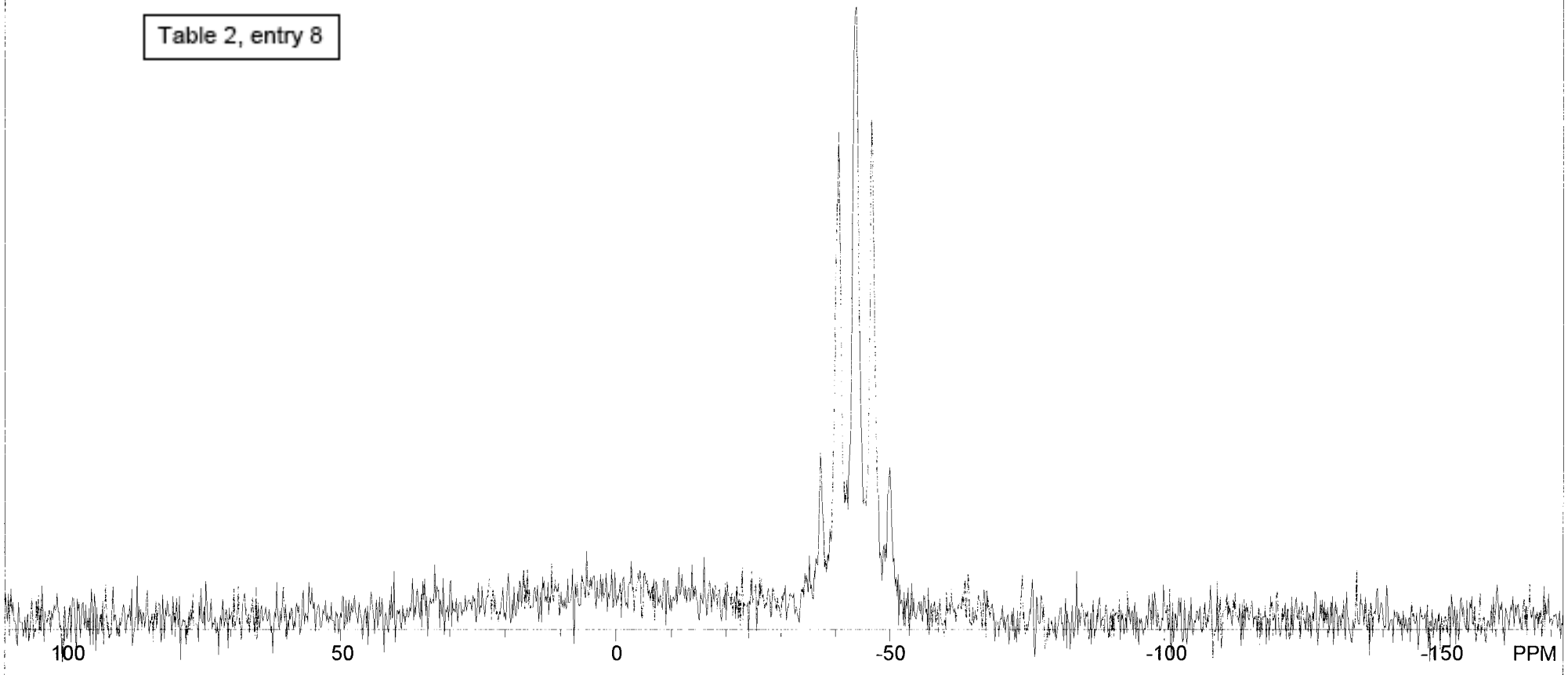
Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	1245	2220	24.10	-1078.27	-37.334
2	1264	6739	73.14	-1170.58	-40.530
3	1283	8461	91.83	-1266.51	-43.851
4	1300	6953	75.46	-1350.43	-46.757
5	1320	2035	22.09	-1445.99	-50.066



$^{11}B/^{1}H$ coupled

Table 2, entry 8



USER: -- DATE: 12/03/07 (10:18)

F1: 28.882 | SW1: 10000 | OF1: 0.0 | PTS1d: 2048

EX: c:\eft\global\zg.ppg | PW: 15.5 usec | PD: 0.1 sec | NA: 128 | LB: 0.0

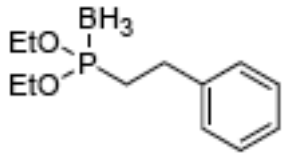


Table 2, entry 8

INDEX	FREQUENCY	PPM	HEIGHT
1	10663.805	141.340	12.0
2	10649.410	141.149	11.2
3	9718.911	128.816	125.1
4	9682.060	128.328	126.0
5	9548.761	126.561	72.2
6	5875.712	77.878	12.4
7	5843.467	77.450	13.1
8	5811.510	77.027	12.9
9	4884.466	64.740	5.6
10	4788.883	63.473	49.2
11	4784.277	63.412	50.8
12	2430.968	32.220	23.1
13	2376.843	31.503	23.2
14	2113.988	28.019	53.0
15	1272.452	16.865	55.2
16	1266.694	16.789	61.0

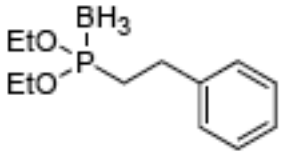
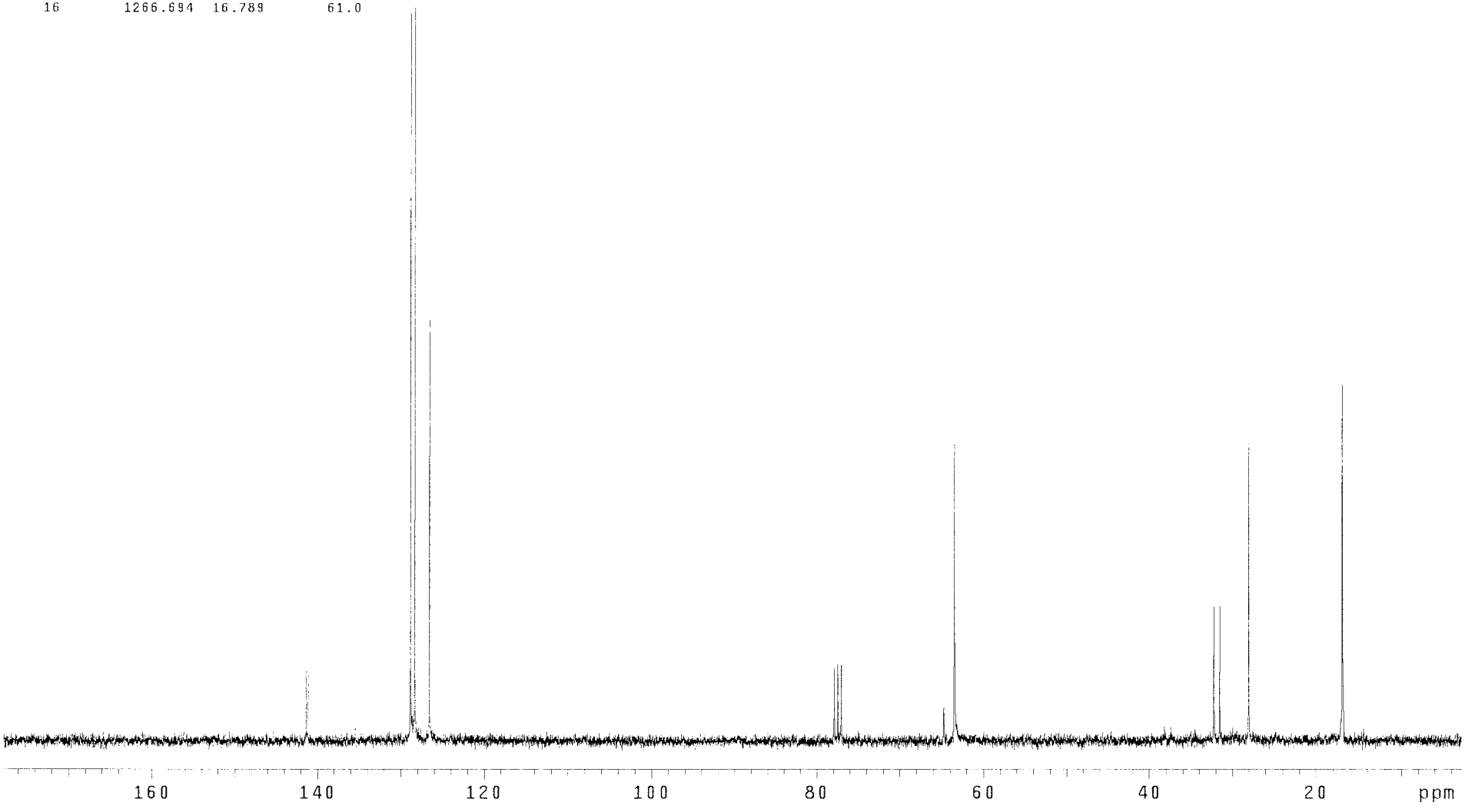
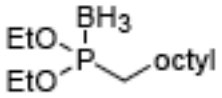


Table 2, entry 8

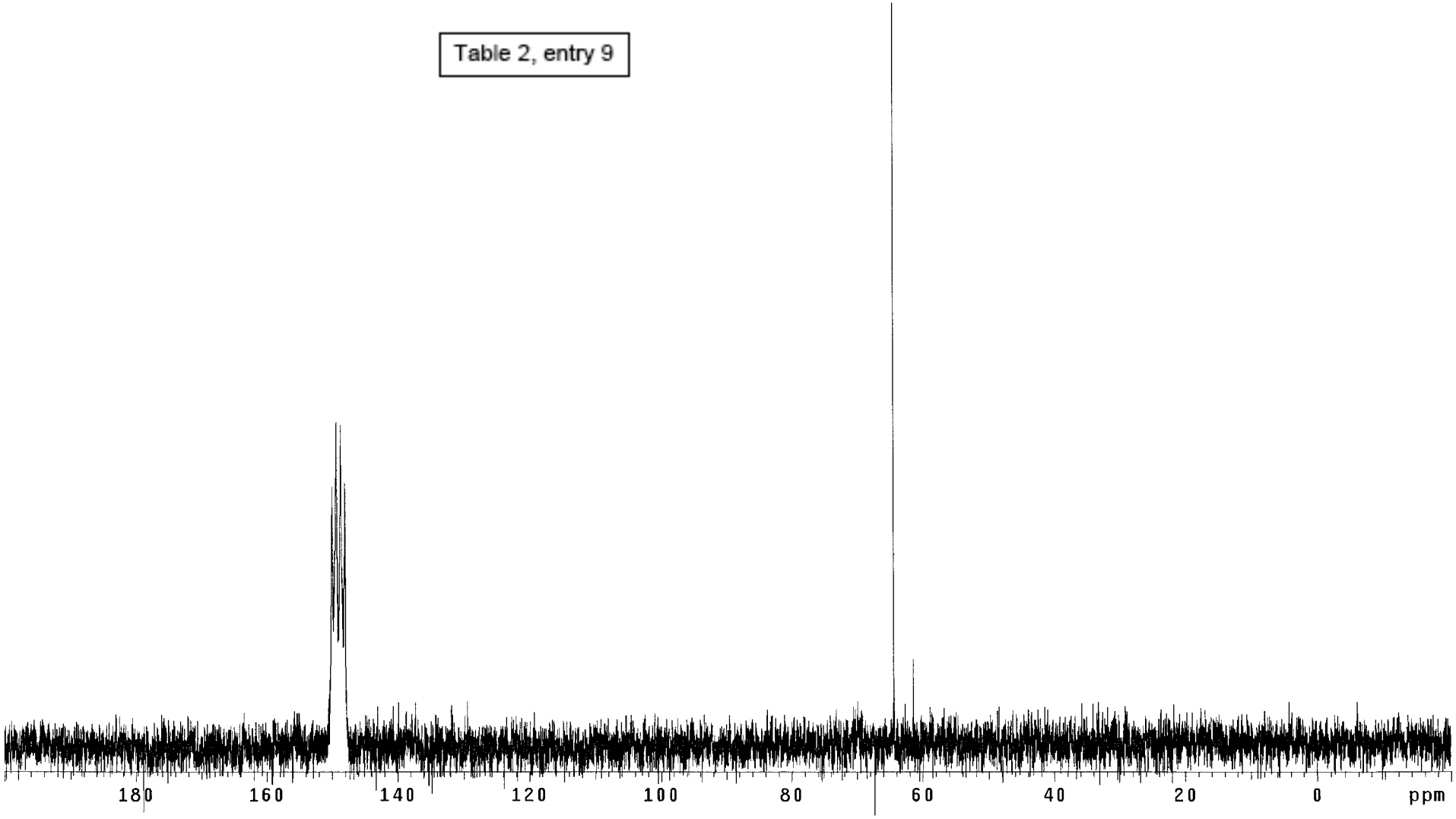


INDEX	FREQUENCY	PPM	HEIGHT
1	18223.226	150.031	44.6
2	18142.445	149.366	55.8
3	18059.623	148.684	55.3
4	17983.737	148.060	45.3
5	7819.915	64.381	128.9
6	7462.516	61.439	14.7



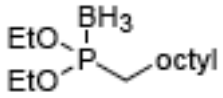
$^{31}P/^{1}H$ decoupled

Table 2, entry 9



Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	1273	19972	90.70	-1218.60	-42.193
2	1290	22082	100.28	-1300.26	-45.020



$^{11}B/^{1}H$ decoupled

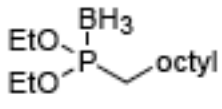
Table 2, entry 9

USER: -- DATE: 12/03/07 (10:03)

F1: 28.882		SW1: 10000		OF1: 0.0		PTS1d: 2048
DS: c:\eft\global\zg.ppg		PW: 15.5 usec	PD: 0.1 sec	NA: 128	LB: 0.0	

Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	1245	2978	23.49	-1080.60	-37.415
2	1264	10059	79.34	-1172.82	-40.607
3	1283	11927	94.08	-1267.29	-43.879
4	1300	9931	78.33	-1349.33	-46.719
5	1320	3336	26.31	-1446.26	-50.075



$^{11}B/^{1}H$ coupled

Table 2, entry 9

USER: -- DATE: 12/03/07 (10:07)

F1: 28.882		SW1: 10000		OF1: 0.0		PTS1d: 2048	
EXP: c:\eft\global\zg.ppg		PW: 15.5 usec	PD: 0.1 scc	NA: 128	LB: 0.0		

170

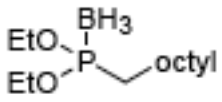


Table 2, entry 9

INDEX	FREQUENCY	PPM	HEIGHT
1	4771.897	63.248	65.3
2	4767.290	63.187	54.5
3	2426.074	32.156	13.8
4	2419.452	32.068	97.7
5	2341.431	31.034	51.6
6	2327.324	30.847	51.6
7	2292.200	30.381	31.1
8	2258.515	29.935	27.2
9	2255.348	29.893	12.7
10	2236.059	29.637	43.8
11	2232.316	29.588	113.2
12	2224.543	29.484	102.9
13	2213.027	29.332	86.1
14	1726.761	22.887	85.1
15	1643.557	21.784	101.7
16	1269.861	16.831	70.1
17	1264.391	16.758	70.9
18	1080.709	14.324	72.0

