

Supporting Information

Strategies for the asymmetric synthesis of *H*-phosphinate esters

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General Chemistry

All reactions were conducted in oven-dried glassware, under nitrogen. Reactions carried out at a temperature below 0 °C employed a CO₂/acetone bath. All reagents and solvents were used as received unless otherwise specified. Concentrated H₃PO₂ was obtained by rotary evaporation (0.5 mmHg) of the 50 wt. % aqueous solution at rt for 20-30 min before reaction. *Caution: overdrying H₃PO₂ may result in the formation of a yellow solid of high phosphorus content that could be pyrophoric.* Anilinum hypophosphite and stock solutions of ethyl hypophosphite were prepared as previously described.^{1,2} Unless otherwise specified, HPLC or reagent grade solvents were purchased from Aldrich and used as received. Acetonitrile was distilled under N₂ from CaH₂, and used immediately. *i*Pr₂NEt was distilled from CaH₂ and stored over 4 Å molecular sieves. Catalysts and ligands were commonly purchased from Aldrich or Strem Chemicals. Analytical thin layer chromatography (TLC) was performed on SiO₂ 60 F-254 plates. Visualization was accomplished by UV irradiation at 254 nm and/or by staining with *para*-anisaldehyde or KMnO₄ solution. Flash column chromatography was performed using SiO₂ 60 (particle size 0.040–0.055 mm, 230–400 mesh). Radial chromatography was carried out using 2 or 4 mm layers of silica gel 60 PF₂₅₄ containing gypsum. Proton, carbon and phosphorus NMR spectra were recorded at 300 MHz/150 MHz/121 MHz (¹H NMR/¹³C NMR/³¹P NMR). Chemical shifts are reported as δ values in parts per million (ppm) as referenced to: (a) internal standard (¹H NMR, Me₄Si, δ = 0.00 ppm), (b) residual solvent (¹³C NMR, CDCl₃, δ = 77.0 ppm), and (c) external standard (³¹P NMR, 85% H₃PO₄, δ = 0.00 ppm). ¹H NMR spectra are reported as follows: chemical shift, multiplicity (s = singlet, bs = broad singlet, d = doublet, dd = doublet of doublets, ddd = doublet of doublet of doublets, t = triplet, q = quartet, quint. = quintuplet, sext. = sextuplet, m = multiplet), number of protons, and coupling constant(s). NMR yields are determined by integration of all the resonances in the ³¹P NMR spectra, an approach that is valid if no phosphorus-containing gas evolves, or if the precipitate in a heterogeneous mixture does not contain phosphorus. The yields determined by NMR are generally accurate within ~10% of the value indicated, and are reproducible. The validity of the method has been carefully verified.^{1,3} Isolated yields are sometimes significantly lower because *H*-phosphinate esters are highly polar compounds and hydrolytically labile. Chiral HPLC resolutions were performed with a (*S,S*)-Whelk-01 Column (250x4.6 mm, 5 μm) from Regis® Technologies, which was accompanied with a guard column (Agilent Zorbax® ODS, 4.6 x 12.5 mm, 5 μm), using hexanes/isopropanol mixtures as the mobile phase. Low resolution mass spectrometry was performed on a Bruker Esquire 6000, Bruker Daltonics, Inc., ± ESI. High resolution mass spectrometry was provided by the Mass Spectrometry Facility of the University of South Carolina.

Preparation of chiral auxiliaries (Tables 1, 2 and 3)

The chiral auxiliaries were either commercially available or synthesized according to known protocols described in the following references:

(a) *Commercially available*: fenchyl alcohol (Table 1, entry 1), (+)-isopinocampheol (Table 1, entry 2), (1*R*,2*S*)-*trans*-2-phenyl-1-cyclohexanol (Table 1, entry 6), 2-methoxy-2-phenylethanol (Table 1, entry 8), 2-*tert*-butylcyclohexanol, mixture of isomers (Table 1, entry 9), (+)-*N,N*-Dicyclohexyl-(1*R*)-isoborneol-10-sulfonamide (Table 1, entry 10), (–)-menthol (Table 1, entry 11).

(b) *Synthesized through known protocols*:

-Isoborneol (Table 1, entry 3): W. Oppolzer, C. Chapis, *Tetrahedron Lett.*, 1984, **25**, 5383-5386.

-2,2-Diphenylcyclopentanol (Table 1, entry 4): E. Bacqué, J.-M. Paris, *Synth. Commun.*, 1992, **22**, 2259-2272.

-Spiro[4.5]decan-6-ol (Table 1, entry 5): R. D. Sands, *J. Org. Chem.*, 1994, **59**, 468-471.

-(1*S*,2*S*)-*N*-(1-hydroxy-1-phenylpropan-2-yl)ethanamide (Table 1, entry 7): H. Tlahuext, R. Contreras, *Tetrahedron: Asymmetry*, 1992, **3**, 727-730.

-(–)-8-Phenylmenthol (Table 1, entry 12 & Table 3, entry 1): O. Ort, *Org. Synth., Coll.*, 1993, **8**, 522; 1987, **65**, 203.

-(–)- and (+)- 8-(2-Naphthyl)menthol (Table 1, entries 13-14): T. Takahashi, N. Kurose, T. Koizumi, *Heterocycles*, 1993, **36**, 1601-1616.

-2-(2-Phenylpropan-2-yl)cyclohexanol (Table 2, entry 1 & Table 3, entry 2), 2-(2-(3,5-diisopropylphenyl)propan-2-yl)cyclohexanol (Table 2, entry 2), 2-(9*H*-xanthen-9-yl)cyclohexanol (Table 2, entry 3), 2-(9,10-dihydroanthracen-9-yl)cyclohexanol (Table 2, entry 4): D. L. Comins, J. M. Salvador, *J. Org. Chem.*, 1993, **58**, 4656-4661.

-2-(9*H*-Fluoren-9-yl)cyclopentanol (Table 2, entry 5), 2-(9*H*-fluoren-9-yl)cyclohexanol (Table 2, entry 6): T. Ohwada, *J. Am. Chem. Soc.*, 1992, **114**, 8818-8827.

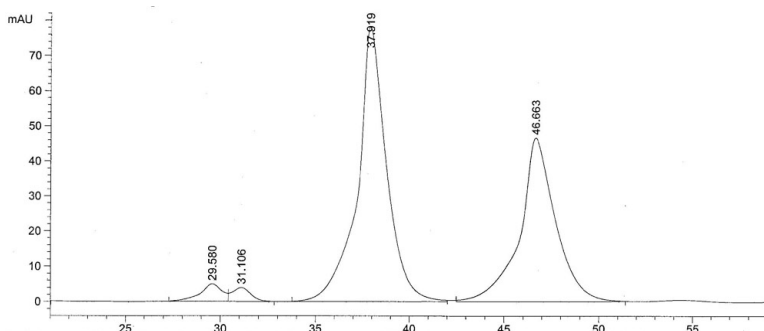
Validation of the ³¹P NMR assay (Scheme 3)

(a) Synthesis of ethyl (1-naphthyl) phosphinate. To a suspension of anilinum hypophosphite (0.382 g, 2.40 mmol, 1.20 equiv) and 3-aminopropyltriethoxysilane (0.531 g, 2.40 mmol, 1.20 equiv) in CH₃CN (12.0 ml) was added 1-bromonaphthalene (0.28 mL, 0.414 g, 2.0 mmol), Pd(OAc)₂ (9.0 mg, 0.040 mmol, 2 mol% Pd) and either, racemic BINAP or (*R*)-BINAP (28.8 mg, 0.044 mmol). The reaction mixture was heated at reflux for 8 h under N₂. After cooling to rt, ³¹P NMR analysis showed the product at ~28 ppm (100%, doublet). The mixture was diluted with EtOAc and washed with aq. HCl (2 M). The aqueous phase was extracted with EtOAc (3 x) and the combined organic phase was washed with saturated aq. NaHCO₃ (1 x) and brine (1 x), dried (MgSO₄) and concentrated to afford the pure title product (0.431 g, 98% yield). ¹H NMR (CDCl₃, 300 MHz) δ 8.43 (d, *J* = 8 Hz, 1 H), 8.09 (dd, *J* = 7, 1 Hz, 1 H), 8.07 (d, *J* = 7 Hz, 1 H), 7.94 (d, *J*_{HP} = 563 Hz, 1 H), 7.93 (d, *J* = 8 Hz, 1 H), 7.75 – 7.89 (m, 1 H), 7.48 – 7.69 (m, 2 H), 4.05 – 4.29 (m, 2 H), 1.37 (t, *J* = 7 Hz, 3 H); ³¹P NMR (CDCl₃, 121.47 MHz) δ 26.76 (dm, *J*_{PH} = 563 Hz).

(b) NMR assay: Derivatization with (*S*)-(-)-(α)-methylbenzylamine/CCl₄. To an NMR tube charged with a solution of ethyl (1-naphthyl) phosphinate (20 mg) in CCl₄ (1 mL) was added an excess of (*S*)-(-)-(α)-methylbenzylamine at rt. The solution was kept for 2-4 h at rt and then analyzed by ³¹P NMR to determine the corresponding de (de = ee) by the difference in heights and integrals.

From (*R*)-BINAP: ³¹P NMR (CDCl₃, 121.47 MHz) δ 20.94 (s, 60.9%, height: 155.7); 20.53 (s, 39.1%, height: 114.8); ee_(heights) = 15%. HPLC analysis: ee = 16%; Product1, *t*_R 37.919 min; Product2, *t*_R 46.663 min; conditions: (*S,S*)-Whelk-01 column (Regis® Technologies, 250 x 4.6 mm, 5 μm) with guard column (Agilent Zorbax® ODS, 4.6 x 12.5 mm, 5 μm), 1 mL/min (isocratic), rt, hexanes/*i*PrOH (7/3, v/v).

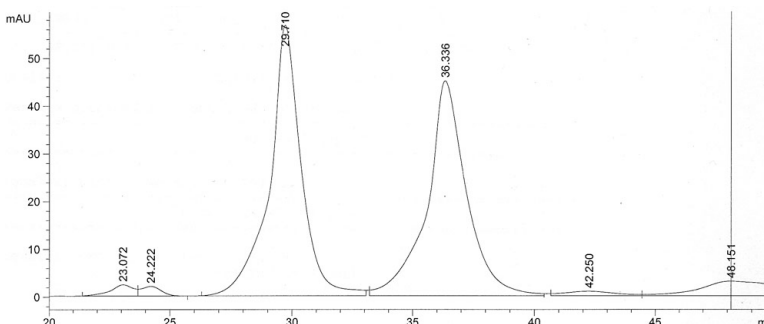
HPLC



From racemic BINAP: ³¹P NMR (CDCl₃, 121.47 MHz) δ 21.507 (s, height: 104.5); 21.413 (s, height: 110.0); ee_(heights) = 2.3%. HPLC analysis: ee = 1.9%; Product1, *t*_R 29.482 min; Product2, *t*_R 35.995 min; conditions: (*S,S*)-Whelk-01 Column (Regis® Technologies, 250 x 4.6 mm, 5 μm) with guard column (Agilent Zorbax® ODS, 4.6 x 12.5 mm, 5 μm), 1 mL/min (isocratic), rt, hexanes/*i*PrOH (7/3, v/v).

MS (ESI⁺) for C₁₂H₁₃O₂P, [M+H]⁺ *m/z* 221.1; [NpP(O)(OH)(H)+H]⁺ *m/z* 193; [NpP(O)+H]⁺ *m/z* 175; [Np+H]⁺ *m/z* 129.1 (Np = 1-naphthyl).

HPLC



Pd-Catalyzed asymmetric hydrophosphinylation (Table 4)

(a) Preparation of a stock solution of EtOP(O)H₂. This was conducted as described in references 1 and 2. In a typical procedure, a mixture of concentrated H₃PO₂ (100 mmol), (EtO)₂SiMe₂ (200 mmol) in the appropriate volume of reagent grade solvent (CH₃CN or toluene) to create a 0.50 M solution, is refluxed for 2 h under N₂. After cooling to rt, the stock solution was stored under N₂ at rt. Less than 10% decomposition was detected after 2 months.

(b) Preparation of Palladium(II) [*N,N'*-Bis(3,5-di-*tert*-butylsalicylidene)-(R,R)-1,2-cyclohexanediamine], [Pd(R,R)-Jacobsen] (Table 4, entries 1b-c).⁴ Prepared from PdCl₂/Et₃N according to the procedure described in reference 4. ¹H NMR (CDCl₃, 300 MHz) δ 7.71 (s, 2 H), 7.43 (s, 2 H), 6.98 (d, *J* = 2 Hz, 2 H), 3.41 (bs, 2 H), 2.59 (bs, 2 H), 1.89 (bs, 2 H), 1.10 – 1.75 (m, 40 H).

(c) General Procedure for the Pd-catalyzed asymmetric hydrophosphinylation. (Table 4). To a 0.50 M solution of EtOP(O)H₂ in CH₃CN or toluene (2.0 equiv) was added 1-octene (1.0 equiv) followed by the appropriate Pd-catalyst (2-3 mol%). In those cases where the Pd-complex was formed *in situ*, a 1/1 ratio of PdCl₂/ligand was used. The solution was heated at reflux for 8-12 h under N₂. After cooling to rt, the mixture was diluted with EtOAc and washed with 2 M aq. HCl (1 x), followed by extraction of the aqueous phase with EtOAc (2 x). The combined organic phase was washed with saturated aq. NaHCO₃ (1 x) and brine (1 x), dried (MgSO₄) and concentrated to yield the crude ethyl octyl-*H*-phosphinate product,^{5,6} which was used without further purification in the ³¹P NMR assay for enantiopurity determination. Note: the title product was isolated by column chromatography (hexanes/EtOAc, 3/1, v/v to EtOAc, 100%) as a colorless oil for identity confirmation.^{5,6} ¹H NMR (CDCl₃, 300 MHz) δ 7.08 (d, *J* = 526 Hz, 1 H), 4.03 – 4.23 (m, 2 H), 1.21 – 1.82 (m, 14 H), 1.37 (t, *J* = 7 Hz, 3 H), 0.88 (t, *J* = 7 Hz, 3 H); ³¹P NMR (CDCl₃, 121.47 MHz) δ 40.3 (d, *J*_{PH} = 526 Hz).

(d) ³¹P NMR assay: Derivatization with (*S*)-(-)-(α)-methylbenzylamine/CCl₄ (Table 4). To an NMR tube charged with a solution of the appropriate crude mixture of ethyl octyl-*H*-phosphinate (20 mg) in CCl₄ (1 mL) was added an excess of (*S*)-(-)-(α)-methylbenzylamine at rt. The solution was kept for 2-4 h at rt and then analyzed by ³¹P NMR to determine the corresponding de (de = ee) by the difference in heights and integrals.

Ethyl octyl-*H*-phosphinate (4, Table 4).

¹H NMR (CDCl₃, 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), 1.34 - 1.39 (t, *J* = 7 Hz, 3 H), 0.86 - 0.90 (t, *J* = 7 Hz, 3 H); ¹³C NMR (CDCl₃, 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; ³¹P NMR (CDCl₃, 121.47 MHz) δ 40.7 (dm, *J* = 530 Hz).

P-Chiral-*H*-phosphinate esters: kinetic resolution (Scheme 4)

Preparation of (1,1-Diethoxy-ethyl)-cinnamyl-phosphinic acid ethyl ester (Scheme 4, molecule 5).⁷ To cinnamyl *H*-phosphinic acid⁸ (5.0 mmol, 1.0 equiv) was added triethyl orthoacetate (5.5 mL, 30.0 mmol, 6.0 equiv) and boron trifluoride etherate (0.13 mL, 1.0 mmol, 0.20 equiv) at rt. The reaction mixture was vigorously stirred for 16-24 h at rt under N₂. The reaction mixture was diluted with EtOAc and washed with 0.50 M aq. NaHCO₃ (1 x) and brine (2 x). The organic layer was dried (MgSO₄), concentrated and purified by chromatography on SiO₂ (hexanes/EtOAc, 6/4, v/v) to afford the title product (1.30 g, 80% yield) as a light yellow oil. ¹H NMR (300 MHz, CDCl₃) δ 7.20 – 7.38 (m, 5 H, *aro* CH), 6.53 (dd, ³*J*_{H,H} = 16.0 Hz, ⁴*J*_{H,CCCP} = 4.0 Hz, 1 H, -CH=), 6.19 – 6.31 (m, 1 H, -CH=), 4.15 – 4.28 (m, 2 H, -CH₂-O), 3.60 – 3.83 (m, 4 H, -CH₂-O), 2.71 – 2.92 (m, 2 H, -CH₂-P), 1.54 (d, ³*J*_{H,CCP} = 11.0 Hz, 3 H, CH₃-C-P), 1.31 (t, *J* = 7.0 Hz, 3 H, CH₃-), 1.22 (t, *J* = 7.0 Hz, 3 H, CH₃-), 1.21 (t, ³*J*_{H,H} = 7.0 Hz, 3 H, CH₃-); ¹³C NMR (75.45 MHz, CDCl₃) δ 137.3, 134.8 (d, *J*_{PCCC} = 12.0 Hz), 128.8 (2 C), 127.7, 126.4 (d, *J*_{PCCCC} = 1.5 Hz, 2 C), 119.1 (d, *J*_{PCC} = 10.5 Hz), 101.5 (d, *J*_{PC} = 139.0 Hz), 62.1 (d, *J*_{POC} = 7.0 Hz), 58.5 (d, *J*_{POCC} = 5.0 Hz), 57.0 (d, *J*_{POC} = 7.0 Hz), 31.7 (d, *J*_{PC} = 82.5 Hz), 20.8 (d, *J*_{PCC} = 12.0 Hz), 17.0 (d, *J*_{POCC} = 5.0 Hz), 15.7, 15.5; ³¹P NMR (121.47 MHz, CDCl₃) δ 45.96 (s); HRMS (EI⁺) *m/z* calcd. for C₁₇H₂₇O₄P ([M-OEt]⁺), 281.1307, found 281.1302.

(1,1-Diethoxy-ethyl)-(2,3-dihydroxy-3-phenyl-propyl)-phosphinic acid ethyl ester (Scheme 4, molecule 6): 2 diastereoisomers 50/50.⁷

Enantioselective dihydroxylation.⁹ To a mixture of *t*-BuOH/H₂O (10.0 mL, 1/1, v/v) were added ADmix-α (1.40 g, 0.4 mol-% OsO₄) and methanesulfonamide (95.0 mg, 1.0 equiv) at rt. After a few minutes of stirring at rt, the mixture became homogeneous. The reaction mixture was cooled down to 0 °C and a solution of ester **5** (0.326 g, 1.0 mmol) in *t*-BuOH/H₂O (0.50 mL) was added. After 16 h at rt, the reaction mixture was cooled down to 0 °C and Na₂SO₃ (1.5 g) was added. After 1 h at rt, the reaction mixture was extracted with EtOAc (3 x). The combined organic phase was washed with 2.0 N aq. KOH (1 x) and with brine (1 x), dried (MgSO₄), concentrated and purified by chromatography on SiO₂

(hexanes/EtOAc, 1/9, v/v to EtOAc/MeOH, 98/2, v/v) to afford the enantioenriched dihydroxylated compound **6** (0.326 g, 91% yield) as a colorless oil. $[\alpha]_D = +0.675^\circ$ (c 1.85, absolute EtOH, 24.9 °C).

Racemic dihydroxylation. To a solution of ester **5** (0.652 g, 2.0 mmol) in acetone (4.0 mL) was added *N*-methylmorpholine *N*-oxide (0.70 g, 6.0 mmol, 3.0 equiv) followed by a 0.050 M aq. solution of osmium tetroxide (0.40 mL, 0.020 mmol, 1 mol-%) at rt. After 17 h at rt, the reaction mixture was cooled down to 0 °C and Na₂SO₃ (3.0 g) was added. After 1 h at rt, the reaction mixture was diluted with brine and extracted with EtOAc (3 x). The combined organic phase was dried (MgSO₄), concentrated and purified by chromatography on SiO₂ (hexanes/EtOAc, 1/9, v/v to EtOAc/MeOH, 98/2, v/v) to afford the racemic dihydroxylated compound **6** (0.555 g, 77% yield) as a colorless oil. $[\alpha]_D = +0.248^\circ$ (c 1.85, absolute EtOH, 24.9 °C).

¹H NMR (300 MHz, CDCl₃) δ 7.28 – 7.41 (m, 5 H, *aro* CH), 4.55 (m, 1 H + 0.5 H, -CH-O), 4.08 – 4.28 (m, 2 H + 0.5 H, -CH₂-O & -CH-O), 3.46 – 3.73 (m, 4 H, -CH₂-O), 1.68 – 2.14 (m, 2 H, -CH₂-P), 1.45 & 1.39 (2 d, $J_{\text{HCCP}} = 11.5$ Hz, 3 H, CH₃-), 1.32 & 1.29 (2 t, $J = 7.0$ Hz, 3 H, CH₃-), 1.12 – 1.23 (m, 6 H, CH₃-); ¹³C NMR (75.45 MHz, CDCl₃) δ 140.3 & 140.2, 128.4 (2 C), 128.1 & 128.0, 127.1 & 127.0 (2 C), 101.1 (d, $J_{\text{PC}} = 144.0$ Hz) & 100.9 (d, $J_{\text{PC}} = 142.0$ Hz), 78.0 & 77.8 (2 d, $J_{\text{PCCC}} = 12.0$ Hz), 71.3 (d, $J_{\text{PCC}} = 5.0$ Hz) & 70.5 (d, $J_{\text{PCC}} = 5.5$ Hz), 62.2 & 61.9 (2 d, $J_{\text{POC}} = 7.0$ Hz), 58.4 (d, $J_{\text{PCOC}} = 5.5$ Hz), 57.9 & 57.8 (2 d, $J_{\text{PCOC}} = 7.0$ Hz), 29.4 (d, $J_{\text{PC}} = 83.0$ Hz) & 29.3 (d, $J_{\text{PC}} = 84.5$ Hz), 20.0 & 19.8 (2 d, $J_{\text{PCC}} = 12.5$ Hz), 16.6 (d, $J_{\text{POCC}} = 5.5$ Hz), 15.4, 15.2; ³¹P NMR (121.47 MHz, CDCl₃) δ 50.81 (s, 50%), 49.87 (s, 50%); HRMS (Ammonium Chem Ion) *m/z* calcd. for C₁₇H₂₉O₆P, ([M-OEt]⁺), 315.1361, found 315.1369.

(2,3-Dihydroxy-3-phenyl-propyl)-H-phosphinic acid (Scheme 4, molecule 7). To a solution of the ester **6** (0.150 g, 0.420 mmol) in EtOH (5.0 mL, 0.10 M) was added Amberlite IR120+ (1.25 g, 3.0 g/mmol). After 16 h at reflux, the reaction mixture was cooled down to rt and filtered through cotton. The filtrate was concentrated in vacuo to afford the deprotected product **7** (0.930 g, 100% yield) as a colorless wax. ¹H NMR (300 MHz, D₂O) δ 7.18 – 7.30 (m, 5 H, *aro* CH), 6.82 (ddd, $J_{\text{HP}} = 563.0$ Hz, $J = 3.5, 1.0$ Hz, 1 H, H-P), 4.43 (d, $J = 6.0$ Hz, 1 H, -CH-O), 4.03 (tdd, $J_{\text{HCCP}} = 10.5$ Hz, $J = 6.0, 3.5$ Hz, 1 H, -CH-O), 1.56 – 1.86 (m, 2 H, -CH₂-P); ¹³C NMR (75.45 MHz, D₂O) δ 139.8, 128.8 (2 C), 128.5, 127.2 (2 C), 77.4 (d, $J_{\text{PCCC}} = 14.0$ Hz), 70.3 (d, $J_{\text{PCC}} = 4.0$ Hz), 33.4 (d, $J_{\text{PC}} = 93.5$ Hz); ³¹P NMR (121.47 MHz, D₂O) δ 29.77 (ddd, $J_{\text{PH}} = 550.0$ Hz, $J_{\text{PCH}} = 18.5$ Hz, $J_{\text{PCHH}} = 12.0$ Hz); HRMS (ESI⁺) *m/z* calcd. for C₉H₁₃O₄P, ([M+H]⁺), 217.0629, found 217.0633.

C-Chiral H-phosphinic acids: asymmetric benzylation (Equations 3 & 4)

Benzylation of H₃PO₂ with (R)-1-(2-naphthyl)ethanol (Equation 3).¹⁰ To a solution of (R)-(+)-1-(2-naphthyl)ethanol (0.344 g, 2.0 mmol, 1.0 equiv)¹¹ and concentrated H₃PO₂ (0.264 g, 4.0 mmol, 2.0 equiv) in *t*-AmylOH (0.20 M, 10.0 mL) were added Pd₂dba₃ (18.3 mg, 0.020 mmol, 1 mol%) and xantphos (25.5 mg, 0.0440 mmol, 2.2 mol%) at rt. The flask was equipped with a Dean-Stark trap (prefilled with *t*-AmylOH) and the reaction mixture was heated at reflux for 16 h under N₂. After cooling down to rt, the reaction was filtered and concentrated in vacuo. The residue was dissolved in EtOAc and extracted with 0.50 M aq. NaHCO₃ (3 x). The aqueous layer was acidified to pH 1 with 10% aq. HCl and extracted with EtOAc (3 x). The combined organic phase was washed with brine (1 x), dried (MgSO₄) and concentrated to afford a white solid [0.440 g, 100% yield, 89 : 11 (**8** : **9**, branched : linear)].

Hydrophosphinylation of 2-vinylnaphthalene with H₃PO₂ (Equation 4).¹⁰ To a solution of 2-vinylnaphthalene (0.318 g, 2.0 mmol, 1.0 equiv) and concentrated H₃PO₂ (0.264 g, 4.0 mmol, 2.0 equiv) in *t*-AmylOH (0.20 M, 10.0 mL) were added Pd₂dba₃ (0.0183 g, 0.020 mmol, 1 mol%) and xantphos (25.5 mg, 0.0440 mmol, 2.2 mol%) at rt. The flask was equipped with a Dean-Stark trap (prefilled with *t*-AmylOH) and the reaction mixture was heated at reflux for 16 h under N₂. After cooling down to rt, the reaction was filtered and concentrated in vacuo. The residue was dissolved in EtOAc and extracted with 0.50 M aq. NaHCO₃ (3 x). The aqueous layer was acidified to pH 1 with 10% aq. HCl and extracted with EtOAc (3 x). The combined organic phase was washed with brine (1 x), dried (MgSO₄) and concentrated to afford a white solid [0.315 g, 72% yield, 78 : 22 (**8** : **9**, branched : linear)].

1-(2-Naphthyl)ethyl-H-phosphinic acid (Branched isomer, molecule 8). ¹H NMR (CDCl₃, 300 MHz) δ 7.25 – 7.81 (m, 7 H), 6.88 (d, $J_{\text{HP}} = 548.0$ Hz, 1 H), 3.21 (dq, $J_{\text{HCP}} = 24.5$ Hz, $J = 7.5$ Hz, 1 H), 1.59 (d, $J_{\text{HCCP}} = 19.0$ Hz, $J = 7.5$ Hz, 3 H); ¹³C NMR (CDCl₃, 75.45 MHz) δ 133.4 (d, $J_{\text{PCCCC}} = 5.5$ Hz), 133.3 (d, $J_{\text{PCC}} = 7.5$ Hz), 132.6 (d, $J_{\text{PCCCC}} = 2.5$ Hz), 128.3 (d, $J_{\text{PCCCC}} = 2.5$ Hz), 127.8, 127.6, 127.4 (d, $J_{\text{PCCC}} = 8.0$ Hz), 126.7 (d, $J_{\text{PCCC}} = 5.0$ Hz), 126.2, 126.0, 40.6 (d, $J_{\text{PC}} = 89.5$ Hz), 12.4; ³¹P NMR (CDCl₃, 121.47 MHz) δ 41.10 (d, $J_{\text{PH}} = 548.0$ Hz); HRMS (EI⁺) *m/z* calcd. for C₁₂H₁₃O₂P, (M⁺) 220.0653, found 220.0657.

2-(2-Naphthyl)ethyl-H-phosphinic acid (Linear isomer, molecule 9). ¹H NMR (CDCl₃, 300 MHz) δ 7.25 – 7.81 (m, 7 H), 7.00 (d, $J_{\text{HP}} = 548.0$ Hz, 1 H), 2.94 – 3.04 (m, 2 H), 2.00 – 2.11 (m, 2 H); ³¹P NMR (CDCl₃, 121.47 MHz) δ 37.39 (d, $J_{\text{PH}} = 548.0$ Hz); HRMS (EI⁺) *m/z* calcd. for C₁₂H₁₃O₂P, (M⁺) 220.0653, found 220.0657.

Determination of the enantioselectivity of the benzylation reaction.¹⁰ 1-(2-Naphthyl)ethyl-*H*-phosphinic acid (5.0 mg) was dissolved in CDCl₃ (0.50 mL) and (*R*)-(+)-(α)-methylbenzylamine (2 drops) were added. The solution was kept for a few minutes at rt and then analyzed by ³¹P NMR. The enantiomeric excess was determined by height and by integration.

³¹P NMR data (CDCl₃ + (*R*)-(+)-(α)-methylbenzylamine, 300 MHz):¹⁰

| Starting material | Linear isomer, % | Branched isomer Integration percent, height, shift | ee, % |
|---|------------------|---|-------|
| Racemic 1-(2-Naphthyl)ethanol | 10 | 43%, 124.4, 30.53 ppm 46%, 124.6, 30.33 ppm | 3 |
| <i>R</i> -(+)-1-(2-Naphthyl)ethanol ¹¹ | 11 | 10%, 15.7, 30.48 ppm 79%, 125.7, 30.25 ppm | 77 |
| 2-Vinylnaphthalene | 22 | 38%, 65.6, 28.84 ppm 41%, 71.4, 29.67 ppm | 3 |

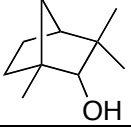
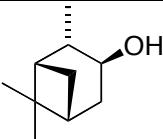
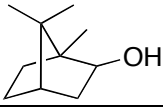
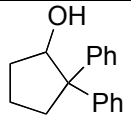
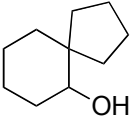
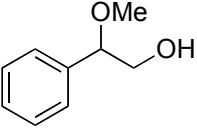
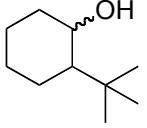
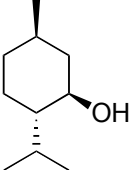
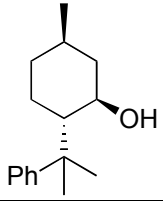
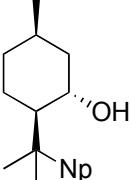
Determination of the absolute configuration: Preparation of 1-(2-naphthyl)ethylphosphonic acid.^{10,12} To the chiral 1-(2-naphthyl)ethyl-*H*-phosphinic acid (**8**) (0.227 g, 1.030 mmol, 1.0 equiv) in DMF (0.15 M, 7.0 mL) were added Pd₂dba₃ (9.40 mg, 0.01030 mmol, 1 mol%) and xantphos (12.40 mg, 0.0216 mmol, 2.2 mol%) at rt. The resulting solution was heated at 110 °C, opened to air for 23 h. After cooling down to rt, the reaction was filtered and concentrated in vacuo. The residue was dissolved in EtOAc and extracted with 0.50 M aq. NaHCO₃ (3 x). The aqueous layer was acidified to pH 1 with 10% aq. HCl and extracted with EtOAc (3 x). The combined organic phase was washed with brine (1 x), dried (MgSO₄) and concentrated to afford a white solid (0.147 g, 61%, 89 : 11, branched : linear). ¹H NMR (CDCl₃, 300 MHz) δ 7.75 – 7.84 (m, 4 H), 7.43 – 7.48 (m, 3 H), 3.15 (dq, $J_{HCP} = 21.5$ Hz, $J = 7.5$ Hz, 1 H), 1.49 (dd, $J_{HCCP} = 17.5$ Hz, $J = 7.5$ Hz, 3 H); ¹³C NMR (DMSO-*d*₆, 75.45 MHz) δ 138.8 (d, $J_{PCC} = 8.0$ Hz), 133.6, 132.5, 128.2 (d, $J_{PCCC} = 5.0$ Hz), 128.1, 128.0, 127.8, 127.2 (d, $J_{PCCC} = 8.0$ Hz), 126.6, 126.0, 39.6 (d, $J_{PC} = 124.0$ Hz), 16.8 (d, $J_{PCC} = 4.5$ Hz); ³¹P NMR (DMSO-*d*₆, 121.47 MHz) δ 25.99 (s, 11%), 25.65 (s, 89%); HRMS (EI⁺) *m/z* calcd. for C₁₂H₁₃O₃P, (M⁺) 236.0602, found 236.0598. [α]_D = -11.106° (c 0.9, MeOH).

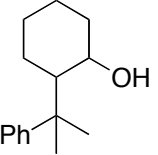
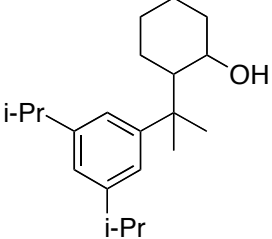
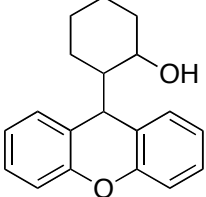
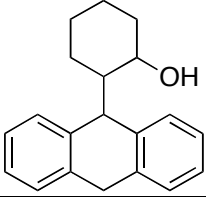
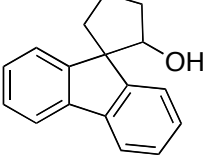
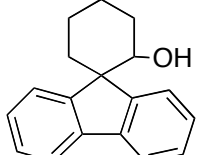
Comparing the specific rotation of the 1-(2-naphthyl)ethylphosphonic acid and the literature data ([α]_D = -3.7 (c 1.2, MeOH) for the (*R*)-1-phenylethylphosphonic acid,¹³ the absolute configuration (*S*) was assigned to 1-(2-naphthyl)ethyl-*H*-phosphonic acid. Therefore the reaction proceeds with overall inversion of configuration.

References

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- ¹¹Purchased from Aldrich, R/S \geq 98.5/1.5.
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SI Table.^a Hypophosphite esters ROP(O)H₂ **1**: ³¹P-NMR data.

| Alcohol ROH | Hypophosphite ROP(O)H ₂ ³¹ P-NMR | solvent |
|---|---|-----------------------------------|
|  | 16.4 (td, $J = 566, 12$ Hz) | CH ₃ CN |
|  | 19.5 (td, $J = 566, 12$ Hz) | CH ₃ CN |
|  | 14.2 (td, $J = 570, 21$ Hz) | CH ₃ CN |
|  | 13.7 (td, $J = 567, 10$ Hz) | CH ₃ CN |
|  | 13.2 (td, $J = 563, 11$ Hz) | CH ₃ CN |
|  | 17.4 (tt, $J = 570, 12$ Hz) | CH ₃ CN |
|  | <i>Trans</i> : 11.7 (ddd, $J = 572, 568, 10$ Hz) <i>Cis</i> : 10.5 (ddd) | CH ₃ CN |
|  | 10.4 (td, $J = 570, 10$ Hz) 11.3 (td, $J = 563, 11$ Hz) | CH ₃ CN cyclohexane |
|  | 13.1 (td, $J = 569, 10$ Hz) 17.0 (td, $J = 569, 10$ Hz) | CH ₃ CN cyclohexane |
|  | 19.9 (td, $J = 570, 8$ Hz) | CH ₃ CN |

| | | |
|---|--|--------------------|
|  | <i>Trans</i> : 10.7 (td, $J = 570, 10$ Hz) | CH ₃ CN |
|  | <i>Trans</i> : 12.6 (td, $J = 569, 10$ Hz) | CH ₃ CN |
|  | <i>Trans</i> : 13.7 (td, $J = 570, 9$ Hz) | CH ₃ CN |
|  | <i>Trans</i> : 13.7 (td, $J = 572, 9$ Hz) | CH ₃ CN |
|  | <i>Trans</i> : 16.2 (ddd, $J = 567, 568, 9$ Hz) | CH ₃ CN |
|  | <i>Trans</i> : 17.9 (ddd, $J = 566, 565, 10$ Hz) | CH ₃ CN |
| EtOP(O)H ₂ | 16.2 (tt, $J = 565, 10$ Hz) | CH ₃ CN |

^a Hypophosphite esters (alkyl phosphinates) ROP(O)H₂ cannot be isolated as pure compounds. Instead, they are prepared in solution (Scheme 2) and used *in situ*.

SD III-157 AFTER 2H AT 0C DECOUPLED

EXP2 PULSE SEQUENCE: STD13C

DATE 08-26-02

SOLVENT CDCL3

FILE P25

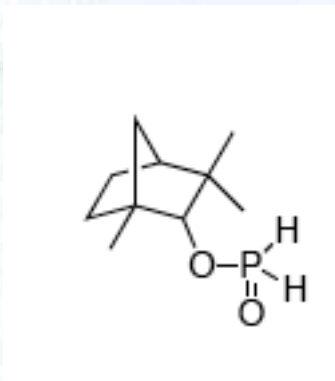
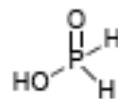
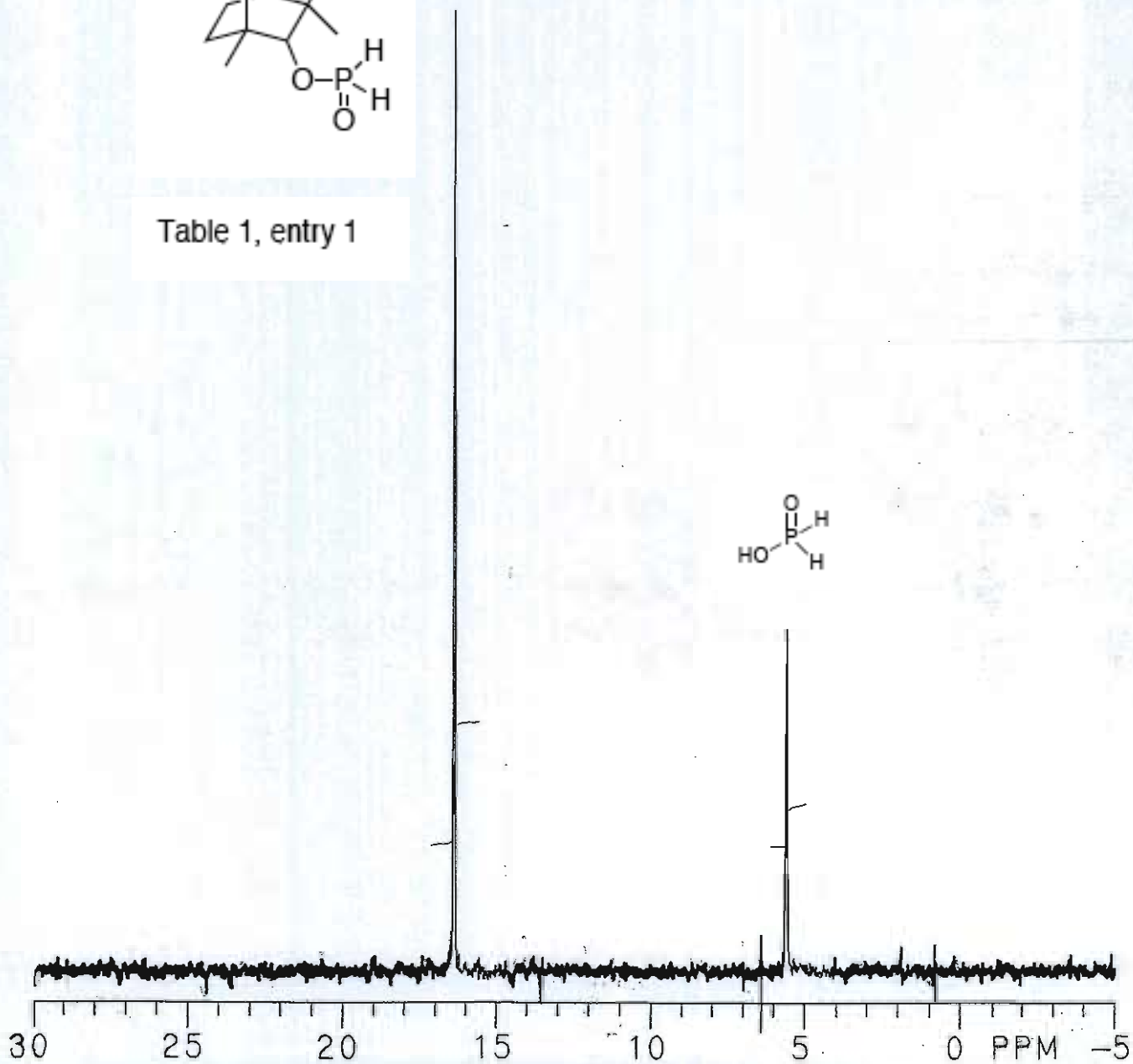


Table 1, entry 1



EXP2 PULSE SEQUENCE: STD13C
DATE 08-26-02
SOLVENT CDCL3
FILE P25

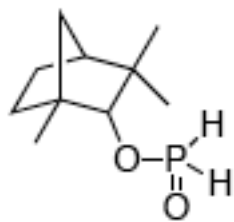
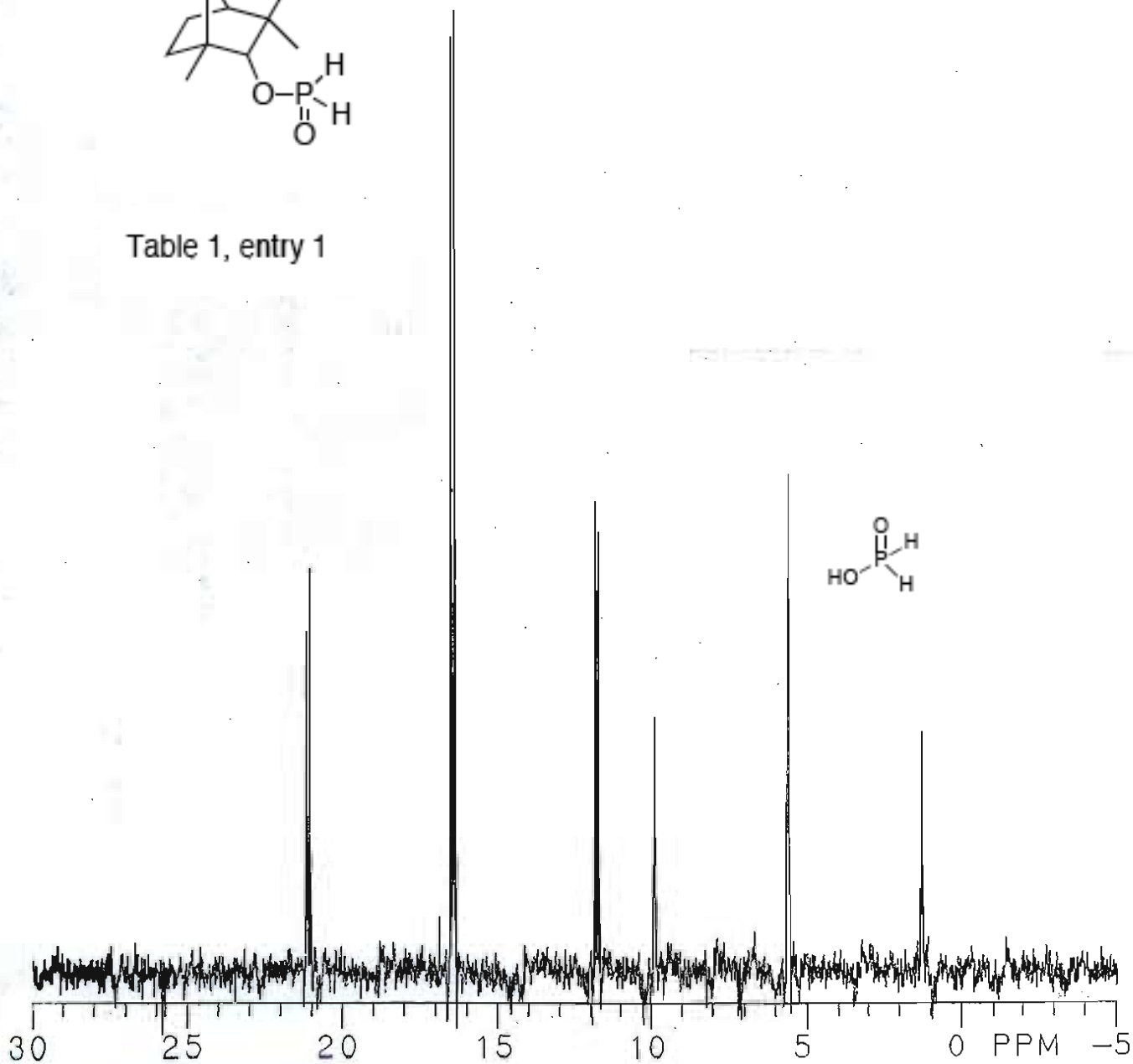
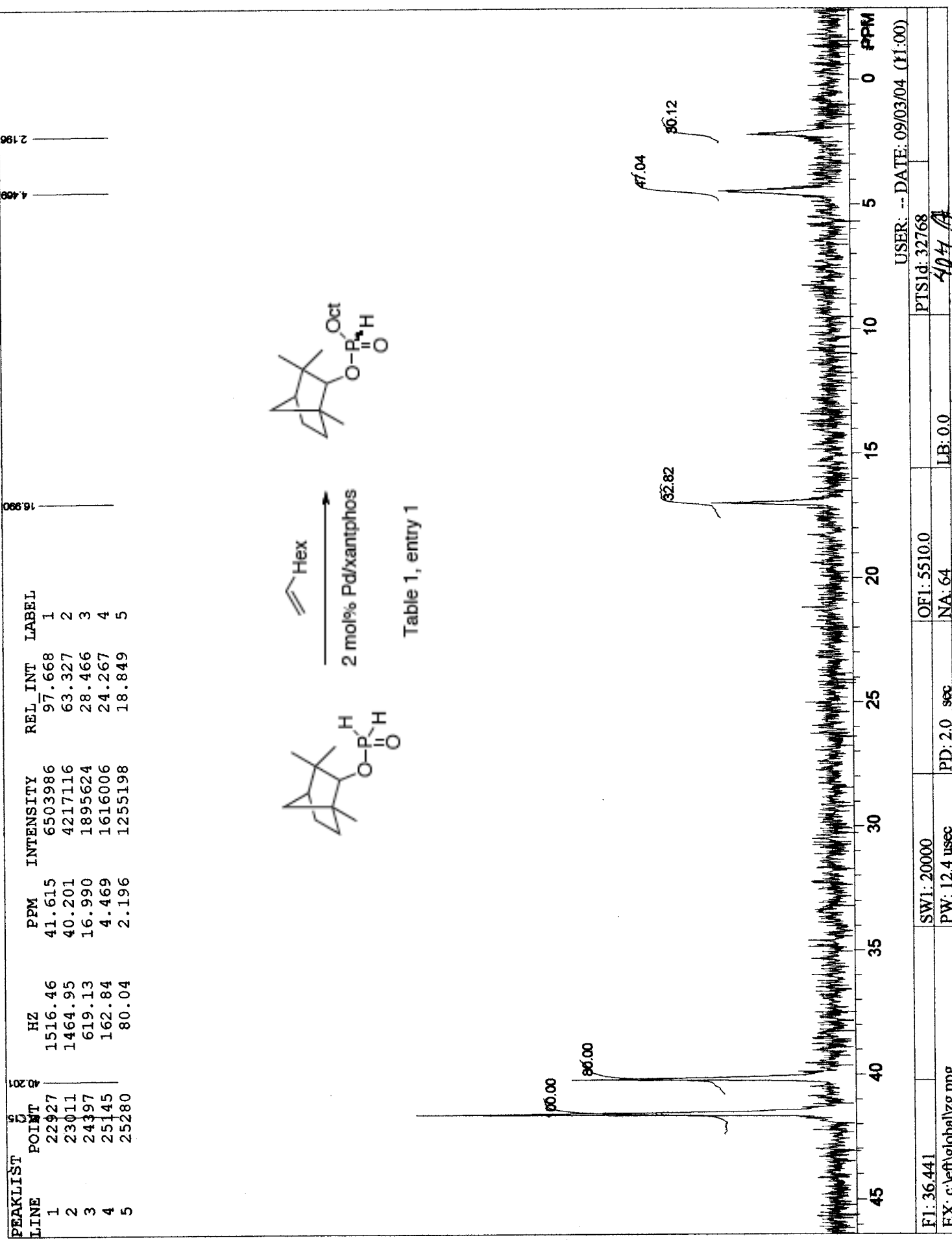


Table 1, entry 1





INDEX 1 FREQUENCY PPM HEIGHT
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 date Sep CDC13 gain not used
 solvent file spin 20
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 SW 26738.0 pw90 18.300
 at 1.598 alfa 20.000
 np 85476
 fb 14800 il
 bs 64 in n
 ss 4 dp y
 d1 1.000 hs nn
 nt 32 lb 2.00
 Ct TRANSMITTER P31 fn not used
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 tof 10608.2 wp 10010.0
 tpwr 55 rfl 2437.3
 pw 7.117 rfp 171.5
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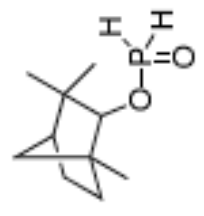
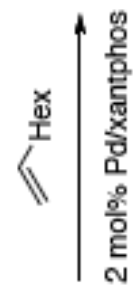
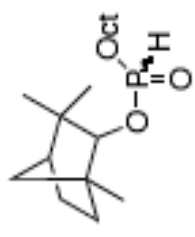
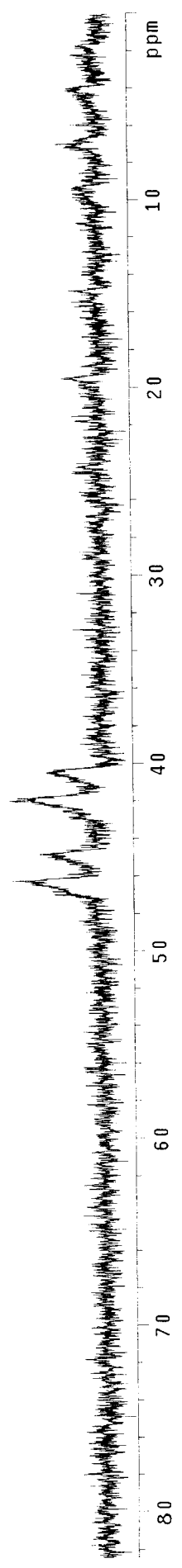


Table 1, entry 1



SD III-160 AFTER 2H AT RT DECOUPLED

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DATE 08-26--02

SOLVENT CDCL3

FILE P25

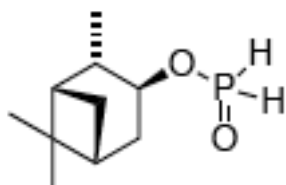
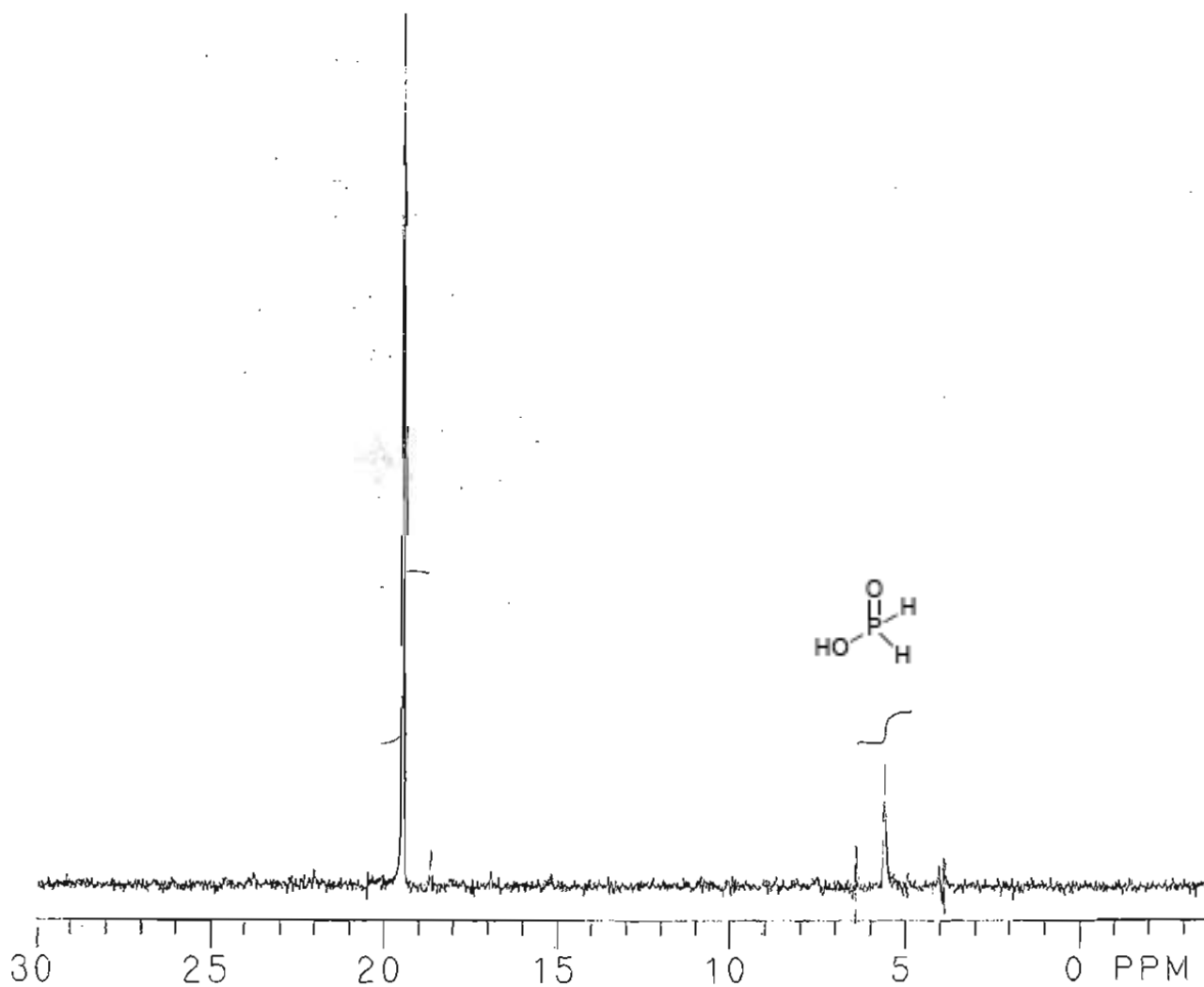


Table 1, entry 2



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EXP2 PULSE SEQUENCE: STD13C

DATE 08-26-02

SOLVENT CDCL3

FILE P25

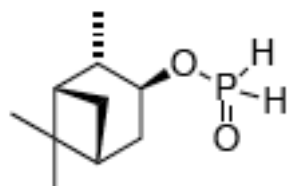
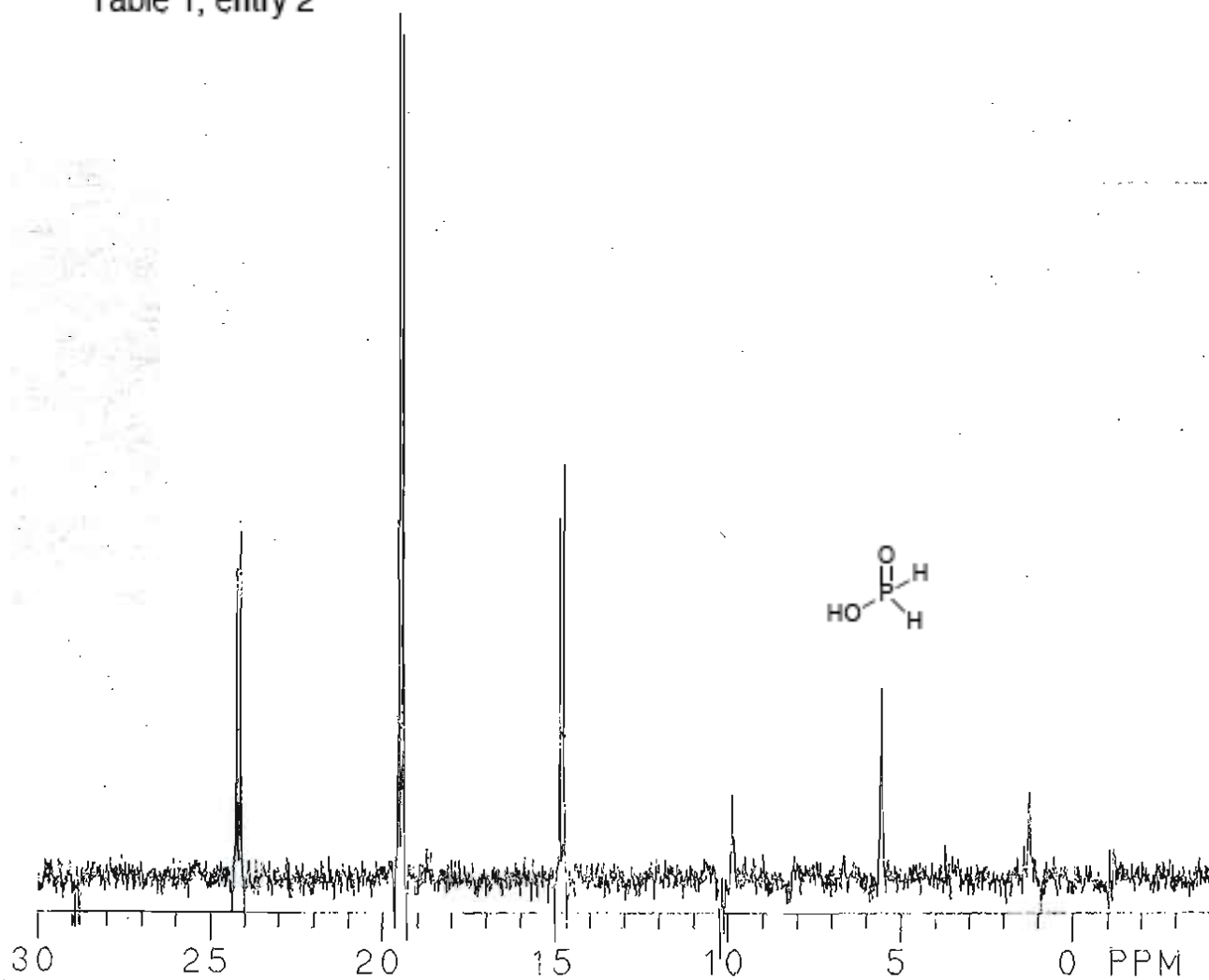


Table 1, entry 2



6.002
3.051
2.378
1.541

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| 1 | 23061 | 1434.67 | 39.370 | 11970211 | 56.988 | 1 |
| 2 | 23200 | 1349.79 | 37.041 | 24363698 | 115.992 | 2 |
| 3 | 23526 | 1150.49 | 31.572 | 872073 | 4.152 | 3 |
| 4 | 25053 | 218.71 | 6.002 | 5897143 | 28.075 | 4 |
| 5 | 25229 | 111.17 | 3.051 | 6584051 | 31.346 | 5 |
| 6 | 25269 | 86.67 | 2.378 | 5494614 | 26.159 | 6 |
| 7 | 25319 | 56.16 | 1.541 | 1152599 | 5.487 | 7 |

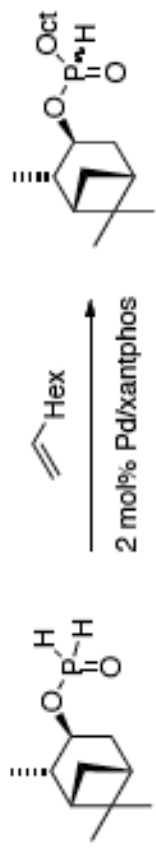
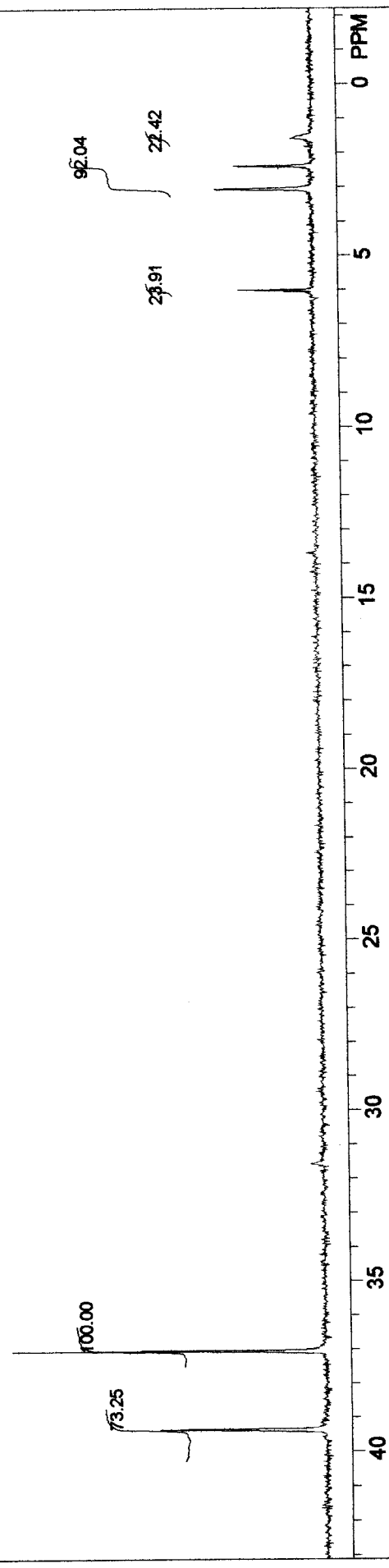


Table 1, entry 2



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 FI: 36.441
 EX: c:\eff\global\zg.ppg

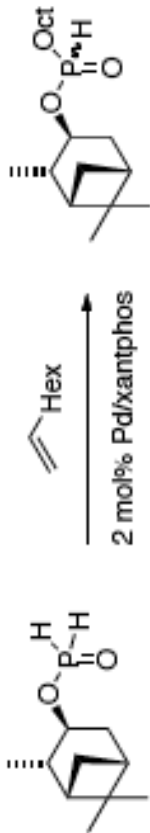
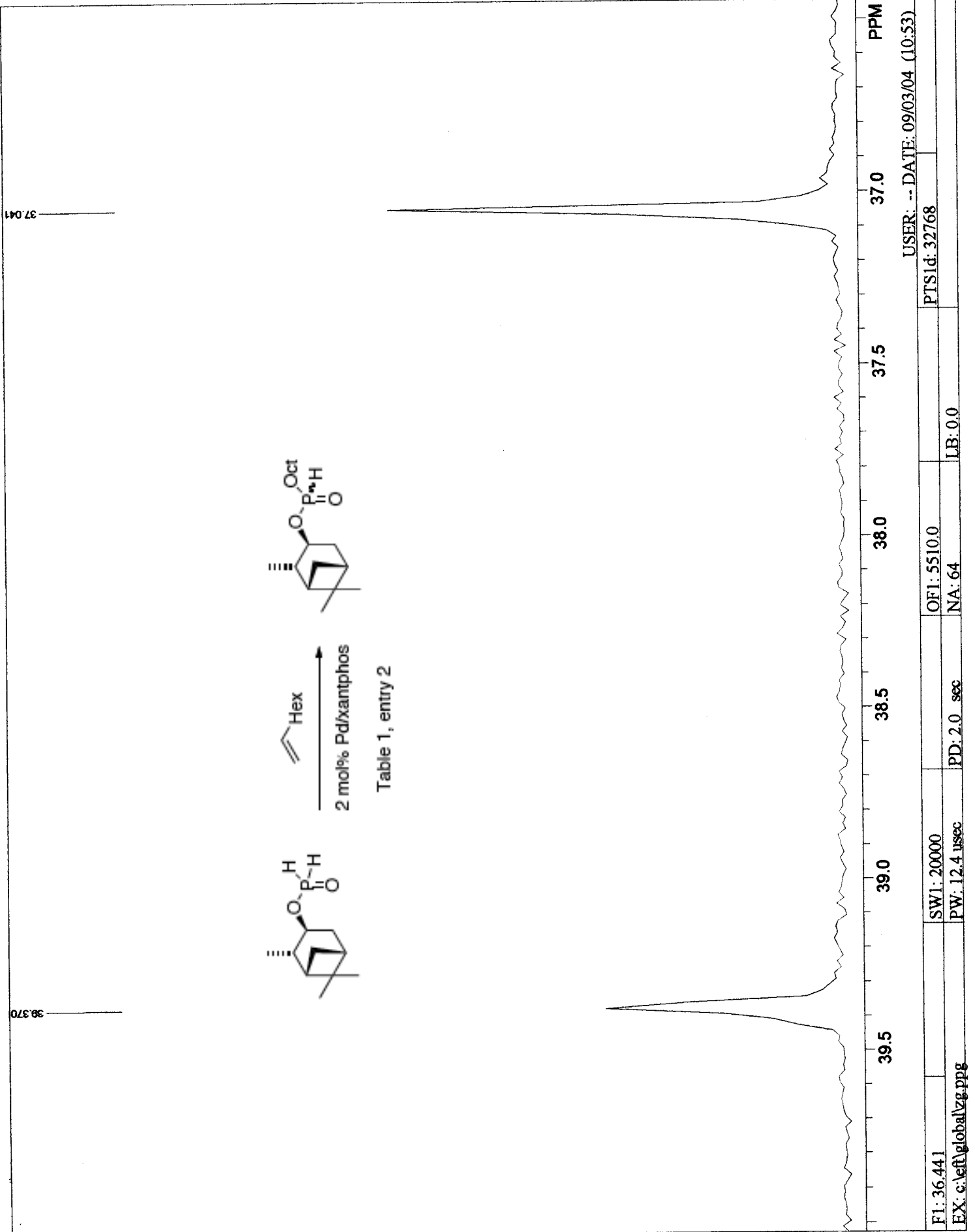


Table 1, entry 2



| | | | | |
|--------------------------|---------------|-------------|--------------|---------------------------------|
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| EX: c:\efl\global\zg.ppg | PW: 12.4 usec | PD: 2.0 sec | LB: 0.0 | |

| INDEX | FREQUENCY | PPM | HEIGHT |
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| 1 | 5340.157 | 43.965 | 16.0 |
| 2 | 5326.286 | 43.851 | 29.8 |
| 3 | 5313.230 | 43.744 | 30.3 |
| 4 | 5300.175 | 43.636 | 18.7 |
| 5 | 5050.485 | 41.581 | 21.6 |
| 6 | 5036.614 | 41.466 | 40.5 |
| 7 | 5025.190 | 41.372 | 38.6 |
| 8 | 5011.318 | 41.258 | 22.2 |
| 9 | 4794.268 | 39.471 | 31.5 |
| 10 | 4782.436 | 39.374 | 29.7 |
| 11 | 4767.749 | 39.253 | 18.7 |
| 12 | 4519.284 | 37.207 | 21.4 |
| 13 | 4505.820 | 37.096 | 40.2 |
| 14 | 4493.988 | 36.999 | 40.5 |
| 15 | 4480.525 | 36.888 | 22.2 |
| 16 | 1336.969 | 11.007 | 24.0 |
| 17 | 1128.079 | 9.287 | 52.5 |
| 18 | 856.358 | 7.050 | 29.8 |
| 19 | 845.343 | 6.960 | 31.1 |
| 20 | 754.769 | 6.214 | 18.9 |
| 21 | 649.508 | 5.347 | 23.2 |
| 22 | 606.669 | 4.995 | 102.7 |
| 23 | 222.751 | 1.834 | 27.5 |
| 24 | 212.144 | 1.747 | 27.9 |
| 25 | 119.530 | 0.984 | 18.7 |
| 26 | 84.851 | 0.699 | 46.9 |

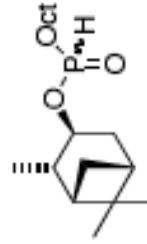
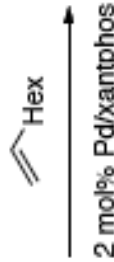
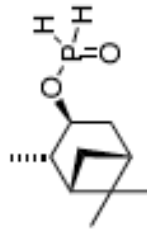
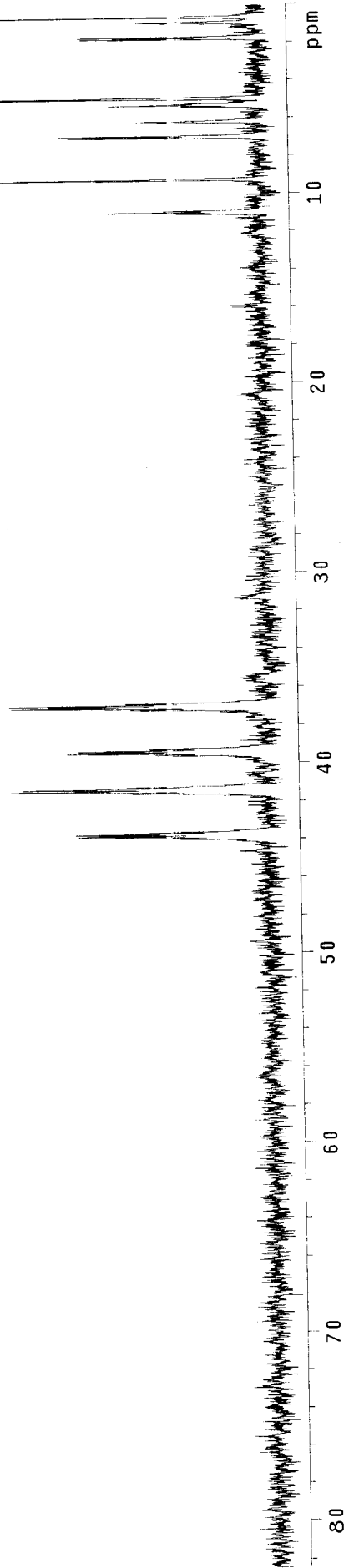


Table 1, entry 2

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 ss 4
 d1 1.000
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 ct 16
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 tof 55
 tpwr 7.117
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 dpwr 6700
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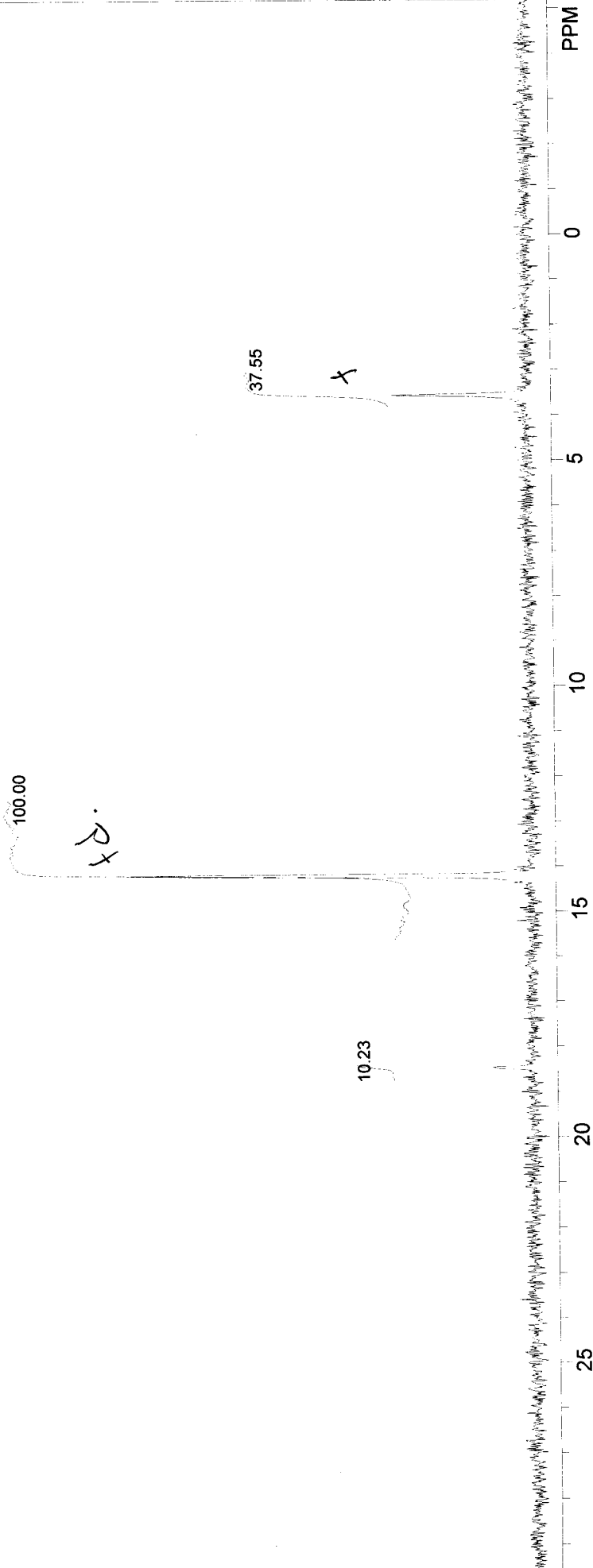


Alkylphosphonate

| LINE | POINT | HZ | PPM | INTENSITY | REL_INT | LABEL |
|------|--------|--------|--------|-----------|---------|-------|
| 1 | 243.0 | 671.98 | 18.441 | 1227650 | 10.084 | 1 |
| 2 | 245.65 | 516.31 | 14.169 | 11823153 | 97.119 | 2 |
| 3 | 251.99 | 129.58 | 3.556 | 4226777 | 34.720 | 3 |



