Supporting Information

Environmentally Benign Synthesis of H-Phosphinic Acids Using a Water-Tolerant, Recyclable Polymer-Supported Catalyst

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General Chemistry. ^{1}H NMR spectra were recorded on a Varian Mercury-300 spectrometer. Chemical shifts for ^{1}H NMR spectra are reported (in parts per million) relative to internal tetramethylsilane (Me_4Si, $\delta = 0.00$ ppm) with $CDCl_3$ as solvent. ^{13}C NMR spectra were recorded at 75 MHz. Chemical shifts for ^{13}C NMR spectra are reported (in parts per million) relative to $CDCl_3$ ($\delta = 77.0$ ppm). ^{31}P NMR spectra were recorded at 121 MHz on a Varian Mercury-300, spectrometer and/or at 36 MHz on an Anasazi EFT-90 spectrometer, and chemical shifts reported (in parts per million) relative to external 85% phosphoric acid ($\delta = 0.0$ ppm). Radial chromatography was carried out with a Harrison Associates Chromatotron using 1, 2, or 4 mm layers of silica gel 60 PF_{254} containing gypsum (E. Merck). Silica gel (200-300 mesh, Natland International Corporation) was used for flash chromatography. Ethyl acetate/hexanes mixtures were used as the eluent for chromatographic purifications. TLC plates were visualized by immersion in anisaldehyde stain (by volume: 93% ethanol, 3.5% sulfuric acid, 1% acetic acid, and 2.5% anisaldehyde) followed by heating. Organic solutions of products were dried over anhydrous $MgSO_4$.

Reagents and Solvents. Palladium catalysts and ligands were purchased from Strem or Aldrich. Polystyrene isocyanate was purchased from Advanced ChemTech or Aldrich, and used as received. Aqueous hypophosphorous acid (50 wt. %), was obtained from Aldrich and used as received. Concentrated hypophosphorous acid was obtaining by concentrating the 50 wt. % aqueous solution in vacuo on a rotary evaporator, at room temperature for 20-30 min before reaction. Anhydrous toluene was freshly distilled from CaH_2 prior to use. All other reagents were used as received. Reagent or HPLC grade acetonitrile was used throughout this study and was not dried prior to use.

^{31}P NMR Yield Measurements. The NMR yields are determined by integration of all the resonances in the ^{31}P NMR spectra, an approach which is valid if no phosphorus-containing gas (ie. PH_3) 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. Isolated yields are sometimes significantly lower because the acids are isolated by extraction, and therefore the corresponding isolated yields directly reflect water-solubility.

Octylphosphinic acid (Table 2, entry 1). References: (a) Deprèle, S.; Montchamp, J.-L. *J. Org. Chem.* **2001**, *66*, 6745; (b) Deprèle, S.; Montchamp, J.-L. *J. Am. Chem. Soc.* **2002**, *124*, 9386; (c) Karanewsky, D. S.; Badia, M. C.; Cushman, D. W.; DeForrest, J. M.; Dejneka, T.; Loots, M. J.; Perri, M. G.; Petrillo Jr., E. W.; Powell, J. R. *J. Med. Chem.* **1988**, *31*, 204; (d) Nifant'ev, E. E.; Magdeeva, R. K.; Shchepet'eva, N. P. *J. Gen. Chem. USSR* **1980**, *50*, 1416; (e) Devedjiev, I.; Ganev, V.; Stefanova, R.; Borisov, G. *Phosphorus Sulfur* **1987**, *31*, 7. ^{1}H NMR ($CDCl_3$)

δ 10.13 (bs, 1H), 7.08 (d, J = 540 Hz, 1 H), 1.70-1.80 (m, 2 H), 1.50-1.65 (m, 2 H), 1.25-1.40 (m, 12 H), 0.88 (t, J = 7 Hz, 3 H); ^{13}C NMR ($CDCl_3$) δ 31.6, 30.3 (d, J_{PCCC} = 16 Hz), 29.1 (d, J_{PC} = 94 Hz), 29.0, 28.9, 22.5, 20.6 (d, J_{PCC} = 3 Hz), 14.0; ^{31}P NMR ($CDCl_3$) δ 38.9 (dt, J_{P-H} = 540, 12 Hz).

Decylphosphinic acid (Table 2, entry 2). References: : (a) Deprèle, S.; Montchamp, J.-L. *J. Org. Chem.* **2001**, *66*, 6745; (b) Karanewsky, D. S.; Badia, M. C.; Cushman, D. W.; DeForrest, J. M.; Dejneka, T.; Loots, M. J.; Perri, M. G.; Petrillo Jr.; E. W.; Powell, J. R. *J. Med. Chem.* **1988**, *31*, 204; (c) Nifant'ev, E. E.; Magdeeva, R. K.; Shchepet'eva, N. P. *J. Gen. Chem. USSR* **1980**, *50*, 1416. ^1H NMR ($CDCl_3$) δ 11.58 (bs, 1H), 7.08 (d, J = 540 Hz, 1 H), 1.20-1.75 (m, 18 H), 0.88 (t, J = 6 Hz, 3 H); ^{13}C NMR ($CDCl_3$) δ 31.9, 30.4 (d, J_{PCCC} = 16 Hz), 29.9, 29.2 (d, J_{PC} = 94 Hz), 29.4, 29.3, 29.2, 22.7, 20.6(d, J_{PCC} = 3 Hz), 14.1; ^{31}P NMR ($CDCl_3$) δ 39.3 (dm, J_{P-H} = 540 Hz).

4-Phenylbutylphosphinic acid (Table 2, entries 3 & 4). References: (a) Deprèle, S.; Montchamp, J.-L. *J. Org. Chem.* **2001**, *66*, 6745; (b) Karanewsky, D. S.; Badia, M. C.; Cushman, D. W.; DeForrest, J. M.; Dejneka, T.; Loots, M. J.; Perri, M. G.; Petrillo Jr.; E. W.; Powell, J. R. *J. Med. Chem.* **1988**, *31*, 204; (c) Anderson, N. G.; Coradetti, M. L.; Cronin, J. A.; Davies, M. L.; Gardineer, M. B.; Kotnis, A. S.; Lust, D. A.; Palaniswamy, V. A. *Org. Process Res. Dev.* **1997**, *1*, 315. ^1H NMR ($CDCl_3$) δ 11.74 (bs, 1H), 7.15-7.25 (m, 5H), 7.04 (d, J = 542 Hz, 1 H), 2.61 (t, J = 7 Hz, 2 H), 1.60-1.75 (m, 6 H); ^{13}C NMR ($CDCl_3$) δ 141.6, 128.3, 125.8,35.3, 32.1 (d, J_{PCCC} = 16 Hz), 29.0 (d, J_{PC} = 94 Hz), 20.3 (d, J_{PCC} = 3 Hz); ^{31}P NMR ($CDCl_3$) δ 38.2 (dm, J_{P-H} = 542 Hz).