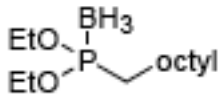
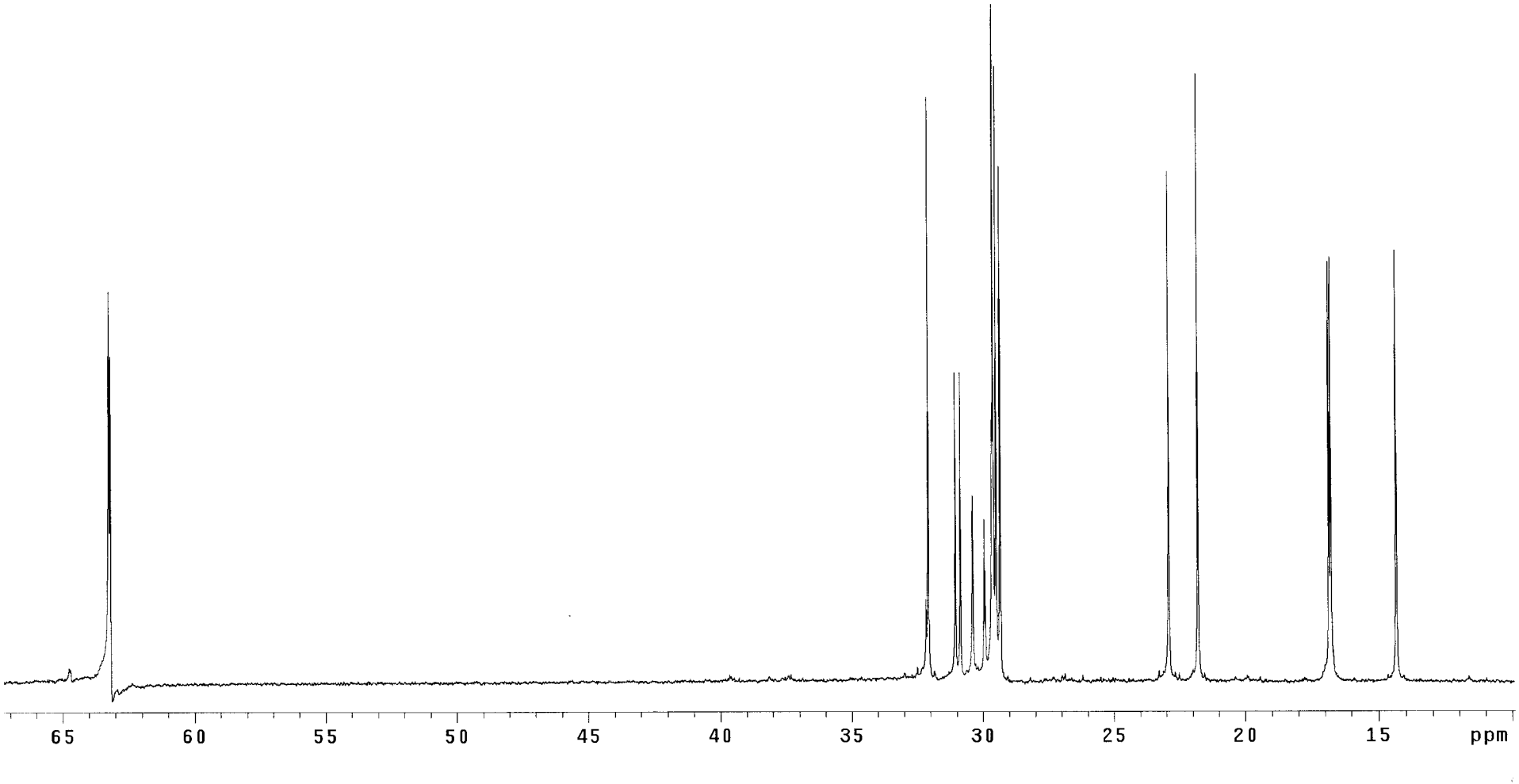
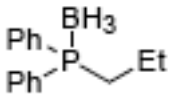


Table 2, entry 9

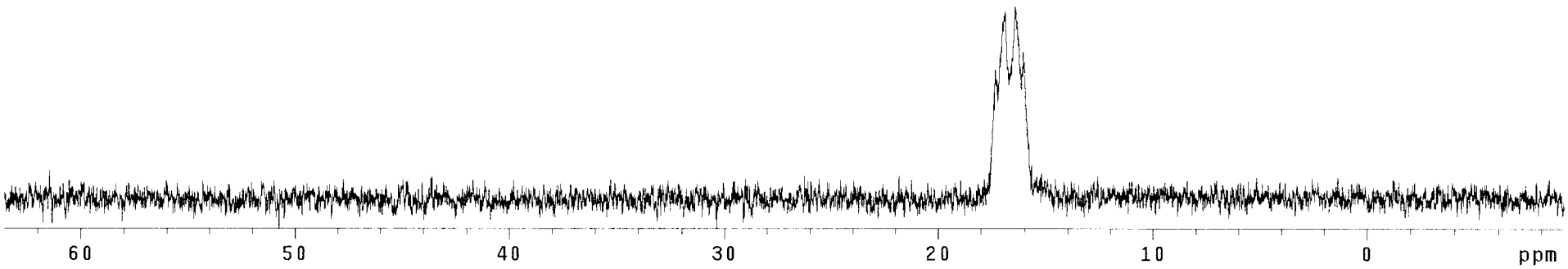


INDEX	FREQUENCY	PPM	HEIGHT
1	2052.174	16.895	30.1
2	1993.424	16.412	30.9

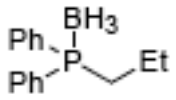


$^{31}P/^{1}H$ decoupled

Table 2, entry 10

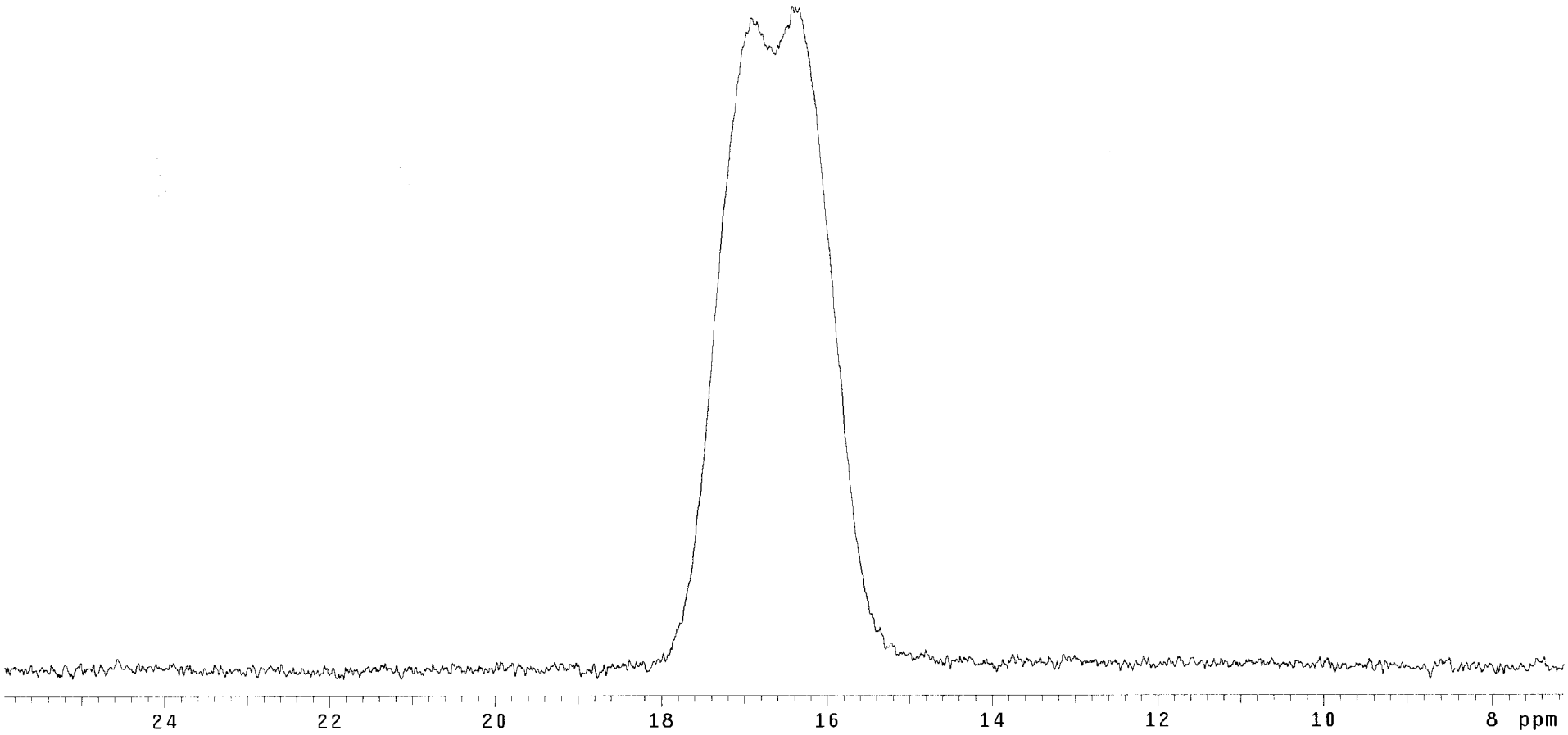


INDEX	FREQUENCY	PPM	HEIGHT
1	2052.990	16.902	104.5
2	1987.304	16.361	106.3



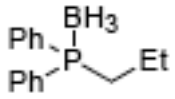
$^{31}P/^{1}H$ coupled

Table 2, entry 10



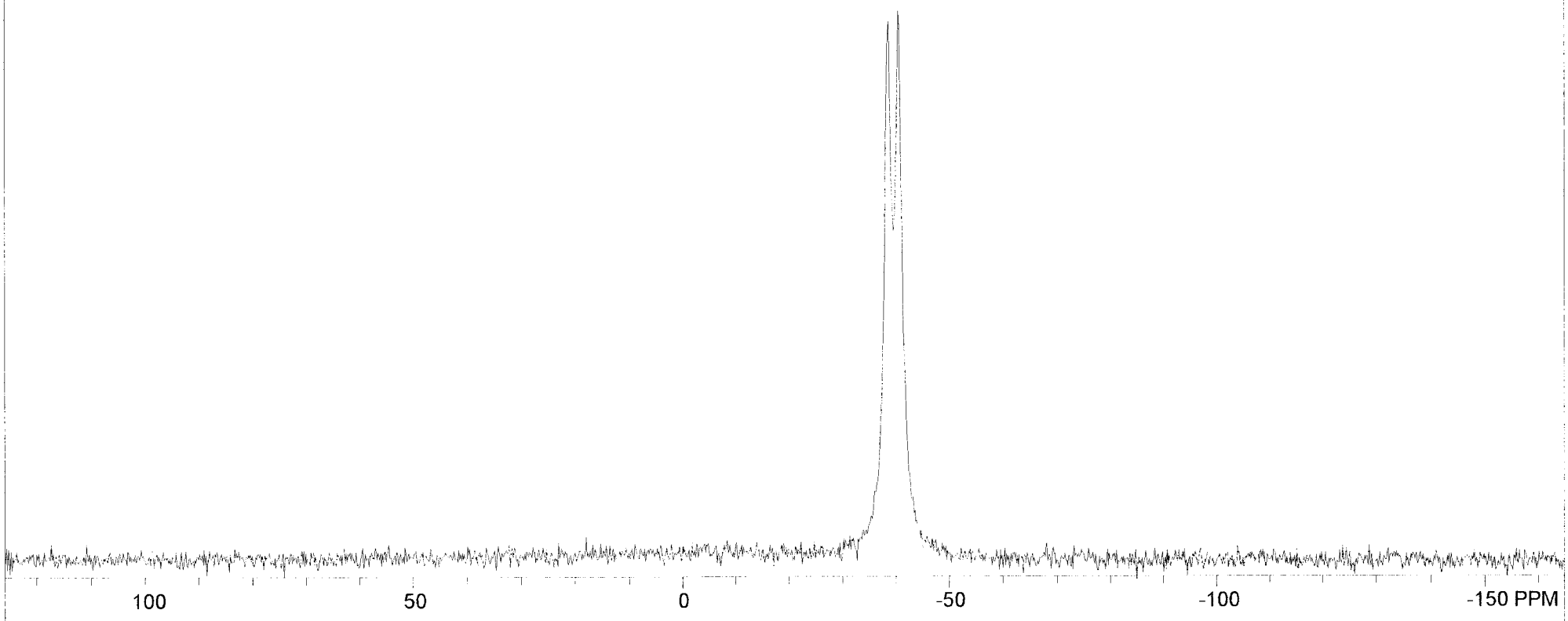
Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	1253	28414	94.62	-1119.82	-38.773
2	1264	29071	96.80	-1172.80	-40.607



$^{11}B/^{1}H$ decoupled

Table 2, entry 10



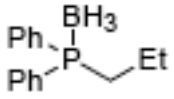
USER: -- DATE: 12/23/07 (15:46)