Table 1, entry 3

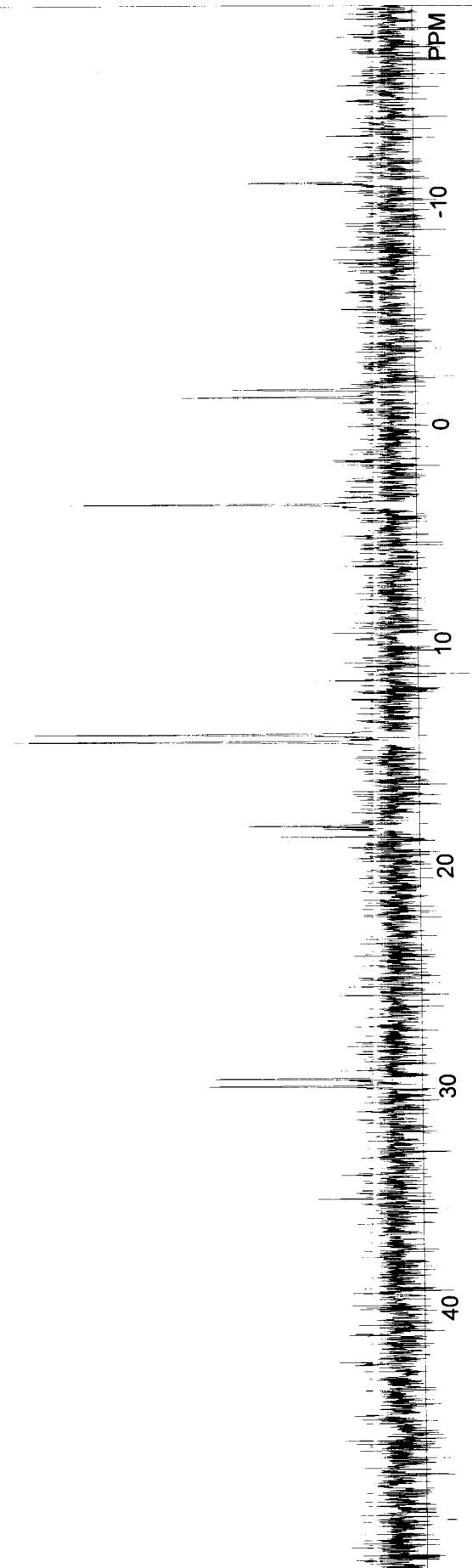


USER: -- DATE: 01/10/05 (19:47)
PTSId: 32768
484c -2h
OF1: 5510.0
NA: 16
LB: 0.0
PD: 2.0 sec
SW1: 20000
PW: 12.4 usec
F1: 36.441
EX: c:\left\global\zg.ppg

| PEAK LINE# | HZ | PPM | INTENSITY | REL INT | LABEL |
|------------|---------|---------|-----------|---------|-------|
| 1 19 | 1094.47 | 30.034 | -91387 | -3.249 | 1 |
| 2 47 | 1076.61 | 29.544 | 1138219 | 40.468 | 2 |
| 3 98 | 679.42 | 18.645 | -252489 | -8.977 | 3 |
| 4 31 | 659.19 | 18.089 | 933707 | 33.197 | 4 |
| 5 52 | 524.48 | 14.393 | 284930 | 10.130 | 5 |
| 6 37 | 503.74 | 13.824 | -20081 | -0.714 | 6 |
| 7 98 | 130.13 | 3.571 | 2220780 | 78.957 | 7 |
| 8 98 | -53.10 | -1.457 | 183817 | 6.535 | 8 |
| 9 09 | -59.65 | -1.637 | 1038531 | 36.924 | 9 |
| 10 66 | -399.79 | -10.971 | 517024 | 18.382 | 10 |



Table 1, entry 3



USER: -- DATE: 01/10/05 (19:50)
 PTSId: 32768
 OF1: 5510.0
 NA: 16
 LB: 0.0
 SW1: 20000
 PD: 2.0 sec
 EX: vzg.ppg
 PW: 12.4 usec

484a *Allyl, 1mm*

| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 4864.034 | 40.045 | 24.6 |
| 2 | 4846.899 | 39.904 | 24.0 |
| 3 | 4782.228 | 38.454 | 126.0 |
| 4 | 826.983 | 6.809 | 103.9 |

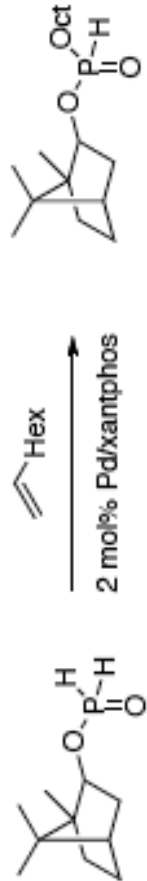


Table 1, entry 3

484a
 exp1 s2pul
 SAMPLE
 date Jan 11 2005 temp not used
 solvent CDC13 gain not used
 file exp spin 20
 ACQUISITION hst 0.008
 at 26738.0 pw90 18.300
 1.598 aifa 20.000
 85476 il
 14800 in n
 64 in n
 4 dp y
 1.000 hs nn
 8
 TRANSMITTER 8 lb
 P31 fn
 121.474 sp
 10608.2 wp 10010.0
 55 rfl 2437.3
 7.117 rfp 0
 DECOUPLER H1 lp -125.8
 H1 lp -302.0
 dof 0
 dm YYY wc 250
 dmm w sc 0
 dpwr 35 vs 24
 dmf 6700 th ai no ph 6



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 4864.034 | 40.045 | 24.6 |
| 2 | 4846.899 | 39.904 | 24.0 |
| 3 | 4792.228 | 39.454 | 126.0 |

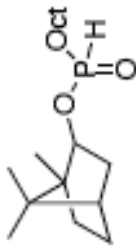
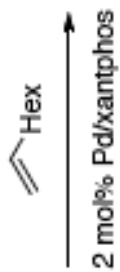
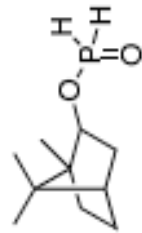
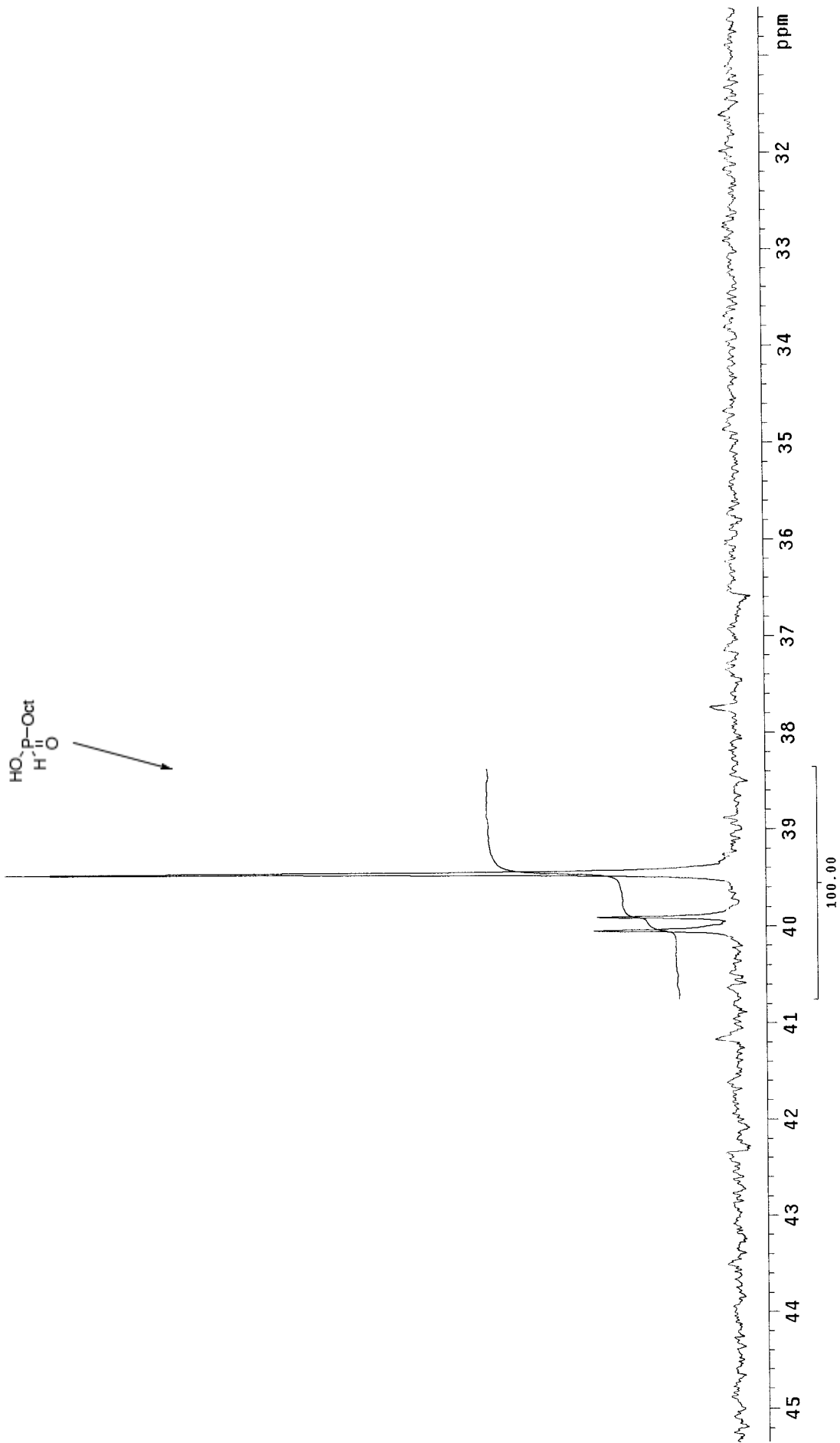


Table 1, entry 3

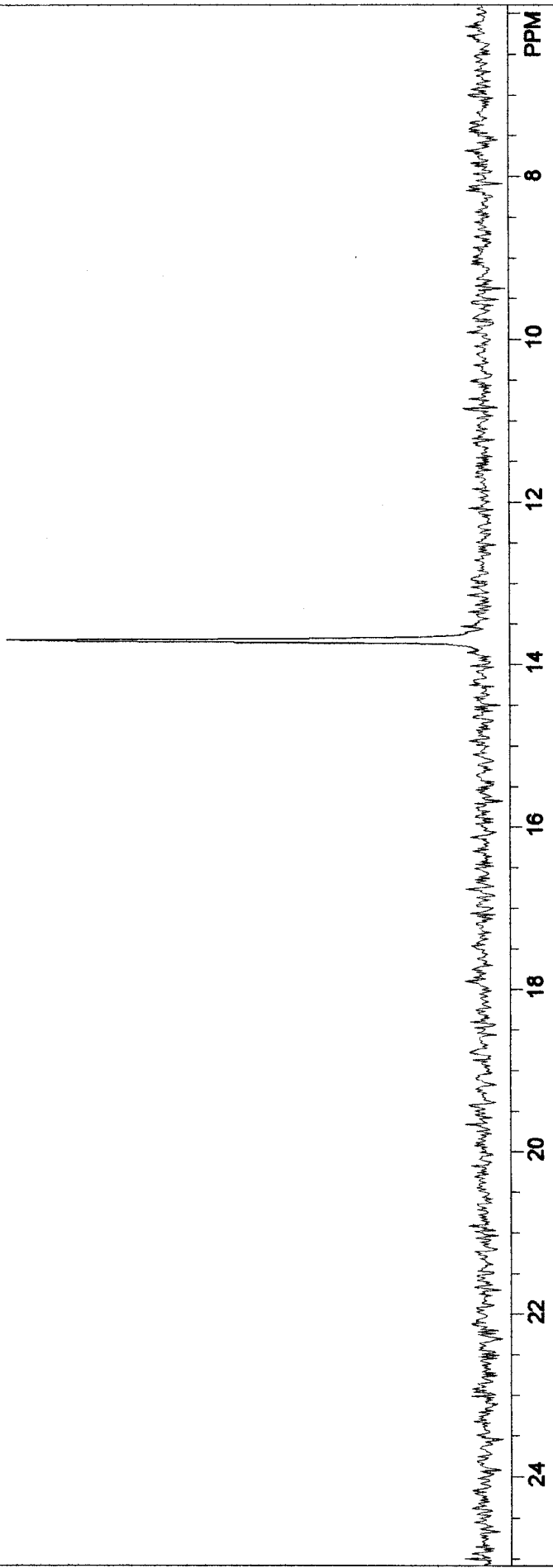


Interpolated Peak Listing

| PEAK | POINT | HEIGHT | REL. HT | HZ | PPM |
|------|-------|--------|---------|--------|--------|
| 1 | 24594 | 5484K | 105.65 | 498.73 | 13.686 |



Table 1, entry 4



| | | | | |
|---------------------------|---------------|-------------|--------------|--------------------------------|
| F1: 36.441 | SW1: 20000 | OF1: 5510.0 | PTS1d: 32768 | USER: --DATE: 09/08/04 (21:21) |
| EX: c:\left\global\zg.ppg | PW: 12.4 usec | NA: 16 | LB: 0.0 | 405 A - 2.5 h m f |

Interpolated Peak List

| PEAK | POINT | HEIGHT | REL. HT | HZ | PPM |
|------|-------|--------|---------|---------|--------|
| 1 | 23658 | 805367 | 53.13 | 1070.26 | 29.370 |
| 2 | 23674 | 707678 | 46.68 | 1060.33 | 29.097 |
| 3 | 24587 | 1405K | 92.71 | 503.26 | 13.810 |
| 4 | 24604 | 1487K | 98.10 | 492.88 | 13.526 |
| 5 | 25516 | 560840 | 37.00 | -64.18 | -1.761 |
| 6 | 25533 | 856394 | 56.49 | -74.16 | -2.035 |

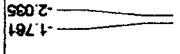
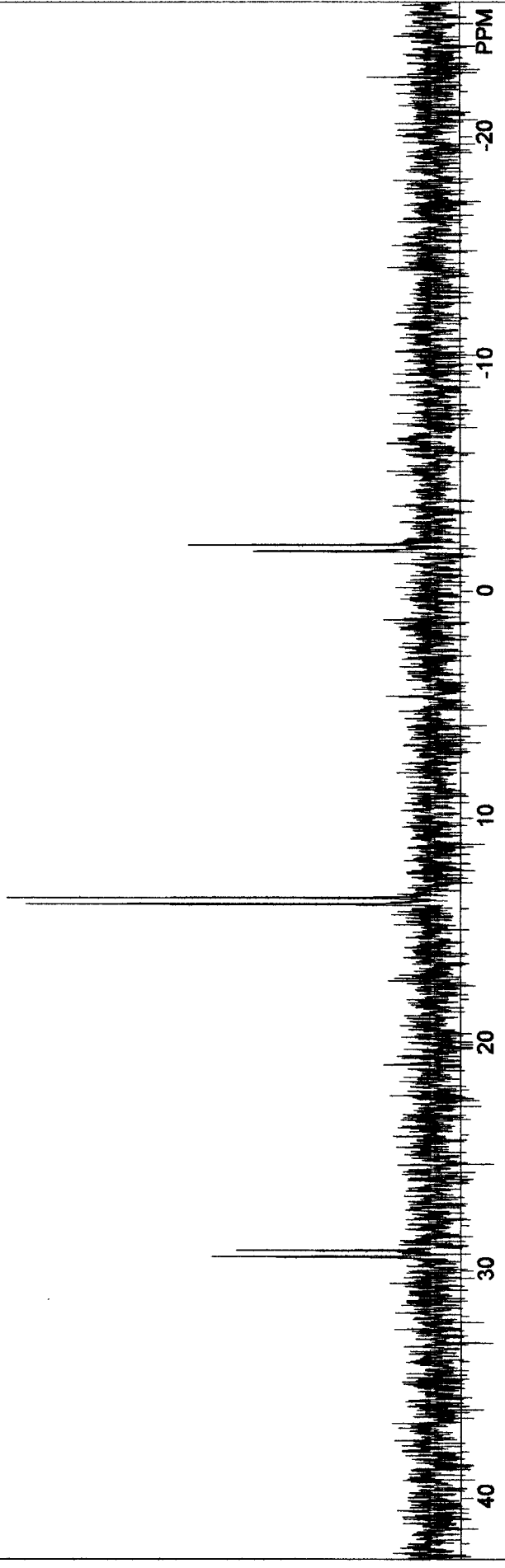
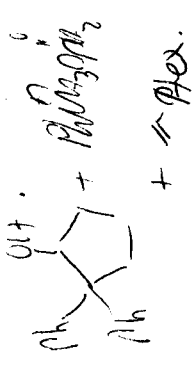


Table 1, entry 4



| | | | |
|--------------------------|---------------|-------------|---------------------------------|
| FI: 36.441 | SW1: 20000 | OF1: 5510.0 | USER: -- DATE: 09/08/04 (21:23) |
| EX: c:\efl\global\zg.ppg | PW: 12.4 usec | NA: 16 | PTSId: 32768 |
| | PD: 2.0 sec | LB: 0.0 | |



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 5234.080 | 43.092 | 18.6 |
| 2 | 4567.834 | 37.607 | 30.5 |
| 3 | 1928.145 | 15.874 | 62.5 |
| 4 | 1023.634 | 8.428 | 62.5 |
| 5 | 848.606 | 6.987 | 33.6 |
| 6 | 703.770 | 5.794 | 23.6 |
| 7 | 506.712 | 4.172 | 87.8 |

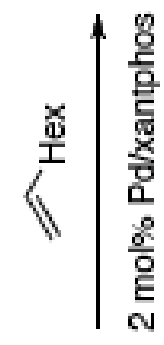
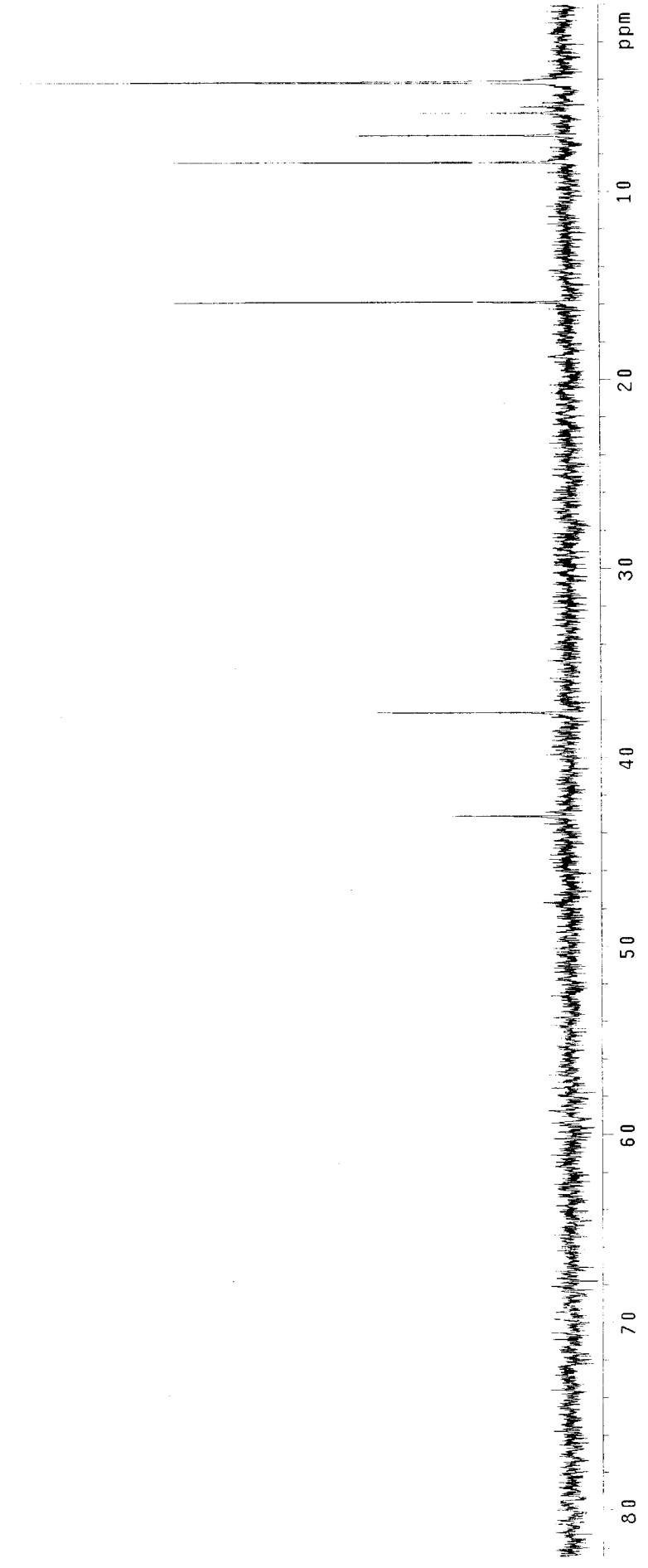


Table 1, entry 4

405a
 exp1 szpu1
 SAMPLE g 2004 temp not used
 Sep CDC13 gain not used
 solvent exp spin 20
 file hst 0.008
 ACQUISITION pw90 18.300
 at 26738.0 pw90 20.000
 1.598 a1fa
 np 85476
 fb 14800 il
 bs 64 in n
 ss 4 dp y
 d1 1.000 hs
 nt 16
 ct TRANSMITTER 16 lb fn
 P31 not used
 tn 121.474 sp
 sfrq 10608.2 wp 10010.0
 tof 55 rfl 2437.3
 tpwr 7.117 rfp
 pw DECOUPLER H1 lp
 dn 0
 dof yyy wc 250
 dm w SC 0
 dmm 35 vs 42
 dpwr 6700 th ai no ph 12
 dmf



PEAKLIST

| LINE | POINT | HZ | PPM | INTENSITY | REL. INT | LABEL |
|------|-------|--------|--------|-----------|----------|-------|
| 1 | 24625 | 479.97 | 13.171 | 3777654 | 96.238 | 1 |
| 2 | 25379 | 20.17 | 0.553 | 27217 | 0.693 | 2 |

0.553

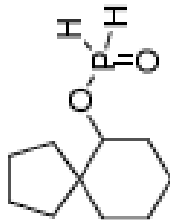
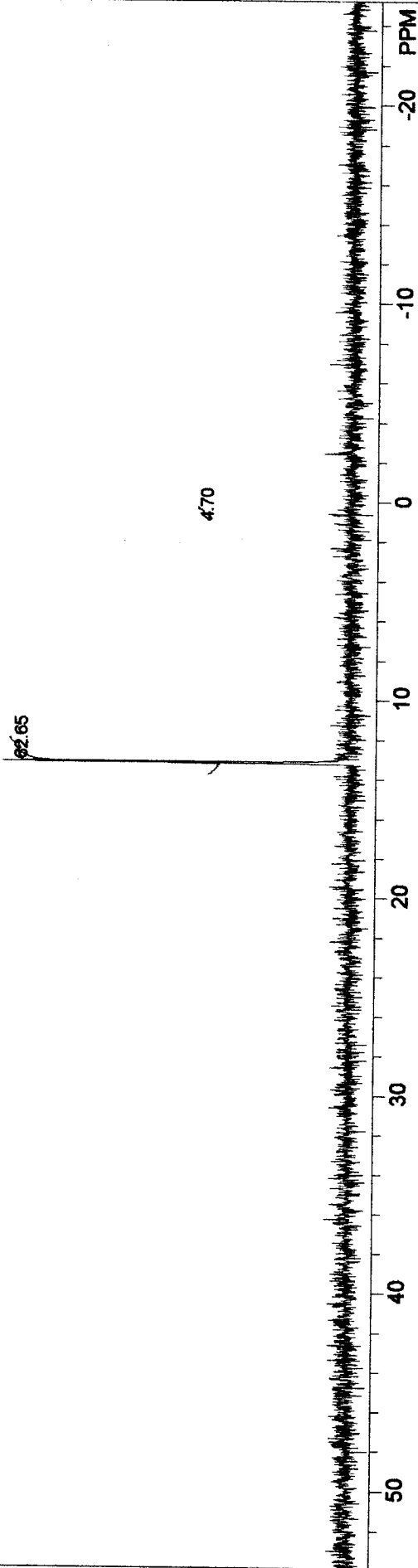


Table 1, entry 5



| | | | |
|--------------------------|---------------|-------------|---------------------------------|
| F1: 36.441 | SW1: 20000 | OF1: 5510.0 | USER: -- DATE: 09/13/04 (14:09) |
| EX: c:\efi\global\zg.ppg | PW: 12.4 usec | PD: 2.0 sec | PTS1d: 32768 |
| | | NA: 16 | LB: 0.0 |
| | | | 7/015 |

Intpolated Peak Listing

| PK | POINT | HEIGHT | REL. HT | HZ | PPM |
|----|-------|--------|---------|---------|--------|
| 1 | 23696 | 602107 | 50.01 | 1046.82 | 28.727 |
| 2 | 23714 | 543174 | 45.12 | 1035.93 | 28.428 |
| 3 | 24619 | 1156K | 96.01 | 483.52 | 13.269 |
| 4 | 24637 | 1109K | 92.09 | 472.59 | 12.969 |
| 5 | 25542 | 739063 | 61.39 | -79.60 | -2.184 |
| 6 | 25560 | 577178 | 47.94 | -90.47 | -2.483 |

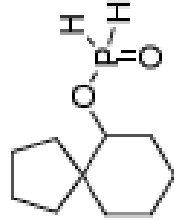
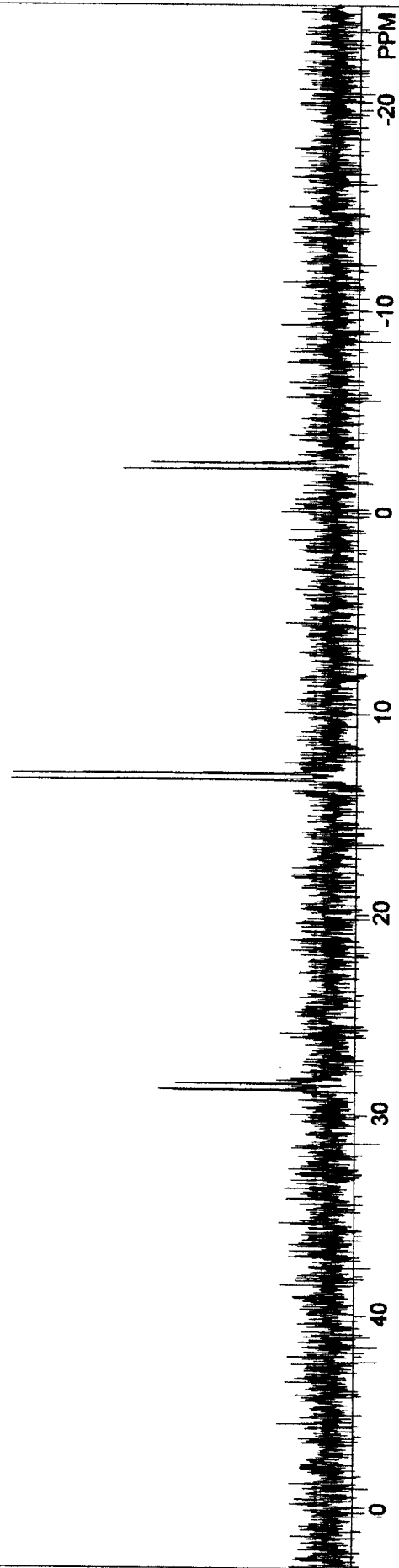
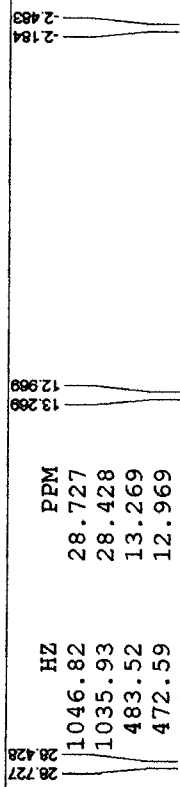
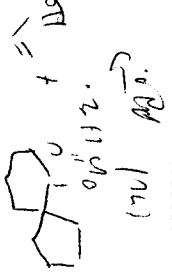


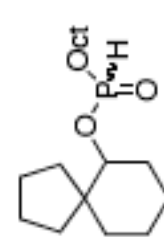
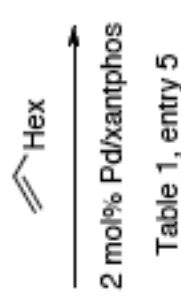
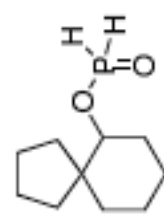
Table 1, entry 5



F1: 341 SW1: 20000 OF1: 5510.0 USER: -- DATE: 09/13/04 (14:10)
 EX: d:\global\vg.ppg PW: 12.4 usec PD: 2.0 sec NA: 16 LB: 0.0 PTS1d: 32768
 Y/1/07

7h (7u)  + \Rightarrow 110x
 0/112.
 (7u) \Rightarrow 110x

| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 5163.090 | 42.508 | 53.1 |
| 2 | 4474.405 | 36.838 | 82.4 |



4108
 exp1 s2pu1

SAMPLE
 date Sep 14 2004
 solvent CDC13
 file exp
 ACQUISITION
 sw 26738.0
 at 1.598
 np 85478
 fb 14800
 bs 64
 ss 4
 di 1.000
 nt 8
 ct 8

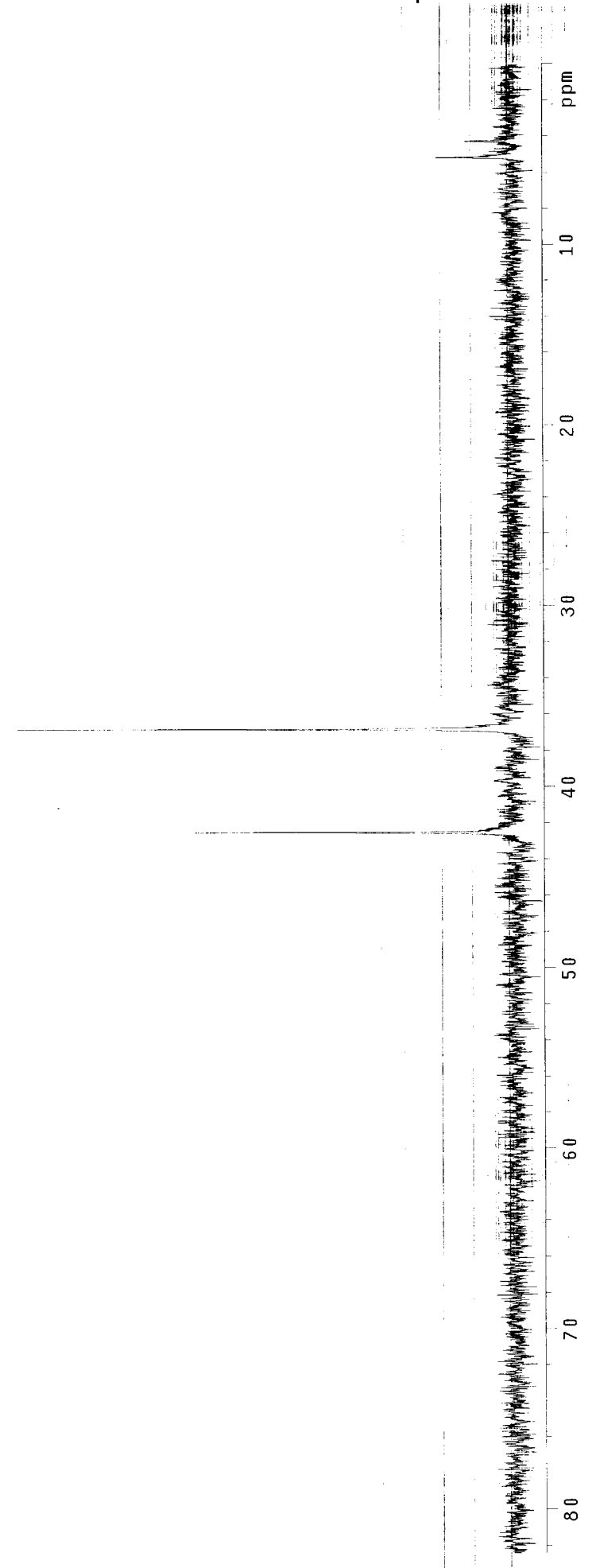
TRANSMITTER P31
 tn 121.474
 tof 10608.2
 tpwr 55
 pw 7.117

DECOUPLER H1
 dn 0
 dof VVY
 dm W
 dmm 35
 dpwr 6700
 dmf

SPECIAL
 temp not used
 gain not used
 spin 20
 hst 0.008
 pw90 18.300
 alfa 20.000

FLAGS
 n n
 y y
 nn nn
 PROCESSING 2.00
 not used
 DISPLAY
 sp 10010.0
 wp 2437.3
 rfl 0
 rfp -148.5
 lp -208.4

PLOT
 wc 250
 sc 0
 vs 30
 th 17
 at no ph



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 5163.090 | 42.508 | 53.1 |
| 2 | 4474.405 | 36.838 | 82.4 |

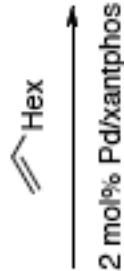
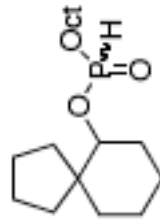
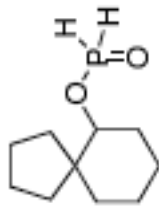
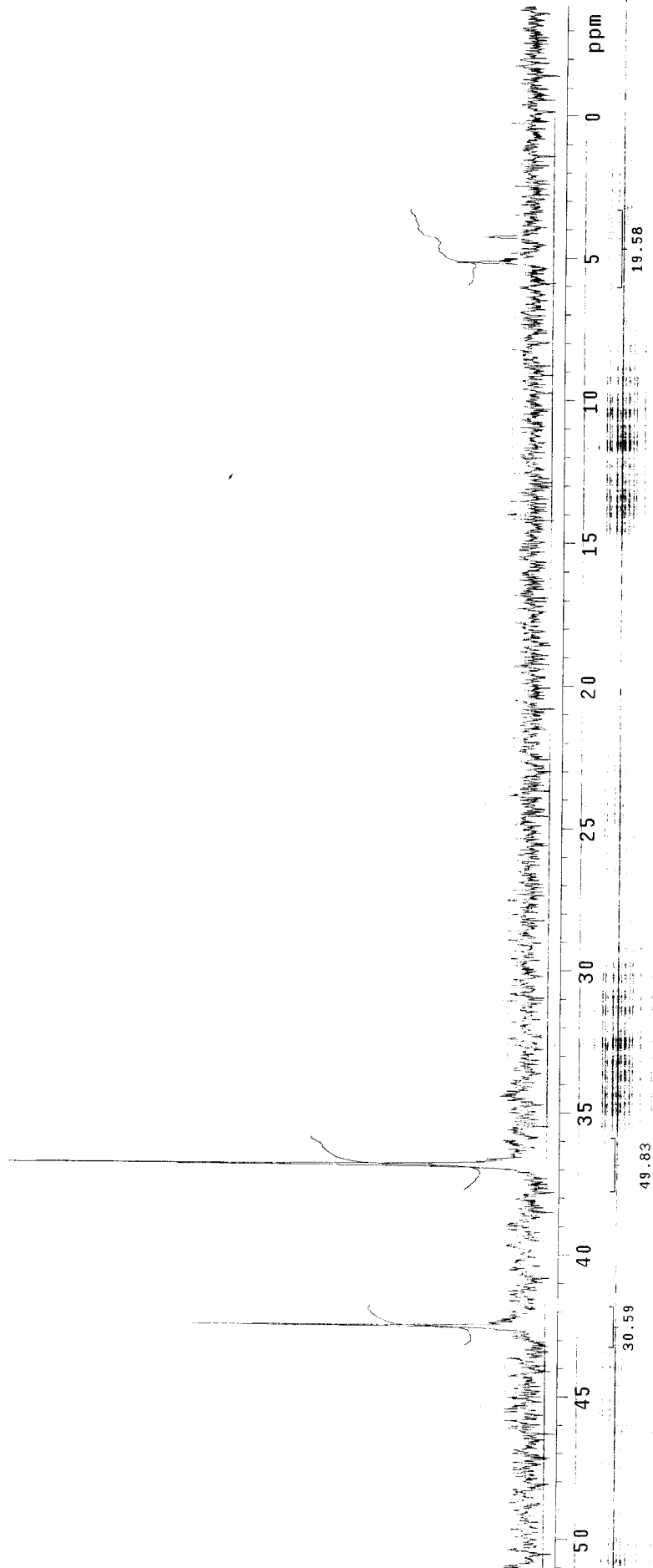


Table 1, entry 5



INDEX FREQUENCY PPM HEIGHT
 1 -2437.337 -20.067 0.0

410B
 exp1 s2pu1

| | | | |
|-------------|-------------|------|----------|
| date | Sep 14 2004 | temp | not used |
| solvent | CDC13 | gain | not used |
| file | | exp | 20 |
| ACQUISITION | | | |
| sw | 26738.0 | pw90 | 0.008 |
| at | 1.598 | alfa | 18.300 |
| np | 85476 | il | 20.000 |
| fb | 14800 | in | n |
| bs | 64 | in | n |
| ss | 4 | dp | y |
| nt | 1.000 | hs | nn |
| ct | 8 | lb | 2.00 |
| TRANSMITTER | | | |
| fn | P31 | fn | not used |
| sfrq | 121.474 | sp | 0 |
| tof | 10608.2 | wp | 10010.0 |
| tpwr | 55 | rfl | 2437.3 |
| pw | 7.117 | rfp | 0 |
| DECOUPLER | | | |
| dn | H1 | lp | -64.5 |
| dof | 0 | lp | -288.8 |
| dm | yinn | wc | 250 |
| dmm | w | sc | 0 |
| dpwr | 35 | vs | 31 |
| dmf | 6700 | th | 11 |
| | | ai | no |
| | | ph | |

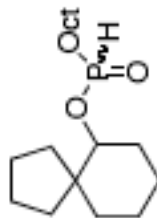
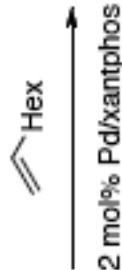
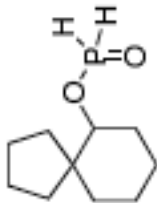
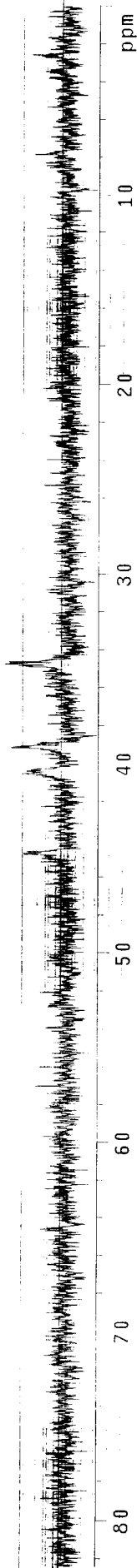
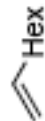
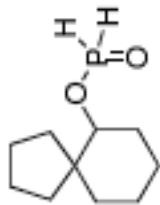


Table 1, entry 5

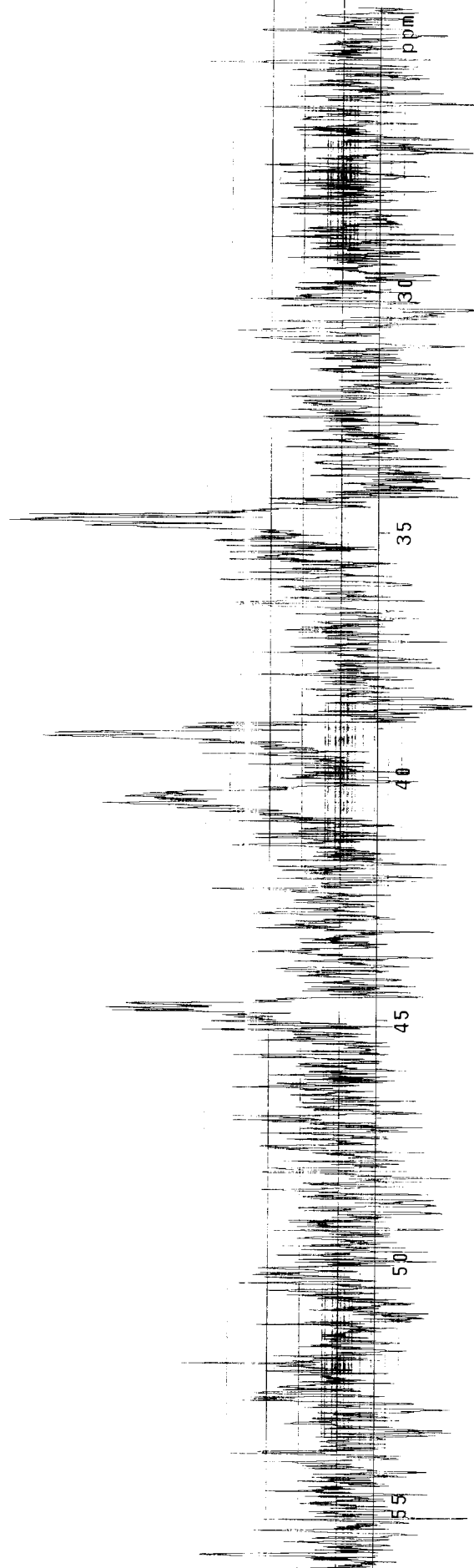
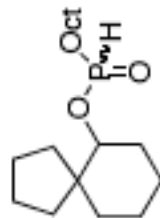


| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 5436.035 | 44.755 | 37.8 |
| 2 | 4925.641 | 40.553 | 38.4 |
| 3 | 4758.773 | 39.179 | 48.1 |
| 4 | 4222.676 | 34.765 | 53.4 |
| 5 | 4208.396 | 34.648 | 50.2 |

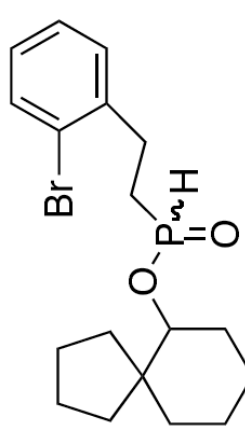
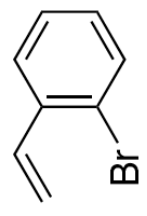


2 mol% Pd/xantphos

Table 1, entry 5

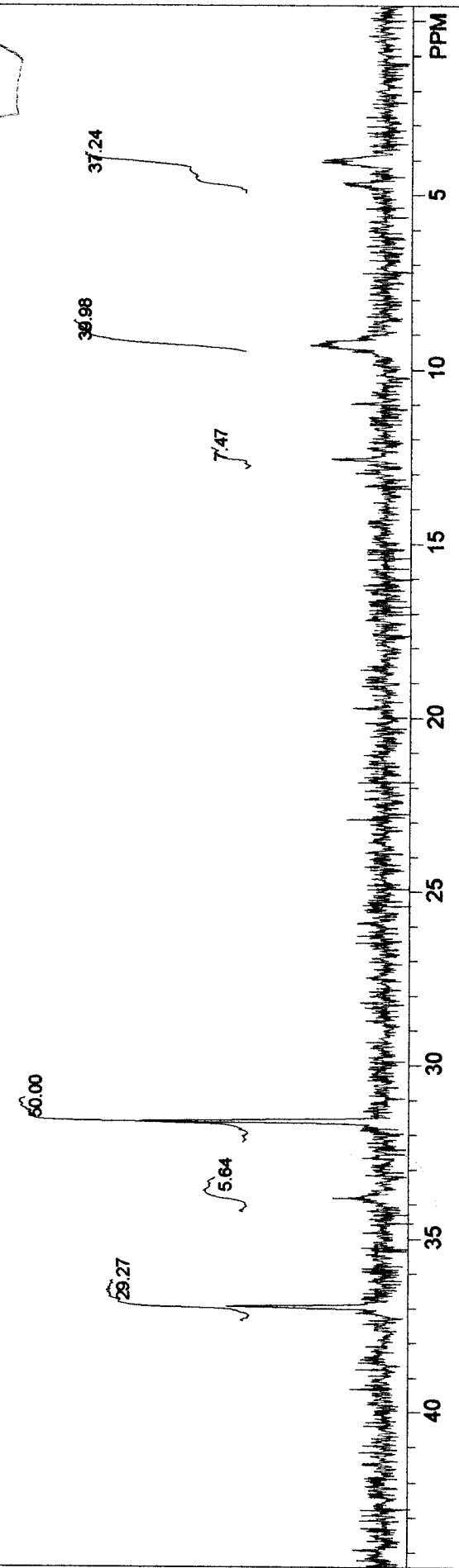
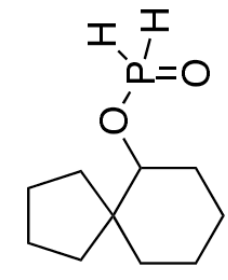


| PEAKLIST LINE | POINT | F ₁ (MHz) | PPM (δ) | INTENSITY | REL INT | N LABEL |
|---------------|-------|----------------------|---------|-----------|---------|---------|
| 1 | 23205 | 1346.60 | 36.953 | 4870023 | 58.987 | 1 |
| 2 | 23392 | 1232.73 | 33.829 | 1625032 | 19.683 | 2 |
| 3 | 23524 | 1152.05 | 31.615 | 7219673 | 87.446 | 3 |
| 4 | 24043 | 835.54 | 22.929 | 964052 | 11.677 | 4 |
| 5 | 24661 | 457.88 | 12.565 | 1784188 | 21.610 | 5 |
| 6 | 24852 | 341.39 | 9.368 | 1821931 | 22.068 | 6 |
| 7 | 25136 | 167.74 | 4.603 | 481880 | 5.837 | 7 |
| 8 | 25173 | 145.26 | 3.986 | 1974972 | 23.921 | 8 |



2 mol% Pd/xantphos

Table 1, entry 5



| | | | | |
|---------------------------|---------------|-------------|--------------|---------------------------------|
| F1: 36.441 | SW1: 20000 | OF1: 5510.0 | PTSId: 32768 | USER: -- DATE: 09/28/04 (12:27) |
| EX: c:\left\global\zg.ppg | PW: 12.4 usec | PD: 2.0 sec | NA: 256 | LB: 0.0 |
| | | | H2H | <i>Cocodchun!</i> |

Interpolated Peak Listing

| PEAK | POINT | HEIGHT | REL. HT | CHZ | PPM |
|------|-------|--------|---------|--------|--------|
| 1 | 24374 | 3218K | 93.86 | 633.39 | 17.382 |

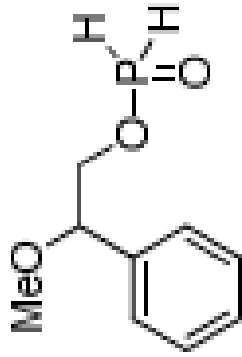
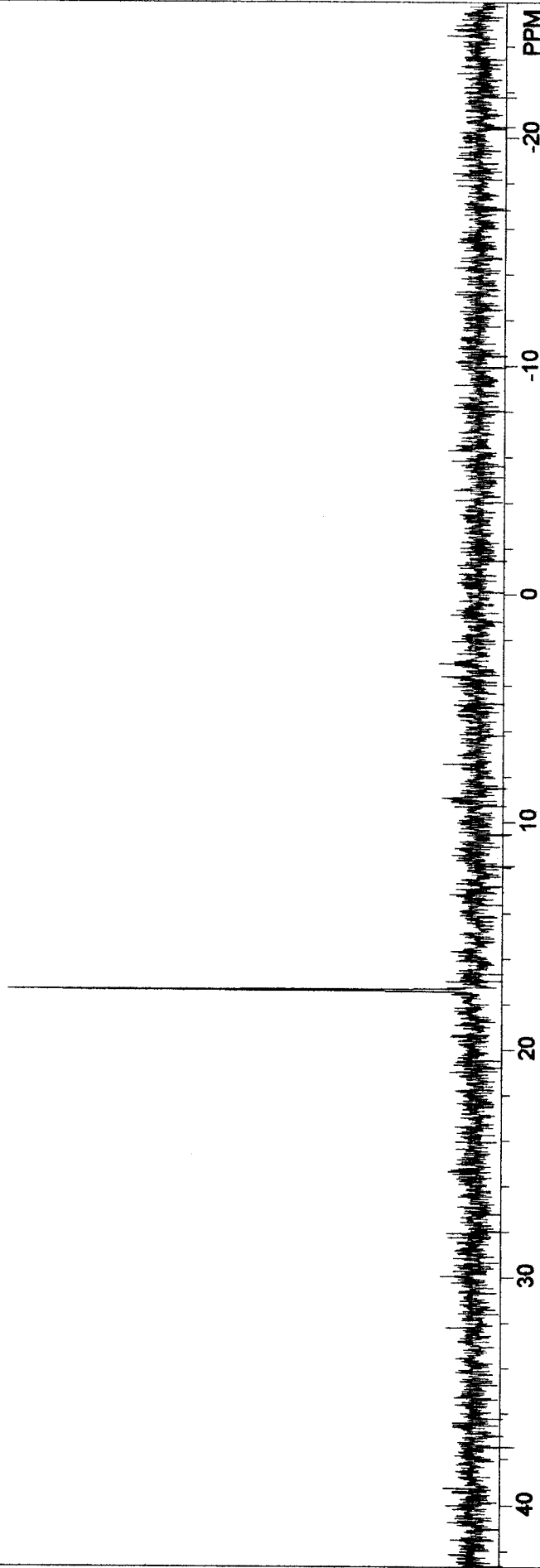


Table 1, entry 8



F1: 36.441
 EX: c:\left\global\zg.ppg
 SW1: 20000
 PW: 12.4 usec
 OF1: 5510.0
 NA: 16
 LB: 0.0
 USER: -- DATE: 09/08/04 (21:25)
 PTSId: 32768
 405 / 426 C - 2.5 h MT

Interpolated Peaks Listing

| PEAK | POINT | HEIGHT | REL. HT | PPM |
|------|-------|--------|---------|--------|
| 1 | 23421 | 306358 | 36.74 | 33.345 |
| 2 | 23442 | 321246 | 38.52 | 32.991 |
| 3 | 23461 | 276286 | 33.13 | 32.673 |
| 4 | 24355 | 638620 | 76.58 | 17.693 |
| 5 | 24375 | 600443 | 72.00 | 17.354 |
| 6 | 24396 | 487654 | 58.48 | 17.004 |
| 7 | 25290 | 304037 | 36.46 | 2.033 |
| 8 | 25311 | 397989 | 47.72 | 1.676 |
| 9 | 25330 | 361172 | 43.31 | 1.363 |

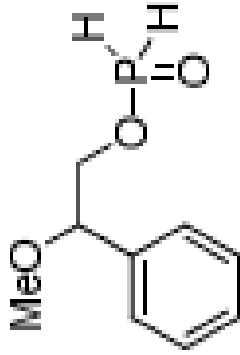
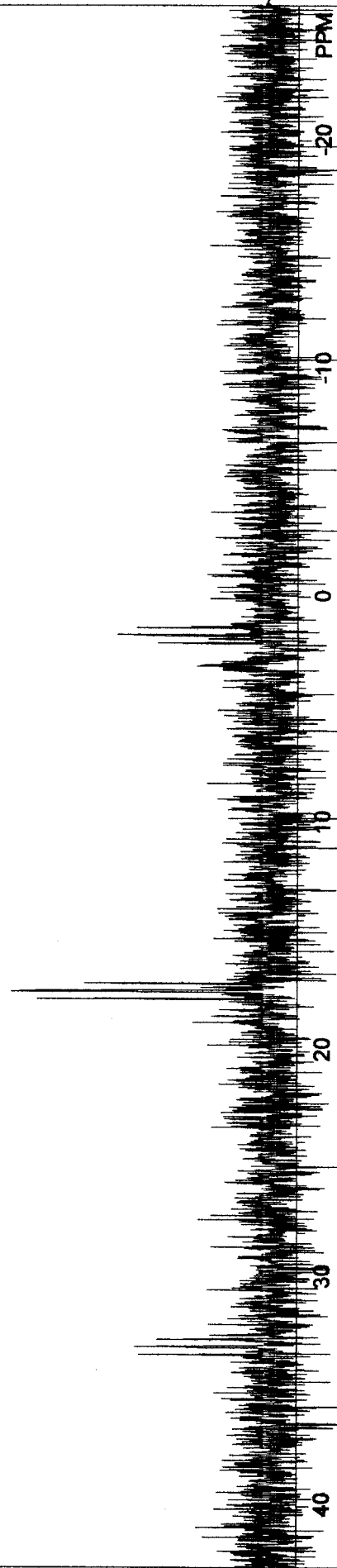


Table 1, entry 8



F1: 36.441 SW1: 20000 OF1: 5510.0 USER: -- DATE: 09/08/04 (21:26)
 EX: c:\leftglobal\zg.ppg PW: 12.4 usec PD: 2.0 sec NA: 16 LB: 0.0 PTS1d: 32768

| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 5313.638 | 43.747 | 20.5 |
| 2 | 5247.544 | 43.203 | 25.9 |
| 3 | 4559.266 | 37.536 | 93.7 |
| 4 | 1372.464 | 11.299 | 19.9 |
| 5 | 1345.537 | 11.078 | 40.1 |
| 6 | 1313.714 | 10.816 | 21.5 |
| 7 | 954.276 | 7.857 | 133.5 |
| 8 | 713.970 | 5.878 | 66.7 |

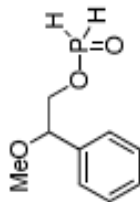
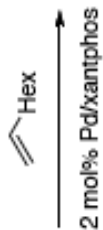
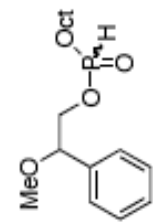
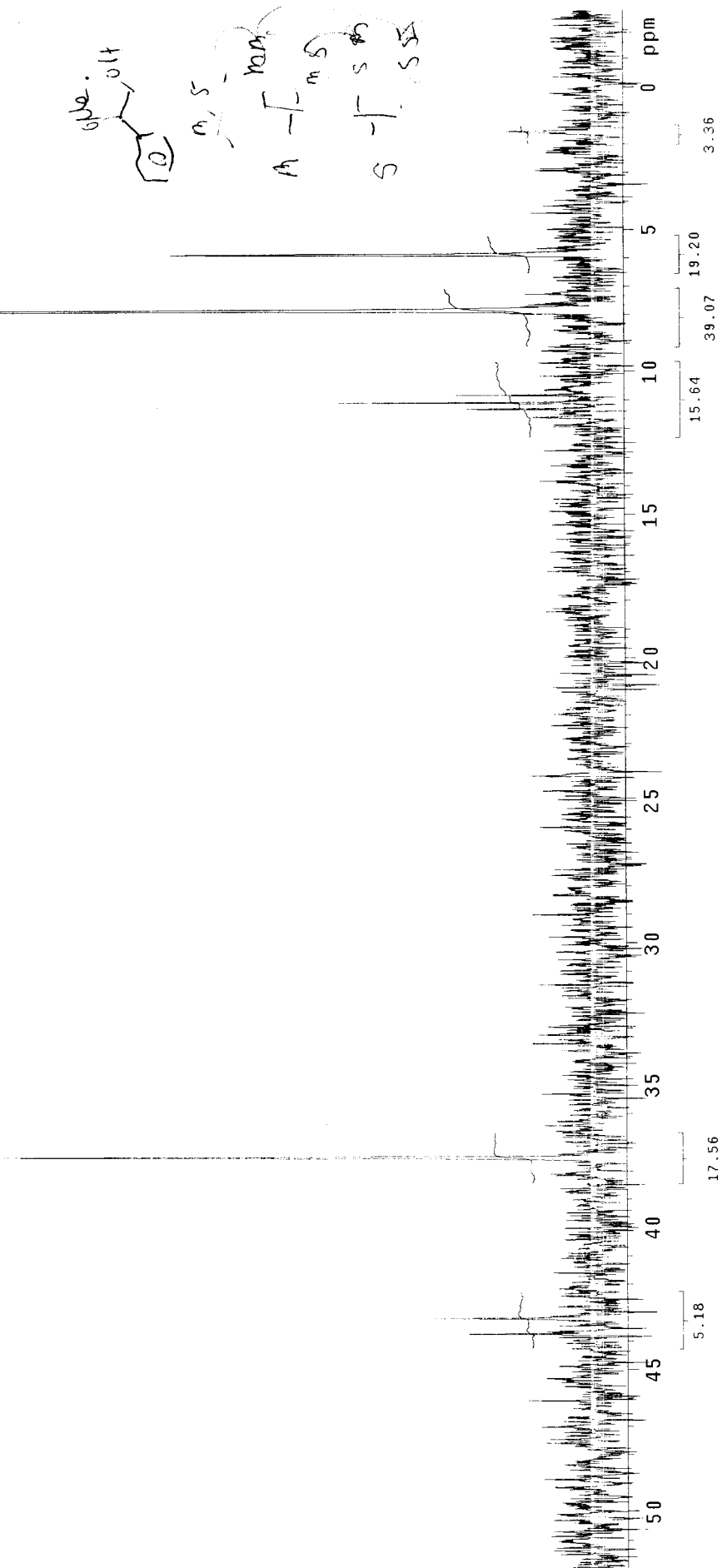


Table 1, entry 8

0.14
 0.5
 A - [m s]
 S - [s s s]



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 6250.789 | 51.463 | -16.6 |
| 2 | 5902.774 | 48.597 | -17.2 |
| 3 | 5789.353 | 47.664 | -16.7 |
| 4 | 5573.527 | 45.887 | 21.2 |
| 5 | 5301.806 | 43.650 | -16.3 |
| 6 | 5045.997 | 41.544 | 16.1 |
| 7 | 4840.779 | 39.854 | 27.1 |
| 8 | 4827.723 | 39.747 | 54.6 |
| 9 | 4813.852 | 39.632 | 39.8 |
| 10 | 4738.782 | 39.014 | -18.4 |
| 11 | 4664.528 | 38.403 | 16.3 |
| 12 | 4290.810 | 35.326 | 45.5 |
| 13 | 3887.717 | 32.007 | -17.8 |
| 14 | 3685.354 | 30.341 | -16.7 |
| 15 | 3619.668 | 29.801 | 16.7 |
| 16 | 3274.917 | 26.962 | -19.9 |
| 17 | 3008.908 | 24.772 | -17.2 |
| 18 | 2526.257 | 20.799 | -16.4 |
| 19 | 2177.835 | 17.930 | 15.9 |
| 20 | 2154.171 | 17.735 | -16.4 |
| 21 | 1935.489 | 15.935 | -17.0 |
| 22 | 1701.711 | 14.010 | 22.8 |
| 23 | 1465.078 | 12.062 | -18.0 |
| 24 | 1291.274 | 10.631 | 146.5 |
| 25 | 1183.157 | 9.741 | -19.2 |
| 26 | 1046.889 | 8.619 | 129.9 |
| 27 | 617.685 | 5.085 | 132.9 |
| 28 | 381.867 | 3.144 | 124.2 |
| 29 | 181.136 | 1.491 | 22.5 |

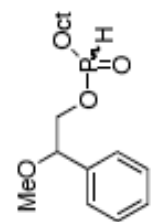
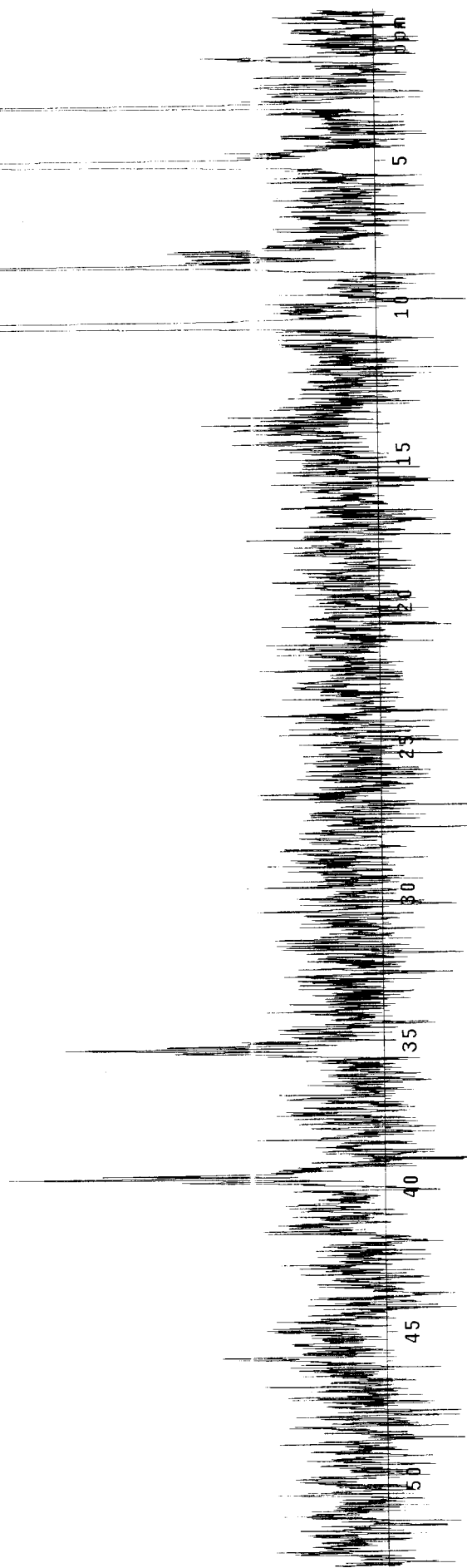
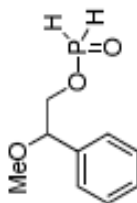


Table 1, entry 8

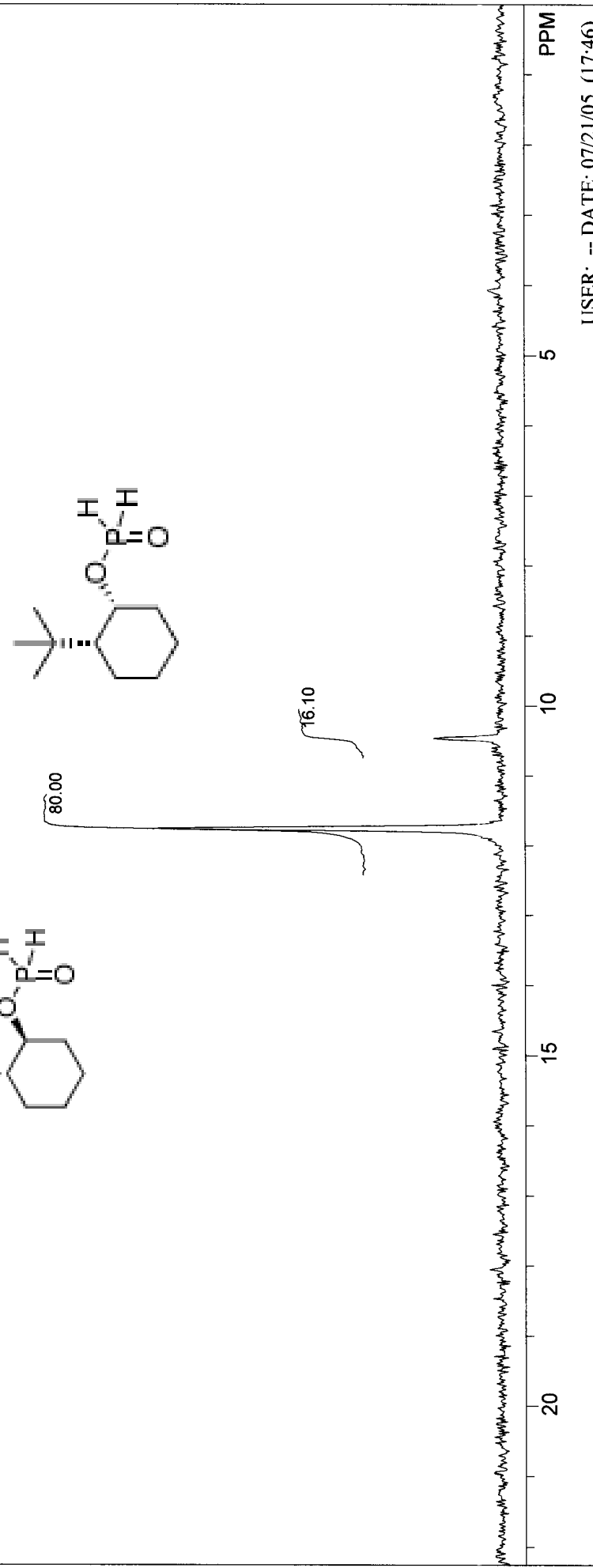
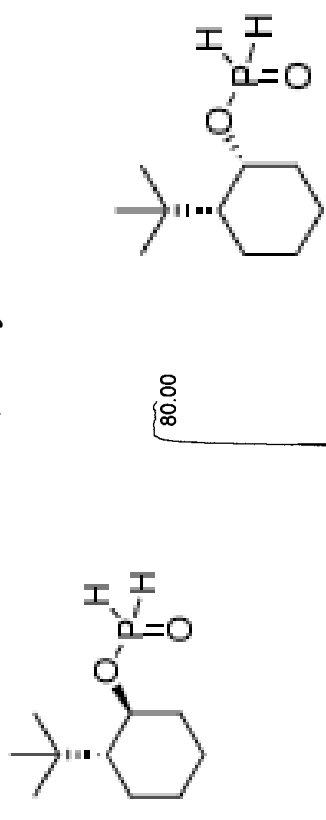


597

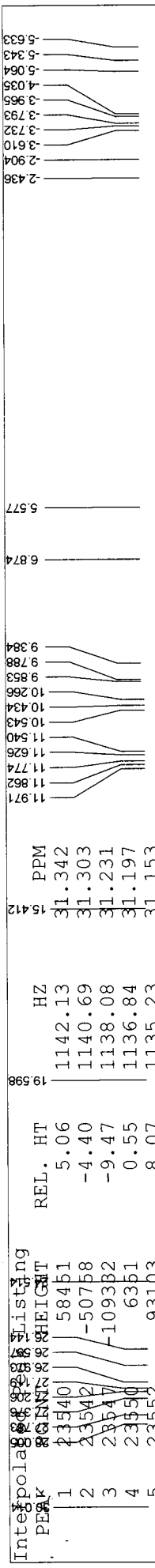
Interpolated Peak Listing

| PEAK | POINT | HEIGHT | REL. HT | HZ | PPM |
|------|-------|--------|---------|--------|--------|
| 1 | 24710 | 8637K | 101.80 | 427.97 | 11.744 |
| 2 | 24787 | 1630K | 19.21 | 381.20 | 10.461 |

Table 1, entry 9

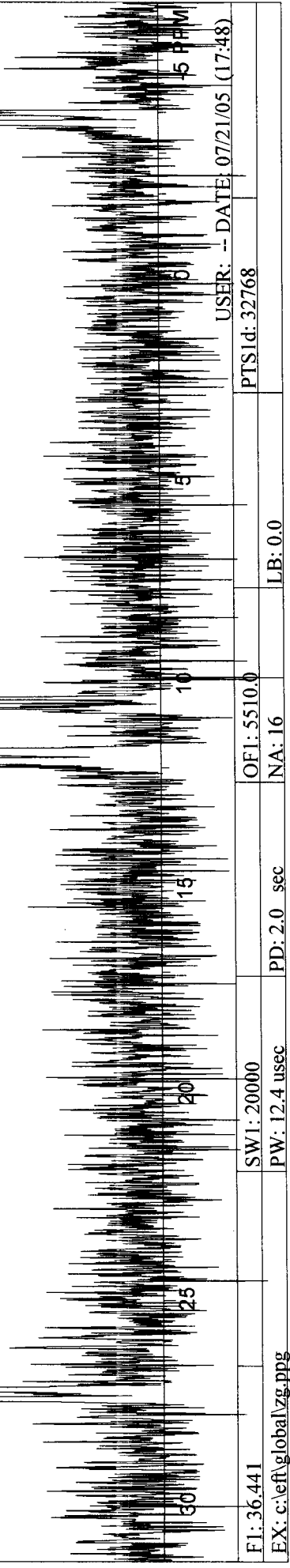
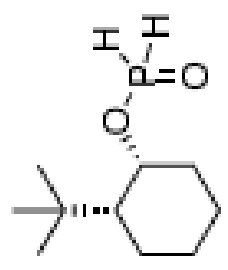
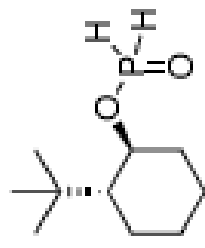


| | | | |
|----------------------------|---------------|-------------|---------------------------------|
| F1: 36.441 | SW1: 20000 | OF1: 5510.0 | USER: -- DATE: 07/21/05 (17:46) |
| E.X: c:\left\global\zg.ppg | PW: 12.4 usec | NA: 16 | PTSId: 32768 |
| | | LB: 0.0 | D esterf- |



| PEAK | PPM | INTEGRATION | REL. HT | HZ |
|------|--------|-------------|---------|---------|
| 1 | 11.971 | 1.1971 | 5.06 | 1142.13 |
| 2 | 11.882 | 1.1882 | -4.40 | 1140.69 |
| 3 | 11.774 | 1.1774 | -9.47 | 1138.08 |
| 4 | 11.626 | 1.1626 | 0.55 | 1136.84 |
| 5 | 11.540 | 1.1540 | 8.07 | 1135.23 |
| 6 | 11.054 | 1.0543 | 1.27 | 1131.83 |
| 7 | 10.434 | 1.0434 | -1.37 | 1130.03 |
| 8 | 10.266 | 1.0266 | -2.60 | 1128.09 |
| 9 | 9.985 | 0.9853 | -68.53 | 1128.63 |
| 10 | 9.788 | 0.9788 | 6.00 | 1117.82 |
| 11 | 9.384 | 0.9384 | -7.33 | 1115.49 |
| 12 | 6.874 | 6.874 | -14.01 | 1114.19 |
| 13 | 5.577 | 5.577 | -14.42 | 1110.97 |
| 14 | 5.343 | 5.343 | -8.24 | 1107.73 |
| 15 | 5.064 | 5.064 | 13.25 | 1103.04 |
| 16 | 4.038 | 4.038 | -2.29 | 1102.05 |
| 17 | 3.965 | 3.965 | 7.93 | 1098.19 |
| 18 | 3.793 | 3.793 | 4.65 | 1095.00 |
| 19 | 3.732 | 3.732 | -11.56 | 1093.74 |
| 20 | 3.610 | 3.610 | -4.70 | 1086.28 |
| 21 | 3.342 | 3.342 | 9.90 | 1083.41 |
| 22 | 3.303 | 3.303 | -7.46 | 1081.87 |
| 23 | 3.231 | 3.231 | 15.97 | 1079.93 |
| 24 | 3.197 | 3.197 | 2.53 | 1078.86 |
| 25 | 3.153 | 3.153 | -1.59 | 1073.78 |
| 26 | 3.106 | 3.106 | -1.10 | 1071.82 |
| 27 | 3.067 | 3.067 | -96.33 | 1075.87 |
| 28 | 3.028 | 3.028 | -6.66 | 1068.59 |
| 29 | 2.989 | 2.989 | -5.77 | 1065.66 |
| 30 | 2.950 | 2.950 | -9.30 | 1063.39 |
| 31 | 2.911 | 2.911 | 12.74 | 1059.34 |
| 32 | 2.872 | 2.872 | -9.40 | 1056.64 |
| 33 | 2.833 | 2.833 | 7.72 | 1054.66 |
| 34 | 2.794 | 2.794 | -4.26 | 1054.14 |
| 35 | 2.755 | 2.755 | -4.37 | 1049.16 |

Table 1, entry 9



| FI: 36.441 | SW1: 20000 | OF1: 5510.0 | PTS1d: 32768 | USER: -- DATE: 07/21/05 (17:48) |
|---------------------------|---------------|-------------|--------------|---------------------------------|
| EX: c:\left\global\zg.ppg | PW: 12.4 usec | NA: 16 | LB: 0.0 | |
| | PD: 2.0 sec | | | |

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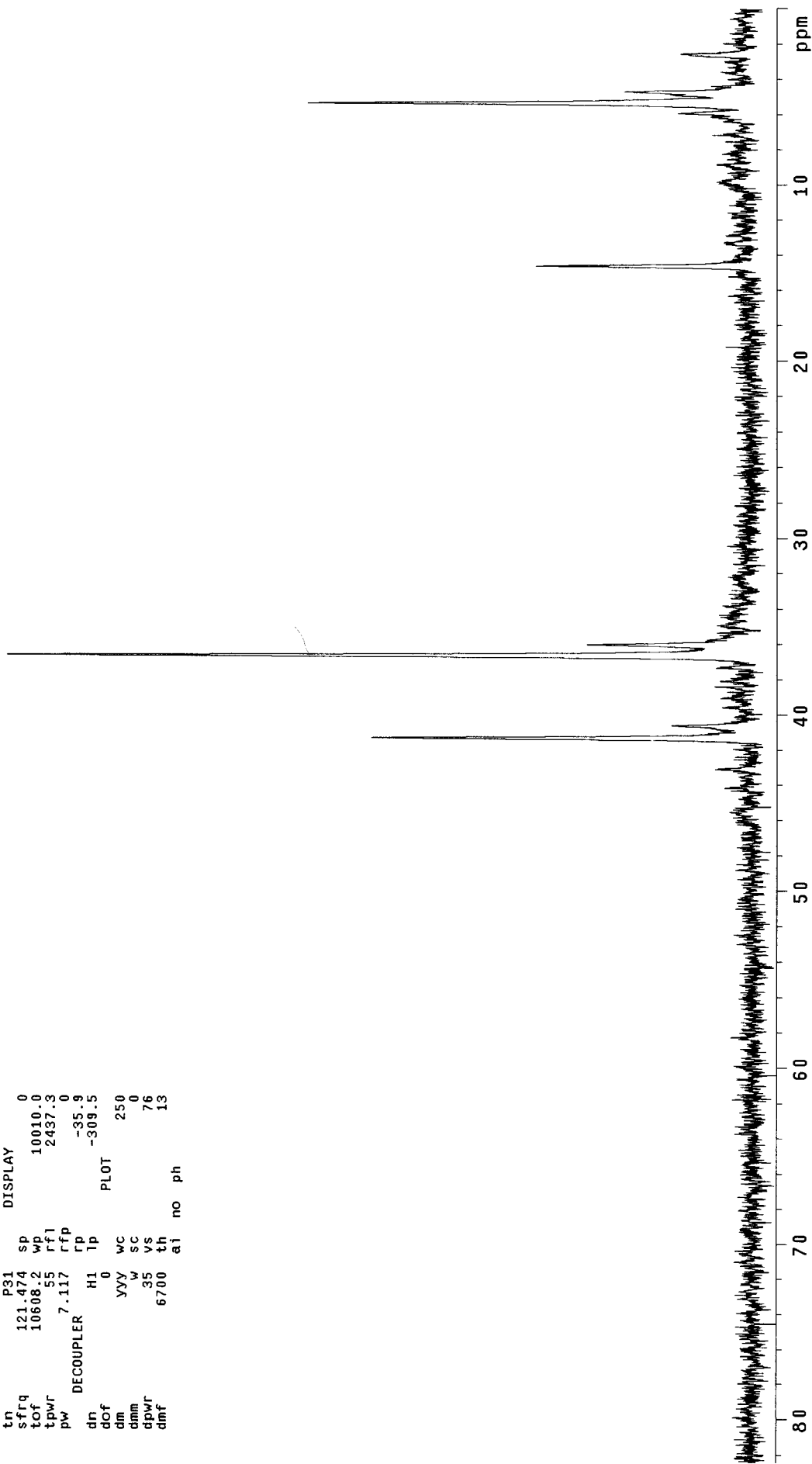
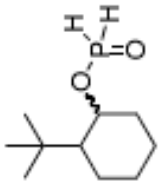
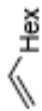
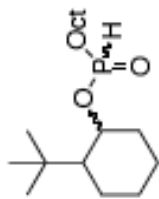
exp2 s2pu1

SAMPLE date Jul 28 2005 temp not used
solvent CCl₄ gain not used
file C013 spin 20
ACQUISITION hst 0.008
sw 26738.0 pw90 18.300
at 1.598 alfa 20.000
fb 85476
bs 14800 ll
ss 64 in n
di 4 dp y
nt 1.000 hs mn
ct 16 lb fn 2.00 not used
TRANSMITTER P31 DISPLAY
tn 121.474 sp 0
sfrq 10608.2 wp 10010.0
tpwr 55 rfl 2437.3
pw 7.117 rfp 0
DECOUPLER H1 lp -35.9
dn 0
dof YVY WC 250
dim 0
dimw SC 76
dpwr 35 VS 13
dmf 6700 th a1 no ph