7-Methyl-2-octenephosphinic acid (Table 2, entry 5). ^1H NMR ($CDCl_3$) δ 10.30 (bs, 1H), 7.08 (d, J = 540 Hz, 1 H), 5.09 (t, J = 1.5 Hz, 1 H); 1.95-2.0 (m, 2 H), 1.55-1.80 (m, 8 H), 1.30-1.45 (m, 6 H); ^{13}C NMR ($CDCl_3$) δ 131.8, 124.6, 30.3 (d, J_{PCCC} = 16 Hz), 29.5 (d, J_{PC} = 94 Hz), 29.6, 27.9, 25.9, 20.8 (d, J_{PCC} = 3 Hz), 17.9; ^{31}P NMR ($CDCl_3$) δ 38.9 (dm, J_{P-H} = 540 Hz).

1-Phenylethylphosphinic acid (Table 2, entry 6). Reference: Buckler, S. A.; Epstein, M. *Tetrahedron* **1962**, *18*, 1211. ^1H NMR ($CDCl_3$) d 9.08 (bs, 1H), 7.20-7.35 (m, 5H), 6.90 (d, J = 552 Hz, 1 H), 3.10-3.20 (m, 1 H), 1.50-1.60 (dd, J = 19, 7 Hz, 3 H); ^{31}P NMR ($CDCl_3$) d 41.2 (dm, J_{P-H} = 552 Hz).

2-Phenylethylphosphinic acid (Table 2, entry 6). References: (a) Arbuzova, S. N.; Gusarova, N. K.; Malysheva, S. F.; Brandsma, L.; Albanov, A. I.; Trofimov, B. A. *Russ. J. Gen. Chem.* **1996**, *66*, 54; (b) Jackson, P. F.; Tays, K. L.; Maclin, K. M.; Ko, Y.-S.; Li, W.; Vitharana, D.; Tsukamoto, T.; Stoermer, D.; Lu, X.-C. M.; Wozniak, K.; Slusher, B. S. *J. Med. Chem.* **2001**, *44*, 4170; (c) Lui, X.; Hu, E.; Tian, X.; Mazur, A.; Ebetino, F. H. *J. Organomet. Chem.* **2002**, *646*, 212; (d) Gusarova, N. K.; Shaikhudinova, S. I.; Sukhov, B. G.; Kazantseva, T. I.; Malysheva, S. F.; Smetannikov, Y. V.; Tarasova, N. P.; Kuimov, V. A.; Trofimov, B. A. *Russ. Chem. Bull.* **2003**, *52*, 511. ^1H NMR ($CDCl_3$) d 9.08 (bs, 1H), 7.20-7.35 (m, 5H), 7.06 (d, J = 548 Hz, 1 H), 2.85-2.95 (m, 2 H), 2.05-2.15 (m, 2H); ^{31}P NMR ($CDCl_3$) d 37.6 (dm, J_{P-H} = 549 Hz).

(1-Cyclohexyl) methylphosphinic acid (Table 2, entry 7). ^1H NMR ($CDCl_3$) δ 10.65 (bs, 1H), 7.18 (d, J = 540 Hz, 1 H), 1.60-1.75 (m, 7 H), 1.40-1 (m, 6 H); ^{13}C NMR ($CDCl_3$) δ 37.0, 36.7, 34.5, 34.4, 31.7, 26.0, 25.9; ^{31}P NMR ($CDCl_3$) δ 37.9 (dm, J_{P-H} = 540 Hz).

[2-(4-Chloro-phenyl)-propyl]-phosphinic acid (Table 2, entry 8).
^{1}H NMR ($CDCl_3$) δ 10.0 (bs, 1H), 7.1-7.4 (m, 4H), 6.85 (d, J = 550 Hz, 1 H), 3.15-3.25 (m, 1 H), 2.0-2.1 (m, 2 H), 1.34 (t, J = 6 Hz, 3 H); ^{13}C NMR ($CDCl_3$) δ 143.9, 143.8, 132.4, 128.8, 128.3, 128.0, 37.6 (d, J_{PC} = 94 Hz), 33.3, 23.6(d, J_{PCC} = 10 Hz), 14.0; ^{31}P NMR ($CDCl_3$) δ 39.7 (dm, J_{P-H} = 550 Hz).

2-Phthalimidylethylphosphinic acid (Table 2, entry 9). ^{1}H NMR (DMSO) δ 7.80-7.85 (m, 4H), 7.0 (d, J = 531 Hz, 1 H), 3.70-3.80 (m, 2 H), 1.95-2.05 (m, 2H); ^{13}C NMR (DMSO) δ 168.3, 135.0, 132.4, 123.7, 31.4, 29.5 (d, J_{PC} = 89 Hz); ^{31}P NMR (DMSO) δ 23.3 (dm, J_{P-H} = 532 Hz).

(1-Phenyl-vinyl)-phosphinic acid (Table 2, entry 10). Reference: Dumond, Y. R.; Montchamp, J.-L. *J. Organomet. Chem.* **2002**, *653*, 252. ^{1}H NMR ($CDCl_3$, $SiMe_4$) δ 12.37 (br, 1 H), 7.4-7.5 (m, 2 H), 7.2-7.4 (m, 3 H), 7.30 (d, J = 571 Hz, 1 H), 6.16 (d, J = 46 Hz, 1 H), 6.14 (d, J = 25 Hz, 1 H); ^{13}C NMR ($CDCl_3$) δ 142.3 (d, J_{PC} = 124 Hz), 135.1 (d, J_{PCC} = 13 Hz), 129.3 (d, J_{PCC} = 13 Hz), 128.7 (2 C), 128.6, 127.3 (d, J_{PCCC} = 6 Hz, 2 C); ^{31}P NMR ($CDCl_3$) δ 25.7 (ddd, J = 571 Hz, J = 46 Hz, J = 24 Hz).