F1: 28.882 | SW1: 10000 | OF1: 0.0 | PTS1d: 2048

EX: c:\cft\global\zg.ppg | PW: 15.5 usec | PD: 0.1 sec | NA: 128 | LB: 0.0

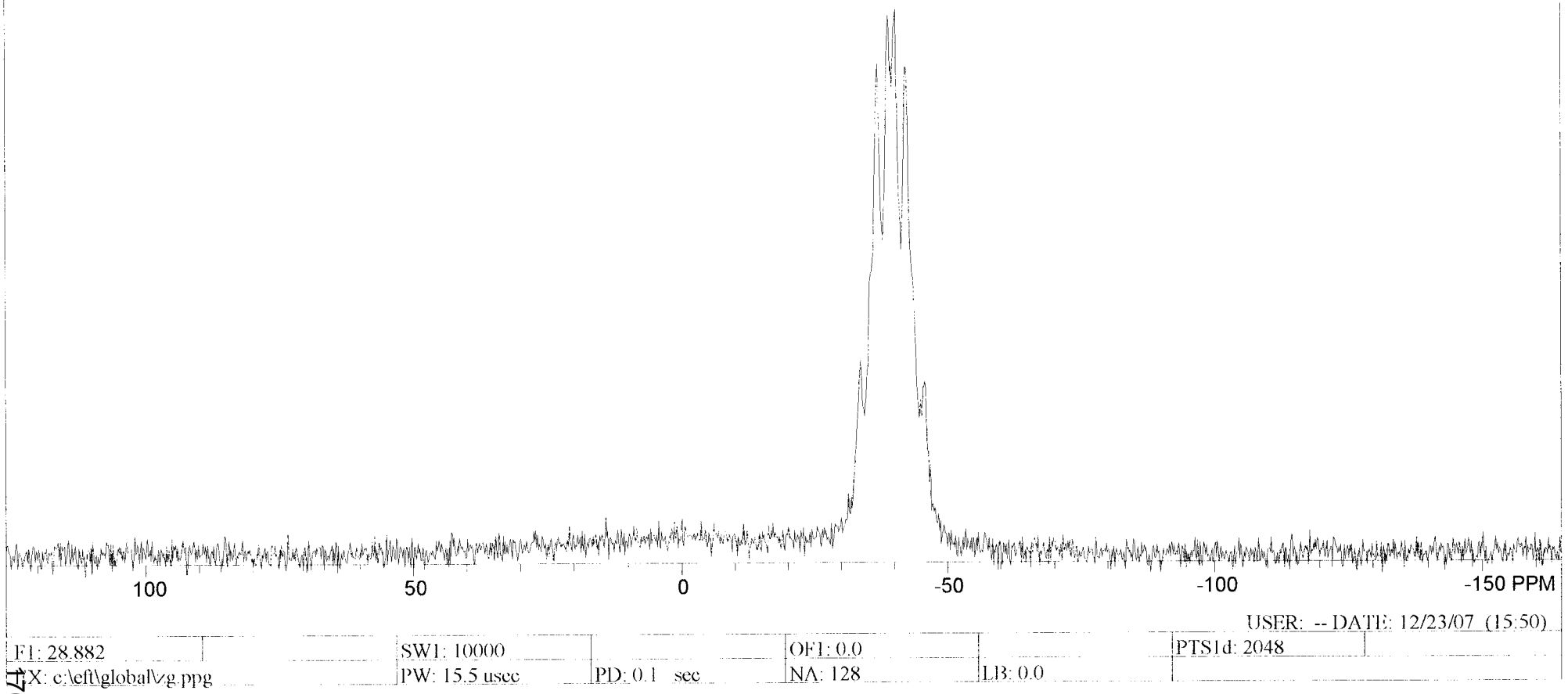
Interpolated Peak Listing

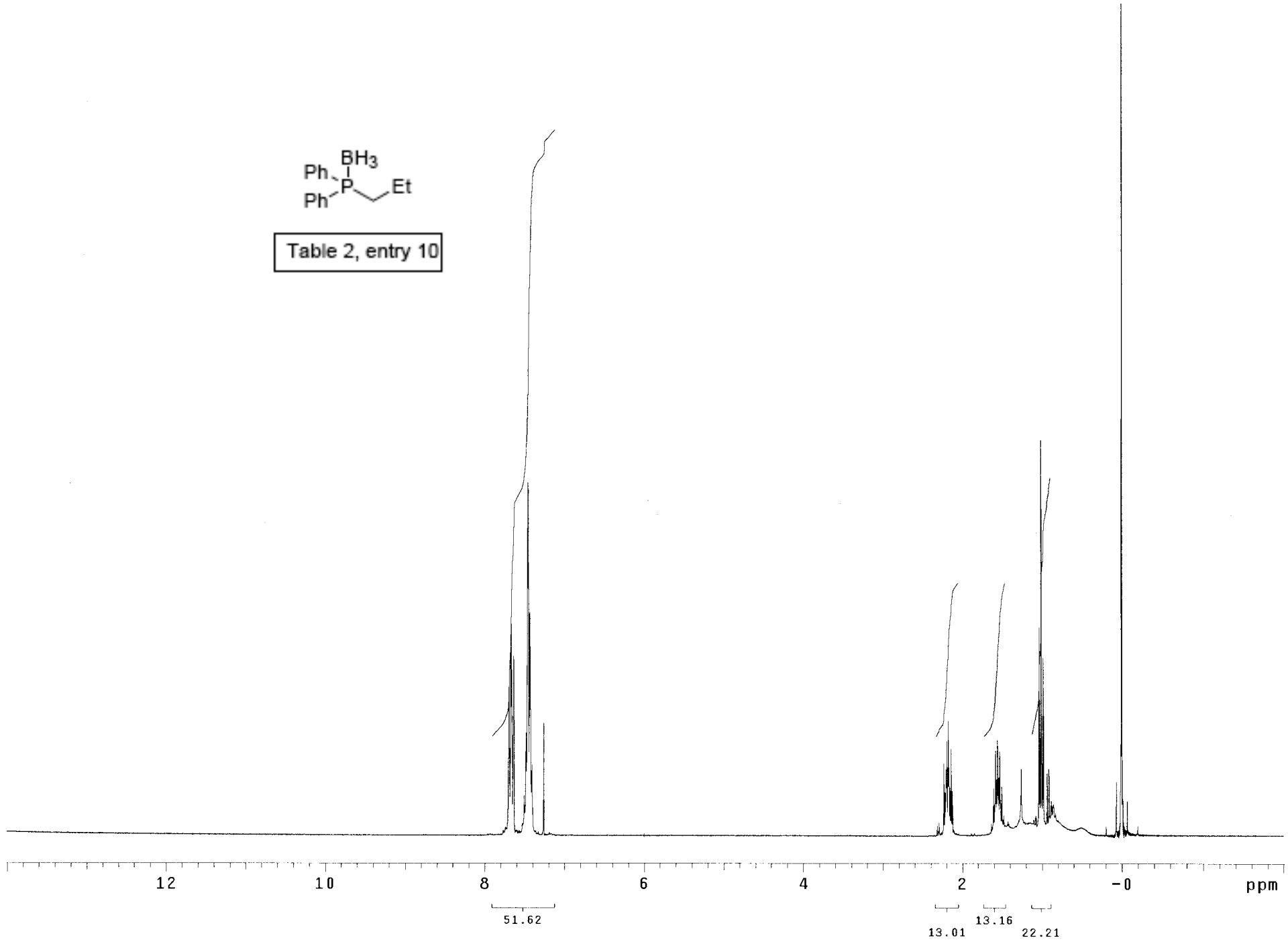
PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	1243	12712	84.65	-1069.00	-37.013
2	1255	14011	93.30	-1128.04	-39.057
3	1263	14238	94.81	-1168.91	-40.472
4	1274	12486	83.14	-1223.69	-42.369



$^{11}B/^{1}H$ coupled

Table 2, entry 10




Ph
BH3
P
Et
Ph
Table 2, entry 10
12
10
8
6
4
2
-0
ppm
51.62
13.01
13.16
22.21

INDEX	FREQUENCY	PPM	HEIGHT
1	9992.130	132.438	115.5
2	9982.917	132.315	111.1
3	9911.518	131.369	74.5
4	9908.926	131.335	73.6
5	9830.617	130.297	12.6
6	9775.628	129.568	15.2
7	9740.792	129.106	120.9
8	9731.003	128.976	119.4
9	5869.954	77.801	20.0
10	5837.709	77.374	20.6
11	5806.040	76.954	20.5
12	2133.854	28.282	27.0
13	2097.002	27.794	26.4
14	1285.407	17.037	59.0
15	1222.645	16.205	30.9
16	1207.386	16.003	31.2

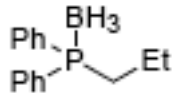
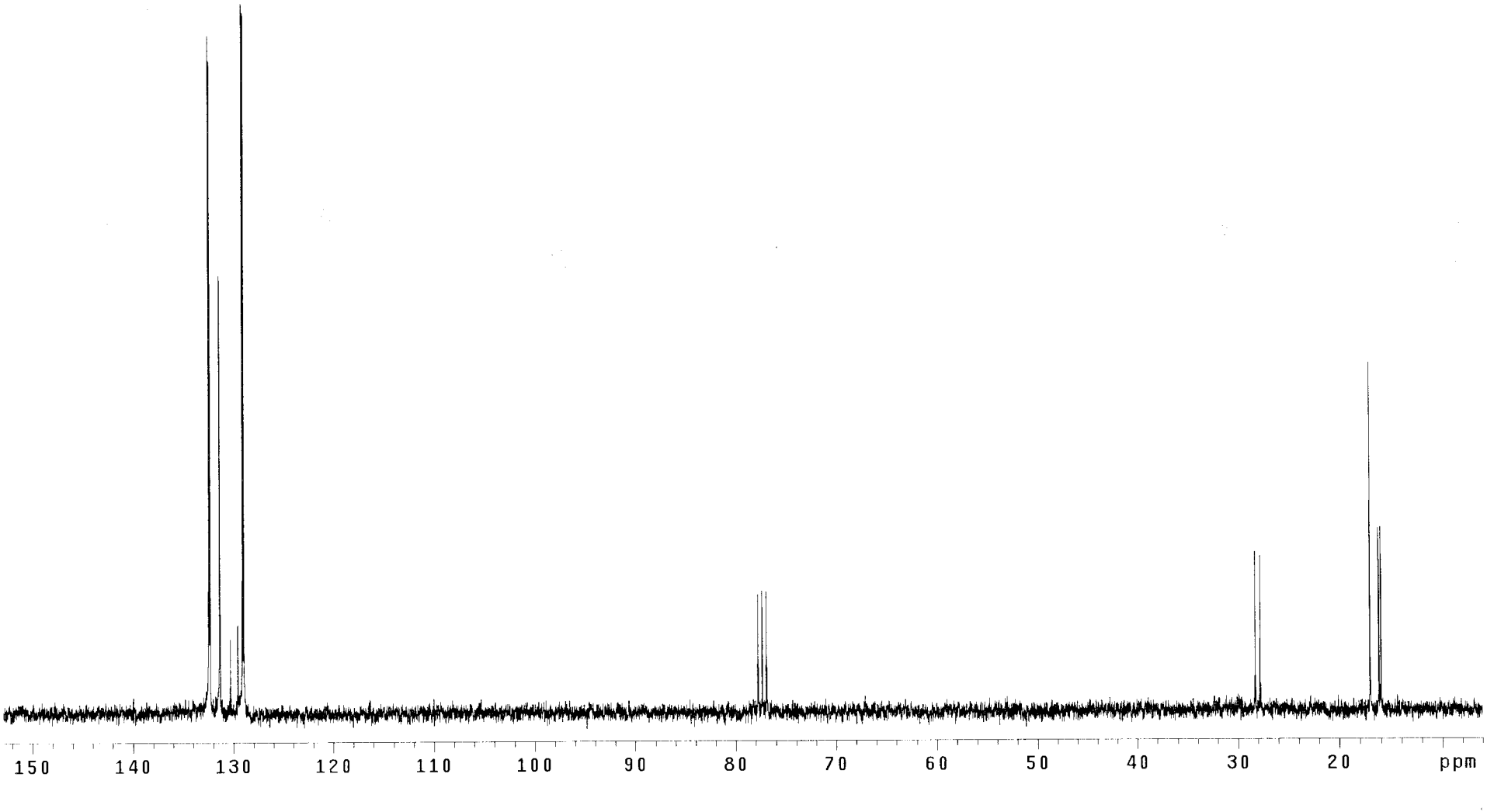
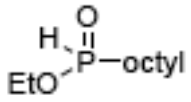