2 mol% Pd/xantphos

Table 1, entry 9

trans/cis = 3:1



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 5023.966 | 41.362 | 99.8 |
| 2 | 4932.984 | 40.613 | 19.6 |
| 3 | 4456.861 | 36.693 | 196.1 |
| 4 | 4378.119 | 36.045 | 41.5 |

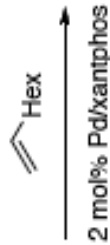
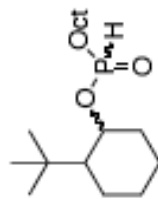
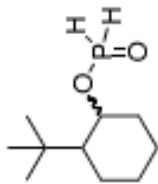
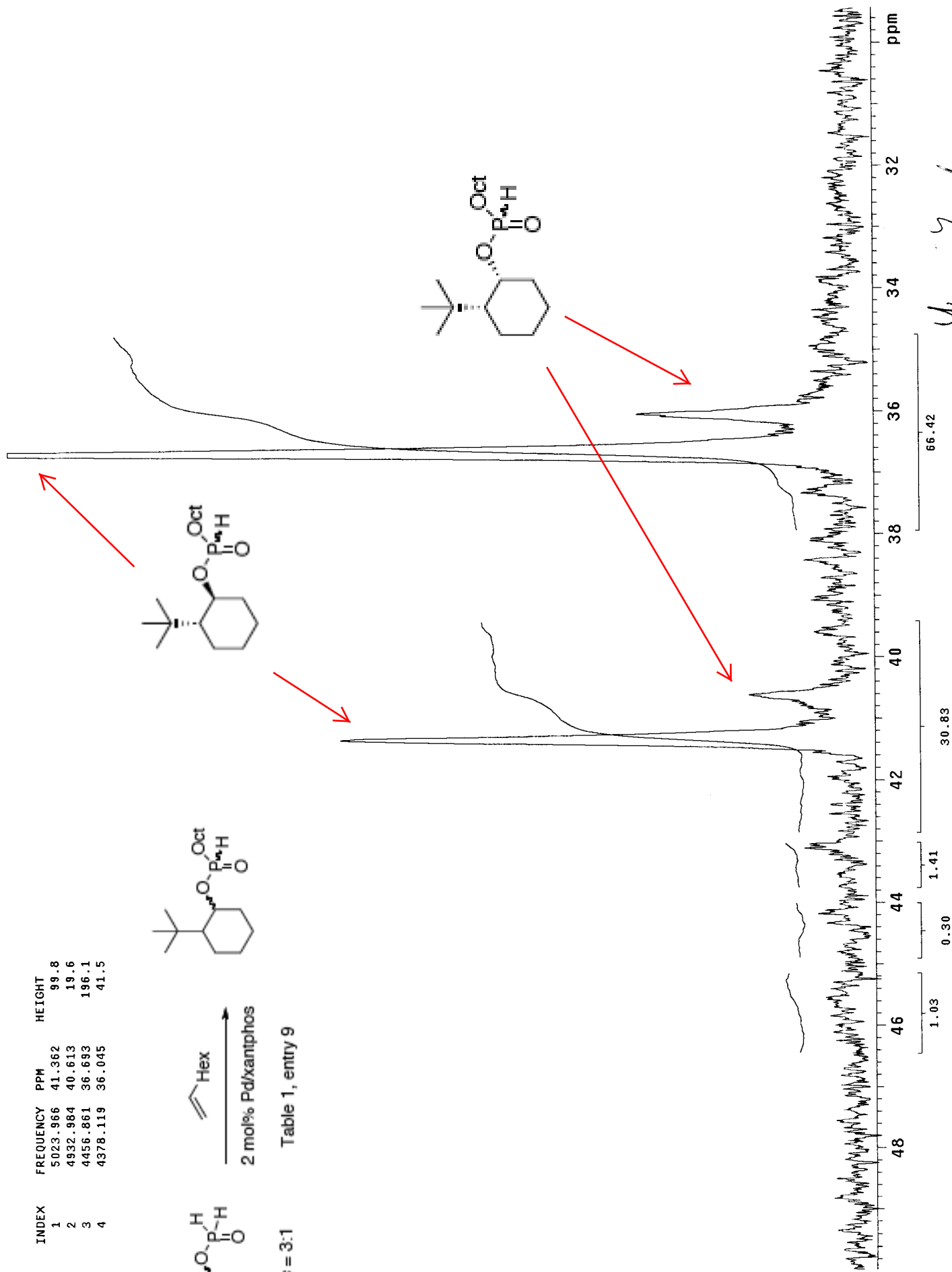


Table 1, entry 9

trans/cis = 3:1



Yves Yand
 J. P. Yand

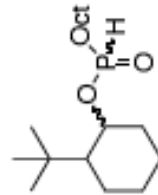
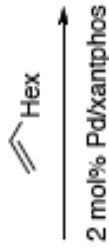
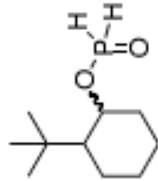
603

exp2 s2pu1

```

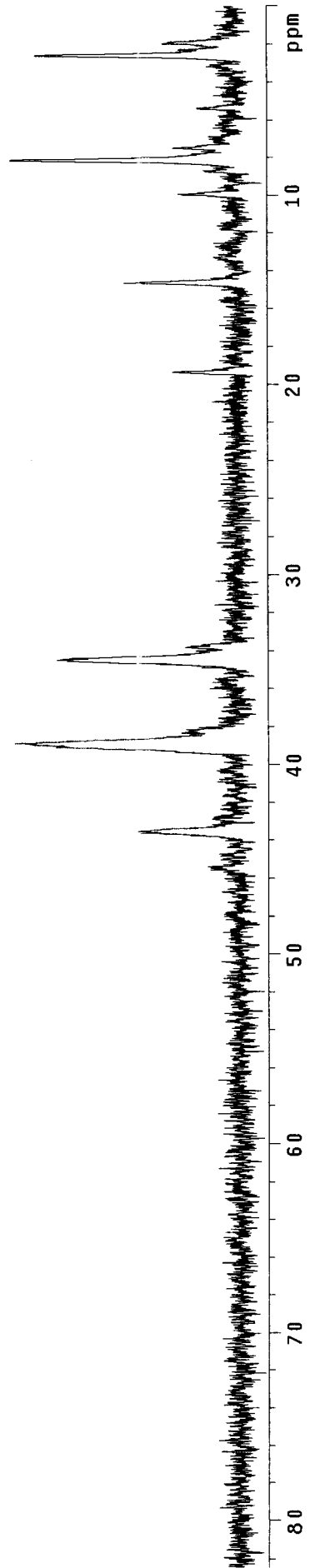
SAMPLE
date Jul 28 2005
solvent CDC13
file exp
ACQUISITION
sw 26738.0
at 1.598
np 85476
fb 14800
bs 64
ss 4
di 1.000
nt 8
ct 8
SPECIAL
temp not used
gain not used
spn 20
hst 0.008
pw90 18.300
alfa 20.000
FLAGS
i1 n
in n
dp y
hs nm
PROCESSING
lb 2.00
fn not used
DISPLAY
sp 0
wd 10010.0
rf1 2437.3
rfp 0
rp -49.3
lp -250.1
PLOT
wc 250
w 0
sc 0
vs 58
th 12
al no
ph

```



trans/cis = 3:1

Table 1, entry 9



Interpolated Peak Listing

| PEAK | POINT | HEIGHT | REL. HT | HZ | PPM |
|------|-------|--------|---------|--------|--------|
| 1 | 24790 | 13038K | 98.68 | 379.53 | 10.415 |

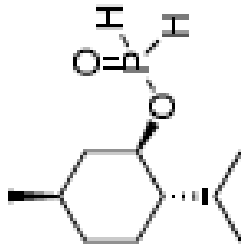
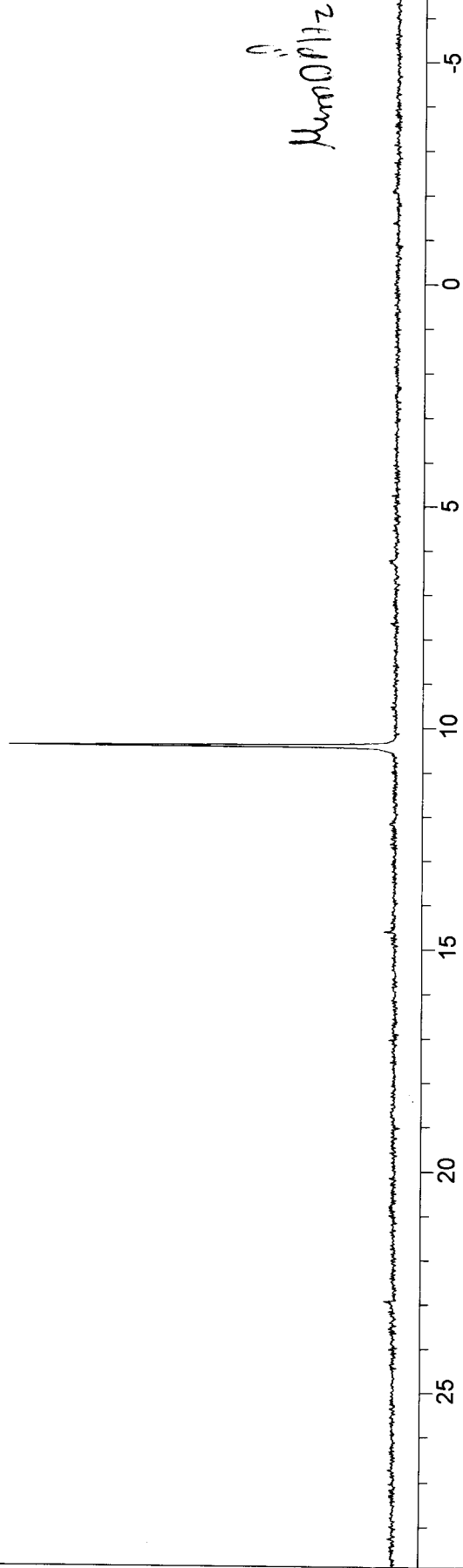


Table 1, entry 11



| | | | | |
|---------------------------|---------------|-------------|--------------|---------------------------------|
| FI: 36.441 | SW1: 20000 | OF1: 5510.0 | PTSLd: 32768 | USER: -- DATE: 03/28/05 (14:24) |
| EX: c:\left\global\zg.ppg | PW: 12.4 usec | NA: 8 | LB: 0.0 | 579 |

Interpreted Peak Listing

| PEAK # | POINT | HEIGHT | REL. HT | HZ | PPM |
|--------|-------|--------|---------|---------|--------|
| 1 | 23849 | 849293 | 47.13 | 953.77 | 26.173 |
| 2 | 23866 | 932739 | 51.77 | 943.36 | 25.888 |
| 3 | 24783 | 1810K | 100.47 | 383.44 | 10.522 |
| 4 | 24800 | 1746K | 96.90 | 373.15 | 10.240 |
| 5 | 25717 | 879833 | 48.83 | -186.71 | -5.124 |
| 6 | 25734 | 811058 | 45.01 | -197.17 | -5.411 |

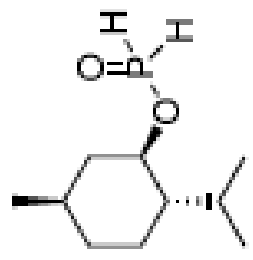


Table 1, entry 11

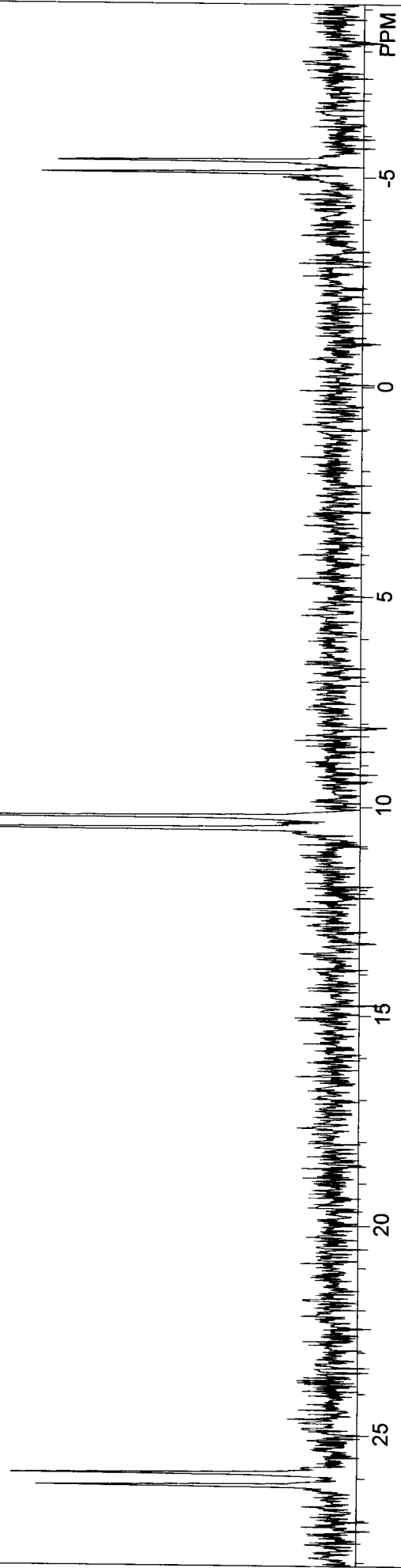
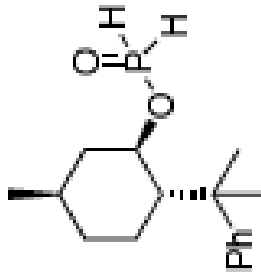
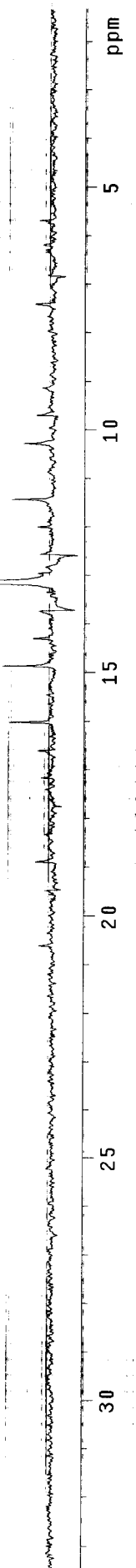


Table 1, entry 12

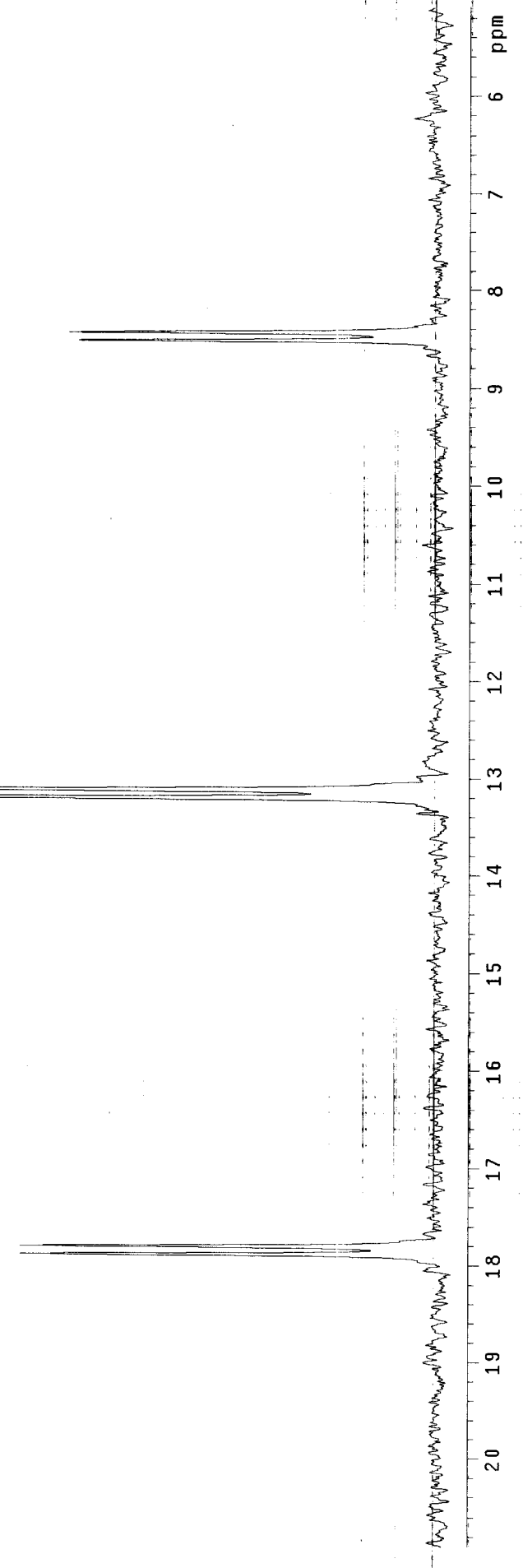
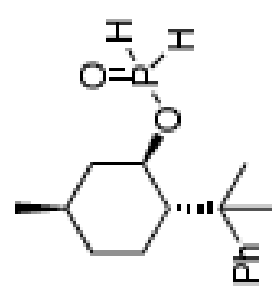


| INDEX | FREQUENCY (PPM) | HEIGHT |
|-------|-----------------|--------|
| 1 | 1388.376 | 6.8 |
| 2 | 1298.844 | 7.9 |
| 3 | 127.5 | 127.5 |
| 4 | 10.282 | 3.9 |
| 5 | 1388.376 | 6.4 |
| 6 | 1298.844 | 4.7 |



| INDEX | FREQUENCY PPM | HEIGHT |
|-------|---------------|--------|
| 1 | 2172.989 | 17.890 |
| 2 | 2162.739 | 17.806 |
| 3 | 1603.794 | 13.204 |
| 4 | 1583.584 | 13.120 |
| 5 | 1034.241 | 8.515 |
| 6 | 1024.450 | 8.434 |

Table 1, entry 12



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 4780.805 | 39.360 | 36.8 |
| 2 | 4287.138 | 35.296 | 3.2 |
| 3 | 4074.984 | 33.549 | 126.0 |

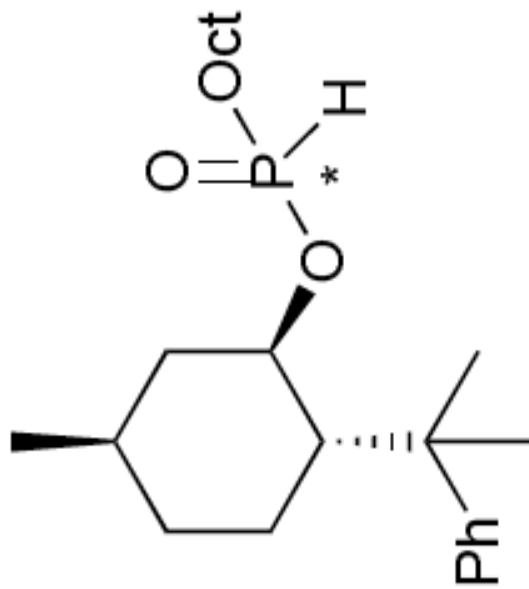
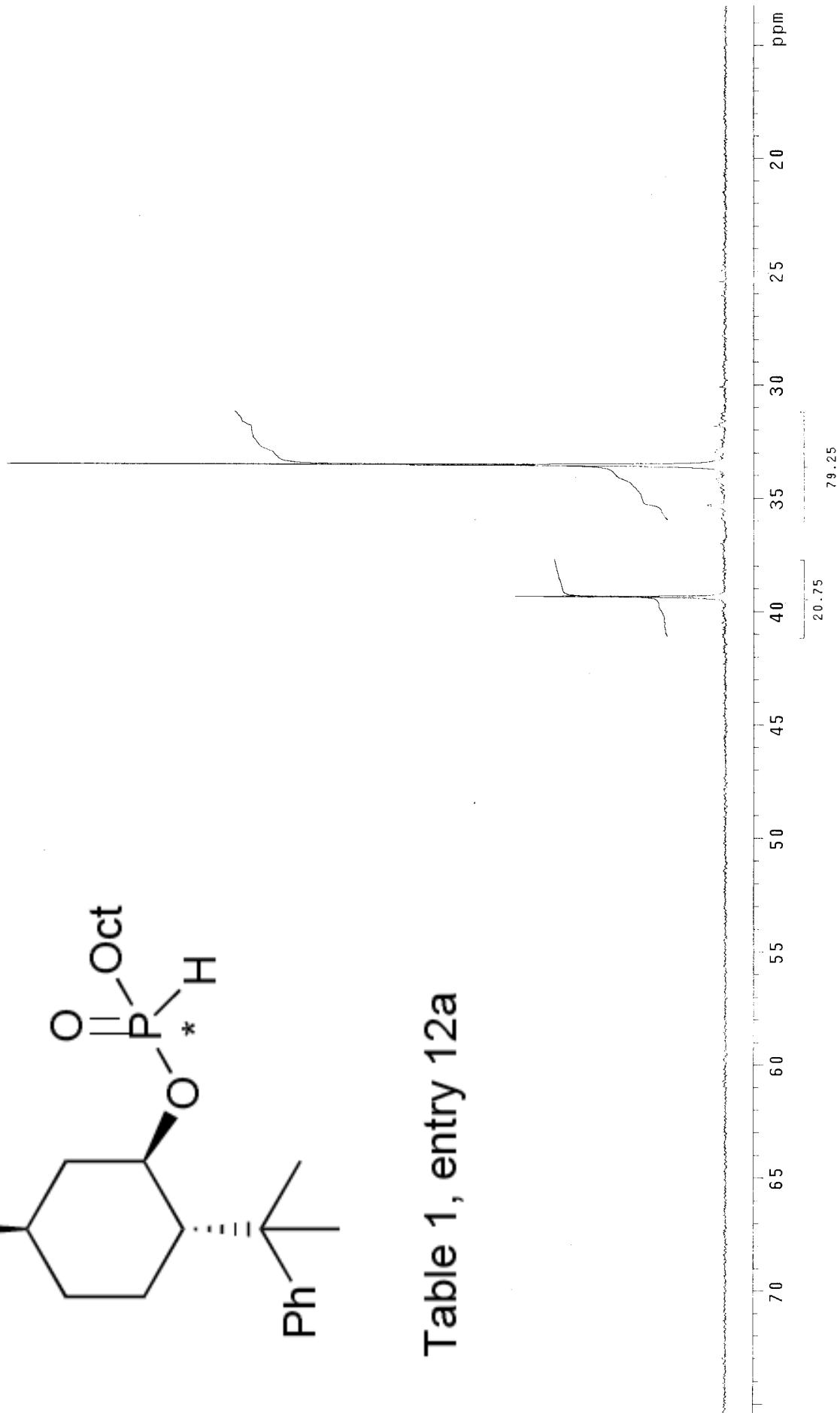


Table 1, entry 12a



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 5052.525 | 41.597 | 32.1 |
| 2 | 5041.102 | 41.503 | 31.9 |
| 3 | 4524.587 | 37.251 | 32.0 |
| 4 | 4513.572 | 37.160 | 32.5 |
| 5 | 4334.465 | 35.686 | 109.1 |
| 6 | 3815.095 | 31.410 | 105.9 |

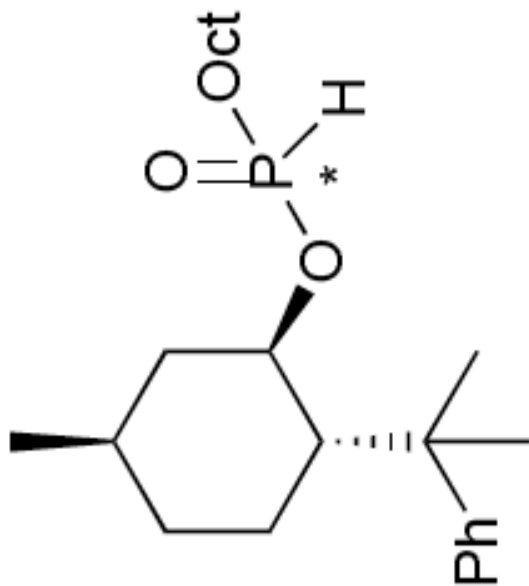
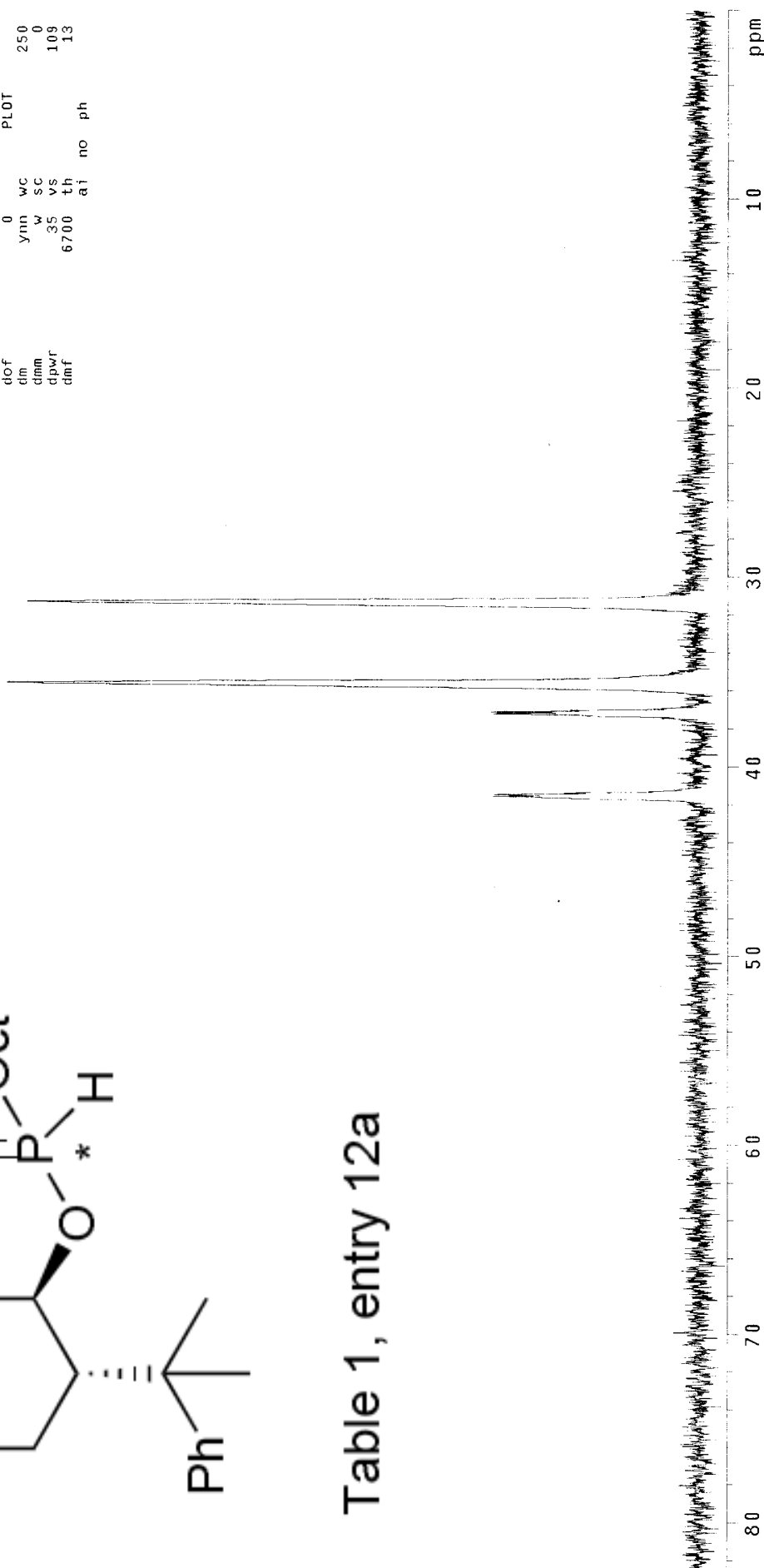


Table 1, entry 12a

```

exp1 s2pu1
SAMPLE
date Sep 8 2004 temp not used
solvent CDC13 gain not used
file exp spin 20
ACQUISITION
sw 26738.0 pw90 0.008
at 1.598 hst 18.300
np 85476 alfa 20.000
fb 14800 il n
bs 64 in n
ss 4 dp y
di 1.000 hs nn
nt 16 hs PROCESSING
ct 16 lb 2.00 not used
TRANSMITTER P31 fh 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 H1 lp 137.0
dh dof 0 -289.2
dm ynn wc 250
dmm w SC 0
dpwr 35 vs 109
dmf 6700 th no 13
ai ph

```



| INDEX | FREQUENCY PPM | HEIGHT | INDEX | FRE | HT |
|-------|---------------|--------|-------|-----|---------|
| 1 | 2367.531 | 7.890 | 40 | 5 | 4.7 |
| 2 | 2365.186 | 7.883 | 41 | 5 | 8.0 |
| 3 | 2206.295 | 7.353 | 42 | 5 | 7.5 |
| 4 | 2204.829 | 7.348 | 43 | 4 | 5.7 |
| 5 | 2202.777 | 7.341 | 44 | 4 | 6.5 |
| 6 | 2197.500 | 7.324 | 45 | 4 | 3.2 |
| 7 | 2196.621 | 7.321 | 46 | 4 | 4.3 |
| 8 | 2187.240 | 7.290 | 47 | 4 | 12.1 |
| 9 | 2185.481 | 7.284 | 48 | 4 | |
| 10 | 2182.256 | 7.273 | 49 | 4 | |
| 11 | 2180.204 | 7.266 | 50 | 4 | |
| 12 | 2171.996 | 7.239 | 51 | 4 | |
| 13 | 2142.680 | 7.141 | 52 | 4 | |
| 14 | 2141.507 | 7.137 | 53 | 4 | |
| 15 | 2140.042 | 7.132 | 54 | 4 | |
| 16 | 2134.472 | 7.114 | 55 | 3 | |
| 17 | 2128.608 | 7.094 | 56 | 3 | |
| 18 | 2127.436 | 7.090 | 57 | 3 | |
| 19 | 1848.058 | 6.159 | 58 | 3 | |
| 20 | 1845.713 | 6.151 | 59 | 3 | |
| 21 | 1331.223 | 4.437 | 60 | 3 | |
| 22 | 1327.412 | 4.424 | 61 | 3 | |
| 23 | 1324.480 | 4.414 | 62 | 3 | |
| 24 | 1320.669 | 4.401 | 63 | 3 | |
| 25 | 1246.784 | 4.155 | 64 | 3 | |
| 26 | 1239.758 | 4.132 | 65 | 3 | |
| 27 | 1232.429 | 4.107 | 66 | 3 | |
| 28 | 1225.394 | 4.084 | 67 | 3 | |
| 29 | 637.322 | 2.124 | 68 | 3 | |
| 30 | 635.856 | 2.119 | 69 | 3 | |
| 31 | 634.097 | 2.113 | 70 | 2 | |
| 32 | 629.700 | 2.099 | 71 | 2 | 286.707 |
| 33 | 621.492 | 2.071 | 72 | 2 | 284.069 |
| 34 | 618.267 | 2.061 | 73 | 2 | 282.603 |
| 35 | 612.990 | 2.043 | 74 | 2 | 279.964 |
| 36 | 609.179 | 2.030 | 75 | 2 | 273.515 |
| 37 | 599.212 | 1.997 | 76 | 2 | 269.118 |
| 38 | 595.694 | 1.985 | 77 | 2 | 267.359 |
| 39 | 517.421 | 1.724 | 78 | 2 | 265.013 |

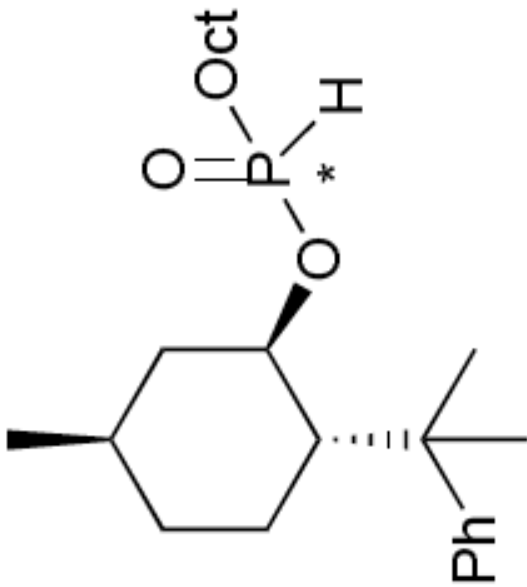
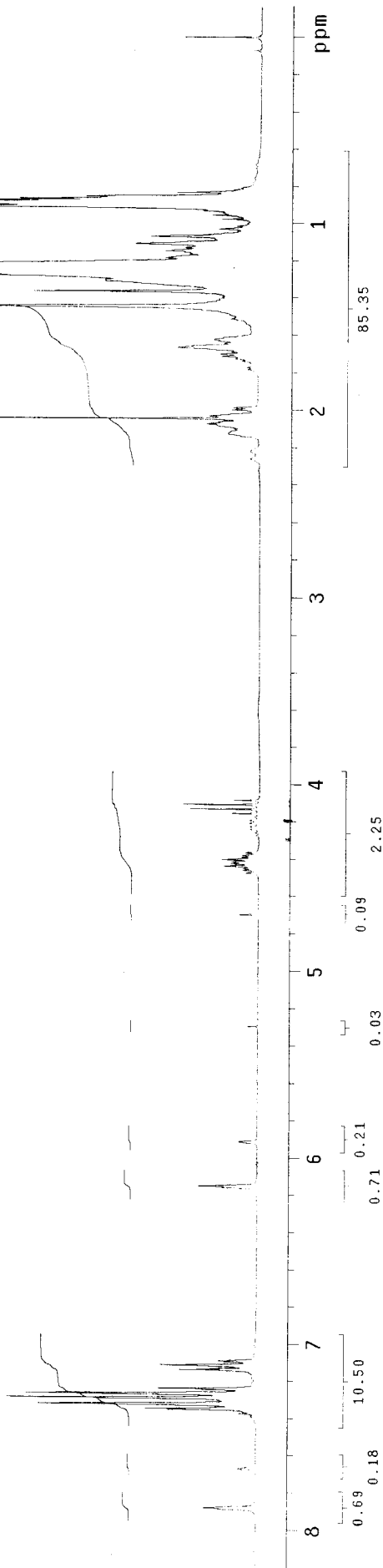


Table 1, entry 12a

| | | | | | |
|----|---------|-------|----|---|---------|
| 32 | 629.700 | 2.099 | 71 | 2 | 286.707 |
| 33 | 621.492 | 2.071 | 72 | 2 | 284.069 |
| 34 | 618.267 | 2.061 | 73 | 2 | 282.603 |
| 35 | 612.990 | 2.043 | 74 | 2 | 279.964 |
| 36 | 609.179 | 2.030 | 75 | 2 | 273.515 |
| 37 | 599.212 | 1.997 | 76 | 2 | 269.118 |
| 38 | 595.694 | 1.985 | 77 | 2 | 267.359 |
| 39 | 517.421 | 1.724 | 78 | 2 | 265.013 |



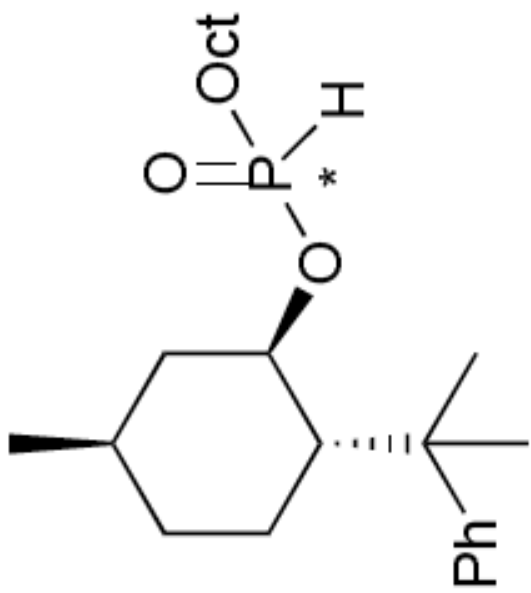
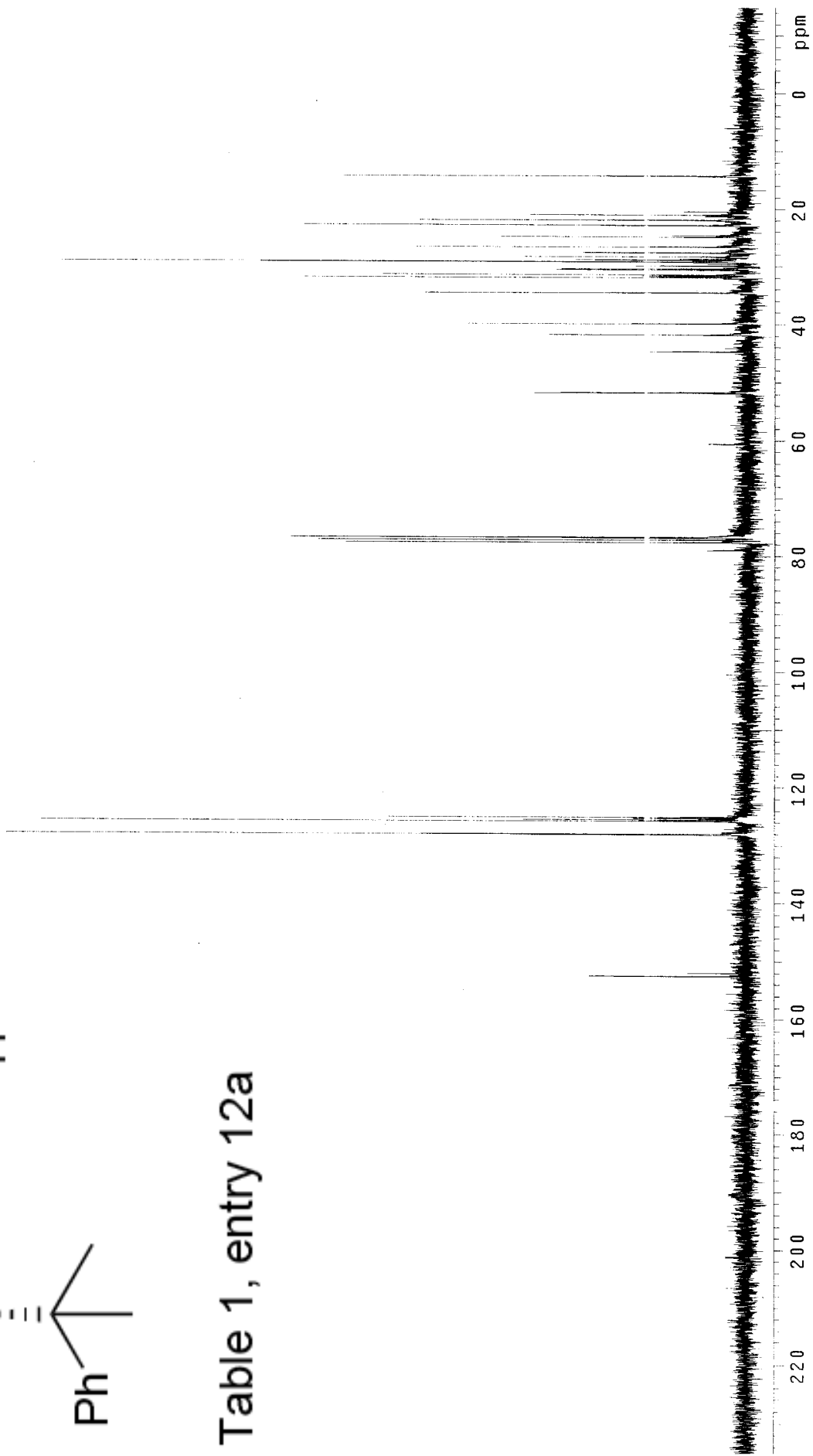


Table 1, entry 12a



| INDEX | FREQUENCY PPM | HEIGHT |
|-------|---------------|--------|
| 1 | 4483.381 | 22.7 |
| 2 | 3775.928 | 126.0 |
| 3 | 1521.380 | 4.1 |

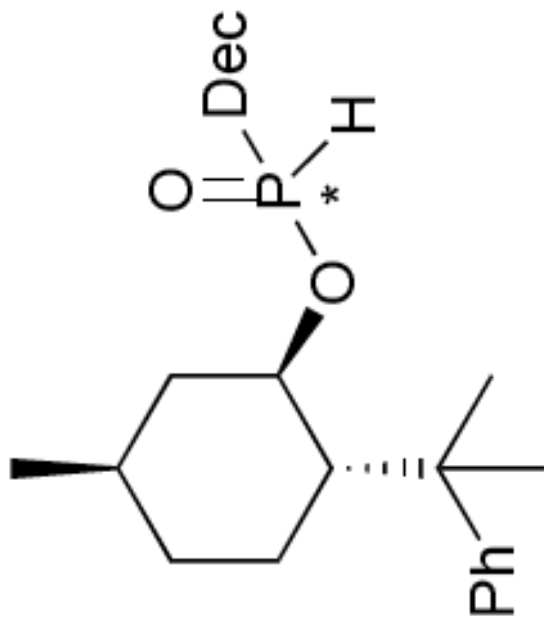
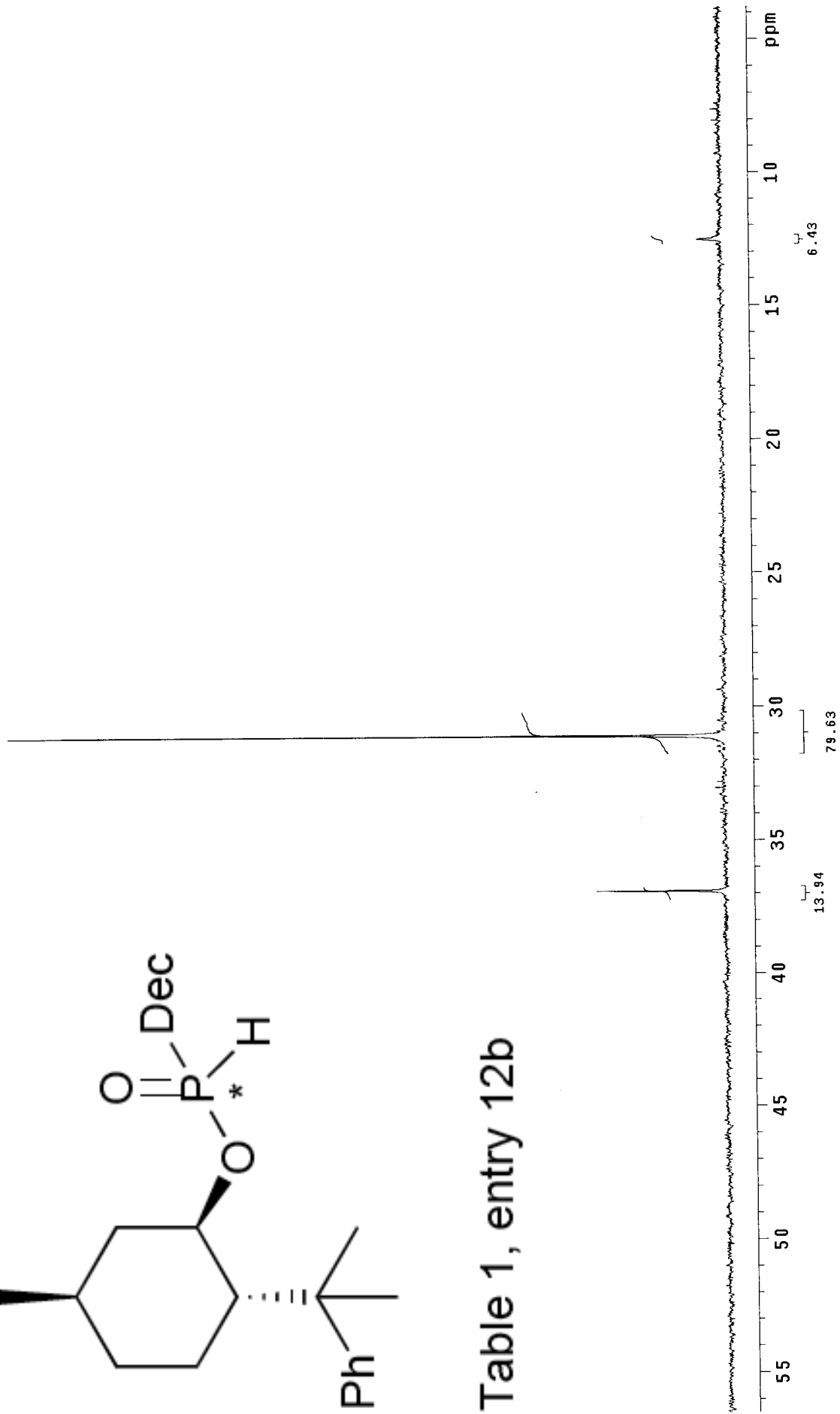


Table 1, entry 12b



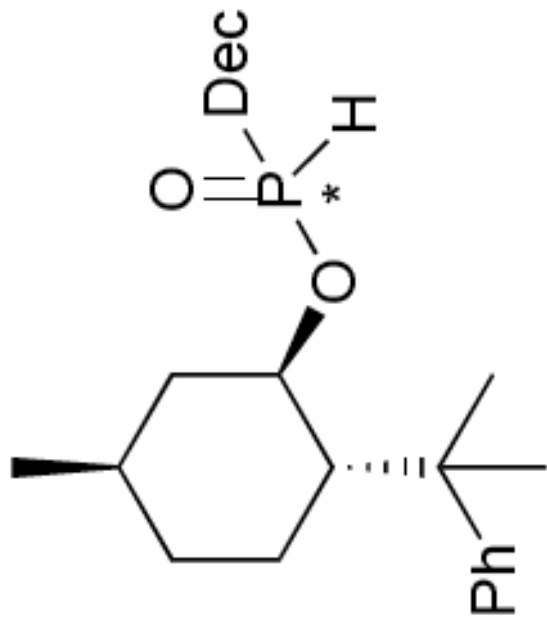
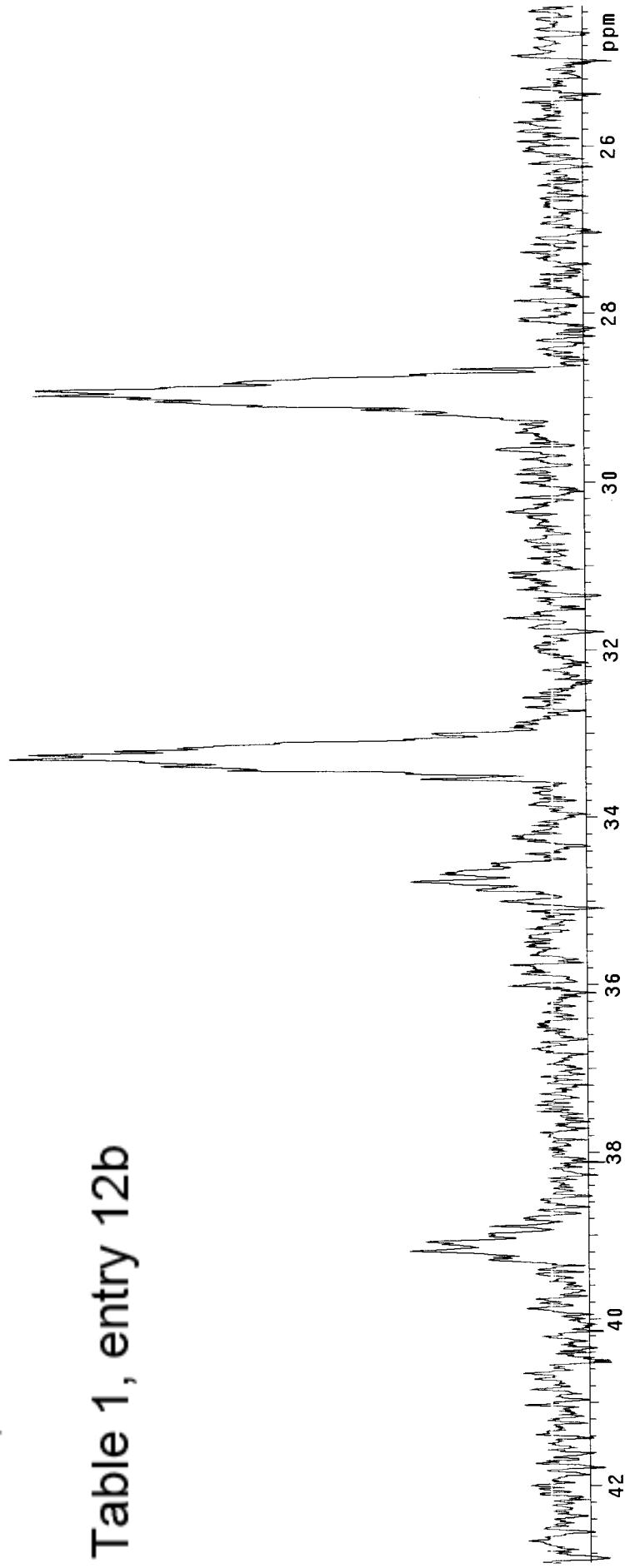


Table 1, entry 12b



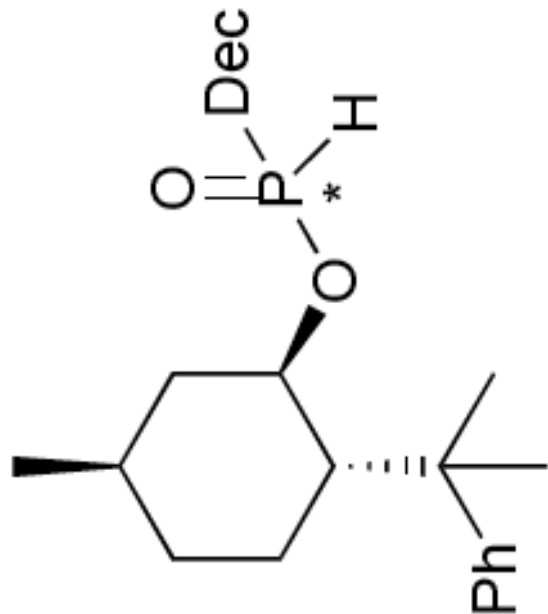
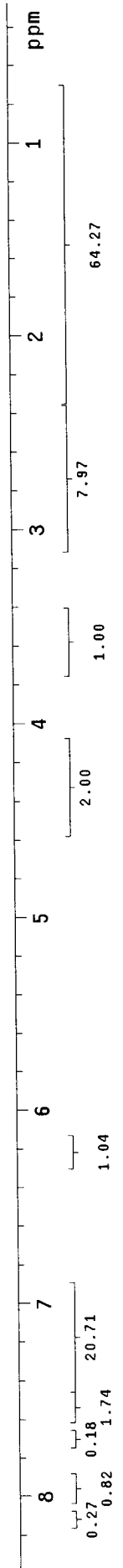


Table 1, entry 12b

| INDEX | FREQUENCY | PPM | HEIGHT | II | IT | INDEX | FREQUENCY | PPM | HEIGHT | IND |
|-------|-----------|-------|--------|----|-----|-------|-----------|-------|--------|-----|
| 1 | 2433.784 | 8.111 | 3.9 | | 1.6 | 118 | 776.864 | 2.589 | 5.4 | 157 |
| 2 | 2386.879 | 7.955 | 10.4 | | 1.0 | 119 | 774.812 | 2.582 | 3.5 | 158 |
| 3 | 2384.827 | 7.948 | 13.0 | | 1.1 | 120 | 771.001 | 2.570 | 5.7 | 159 |
| 4 | 2383.948 | 7.945 | 13.6 | | 1.6 | 121 | 768.656 | 2.562 | 3.0 | 160 |
| 5 | 2381.896 | 7.938 | 10.1 | | 1.2 | 122 | 652.566 | 2.175 | 13.3 | 161 |
| 6 | 2308.900 | 7.895 | 4.0 | | 1.5 | 123 | 641.719 | 2.139 | 4.0 | 162 |
| 7 | 2264.047 | 7.546 | 4.2 | | 1.6 | 124 | 635.856 | 2.119 | 7.2 | 163 |
| 8 | 2262.581 | 7.541 | 4.8 | | 1.3 | 125 | 629.993 | 2.100 | 9.7 | 164 |
| 9 | 2261.115 | 7.536 | 18.5 | | 1.2 | 126 | 626.768 | 2.089 | 11.4 | 165 |
| 10 | 2259.942 | 7.532 | 19.2 | | 1.0 | 127 | 619.753 | 2.065 | 11.2 | 166 |
| 11 | 2256.131 | 7.519 | 4.9 | | 1.4 | 128 | 618.267 | 2.061 | 11.2 | 167 |
| 12 | 2254.666 | 7.514 | 5.5 | | 1.5 | 129 | 614.749 | 2.049 | 8.0 | 168 |
| 13 | 2253.200 | 7.509 | 21.1 | | 1.6 | 130 | 608.006 | 2.026 | 7.7 | 169 |
| 14 | 2252.027 | 7.505 | 21.0 | | 3.9 | 131 | 604.488 | 2.015 | 6.5 | 170 |
| 15 | 2225.936 | 7.419 | 10.8 | | 3.4 | 132 | 560.222 | 1.867 | 3.6 | 171 |
| 16 | 2224.471 | 7.414 | 14.9 | | 2.8 | 133 | 558.170 | 1.860 | 4.0 | 172 |
| 17 | 2222.418 | 7.407 | 4.2 | | 3.3 | 134 | 554.066 | 1.847 | 2.8 | 173 |
| 18 | 2218.314 | 7.393 | 11.9 | | 1.0 | 135 | 551.720 | 1.839 | 3.2 | 174 |
| 19 | 2217.435 | 7.390 | 22.8 | | 4.5 | 136 | 547.909 | 1.826 | 4.0 | 175 |
| 20 | 2216.262 | 7.386 | 22.9 | | 3.5 | 137 | 545.271 | 1.817 | 4.4 | 176 |
| 21 | 2214.210 | 7.379 | 4.6 | | 4.7 | 138 | 540.874 | 1.803 | 4.3 | 177 |
| 22 | 2211.278 | 7.370 | 22.5 | | 3.8 | 139 | 538.528 | 1.795 | 2.9 | 178 |
| 23 | 2209.813 | 7.365 | 29.4 | | 5.6 | 140 | 534.717 | 1.782 | 3.0 | 179 |
| 24 | 2208.054 | 7.359 | 9.4 | | 4.7 | 141 | 532.958 | 1.776 | 3.0 | 180 |
| 25 | 2204.536 | 7.347 | 28.7 | | 5.5 | 142 | 530.027 | 1.766 | 6.1 | 181 |
| 26 | 2202.777 | 7.341 | 49.5 | | 5.7 | 143 | 526.509 | 1.755 | 9.5 | 182 |
| 27 | 2201.604 | 7.337 | 46.3 | | 5.5 | 144 | 522.991 | 1.743 | 11.6 | 183 |
| 28 | 2199.552 | 7.331 | 10.1 | | 5.8 | 145 | 519.766 | 1.732 | 15.6 | 184 |
| 29 | 2197.207 | 7.323 | 22.2 | | 5.8 | 146 | 517.128 | 1.723 | 26.1 | 185 |
| 30 | 2195.741 | 7.318 | 12.5 | | 5.4 | 147 | 513.903 | 1.713 | 14.1 | 186 |
| 31 | 2193.982 | 7.312 | 7.3 | | 6.2 | 148 | 509.506 | 1.698 | 17.7 | 187 |
| 32 | 2191.051 | 7.302 | 10.1 | | 6.1 | 149 | 507.747 | 1.692 | 17.8 | 188 |
| 33 | 2189.292 | 7.296 | 16.6 | | 6.0 | 150 | 506.281 | 1.687 | 18.5 | 189 |
| 34 | 2186.653 | 7.288 | 45.6 | | 6.0 | 151 | 503.349 | 1.678 | 24.7 | 190 |
| 35 | 2184.308 | 7.280 | 18.7 | | 5.8 | 152 | 497.193 | 1.657 | 13.3 | 191 |
| 36 | 2182.842 | 7.275 | 15.3 | | 5.3 | 153 | 493.382 | 1.644 | 11.0 | 192 |
| 37 | 2179.031 | 7.262 | 148.4 | | 6.1 | 154 | 491.330 | 1.637 | 12.8 | 193 |
| 38 | 2176.686 | 7.254 | 26.1 | | 6.5 | 155 | 488.682 | 1.629 | 12.5 | 194 |
| 39 | 2175.513 | 7.250 | 25.6 | | 3.1 | 156 | 486.053 | 1.620 | 13.2 | 195 |

| | | | |
|-----|-----|---------|-------|
| 3.4 | 111 | 791.229 | 2.637 |
| 3.0 | 112 | 788.884 | 2.629 |
| 3.8 | 113 | 787.711 | 2.625 |
| 3.6 | 114 | 785.366 | 2.617 |
| 8.3 | 115 | 782.728 | 2.609 |
| 6.3 | 116 | 781.555 | 2.605 |
| 6.2 | 117 | 779.210 | 2.597 |



414 pure

```
exp1 s2pu1
SAMPLE
date Sep 24 2004 temp not used
solvent CDC13 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
fb 10400
bs 64
ss 4
d1 1.000 hs
nt 256 lb
ct 256 fn
TRANSMITTER C13
sfrq 75.456 SP
tof 737.2 WP
tpwr 58 rf1
pw 9.250 rfp
DECOUPLER H1 lp
dn 0
dof 0 WC 250
dm YVY W SC 0
dim 35 VS 151
dpwr 6700 tn 18
dmf ai no ph
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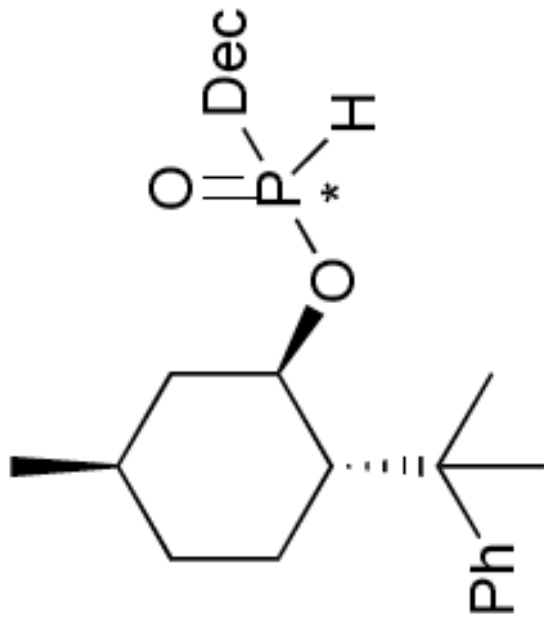
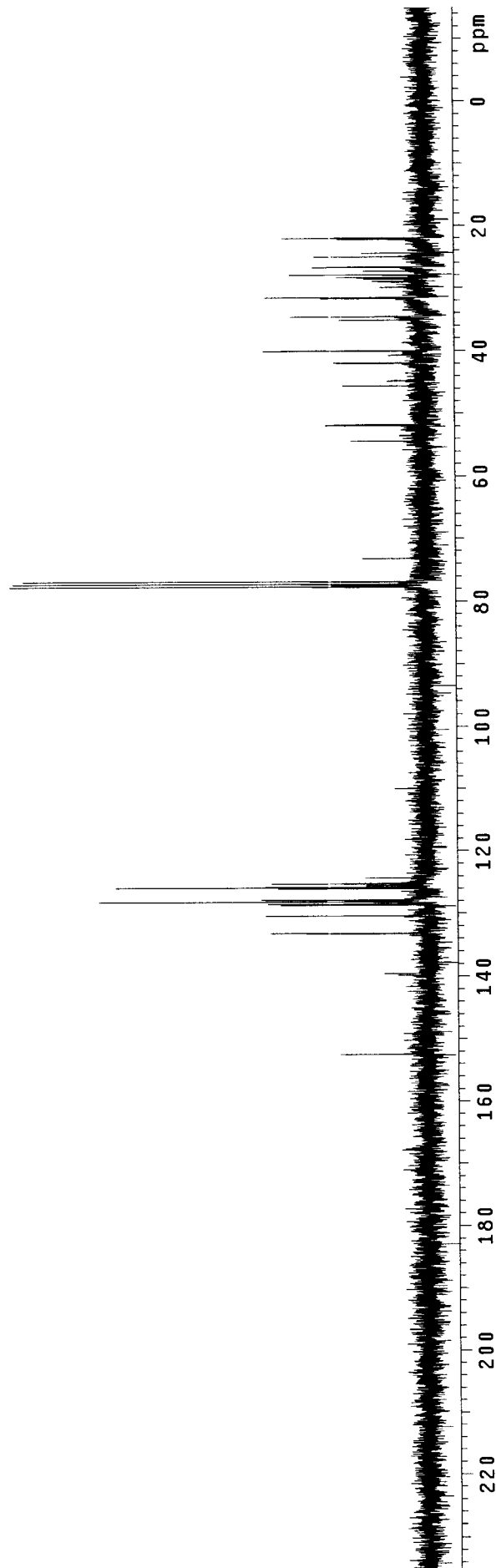


Table 1, entry 12b



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 2419.772 | 19.922 | 48.1 |
| 2 | 2101.949 | 17.305 | 19.6 |

4910 - 61 km/h centrm
a - 1.5 h.

exp1 s2pu1

| SAMPLE | | SPECIAL | |
|-----------------|-------------|------------|----------|
| date | Jan 13 2005 | temp | not used |
| solvent | CDCl3 | gain | not used |
| file | | spin | 20 |
| ACQUISITION | | | |
| 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 |
| dl | 1.000 | hs | nn |
| nt | 8 | PROCESSING | |
| ct | 8 | lb | 2.00 |
| TRANSMITTER P31 | | | |
| tn | | fn | not used |
| sfrq | 121.474 | sp | DISPLAY |
| tof | 10608.2 | wp | 10010.0 |
| tpwr | 55 | rfl | 2437.3 |
| pw | 7.117 | rff | 0 |
| DECOUPLER | | | |
| dn | H1 | tp | -0.1 |
| dof | 0 | PLOT | |
| dm | YYY | wc | 250 |
| dimm | 3 | sc | 0 |
| dpwr | 35 | vs | 27 |
| dmf | 6700 | th | 12 |
| | | ai | no |
| | | ph | |

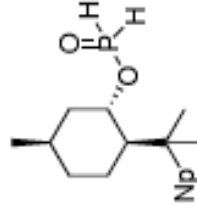
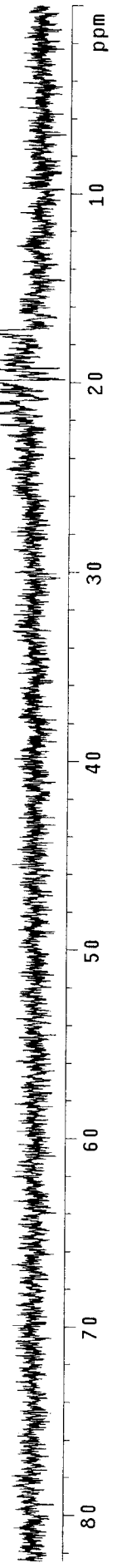


Table 1, entry 13



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 2699.653 | 22.226 | 14.2 |
| 2 | 2627.031 | 21.628 | 21.5 |
| 3 | 2567.056 | 21.135 | 19.3 |
| 4 | 2490.762 | 20.506 | 18.4 |
| 5 | 2418.956 | 19.915 | 129.9 |
| 6 | 2347.966 | 19.331 | 22.5 |
| 7 | 2286.768 | 18.827 | 16.4 |
| 8 | 2101.541 | 17.302 | 53.7 |

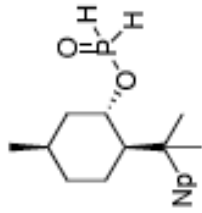
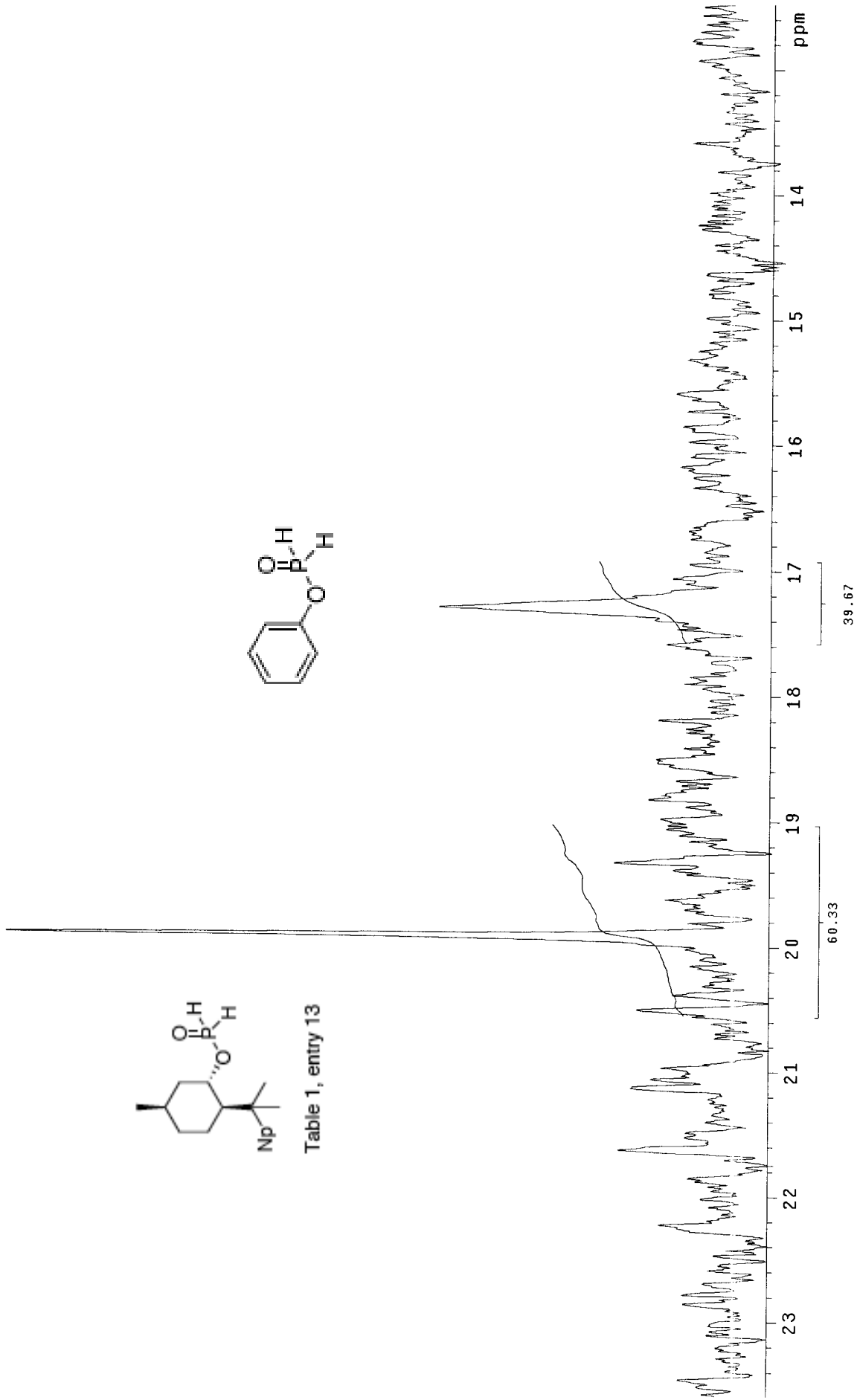


Table 1, entry 13



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 2992.181 | 24.635 | 21.5 |
| 2 | 2984.837 | 24.574 | 22.6 |
| 3 | 2669.869 | 21.981 | 22.8 |
| 4 | 2422.628 | 19.945 | 36.2 |
| 5 | 2102.765 | 17.312 | 41.5 |
| 6 | 1847.772 | 15.213 | 20.6 |
| 7 | 1534.844 | 12.636 | 18.6 |

```

a
exp1 s2pu1
SAMPLE
date Jan 13 2005 temp not used
solvent CDC13 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
fb 14800 il n
bs 64 in n
ss 4 dp y
d1 1.000 hs nn
nt 8
ct TRANSMITTER 8 lb 2.00
P31 fn not used
sfrq 121.474 sp DISPLAY 0
tof 10608.2 wp 10010.0
tpwr 55 rfi 2437.3
pw 7.117 rfp 0
DECOUPLER H1 lp -39.5
dn dof 0 PLOT -266.3
dm ynn wc 250
dmm w sc 0
dpwr 35 vs 51
dmf 6700 th ai no ph 13

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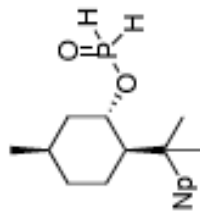
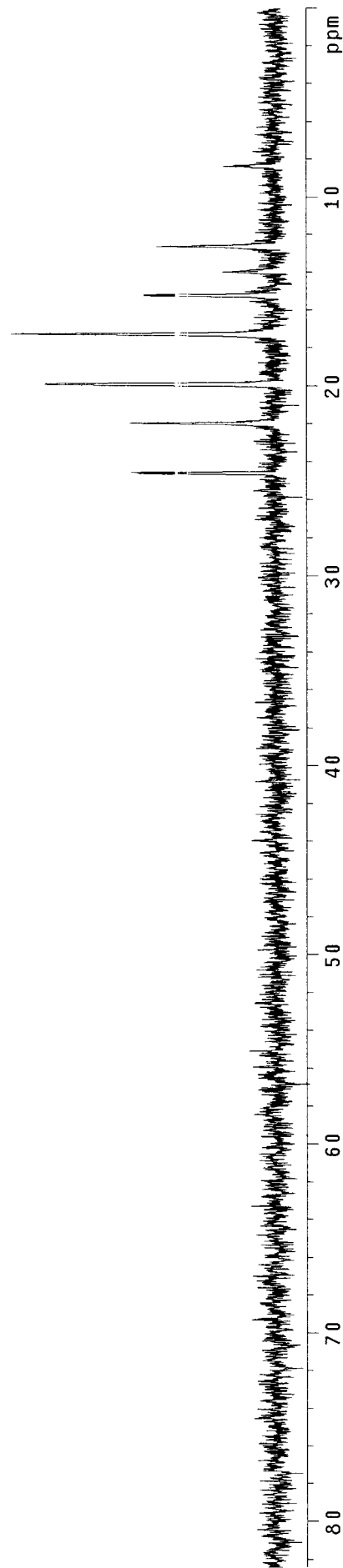


Table 1, entry 13



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 2992.181 | 24.835 | 72.8 |
| 2 | 2984.837 | 24.574 | 76.7 |
| 3 | 2669.869 | 21.981 | 77.3 |
| 4 | 2422.628 | 19.945 | 122.7 |
| 5 | 2102.765 | 17.312 | 140.9 |
| 6 | 1847.772 | 15.213 | 69.9 |
| 7 | 1700.487 | 14.000 | 27.8 |
| 8 | 1534.844 | 12.836 | 63.0 |
| 9 | 1015.474 | 8.360 | 27.2 |

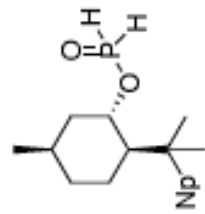
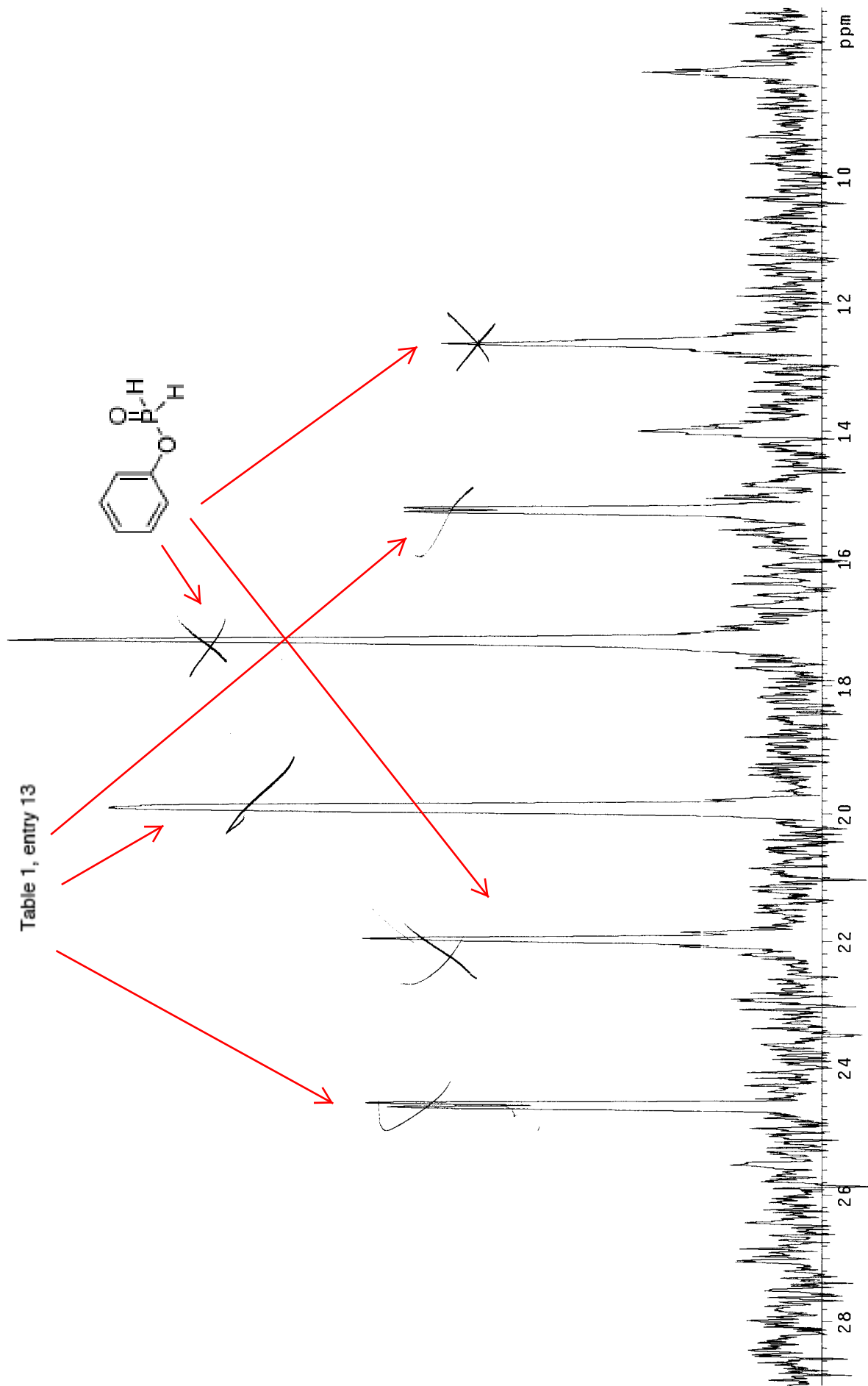
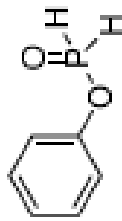