***Trans*-Oct-1-enyl-phosphinic acid (Table 2, entry 11).** References: (a) Dumond, Y. R.; Montchamp, J.-L. *J. Organomet. Chem.* **2002**, *653*, 252; (b) Nifant'ev, E. E.; Magdeeva, R. K.; Maslennikova, V. I.; Taber, A. M.; Kalechits, I. V. *J. Gen. Chem. USSR* **1982**, *52*, 2173; (c) Belakhov, V. V.; Yudelevich, V. I.; Fetter, A. P.; Ionin, B. I. *J. Gen. Chem. USSR* **1986**, *56*, 680. ^{1}H NMR ($CDCl_3$, $SiMe_4$) δ 10.64 (br, 1 H), 7.2 (d, J = 562 Hz, 1 H), 6.6-6.8 (m, 1 H), 5.82 (dd, J = 17 Hz, J = 24 Hz, 1 H), 2.2-2.3 (m, 2 H), 1.4-1.5 (m, 2 H), 1.2-1.4 (m, 6 H), 0.88 (d, J = 6 Hz, 3 H); ^{13}C NMR ($CDCl_3$) δ 153.7 (d, J_{PCC} = 6 Hz), 120.5 (d, J_{PC} = 134 Hz), 34.1 (d, J_{PCCC} = 20 Hz), 31.5, 28.7, 27.6, 22.5, 14.0; ^{31}P NMR ($CDCl_3$) 23.9 (d, J = 562 Hz).
(1-Hexyl-vinyl)-phosphinic acid (Table 2, entry 11). Reference: Dumond, Y. R.; Montchamp, J.-L. *J. Organomet. Chem.* **2002**, *653*, 252. ^{1}H NMR ($CDCl_3$, $SiMe_4$) δ 12.78 (br, 1 H), 7.15 (d, J = 557 Hz, 1 H), 5.92 (d, J = 25 Hz, 1 H), 5.79 (d, J = 49 Hz, 1 H), 2.30 (m, 2 H), 1.5-1.6 (m, 2 H), 1.2-1.4 (m, 6 H), 0.88 (t, J = 7 Hz, 3 H); ^{13}C NMR ($CDCl_3$) δ 142.6 (d, J_{PC} = 121 Hz), 127.4 (d, J_{PCC} = 14 Hz), 31.46, 30.5 (d, J_{PCC} = 13 Hz), 28.7, 27.7 (d, J_{PCCC} = 5 Hz), 22.5, 13.9; ^{31}P NMR ($CDCl_3$) δ 27.4 (dm, J = 557 Hz).

(1-Butyl-hex-1-enyl) phosphinic acid (Table 2, entry 12). ^{1}H NMR ($CDCl_3$) δ 12.77 (bs, 1 H), 7.10 (d, J = 550 Hz, 1 H), 6.32 (dt, J = 27 Hz, J = 7 Hz, 1 H), 2.15-2.3 (m, 4 H), 1.25-1.50 (m, 8 H), 0.89 (d, J = 7 Hz, 6 H); ^{13}C NMR ($CDCl_3$) δ 145.3 (d, J_{PCC} = 14 Hz), 132.8 (d, J_{PC} = 127 Hz), 31.3, 30.6, 28.0, 27.8, 24.8 (d, J_{PCCC} = 13 Hz), 22.7, 22.3, 13.7; ^{31}P NMR ($CDCl_3$) 30.73 (dm, J = 550).

Preparation of supported-ligand 4. Dry toluene (15 mL) was added to a mixture of polystyrene isocyanate resin (1.003 g, 2.4 mmol; Advanced Chemtech, 100-200 Mesh, 2.4 mmol/g), and 4,6-bis(diphenylphosphino)phenoxazine **2** (0.653 g, 1.185 mmol; Strem, nixantphos). The suspension was stirred and heated at 115 ºC overnight, under N_2. To the resulting mixture was added anhydrous diisopropylamine (0.5 ml, 3.56 mmol) in order to neutralize the unreacted isocyanate groups. The reaction mixture was stirred at room

temperature for 6 hours, under N_2. After filtration, the collected resin (1.2 g) was rinsed with dichloromethane and dried overnight under vacuum, over P_2O_5; while the filtrate was evaporated to recover unreacted nixantphos as an off-white powder. This amount was taken into account to calculate the loading of the resin. The supported ligand's loading was found to be 0.14 mmol/g.

Representative hydrophosphinylation procedure with 50 % aqueous H_3PO_2. Aqueous hypophosphorous acid (50 wt. %) was first weighted in a round bottom flask, (0.2723 g, 2.06 mmol). Acetonitrile (2 mL), was then added to the flask. Addition of 4-phenylbutene (0.15 mL, 0.99 mmol), Pd_2dba_3 (4.6 mg, 1 mol %), and supported nixantphos, (0.14 mmol/g, 64.8 mg, 1.08 mol %) followed. In order to avoid any potential leak of solvent, the neck of the flask was washed with another portion of acetonitrile (2 mL). The resulting mixture was stirred and refluxed overnight under nitrogen. After measuring the crude yield by ^{31}P NMR, the reaction mixture was cooled down to room temperature and then filtered to collect the resin. The filtrate was then extracted with hexane, once. The acetonitrile layer was concentrated down and the residue underwent a single extraction with ethyl acetate and brine. The organic layer was dried over $MgSO_4$. After filtration and concentration the resulting yellow oil was dried overnight under vacuum, over P_2O_5. The reaction yielded 4-phenylbutylphosphinic acid (0.193 g, 97%) as a yellow oil.

Hydrophosphinylation procedure with concentrated H_3PO_2. These runs were conducted as above with the 50 wt. % solution except the solution was first concentrated in vacuo at room temperature for 20-30 min, prior to the addition of the substrate and catalyst.

Reaction in air and at room temperature. To a solution of 50 % aq H_3PO_2 (0.211 g, 1.60 mmol) was added some HPLC grade CH_3CN, 4-phenylbutene (0.15 ml, 0.99 mmol), Pd_2dba_3 (5 mg, 1.1 mol %), and supported nixantphos (0.33 mmol/g, 33.9 mg, 1.1 mol %). The reaction was stirred at room temperature in an open flask for 6 hours. After taking a ^{31}P NMR of the crude mixture, the yield of the desired phosphinic acid was 78 %. The corresponding isolated yield was 51% (after extraction as above with brine and ethyl acetate).

Reaction in air and at reflux. Aqueous hypophosphorous acid (50 wt. %, 0.356 g, 2.69 mmol) and CH_3CN (10 mL) were added to 1-octene (0.25 ml, 1.59 mmol), along with some Pd_2dba_3 (8 mg, 1.1 mol %) and supported nixantphos (0.33 mmol/g, 67 mg, 1.4 mol %). The reaction was refluxed overnight open to air. After measuring the yield by ^{31}P NMR, concentration and partition between ethyl acetate and brine of the corresponding crude phosphinic acid was accomplished. Drying the residue overnight under vacuum over P_2O_5 gave the product (0.188 g, 89%) as yellow oil.