Table 2, entry 10

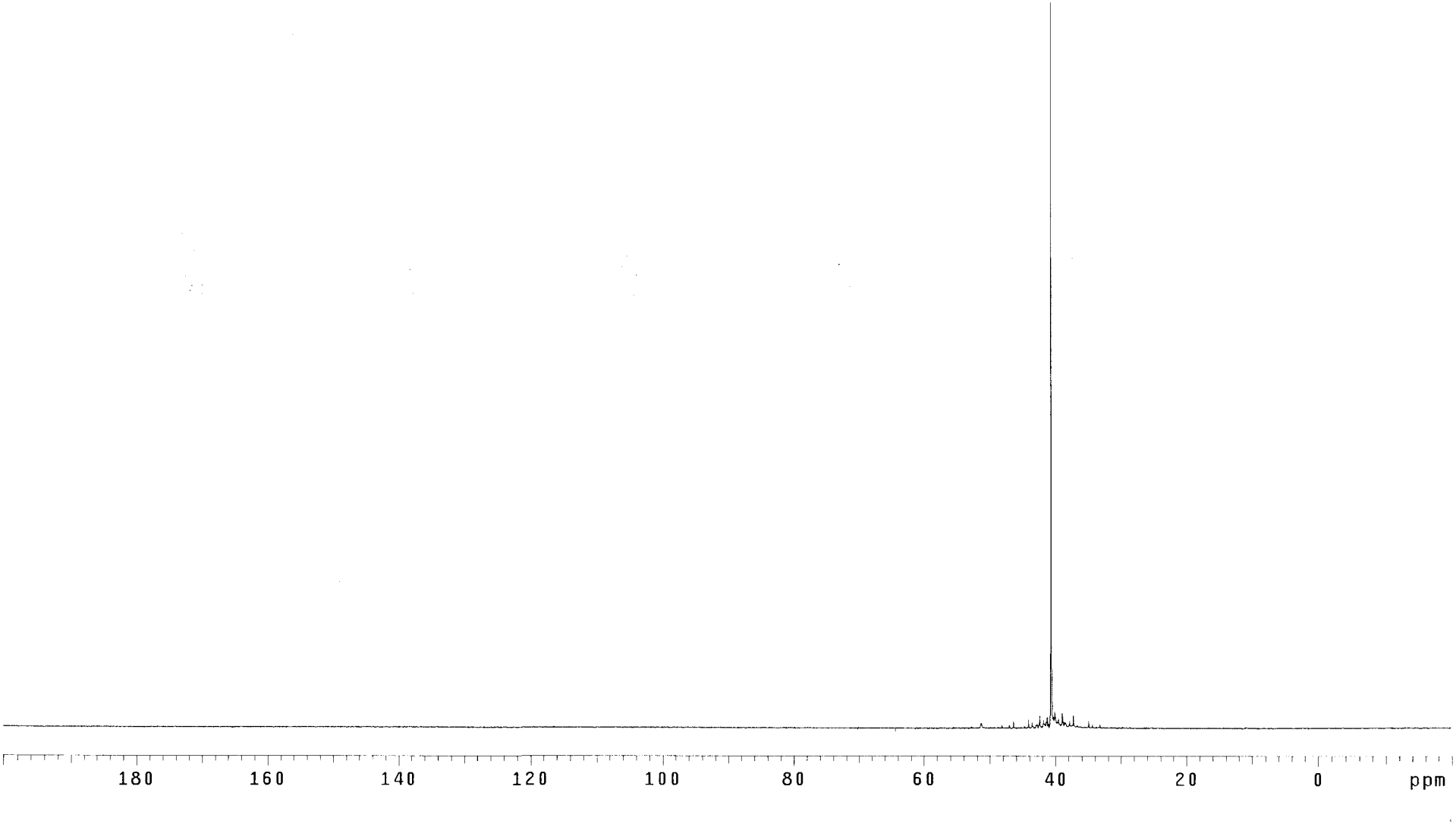


INDEX	FREQUENCY	PPM	HEIGHT
1	4945.224	40.714	126.0

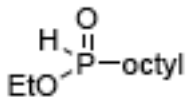


$^{31}P/^{1}H$ decoupled

5 Scheme 5

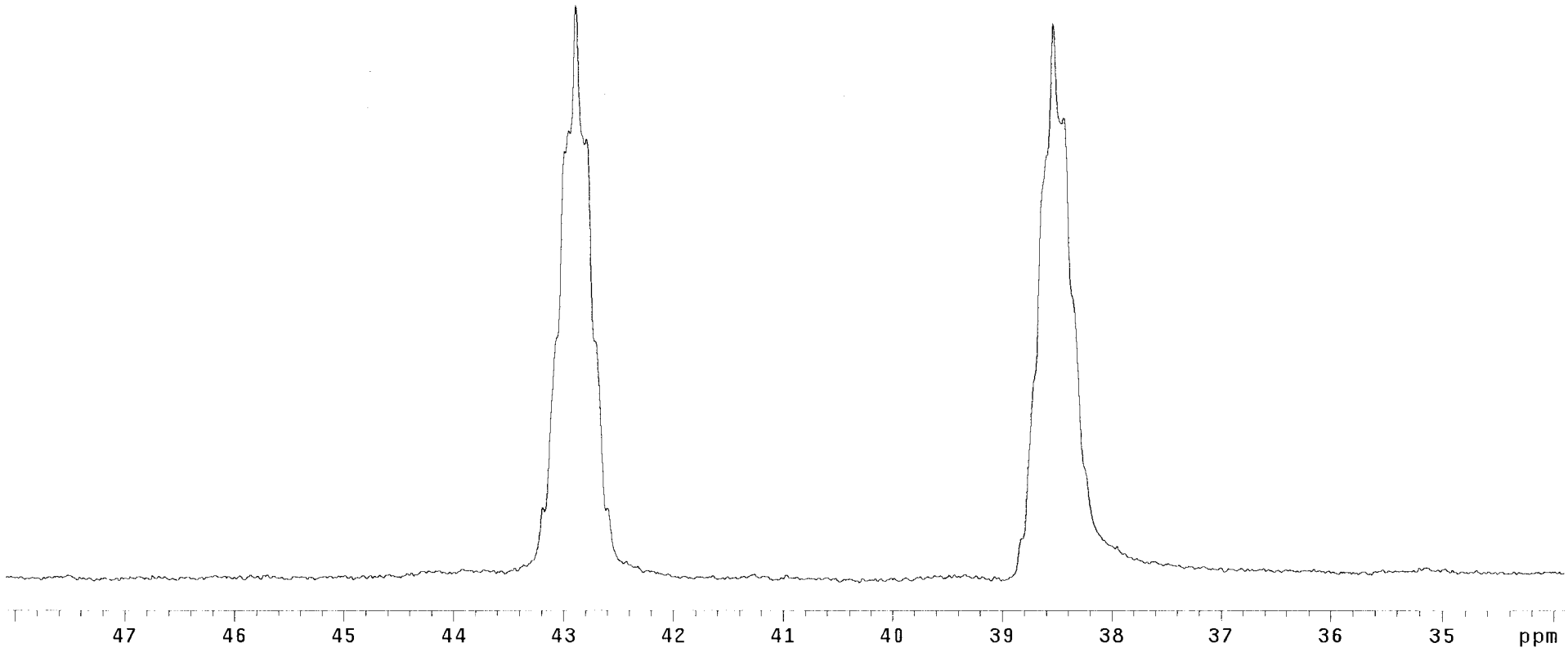


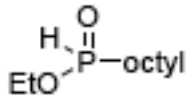
INDEX	FREQUENCY	PPM	HEIGHT
1	5209.193	42.887	92.8
2	5197.361	42.790	71.1
3	4679.623	38.527	89.8
4	4667.792	38.430	74.5



$^{31}P/^{1}H$ coupled

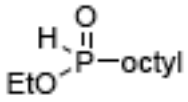
5 Scheme 5



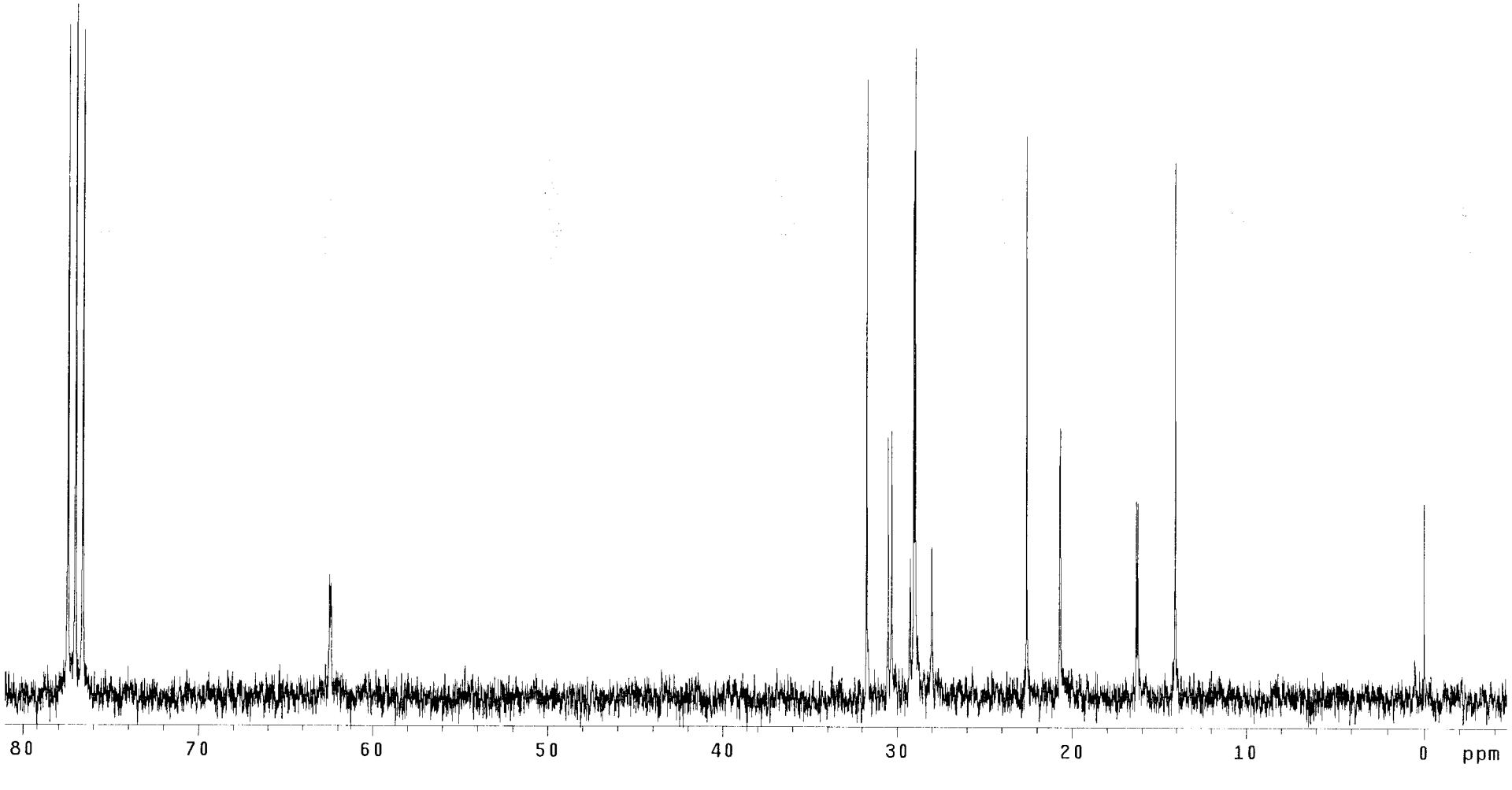


5 Scheme 5

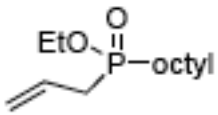
INDEX	FREQUENCY	PPM	HEIGHT
1	5845.844	77.482	112.3
2	5813.887	77.058	115.8
3	5781.929	76.635	111.5
4	4714.390	62.485	20.4
5	2397.069	31.771	103.6
6	2303.789	30.535	43.5
7	2288.243	30.329	44.6
8	2207.918	29.264	23.4
9	2192.947	29.066	91.7
10	2187.765	28.997	108.9
11	2114.350	28.024	25.3
12	1706.681	22.621	94.1
13	1563.594	20.724	40.4
14	1560.715	20.686	45.1
15	1231.068	16.317	33.1
16	1225.022	16.237	32.8
17	1062.069	14.077	89.7
18	-0.000	-0.000	32.7



5 Scheme 5



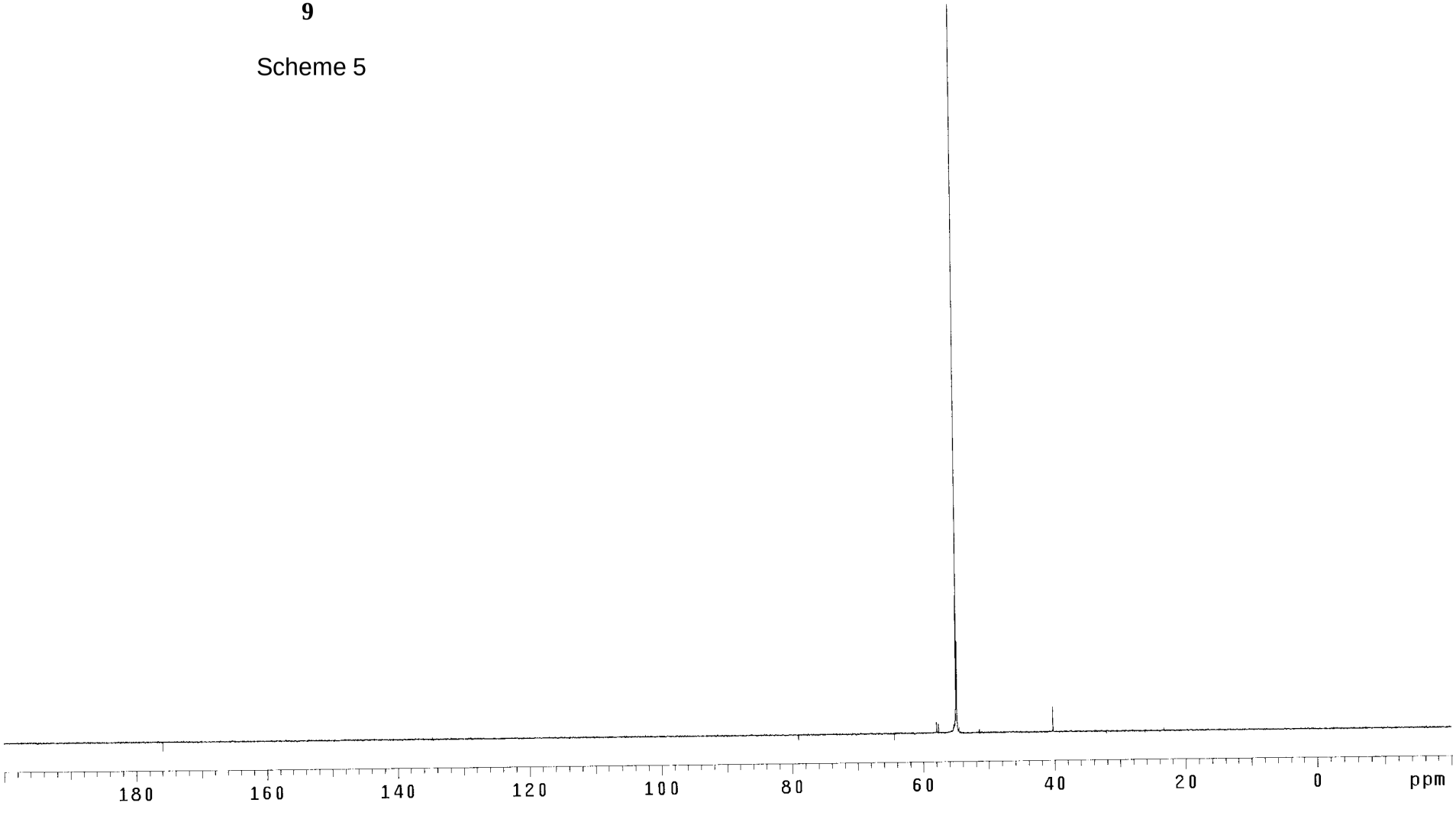
INDEX	FREQUENCY	PPM	HEIGHT
1	6671.426	54.926	126.0



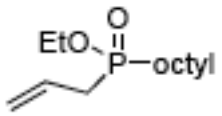
9

$^{31}P/^{1}H$ decoupled

Scheme 5



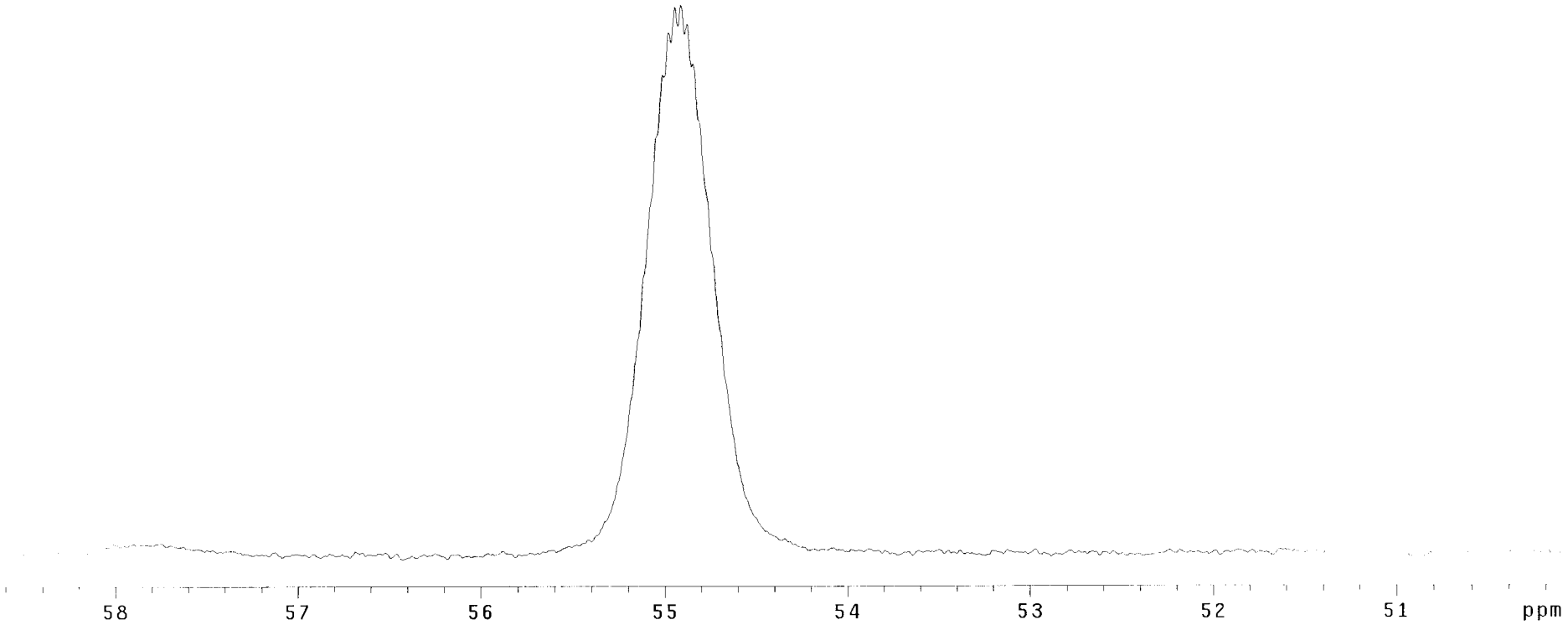
INDEX	FREQUENCY	PPM	HEIGHT
1	6677.137	54.973	82.2
2	6673.058	54.939	86.3
3	6668.978	54.906	86.6
4	6664.898	54.872	83.6