Table 1, entry 13



Interpolated Peak Listing

| PEAK | POINT | HEIGHT | REL. HT | HZ | PPM |
|------|-------|--------|---------|--------|--------|
| 1 | 23872 | 105280 | 96.32 | 939.39 | 25.779 |

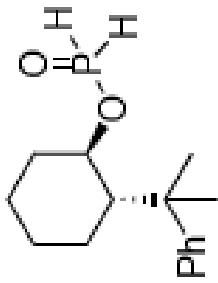
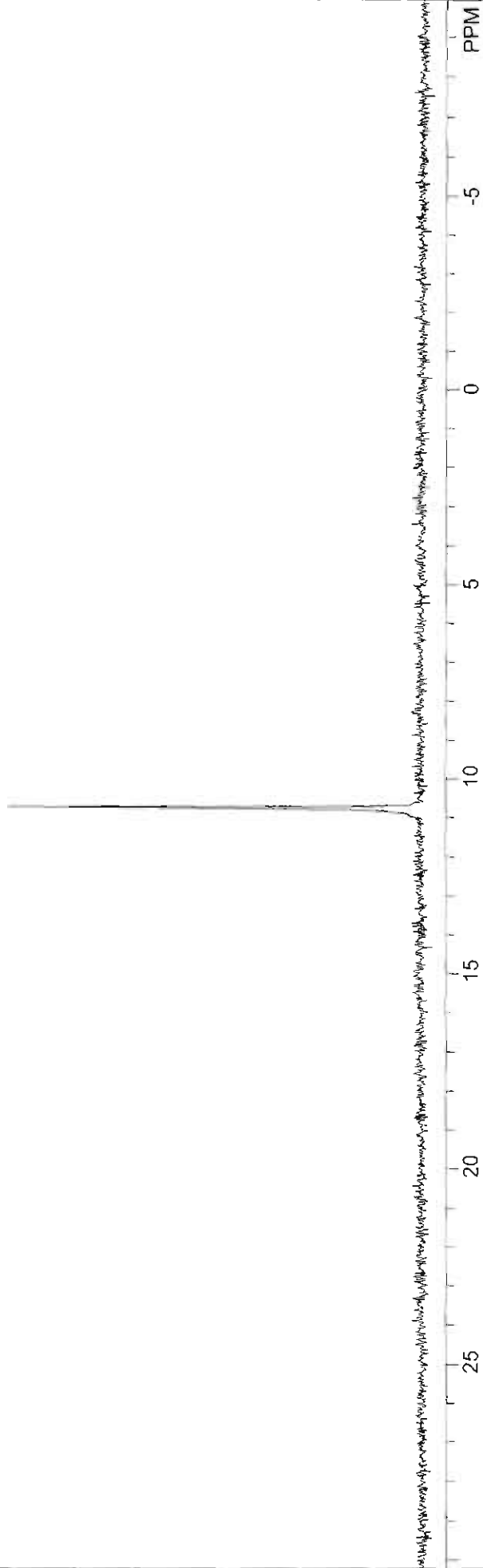


Table 2, entry 1



| | | | | |
|--------------------------|---------------|------------|--------------|---------------------------------|
| FI: 36.441 | SW1: 20000 | OF: 5510.0 | PTSId: 32768 | USER: -- DATE: 09/23/05 (13:21) |
| EX: c:\efl\global\zg.ppg | PW: 12.4 usec | NA: 16 | LB: 0.0 | |

173.3
-183.3

| LINE | POINT | HZ | PPM | INTENSITY | REF_INT | LABEL |
|------|-------|---------|--------|-----------|---------|-------|
| 1 | 23829 | 965.84 | 26.504 | 15172 | 48.715 | 1 |
| 2 | 23846 | 955.19 | 26.212 | 13058 | 41.928 | 2 |
| 3 | 24763 | 395.46 | 10.852 | 26975 | 86.614 | 3 |
| 4 | 24779 | 385.97 | 10.592 | 29026 | 93.199 | 4 |
| 5 | 25696 | -173.56 | -4.763 | 12626 | 40.540 | 5 |
| 6 | 25712 | -183.30 | -5.030 | 13814 | 44.354 | 6 |

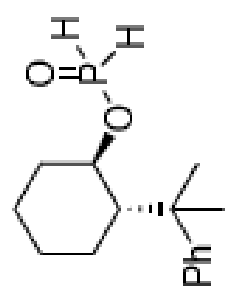
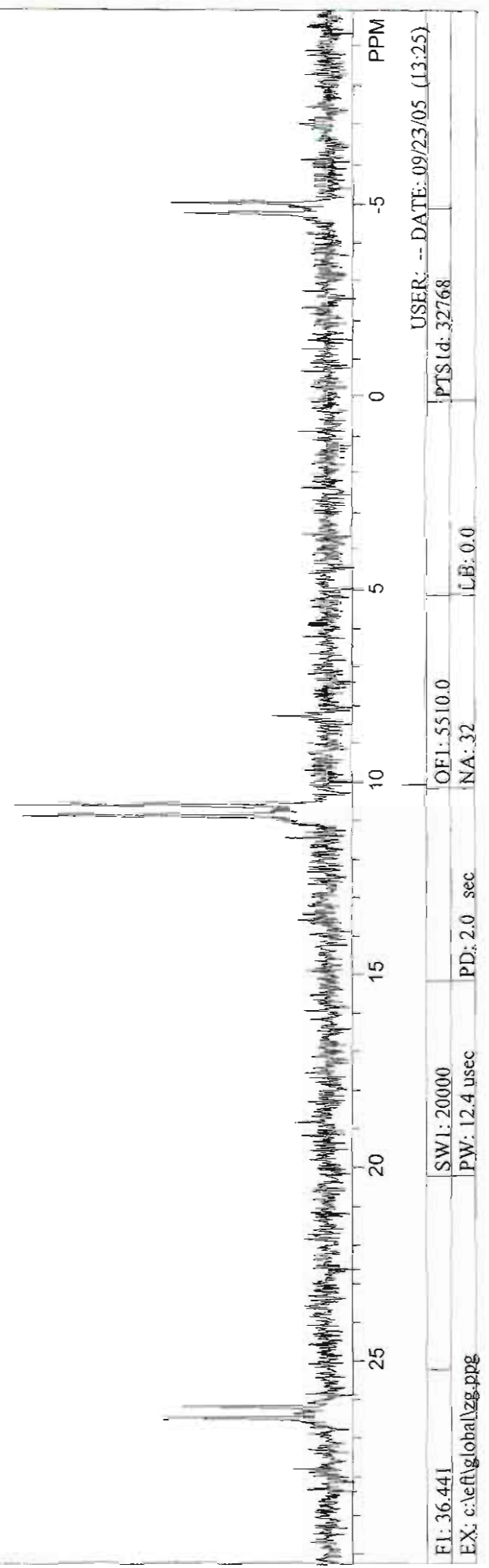


Table 2, entry 1



FI: 36.441
 EX: c:\efl\global\zg.ppg
 SW1: 20000
 PW: 12.4 usec
 PD: 2.0 sec
 OF1: 5510.0
 NA: 32
 LB: 0.0
 PTSId: 32768
 USER: -- DATE: 09/23/05 (13:25)

| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 4764.893 | 39.229 | 45.4 |
| 2 | 4058.256 | 33.412 | 126.0 |

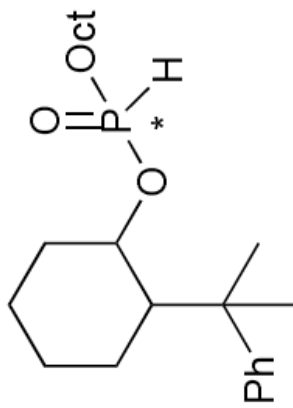
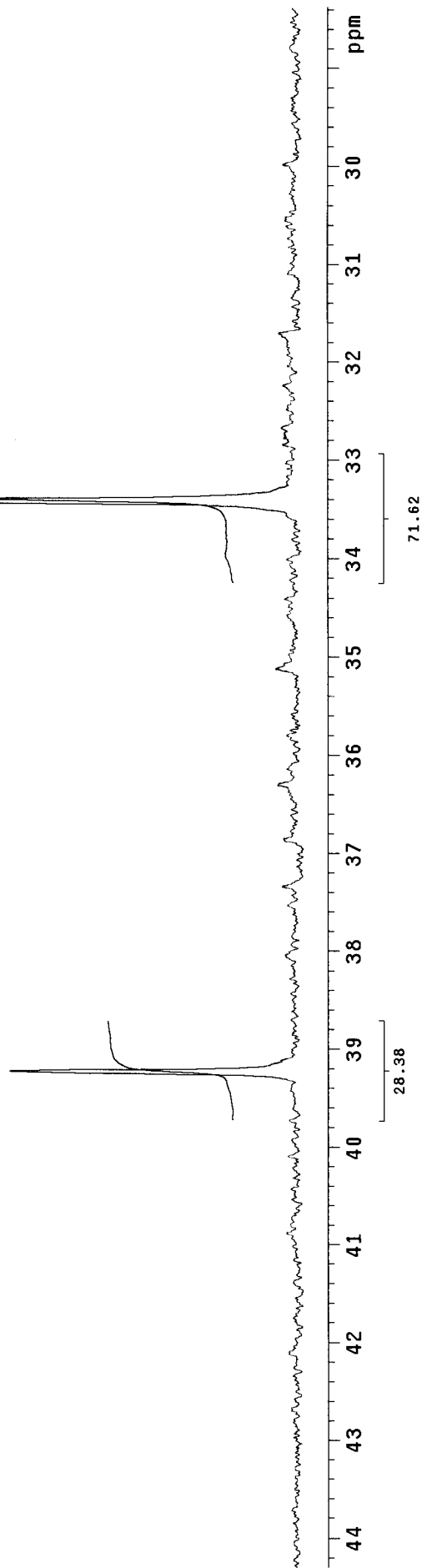


Table 2, entry 1a



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 5076.189 | 41.792 | 42.4 |
| 2 | 5065.173 | 41.701 | 34.7 |
| 3 | 5051.301 | 41.587 | 19.9 |
| 4 | 4547.027 | 37.436 | 29.3 |
| 5 | 4534.787 | 37.335 | 34.9 |
| 6 | 4339.352 | 35.890 | 124.0 |
| 7 | 4353.232 | 35.840 | 124.1 |
| 8 | 3832.230 | 31.551 | 124.9 |

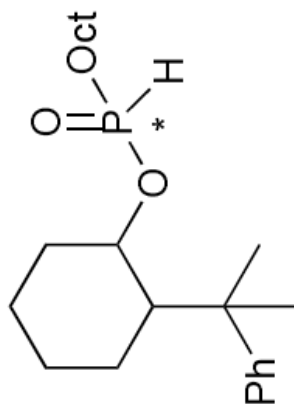
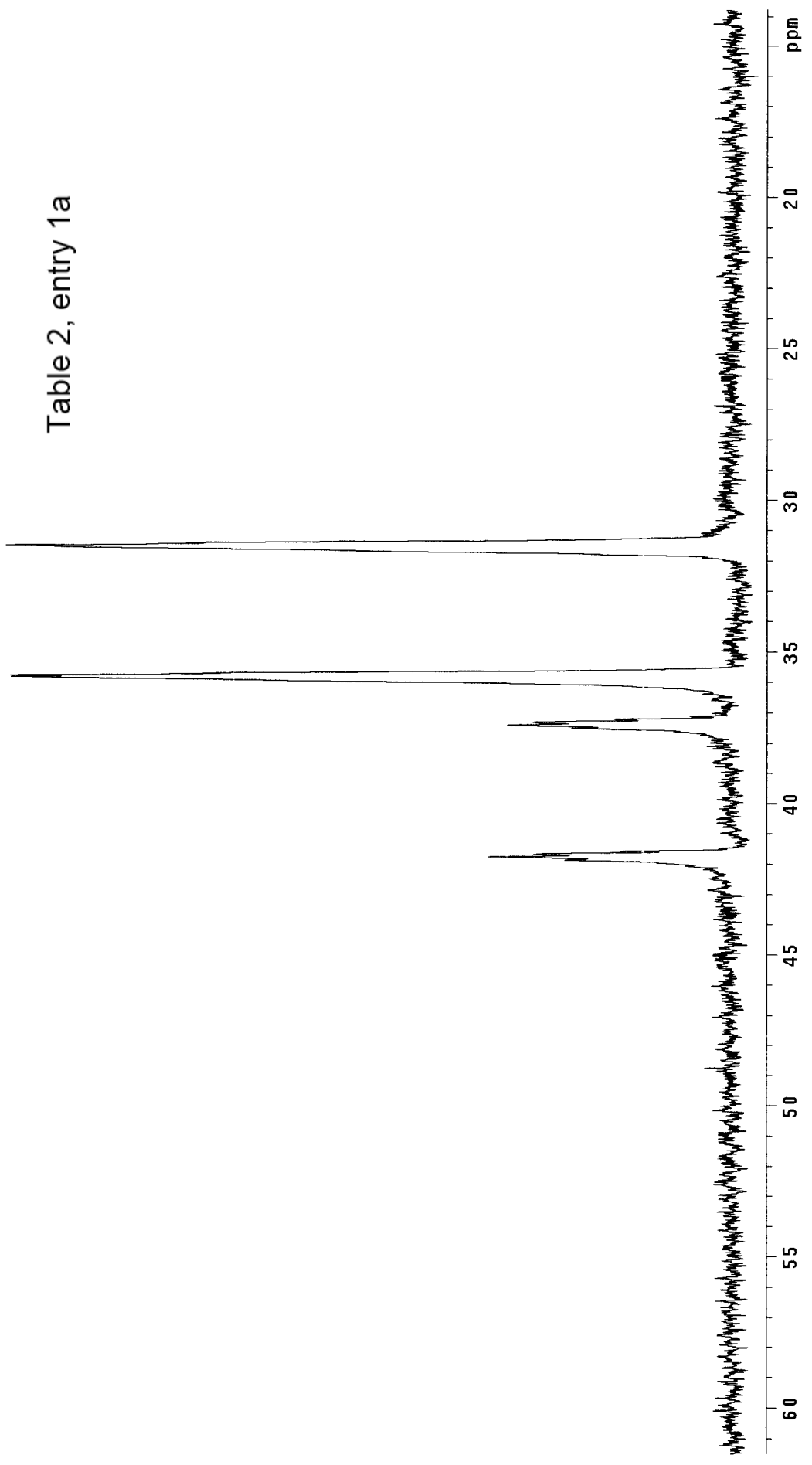


Table 2, entry 1a



100% FREQUENCY 800 MHz

FREQUENCY (PPM) HEIGHT

INDEX

HEIGHT

FREQUENCY PPM

INDEX

HEIGHT

FREQUENCY PPM

INDEX

FREQUENCY PPM

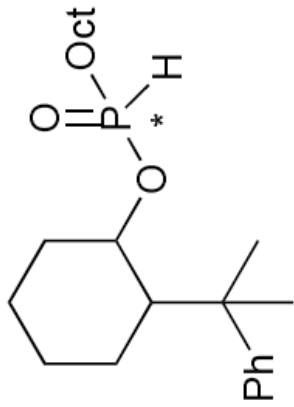
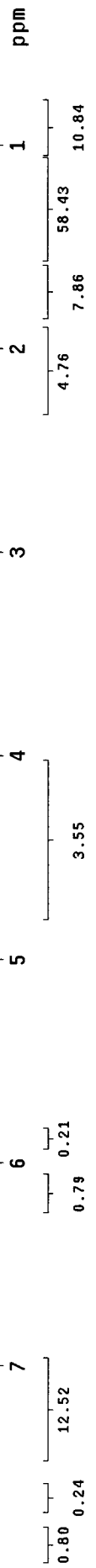


Table 2, entry 1a

| | | | | | | | |
|----|----------|-------|------|----|---------|-------|-------|
| 1 | 2360.788 | 7.868 | 8.9 | 40 | 645.823 | 2.152 | 5.6 |
| 2 | 2293.949 | 7.645 | 2.8 | 41 | 642.012 | 2.140 | 5.0 |
| 3 | 2293.069 | 7.642 | 2.6 | 42 | 633.804 | 2.112 | 9.9 |
| 4 | 2225.057 | 7.416 | 2.1 | 43 | 623.544 | 2.078 | 8.4 |
| 5 | 2223.591 | 7.411 | 2.8 | 44 | 610.938 | 2.036 | 12.0 |
| 6 | 2216.555 | 7.387 | 4.5 | 45 | 539.115 | 1.797 | 2.5 |
| 7 | 2215.383 | 7.383 | 4.5 | 46 | 522.111 | 1.740 | 10.9 |
| 8 | 2206.881 | 7.355 | 13.3 | 47 | 519.180 | 1.730 | 10.9 |
| 9 | 2205.709 | 7.351 | 17.6 | 48 | 508.040 | 1.693 | 17.1 |
| 10 | 2203.856 | 7.344 | 8.1 | 49 | 496.900 | 1.656 | 9.7 |
| 11 | 2198.380 | 7.327 | 34.9 | 50 | 467.291 | 1.557 | 2.1 |
| 12 | 2197.500 | 7.324 | 33.0 | 51 | 464.067 | 1.547 | 2.3 |
| 13 | 2187.533 | 7.291 | 39.7 | 52 | 454.392 | 1.514 | 3.6 |
| 14 | 2183.135 | 7.276 | 15.0 | 53 | 450.874 | 1.503 | 3.2 |
| 15 | 2180.497 | 7.267 | 35.7 | 54 | 435.044 | 1.450 | 95.9 |
| 16 | 2172.289 | 7.240 | 16.4 | 55 | 429.767 | 1.432 | 23.6 |
| 17 | 2159.683 | 7.198 | 2.6 | 56 | 415.989 | 1.386 | 9.3 |
| 18 | 2152.647 | 7.174 | 2.9 | 57 | 410.712 | 1.369 | 35.7 |
| 19 | 2150.595 | 7.167 | 2.1 | 58 | 392.536 | 1.308 | 26.5 |
| 20 | 2146.198 | 7.153 | 3.6 | 59 | 387.553 | 1.292 | 42.1 |
| 21 | 2141.214 | 7.136 | 11.6 | 60 | 377.292 | 1.257 | 82.0 |
| 22 | 2139.748 | 7.131 | 7.7 | 61 | 369.377 | 1.231 | 162.0 |
| 23 | 2133.885 | 7.112 | 15.3 | 62 | 365.859 | 1.219 | 95.5 |
| 24 | 2128.315 | 7.093 | 5.1 | 63 | 346.511 | 1.155 | 14.0 |
| 25 | 2126.849 | 7.088 | 6.6 | 64 | 343.286 | 1.144 | 14.8 |
| 26 | 1842.781 | 6.142 | 5.7 | 65 | 335.957 | 1.120 | 11.6 |
| 27 | 1840.436 | 6.134 | 9.8 | 66 | 333.319 | 1.111 | 12.1 |
| 28 | 1763.922 | 5.879 | 3.1 | 67 | 330.680 | 1.102 | 12.3 |
| 29 | 1586.269 | 5.287 | 6.4 | 68 | 320.127 | 1.067 | 9.0 |
| 30 | 1327.705 | 4.425 | 2.7 | 69 | 308.107 | 1.027 | 6.2 |
| 31 | 1324.480 | 4.414 | 2.7 | 70 | 305.176 | 1.017 | 5.8 |
| 32 | 1321.256 | 4.403 | 5.3 | 71 | 296.088 | 0.987 | 3.5 |
| 33 | 1317.152 | 4.390 | 4.9 | 72 | 284.655 | 0.949 | 3.1 |
| 34 | 1314.513 | 4.381 | 4.7 | 73 | 273.515 | 0.912 | 26.9 |
| 35 | 1310.409 | 4.367 | 5.4 | 74 | 267.065 | 0.890 | 67.6 |
| 36 | 1306.891 | 4.356 | 2.9 | 75 | 264.134 | 0.880 | 20.8 |
| 37 | 1304.253 | 4.347 | 2.4 | 76 | 260.030 | 0.867 | 30.7 |
| 38 | 1299.855 | 4.332 | 2.2 | 77 | 256.805 | 0.856 | 11.3 |
| 39 | 649.635 | 2.165 | 6.1 | 78 | 249.183 | 0.830 | 4.5 |



655-2

exp1 s2pu1

SAMPLE SPECIAL
date Oct 4 2005 temp not used
solvent CDC13 gain not used
file exp 20
ACQUISITION hst 0.008
sw 18867.9 pw90 18.500
at 1.815 alfa 20.000
np 68492
fb 10400 il
bs 64 in n
ss 4 dp in n
d1 1.000 hs rn
nt 1000
ct TRANSMITTER 576 1b 1.00 not used
tn C13 DISPLAY
sfrq 75.456 sp -150.9
tof 737.2 wp 15844.1
tpwr 58 rfl 1134.7
pw 9.250 rfp 0
DECOUPLER H1 lp -241.8
dn 0
dof yyv wc 250
dm w sc 0
dmm 35 vs 172
dpwr 6700 th 25
dmf ai no ph

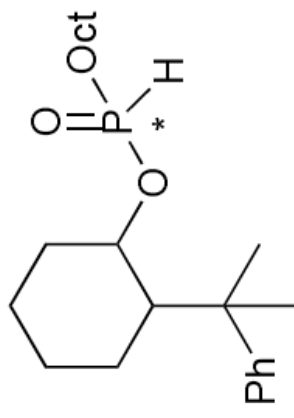
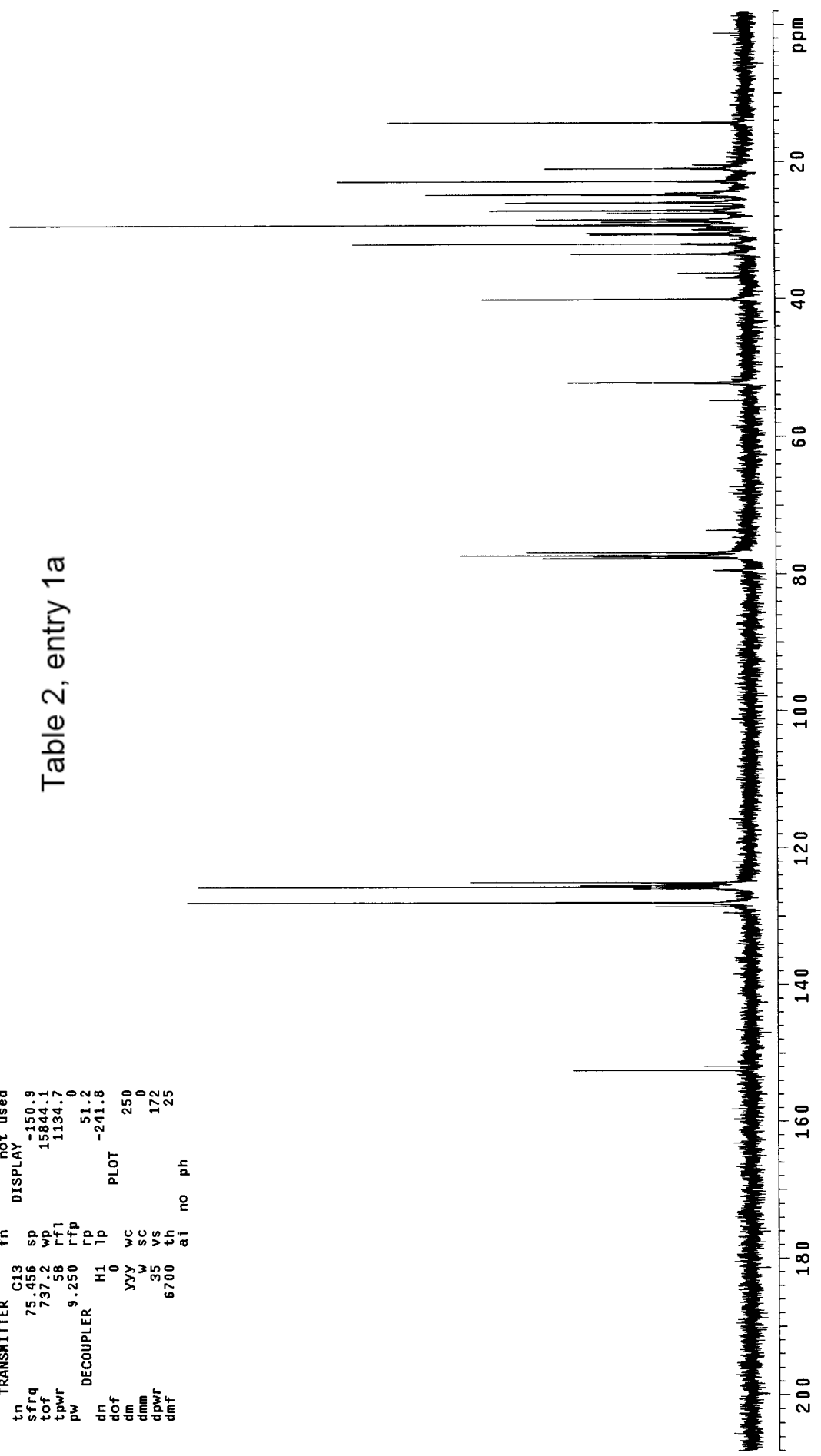
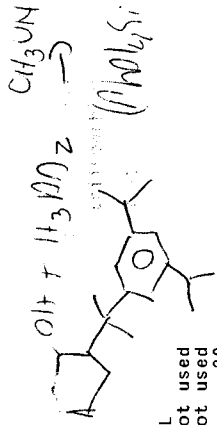


Table 2, entry 1a



754 (ester)



triisopropylbenz-cyclohex

expi s2pul

| | | | |
|-------------|------------|------|----------|
| date | Nov 7 2005 | temp | not used |
| solvent | CDC13 | gain | not used |
| file | | spin | 20 |
| ACQUISITION | | | |
| sw | 26738.0 | hst | 0.008 |
| at | 1.598 | pw90 | 18.300 |
| np | 85476 | alfa | 20.000 |
| bs | 14800 | ll | n |
| fb | 64 | in | n |
| ss | 4 | dp | y |
| d1 | 1.000 | hs | nm |
| nt | 16 | lb | nm |
| ct | 16 | fn | not used |
| TRANSMITTER | | | |
| tn | P31 | sp | 0 |
| sfrq | 121.474 | wp | 10010.0 |
| tof | 10608.2 | rfl | 2437.3 |
| tpwr | 55 | rff | 0 |
| pw | 7.117 | rp | -160.8 |
| DECOUPLER | | | |
| dn | H1 | lp | -38.4 |
| dof | 0 | vc | 250 |
| dm | VVY | w | 0 |
| dmm | W | sc | 0 |
| dpwr | 35 | vs | 12 |
| dmf | 6700 | th | 3 |
| | | ai | no |
| | | ph | |

SPECIAL

not used

not used

0.008

18.300

20.000

FLAGS

n

n

y

nm

2.00

not used

DISPLAY

0

10010.0

2437.3

0

-160.8

-38.4

PLOT

250

0

12

3

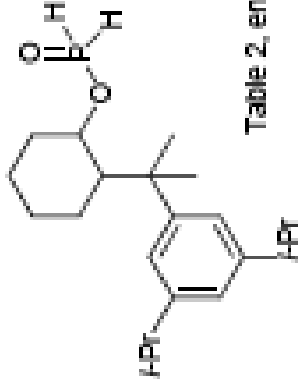
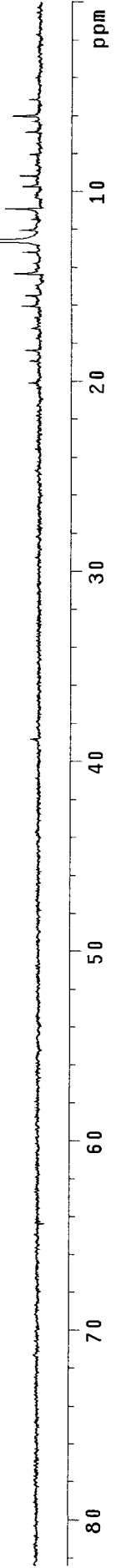


Table 2, entry 2



INDEX FREQUENCY PPM HEIGHT

| | | | |
|---|----------|--------|-------|
| 1 | 1742.102 | 14.343 | 4.2 |
| 2 | 1534.028 | 12.630 | 126.0 |
| 3 | 1463.854 | 12.052 | 3.3 |
| 4 | 1325.546 | 10.913 | 5.5 |
| 5 | 1115.839 | 9.187 | 3.3 |
| 6 | 733.554 | 6.039 | 4.3 |

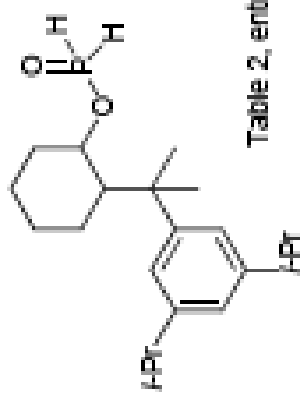
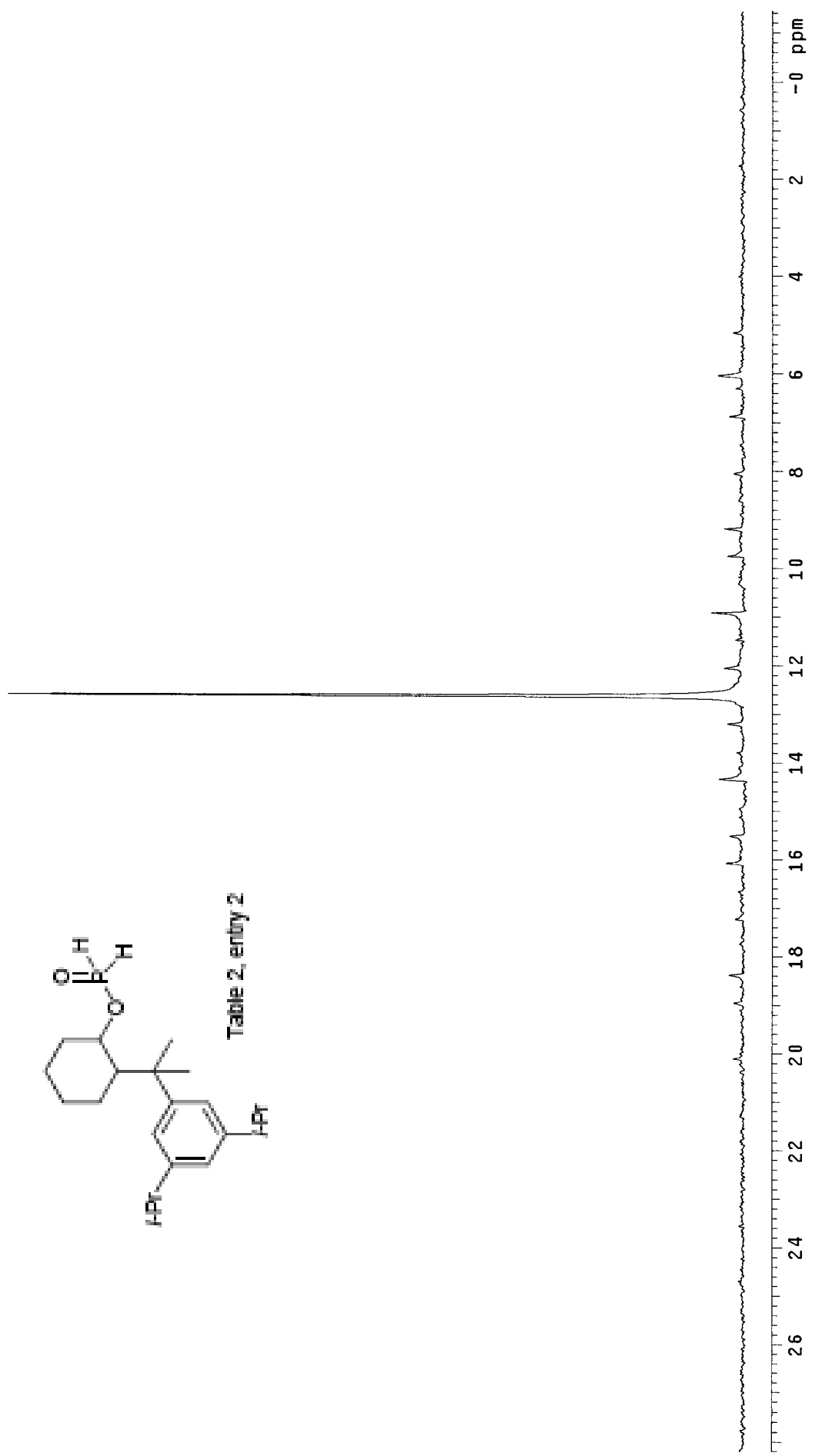


Table 2, entry 2



triisopropylbenz-cyclohex

exp1 s2pu1

| | | | |
|-------------|-------------|------------|----------|
| date | Nov 7 2005 | SPECIAL | not used |
| solvent | CDCl3 | gain | not used |
| file | | spin | 20 |
| ACQUISITION | exp | hst | 0.008 |
| sw | 26738.0 | pw90 | 18.300 |
| at | 1.598 | alfa | 20.000 |
| np | 85476 | ll | n |
| fb | 14800 | in | n |
| bs | 64 | in | n |
| ss | 4 | dp | y |
| d1 | 1.000 | hs | nm |
| nt | 8 | PROCESSING | 2.00 |
| ct | 8 | lb | not used |
| tn | TRANSMITTER | fn | not used |
| tn | P31 | DISPLAY | |
| sfrq | 121.474 | sp | 0 |
| tof | 10608.2 | wp | 10010.0 |
| tpwr | 55 | rf1 | 2437.3 |
| pw | 7.117 | rfp | 0 |
| DECOUPLER | H1 | rp | -148.1 |
| dn | 0 | lp | -83.4 |
| dof | | PLOT | |
| dm | Ynn | wc | 250 |
| dmm | w | sc | 0 |
| dpwr | 35 | vs | 35 |
| dmf | 6700 | th | 7 |
| | ai | no | |
| | | ph | |

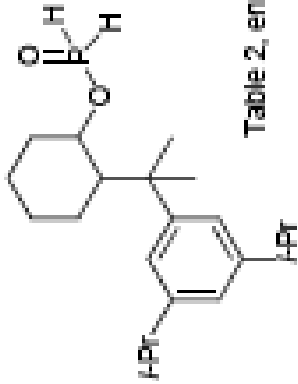
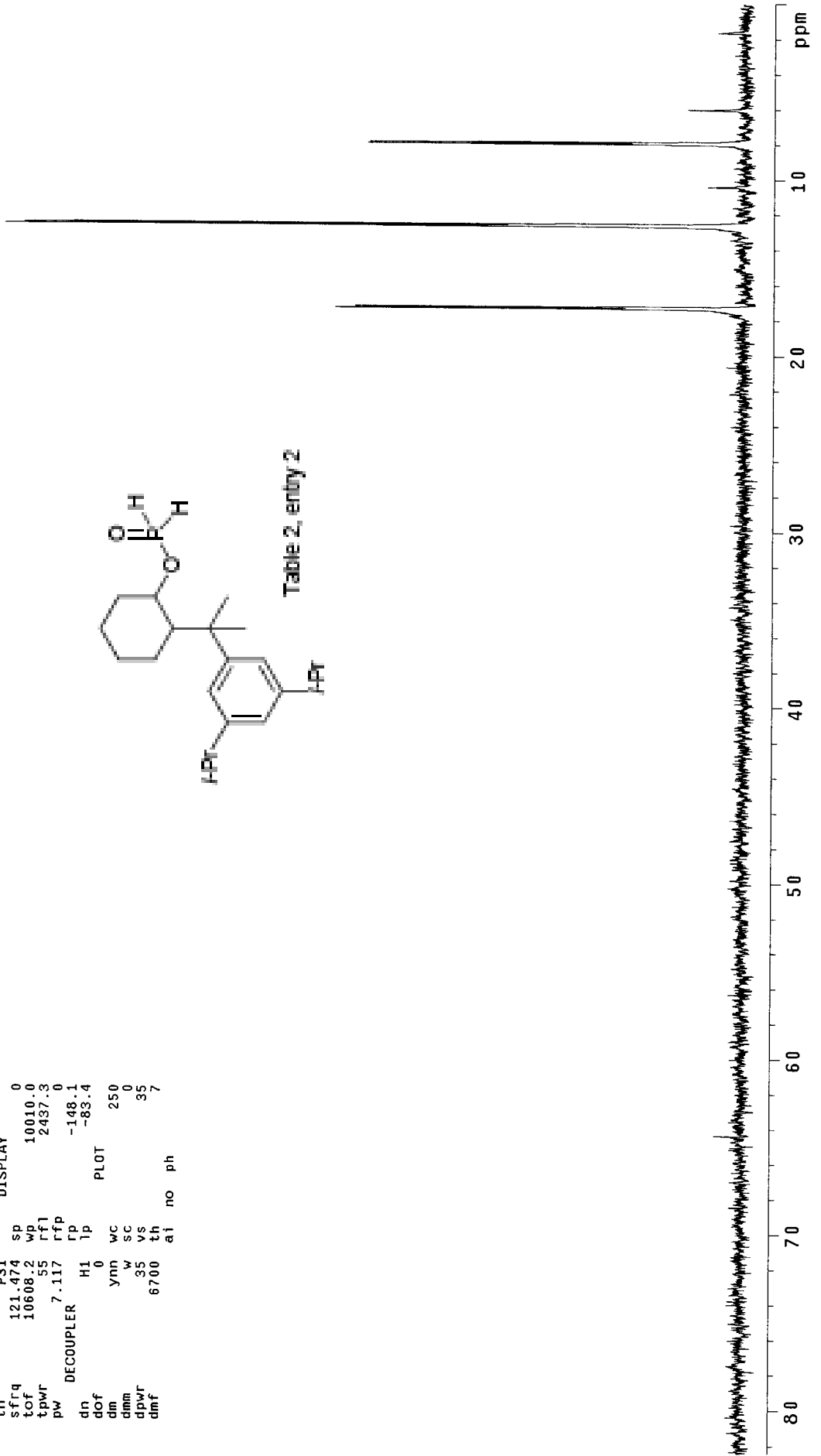


Table 2, entry 2



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 2107.253 | 17.349 | 69.7 |
| 2 | 2097.053 | 17.265 | 66.3 |
| 3 | 1538.108 | 12.663 | 126.0 |
| 4 | 1527.908 | 12.579 | 122.8 |
| 5 | 968.963 | 7.977 | 64.3 |
| 6 | 958.763 | 7.893 | 63.9 |
| 7 | 732.330 | 6.029 | 9.8 |

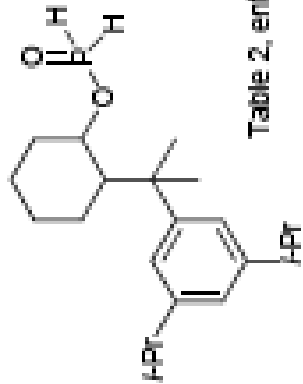
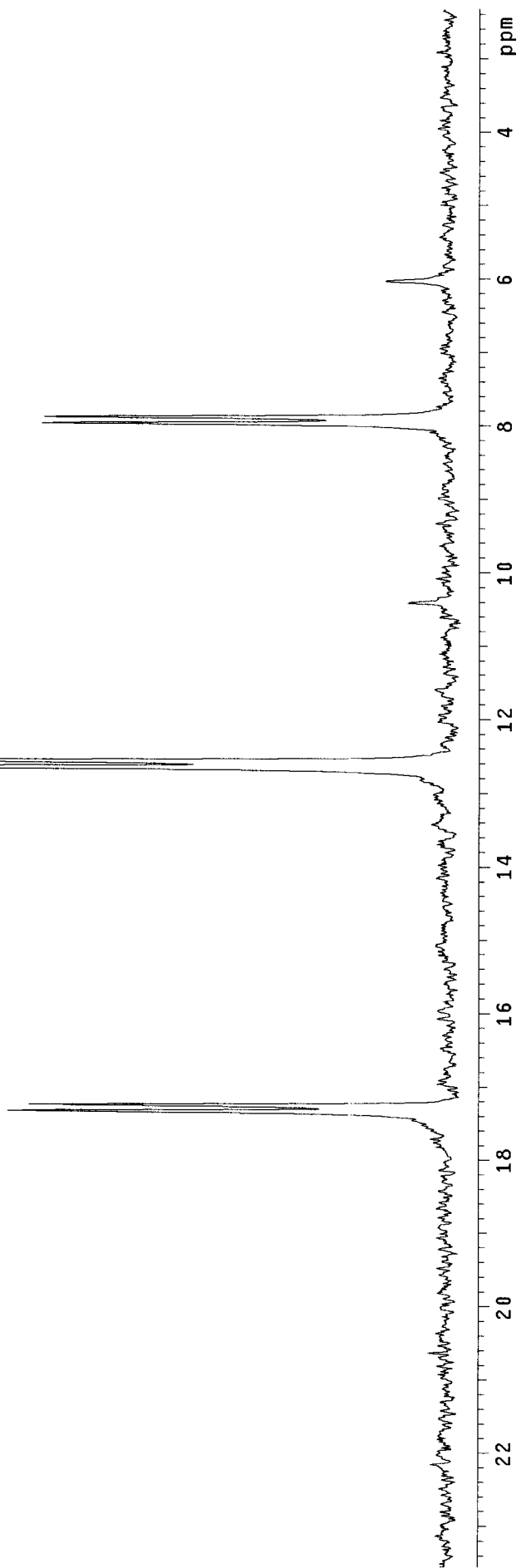


Table 2, entry 2



| INDEX | FREQUENCY PPM | HEIGHT |
|-------|---------------|--------|
| 1 | 4878.314 | 33.4 |
| 2 | 4227.571 | 85.1 |
| 3 | 4178.205 | 21.5 |

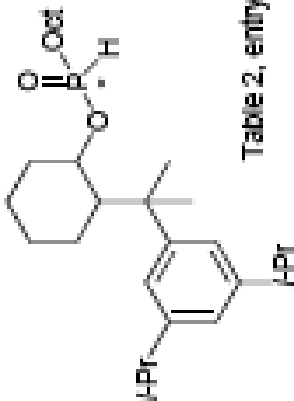
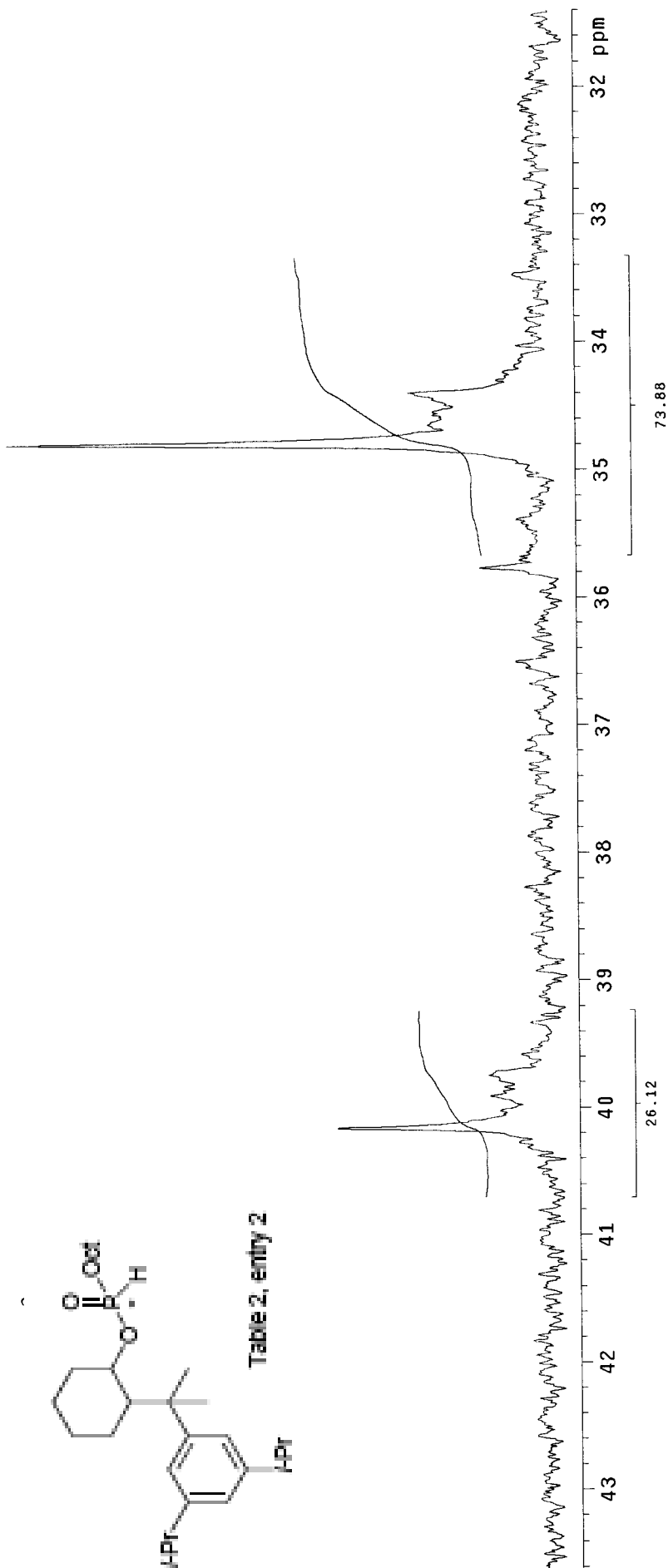


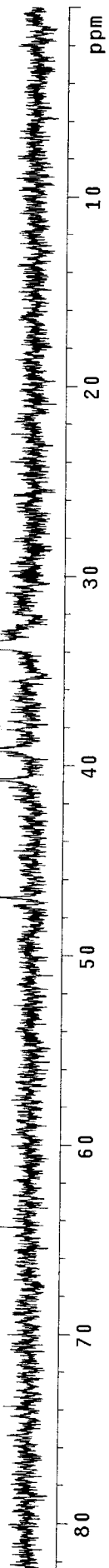
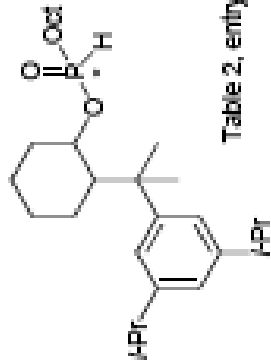
Table 2, entry 2



754

exp1 s2pu1

SAMPLE 9 2005
 date Nov 9 2005 temp not used
 solvent CDCl3 gain not used
 file CDC13 exp 20
 ACQUISITION
 sw 26738.0 hst 0.008
 at 1.598 pw90 18.300
 fb 85478 alfa 20.000
 bs 14800 il
 ss 64 in n
 di 4 dp y
 nt 1.000 hs nn
 ct 8 lb
 TRANSMITTER P31
 tn not used
 sfrq 121.474 sp
 tof 10608.2 wp 10010.0
 tpwr 55 rfl 2437.3
 pw 7.117 rfp 0
 DECOUPLER H1 lp
 dn 0
 dof YYY WC 250
 dm W SC 0
 dmm 3S VS 45
 dpwr 6700 th ai no ph 12
 dmf



754

exp1 s2pu1

| | | | |
|-------------|-------------|-------|------------|
| date | 9 2005 | temp | not used |
| solvent | Nov CDC13 | 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 | il | FLAGS |
| fb | 14800 | in | n |
| bs | 64 | in | n |
| ss | 4 | dp | y |
| d1 | 1.000 | hs | mn |
| nt | 16 | hs | PROCESSING |
| ct | 16 | lb | 2.00 |
| tn | TRANSMITTER | fn | not used |
| tn | p31 | fn | DISPLAY |
| sfrq | 121.474 | sp | 0 |
| tof | 10608.2 | wp | 10010.0 |
| tpwr | 55 | rfl | 2437.3 |
| pw | 7.117 | rff | 0 |
| DECOUPLER | H1 | rp | -110.1 |
| dn | H1 | lp | -706.6 |
| dof | 0 | lp | PLOT |
| dnn | ymn | wc | 250 |
| dmm | w | sc | 0 |
| dpwr | 35 | vs | 35 |
| dmf | 6700 | th | 12 |
| | ai | no | ph |

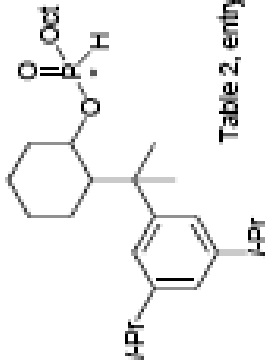
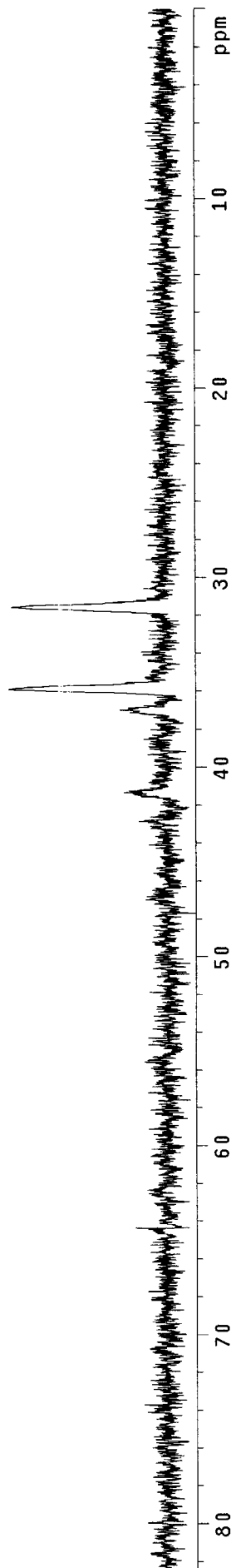


Table 2, entry 2



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 4763.669 | 39.219 | 29.5 |
| 2 | 4099.055 | 33.747 | 126.0 |

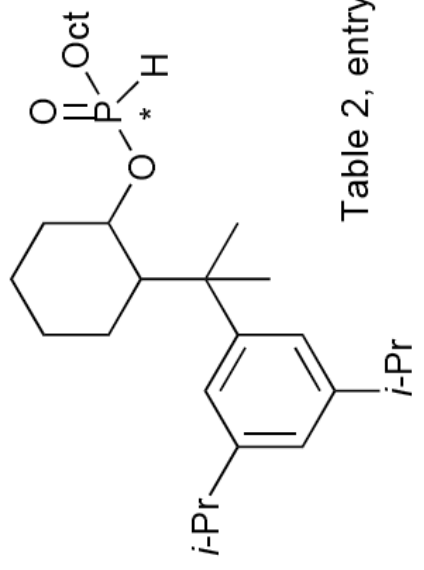
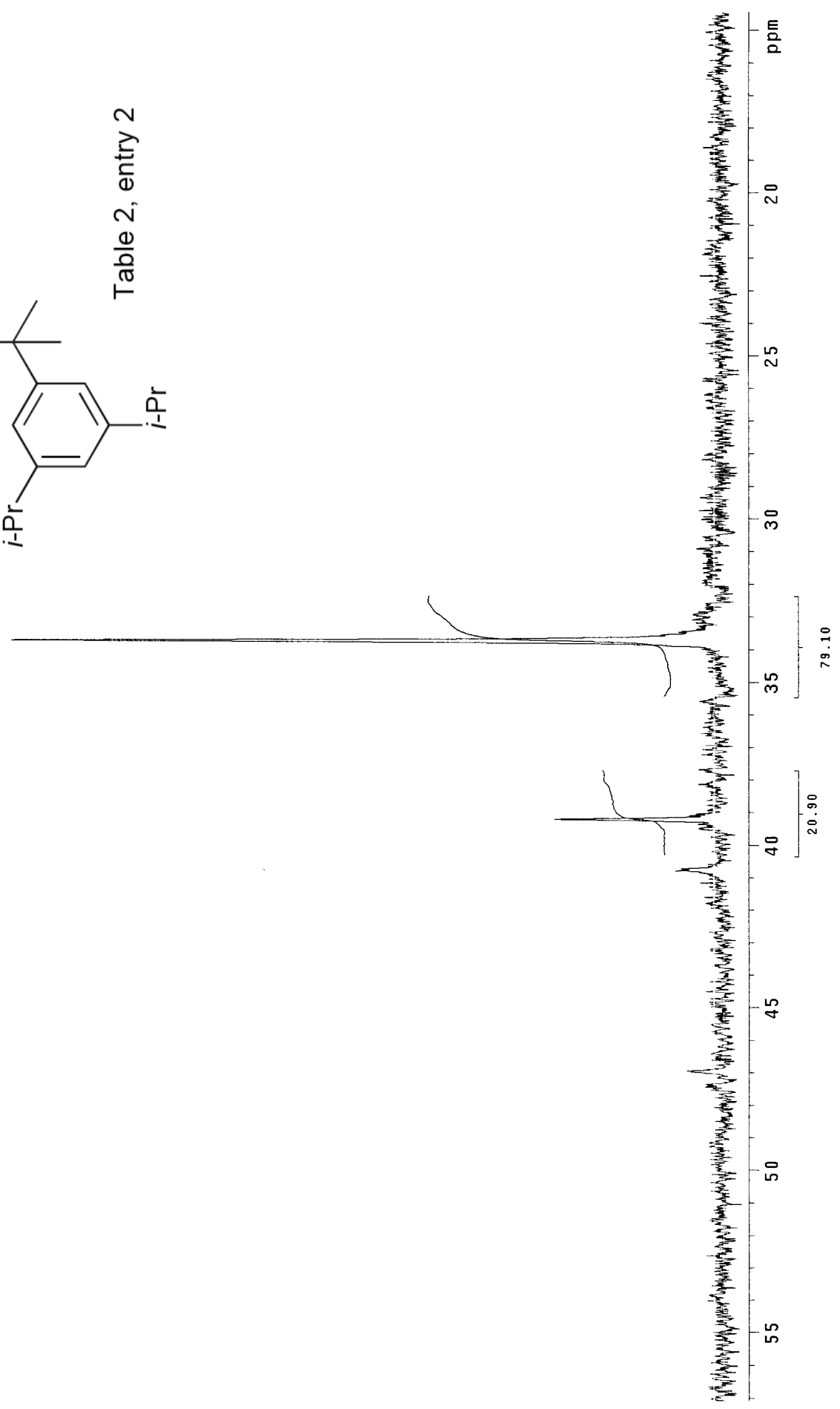


Table 2, entry 2



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 5019.886 | 41.329 | 34.7 |
| 2 | 4494.804 | 37.006 | 38.4 |
| 3 | 4361.392 | 35.907 | 134.4 |
| 4 | 3837.126 | 31.591 | 131.7 |

100% CDCl₃ (100 MHz)
 100% CDCl₃ (100 MHz)
 100% CDCl₃ (100 MHz)

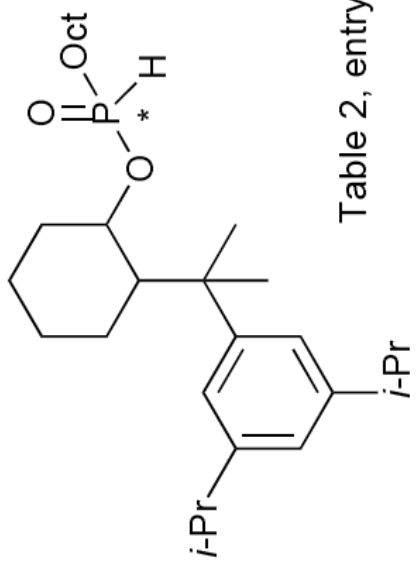
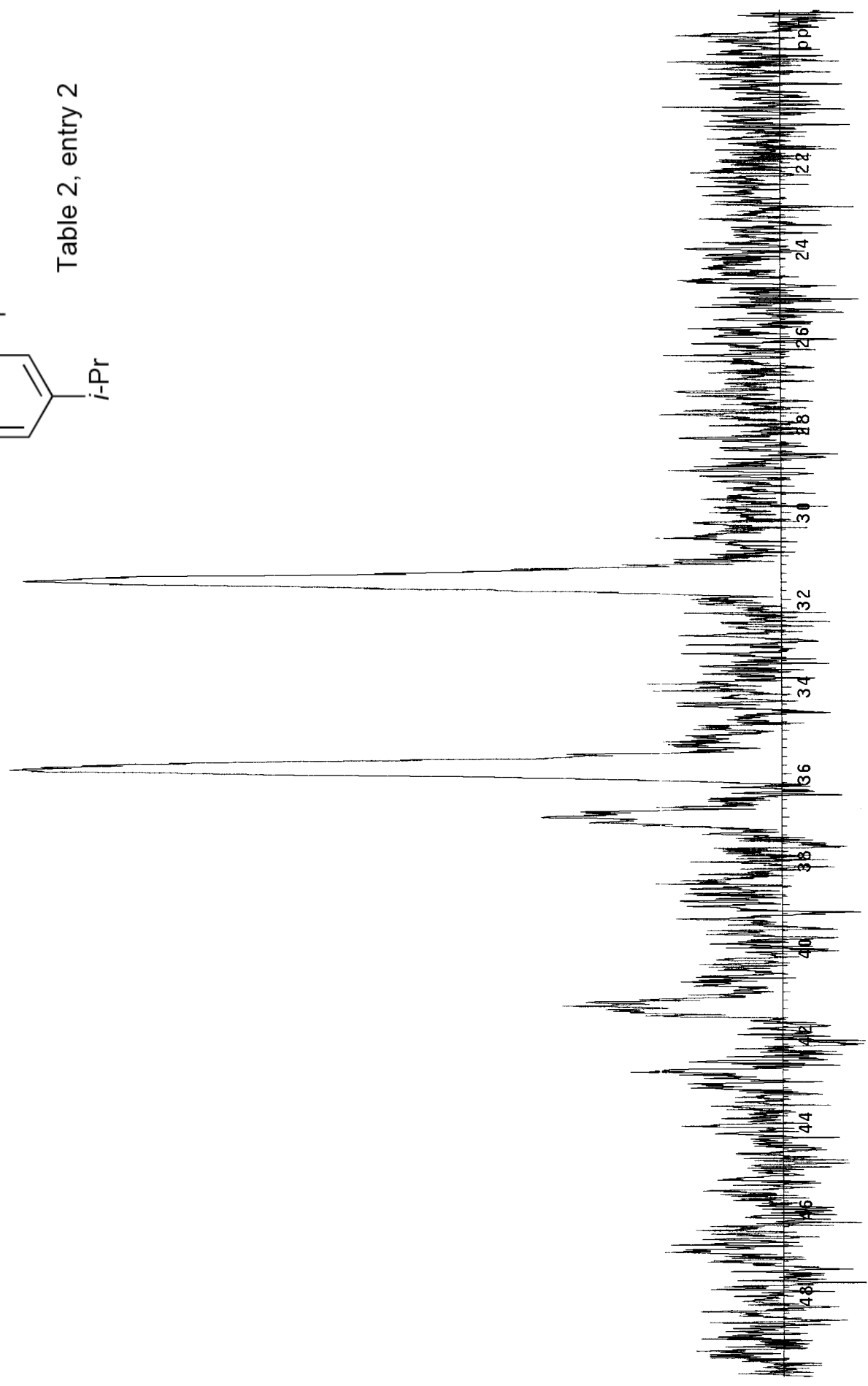


Table 2, entry 2



754

exp1 s2pu1

| | | | |
|-------------|------------|------|----------|
| date | Nov 9 2005 | temp | not used |
| solvent | CDC13 | gain | not used |
| file | exp | spin | 20 |
| ACQUISITION | | hst | 0.008 |
| sw | 4803.1 | pw90 | 17.200 |
| at | 1.994 | alfa | 20.000 |
| np | 19158 | il | |
| fb | not used | in | n |
| bs | 16 | in | n |
| ss | 4 | dp | y |
| d1 | 1.000 | hs | nn |
| nt | 8 | fn | not used |
| ct | 8 | fn | not used |
| TRANSMITTER | H1 | SP | -60.0 |
| tn | 300.053 | wp | 3060.5 |
| sfrq | 300.9 | rfl | 601.0 |
| tof | 55 | rfp | 0 |
| tpwr | 8.600 | fp | -111.2 |
| pw | | lp | -83.0 |
| DECOUPLER | C13 | PLOT | |
| dn | 0 | wc | 250 |
| dof | 0 | sc | 0 |
| dm | nnn | vs | 204 |
| dmm | C | th | 3 |
| dpwr | 45 | ai | cdc |
| dmf | 13100 | ph | |

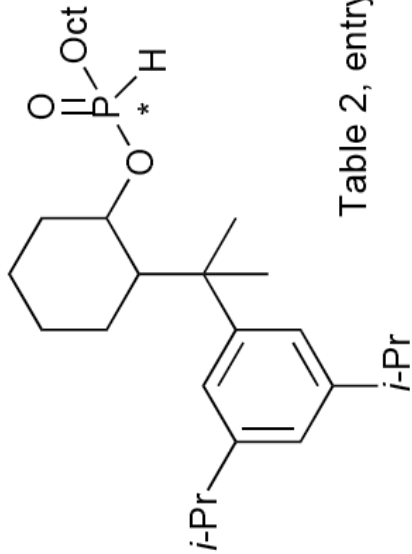
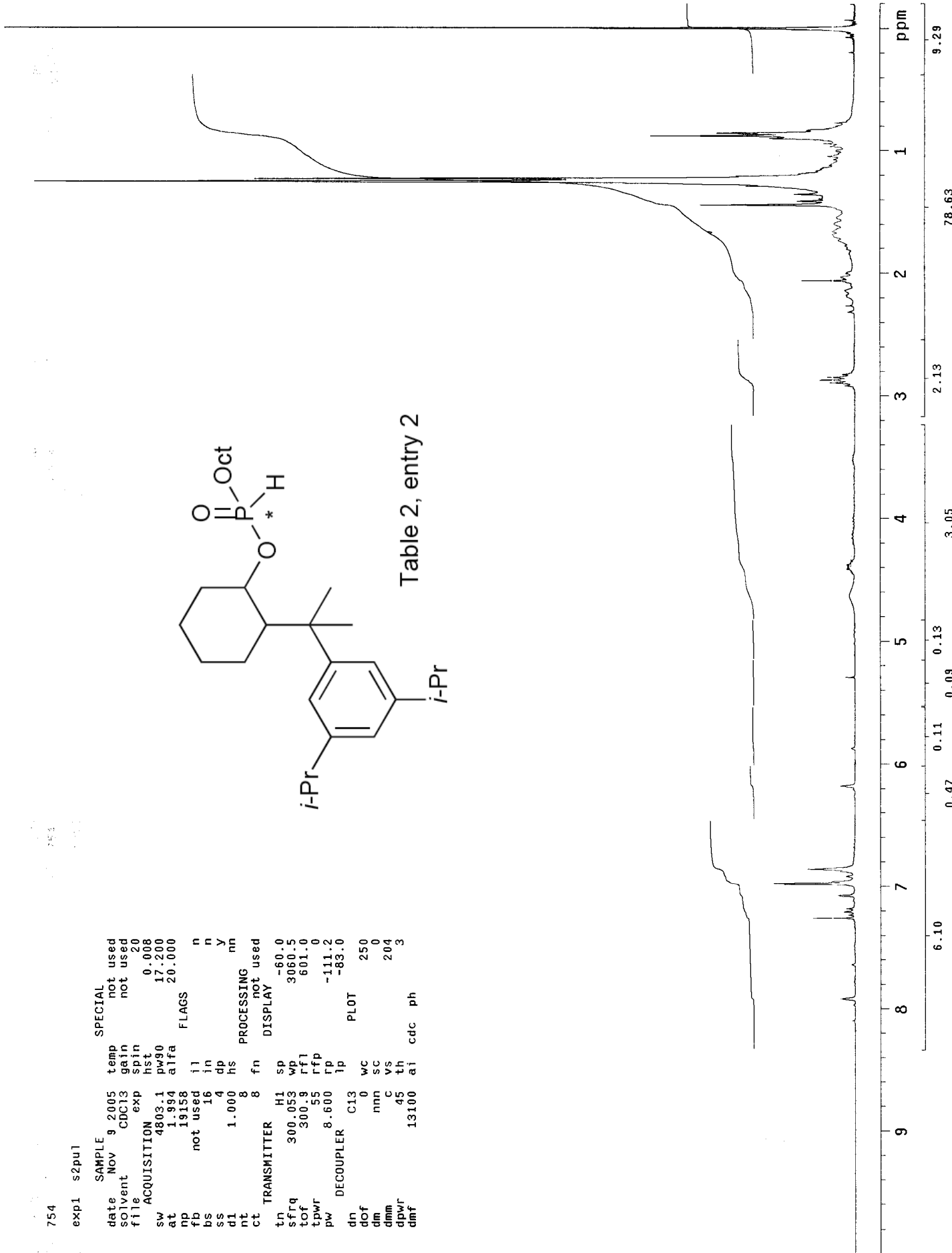


Table 2, entry 2



| INDEX | FREQUENCY PPM | HEIGHT |
|-------|---------------|--------|
| 1 | 2580.112 | 24.7 |
| 2 | 1872.659 | 6.2 |
| 3 | 1732.311 | 6.8 |
| 4 | 1662.544 | 126.0 |
| 5 | 1592.778 | 7.8 |
| 6 | 1452.430 | 5.7 |
| 7 | 1412.039 | 24.4 |

| INDEX | FREQUENCY PPM | HEIGHT |
|-------|---------------|--------|
| 1 | 24.7 | 24.7 |
| 2 | 6.2 | 6.2 |
| 3 | 6.8 | 6.8 |
| 4 | 126.0 | 126.0 |
| 5 | 7.8 | 7.8 |
| 6 | 5.7 | 5.7 |
| 7 | 24.4 | 24.4 |

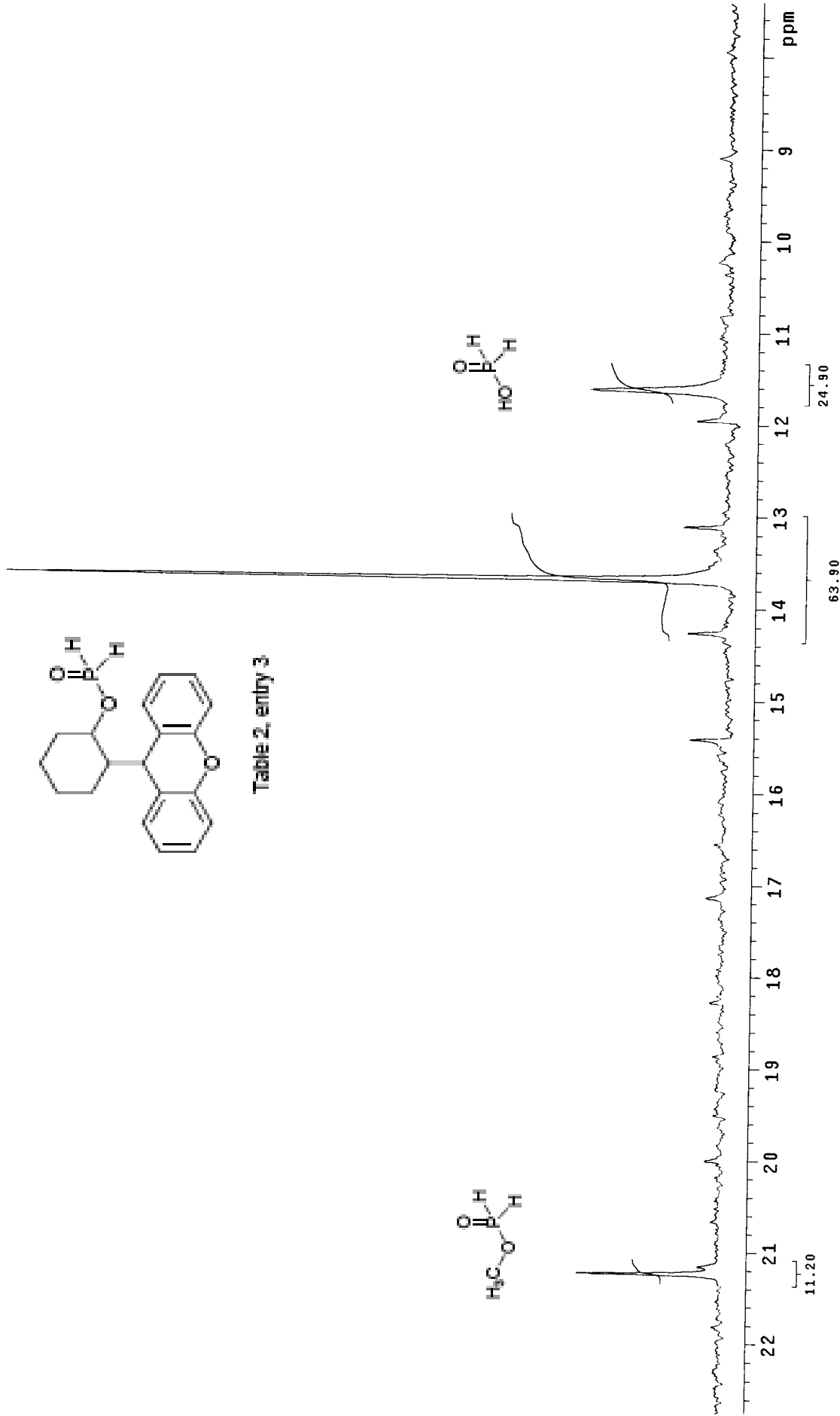


Table 2, entry 3

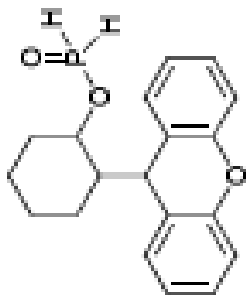
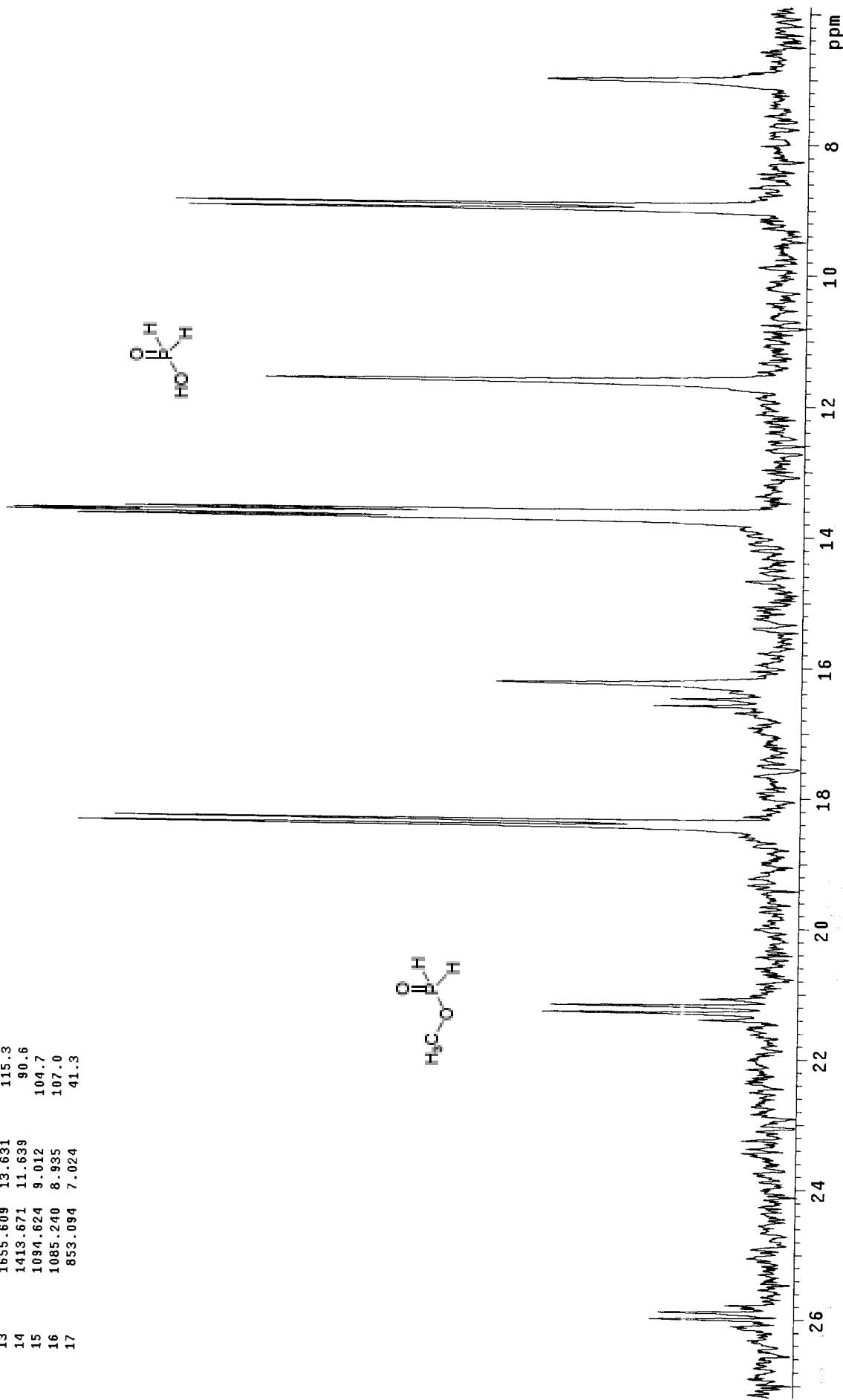
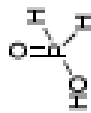


Table 2, entry 3

| INDEX | FREQUENCY .PPM | HEIGHT | INDEX | FREQUENCY .PPM |
|-------|----------------|--------|-------|----------------|
| 1 | 3158.232 | 26.002 | 1 | 159.239 |
| 2 | 3145.585 | 25.898 | 2 | 159.239 |
| 3 | 2587.048 | 21.299 | 3 | 159.239 |
| 4 | 2573.992 | 21.192 | 4 | 159.239 |
| 5 | 2241.073 | 18.451 | 5 | 159.239 |
| 6 | 2231.689 | 18.373 | 6 | 159.239 |
| 7 | 2015.455 | 16.593 | 7 | 159.239 |
| 8 | 2002.807 | 16.489 | 8 | 159.239 |
| 9 | 1974.656 | 16.257 | 9 | 159.239 |
| 10 | 1670.704 | 13.755 | 10 | 159.239 |
| 11 | 1664.992 | 13.708 | 11 | 159.239 |
| 12 | 1661.321 | 13.678 | 12 | 159.239 |
| 13 | 1655.609 | 13.631 | 13 | 159.239 |
| 14 | 1413.671 | 11.639 | 14 | 159.239 |
| 15 | 1094.624 | 9.012 | 15 | 159.239 |
| 16 | 1085.240 | 8.935 | 16 | 159.239 |
| 17 | 853.094 | 7.024 | 17 | 159.239 |



| INDEX | FREQUENCY PPM | HEIGHT |
|-------|---------------|--------|
| 1 | 5542.112 | 45.628 |
| 2 | 4915.441 | 40.469 |
| 3 | 4811.404 | 39.612 |
| 4 | 4465.429 | 36.764 |
| 5 | 1640.921 | 13.510 |
| 6 | 1414.078 | 11.642 |
| 7 | 773.536 | 6.369 |
| 8 | 748.649 | 6.164 |

| INDEX | FREQUENCY PPM | HEIGHT |
|-------|---------------|--------|
| 1 | 1092.11 | 71.1 |
| 2 | | 53.5 |
| 3 | | 58.4 |
| 4 | | 137.2 |
| 5 | | 23.2 |
| 6 | | 35.9 |
| 7 | | 19.4 |
| 8 | | 26.9 |