Multi run reactions (Table 2, entry 6). To aqueous hypophosphorous acid (50 wt. %, 0.490 g, 4 mmol) was added styrene (0.30 ml, 2.62 mmol), Pd_2dba_3 (11.6 mg, 0.96 mol %) and supported Nixantphos (0.33 mmol/g, 95.6 mg, 1.2 mol %). The reaction mixture was refluxed overnight under N_2. The next day, after measuring the crude yield by ^{31}P NMR, the mixture was filtered in order to separate the resin from the filtrate. The resin was washed with CH_3CN and kept in a dessicator, over P_2O_5, between the runs. The filtrate was kept at in a stoppered Erlenmeyer. The resin was then used in the next run without any further precautions. Aqueous hypophosphorous acid (50 wt. %, 0.490 g, 4 mmol), styrene (0.3 ml, 2.62 mmol) and the previously collected resin

were mixed together in CH_3CN (10 mL). The reaction mixture was again refluxed overnight under N_2. The next day, after measuring the crude yield by ^{31}P NMR, the mixture was filtered in order to separate the resin and the filtrate. The resin was washed with CH_3CN, and collected for another run. The filtrate was kept in the same Erlenmeyer as before. For the third time, the reaction was run, with 50 % aq H_3PO_2 (0.390 g, 3.17 mmol), styrene (0.30 ml, 2.62 mmol) and the previously collected resin. The same procedures were used to run the reaction, control the NMR yield and separate the resin from the desired product. The experiment was then run for one last time: 50 % aq H_3PO_2 (0.499 g, 4 mmol), styrene (0.30 ml, 2.62 mmol) and the previously collected resin. At the end of the sequence, all the filtrates were in a single Erlenmeyer. After concentration, partition between EtOAc and brine, the product (2.00 g, 90% over 4 runs) was obtained as a yellow oil and a mixture of regioisomers.

INDEX	FREQUENCY	PPM	HEIGHT
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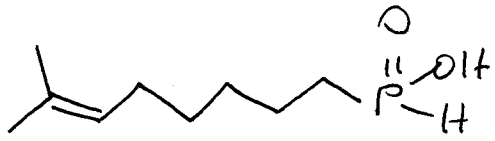

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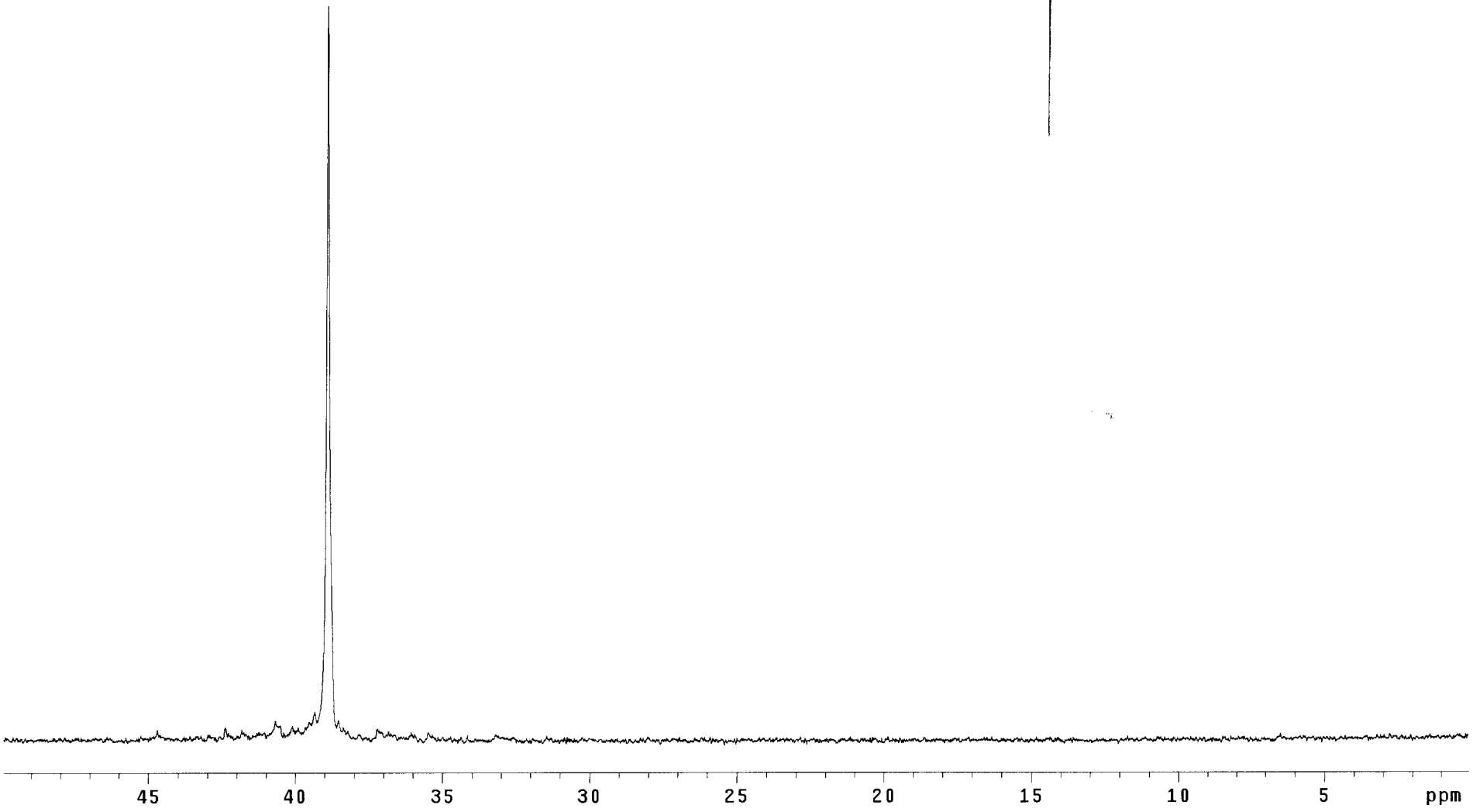

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3	4987.517	41.061	103.4
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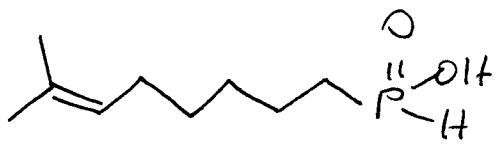
5	4461.103	36.727	126.0
6	4447.785	36.617	103.5