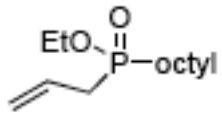
9

$^{31}P/^{1}H$ coupled

Scheme 5



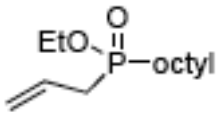
INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	2193.103	7.309	5.7	40	1233.895	4.112	17.2	79	465.239	1.551	8.2
2	1769.492	5.897	2.2	41	1230.084	4.100	20.2	80	462.308	1.541	9.9
3	1763.922	5.879	2.5	42	1226.273	4.087	24.4	81	454.392	1.514	5.3
4	1761.870	5.872	5.3	43	1222.462	4.074	25.0	82	427.715	1.425	3.8
5	1759.818	5.865	2.7	44	1219.237	4.063	20.2	83	423.025	1.410	10.3
6	1756.593	5.854	5.4	45	1215.426	4.051	20.2	84	416.575	1.388	16.1
7	1754.248	5.846	5.6	46	1212.202	4.040	8.7	85	415.403	1.384	17.1
8	1752.196	5.840	6.6	47	1208.391	4.027	6.5	86	409.539	1.365	23.6
9	1748.971	5.829	4.4	48	1205.459	4.018	4.0	87	402.211	1.340	91.4
10	1746.626	5.821	7.9	49	1114.581	3.715	2.6	88	395.175	1.317	162.0
11	1744.574	5.814	7.3	50	1101.095	3.670	3.2	89	392.830	1.309	42.7
12	1742.228	5.806	5.0	51	1098.750	3.662	6.4	90	391.364	1.304	41.3
13	1739.004	5.796	7.7	52	1094.353	3.647	86.7	91	388.139	1.294	108.5
14	1736.951	5.789	6.5	53	1087.903	3.626	2.6	92	385.501	1.285	68.3
15	1734.606	5.781	7.6	54	795.626	2.652	23.0	93	380.224	1.267	111.5
16	1731.675	5.771	3.3	55	788.004	2.626	23.0	94	351.788	1.172	2.3
17	1729.329	5.763	7.3	56	778.330	2.594	22.1	95	340.941	1.136	2.6
18	1727.277	5.757	3.9	57	771.001	2.570	21.9	96	333.905	1.113	2.1
19	1721.707	5.738	3.2	58	608.006	2.026	36.3	97	284.362	0.948	2.9
20	1575.422	5.251	3.1	59	530.027	1.766	8.3	98	270.583	0.902	26.5
21	1573.957	5.246	9.6	60	528.854	1.763	9.0	99	264.134	0.880	87.5
22	1572.491	5.241	14.9	61	523.284	1.744	13.4	100	257.098	0.857	31.7
23	1571.025	5.236	14.7	62	521.232	1.737	10.8	101	21.400	0.071	11.3
24	1570.146	5.233	18.2	63	517.421	1.724	11.7	102	20.228	0.067	3.7
25	1569.266	5.230	19.2	64	513.317	1.711	24.9	103	3.225	0.011	2.3
26	1567.800	5.225	22.5	65	510.385	1.701	12.1	104	-0.000	-0.000	63.5
27	1566.334	5.220	22.4	66	507.161	1.690	9.3	105	-2.638	-0.009	3.1
28	1559.885	5.199	9.3	67	504.522	1.681	9.1	106	-3.225	-0.011	3.0
29	1558.712	5.195	13.0	68	502.763	1.676	9.0				
30	1557.833	5.192	13.9	69	500.711	1.669	17.8				
31	1556.660	5.188	20.8	70	499.245	1.664	14.4				
32	1555.195	5.183	21.7	71	492.503	1.641	7.4				
33	1554.022	5.179	12.0	72	490.451	1.635	5.4				
34	1551.970	5.172	13.0	73	486.933	1.623	9.3				
35	1550.504	5.167	11.2	74	484.294	1.614	10.3				
36	1549.038	5.163	5.4	75	482.829	1.609	10.2				
37	1243.862	4.146	3.6	76	478.138	1.594	16.0				
38	1240.931	4.136	5.7	77	474.620	1.582	12.5				
39	1236.827	4.122	8.4	78	469.637	1.565	14.2				



9

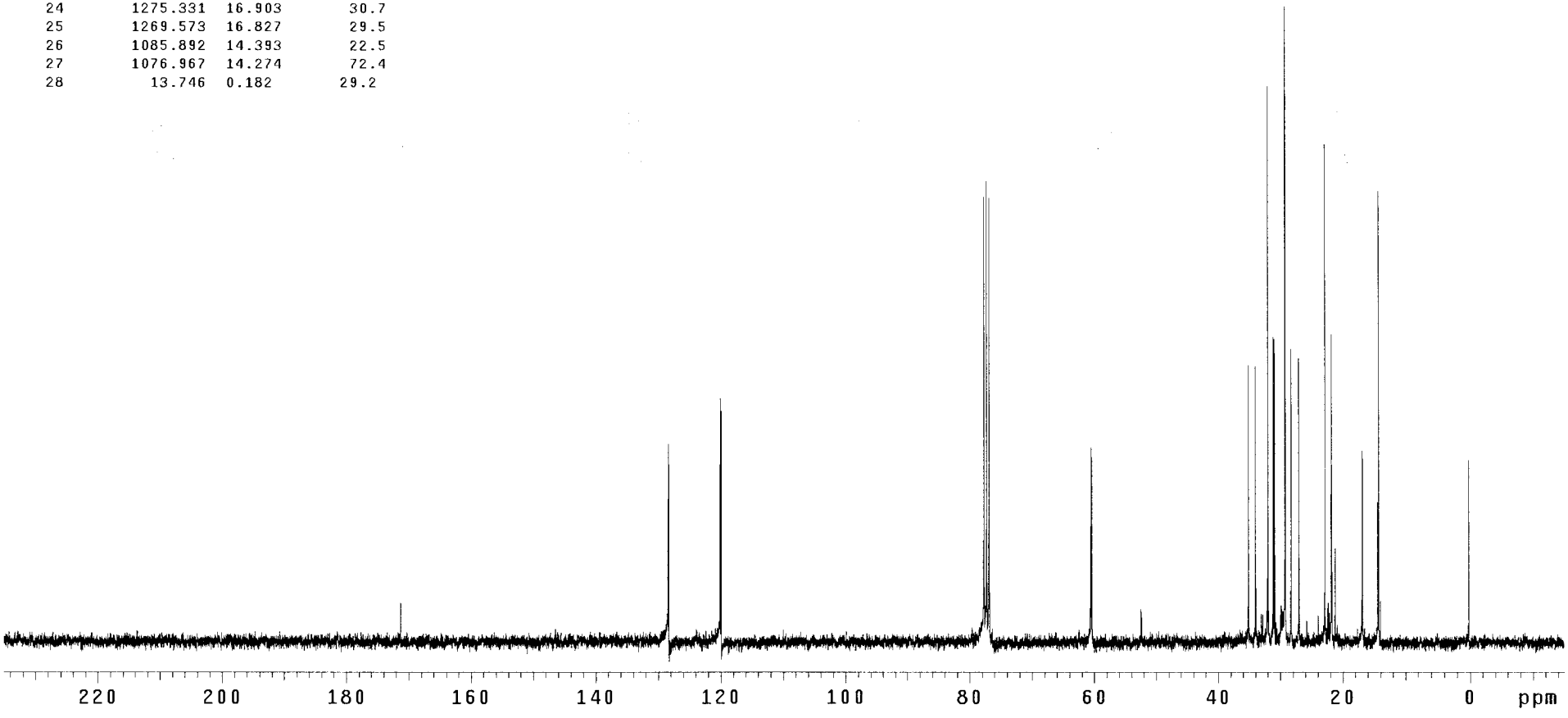
Scheme 5

INDEX	FREQUENCY	PPM	HEIGHT
1	9690.121	128.435	31.8
2	9681.484	128.320	28.6
3	9063.071	120.124	39.2
4	9050.980	119.963	37.1
5	5861.893	77.695	71.4
6	5829.936	77.271	74.1
7	5797.978	76.847	71.3
8	4570.366	60.576	18.7
9	4563.456	60.485	31.2
10	4556.834	60.397	29.7
11	2650.637	35.132	44.5
12	2564.843	33.995	44.3
13	2413.406	31.988	89.4
14	2348.341	31.125	48.9
15	2333.082	30.923	48.6
16	2207.557	29.259	93.7
17	2205.253	29.229	102.3
18	2132.702	28.267	47.0
19	2039.998	27.039	45.6
20	1721.866	22.822	80.0
21	1647.588	21.837	49.3
22	1643.557	21.784	47.0
23	1602.387	21.238	15.1
24	1275.331	16.903	30.7
25	1269.573	16.827	29.5
26	1085.892	14.393	22.5
27	1076.967	14.274	72.4
28	13.746	0.182	29.2

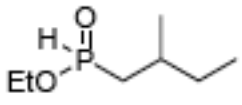


9

Scheme 5



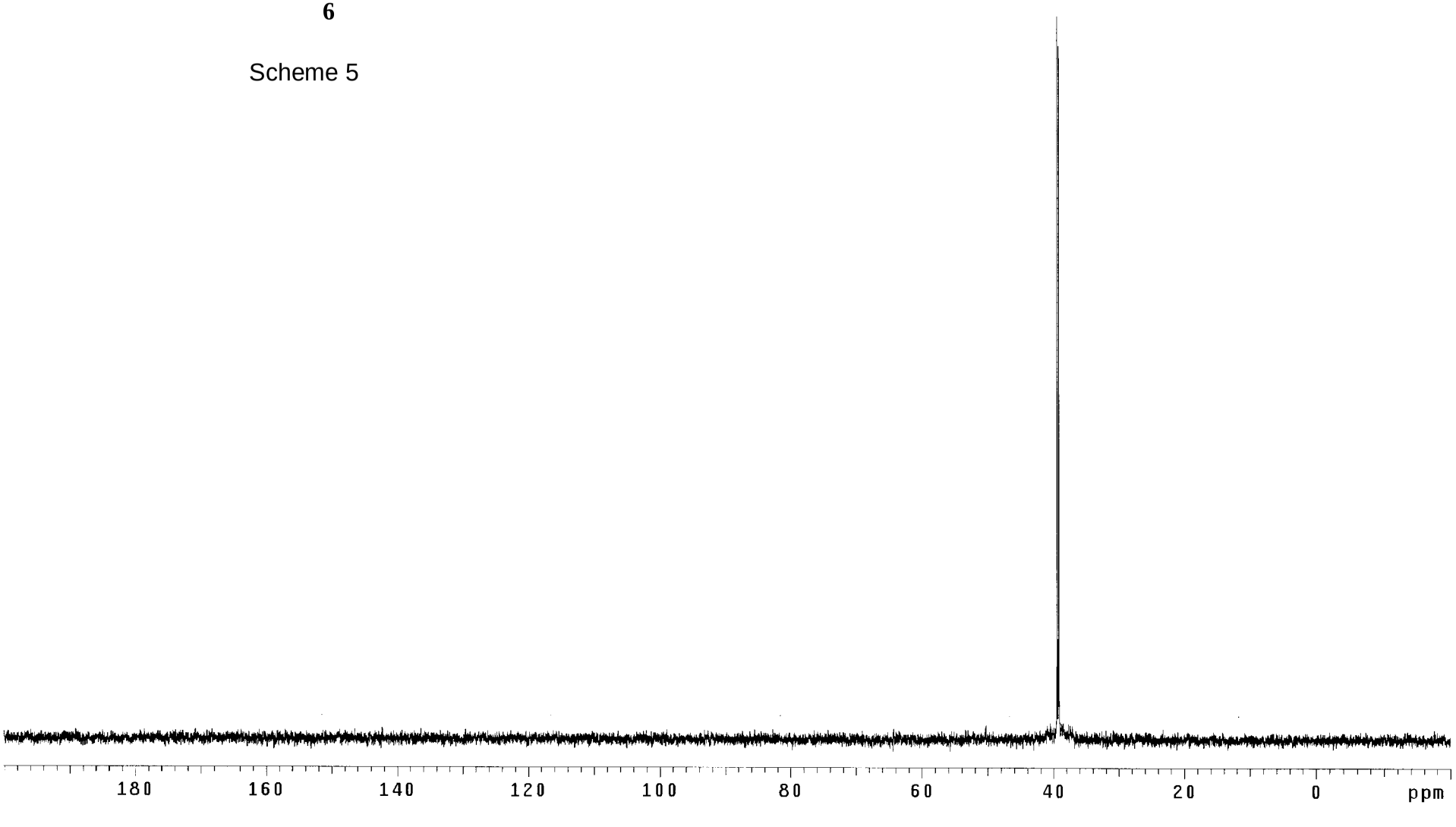
INDEX	FREQUENCY	PPM	HEIGHT
1	4793.044	39.461	126.0
2	4762.037	39.206	120.9