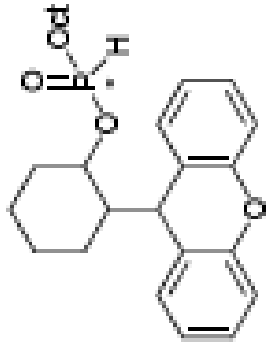
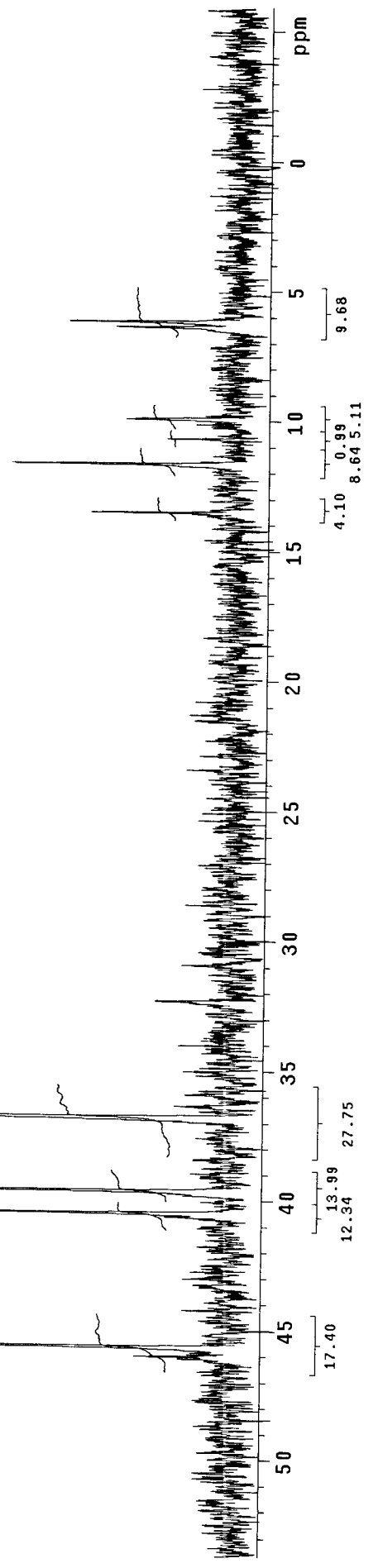
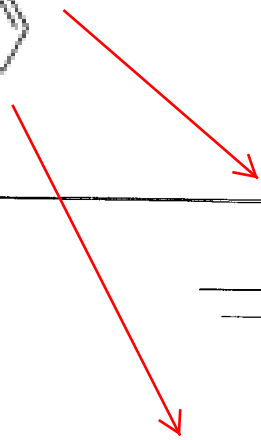


Table 2, entry 3



| INDEX | FREQUENCY PPM | HEIGHT |
|-------|---------------|--------|
| 1 | 2394.477 | 18.6 |
| 2 | 1669.480 | 72.8 |
| 3 | 1107.679 | 21.4 |

| INDEX | FREQUENCY PPM | HEIGHT |
|-------|---------------|--------|
| 1 | 19.714 | 18.6 |
| 2 | 13.745 | 72.8 |
| 3 | 9.119 | 21.4 |

| INDEX | FREQUENCY PPM | HEIGHT |
|-------|---------------|--------|
| 1 | 19.714 | 18.6 |
| 2 | 13.745 | 72.8 |
| 3 | 9.119 | 21.4 |

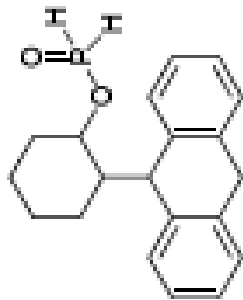
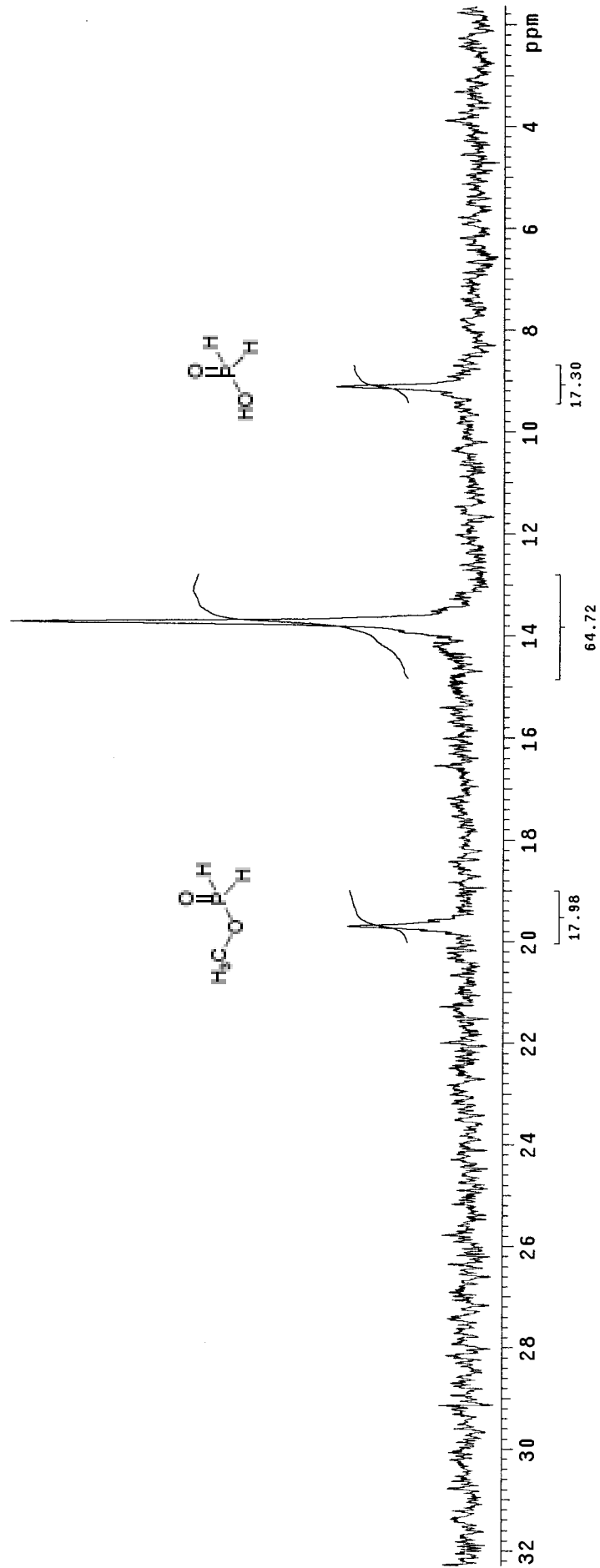


Table 2, entry 4



| INDEX | FREQUENCY | PPM | HEIGHT | INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|-------|-----------|-----|--------|
| 1 | 2241.481 | 18.454 | 69.2 | 1 | 69.2 | | 69.2 |
| 2 | 1845.281 | 16.015 | 15.7 | 2 | 15.7 | | 15.7 |
| 3 | 1822.476 | 15.004 | 22.4 | 3 | 22.4 | | 22.4 |
| 4 | 1668.664 | 13.738 | 147.6 | 4 | 13.738 | | 147.6 |
| 5 | 1113.391 | 9.167 | 80.3 | 5 | 9.167 | | 80.3 |

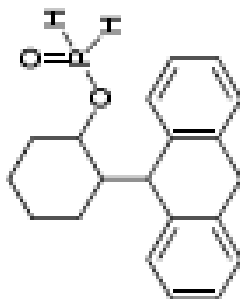
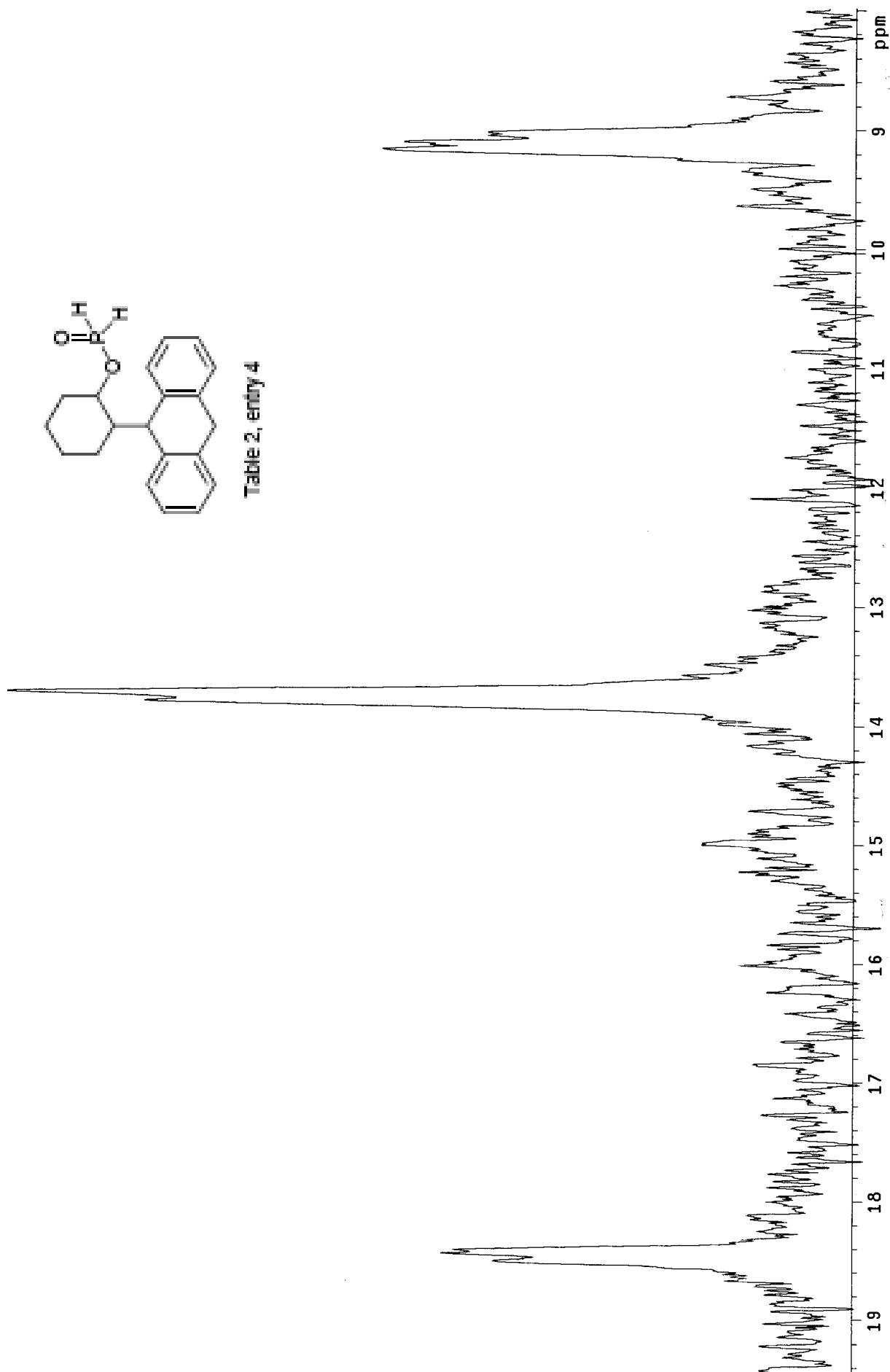


Table 2, entry 4



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 2469.955 | 20.335 | 23.8 |
| 2 | 1966.904 | 16.193 | 126.0 |
| 3 | 593.205 | 4.884 | 19.1 |

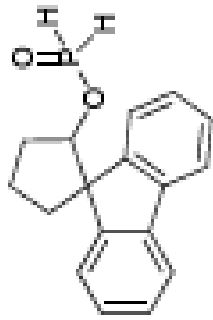
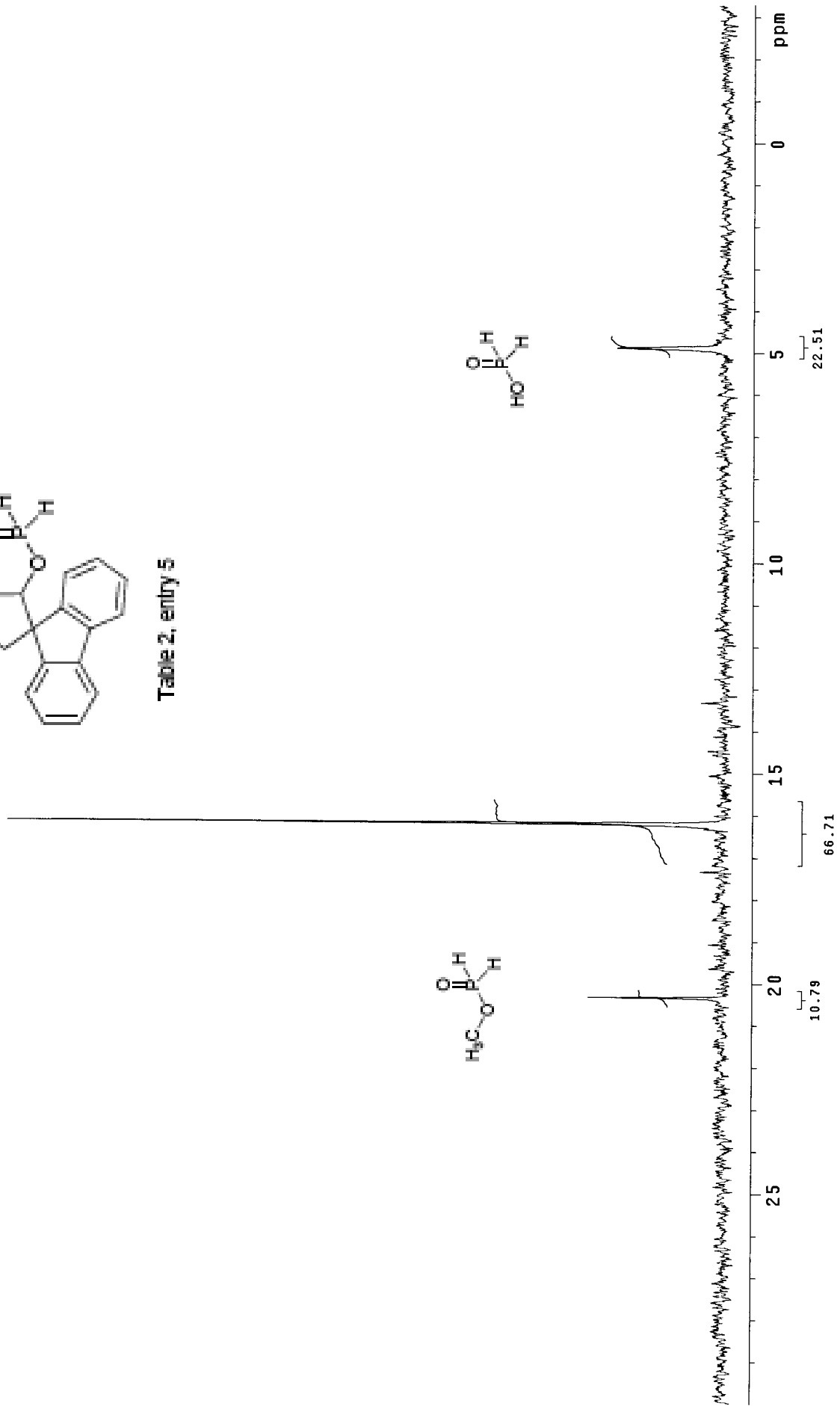


Table 2, entry 5



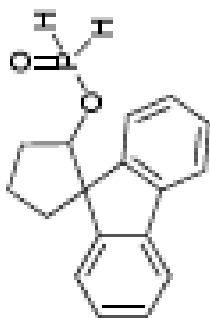
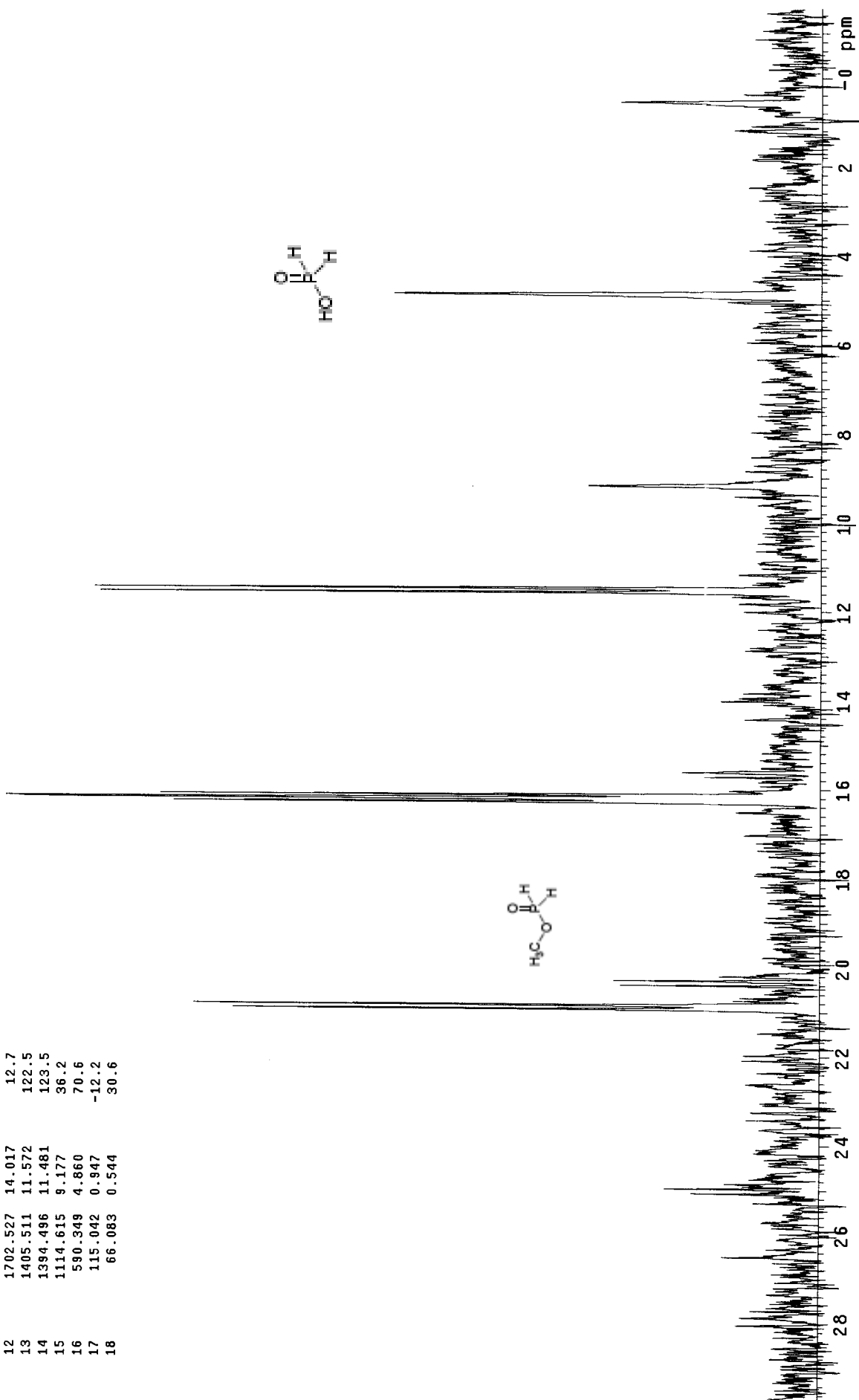
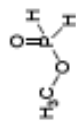
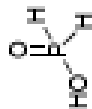


Table 2, entry 5

| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 3219.431 | 26.505 | 12.2 |
| 2 | 3046.035 | 25.078 | 17.8 |
| 3 | 3032.980 | 24.970 | 22.5 |
| 4 | 2543.801 | 20.943 | 98.9 |
| 5 | 2532.377 | 20.849 | 105.8 |
| 6 | 2478.115 | 20.402 | 30.4 |
| 7 | 2465.059 | 20.295 | 31.5 |
| 8 | 1978.736 | 16.291 | 109.5 |
| 9 | 1967.720 | 16.200 | 139.4 |
| 10 | 1959.153 | 16.130 | 111.7 |
| 11 | 1897.138 | 15.619 | 19.6 |
| 12 | 1702.527 | 14.017 | 12.7 |
| 13 | 1405.511 | 11.572 | 122.5 |
| 14 | 1394.496 | 11.481 | 123.5 |
| 15 | 1114.615 | 9.177 | 36.2 |
| 16 | 590.349 | 4.860 | 70.6 |
| 17 | 115.042 | 0.947 | -12.2 |
| 18 | 66.083 | 0.544 | 30.6 |



Workup.

28b

exp1 s2pu1

| SAMPLE | | SPECIAL | |
|-------------|-------------|---------|----------|
| date | Jul 21 2006 | temp | not used |
| solvent | CDCl3 | gain | not used |
| file | | spin | 20 |
| ACQUISITION | | | |
| sw | 26738.0 | hst | 0.008 |
| at | 1.598 | pw90 | 18.300 |
| np | 85478 | alfa | 20.000 |
| fb | 14800 | ll | |
| bs | 64 | in | n |
| ss | 4 | dp | n |
| d1 | 1.000 | hs | y |
| nt | 16 | nm | nm |
| ct | 16 | lb | 2.00 |
| TRANSMITTER | | | |
| tn | P31 | fn | not used |
| sfrq | 121.474 | sp | DISPLAY |
| tof | 10608.2 | wp | 10010.0 |
| tpwr | 55 | rfl | 2437.3 |
| pw | 7.117 | rff | 0 |
| DECOUPLER | | | |
| dn | H1 | rp | 135.3 |
| dof | 0 | lp | -2472.4 |
| dm | yyy | wc | 250 |
| dmm | w | sc | 0 |
| dpwr | 35 | vs | 36 |
| dmtf | 6700 | th | 15 |
| | | ai | no |
| | | ph | |

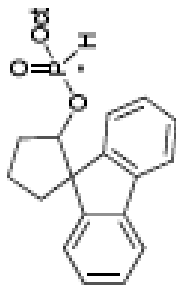
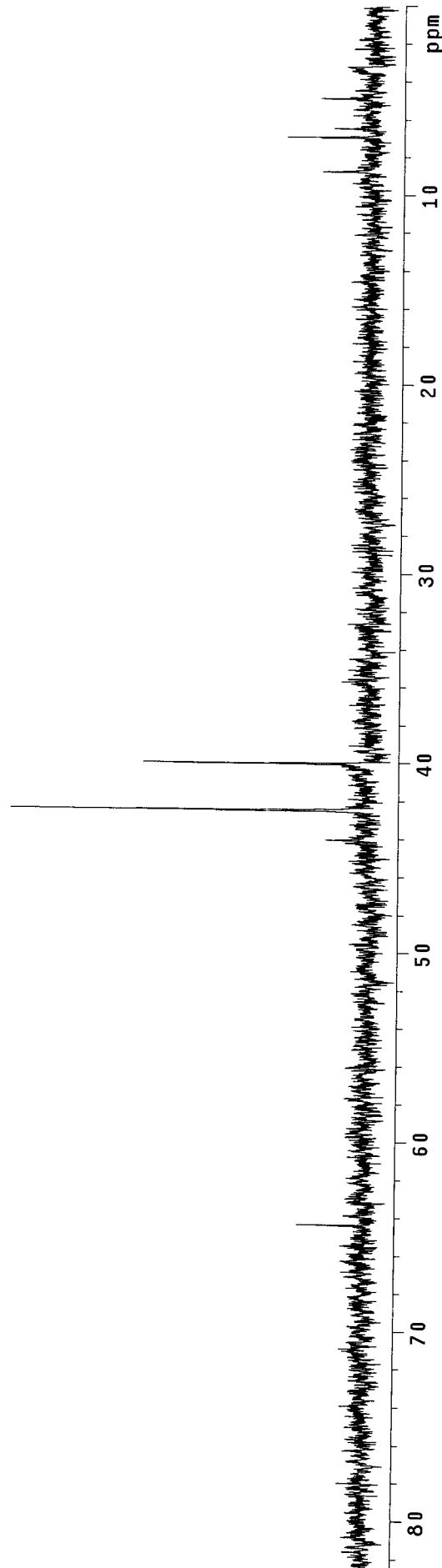


Table 2, entry 5



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 5164.314 | 42.518 | 107.8 |
| 2 | 4863.218 | 40.039 | 68.1 |

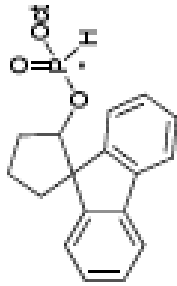
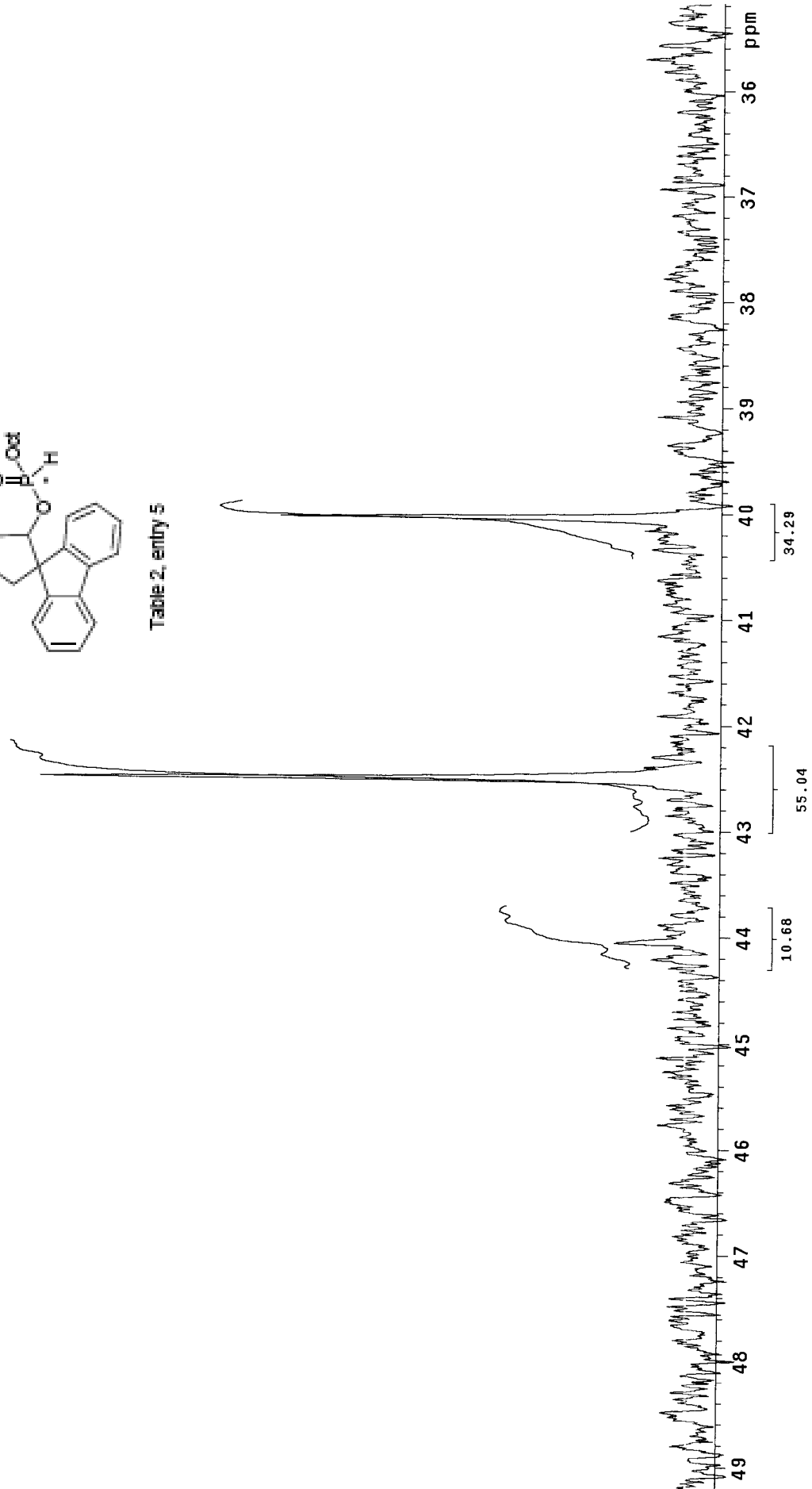


Table 2, entry 5



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 1953.441 | 16.083 | 108.2 |
| 2 | 937.548 | 7.719 | 61.8 |

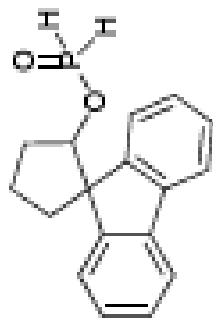
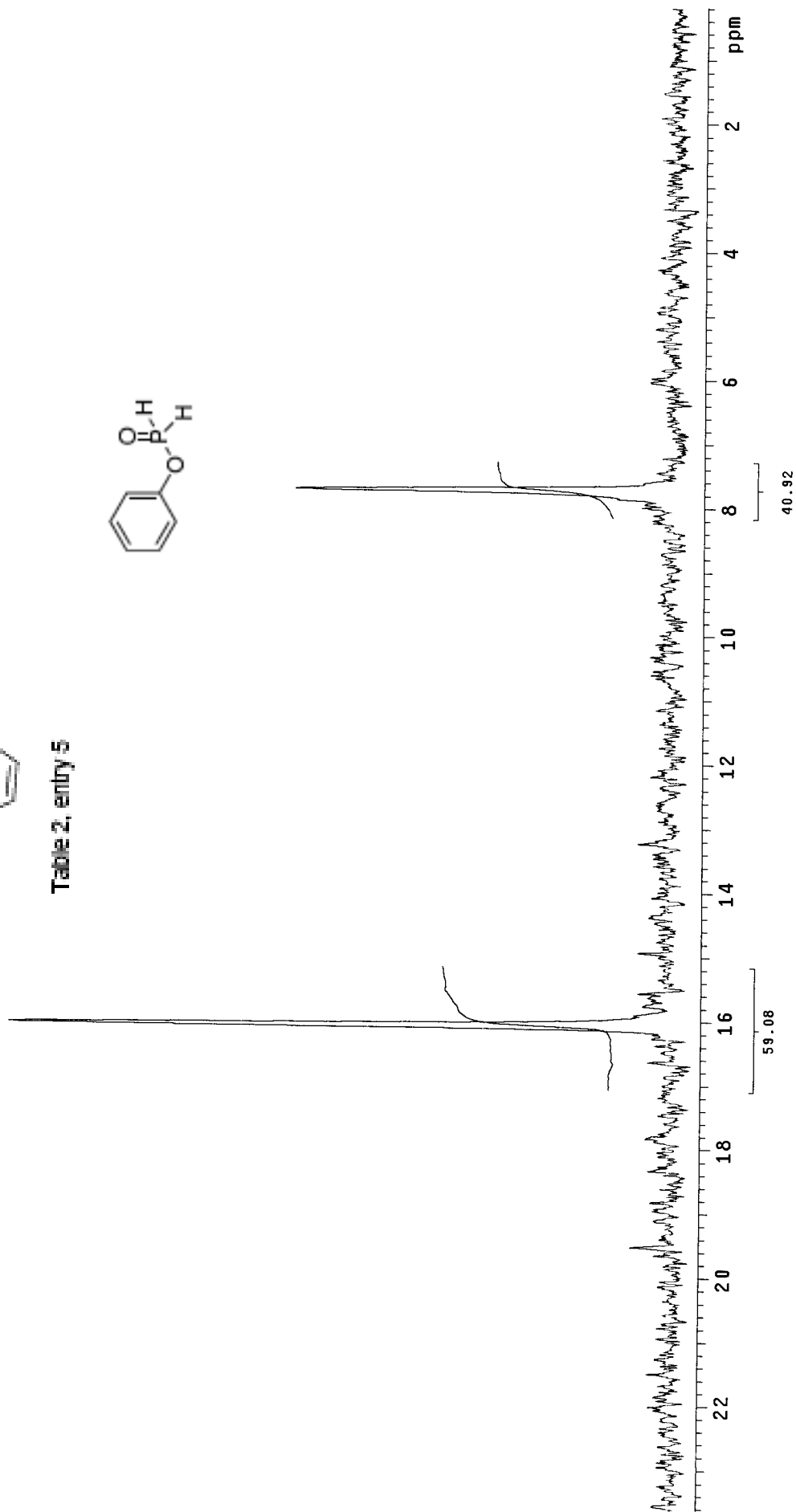
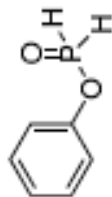


Table 2, entry 5



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 2534.009 | 20.862 | 75.3 |
| 2 | 2522.993 | 20.772 | 71.5 |
| 3 | 1970.168 | 16.320 | 90.4 |
| 4 | 1859.968 | 16.136 | 126.7 |
| 5 | 1950.177 | 16.056 | 65.1 |
| 6 | 1718.847 | 14.151 | -12.0 |
| 7 | 1485.069 | 12.227 | 74.0 |
| 8 | 1395.720 | 11.491 | 63.1 |
| 9 | 1386.336 | 11.414 | 61.5 |
| 10 | 1219.468 | 10.040 | -11.8 |
| 11 | 942.852 | 7.762 | 129.2 |
| 12 | 401.042 | 3.302 | 62.7 |

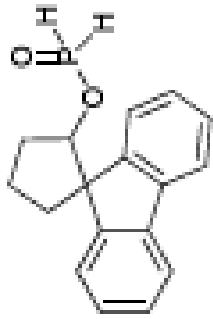
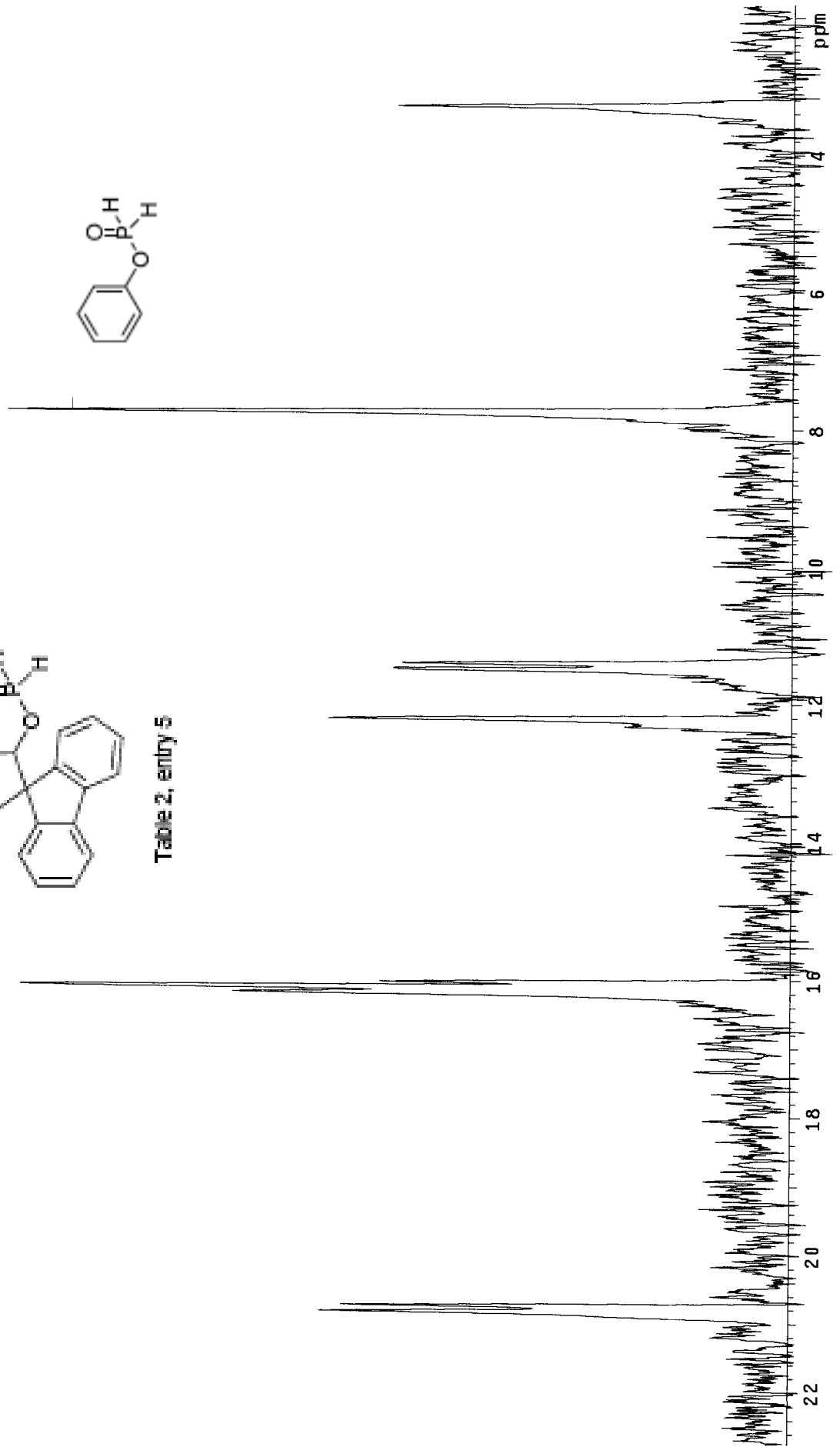
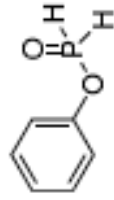


Table 2, entry 5



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 5134.939 | 42.276 | 37.6 |
| 2 | 4819.155 | 39.676 | 39.0 |
| 3 | 4268.778 | 35.145 | 10.4 |

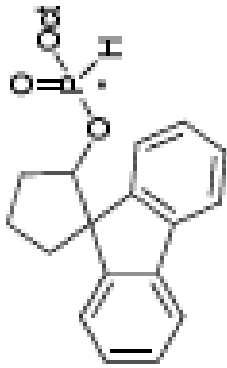
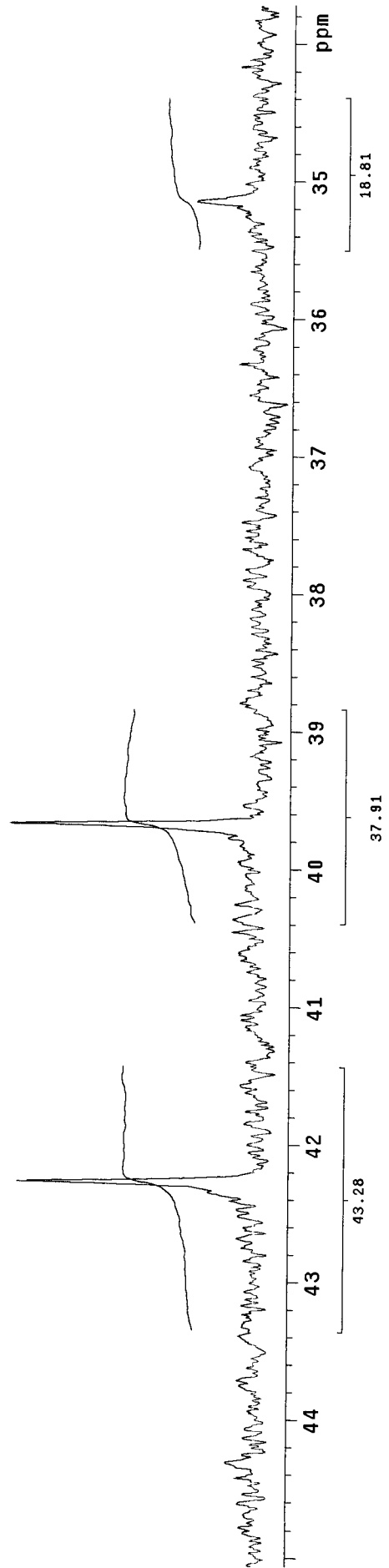


Table 2, entry 5



28a
UNMkep

```
exp1 s2pu1
SAMPLE
date Jul 21 2006
solvent CDC13
file ACQUISITION exp
SW 26738.0
at 1.598
rp 85476
fb 14800
bs 64
ss 4
d1 1.000
nt 16
Ct 16
SPECIAL
temp not used
gain not used
spin 20
hst 0.008
pw90 18.300
alfa 20.000
FLAGS
n
n
n
Y
nn
PROCESSING 2.00
lb
fn
DISPLAY
P31
sfrq 121.474
tof 10608.2
tpwr 55
pw 7.117
DECOUPLER H1 lp
dn
dof
dm
dmm
dpwr 35
dmf 6700
wc 250
sc 0
vs 38
th 10
ai no ph
```

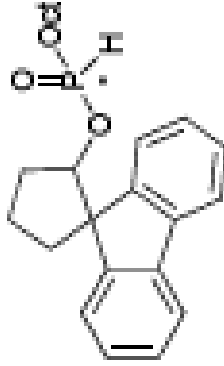
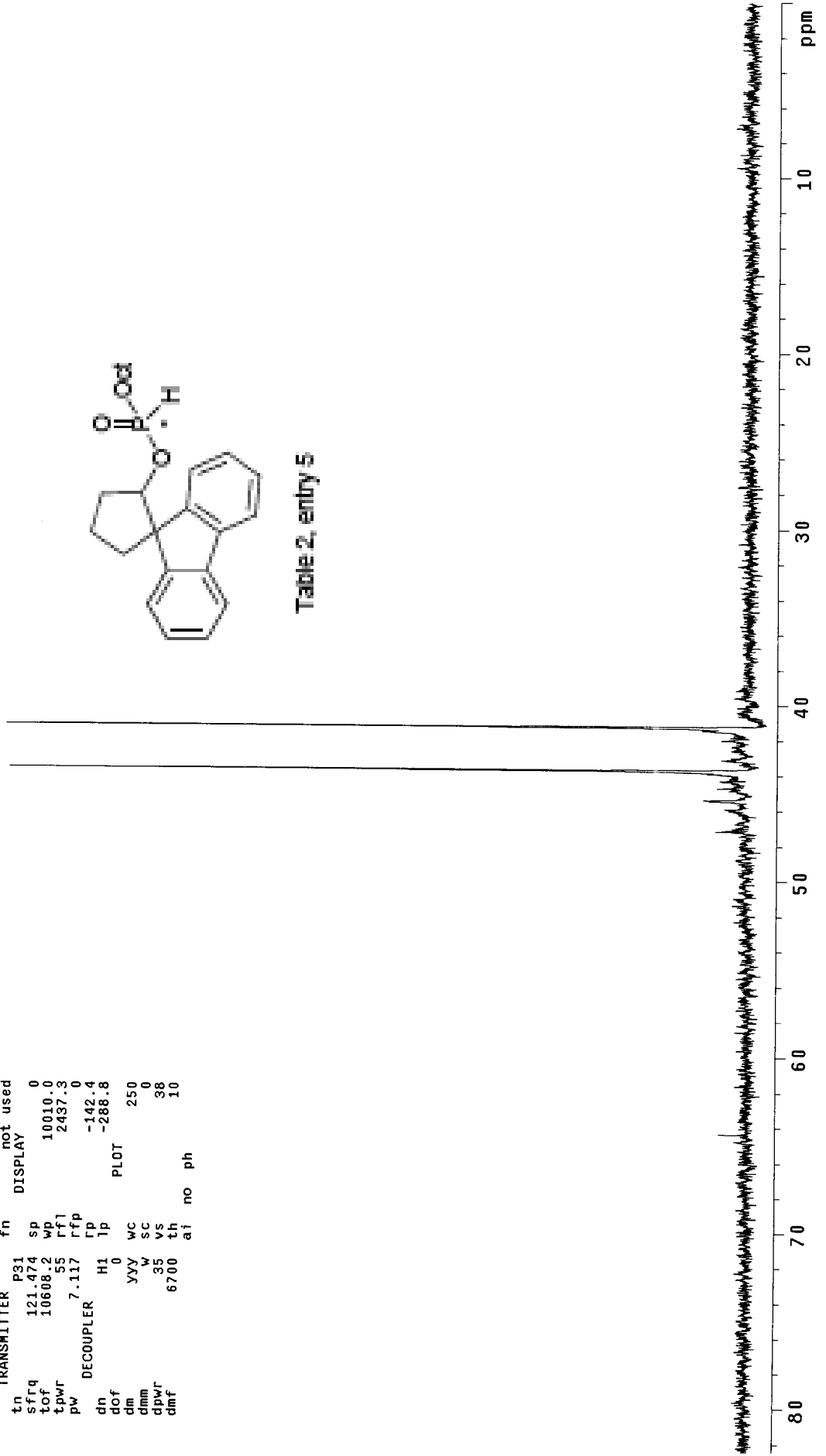


Table 2, entry 5



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 6196.934 | 51.019 | 15.1 |
| 2 | 6066.786 | 49.948 | 14.9 |
| 3 | 5591.479 | 46.035 | 98.3 |
| 4 | 5578.831 | 45.930 | 109.9 |
| 5 | 5285.487 | 43.515 | 111.1 |
| 6 | 5050.485 | 41.581 | 107.1 |
| 7 | 5037.838 | 41.476 | 108.3 |
| 8 | 4734.702 | 38.981 | 109.4 |
| 9 | 4446.254 | 36.606 | 14.2 |

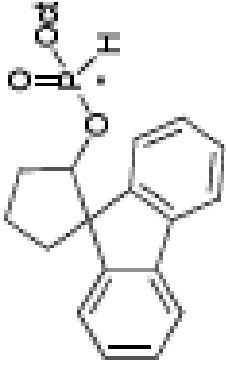
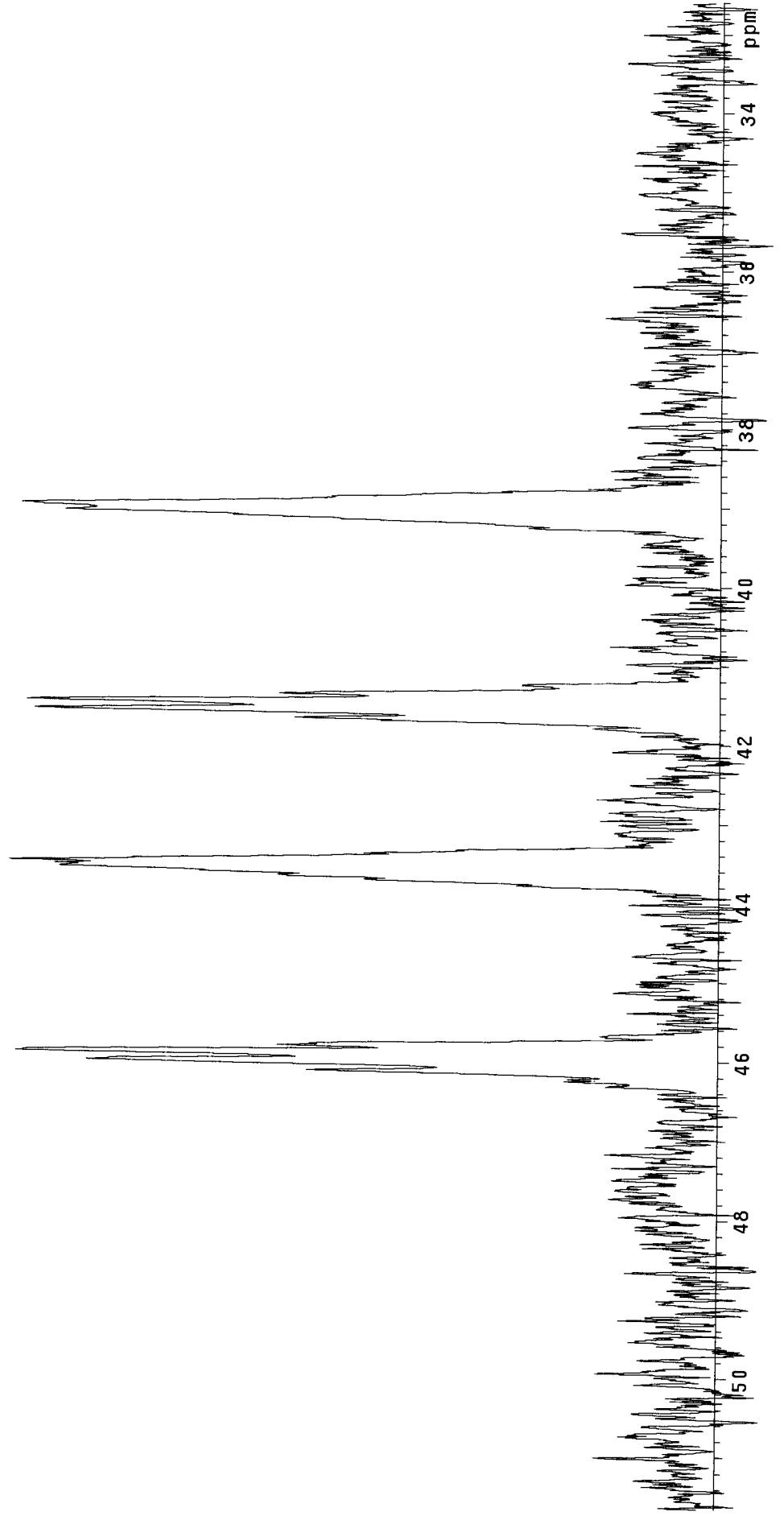


Table 2, entry 5



28A

```

exp1 s2pu1
SAMPLE
date Jul 21 2006
solvent CDC13
file
ACQUISITION
sw 4803.1
np 1.994
bs 19158
fb not used
nt 16
ss 4
d1 1.000
nt 8
Ct TRANSMITTER H1
tn 8
sfrq 300.053
tof 300.9
tpwr 55
pw 8.600
DECOUPLER C13
dn 0
dof 250
dm nnn
dmm C
dpwr 45
dmf 13100 ai cdc ph
SPECIAL
temp not used
gain not used
spin 20
hst 0.008
pw90 17.200
alfa 20.000
FLAGS
n
n
y
nn
PROCESSING
fn not used
DISPLAY
-60.0
3060.5
611.8
-101.1
-68.4
PLOT
250
0
951
3

```

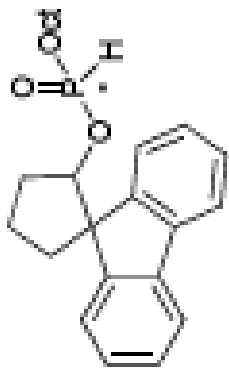
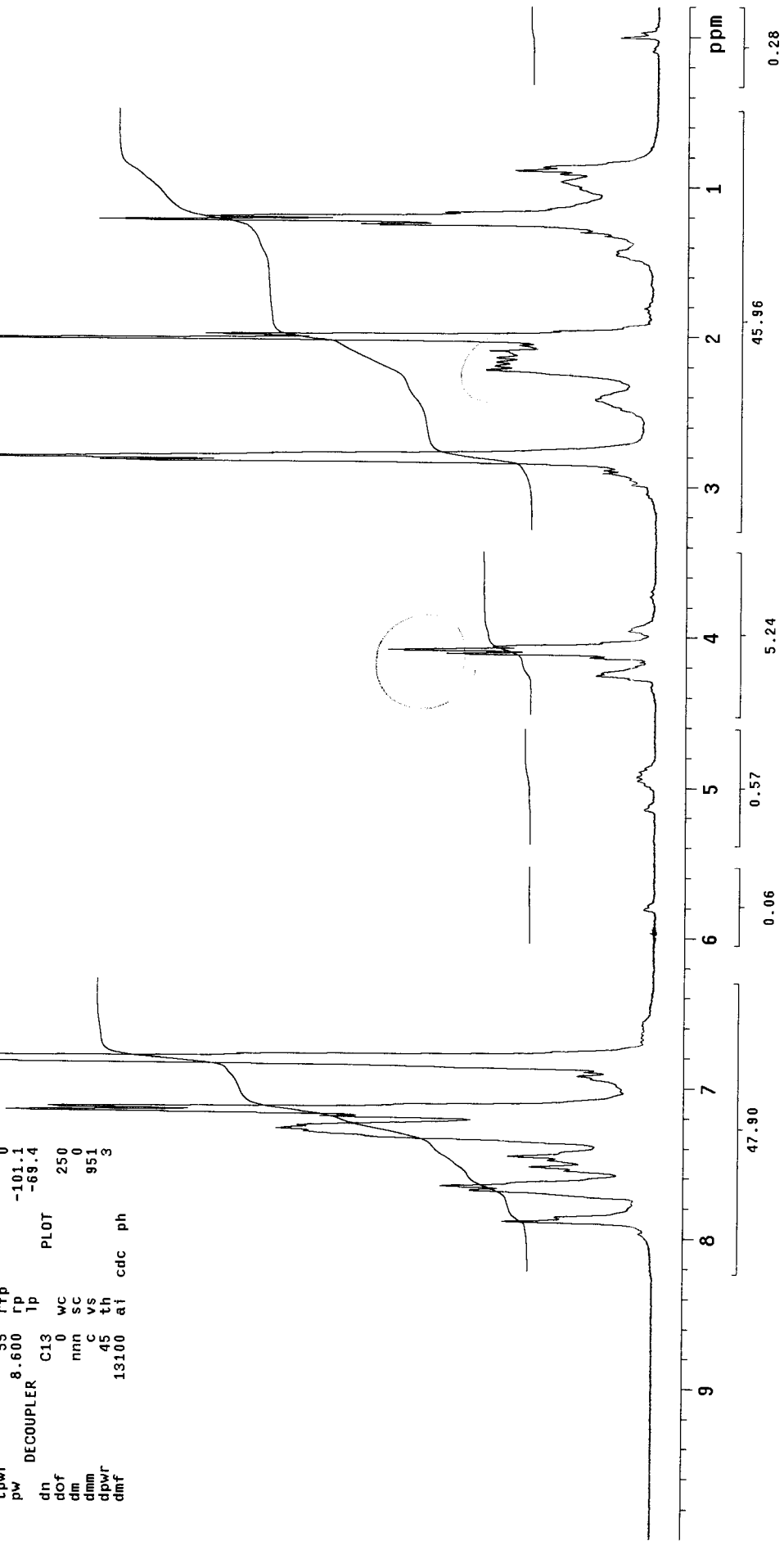


Table 2, entry 5



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 2177.427 | 17.927 | 150.3 |
| 2 | 1579.723 | 13.006 | 58.9 |

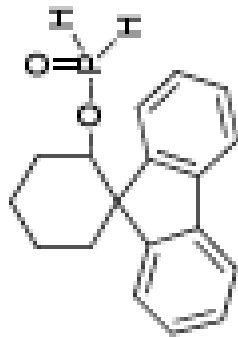
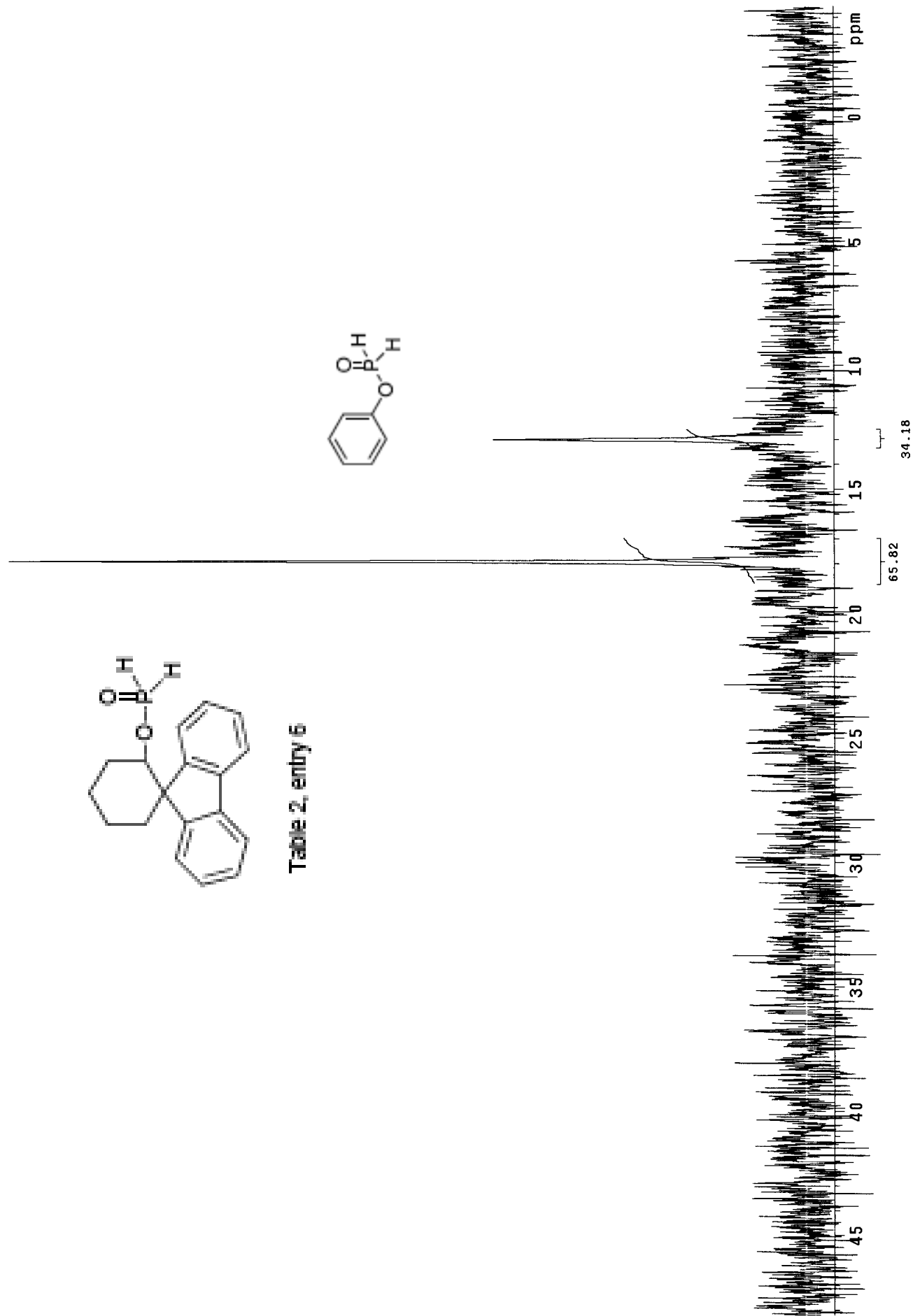
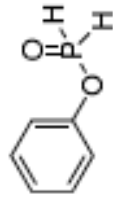


Table 2, entry 6



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 3361.411 | 27.674 | -30.8 |
| 2 | 3135.793 | 25.817 | 32.3 |
| 3 | 2749.835 | 22.639 | 84.1 |
| 4 | 2181.099 | 17.957 | 150.1 |
| 5 | 1610.322 | 13.258 | 105.5 |
| 6 | 1012.618 | 8.337 | 50.5 |

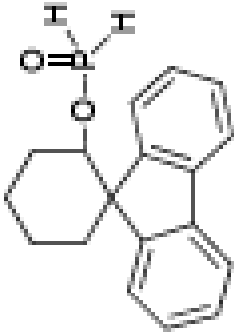
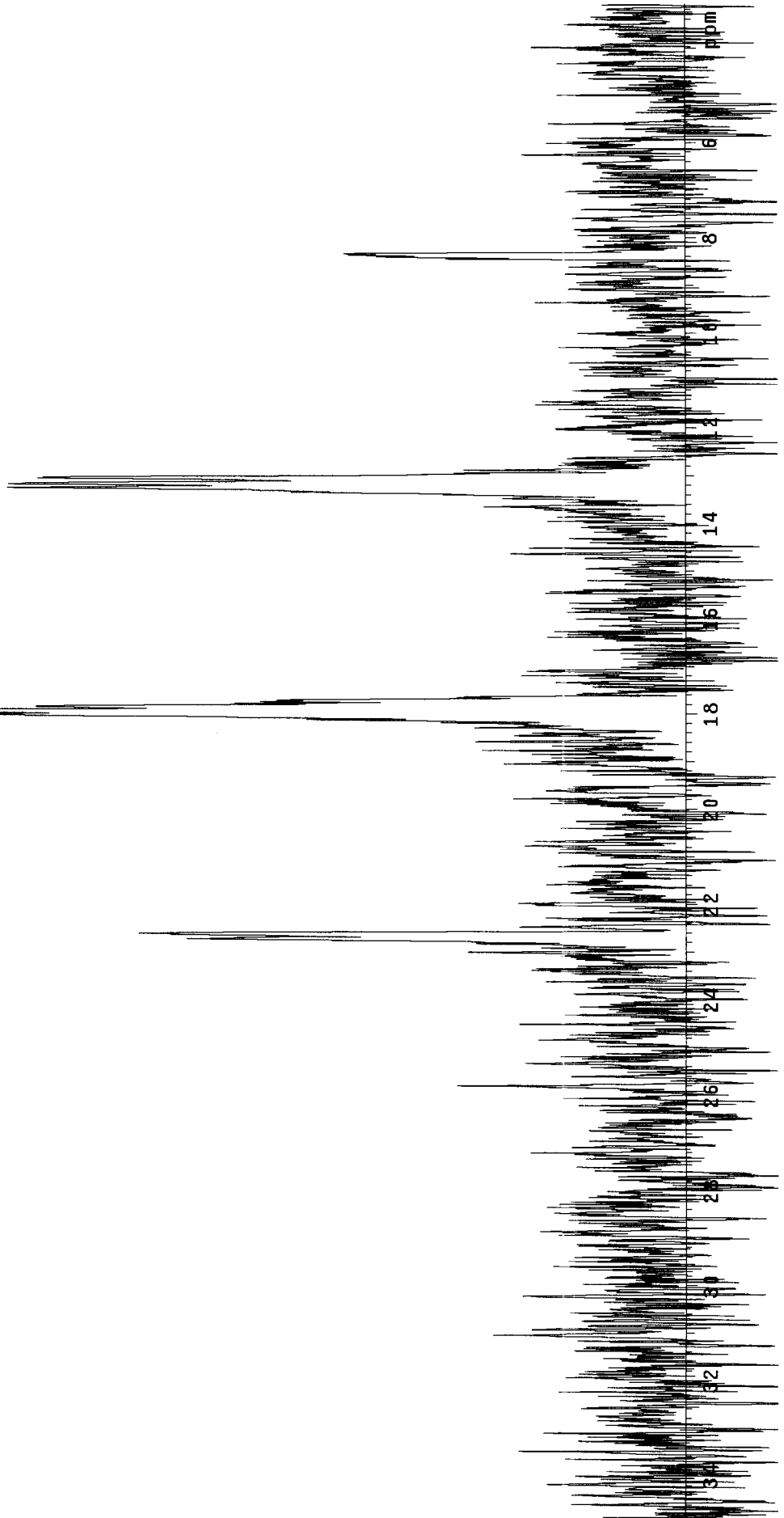


Table 2, entry 6



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 5507.025 | 45.339 | 59.6 |
| 2 | 4902.385 | 40.361 | 74.3 |

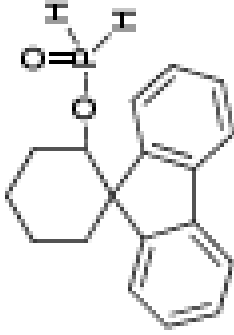
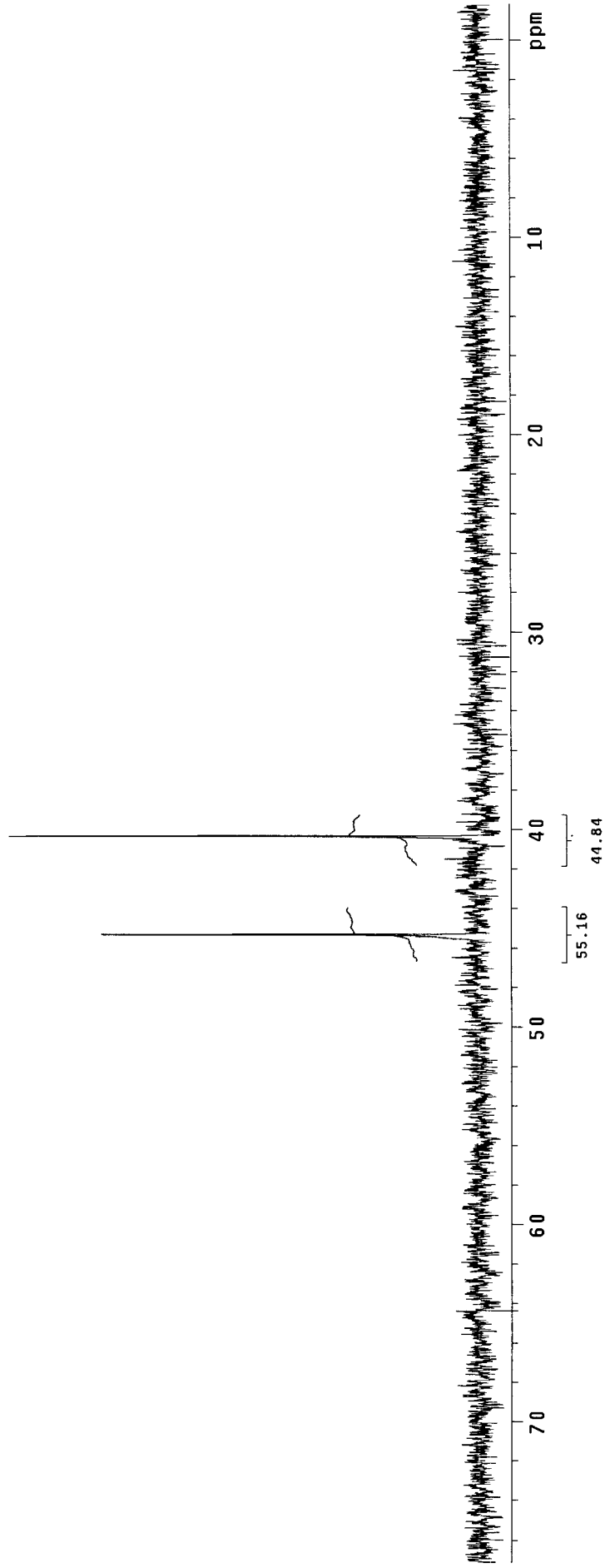


Table 2, entry 6



29a

exp1 s2pu1

```

SAMPLE
date Jul 21 2006
solvent CDCl3
file ACQUISITION exp
sw 4803.1
at 1.994
fb 19158
bs not used
ss 16
d1 4
d1 1.000
nt 8
ct 8
  TRANSMITTER H1
  tn 300.053
  sfrq 300.9
  tof 55
  tpwr 8.600
  pw DECOUPLER C13
  dn 0
  dof 0
  dm nmh
  dmm c
  dpwr 45
  dmf 13100
  ai cdc
  ph

SPECIAL
temp not used
gain not used
spin 20
hst 0.008
pw90 17.200
alfa 20.000
  FLAGS n
  in n
  dp y
  hs nm
  fn not used
  DISPLAY -60.0
  sp 3060.5
  wp 608.6
  rf1 0
  rfp -91.1
  rp -105.6
  PLOT 250
  wc 0
  sc 0
  vs 614
  th 3
  ai cdc
  ph

```

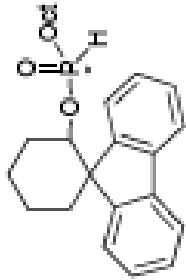
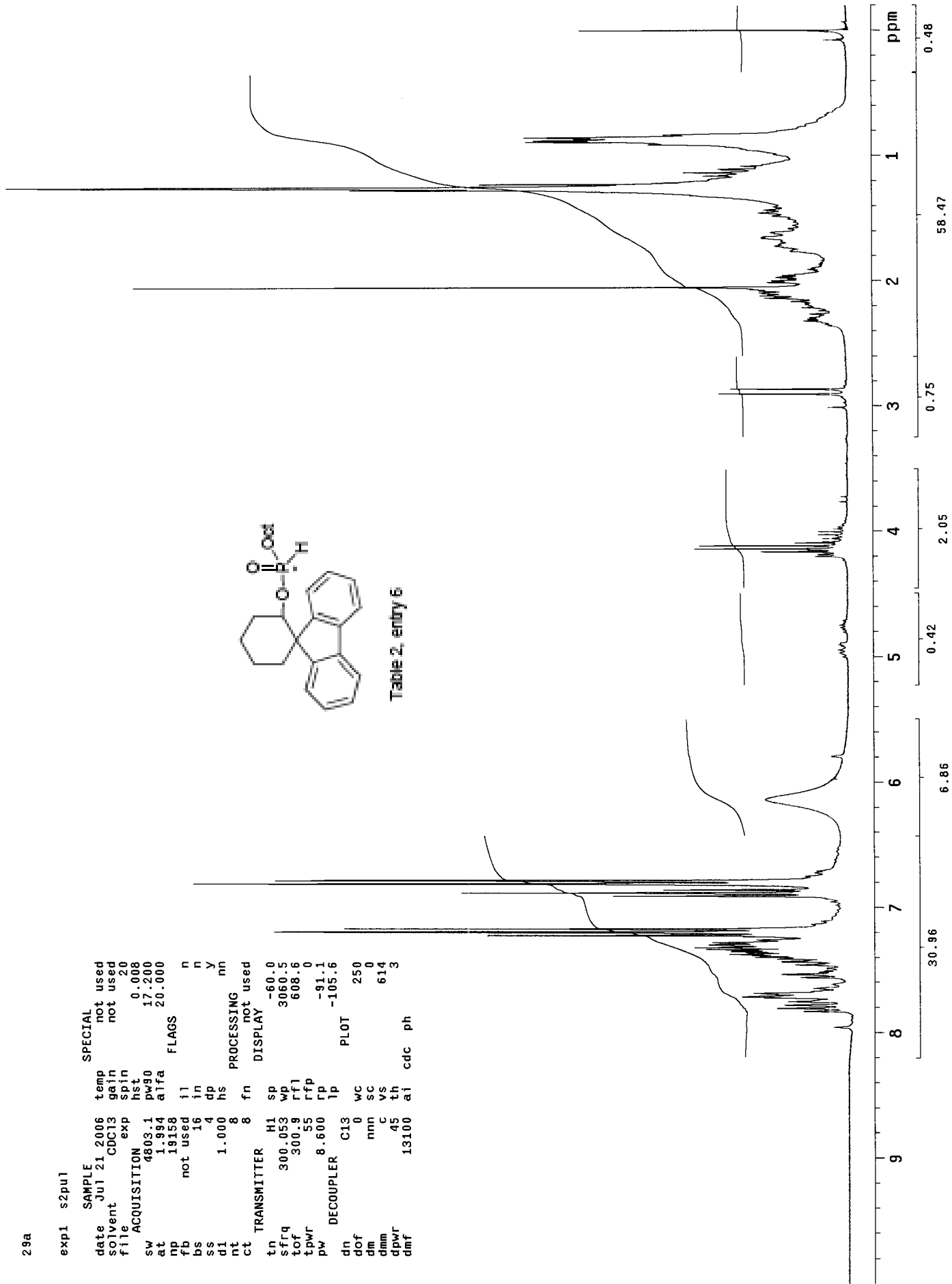


Table 2, entry 6



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 2936.286 | 24.174 | 106.2 |
| 2 | 2349.190 | 19.341 | 122.2 |

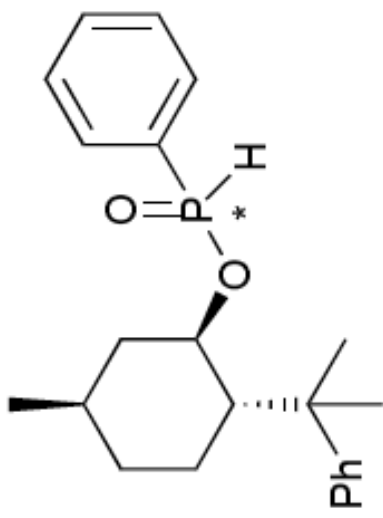
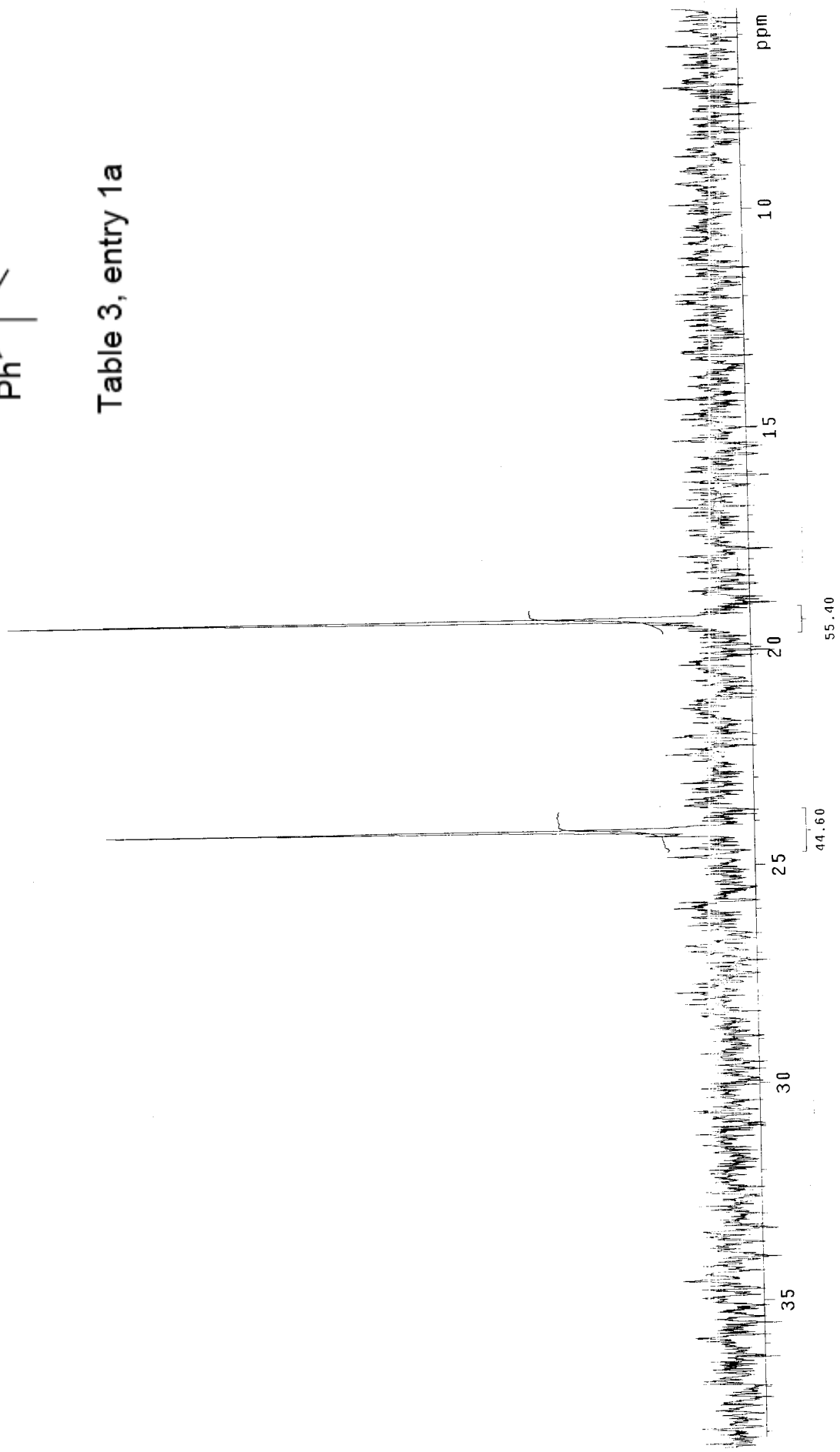