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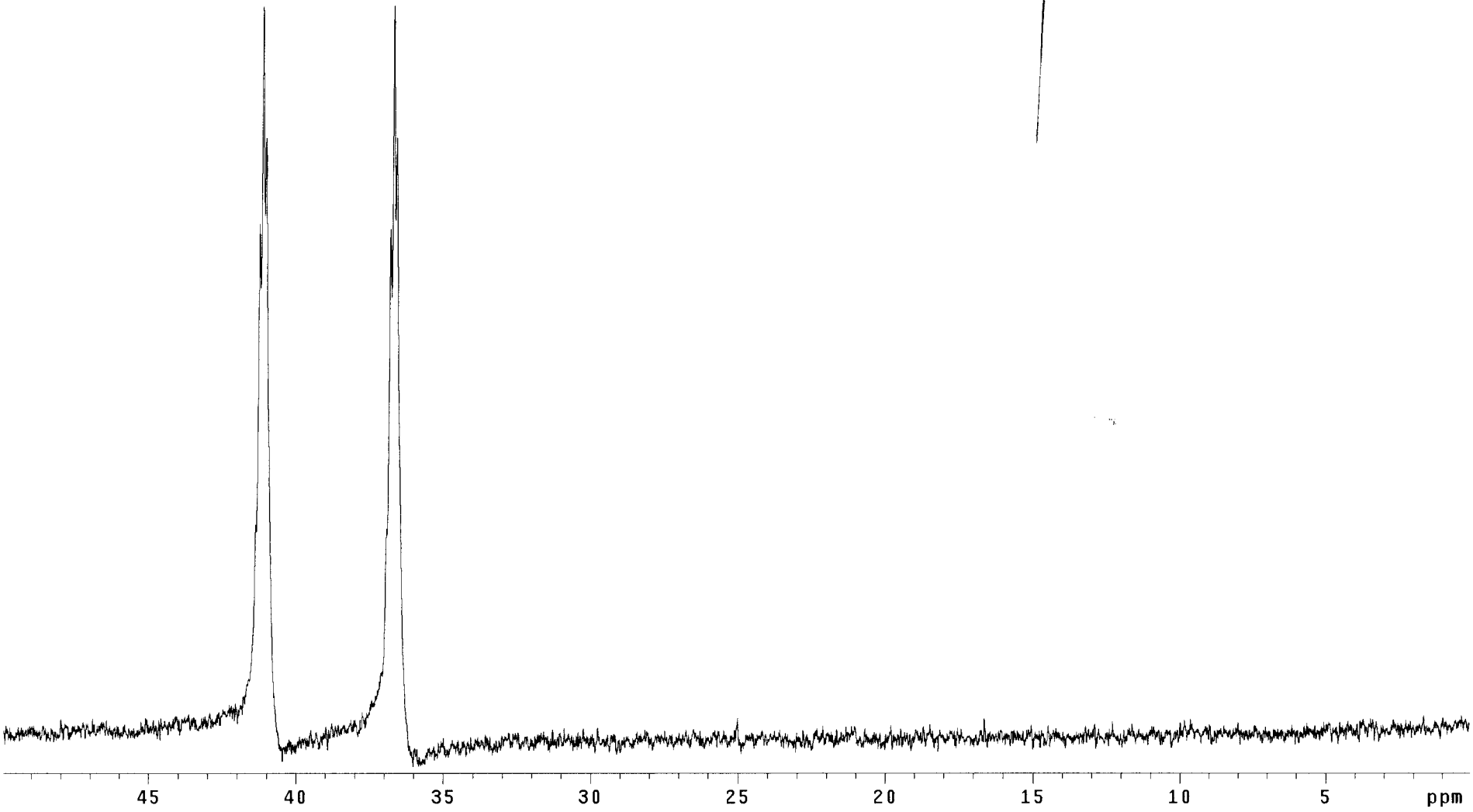

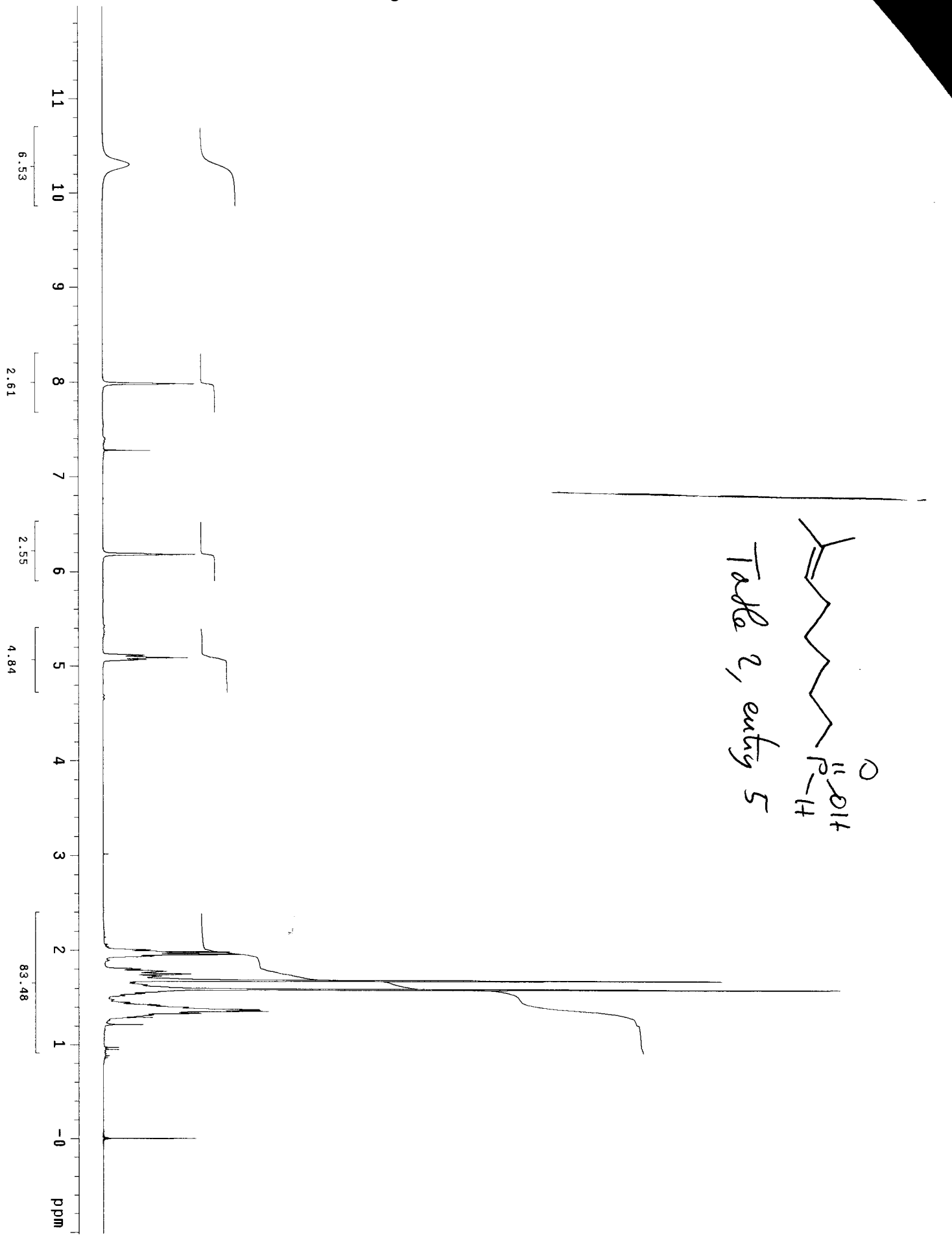

Table 2, entry 5

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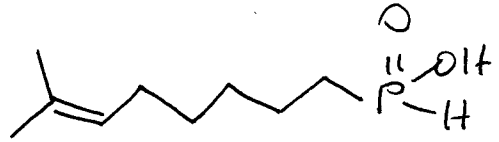
4	5829.476	77.262	129.8
5	5797.606	76.840	127.6
6	2291.840	30.375	57.1
7	2275.905	30.164	60.5
8	2269.627	30.081	47.7
9	2230.031	29.556	114.0
10	2175.947	28.839	43.1
11	2105.446	27.905	145.7
12	1956.716	25.934	133.9
13	1569.923	20.807	56.1
14	1567.026	20.769	58.2
15	1350.209	17.895	92.5