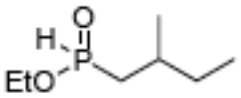
6

Scheme 5

$^{31}P/^{1}H$ decoupled



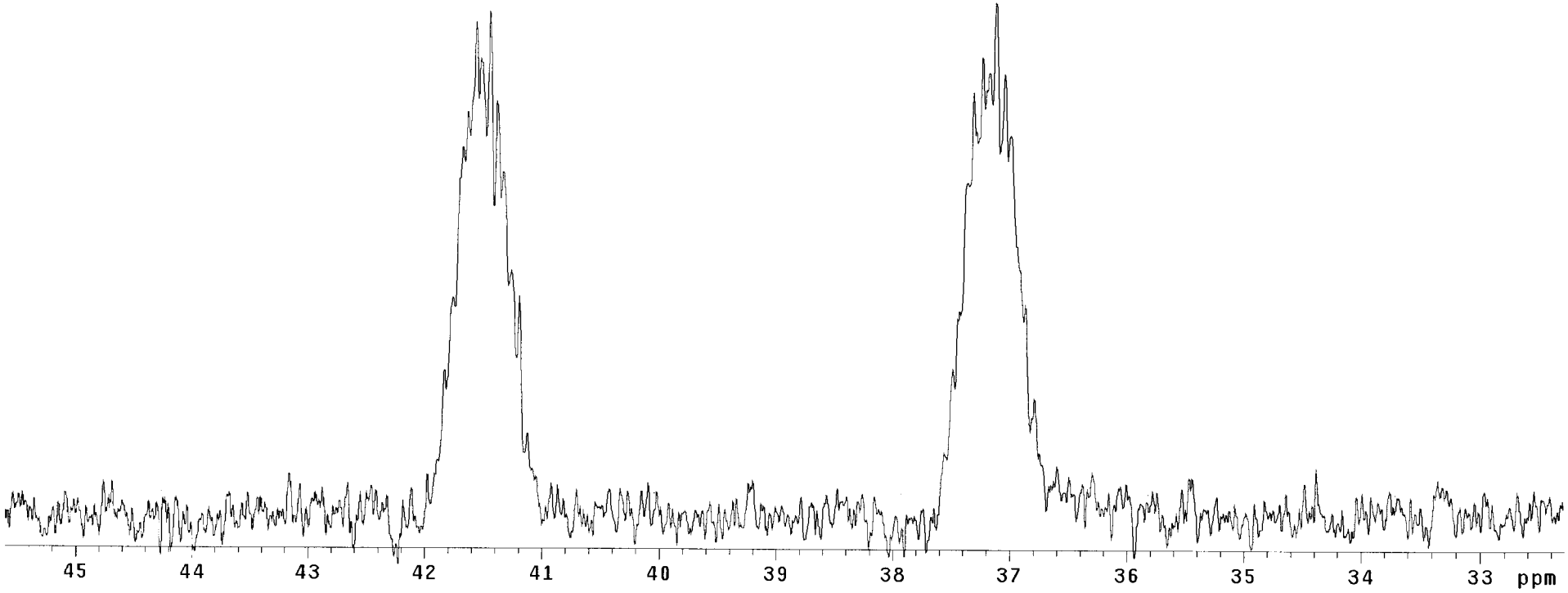
INDEX	FREQUENCY	PPM	HEIGHT
1	5048.853	41.567	80.1
2	5034.982	41.453	81.8
3	5027.230	41.389	67.2
4	4507.452	37.110	83.5

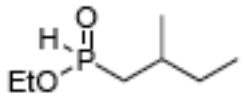


$^{31}P/^{1}H$ coupled

6

Scheme 5

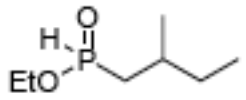




6

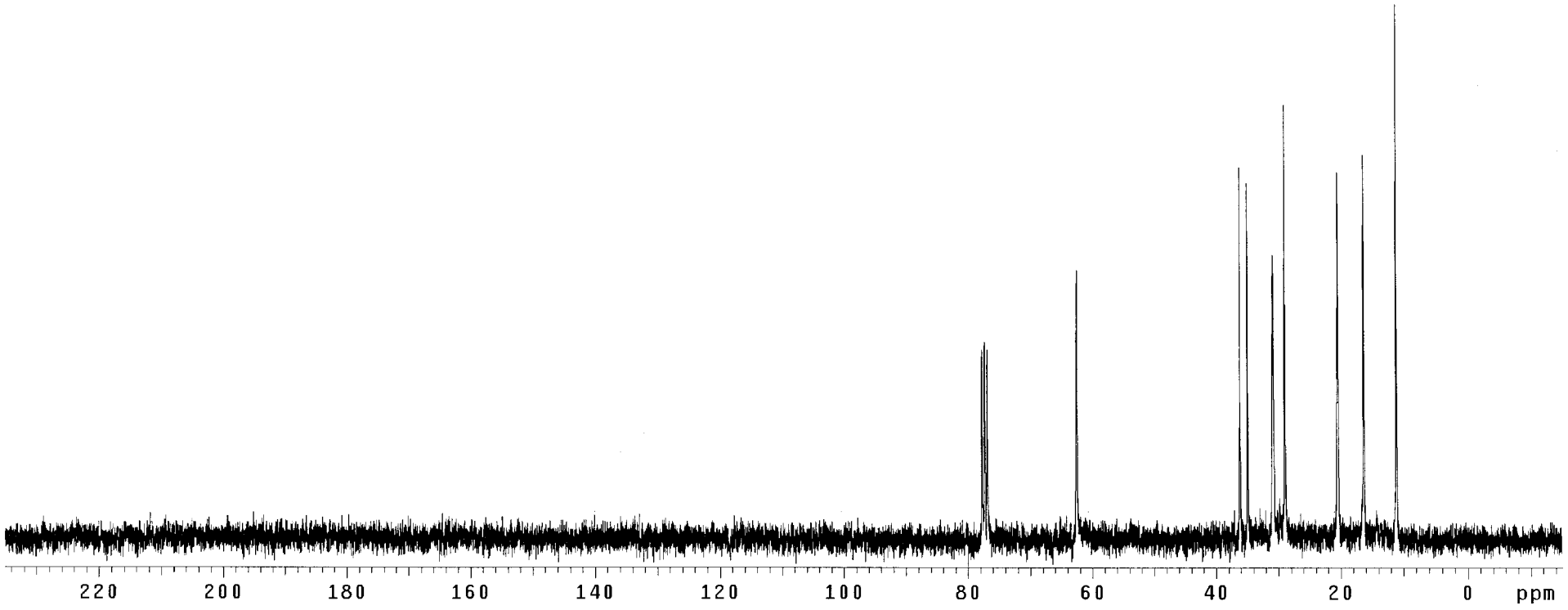
Scheme 5

INDEX	FREQUENCY	PPM	HEIGHT
1	5871.105	77.817	30.3
2	5839.148	77.393	31.6
3	5828.784	77.256	18.8
4	5806.903	76.966	30.3
5	5796.827	76.832	15.8
6	4722.954	62.599	41.1
7	4719.211	62.549	43.1
8	2734.129	36.239	59.7
9	2640.849	35.002	57.2
10	2343.158	31.057	32.9
11	2337.400	30.980	42.2
12	2330.203	30.885	45.6
13	2324.733	30.812	42.7
14	2190.858	29.038	69.7
15	1557.475	20.643	31.4
16	1550.565	20.551	58.9
17	1543.655	20.460	53.9
18	1239.919	16.434	50.4
19	1233.873	16.354	61.7
20	840.887	11.145	86.2

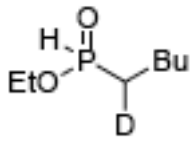


6

Scheme 5



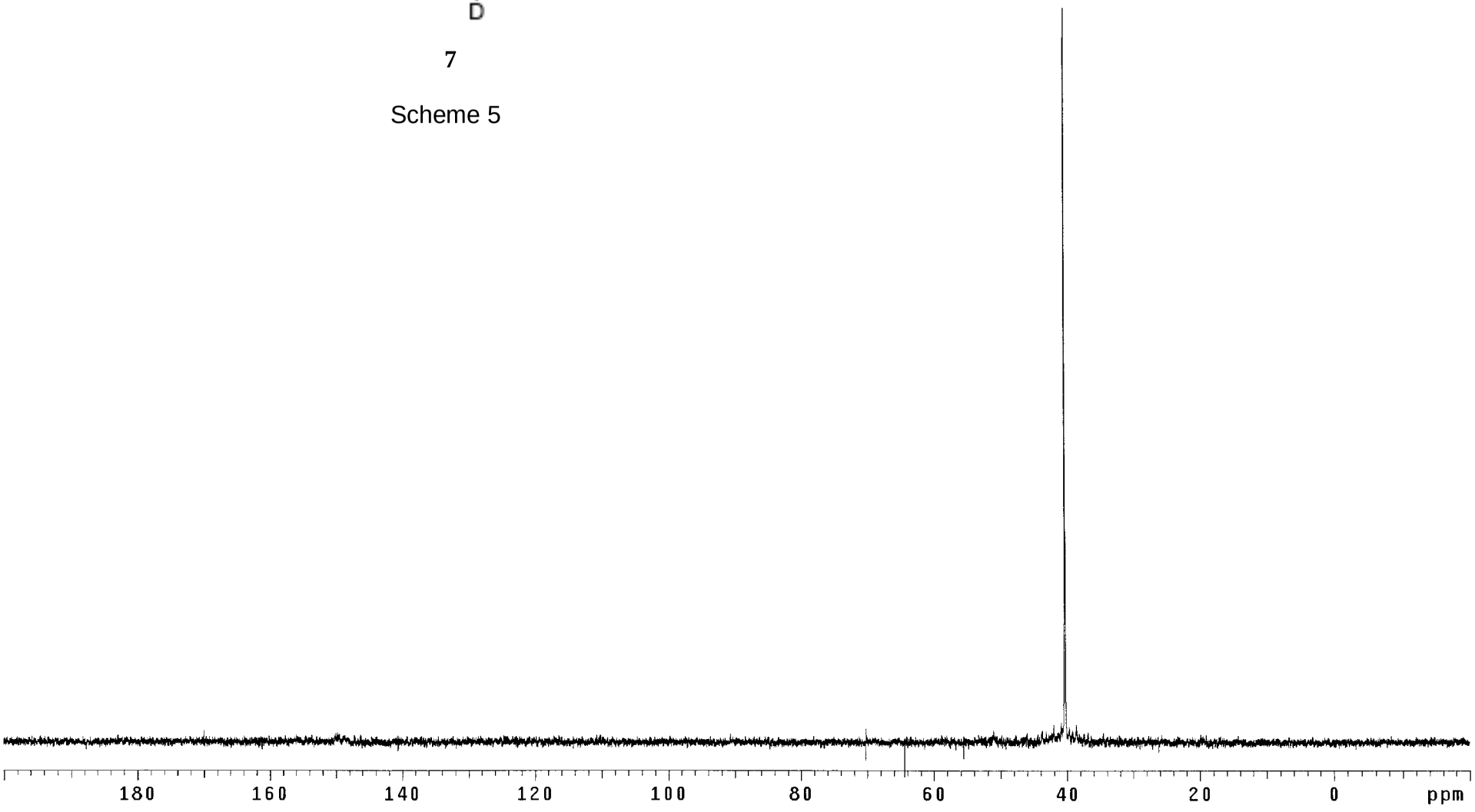
INDEX	FREQUENCY	PPM	HEIGHT
1	4896.673	40.314	126.3



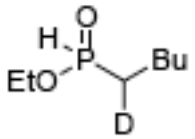
$^{31}P/^{1}H$ decoupled

7

Scheme 5



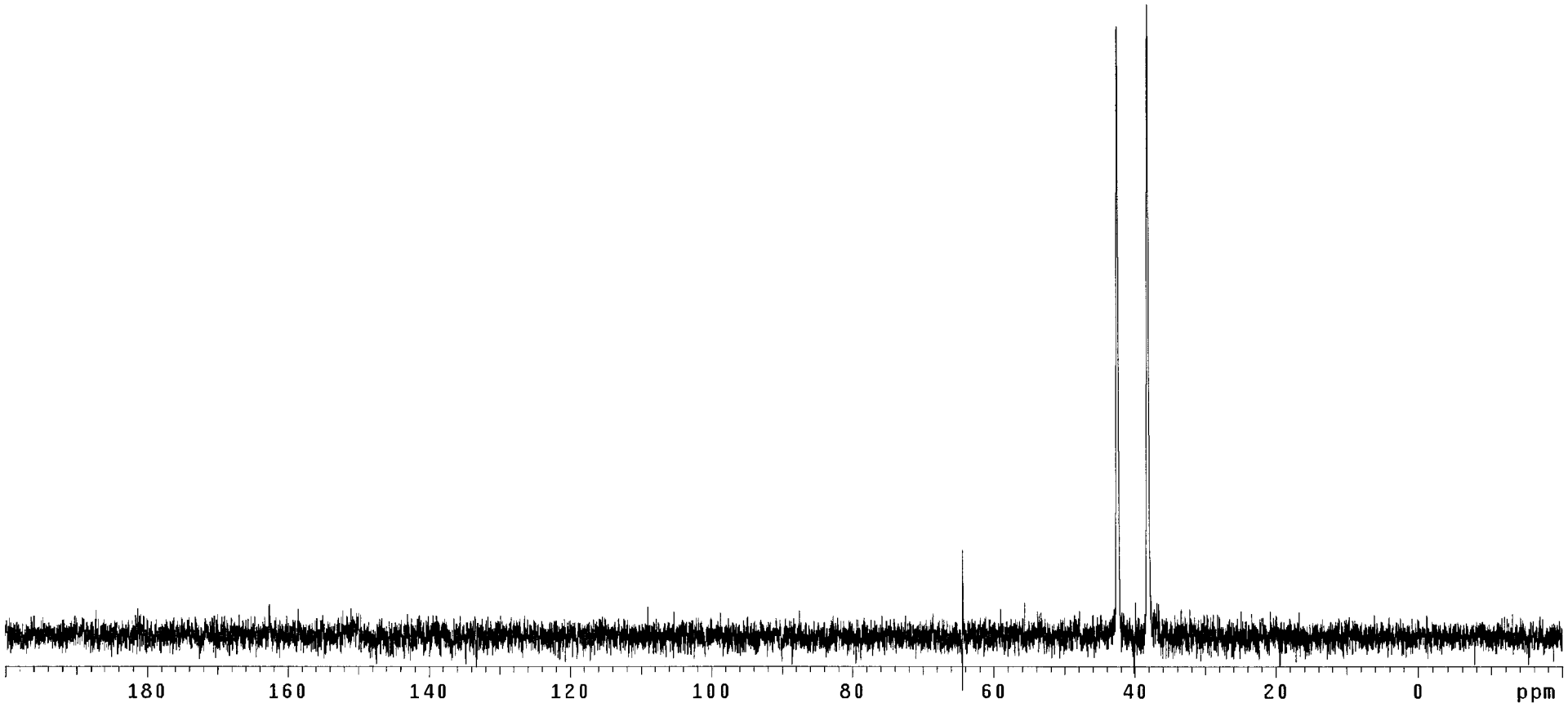
INDEX	FREQUENCY	PPM	HEIGHT
1	5154.522	42.437	98.8
2	4627.401	38.097	102.4