Table 3, entry 1a



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 3213.311 | 26.455 | 49.8 |
| 2 | 2659.262 | 21.894 | 69.9 |
| 3 | 2080.325 | 17.127 | 58.4 |

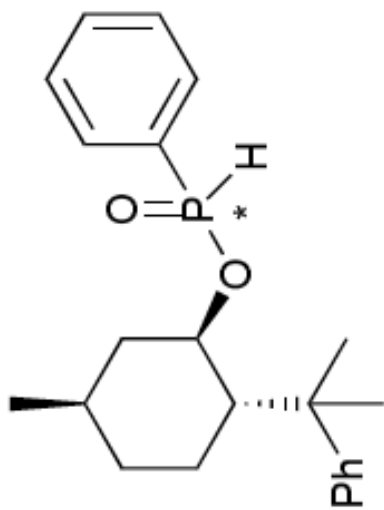
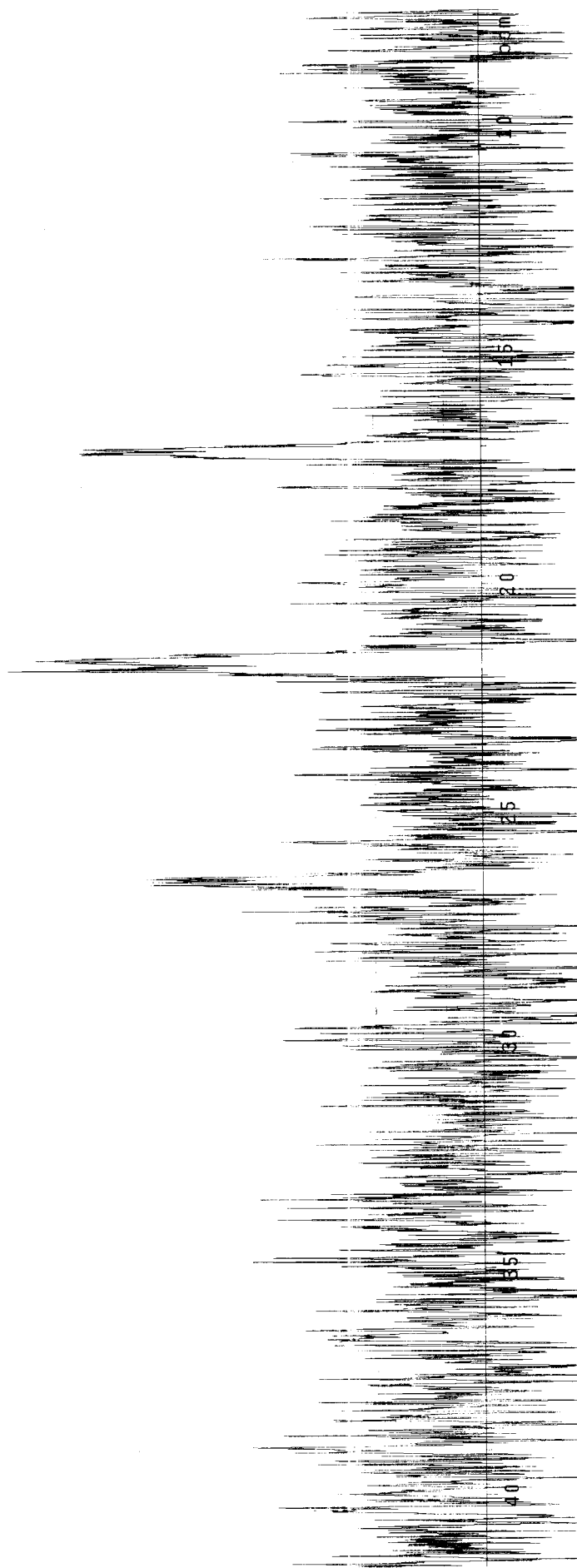
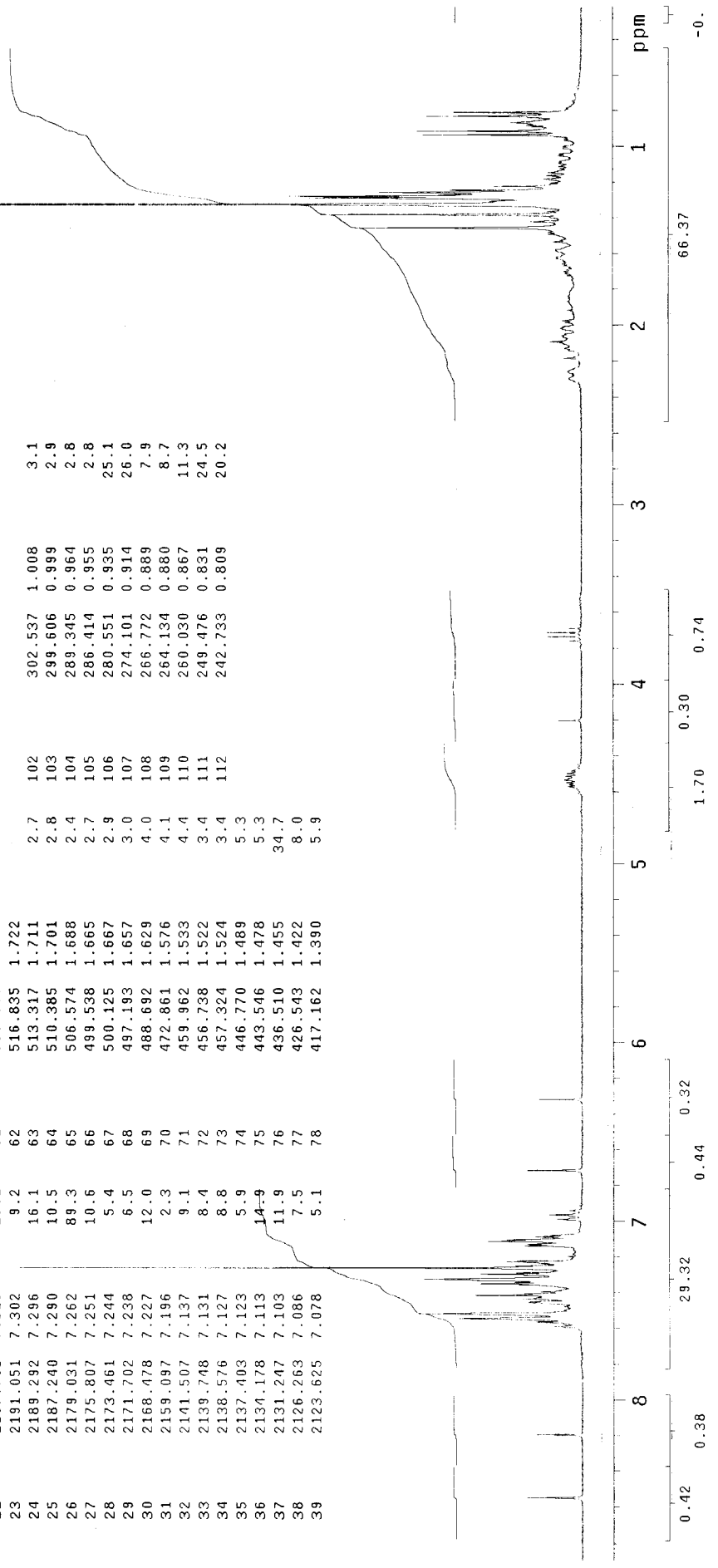
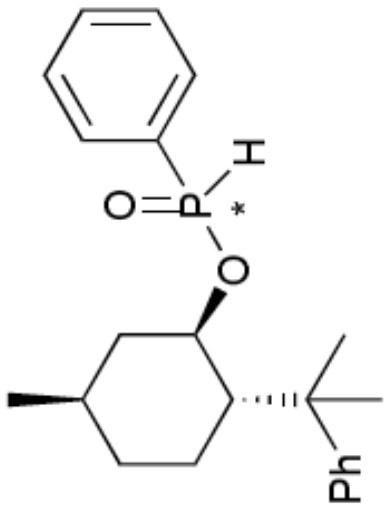


Table 3, entry 1a



| INDEX | FREQUENCY PPM | HEIGHT | INDEX | FREQUENCY PPM | HEIGHT | INDEX | FREQUENCY PPM | HEIGHT |
|-------|---------------|--------|-------|---------------|--------|-------|---------------|--------|
| 1 | 2565.118 | 8.549 | 40 | 2096.361 | 6.987 | 40 | 2096.361 | 6.987 |
| 2 | 2459.289 | 8.196 | 41 | 2089.032 | 6.962 | 41 | 2089.032 | 6.962 |
| 3 | 2277.239 | 7.590 | 42 | 2014.571 | 6.714 | 42 | 2014.571 | 6.714 |
| 4 | 2270.496 | 7.567 | 43 | 1895.256 | 6.316 | 43 | 1895.256 | 6.316 |
| 5 | 2269.030 | 7.562 | 44 | 1371.385 | 4.571 | 44 | 1371.385 | 4.571 |
| 6 | 2264.926 | 7.548 | 45 | 1367.868 | 4.559 | 45 | 1367.868 | 4.559 |
| 7 | 2263.753 | 7.545 | 46 | 1364.643 | 4.548 | 46 | 1364.643 | 4.548 |
| 8 | 2256.131 | 7.519 | 47 | 1359.952 | 4.532 | 47 | 1359.952 | 4.532 |
| 9 | 2250.561 | 7.501 | 48 | 1354.089 | 4.513 | 48 | 1354.089 | 4.513 |
| 10 | 2243.233 | 7.476 | 49 | 1349.399 | 4.497 | 49 | 1349.399 | 4.497 |
| 11 | 2236.783 | 7.455 | 50 | 1260.572 | 4.201 | 50 | 1260.572 | 4.201 |
| 12 | 2235.024 | 7.449 | 51 | 1121.030 | 3.736 | 51 | 1121.030 | 3.736 |
| 13 | 2230.334 | 7.433 | 52 | 1113.994 | 3.713 | 52 | 1113.994 | 3.713 |
| 14 | 2227.402 | 7.423 | 53 | 654.911 | 2.183 | 53 | 654.911 | 2.183 |
| 15 | 2224.471 | 7.414 | 54 | 638.495 | 2.128 | 54 | 638.495 | 2.128 |
| 16 | 2221.832 | 7.405 | 55 | 627.061 | 2.090 | 55 | 627.061 | 2.090 |
| 17 | 2218.607 | 7.394 | 56 | 617.094 | 2.057 | 56 | 617.094 | 2.057 |
| 18 | 2207.174 | 7.356 | 57 | 604.488 | 2.015 | 57 | 604.488 | 2.015 |
| 19 | 2205.415 | 7.350 | 58 | 595.401 | 1.984 | 58 | 595.401 | 1.984 |
| 20 | 2202.777 | 7.341 | 59 | 591.883 | 1.973 | 59 | 591.883 | 1.973 |
| 21 | 2189.845 | 7.332 | 60 | 574.586 | 1.915 | 60 | 574.586 | 1.915 |
| 22 | 2197.793 | 7.325 | 61 | 559.636 | 1.865 | 61 | 559.636 | 1.865 |
| 23 | 2191.051 | 7.302 | 62 | 516.835 | 1.722 | 62 | 516.835 | 1.722 |
| 24 | 2189.292 | 7.296 | 63 | 513.317 | 1.711 | 63 | 513.317 | 1.711 |
| 25 | 2187.240 | 7.290 | 64 | 510.385 | 1.701 | 64 | 510.385 | 1.701 |
| 26 | 2179.031 | 7.262 | 65 | 506.574 | 1.688 | 65 | 506.574 | 1.688 |
| 27 | 2175.807 | 7.251 | 66 | 499.538 | 1.665 | 66 | 499.538 | 1.665 |
| 28 | 2173.461 | 7.244 | 67 | 500.125 | 1.667 | 67 | 500.125 | 1.667 |
| 29 | 2171.702 | 7.238 | 68 | 497.193 | 1.657 | 68 | 497.193 | 1.657 |
| 30 | 2168.478 | 7.227 | 69 | 488.892 | 1.629 | 69 | 488.892 | 1.629 |
| 31 | 2159.097 | 7.196 | 70 | 472.861 | 1.576 | 70 | 472.861 | 1.576 |
| 32 | 2141.507 | 7.137 | 71 | 459.962 | 1.533 | 71 | 459.962 | 1.533 |
| 33 | 2139.748 | 7.131 | 72 | 456.738 | 1.522 | 72 | 456.738 | 1.522 |
| 34 | 2138.576 | 7.127 | 73 | 457.324 | 1.524 | 73 | 457.324 | 1.524 |
| 35 | 2137.403 | 7.123 | 74 | 446.770 | 1.489 | 74 | 446.770 | 1.489 |
| 36 | 2134.178 | 7.113 | 75 | 443.546 | 1.478 | 75 | 443.546 | 1.478 |
| 37 | 2131.247 | 7.103 | 76 | 436.510 | 1.455 | 76 | 436.510 | 1.455 |
| 38 | 2126.263 | 7.086 | 77 | 426.543 | 1.422 | 77 | 426.543 | 1.422 |
| 39 | 2123.625 | 7.078 | 78 | 417.162 | 1.390 | 78 | 417.162 | 1.390 |

Table 3, entry 1a



403C

exp1 s2pu1

SAMPLE Sep 15 2004
date Sep 15 2004
solvent CDC13
file exp
ACQUISITION
sw 18867.9
at 1.815
np 68482
fb 10400
bs 64
ss 4
d1 1.000
nt 1000
ct 1000
TRANSMITTER C13
tn 75.456
sfrq 737.2
tof 58
tpwr 9.250
pw DECOUPLER H1
dn 0
dof YYY
dm W
dmm SC
dpwr 35
dmf 6700
SPECIAL not used
gain not used
spin 20
hst 0.008
pw90 18.500
alfa 20.000
FLAGS
n
n
y
nh
hs
lb
fn
sp
wp
rfl
rfp
rp
lp
wc
sc
vs
th
ai
no
ph
1.00
not used
DISPLAY
-1134.4
18867.6
1134.7
0
112.0
-338.3
250
0
297
16
PLOT

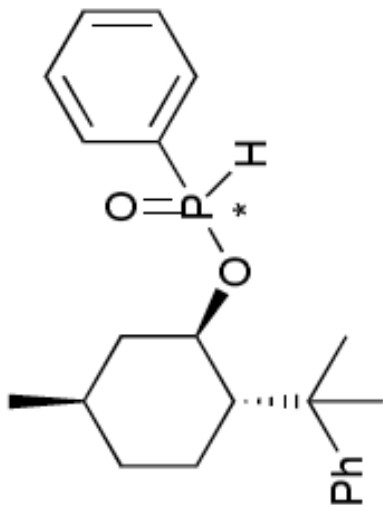
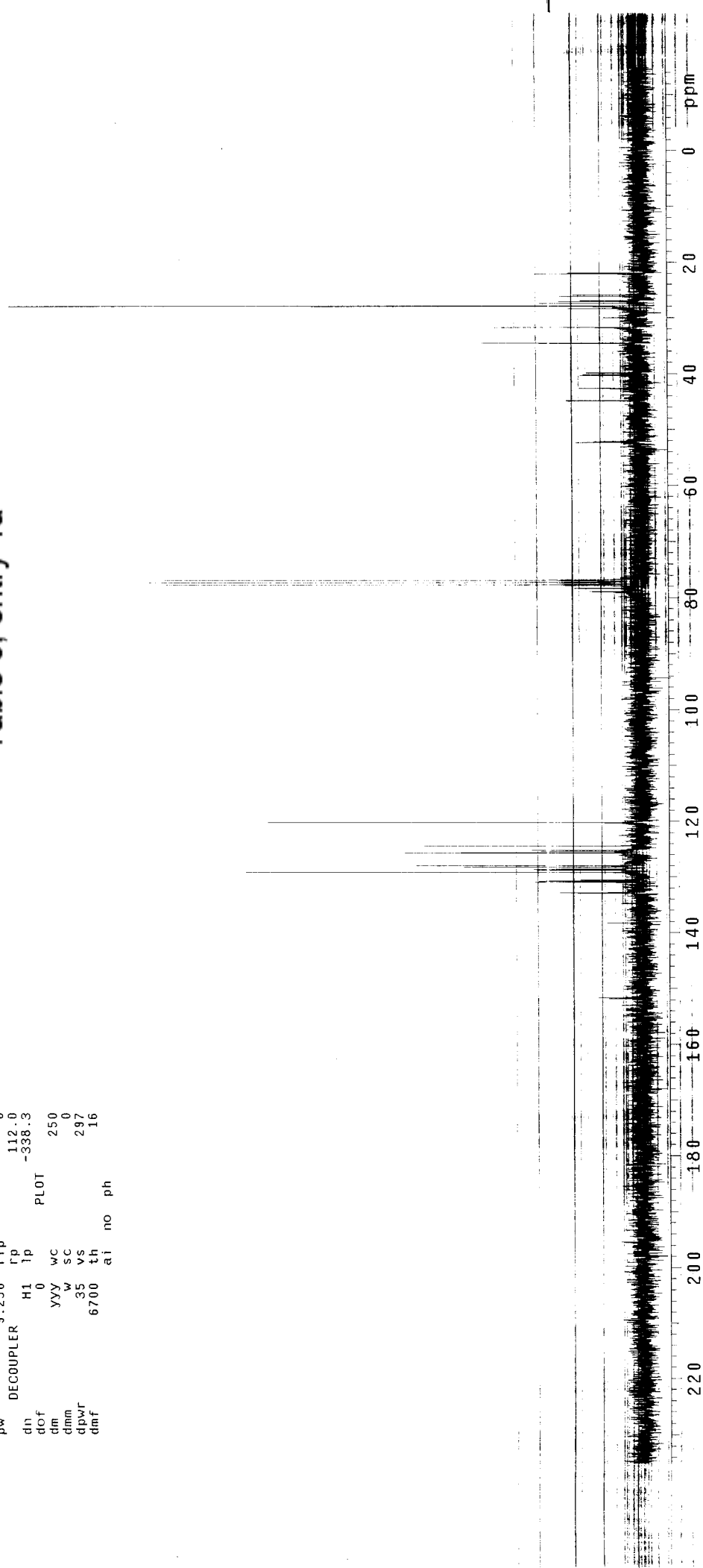


Table 3, entry 1a



Eq 404, Ester formation
403

| LINE | POINT | PPM | INTENSITY | REL_INT | LABEL |
|------|-------|--------|-----------|---------|-------|
| 1 | 24148 | 21.159 | 277032 | 5.157 | 1 |
| 2 | 24398 | 16.980 | 5273578 | 98.177 | 2 |
| 3 | 25237 | 2.921 | 847354 | 15.775 | 3 |

| PPM | Hz |
|--------|----|
| 771.05 | |
| 618.77 | |
| 106.45 | |

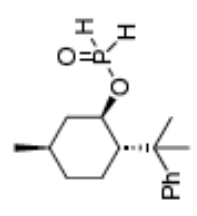
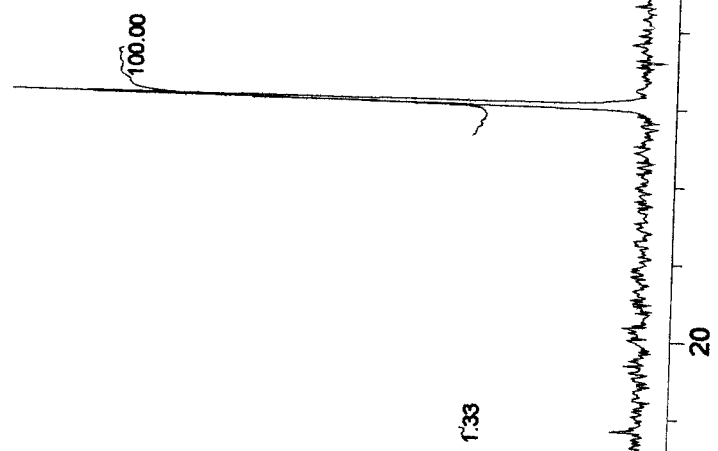


Table 3, entry 1c



| PPM | USER | DATE |
|--------|------|------------------|
| 100.00 | | 09/02/04 (19:39) |
| 27.77 | | |
| 1.33 | | |

| | | | |
|---------------------------|---------------|-------------|---------|
| F1: 36.441 | SW1: 20000 | OF1: 5510.0 | LB: 0.0 |
| EX: c:\left\global\zg.ppg | PW: 12.4 usec | NA: 8 | |
| | PD: 2.0 sec | | |

404 A

Interpolated Peak Listing

| PEAK | POINT | HEIGHT | REL. HT | HZ | PPM |
|------|-------|--------|---------|--------|--------|
| 1 | 24576 | 6211K | 109.46 | 509.82 | 13.990 |
| 2 | 25242 | 2637K | 46.47 | 103.22 | 2.832 |

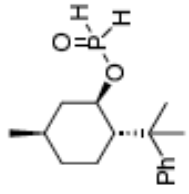
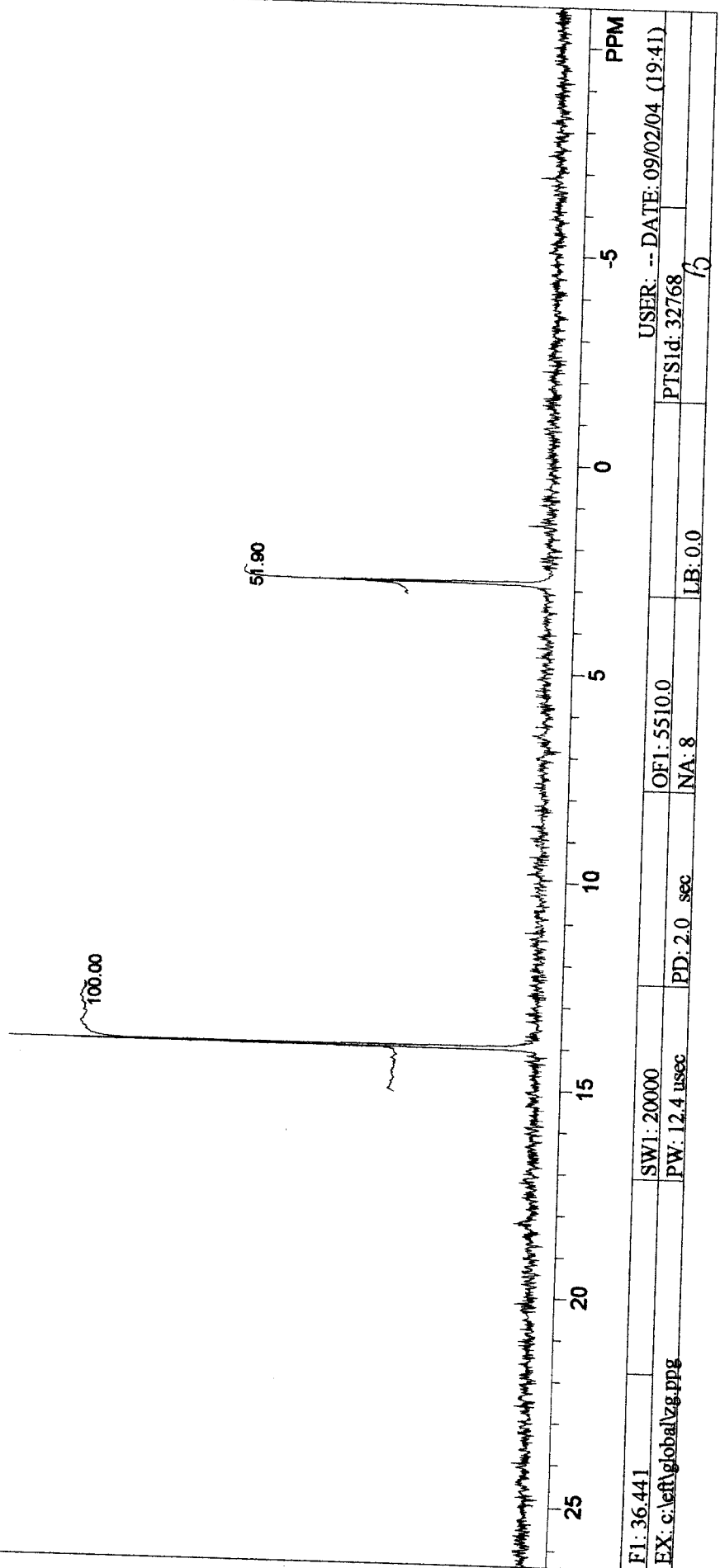


Table 3, entry 1c



| | | | | |
|---------------------------|---------------|-------------|---------|---------------------------------|
| FI: 36.441 | SW1: 20000 | OF1: 5510.0 | LB: 0.0 | USER: -- DATE: 09/02/04 (19:41) |
| EX: c:\left\global\zg.ppg | PW: 12.4 usec | NA: 8 | | PTSid: 32768 |

| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 3804.079 | 31.319 | 58.6 |
| 2 | 3037.059 | 25.004 | 126.0 |

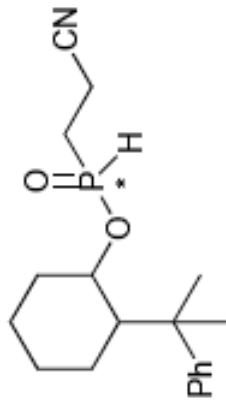
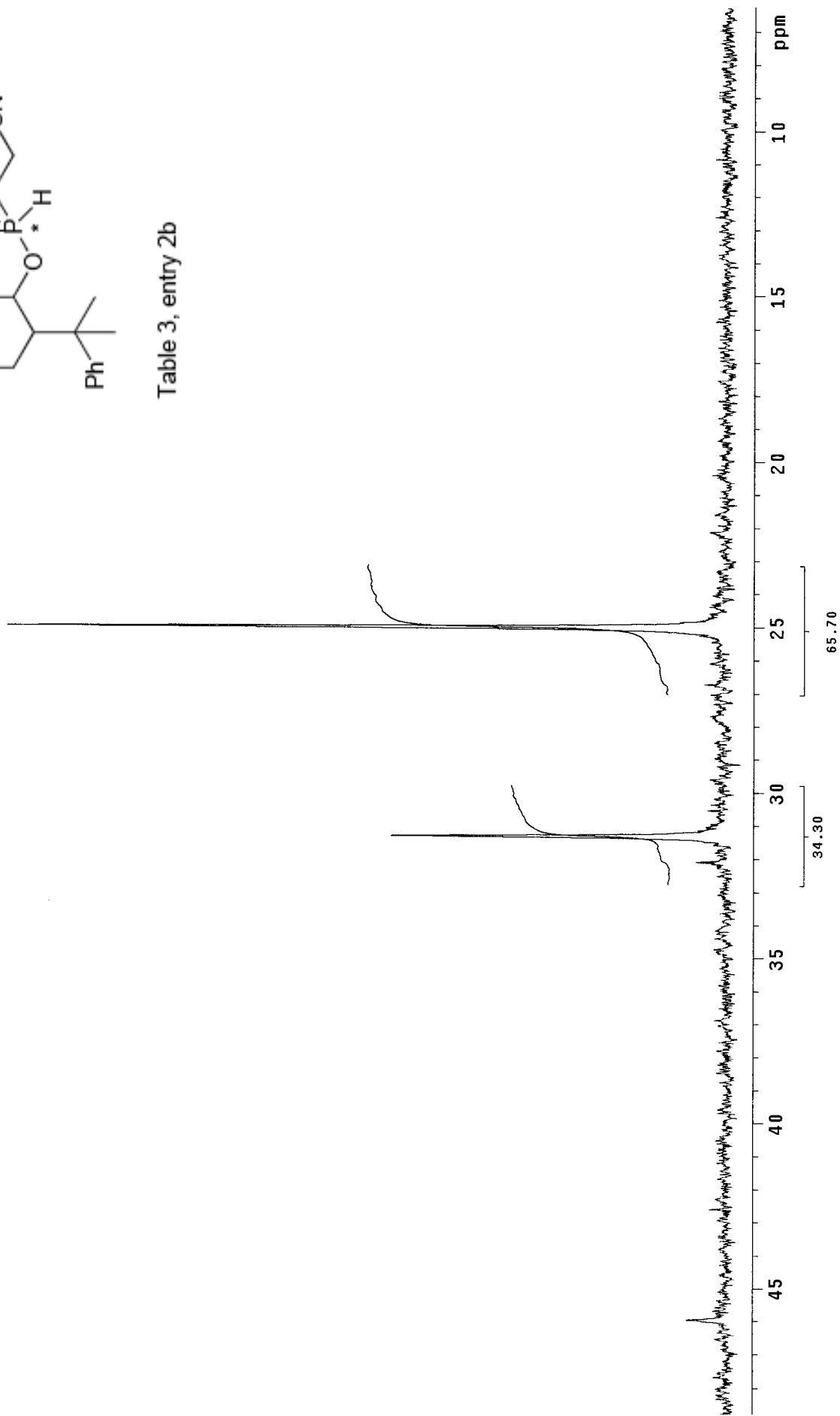


Table 3, entry 2b



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 4076.207 | 33.559 | 36.8 |
| 2 | 3523.382 | 29.008 | 35.8 |
| 3 | 3305.924 | 27.218 | 73.4 |
| 4 | 2766.971 | 22.780 | 66.6 |

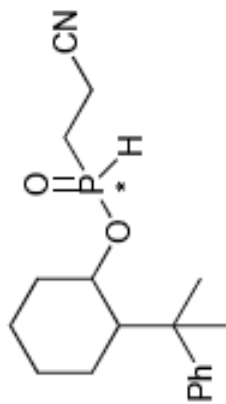
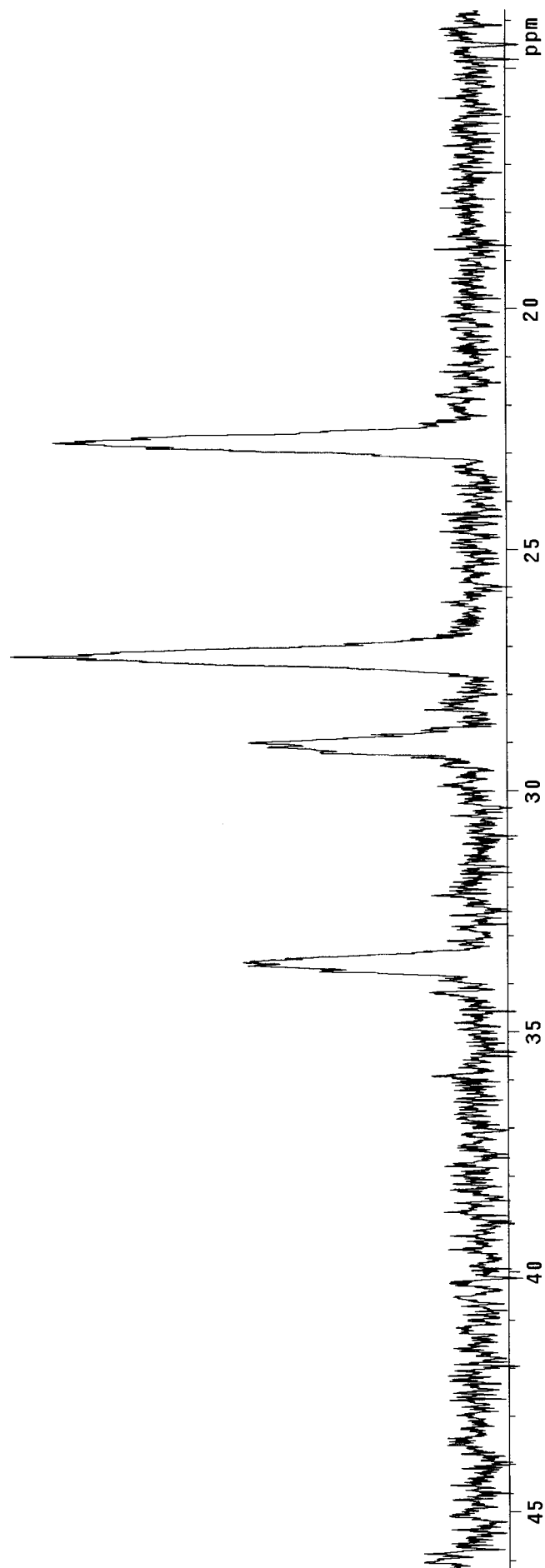


Table 3, entry 2b



660

exp1 s2pu1

| | | | |
|---------|-------------|---------|----------|
| date | Sep 29 2005 | SPECIAL | not used |
| solvent | CDCl3 | gain | not used |
| file | exp | spin | 20 |
| sw | 4803.1 | hst | 0.008 |
| at | 1.994 | pw90 | 17.200 |
| np | 19158 | alfa | 20.000 |
| fb | not used | il | n |
| bs | 16 | in | n |
| ss | 4 | dp | y |
| d1 | 1.000 | hs | nn |
| nt | 8 | fn | not used |
| ct | 8 | fn | not used |
| tn | H1 | sp | -60.0 |
| sfrq | 300.053 | wp | 3060.5 |
| tof | 300.9 | rfl | 597.2 |
| tpwr | 55 | rfl | 0 |
| pw | 8.600 | rp | -108.5 |
| de | C13 | lp | -73.2 |
| dh | 0 | wc | 250 |
| dof | nn | sc | 0 |
| dm | C | vs | 281 |
| dmm | 45 | th | 2 |
| dpwr | 13100 | ai | cdc |
| dmf | | ph | |

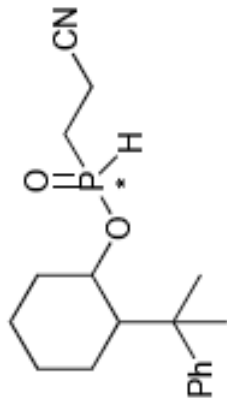
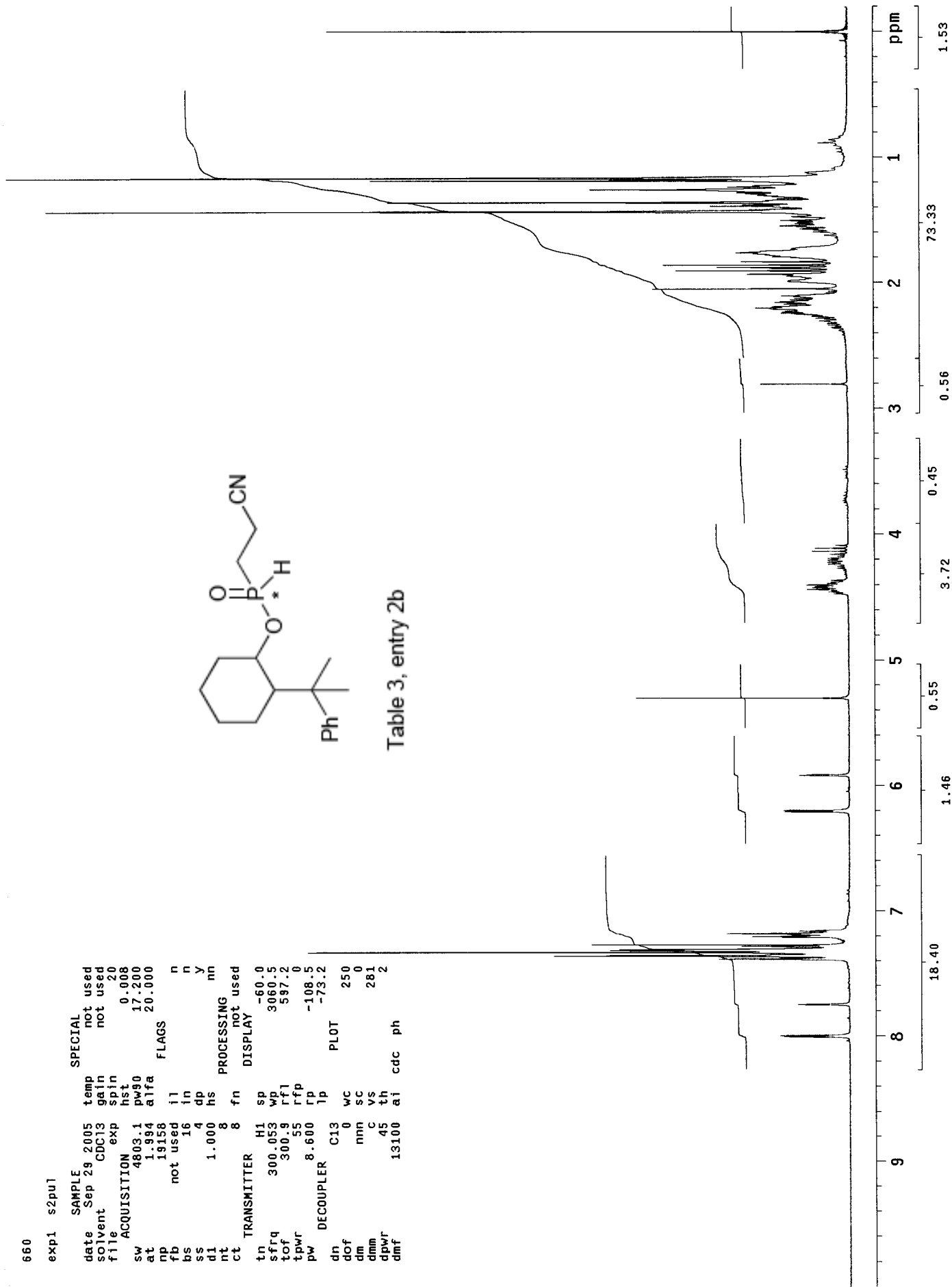


Table 3, entry 2b



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 4191.868 | 34.510 | 126.0 |
| 2 | 3346.723 | 27.553 | 87.0 |

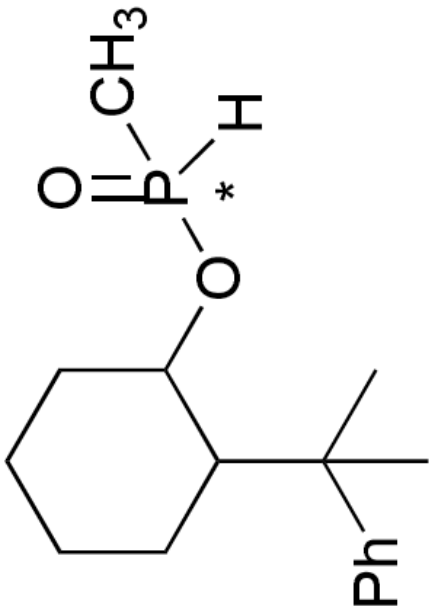
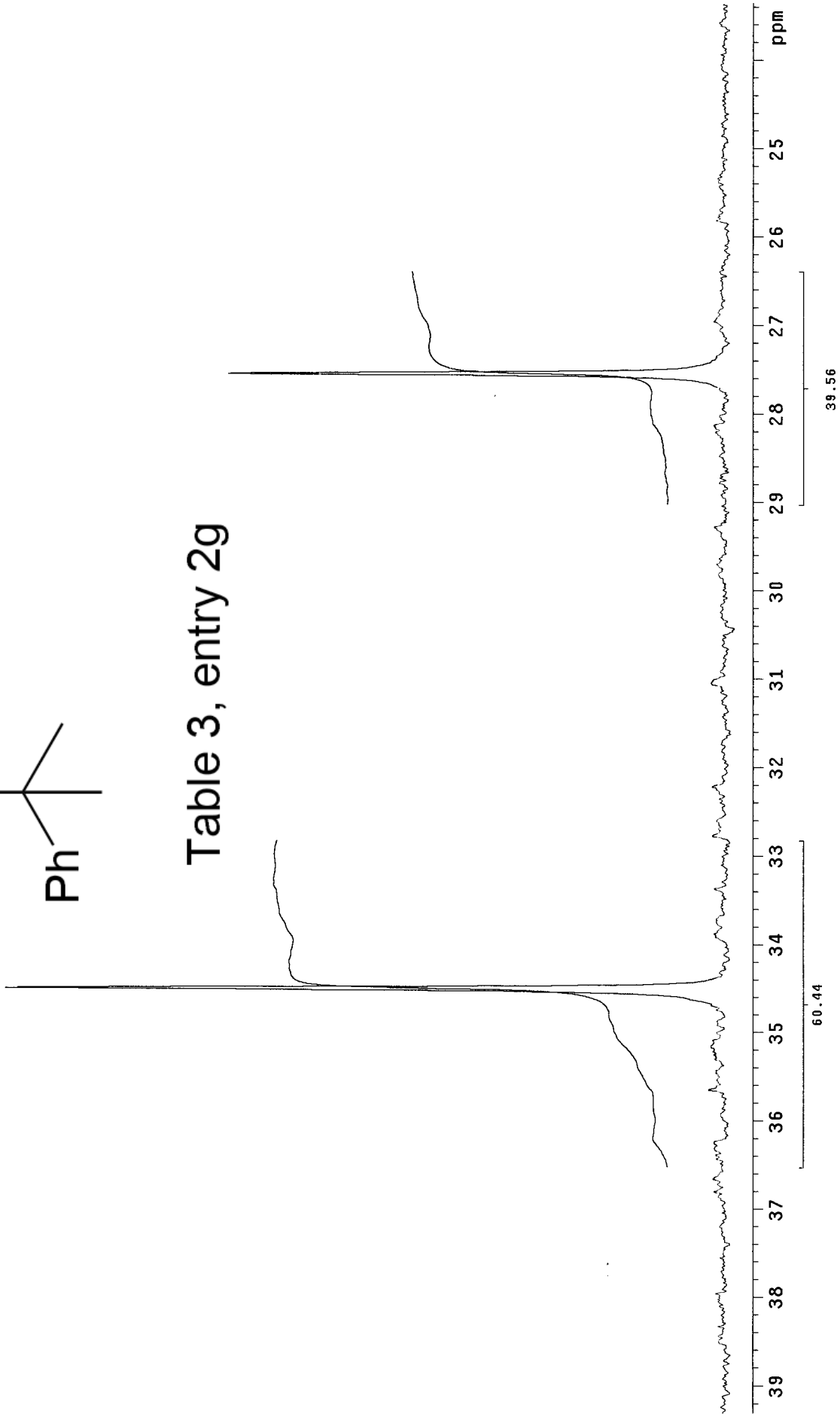
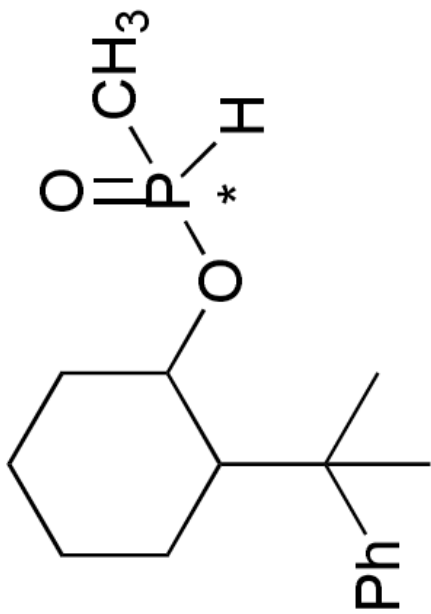


Table 3, entry 2g

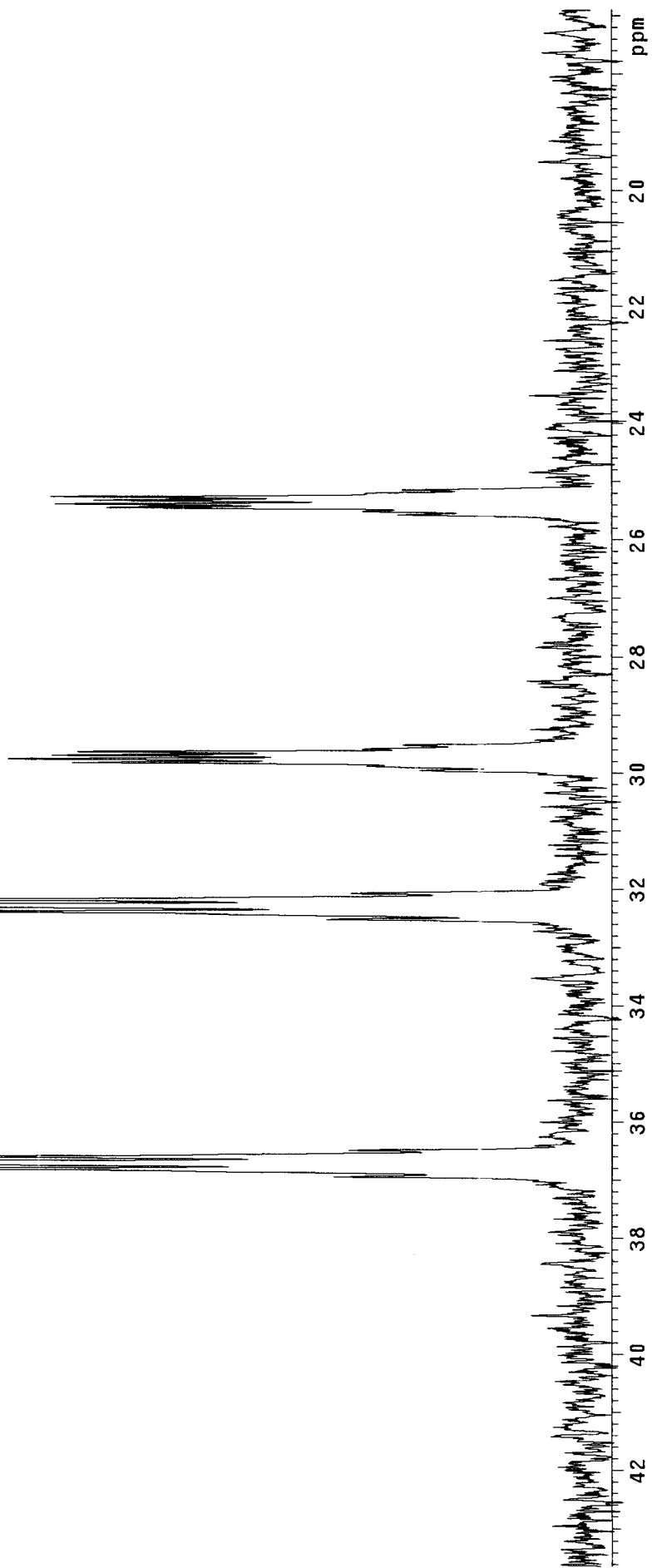




| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 4487.868 | 36.949 | 38.0 |
| 2 | 4473.181 | 36.828 | 117.3 |
| 3 | 4462.981 | 36.744 | 132.2 |
| 4 | 4458.085 | 36.703 | 127.0 |
| 5 | 4448.293 | 36.623 | 112.9 |
| 6 | 4432.790 | 36.495 | 36.7 |
| 7 | 3950.955 | 32.528 | 40.1 |
| 8 | 3935.859 | 32.404 | 109.7 |
| 9 | 3925.660 | 32.320 | 122.4 |
| 10 | 3921.172 | 32.283 | 123.6 |
| 11 | 3910.972 | 32.199 | 113.7 |
| 12 | 3896.284 | 32.078 | 36.3 |
| 13 | 3624.156 | 29.838 | 60.5 |
| 14 | 3616.812 | 29.777 | 90.7 |
| 15 | 3608.652 | 29.710 | 83.7 |
| 16 | 3601.308 | 29.649 | 79.7 |
| 17 | 3091.730 | 25.454 | 76.0 |
| 18 | 3084.794 | 25.397 | 83.2 |
| 19 | 3076.634 | 25.330 | 77.1 |
| 20 | 3069.231 | 25.269 | 83.9 |

537

Table 3, entry 2g



666

exp1 s2pu1

| | | | | |
|---------|-------------|---------|----------|----------|
| date | Sep 29 2005 | SPECIAL | temp | not used |
| solvent | CDCl3 | gain | not used | |
| file | exp | spin | 20 | |
| sw | 4803.1 | nst | 0.008 | |
| at | 1.994 | pw90 | 17.200 | |
| fb | 19158 | alpha | 20.000 | |
| bs | not used | il | | n |
| ss | 16 | in | | n |
| d1 | 4 | dp | | y |
| ct | 1.000 | hs | | nn |
| tn | 8 | fn | | not used |
| trf | 8 | fn | | not used |
| sfrq | H1 | sp | -60.0 | |
| tof | 300.053 | wp | 3060.5 | |
| tpwr | 300.9 | rfl | 597.7 | |
| pw | 55 | rfl | 0 | |
| dn | 8.600 | rp | -110.7 | |
| dof | lp | tp | -69.3 | |
| dm | C13 | pl | | |
| dmm | 0 | wc | 250 | |
| dpwr | 45 | vs | 279 | |
| dmf | 13100 | ai | cdc | ph |

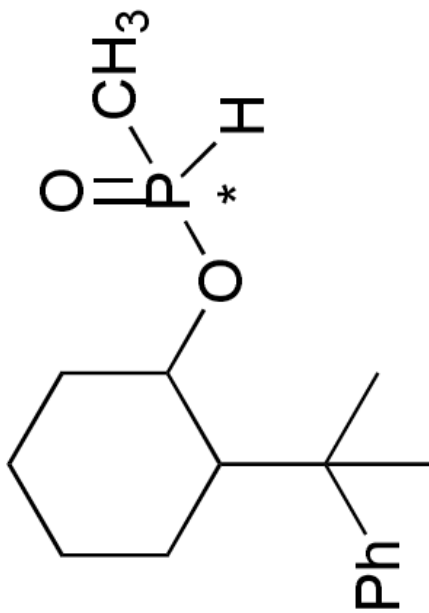
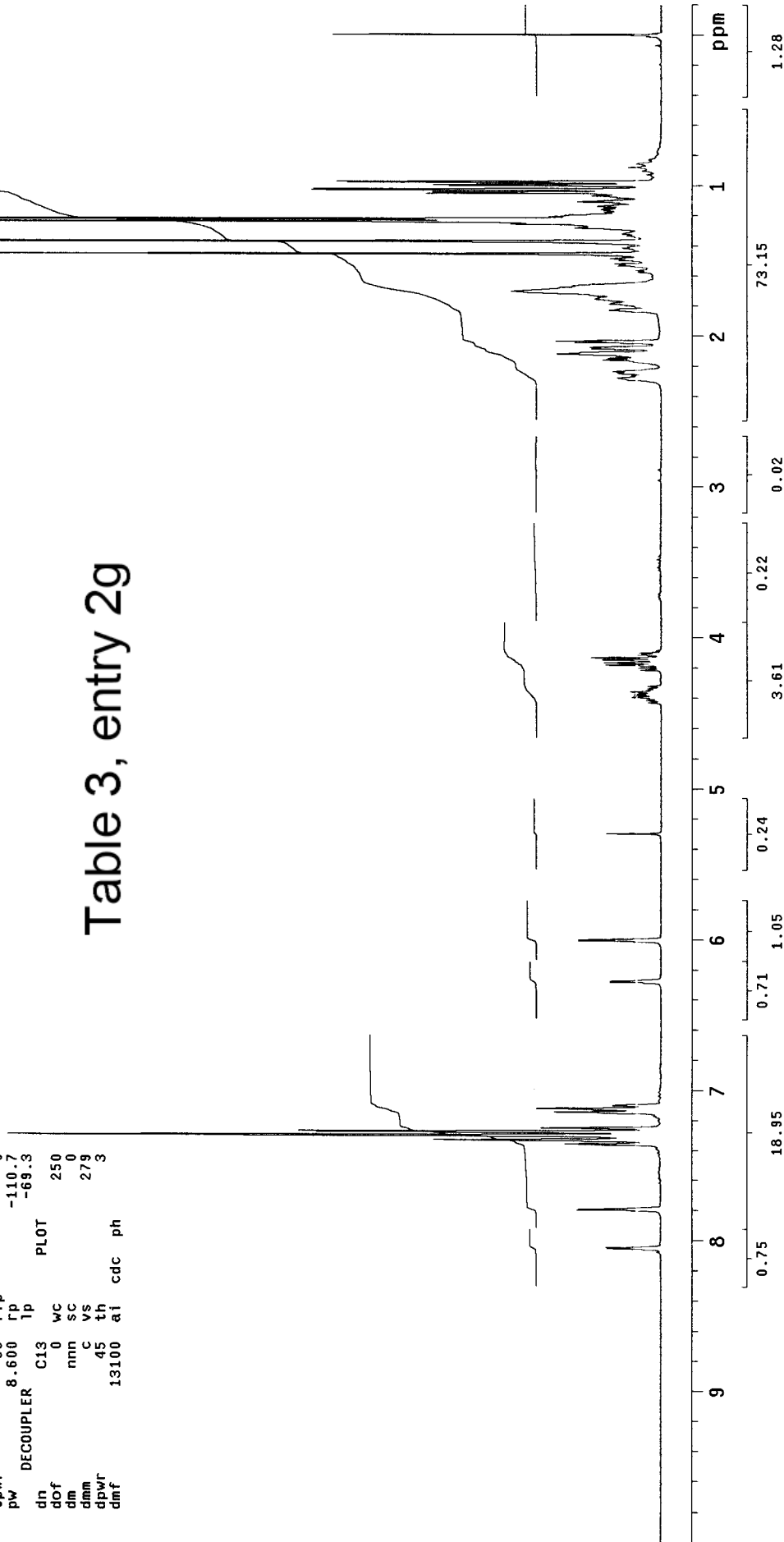
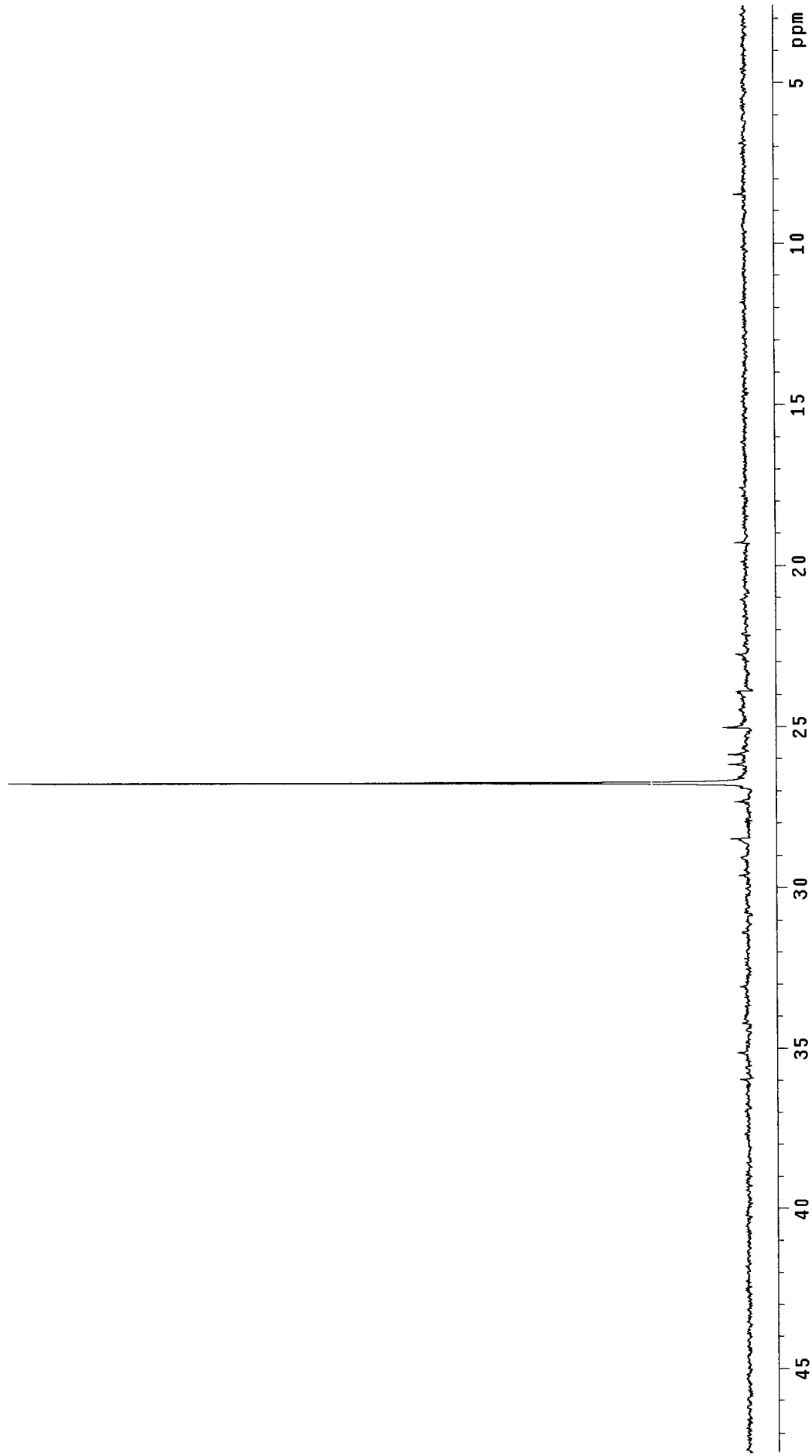


Table 3, entry 2g



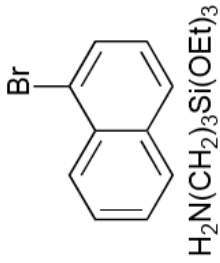
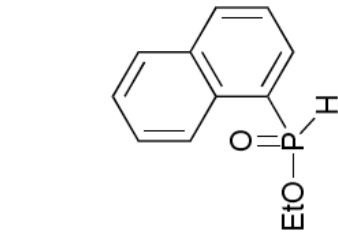
| INDEX | FREQUENCY PPM | HEIGHT |
|-------|---------------|--------|
| 1 | 3250.438 | 126.0 |
| 2 | 3040.323 | 4.0 |



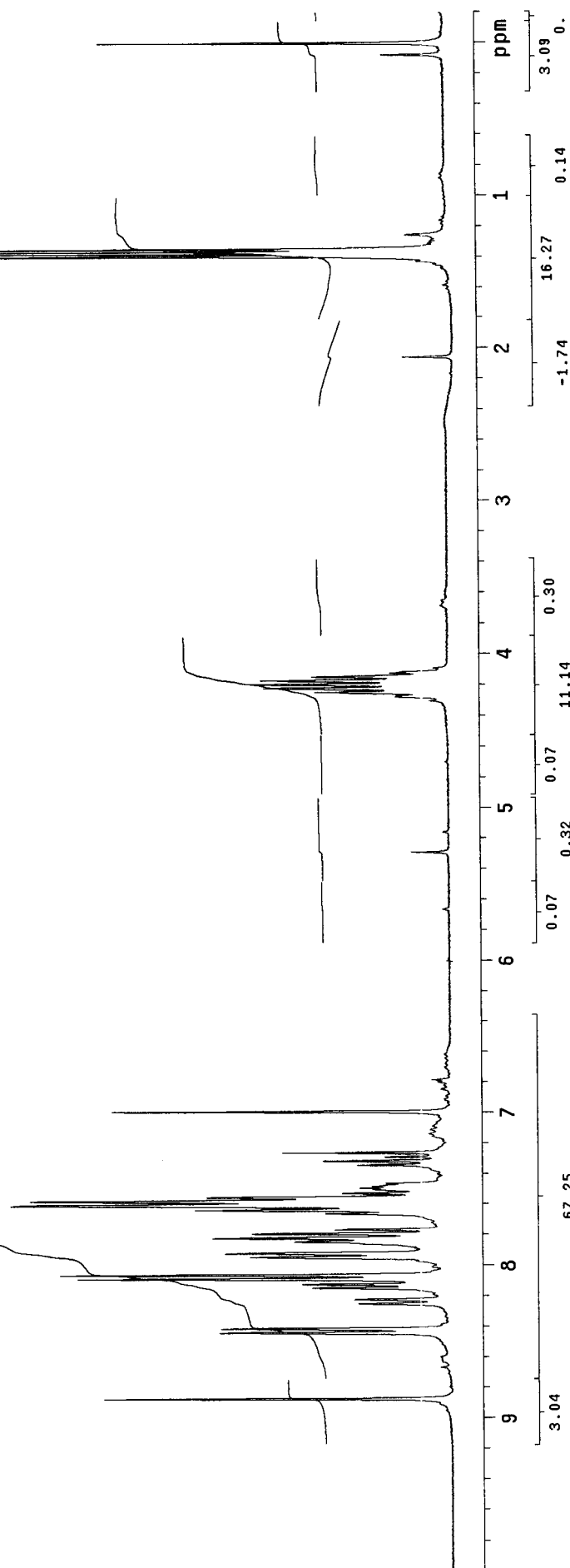
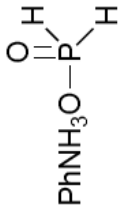
546b

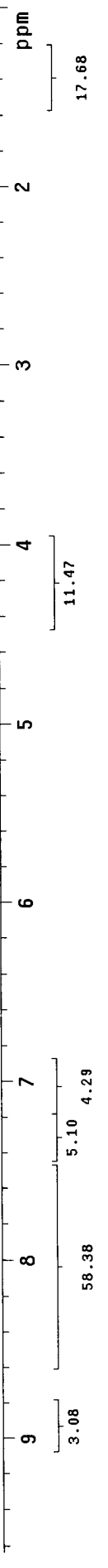
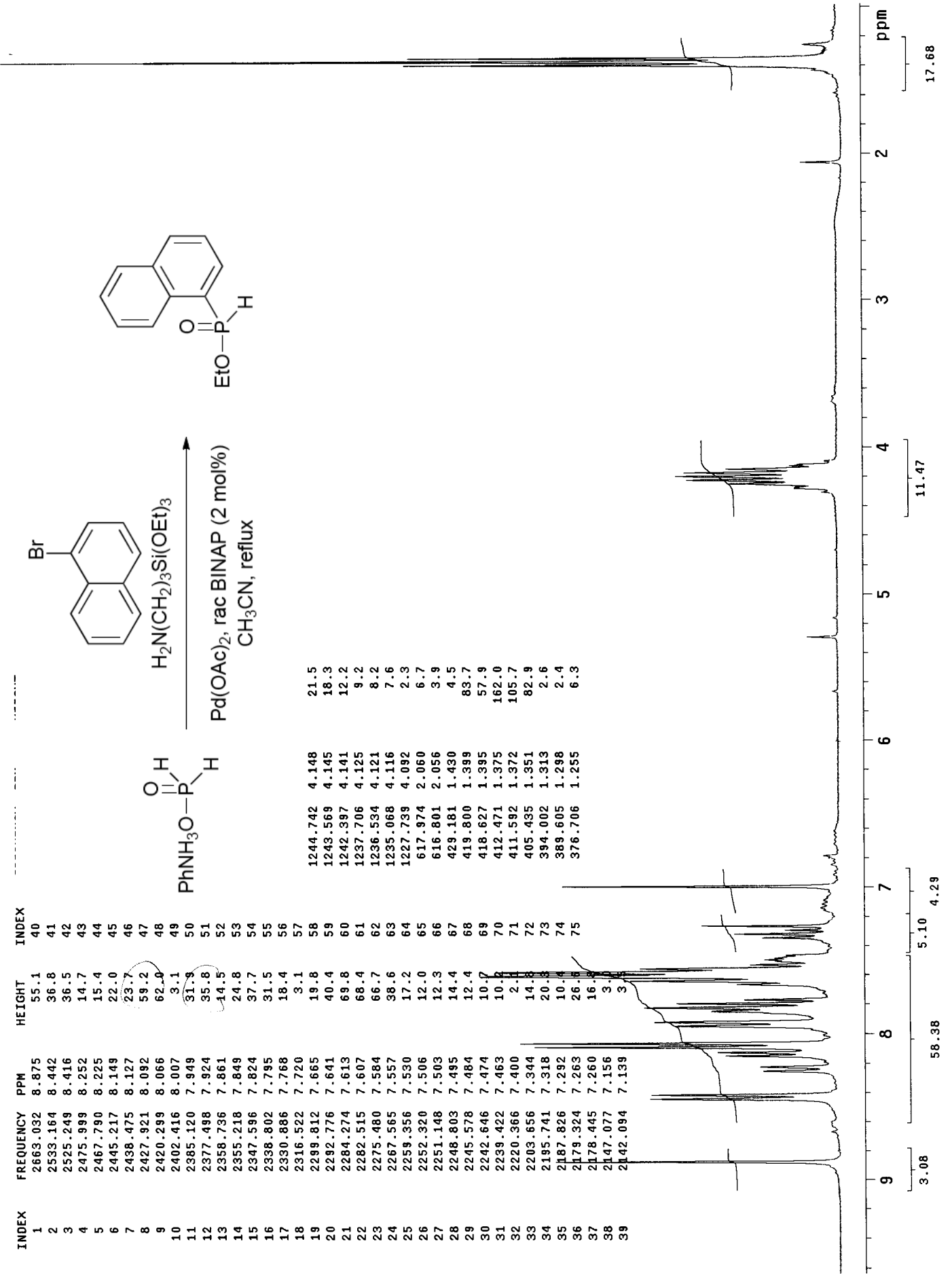
exp1 s2pu1

SAMPLE SPECIAL
date Jul 12 2005 temp not used
solvent CDC13 gain not used
file exp 20
ACQUISITION hst 0.008
sw 4803.1 pw90 17.200
at 1.994 alfa 20.000
np not used il n
fb 16 in n
bs 4 dp y
ss 1.000 hs fn
nt 8 fn not used
ct TRANSMITTER 8 fn not used
tn H1 sp
sfrq 300.053 wp 3060.5
tof 300.9 rfl 599.8
tpwr 55 rfp 0
pw 8.600 rp -114.4
DECOUPLER C13 lp -56.7
dn WC PLOT 250
dof 0
dm nmn SC 0
dmm C VS 250
dpwr 45 th 2
dmf 13100 ai cdc ph



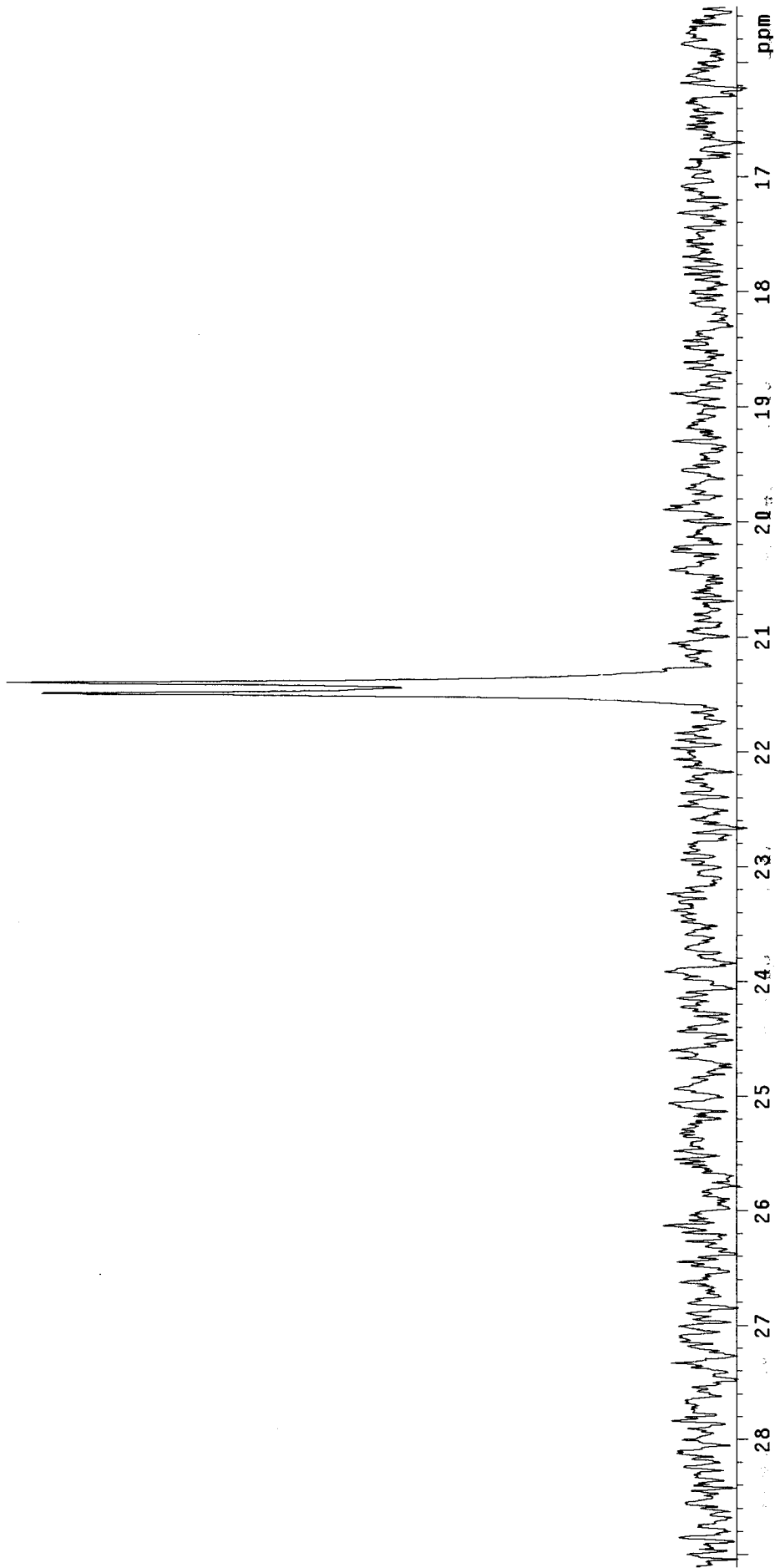
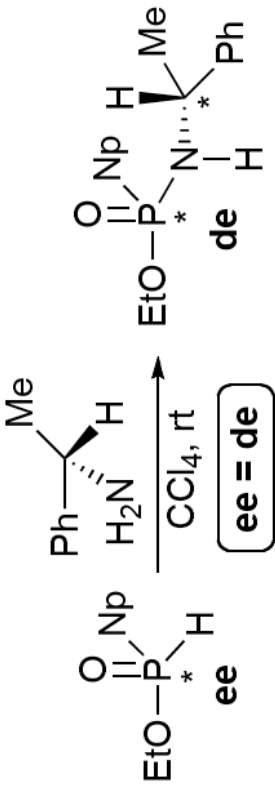
Pd(OAc)₂, rac BINAP (2 mol%)
CH₃CN, reflux





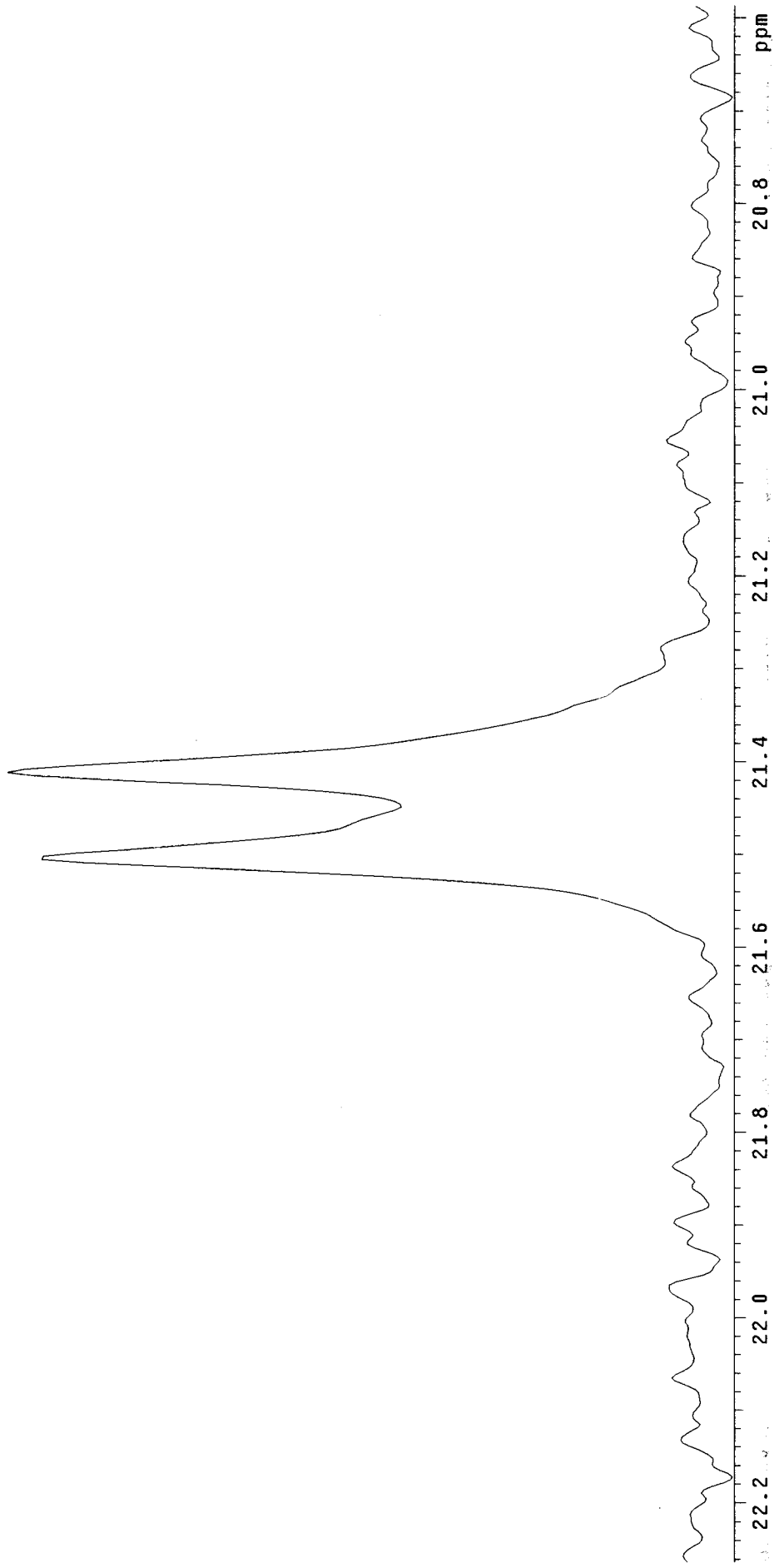
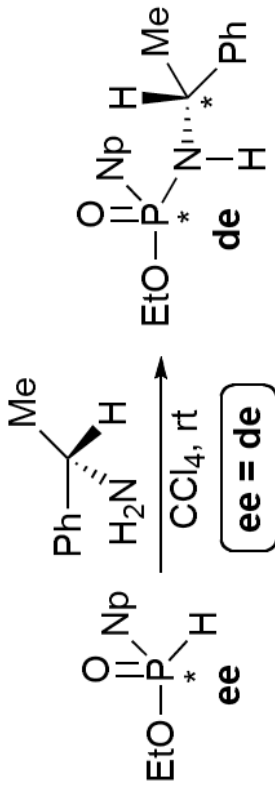
| INDEX | FREQUENCY | PPM |
|-------|-----------|--------|
| 1 | 2612.343 | 21.507 |
| 2 | 2600.919 | 21.413 |