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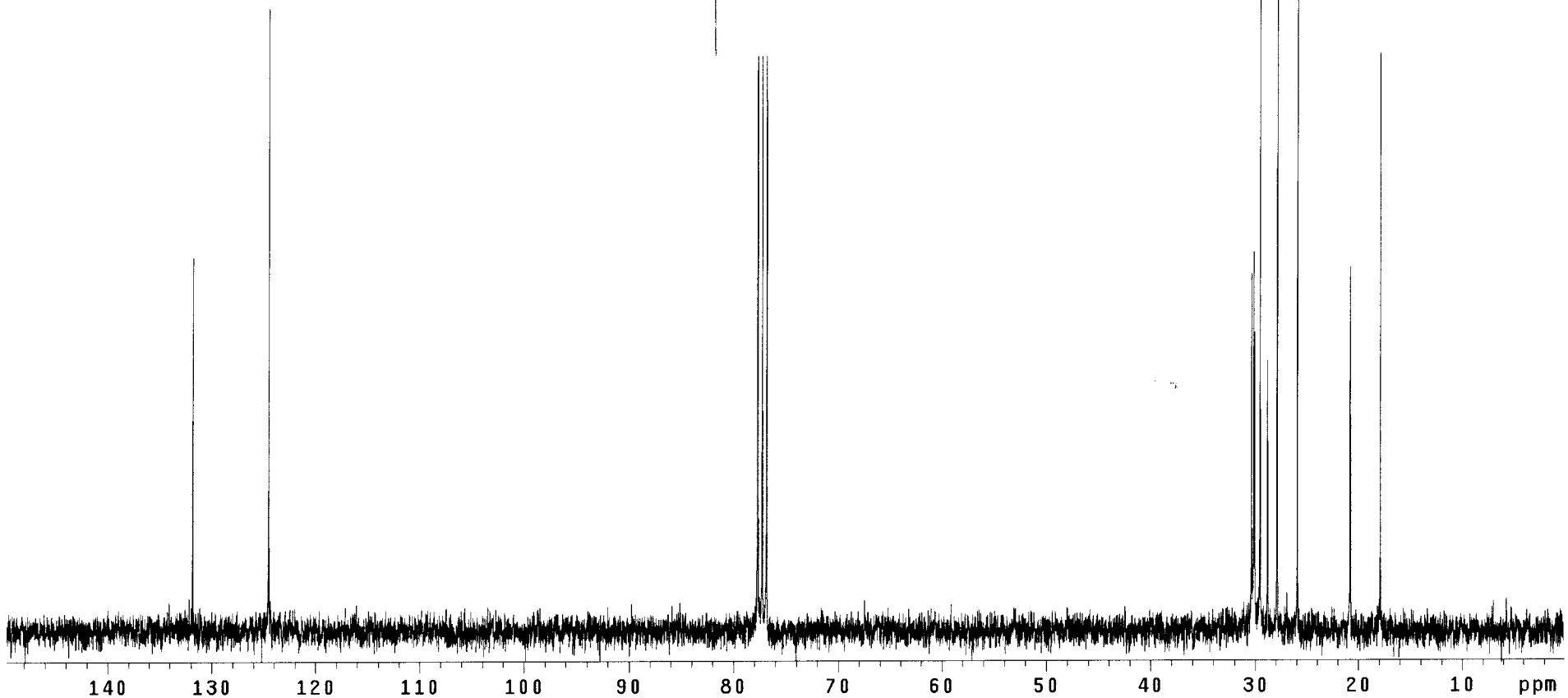

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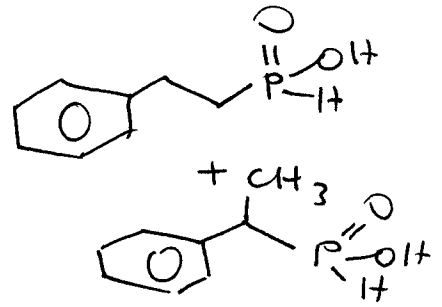

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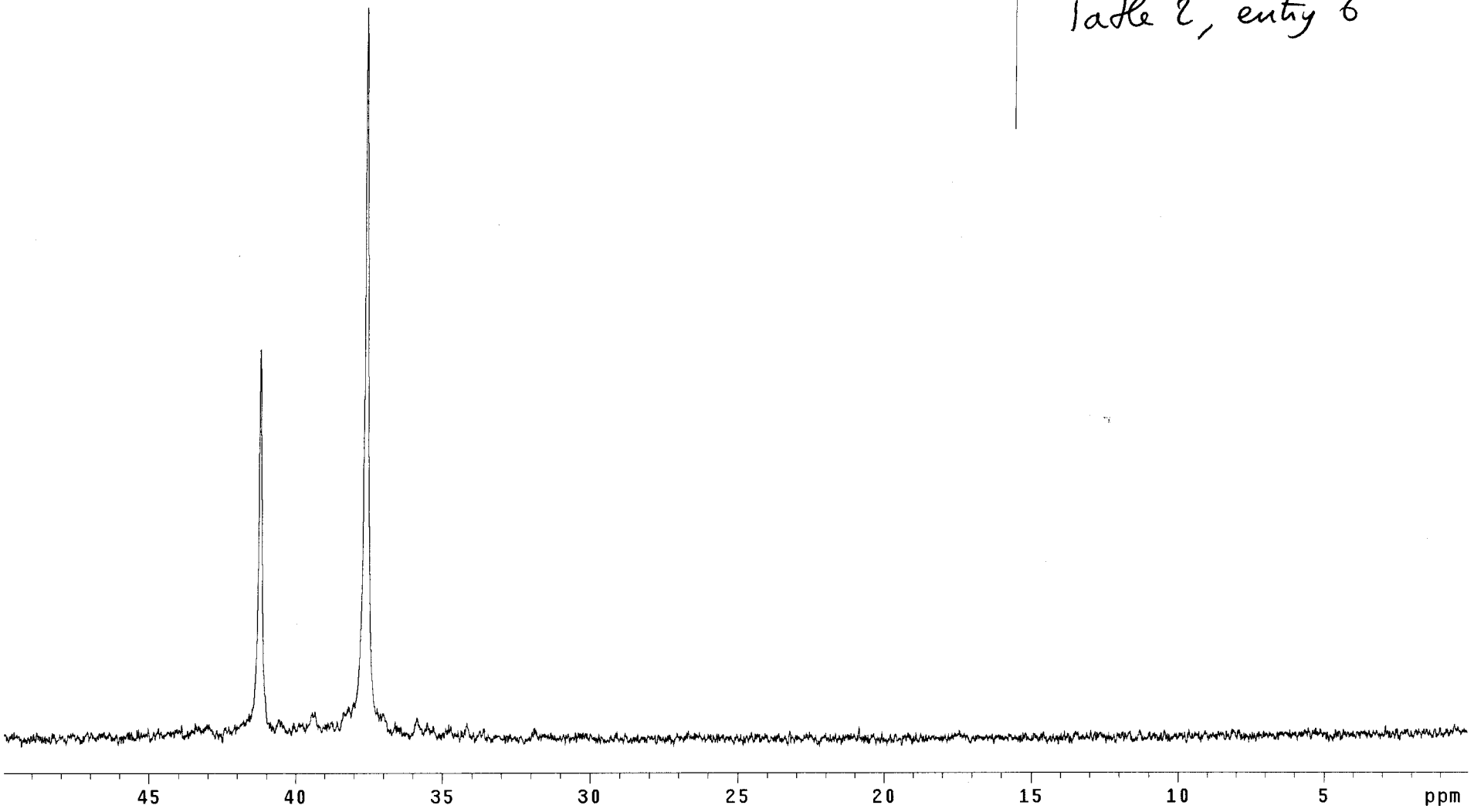