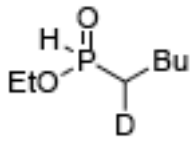


$^{31}P/^{1}H$ coupled

7

Scheme 5

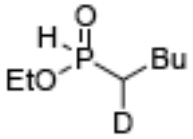




7

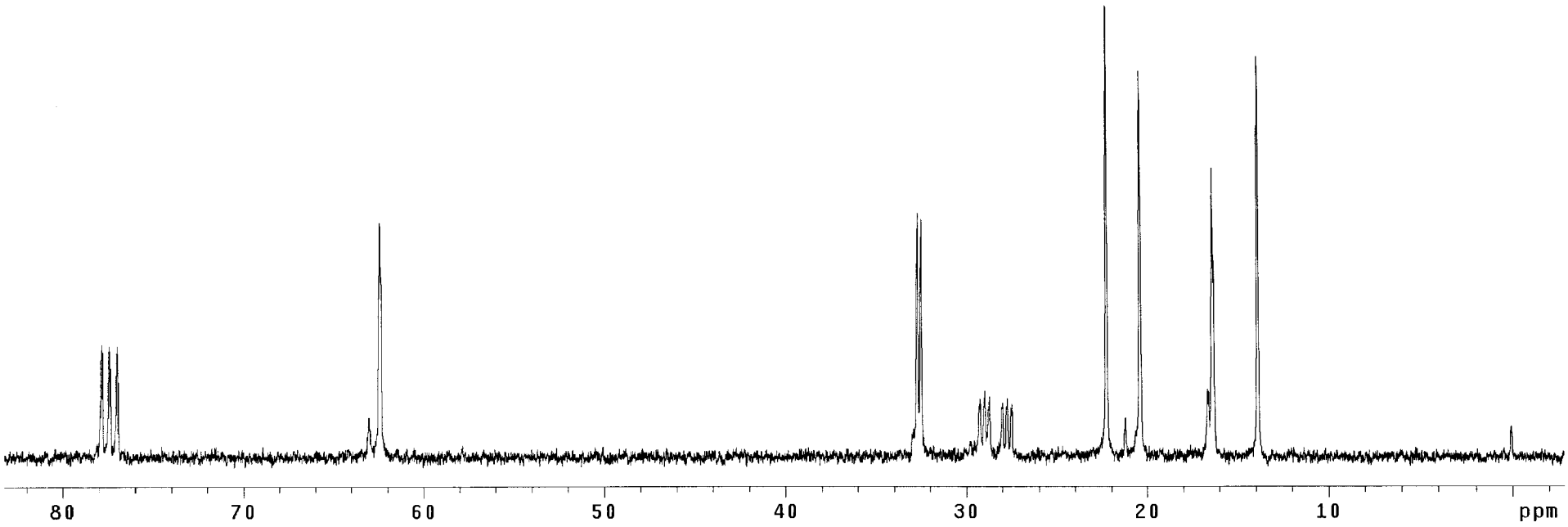
Scheme 5

INDEX	FREQUENCY	PPM	HEIGHT
1	5872.257	77.832	18.1
2	5840.876	77.416	17.8
3	5808.343	76.985	17.9
4	4710.286	62.431	37.7
5	2465.229	32.675	39.2
6	2449.682	32.469	38.2
7	2204.102	29.214	9.1
8	2184.812	28.958	10.5
9	2165.523	28.702	9.6
10	2110.822	27.977	8.5
11	2091.244	27.718	9.3
12	2072.243	27.466	8.3
13	1676.954	22.227	72.6
14	1536.746	20.368	62.0
15	1255.754	16.644	10.8
16	1233.585	16.350	46.4
17	1227.827	16.274	32.4
18	1042.706	13.820	64.4

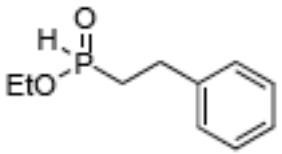


7

Scheme 5



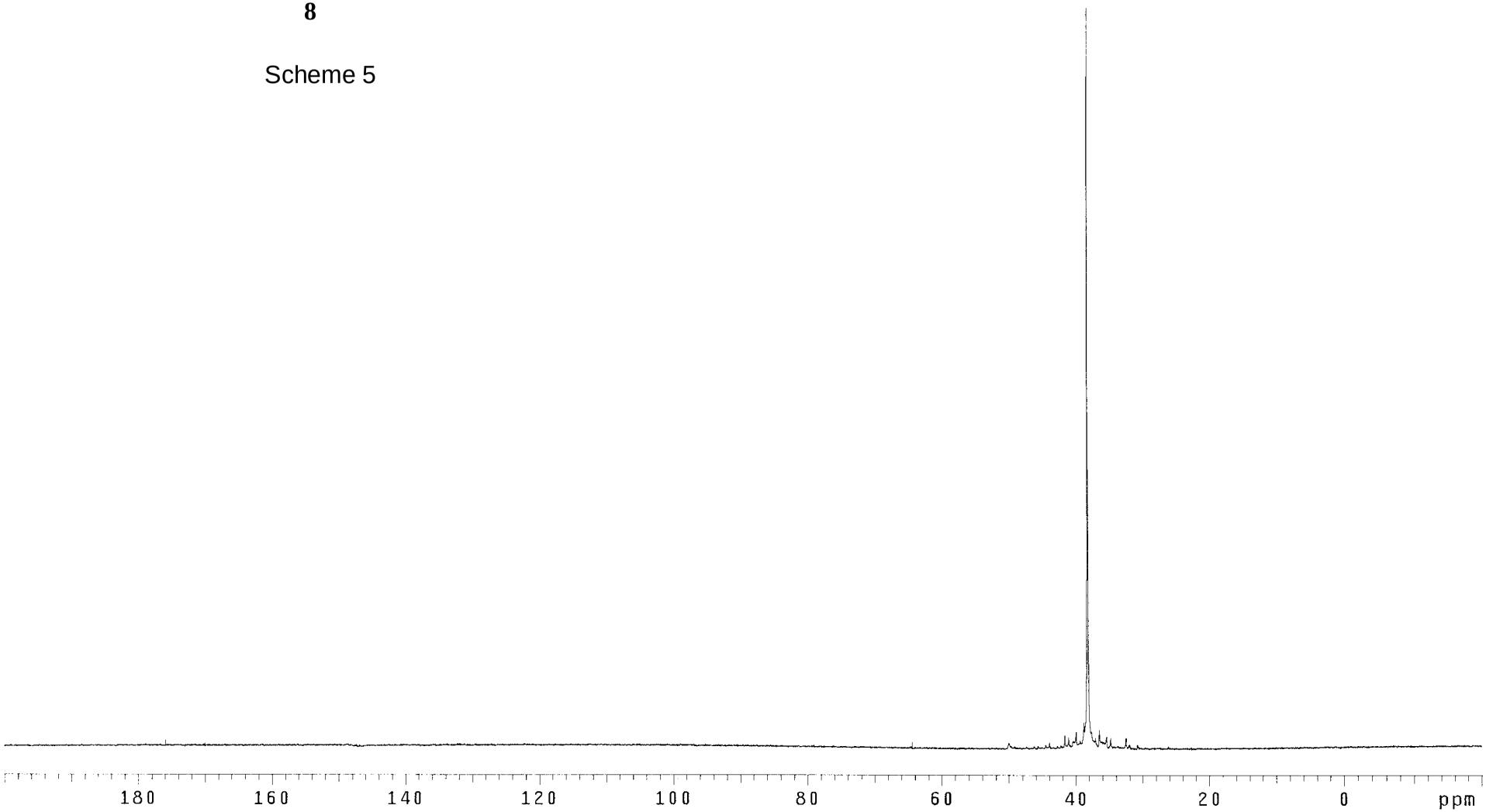
INDEX	FREQUENCY	PPM	HEIGHT
1	4642.496	38.222	126.0



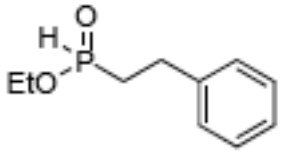
8

Scheme 5

$^{31}P/^{1}H$ decoupled



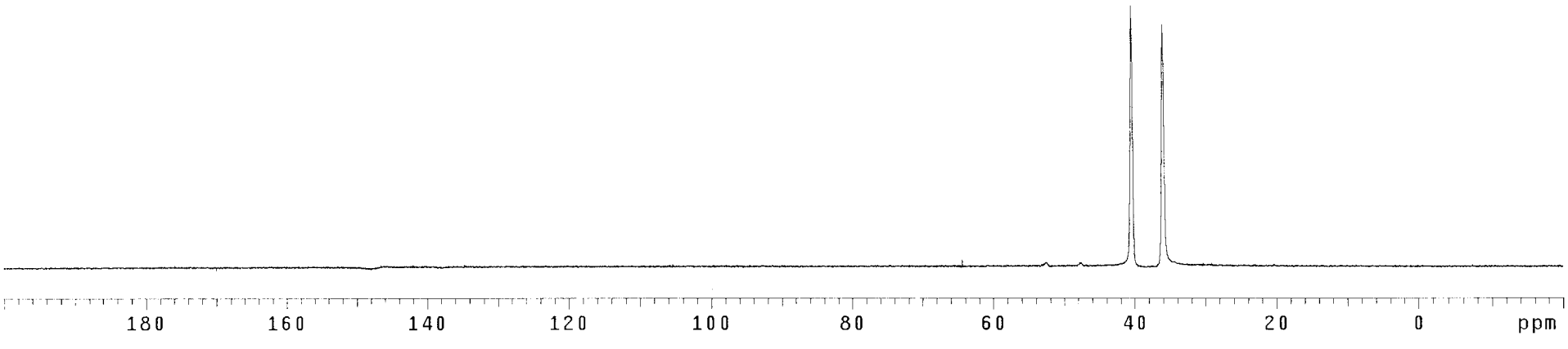
INDEX	FREQUENCY	PPM	HEIGHT
1	4909.321	40.418	42.3
2	4374.855	36.018	39.3

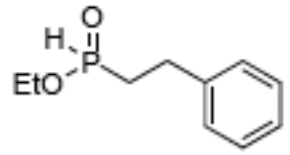


8

$^{31}P/^{1}H$ decoupled

Scheme 5

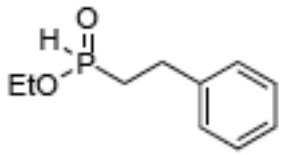




8

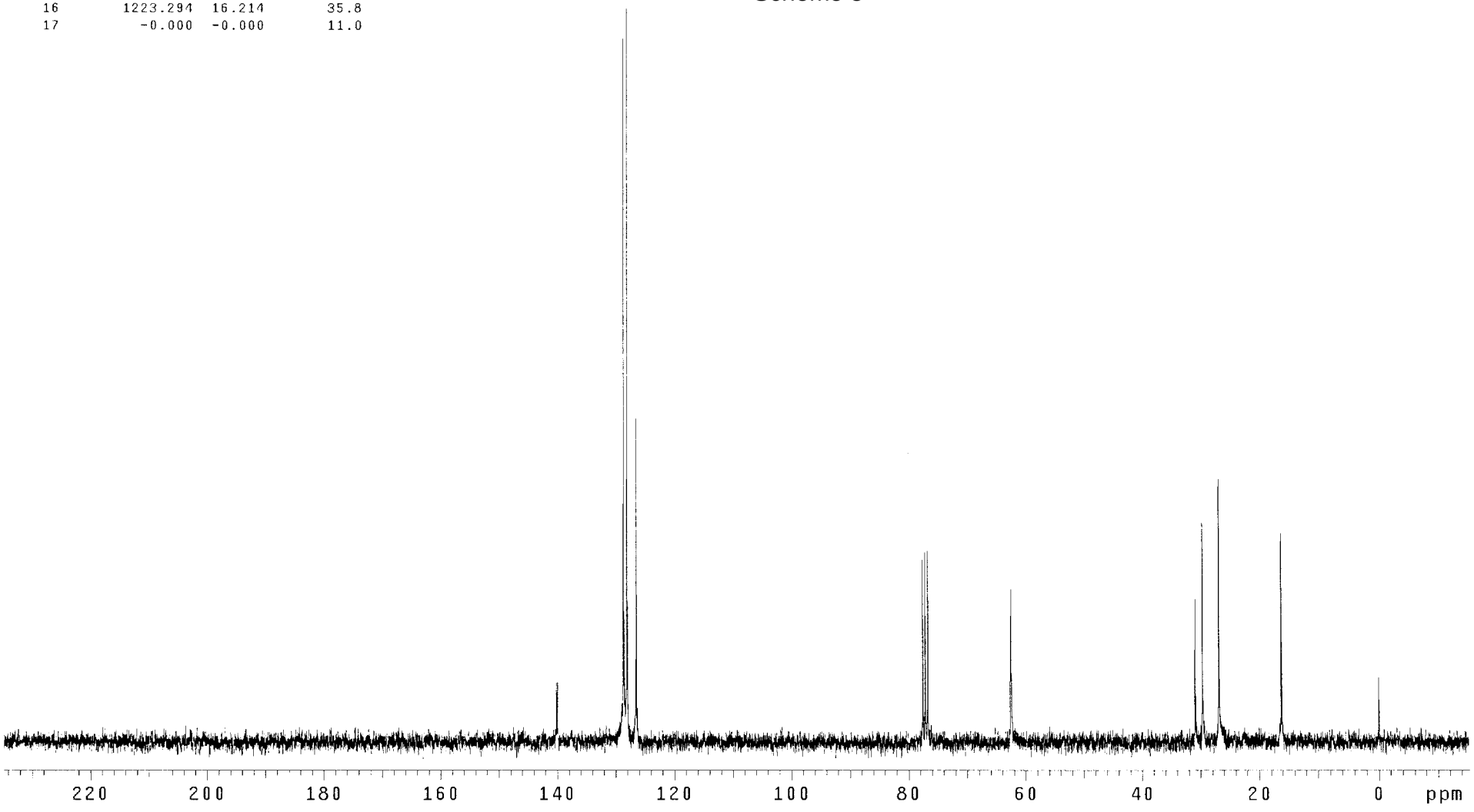
Scheme 5

INDEX	FREQUENCY	PPM	HEIGHT
1	10575.780	140.173	10.1
2	10560.234	139.967	10.1
3	9708.908	128.684	120.7
4	9698.256	128.542	10.9
5	9664.572	128.096	125.9
6	9547.396	126.543	55.3
7	5854.481	77.596	31.3
8	5822.524	77.173	32.5
9	5790.566	76.749	32.7
10	4724.754	62.623	21.6
11	4718.421	62.539	26.2
12	2332.579	30.916	24.5
13	2239.299	29.680	37.6
14	2026.828	26.864	45.0
15	1229.340	16.294	34.4
16	1223.294	16.214	35.8
17	-0.000	-0.000	11.0

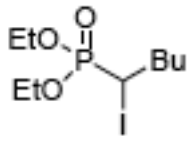


8

Scheme 5



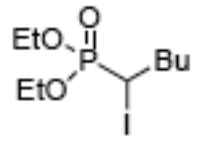
INDEX	FREQUENCY	PPM	HEIGHT
1	7819.915	64.381	-15.5
2	2813.889	23.167	126.5