³¹P NMR assay

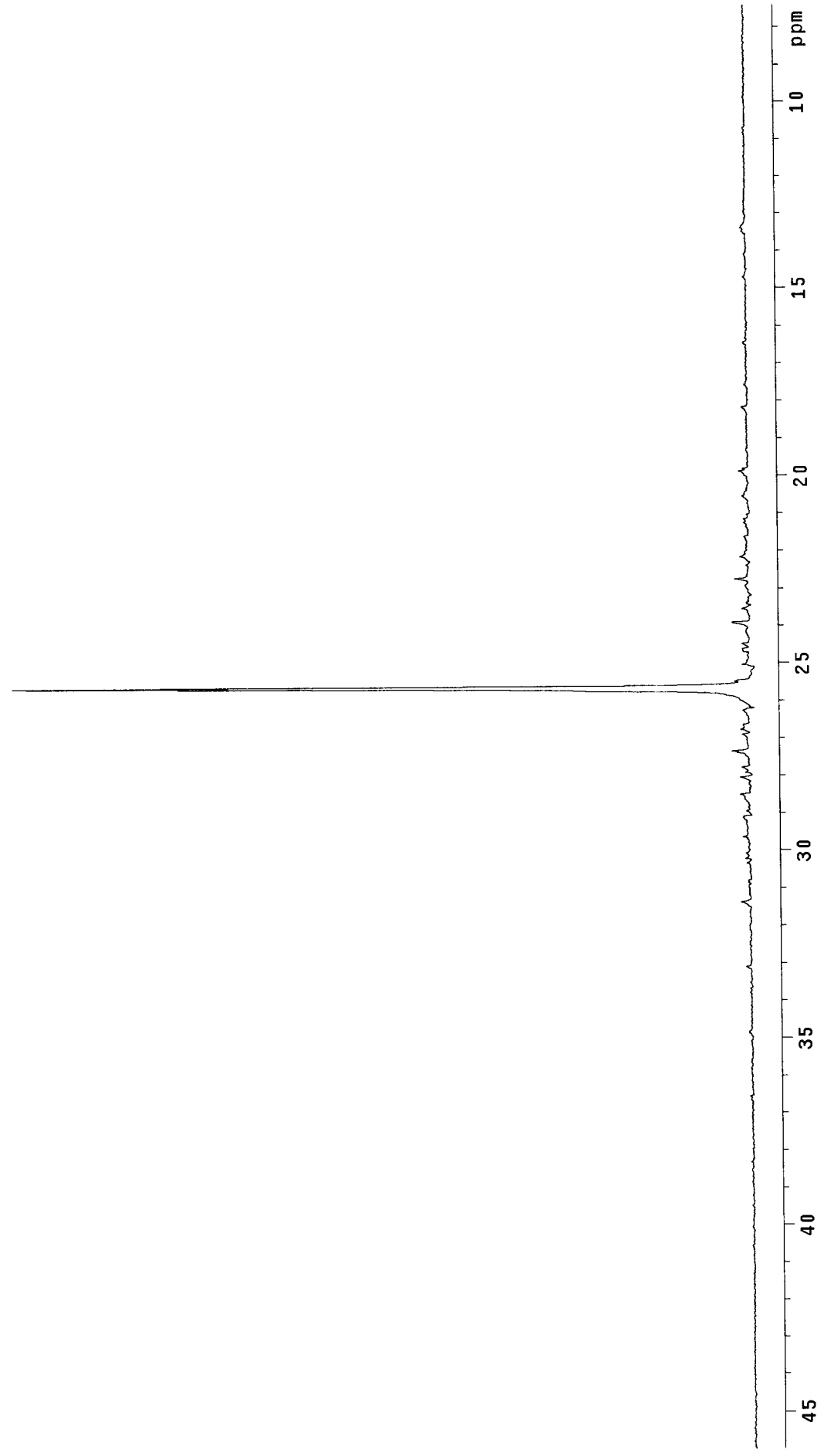
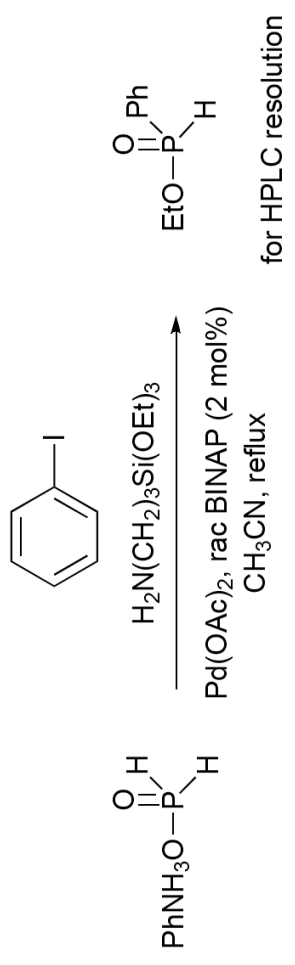


| INDEX | FREQUENCY (PPM) | HEIGHT |
|-------|-----------------|--------|
| 1 | 2612.343 | 104.5 |
| 2 | 2600.919 | 110.0 |

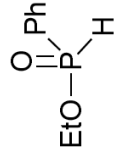
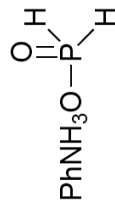
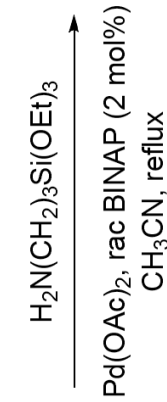
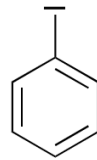
³¹P NMR assay



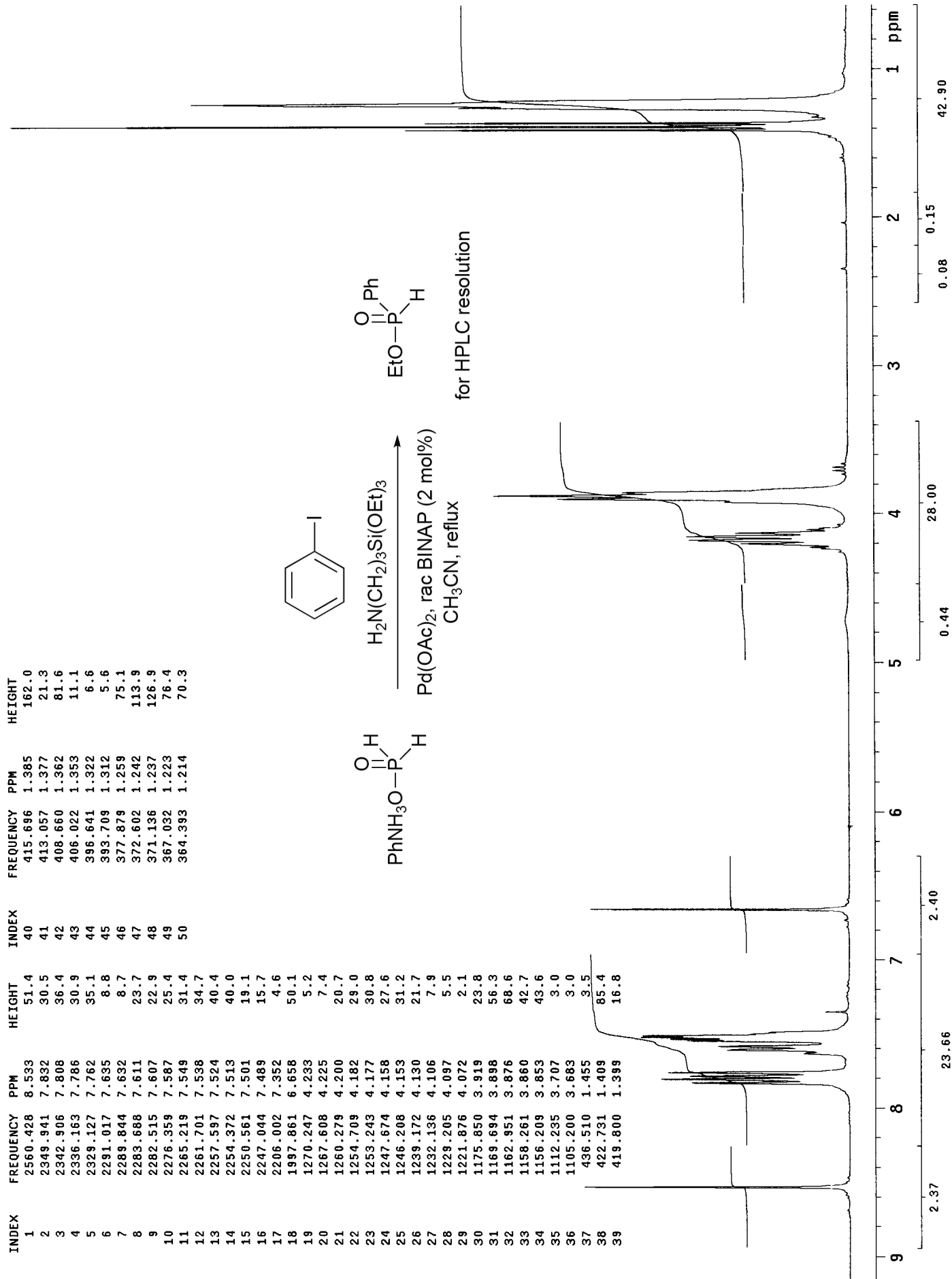
| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 3322.652 | 27.355 | 3.1 |
| 2 | 3117.025 | 25.662 | 97.7 |
| 3 | 3113.353 | 25.632 | 126.0 |
| 4 | 2904.463 | 23.912 | 3.0 |



| INDEX | FREQUENCY PPM | HEIGHT | INDEX | FREQUENCY PPM | HEIGHT |
|-------|---------------|--------|-------|---------------|--------|
| 1 | 2560.428 | 8.533 | 40 | 415.696 | 1.385 |
| 2 | 2349.941 | 7.832 | 41 | 413.057 | 1.377 |
| 3 | 2342.906 | 7.808 | 42 | 408.660 | 1.362 |
| 4 | 2336.163 | 7.786 | 43 | 406.022 | 1.353 |
| 5 | 2329.127 | 7.762 | 44 | 396.641 | 1.322 |
| 6 | 2291.017 | 7.635 | 45 | 393.709 | 1.312 |
| 7 | 2289.844 | 7.632 | 46 | 377.879 | 1.259 |
| 8 | 2283.688 | 7.611 | 47 | 372.602 | 1.242 |
| 9 | 2282.515 | 7.607 | 48 | 371.136 | 1.237 |
| 10 | 2276.359 | 7.587 | 49 | 367.032 | 1.223 |
| 11 | 2265.219 | 7.549 | 50 | 364.393 | 1.214 |
| 12 | 2261.701 | 7.538 | | | |
| 13 | 2257.597 | 7.524 | | | |
| 14 | 2254.372 | 7.513 | | | |
| 15 | 2250.561 | 7.501 | | | |
| 16 | 2247.044 | 7.489 | | | |
| 17 | 2206.002 | 7.352 | | | |
| 18 | 1997.861 | 6.658 | | | |
| 19 | 1270.247 | 4.233 | | | |
| 20 | 1267.608 | 4.225 | | | |
| 21 | 1260.279 | 4.200 | | | |
| 22 | 1254.709 | 4.182 | | | |
| 23 | 1253.243 | 4.177 | | | |
| 24 | 1247.674 | 4.158 | | | |
| 25 | 1246.208 | 4.153 | | | |
| 26 | 1239.172 | 4.130 | | | |
| 27 | 1232.136 | 4.106 | | | |
| 28 | 1229.205 | 4.097 | | | |
| 29 | 1221.876 | 4.072 | | | |
| 30 | 1175.850 | 3.919 | | | |
| 31 | 1169.694 | 3.898 | | | |
| 32 | 1162.951 | 3.876 | | | |
| 33 | 1158.261 | 3.860 | | | |
| 34 | 1156.209 | 3.853 | | | |
| 35 | 1112.235 | 3.707 | | | |
| 36 | 1105.200 | 3.683 | | | |
| 37 | 436.510 | 1.455 | | | |
| 38 | 422.731 | 1.409 | | | |
| 39 | 419.800 | 1.399 | | | |



for HPLC resolution



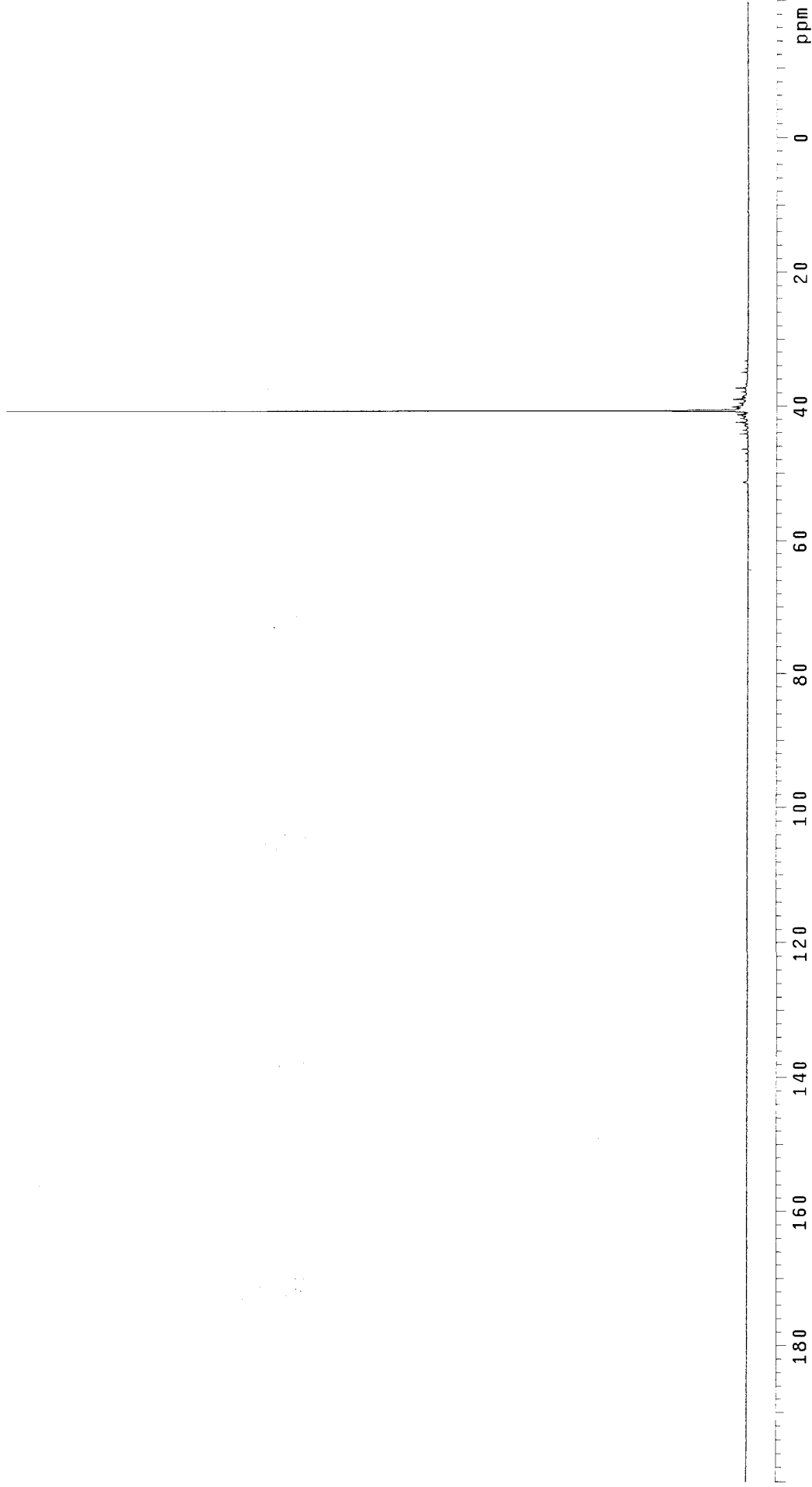
INDEX
1

FREQUENCY PPM
4945.224 40.714

HEIGHT
126.0



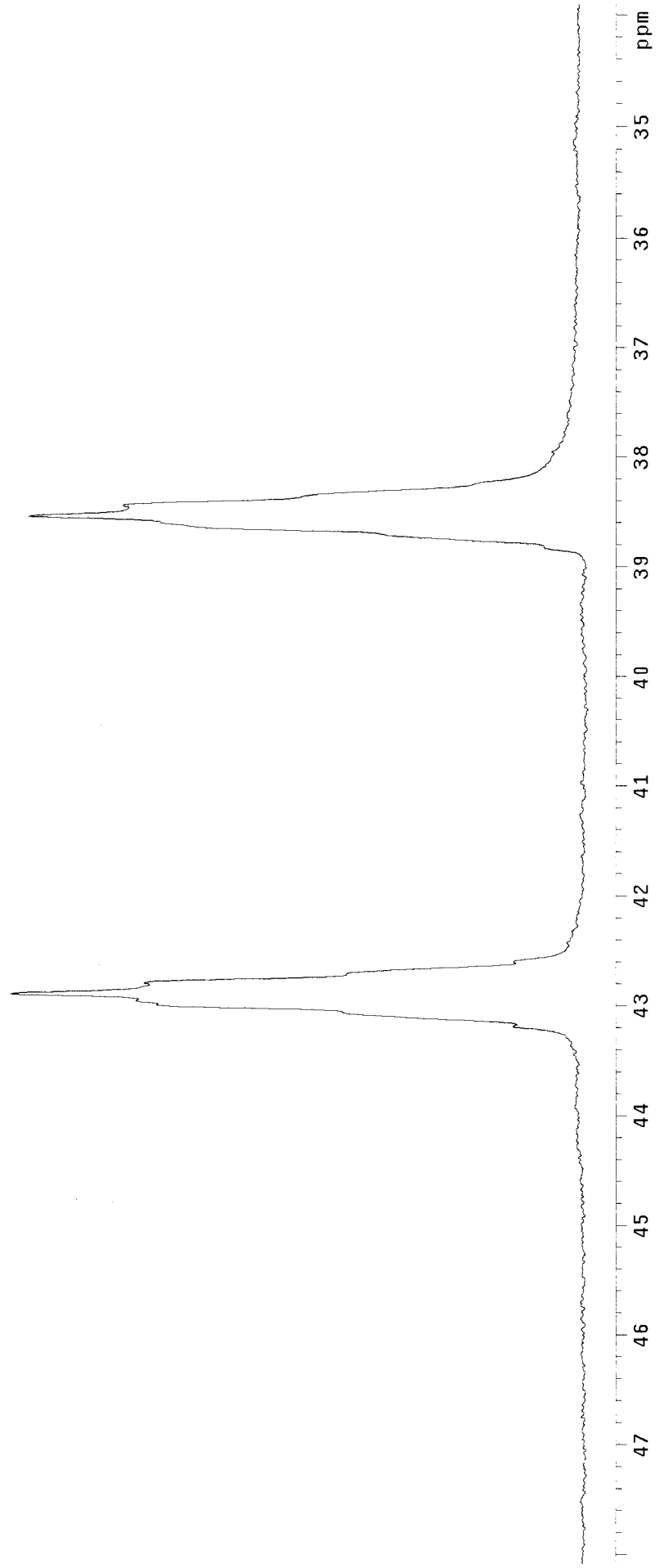
Scheme 4



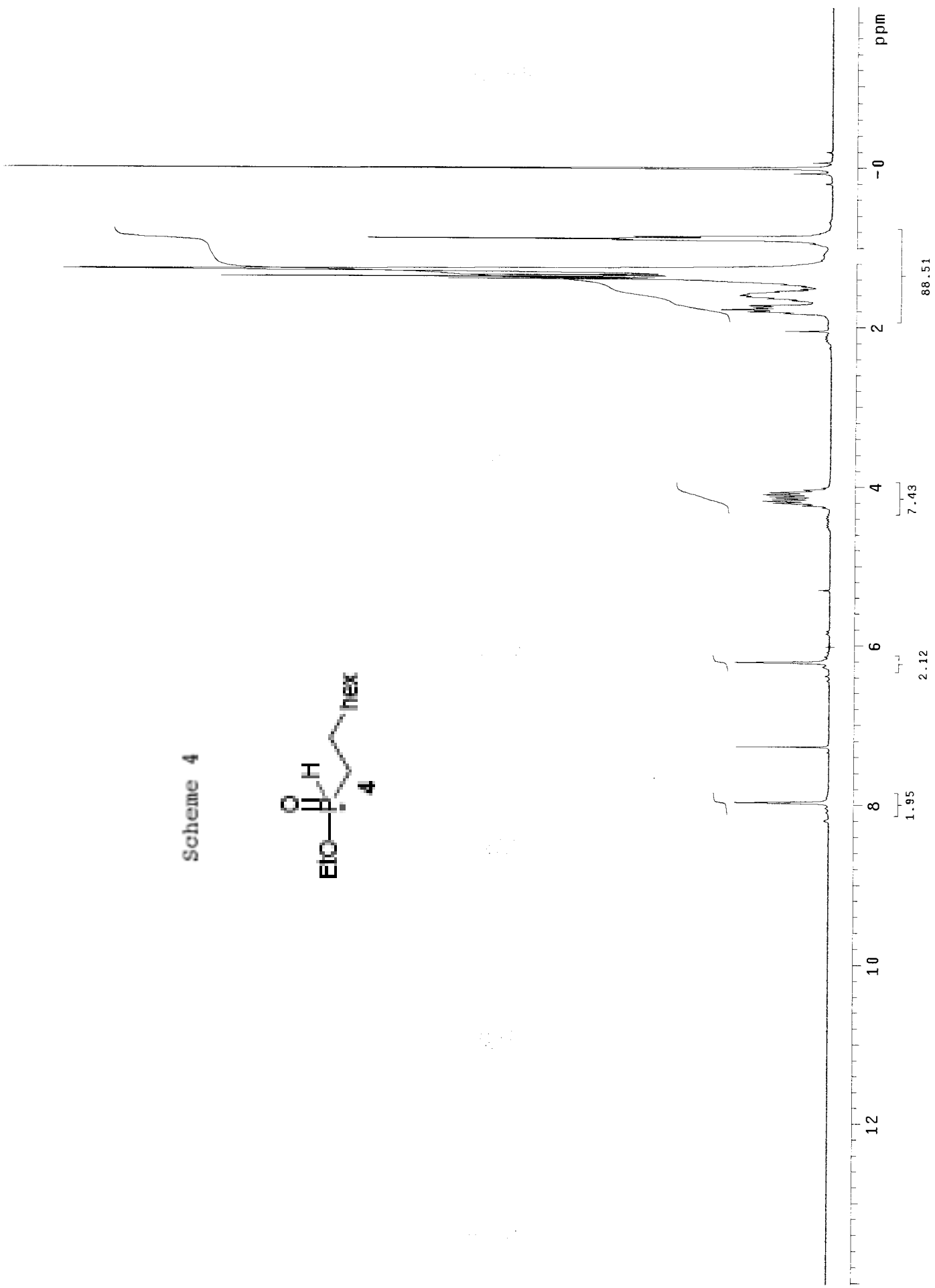
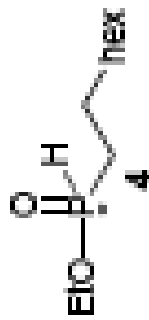
| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 5209.193 | 42.887 | 92.8 |
| 2 | 5197.361 | 42.790 | 71.1 |
| 3 | 4679.623 | 38.527 | 89.8 |
| 4 | 4667.792 | 38.430 | 74.5 |



Scheme 4



Scheme 4



| 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 |



Scheme 4

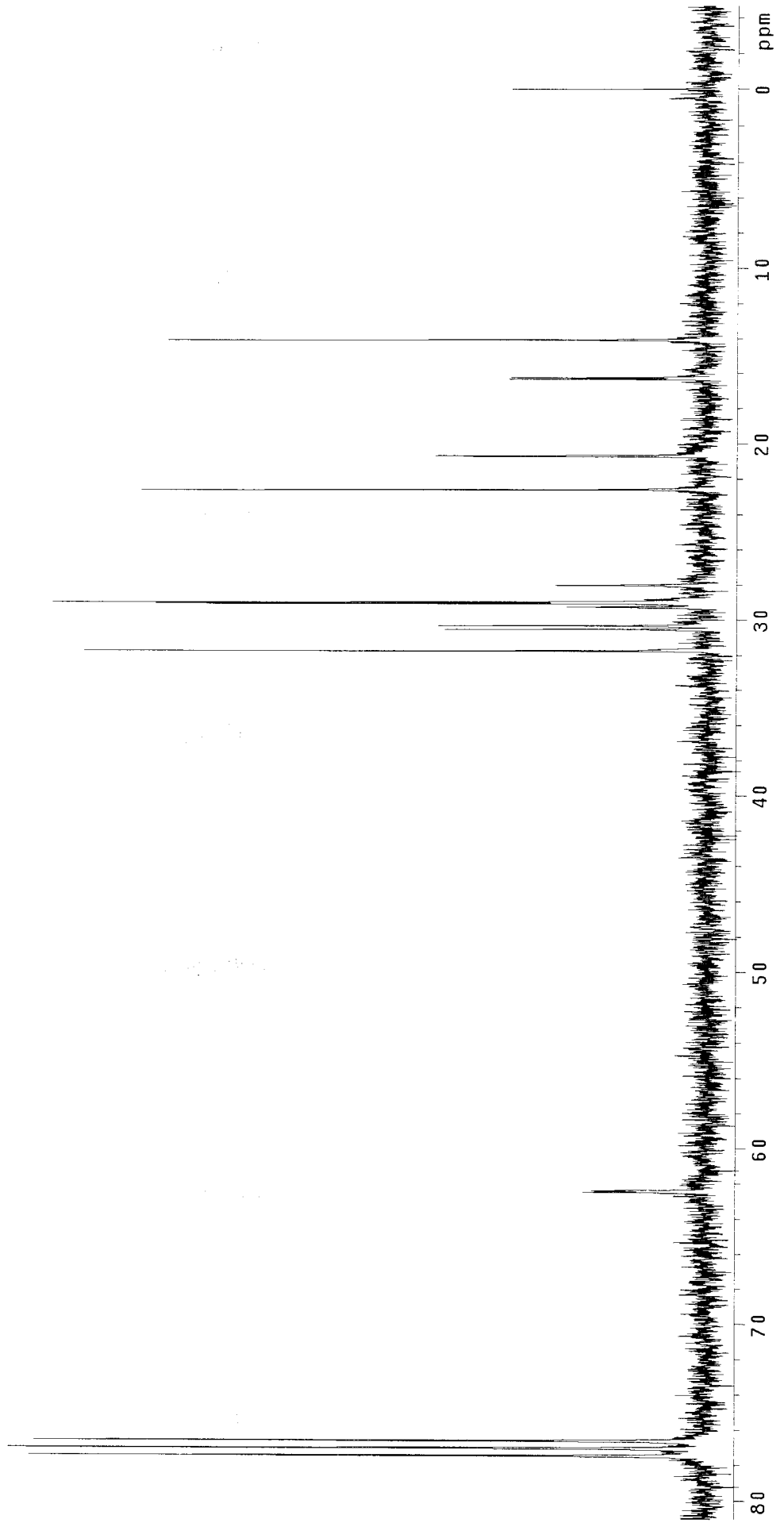
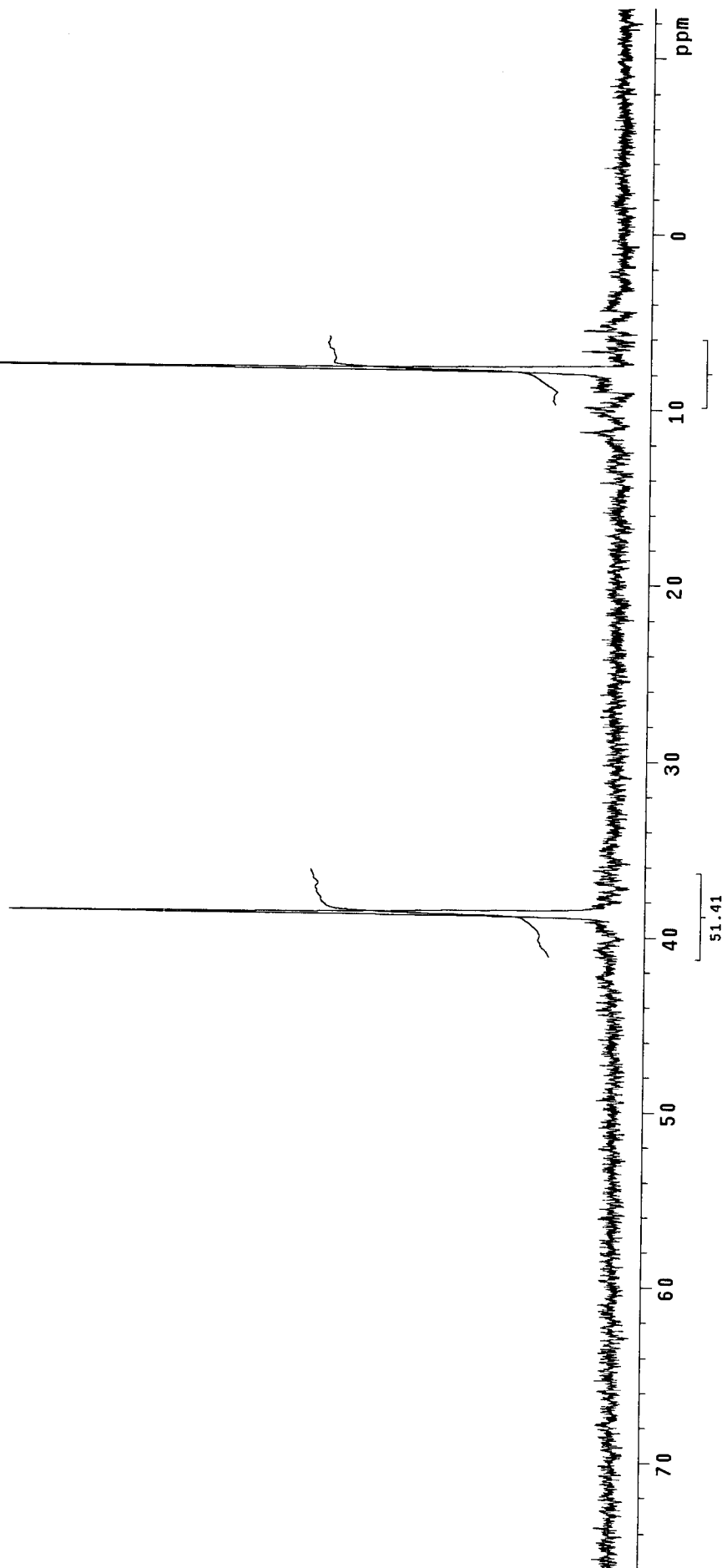
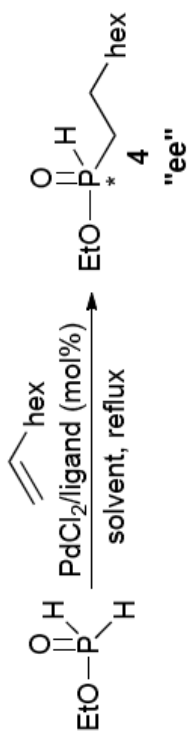


Table 4, entry 1a

| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 4710.630 | 38.783 | 95.4 |
| 2 | 949.380 | 7.816 | 126.0 |

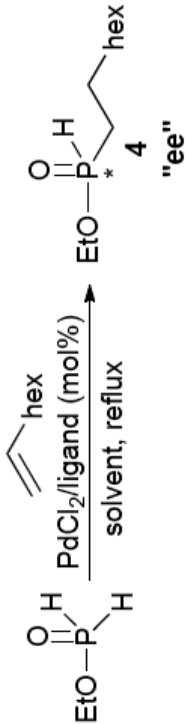


939 = 941 (NMM crude)

939

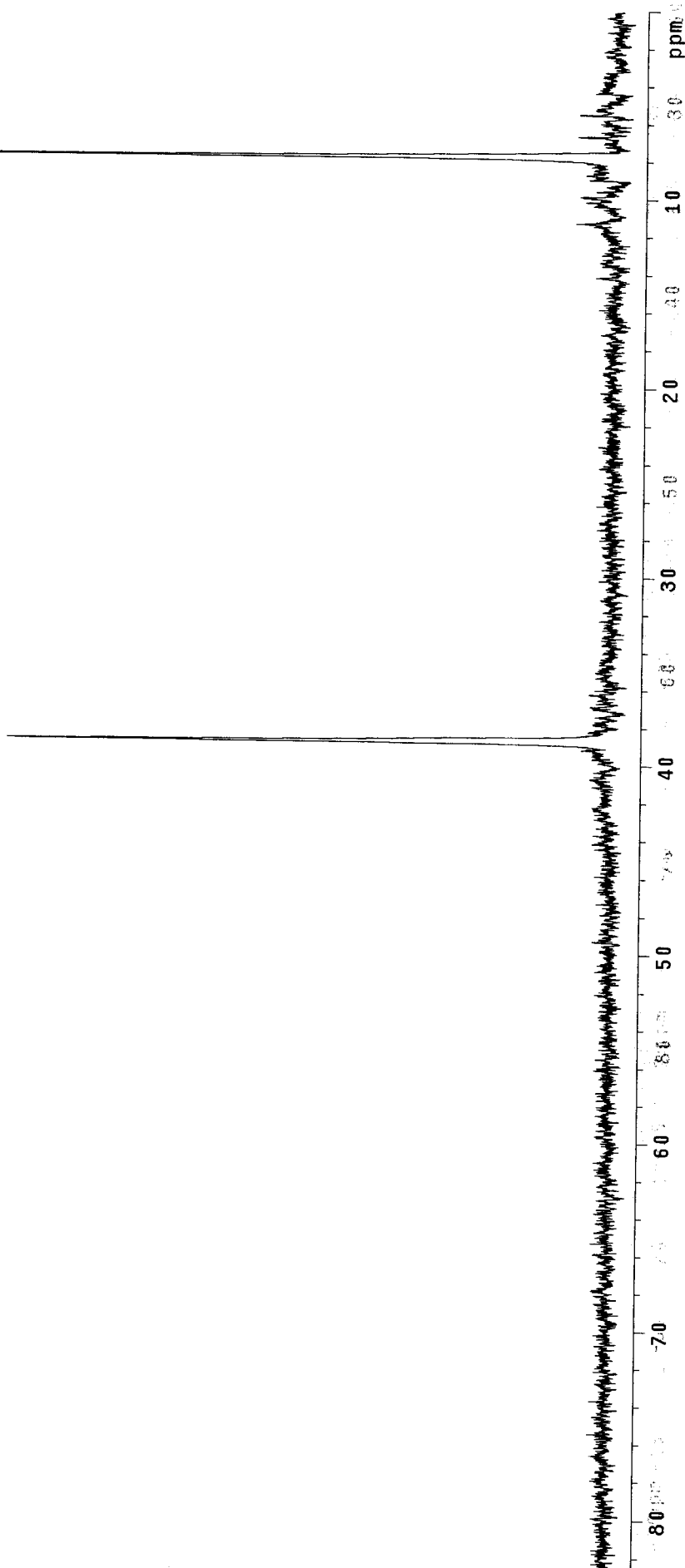
exp1 s2pu1

SAMPLE date May 4 2006
 solvent CDC13
 file
 ACQUISITION exp 26738.0
 at 1.598
 np 85476
 bs 14800
 ss 64
 d1 4
 d1 1.000
 nt 8
 ct 8
 tn TRANSMITTER P31
 sfrq 121.474
 tof 10608.2
 tpwr 55
 pw 7.117
 DECOUPLER H1
 dn 0
 dof VVY
 dm W
 dmm SC
 dpwr 35
 dimf 6700
 SPECIAL
 temp not used
 gain not used
 spin 20
 hst 0.008
 pw80 18.300
 aifa 20.000
 FLAGS
 ll n
 in n
 dp y
 hs mn
 hs 2.00
 lb not used
 fn DISPLAY
 sp 0
 wp 10010.0
 rfl 2437.3
 rfp 0
 rp 123.8
 lp -318.3
 PLOT
 wc 250
 sc 0
 vs 62
 th 9
 ai no ph



US:00

Table 4, entry 1a



| INDEX | FREQUENCY PPM | HEIGHT | INDEX | FREQUENCY PPM | HEIGHT |
|-------|---------------|--------|-------|---------------|--------|
| 1 | 4275.714 | 126.0 | 1 | 4275.714 | 35.203 |
| 2 | 4183.917 | 97.8 | 2 | 4183.917 | 34.446 |

31P NMR assay

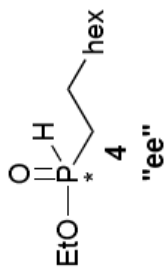
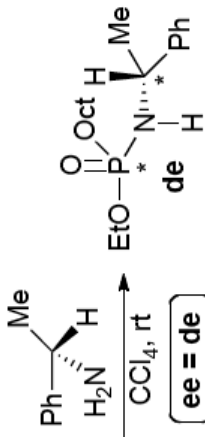
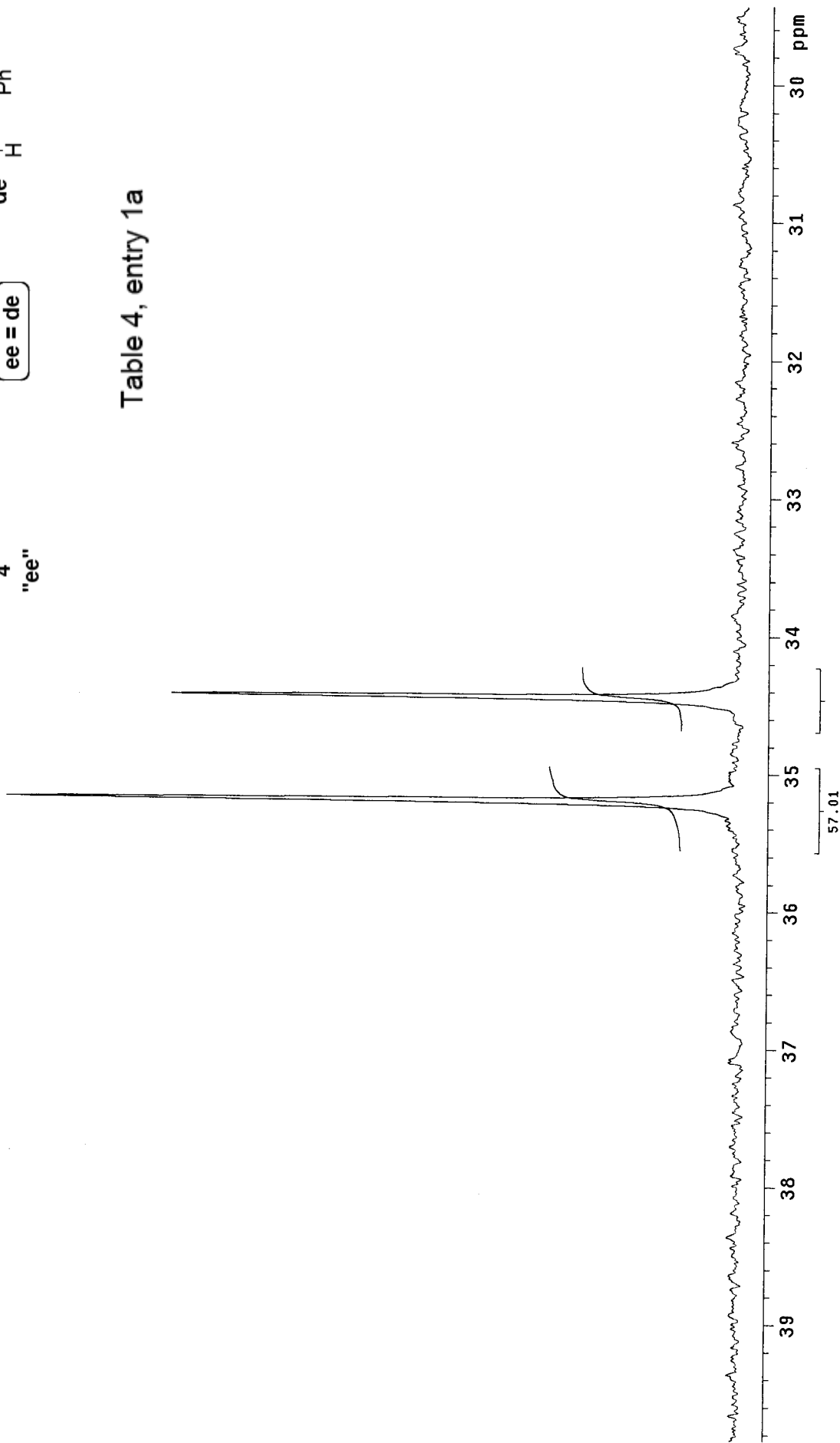


Table 4, entry 1a



976
exp1 s2pul

SAMPLE
 date May 31 2006
 solvent CDCl3
 file
 ACQUISITION exp 20
 sw 26738.0
 at 1.598
 np 85476
 fb 14800
 bs 64
 ss 4
 dl 1.000
 nt 8
 ct 8

SPECIAL
 temp not used
 gain not used
 sp in 20
 hst 0.008
 pw90 18.300
 alfa 20.000

FLAGS
 il n
 in n
 dp y
 hs nn
 lb 2.00
 fn not used

PROCESSING
 DISPLAY 10010.0
 2437.3
 0
 -42.0
 -233.9

TRANSMITTER P31
 sfrq 121.474
 tof 10608.2
 tpwr 7.117

DECOUPLER H1
 dn 0
 dof VVY
 dm W
 dmm SC
 dpwr 35
 dmf 6700

al no ph
 250
 10
 3

976
exp1 s2pul

SAMPLE
 date May 31 2006
 solvent CDCl3
 file
 ACQUISITION exp 20
 sw 26738.0
 at 1.598
 np 85476
 fb 14800
 bs 64
 ss 4
 dl 1.000
 nt 8
 ct 8

SPECIAL
 temp not used
 gain not used
 sp in 20
 hst 0.008
 pw90 18.300
 alfa 20.000

FLAGS
 il n
 in n
 dp y
 hs nn
 lb 2.00
 fn not used

PROCESSING
 DISPLAY 10010.0
 2437.3
 0
 -42.0
 -233.9

TRANSMITTER P31
 sfrq 121.474
 tof 10608.2
 tpwr 7.117

DECOUPLER H1
 dn 0
 dof VVY
 dm W
 dmm SC
 dpwr 35
 dmf 6700

al no ph
 250
 10
 3

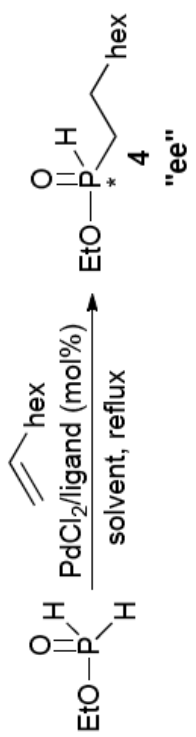
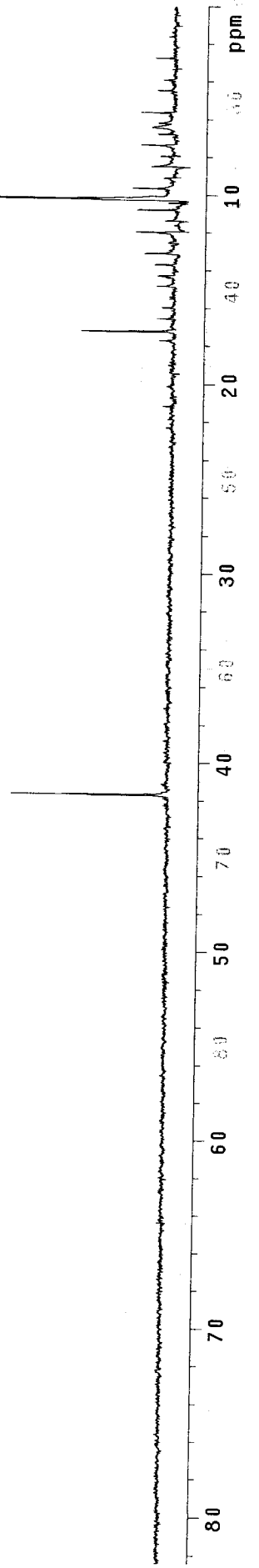
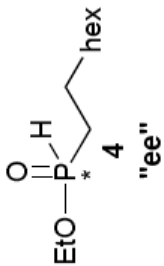


Table 4, entry 2b



| INDEX | FREQUENCY PPM | HEIGHT | INDEX | FREQUENCY PPM | HEIGHT |
|-------|---------------|--------|-------|---------------|--------|
| 1 | 4433.606 | -7.4 | 1 | 4433.606 | 35.51 |
| 2 | 4326.713 | 119.4 | 2 | 4326.713 | 35.62 |
| 3 | 4271.226 | -5.3 | 3 | 4271.226 | 35.16 |
| 4 | 4236.139 | 84.3 | 4 | 4236.139 | 34.67 |
| 5 | 4174.533 | -7.8 | 5 | 4174.533 | 34.36 |



³¹P NMR assay

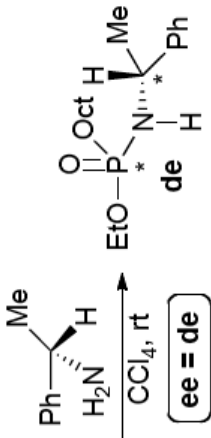
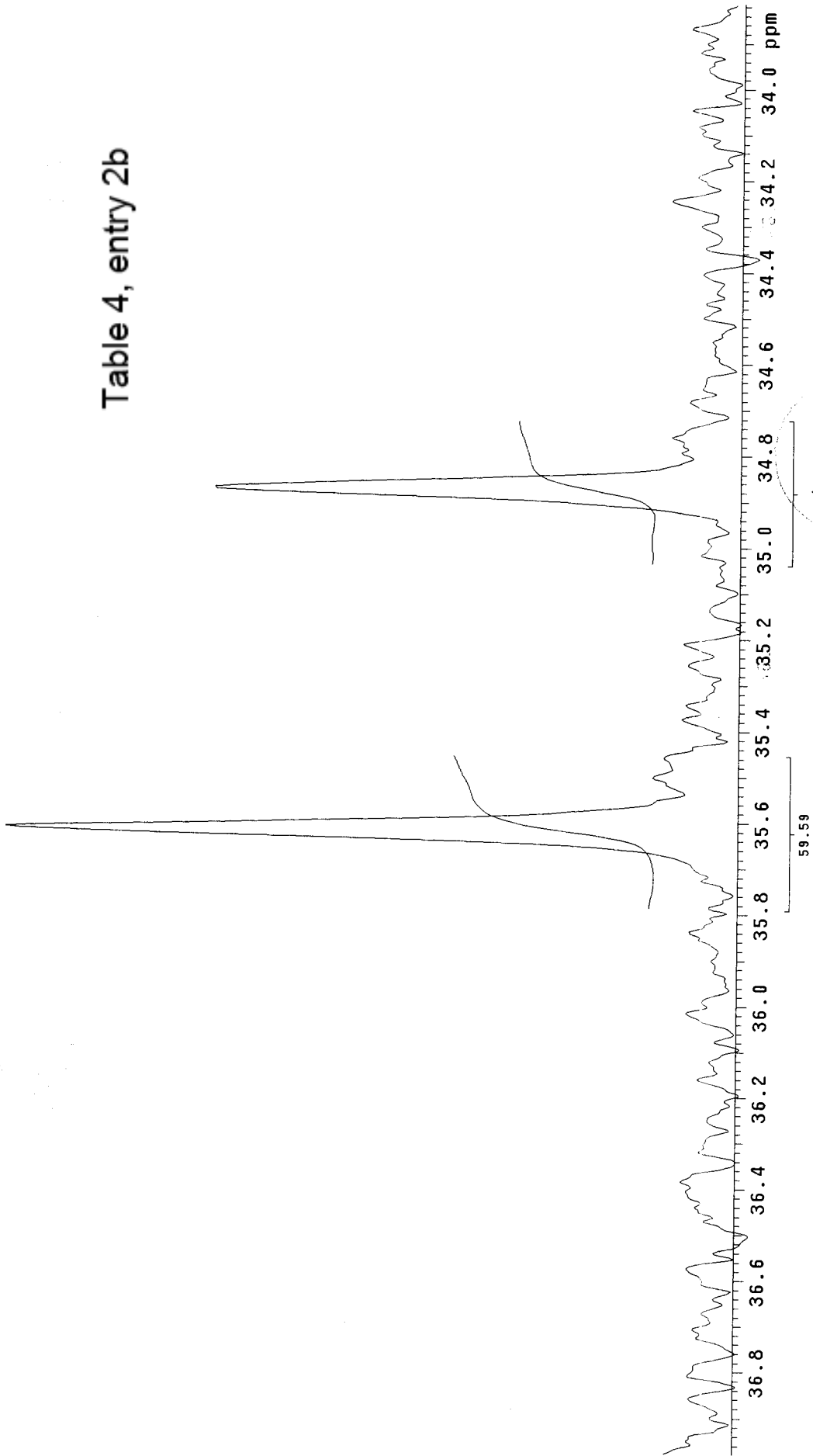


Table 4, entry 2b



191

973a

exp1 s2pu1

SAMPLE date May 31 2006 temp not used
 solvent CDC13 gain not used
 file ACQUISITION exp 20
 26738.0 hst 0.008
 1.598 pw80 18.300
 85476 atfa 20.000
 14800 il n
 64 in n
 4 dp y
 1.000 hs nm
 8 ct PROCESSING 2.00
 8 lb fn not used
 TRANSMITTER p31 DISPLAY
 sfrq 121.474 sp 10010.0
 tof 10608.2 wp 2437.3
 tpwr 55 rfp 110.6
 pw 7.117 rp -274.6
 DECOUPLER H1 lp PLOT
 dof 0 YVY WC 250
 dm W SC 0
 dmm W SC 15
 dpwr 35 vs
 dmf 6700 th 3
 at no ph

exp1 s2pu1

SAMPLE date May 31 2006 temp not used
 solvent CDC13 gain not used
 file ACQUISITION exp 20
 26738.0 hst 0.008
 1.598 pw80 18.300
 85476 atfa 20.000
 14800 il n
 64 in n
 4 dp y
 1.000 hs nm
 8 ct PROCESSING 2.00
 TRANSMITTER p31 DISPLAY
 sfrq 121.474 sp 10010.0
 tof 10608.2 wp 2437.3
 tpwr 55 rfp 110.6
 pw 7.117 rp -274.6
 DECOUPLER H1 lp PLOT
 dof 0 YVY WC 250
 dm W SC 0
 dmm W SC 15
 dpwr 35 vs
 dmf 6700 th 3
 at no ph

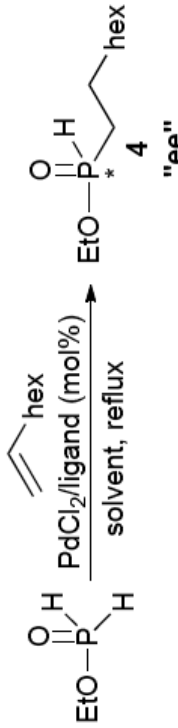
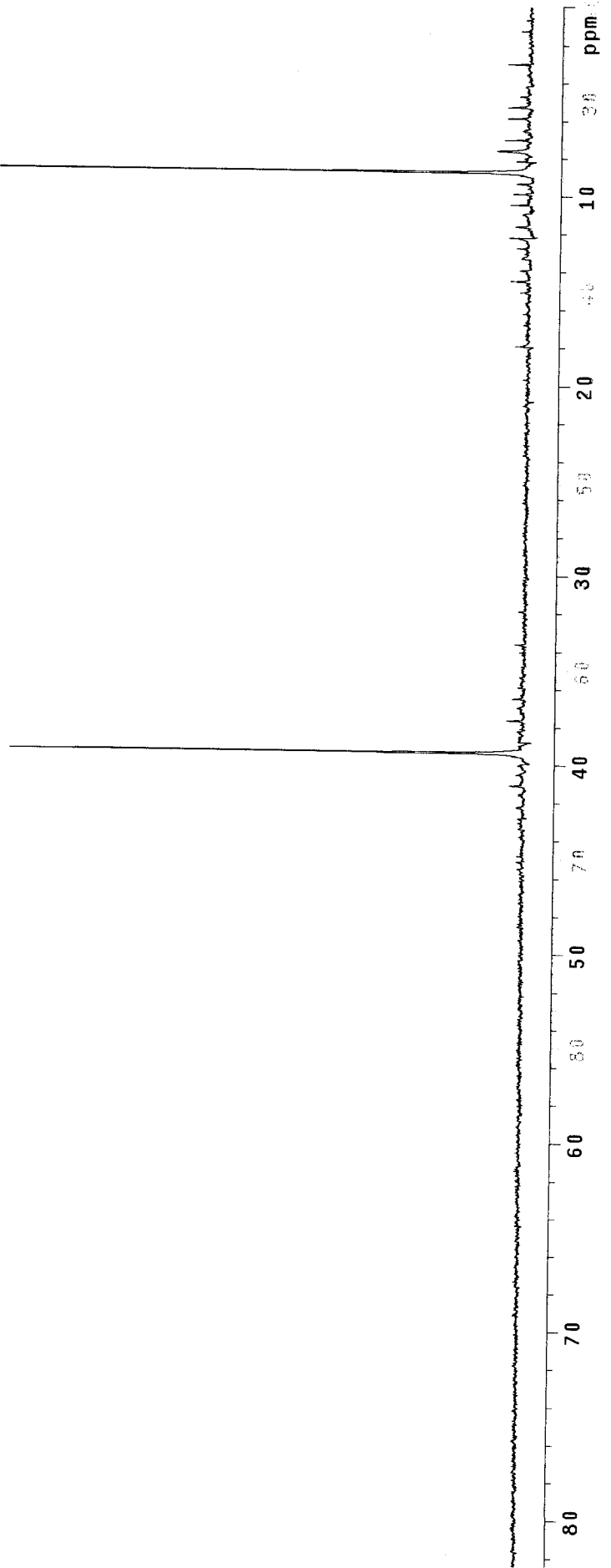


Table 4, entry 6



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 4358.352 | 35.890 | 54.2 |
| 2 | 4275.714 | 35.202 | 45.4 |

| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 4358.352 | 35.890 | 54.2 |
| 2 | 4275.714 | 35.202 | 45.4 |

| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 4358.352 | 35.890 | 54.2 |
| 2 | 4275.714 | 35.202 | 45.4 |

³¹P NMR assay

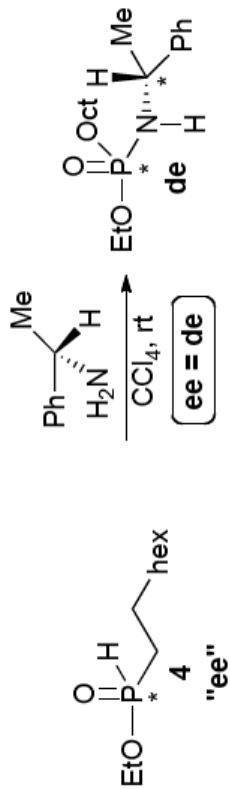
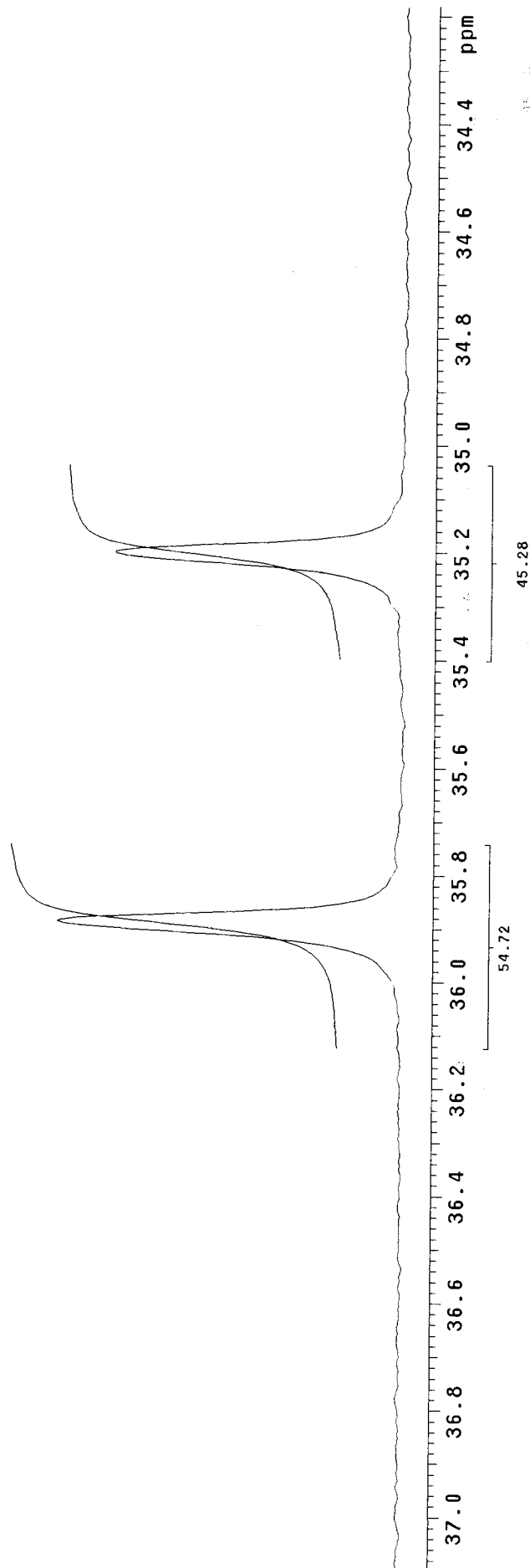


Table 4, entry 6



| INDEX | FREQUENCY | PPM | HEIGHT | INDEX | FREQUENCY | PPM | HEIGHT | INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|-------|-----------|--------|--------|-------|-----------|--------|--------|
| 1 | 4900.345 | 40.344 | 34.1 | 1 | 9900.345 | 40.344 | 34.1 | 1 | 9900.345 | 40.344 | 34.1 |
| 2 | 1089.728 | 8.972 | 126.0 | 2 | 1089.728 | 8.972 | 126.0 | 2 | 1089.728 | 8.972 | 126.0 |

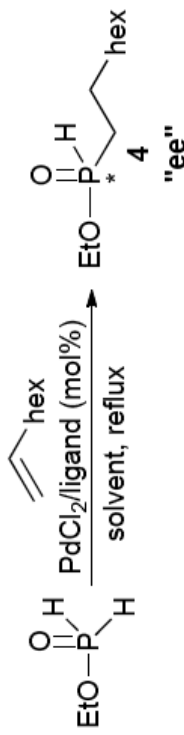
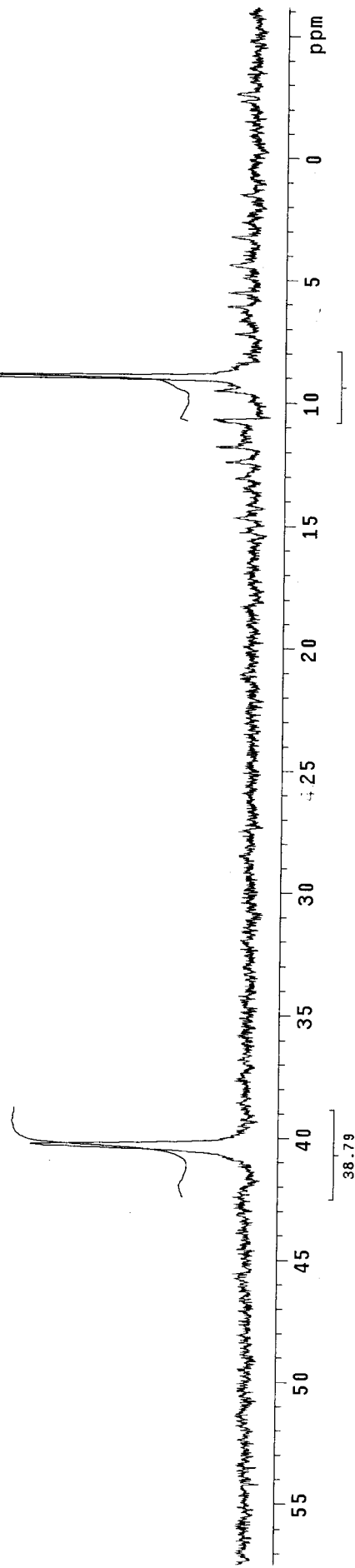


Table 4, entry 8a



38.79

61.21

arsay

973b

expt s2pu1

SAMPLE

date Jun 2 2006
 solvent CDCl3
 file

ACQUISITION exp

sw 26738.0
 at 1.598
 np 85476
 fb 14800
 bs 64
 ss 4
 d1 1.000
 nt 16
 ct 16

TRANSMITTER

tn P81
 sfrq 121.474
 tof 10608.2
 tpwr 55
 pw 7.117

DECOUPLER

dn H1
 dof 0
 dm YVY
 dmm W
 dpwr 35
 dmf 6700

SPECIAL

temp not used
 gain not used
 spin 20
 hst 0.008
 pw90 18.300
 alfa 20.000

FLAGS

il n
 in n
 dp y
 hs mn
 PROCESSING 2.00
 not used

DISPLAY

sp 0
 wp 10010.0
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 rfp 0
 lp 104.6
 tp -268.8

PLOT

wc 250
 sc 0
 vs 18
 th 2
 ai no ph

31P NMR assay

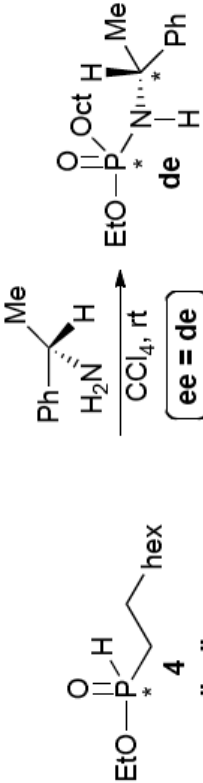
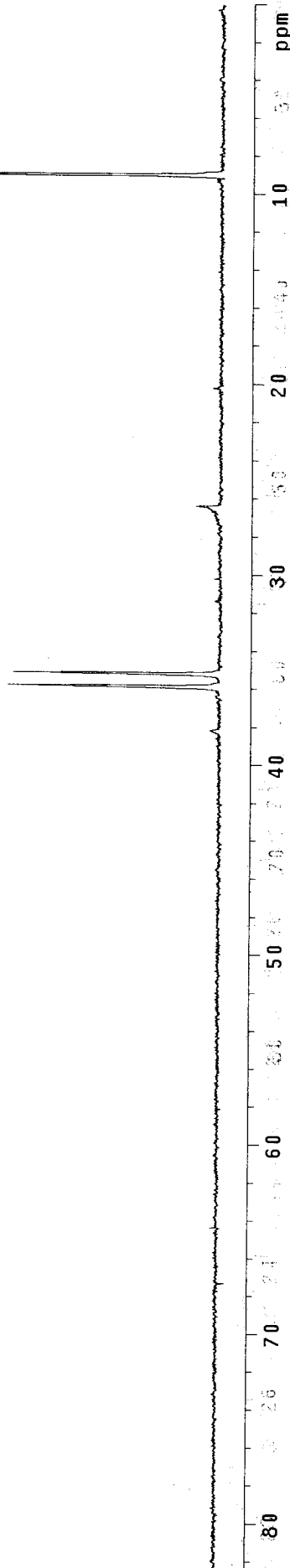


Table 4, entry 8a



| INDEX | FREQUENCY | PPM | HEIGHT | INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|-------|-----------|--------|--------|
| 1 | 4353.232 | 35.840 | 33.1 | 1 | 4353.232 | 35.840 | 33.1 |
| 2 | 4271.226 | 35.165 | 32.2 | 2 | 4271.226 | 35.165 | 32.2 |

³¹P NMR assay

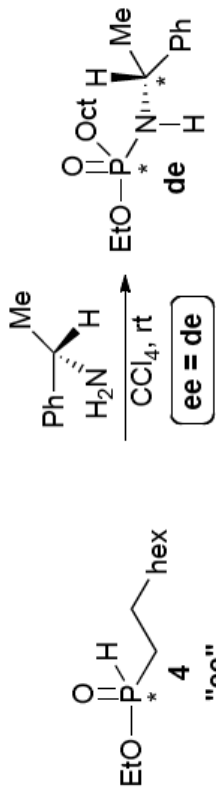
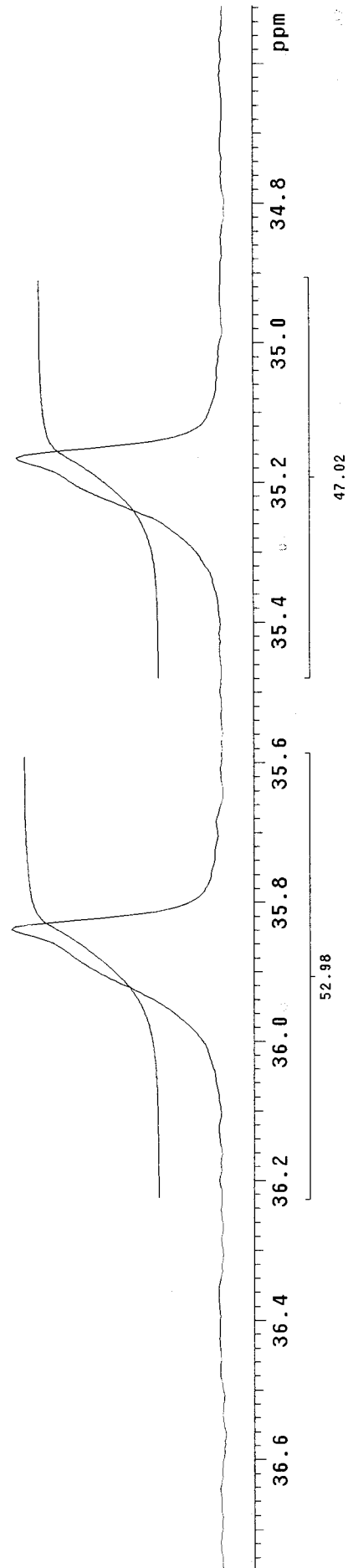
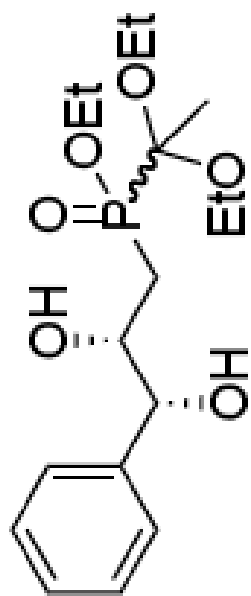


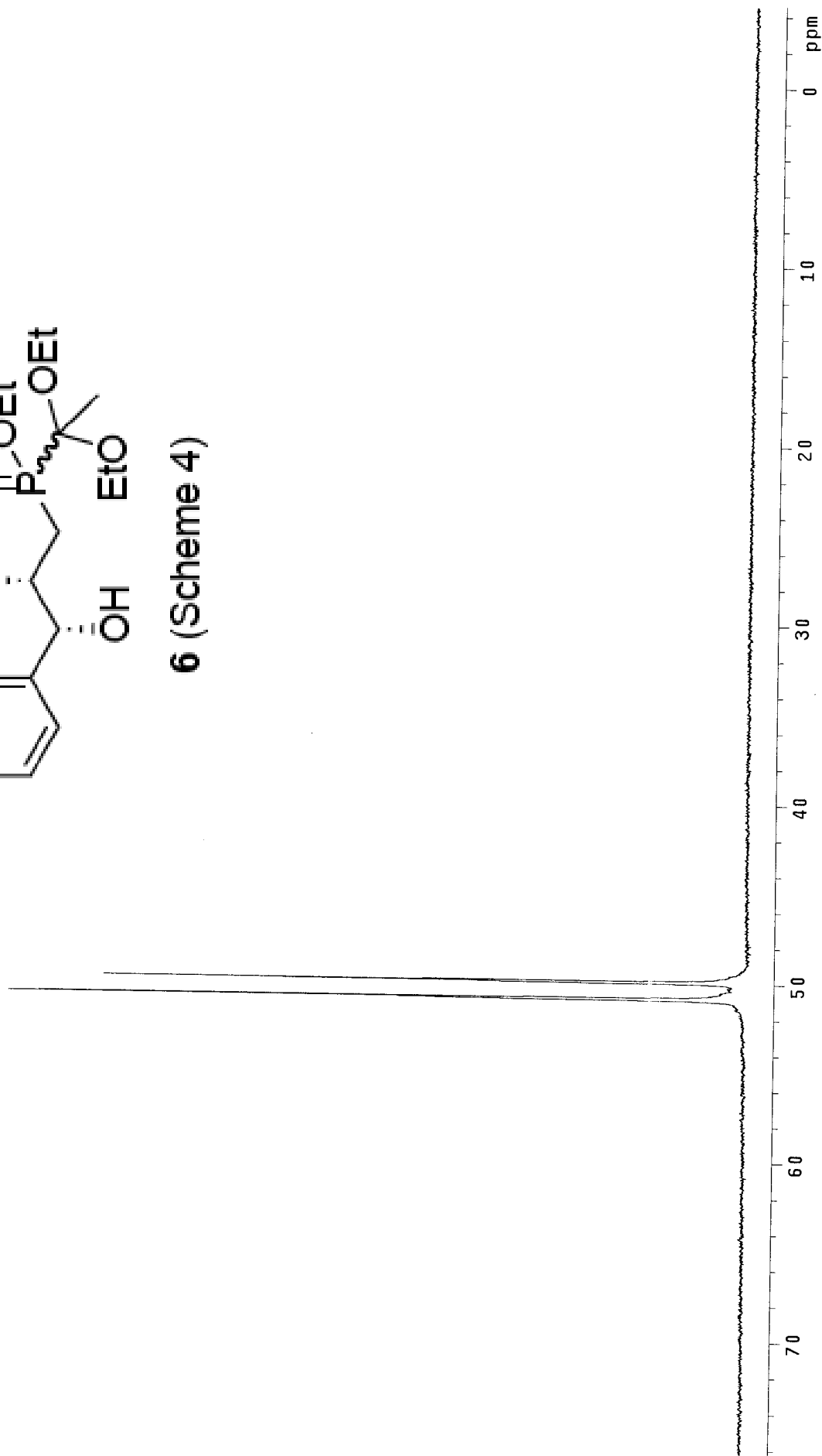
Table 4, entry 8a



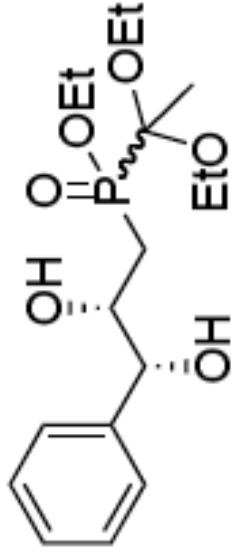
| INDEX | FREQUENCY PPM | HEIGHT |
|-------|---------------|--------|
| 1 | 6171.639 | 125.0 |
| 2 | 6056.994 | 108.9 |



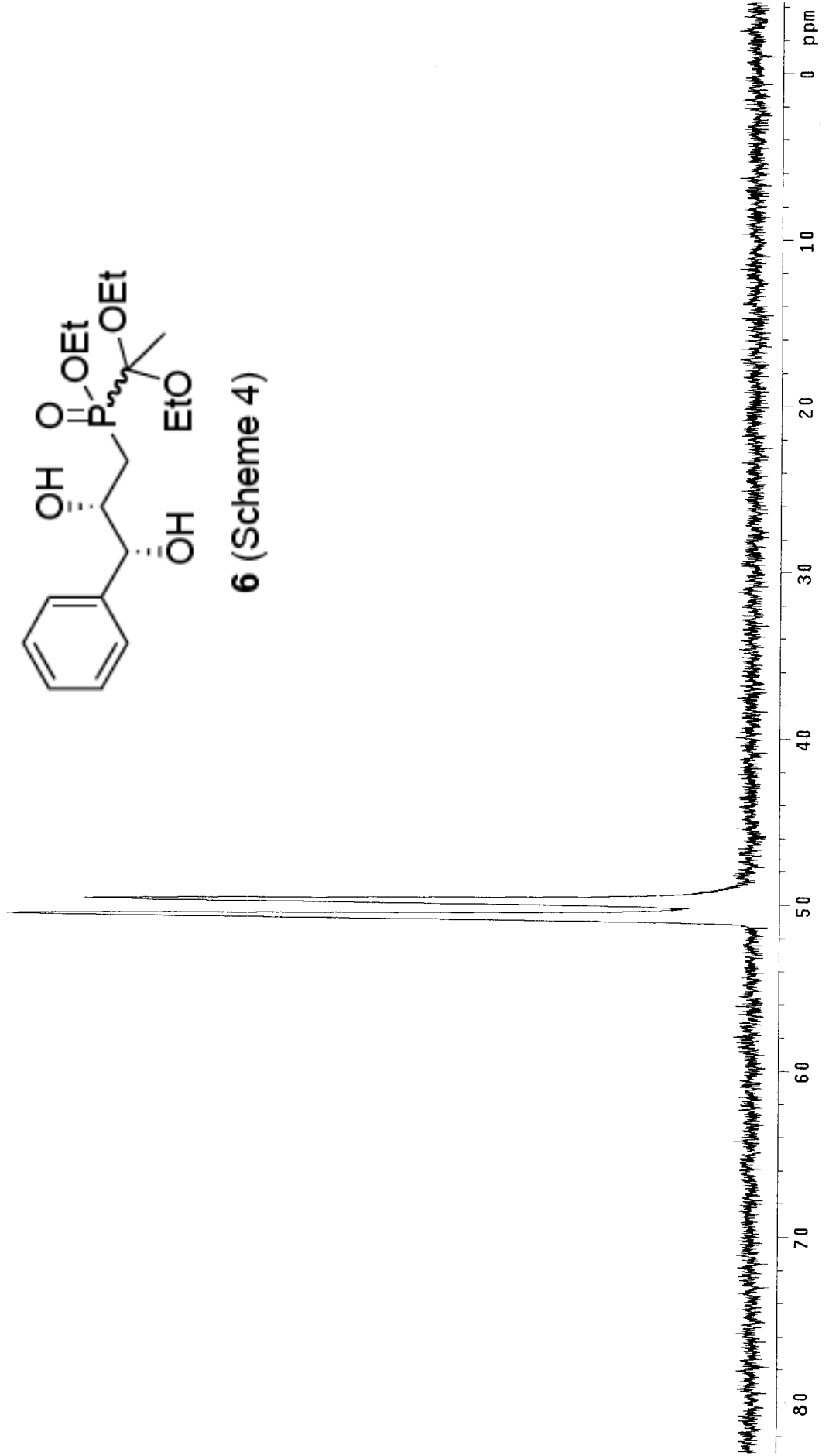
6 (Scheme 4)