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1	5284.203	43.503	41.4
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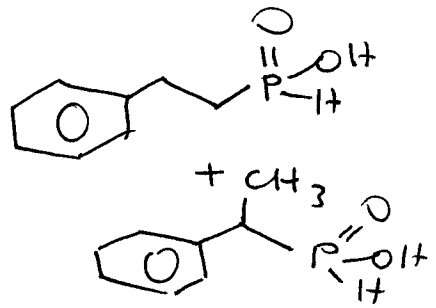
4	4292.784	35.341	92.2



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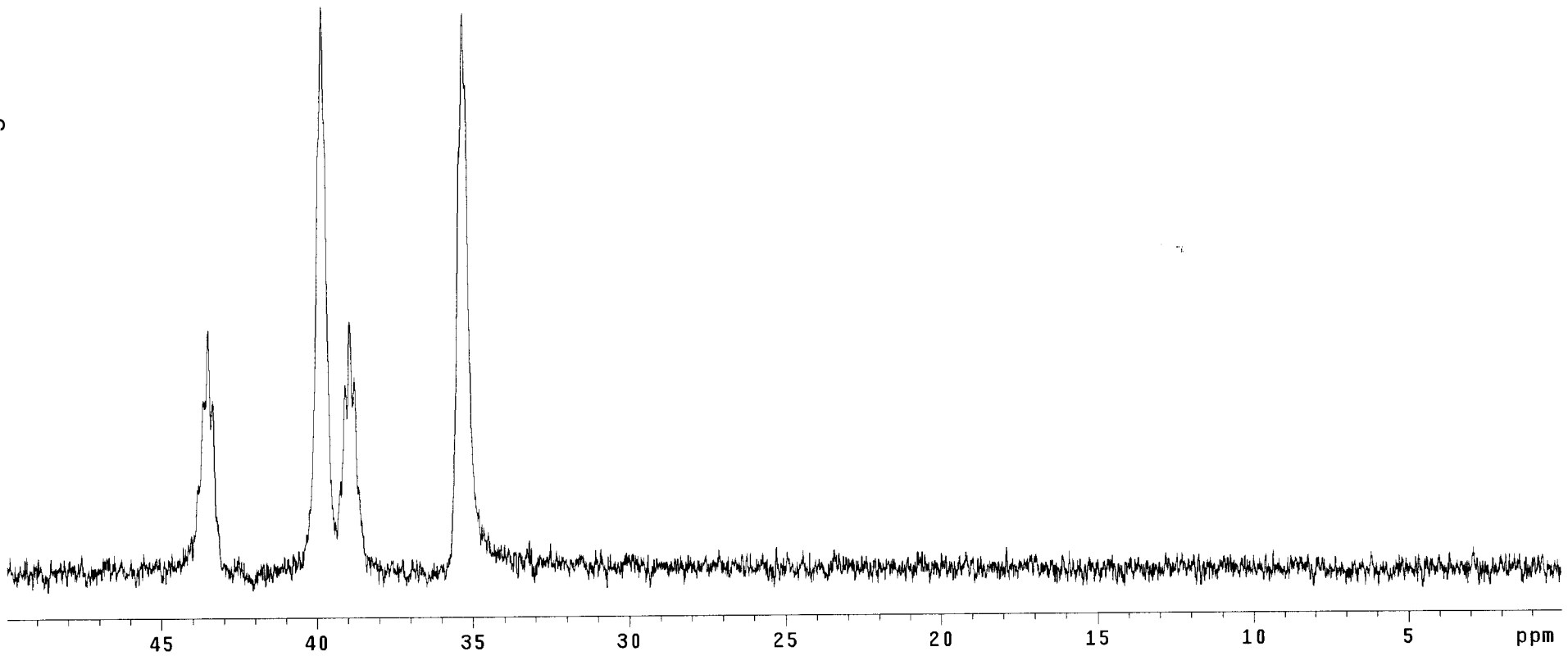