3b-I

Scheme 6

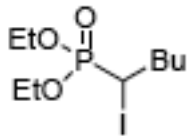
$^{31}P/^{1}H$ decoupled



3b-I

Scheme 6

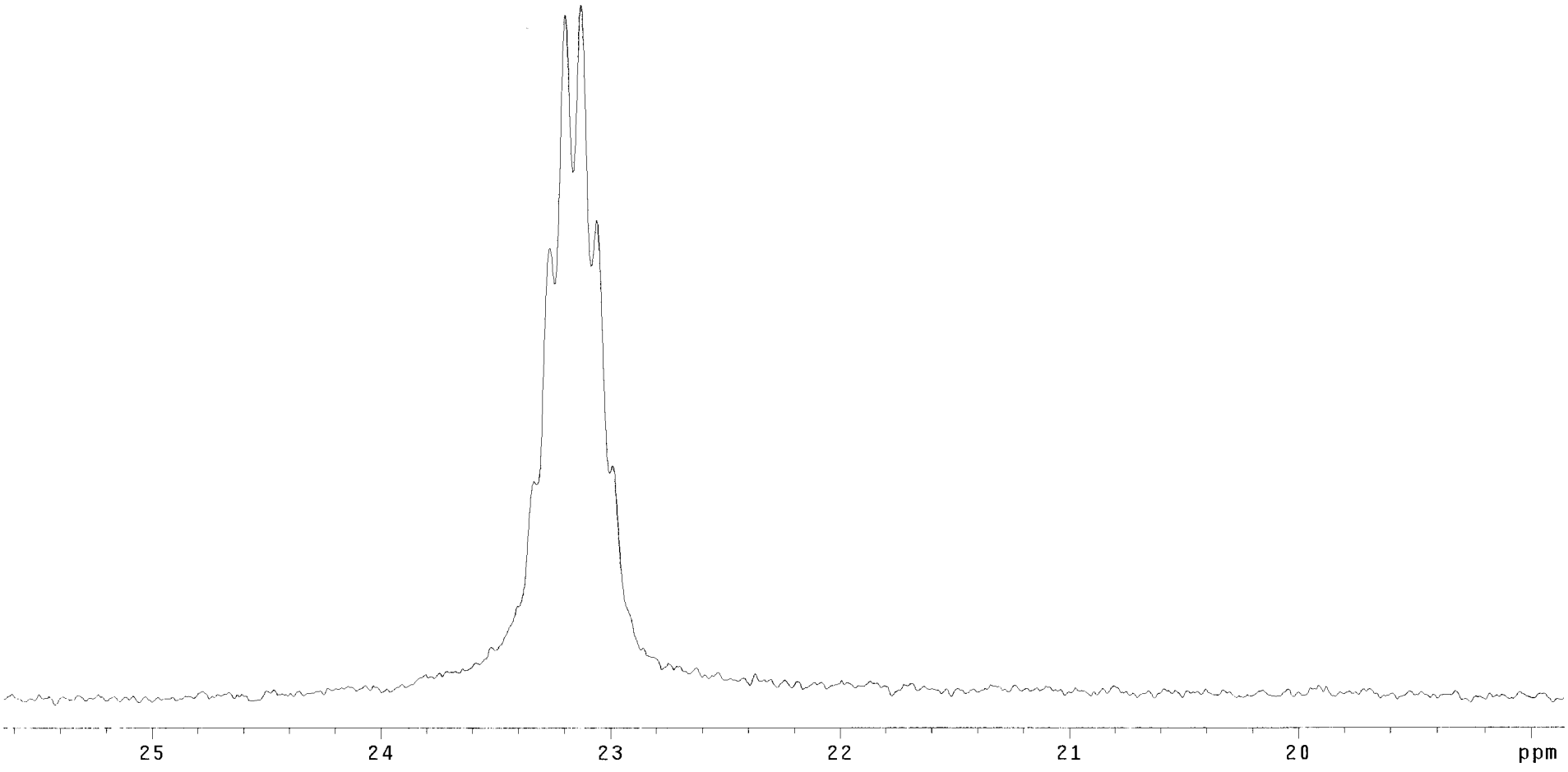
INDEX	FREQUENCY	PPM	HEIGHT
1	2825.313	23.261	72.0
2	2817.153	23.194	109.6
3	2808.586	23.123	111.2
4	2800.426	23.056	76.5



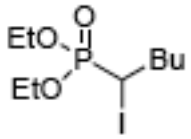
3b-I

$^{31}P/^{1}H$ coupled

Scheme 6

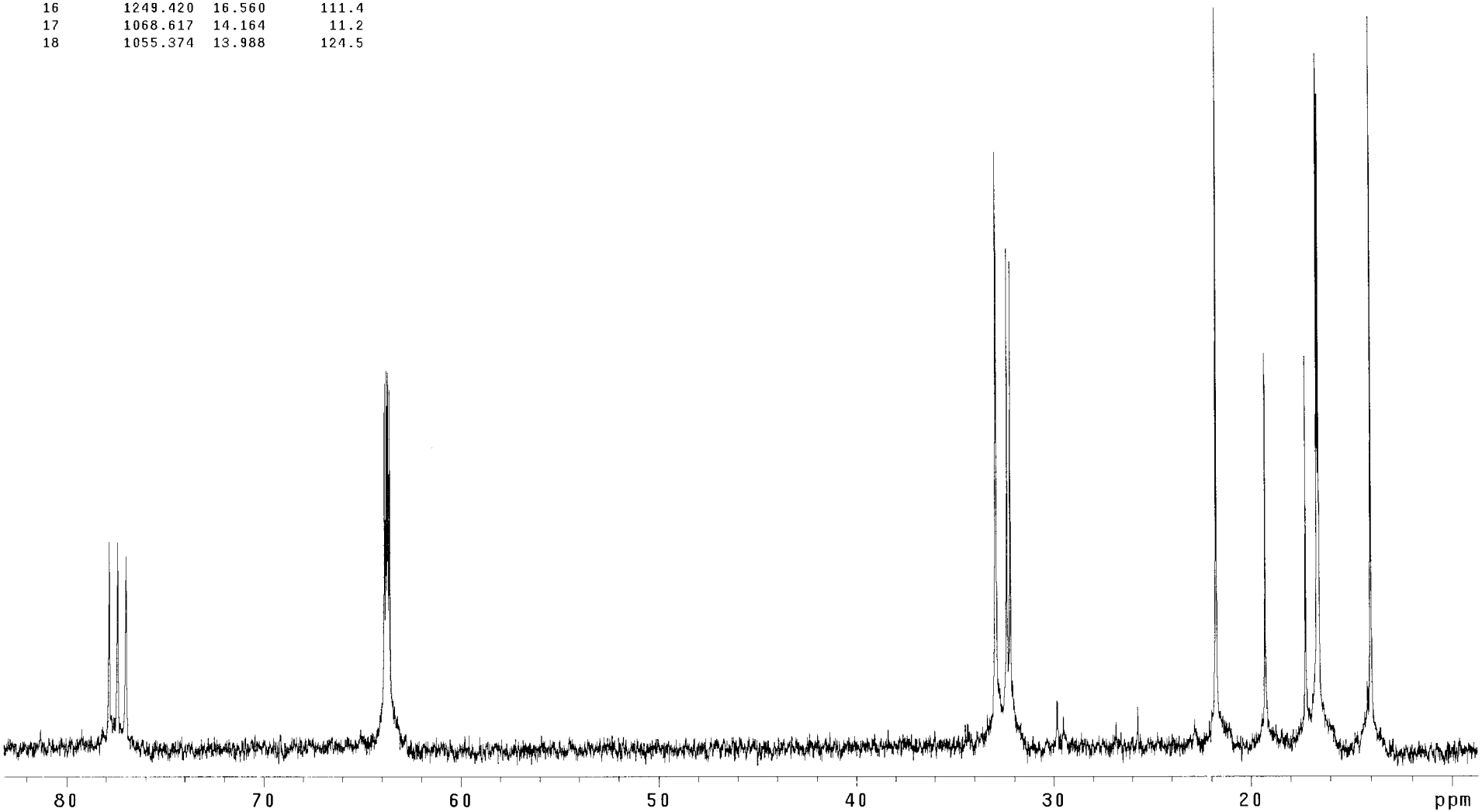


INDEX	FREQUENCY	PPM	HEIGHT
1	5871.681	77.824	35.0
2	5839.436	77.397	34.9
3	5807.479	76.973	32.6
4	4819.113	63.873	61.9
5	4812.203	63.782	64.1
6	4806.157	63.702	63.9
7	4799.247	63.610	60.8
8	2484.230	32.926	101.5
9	2481.927	32.896	90.5
10	2440.469	32.346	85.0
11	2427.801	32.179	82.7
12	1640.390	21.742	126.0
13	1453.254	19.262	67.1
14	1299.515	17.224	66.6
15	1255.178	16.636	118.2
16	1249.420	16.560	111.4
17	1068.617	14.164	11.2
18	1055.374	13.988	124.5

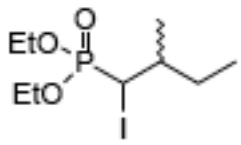


3b-I

Scheme 6



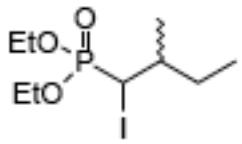
INDEX	FREQUENCY	PPM	HEIGHT
1	2739.228	22.552	126.0
2	2702.101	22.246	115.9



$^{31}P/^{1}H$ decoupled

Diethyl 1–iodo–2-methylbutylphosphonate Scheme 6

INDEX	FREQUENCY	PPM	HEIGHT
1	2733.108	22.502	126.0
2	2695.981	22.196	112.2



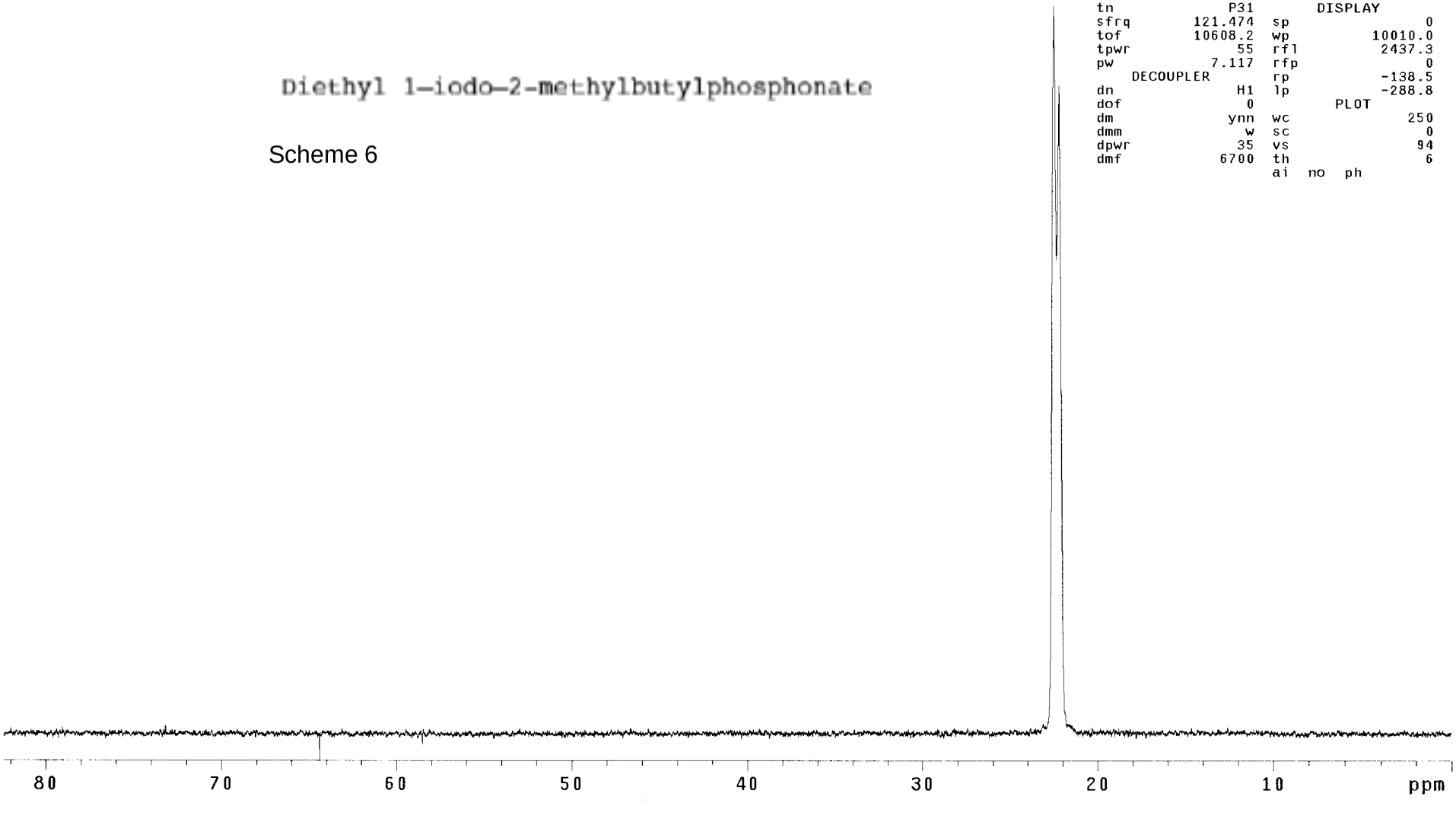
$^{31}P/^{1}H$ decoupled

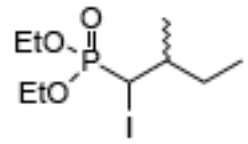
Diethyl 1–iodo–2-methylbutylphosphonate

Scheme 6

exp1 s2pul

SAMPLE		SPECIAL	
date	Dec 29 2007	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	64	PROCESSING	
ct	47	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	-138.5
dn	H1	lp	-288.8
dof	0	PLOT	
dm	ynn	wc	250
dmm	w	sc	0
dpwr	35	vs	94
dmf	6700	th	6
		ai no ph	





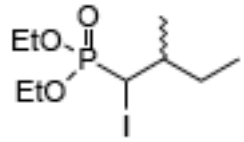
Diethyl 1–iodo–2-methylbutylphosphonate

Scheme 6

Monika

Pulse Sequence: s2pul

INDEX	FREQUENCY	PPM	HEIGHT
1	6405.234	63.688	24.7
2	6402.625	63.662	24.4
3	6398.451	63.621	22.6
4	6395.320	63.589	23.5
5	6378.622	63.423	24.1
6	6371.316	63.351	21.6
7	6364.011	63.278	21.1
8	6357.228	63.211	18.6
9	3656.854	36.361	51.3
10	3543.099	35.229	48.5
11	3093.298	30.757	43.9
12	3077.643	30.601	42.0
13	3033.289	30.160	22.5
14	2961.801	29.450	29.3
15	2883.008	28.666	26.0
16	2830.827	28.147	46.2
17	2829.261	28.132	43.4
18	2810.476	27.945	30.9
19	1995.407	19.841	38.3
20	1980.796	19.695	41.1
21	1901.481	18.907	57.0
22	1655.186	16.458	126.2
23	1649.446	16.401	124.0
24	1165.727	11.591	71.4
25	1140.680	11.342	56.5



Diethyl 1–iodo–2-methylbutylphosphonate

Scheme 6