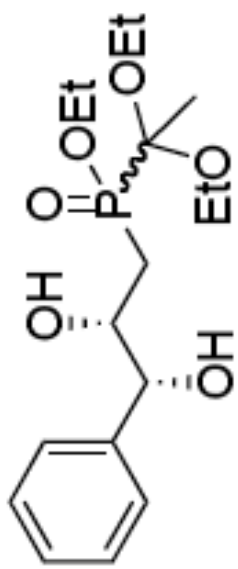


| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 6172.455 | 50.818 | 126.0 |
| 2 | 6056.178 | 49.860 | 113.0 |

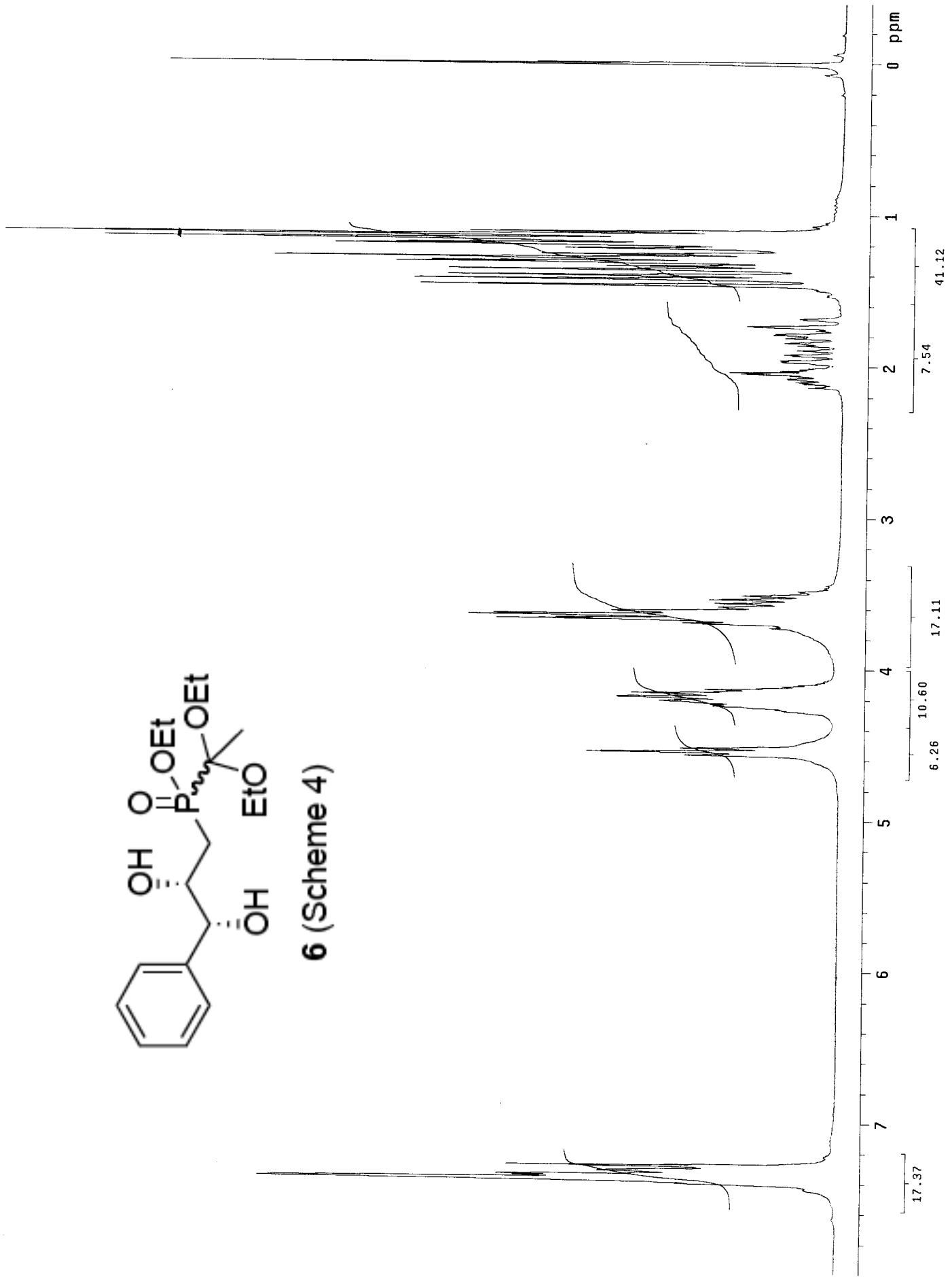


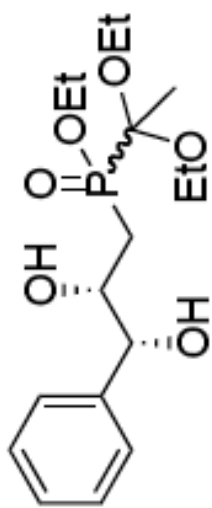
6 (Scheme 4)



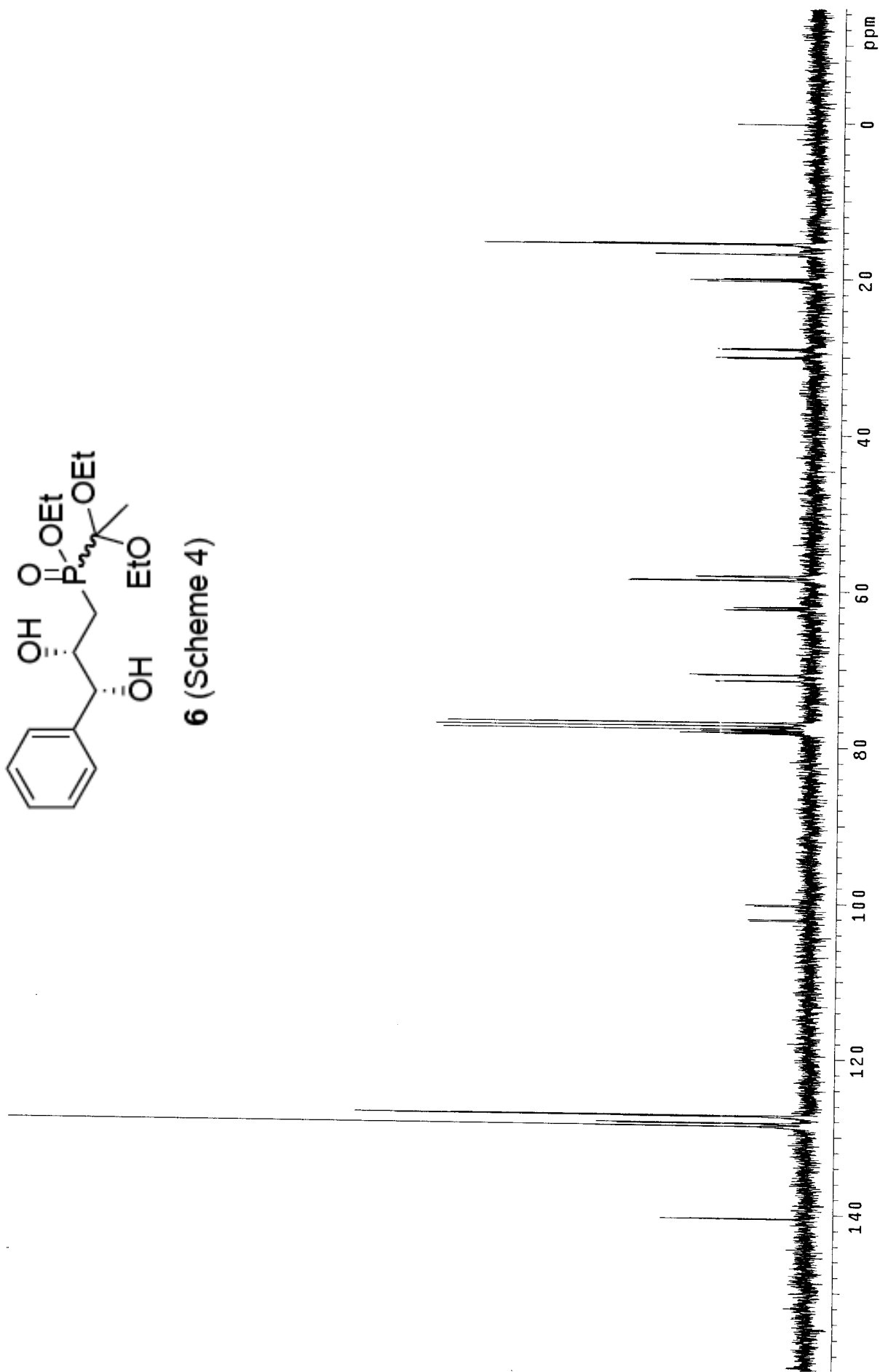


6 (Scheme 4)

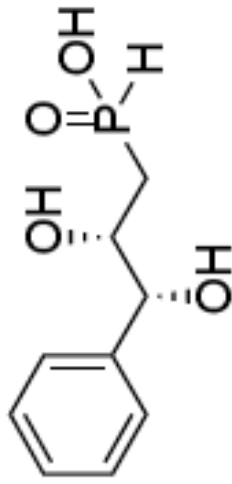




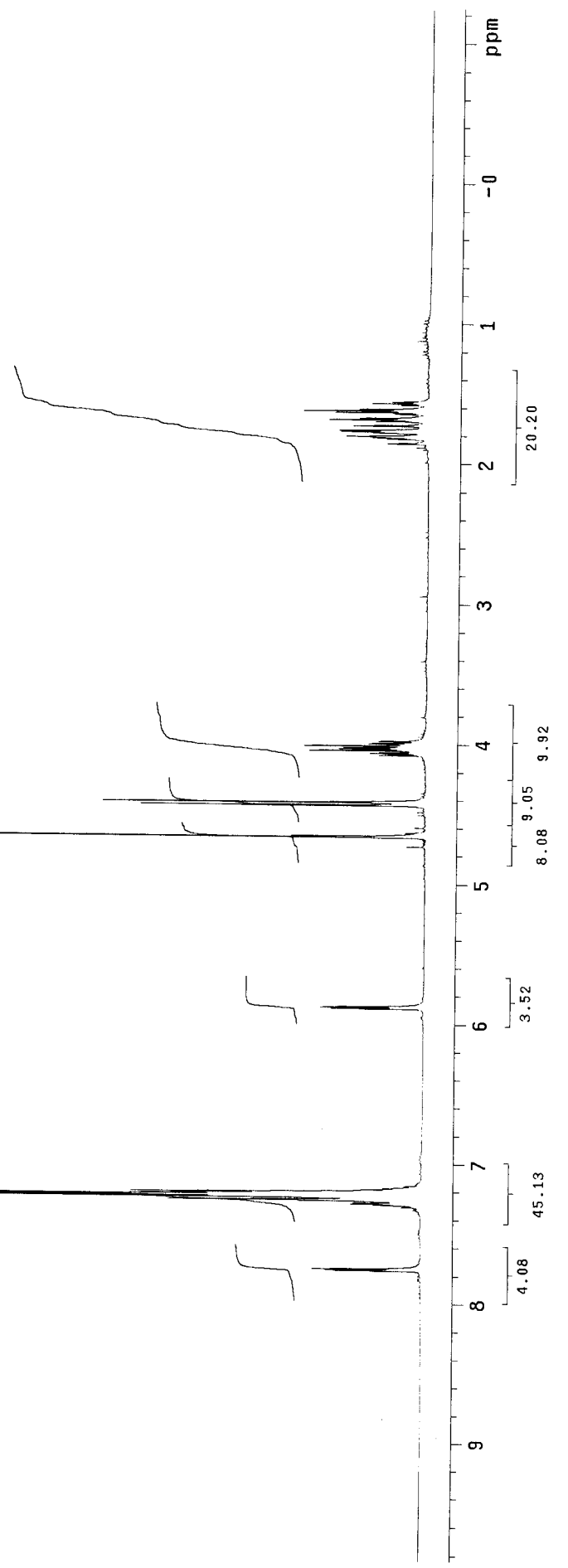
6 (Scheme 4)

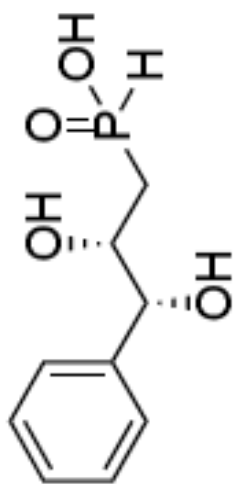


| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|-------|--------|
| 1 | 2325.950 | 7.752 | 17.1 |
| 2 | 2179.958 | 7.265 | 19.4 |
| 3 | 2178.492 | 7.260 | 39.9 |
| 4 | 2176.733 | 7.255 | 26.7 |
| 5 | 2172.629 | 7.241 | 101.4 |
| 6 | 2168.818 | 7.228 | 162.0 |
| 7 | 2165.007 | 7.215 | 44.1 |
| 8 | 2161.782 | 7.205 | 45.7 |
| 9 | 1398.989 | 4.662 | 124.8 |
| 10 | 1396.644 | 4.655 | 21.1 |
| 11 | 1330.977 | 4.436 | 44.8 |
| 12 | 1324.821 | 4.415 | 50.8 |
| 13 | 1213.128 | 4.043 | 18.4 |
| 14 | 1202.867 | 4.009 | 19.2 |
| 15 | 485.807 | 1.619 | 19.8 |

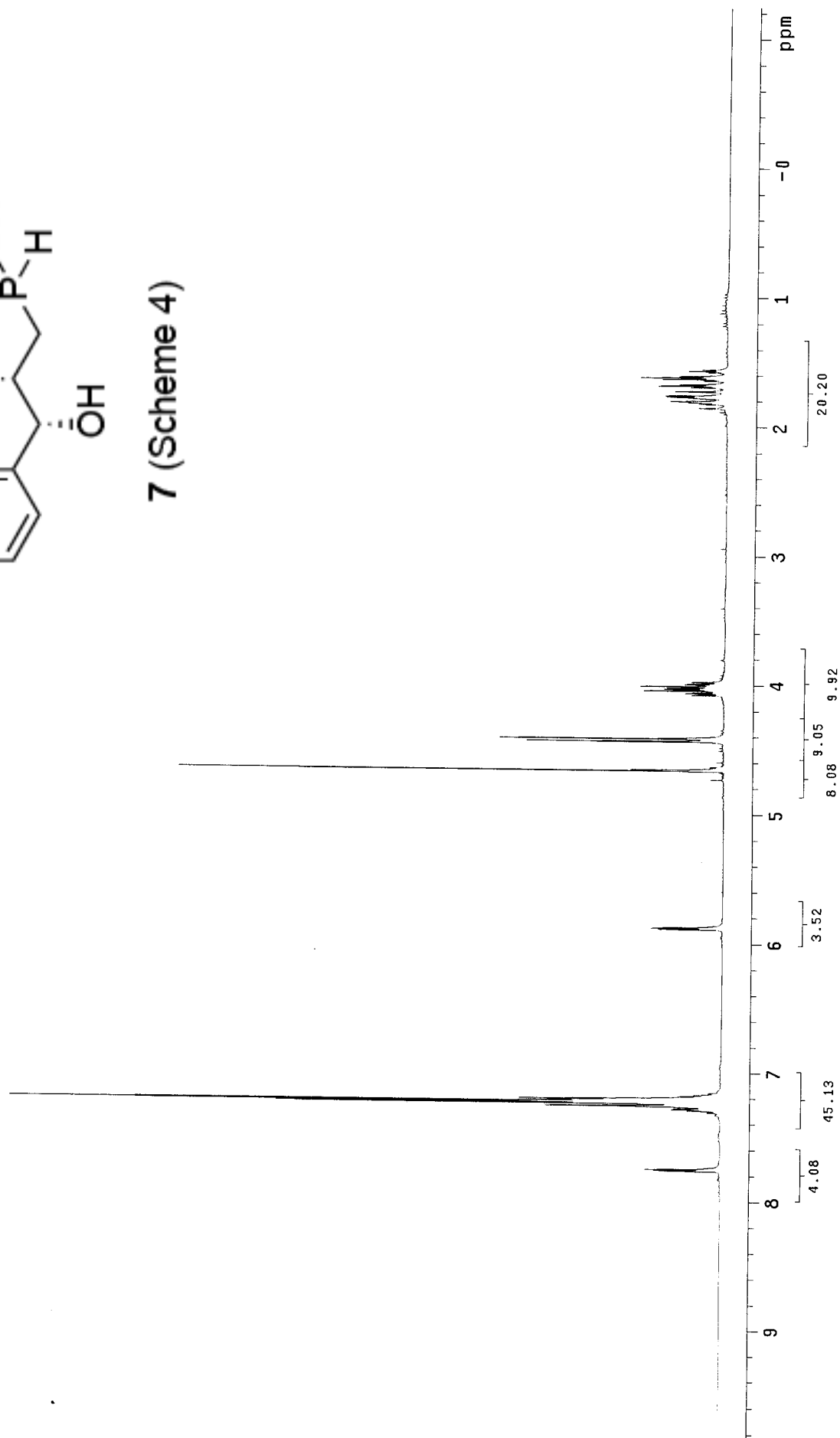


7 (Scheme 4)

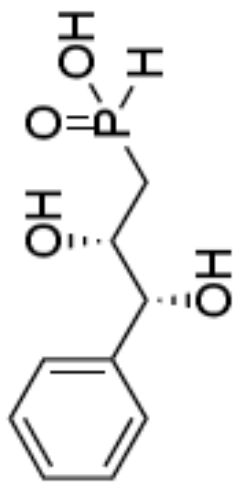




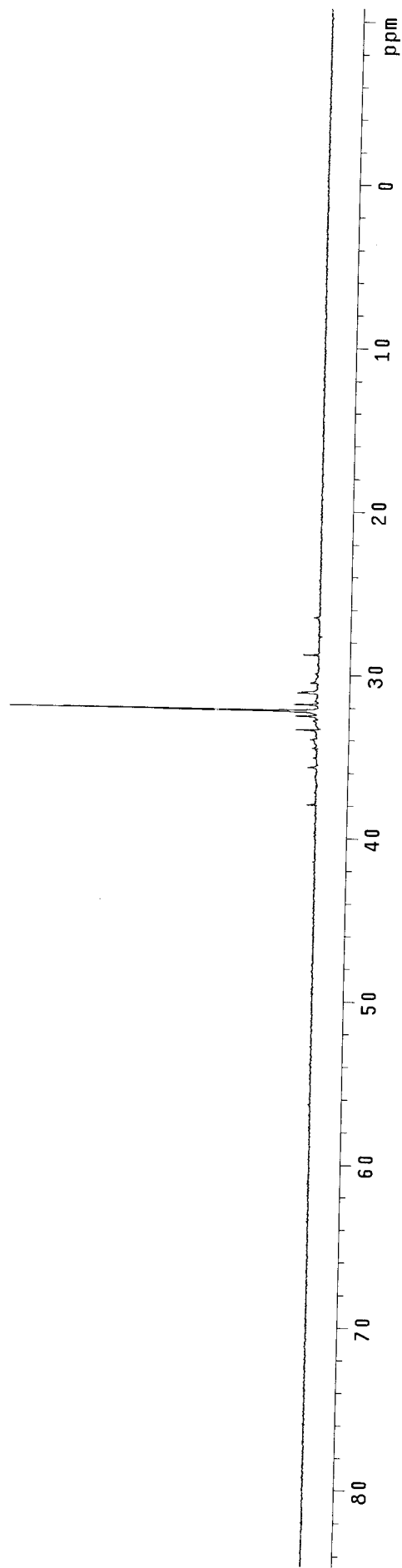
7 (Scheme 4)



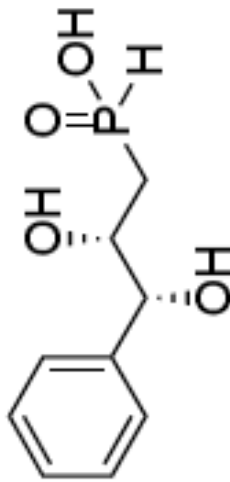
INDEX
1 FREQUENCY PPM HEIGHT
3913.451 32.219 48.5



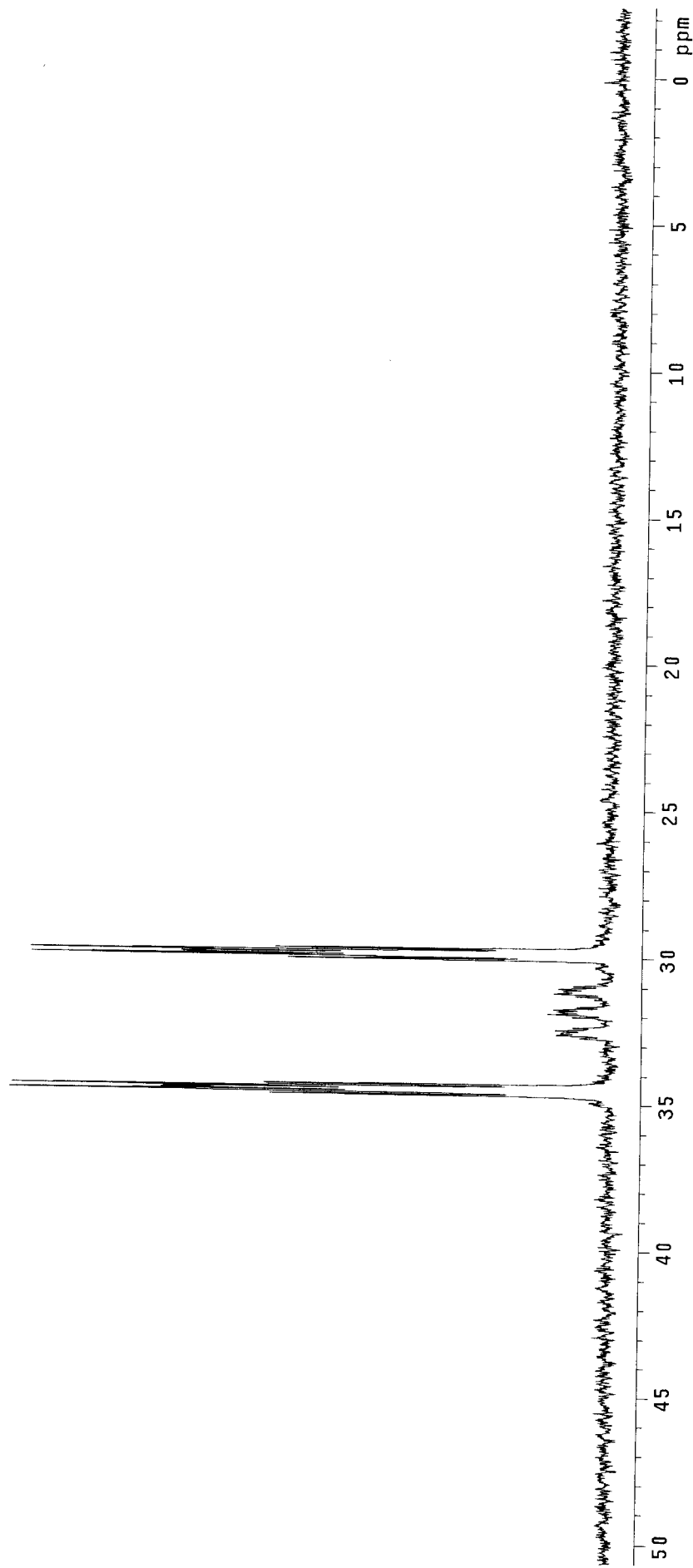
7 (Scheme 4)

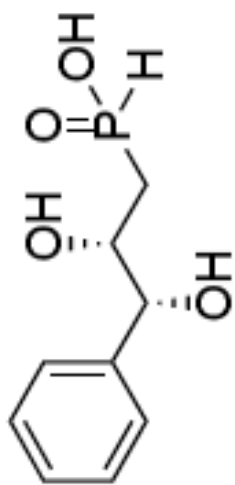


| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 4216.587 | 34.715 | 53.3 |
| 2 | 4205.163 | 34.621 | 94.6 |
| 3 | 4197.003 | 34.554 | 70.6 |
| 4 | 4193.739 | 34.527 | 70.2 |
| 5 | 4185.987 | 34.463 | 94.2 |
| 6 | 4174.156 | 34.366 | 54.3 |
| 7 | 3653.970 | 30.083 | 50.7 |
| 8 | 3642.546 | 29.989 | 91.2 |
| 9 | 3631.122 | 29.895 | 67.5 |
| 10 | 3623.371 | 29.831 | 91.5 |
| 11 | 3611.539 | 29.734 | 52.7 |

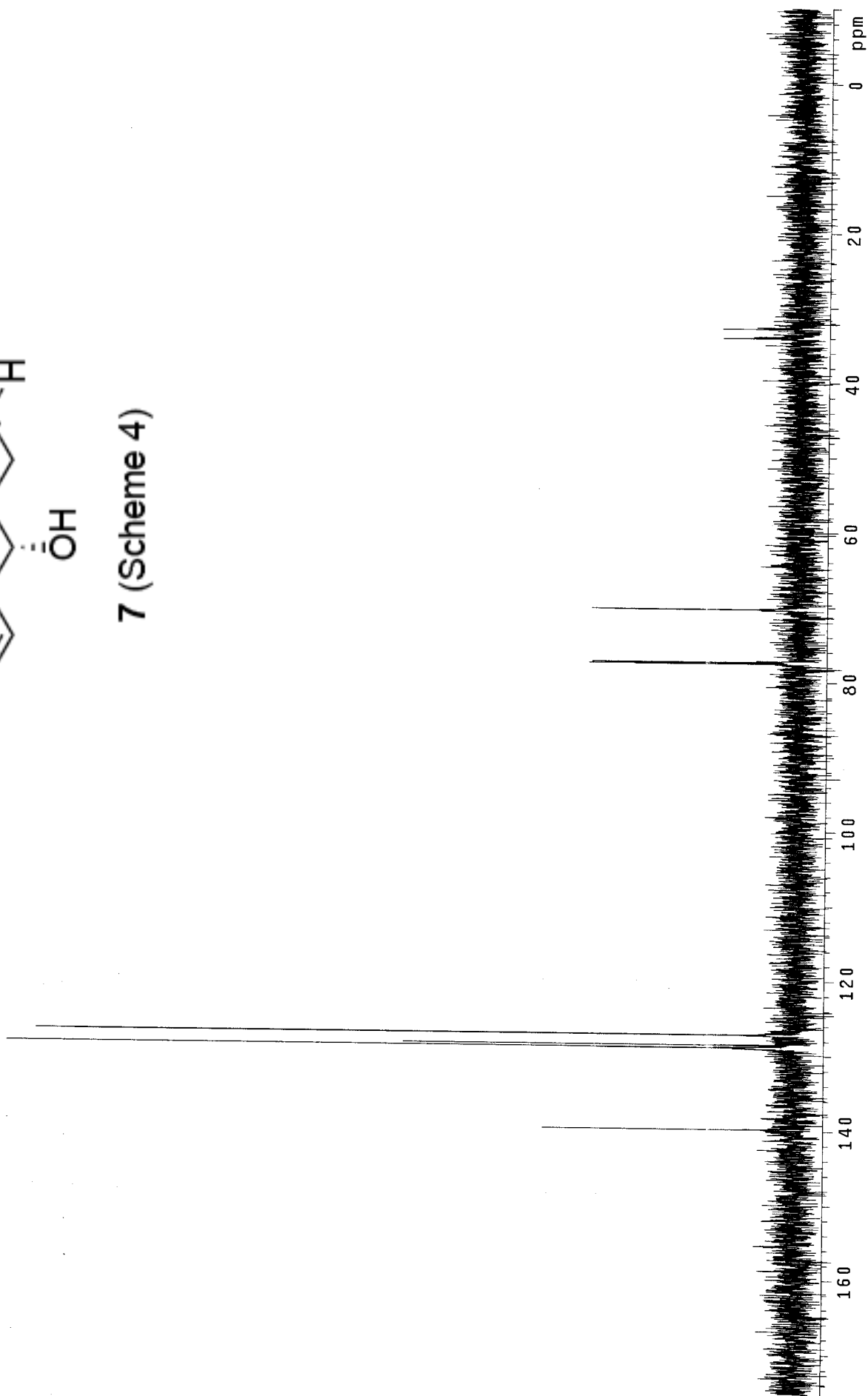


7 (Scheme 4)





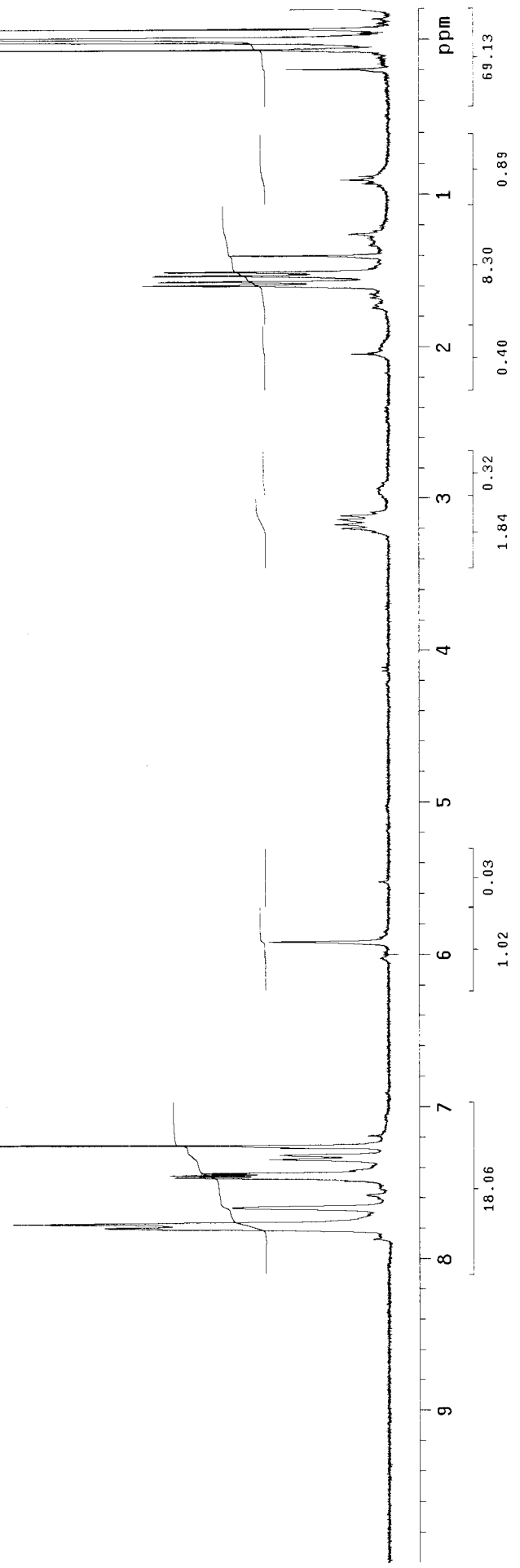
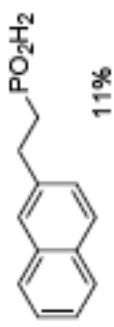
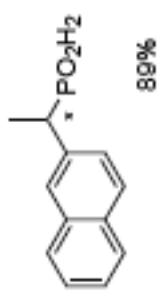
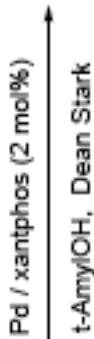
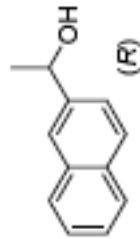
7 (Scheme 4)

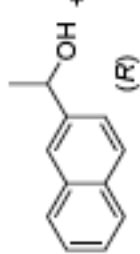


1c915b

exp1 s2pu1

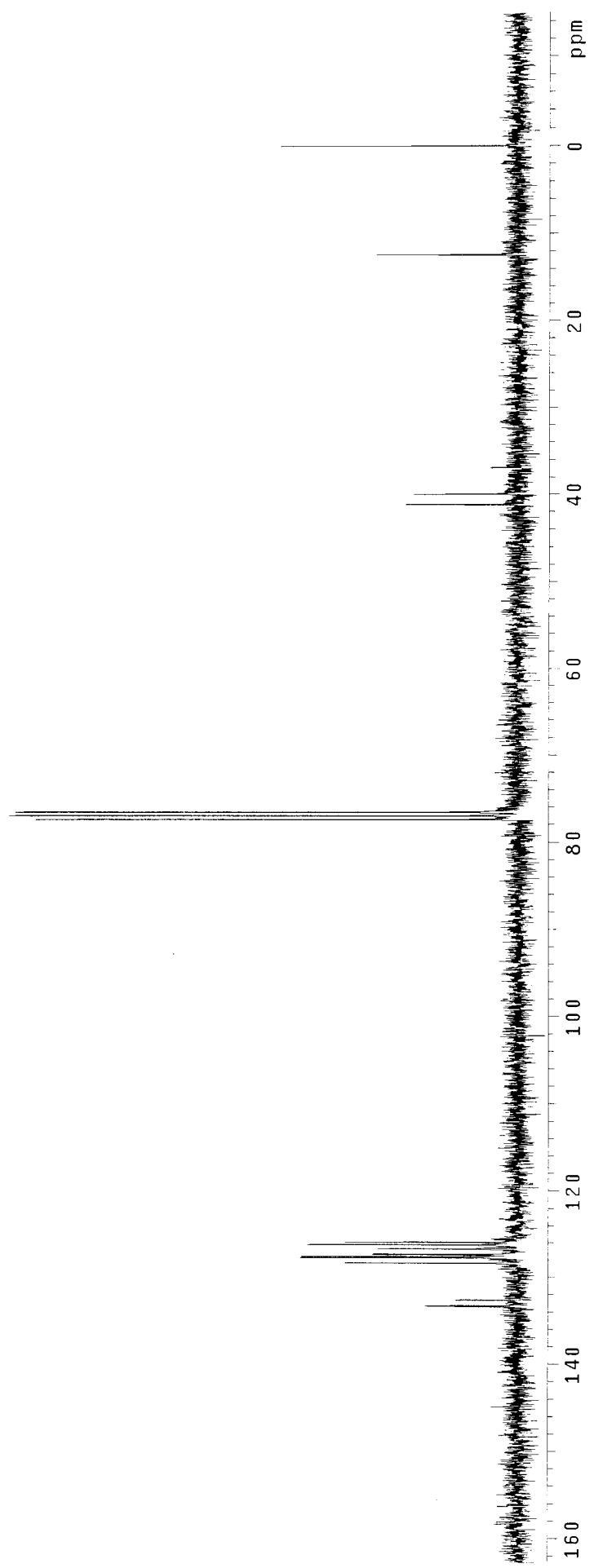
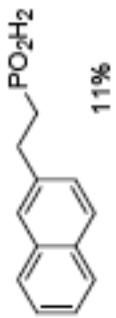
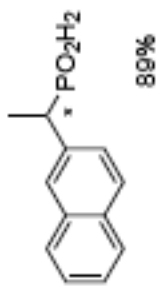
SAMPLE date Jan 17 2008 temp not
 solvent cdc13 gain not
 file ACQUISITION exp hst (1)
 sw 4803.1 pw90 (2)
 at 1.994 aifa FLAGS
 np not used il
 fb 16 in
 bs 4 dp
 ss 1.000 hs
 d1 16 fn not used
 nt 16
 Ct TRANSMITTER HI sp DISPLAY -60.0
 tn 300.053 wp 3060.5
 sfrq 300.9 rf1 601.6
 tof 55 rfp 0
 tpwr 8.600 rp -100.4
 pw DECOUPLER C13 lp -77.8
 dn PLOT
 dof 250
 dm nnn SC 0
 dmm C VS 947
 dpwr 45 th 3
 dmf 13100 ai cdc ph



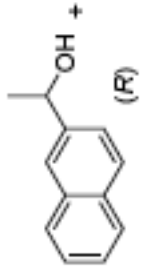


concentrated H_3PO_2

Pd / xantphos (2 mol%)
t-AmylOH, Dean Stark



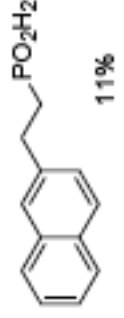
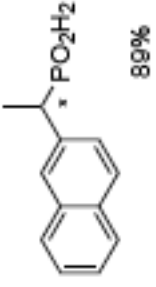
| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 5185.530 | 42.692 | 150.7 |
| 2 | 4759.181 | 39.182 | 14.2 |
| 3 | 3888.941 | 32.018 | -11.7 |
| 4 | 3659.243 | 30.126 | -11.6 |
| 5 | 3538.886 | 29.136 | -17.9 |
| 6 | 677.659 | 5.579 | 13.7 |



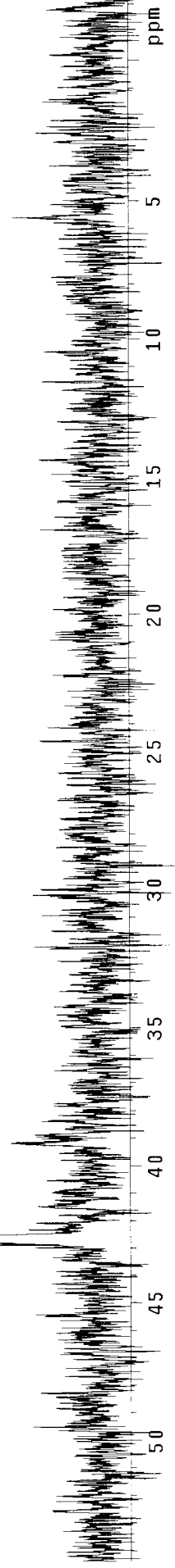
concentrated H_3PO_2

Pd / xantphos (2 mol%)

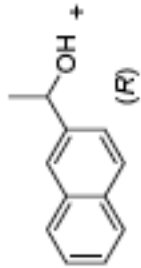
t-AmylOH, Dean Stark



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

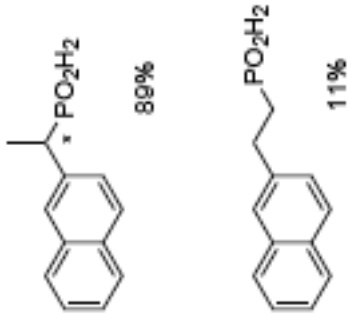


| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|---------|--------|
| 1 | 3702.490 | 30.7483 | 15.7 |
| 2 | 3674.338 | 30.251 | 125.7 |
| 3 | 3113.761 | 25.656 | 14.0 |

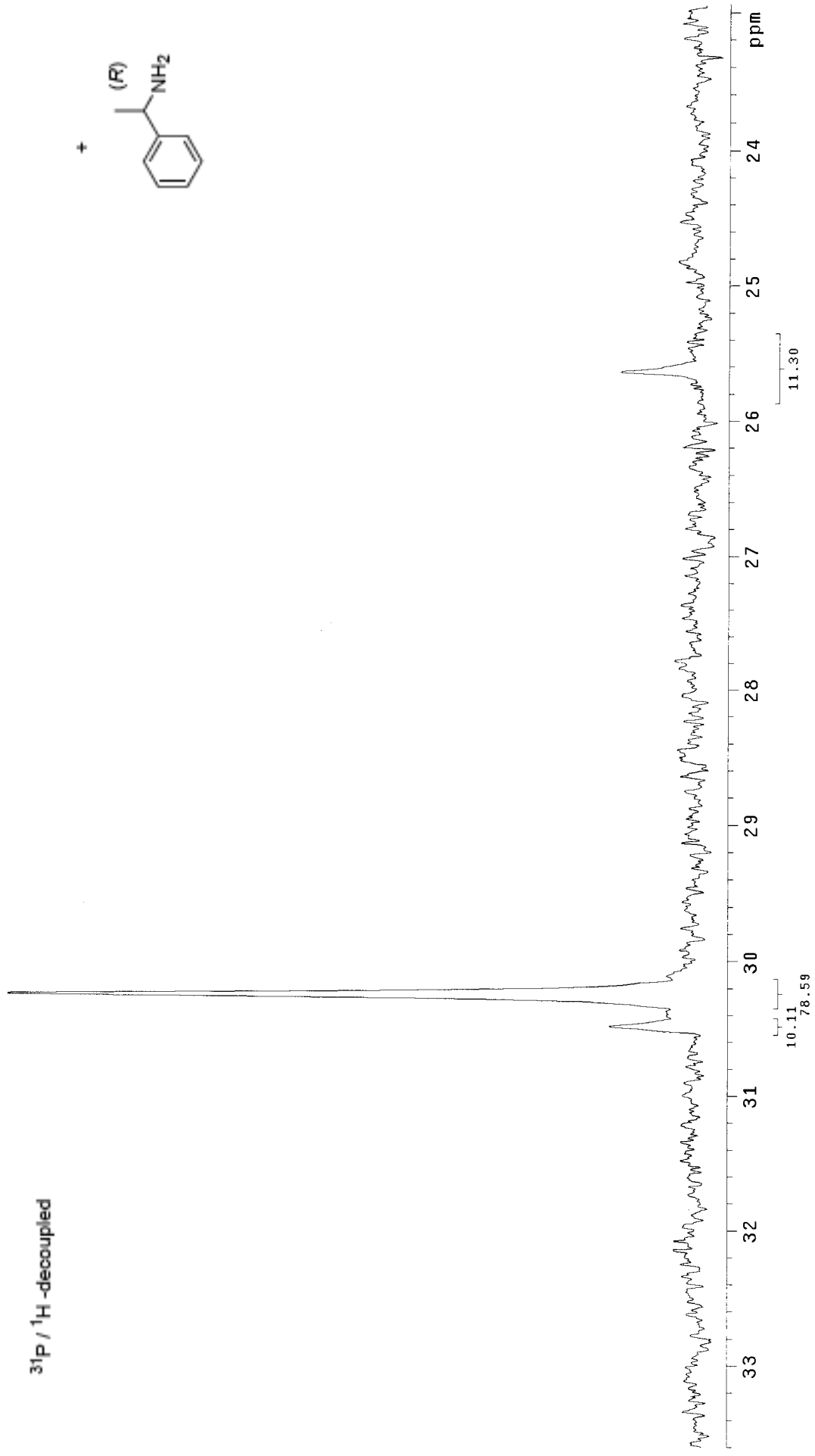


concentrated H₃PO₂

Pd / xantphos (2 mol%)
t-AmylOH, Dean Stark



Enantiomeric excess determination



1c918

exp1 s2pu1

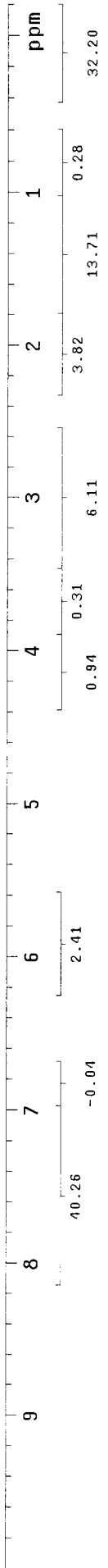
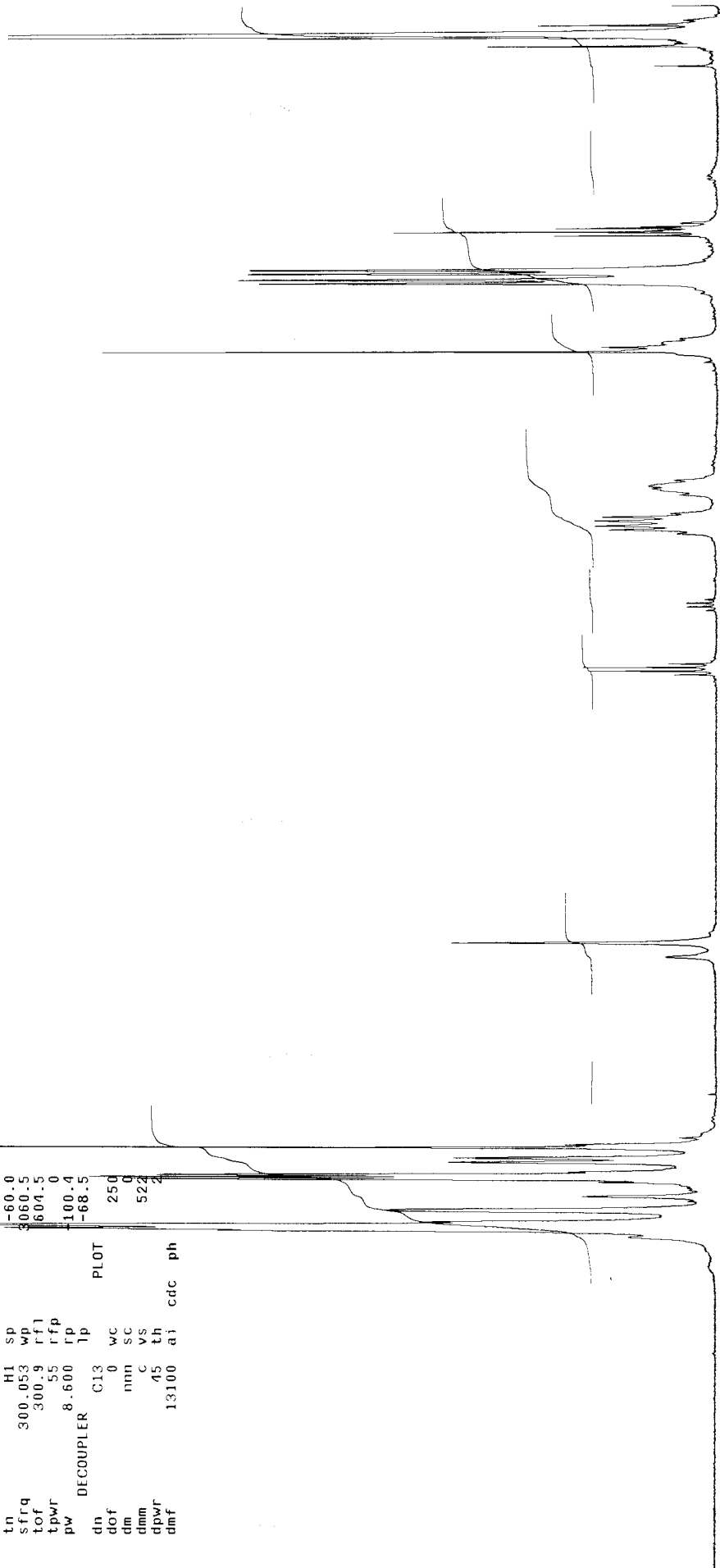
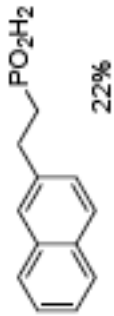
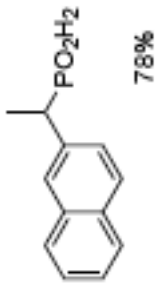
SAMPLE date Jan 18 2008
 solvent CDC13
 file exp
 ACQUISITION 4803.1
 at 1.994
 np 19158
 rb not used
 bs 16
 ss 4
 d1 1.000
 nt 16
 ct 16
 TRANSMITTER H1 SP
 tn 300.053 wp
 sfrq 300.9 rfl
 tof 55 rfp
 tpwr 8.600 rp
 pw DECOUPLER C13
 dn 0 wc
 dof nnn SC
 dm 522 C VS
 dnm 45 th
 dpwr 13100 a1 cdc ph
 dmf

SPECIAL not used
 not used
 gain 20
 spin 0.008
 hst 17.200
 pw90 20.000
 alfa
 FLAGS n n n
 n n y
 nn
 PROCESSING
 not used
 DISPLAY -60.0
 3060.5
 604.5
 0
 -100.4
 -68.5
 250
 0
 522
 4



+ concentrated H₃PO₂

Pd / xantphos (2 mol%)
 t-AmylOH, Dean Stark

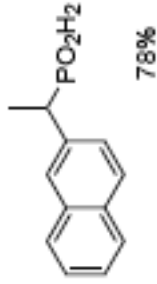


| INDEX | FREQUENCY | PPM | HIGHT |
|-------|-----------|--------|-------|
| 1 | 7819.507 | 64.578 | 39.6 |
| 2 | 5055.798 | 41.460 | 125.8 |
| 3 | 4501.289 | 37.882 | 36.3 |

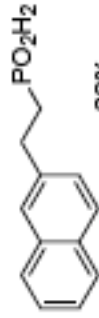


+ concentrated H₃PO₂

Pd / xantphos (2 mol%)
t-AmylOH, Dean Stark

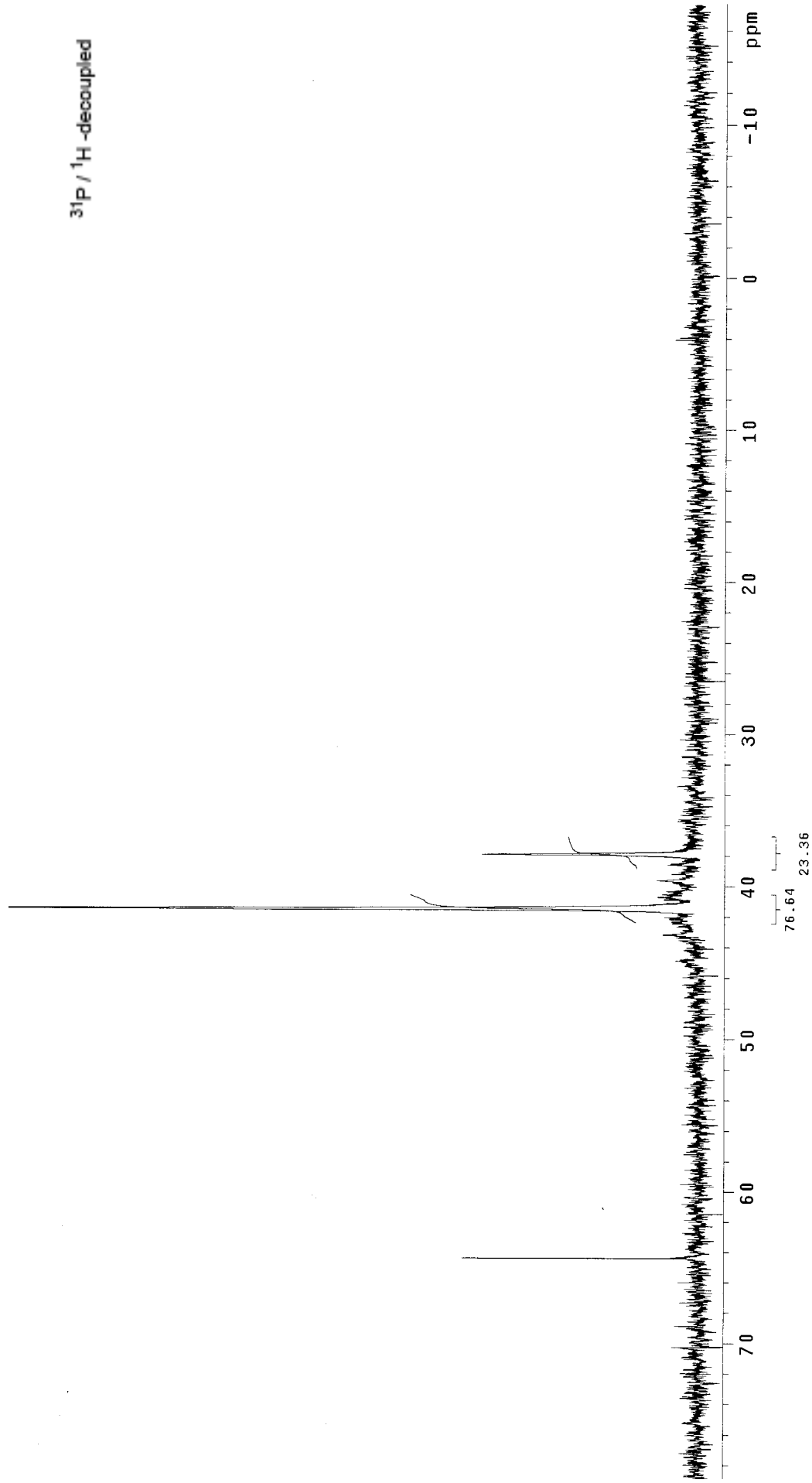


78%

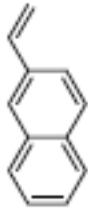


22%

³¹P / ¹H -decoupled



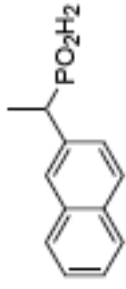
| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|------|----------|
| 1 | 5.114 | 6.08 | 4.5 24.7 |
| 2 | 4.668 | 6.10 | 4.0 08.6 |
| 3 | 4.758 | 7.74 | 5.9 17.9 |
| 4 | 4.174 | 6.74 | 5.5 6.05 |



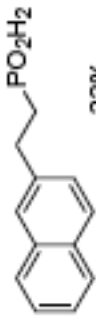
+ concentrated H₃PO₂

Pd / xantphos (2 mol%)

t-AmylOH, Dean Stark

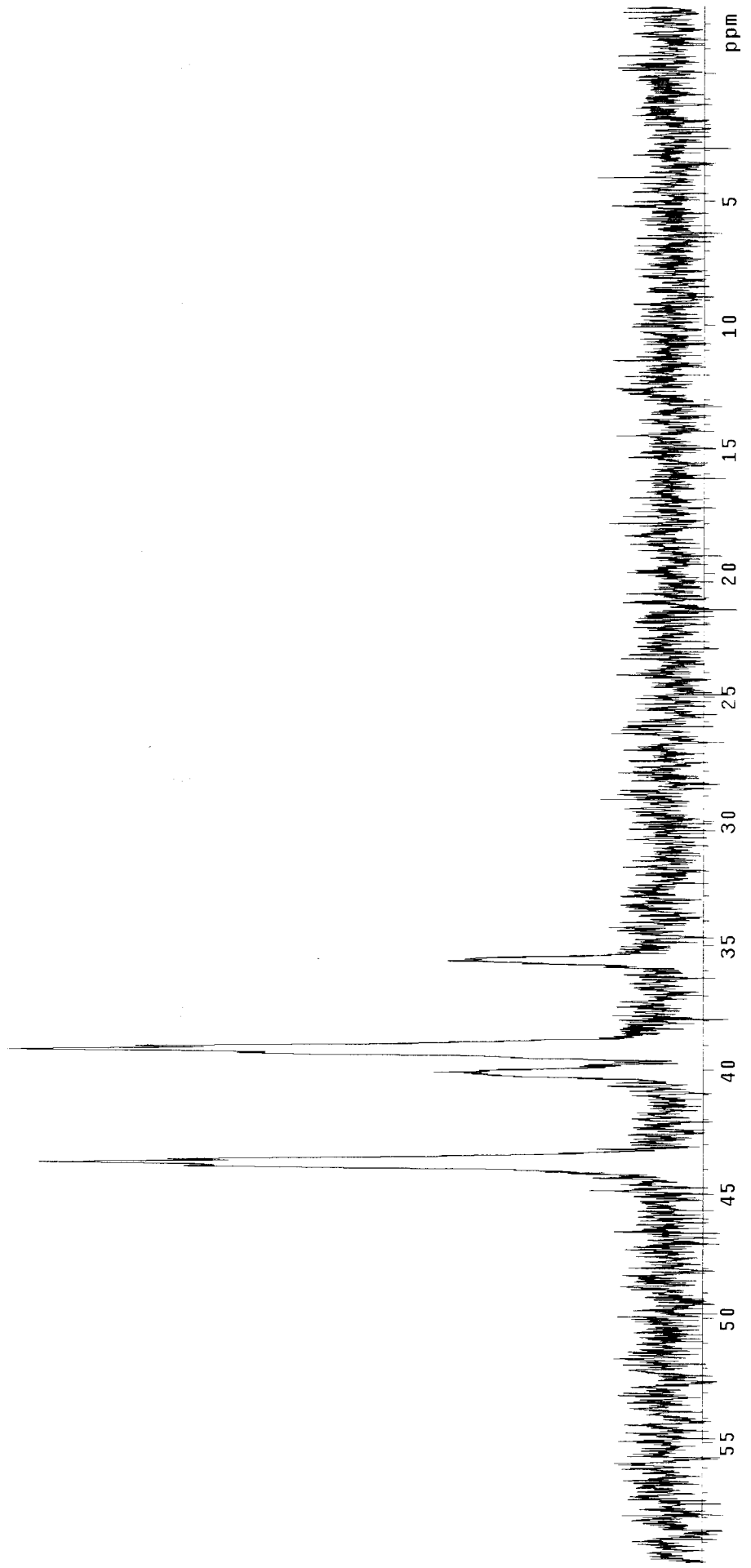


78%



22%

³¹P / ¹H -coupled



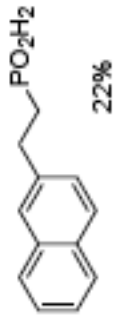
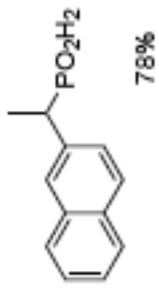
| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 3624.156 | 29.838 | 65.6 |
| 2 | 3604.164 | 29.673 | 71.4 |
| 3 | 3041.955 | 25.044 | 35.9 |



+ concentrated H₃PO₂

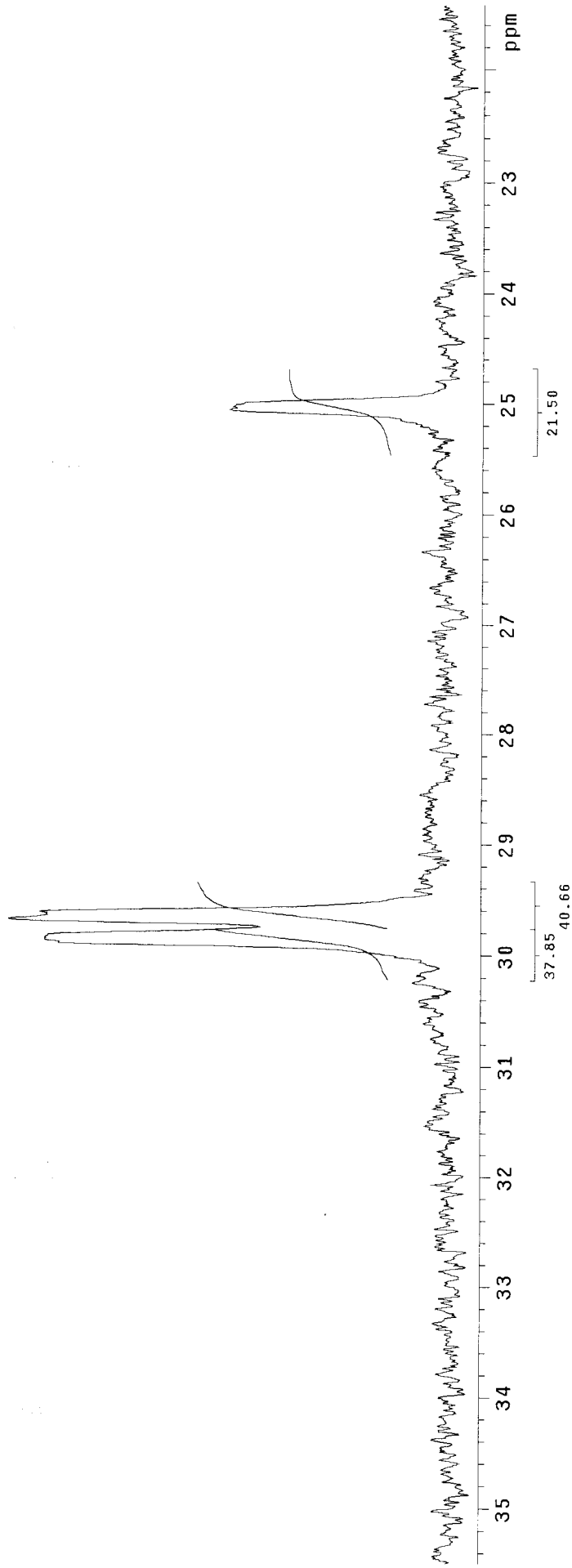
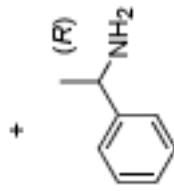
Pd / xantphos (2 mol%)

t-AmylOH, Dean Stark

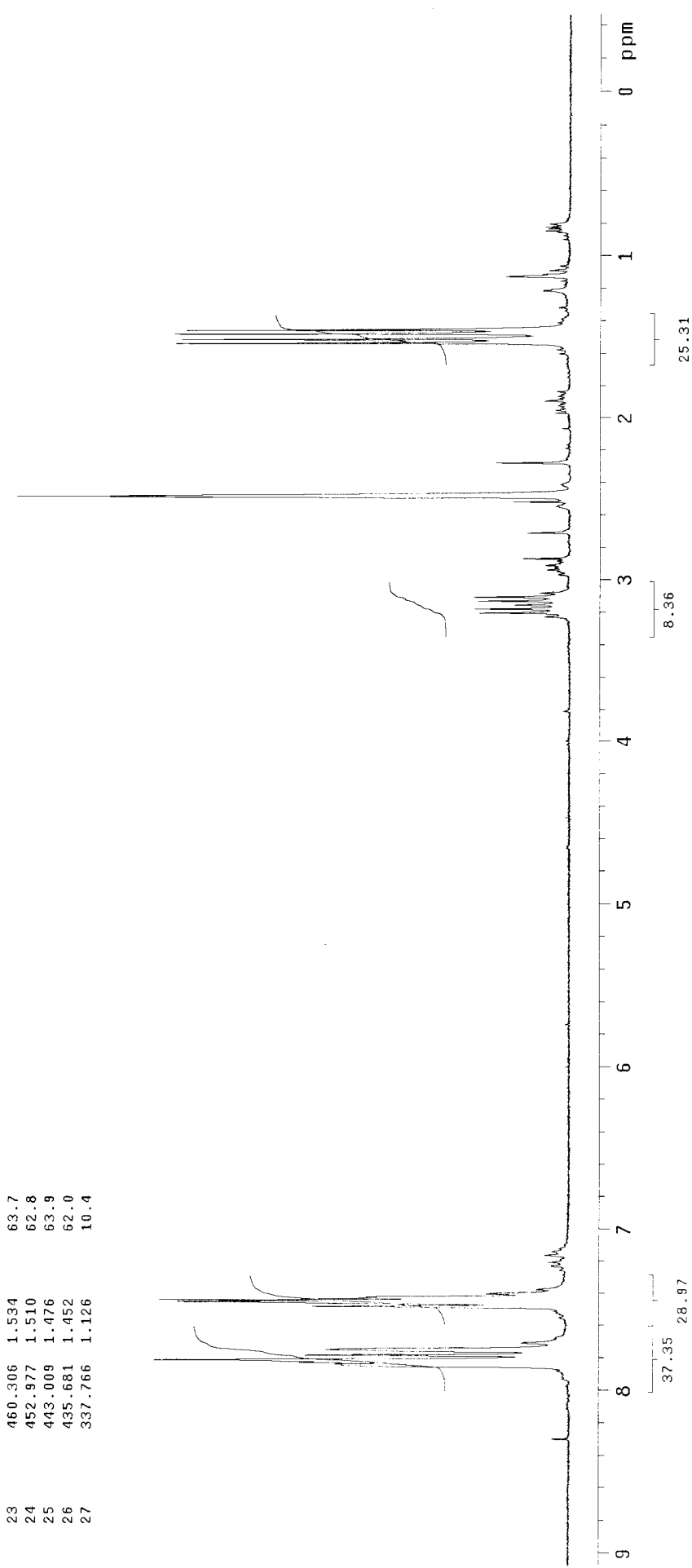
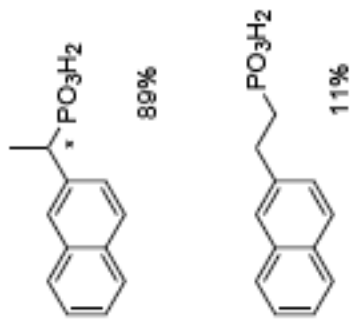


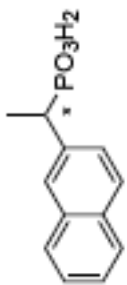
Enantiomeric excess determination

³¹P / ¹H -decoupled



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|-------|--------|
| 1 | 2352.923 | 7.842 | 38.1 |
| 2 | 2349.992 | 7.832 | 43.5 |
| 3 | 2345.008 | 7.815 | 67.1 |
| 4 | 2336.213 | 7.786 | 42.5 |
| 5 | 2325.953 | 7.752 | 39.1 |
| 6 | 2245.628 | 7.484 | 41.4 |
| 7 | 2240.351 | 7.467 | 38.7 |
| 8 | 2237.126 | 7.456 | 63.3 |
| 9 | 2233.902 | 7.445 | 66.2 |
| 10 | 2230.677 | 7.434 | 40.3 |
| 11 | 2227.745 | 7.425 | 32.7 |
| 12 | 2222.469 | 7.407 | 13.5 |
| 13 | 961.017 | 3.203 | 14.6 |
| 14 | 953.688 | 3.178 | 15.3 |
| 15 | 946.359 | 3.154 | 8.9 |
| 16 | 939.030 | 3.130 | 14.7 |
| 17 | 931.408 | 3.104 | 15.5 |
| 18 | 756.394 | 2.521 | 9.1 |
| 19 | 746.426 | 2.488 | 63.9 |
| 20 | 744.667 | 2.482 | 89.5 |
| 21 | 742.908 | 2.476 | 71.3 |
| 22 | 683.984 | 2.280 | 11.9 |
| 23 | 460.306 | 1.534 | 63.7 |
| 24 | 452.977 | 1.510 | 62.8 |
| 25 | 443.009 | 1.476 | 63.9 |
| 26 | 435.681 | 1.452 | 62.0 |
| 27 | 337.766 | 1.126 | 10.4 |

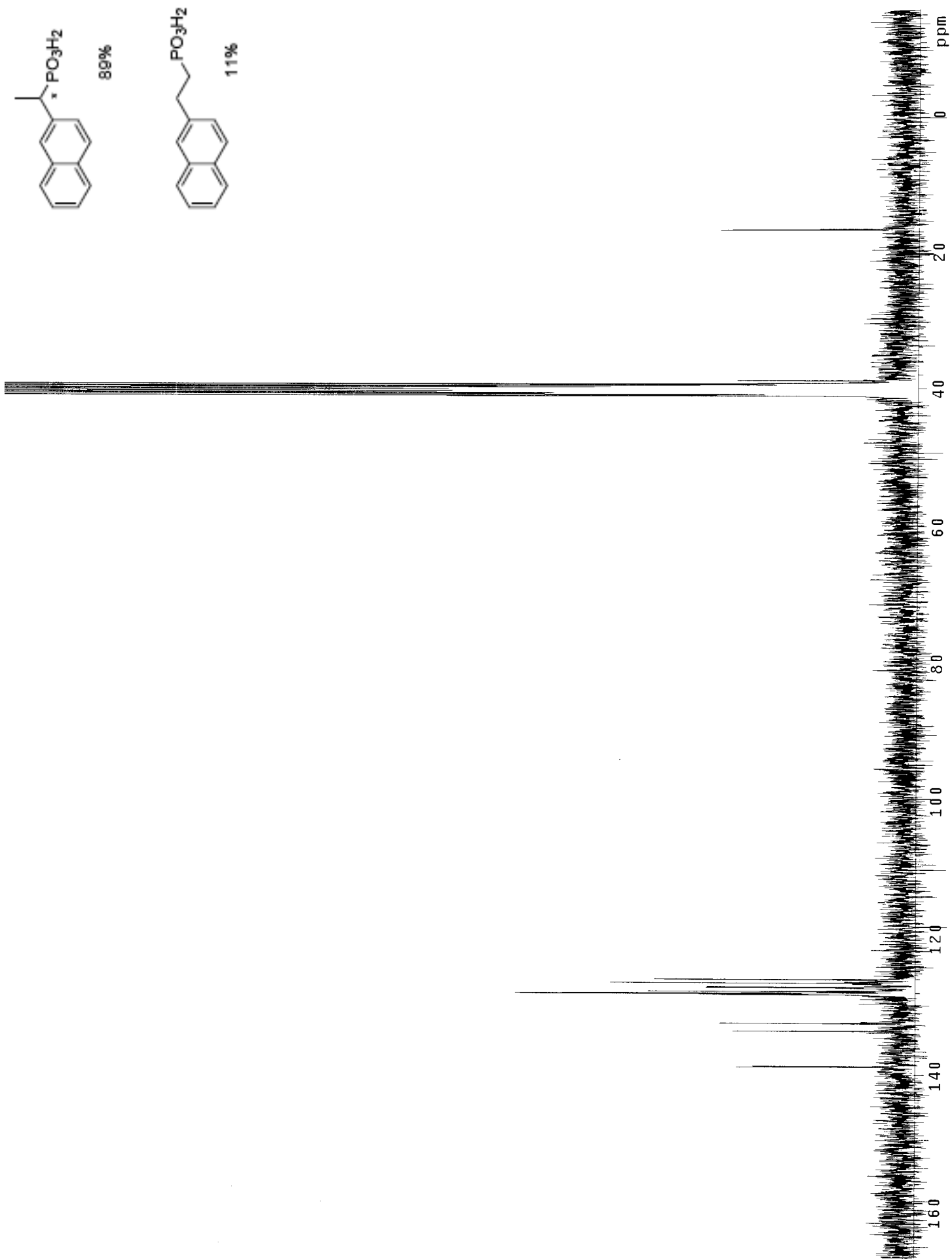




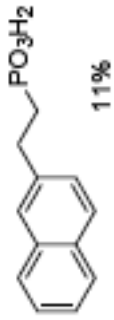
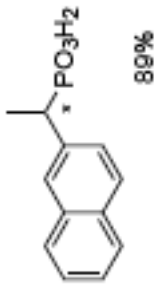
89%



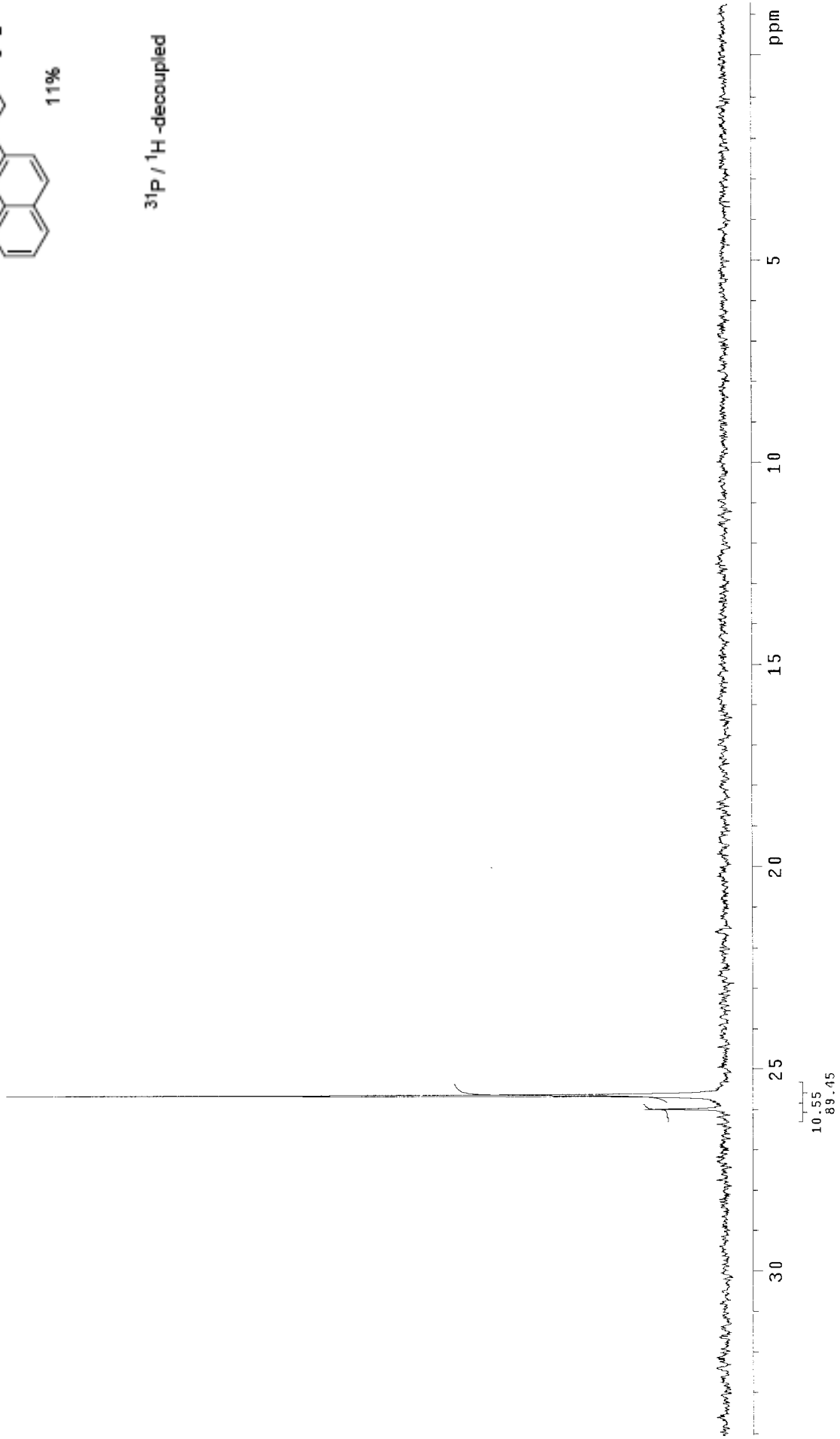
11%



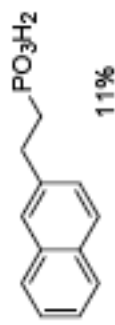
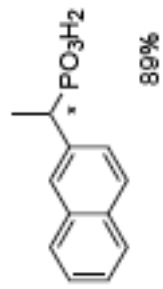
| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 3156.664 | 25.989 | 14.2 |
| 2 | 3115.049 | 25.646 | 126.0 |



$^{31}\text{P} / ^1\text{H}$ -decoupled



| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|--------|--------|
| 1 | 3148.504 | 25.921 | 16.0 |
| 2 | 3133.409 | 25.797 | 43.4 |
| 3 | 3114.641 | 25.643 | 60.5 |
| 4 | 3096.282 | 25.491 | 43.4 |
| 5 | 3077.106 | 25.334 | 15.3 |



$^{31}\text{P} / ^1\text{H}$ -coupled