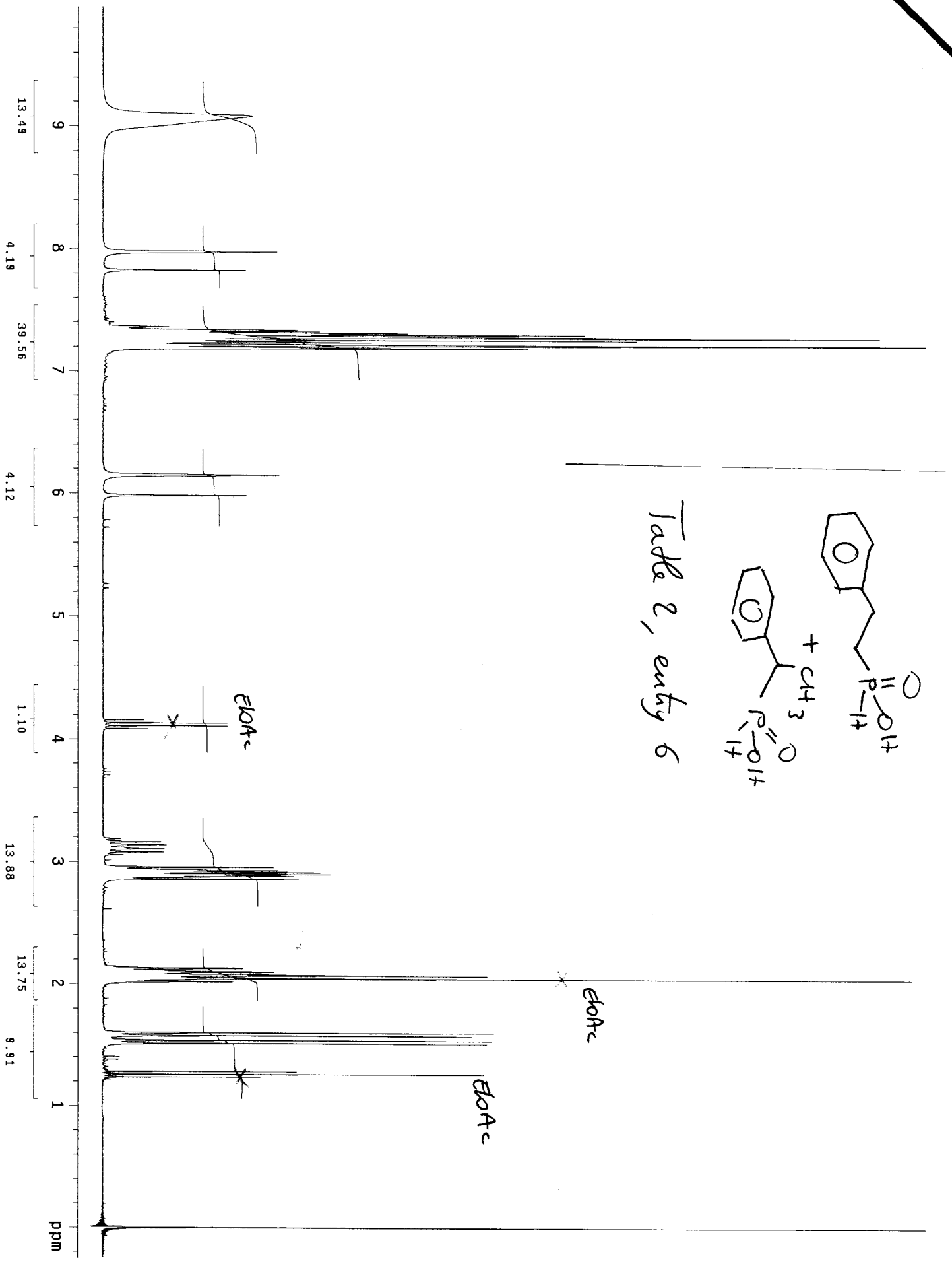

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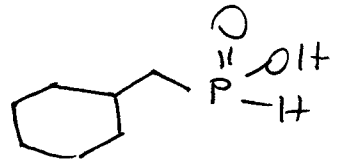
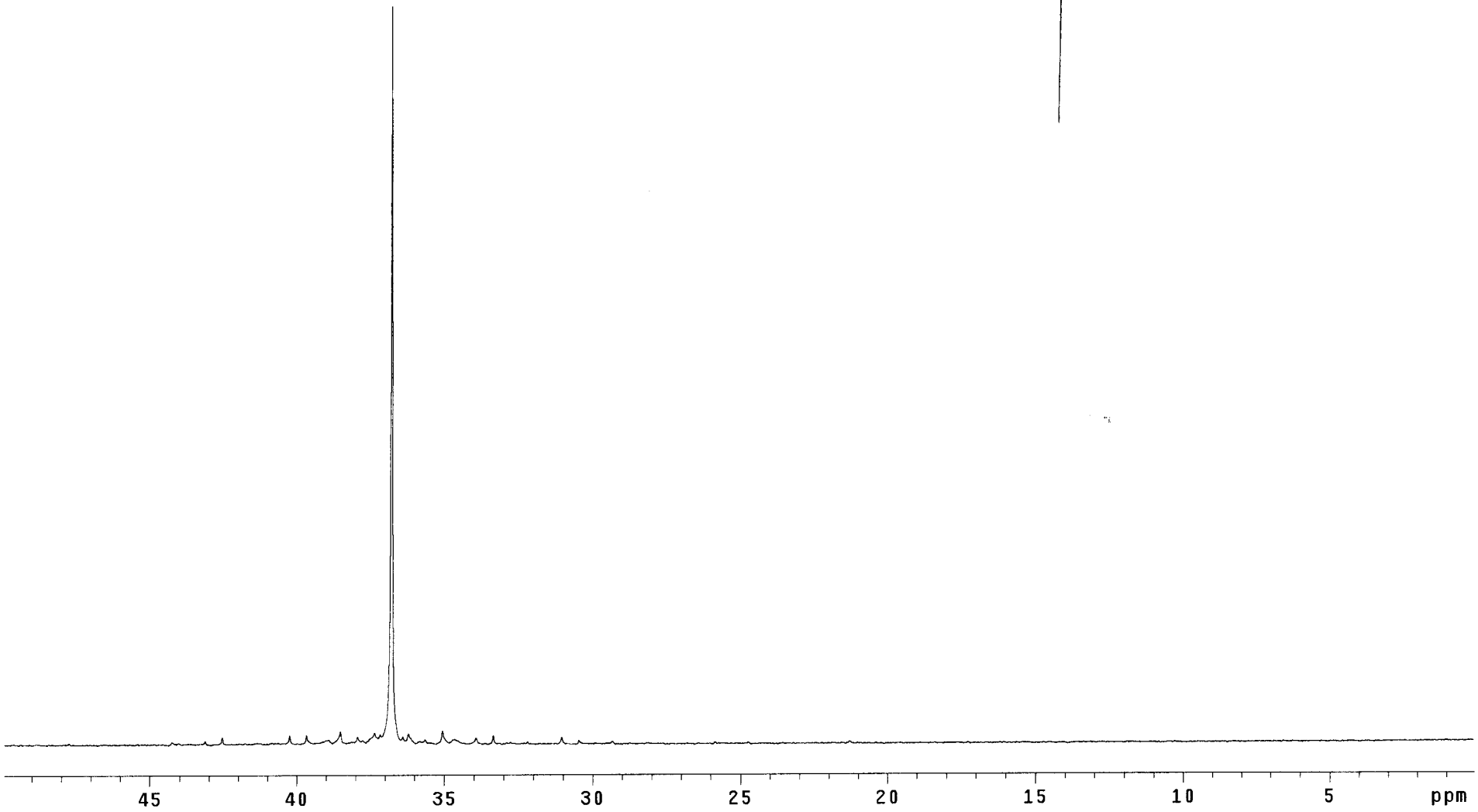
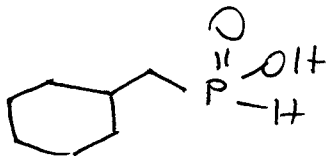


Table 2, entry 7



INDEX	FREQUENCY	PPM	HEIGHT
1	4759.269	39.182	56.0
2	4744.841	39.063	126.0
3	4731.894	38.956	120.7
4	4716.726	38.831	53.2
5	4219.537	34.738	56.0
6	4205.110	34.619	124.8
7	4191.792	34.510	119.3
8	4176.625	34.385	51.5



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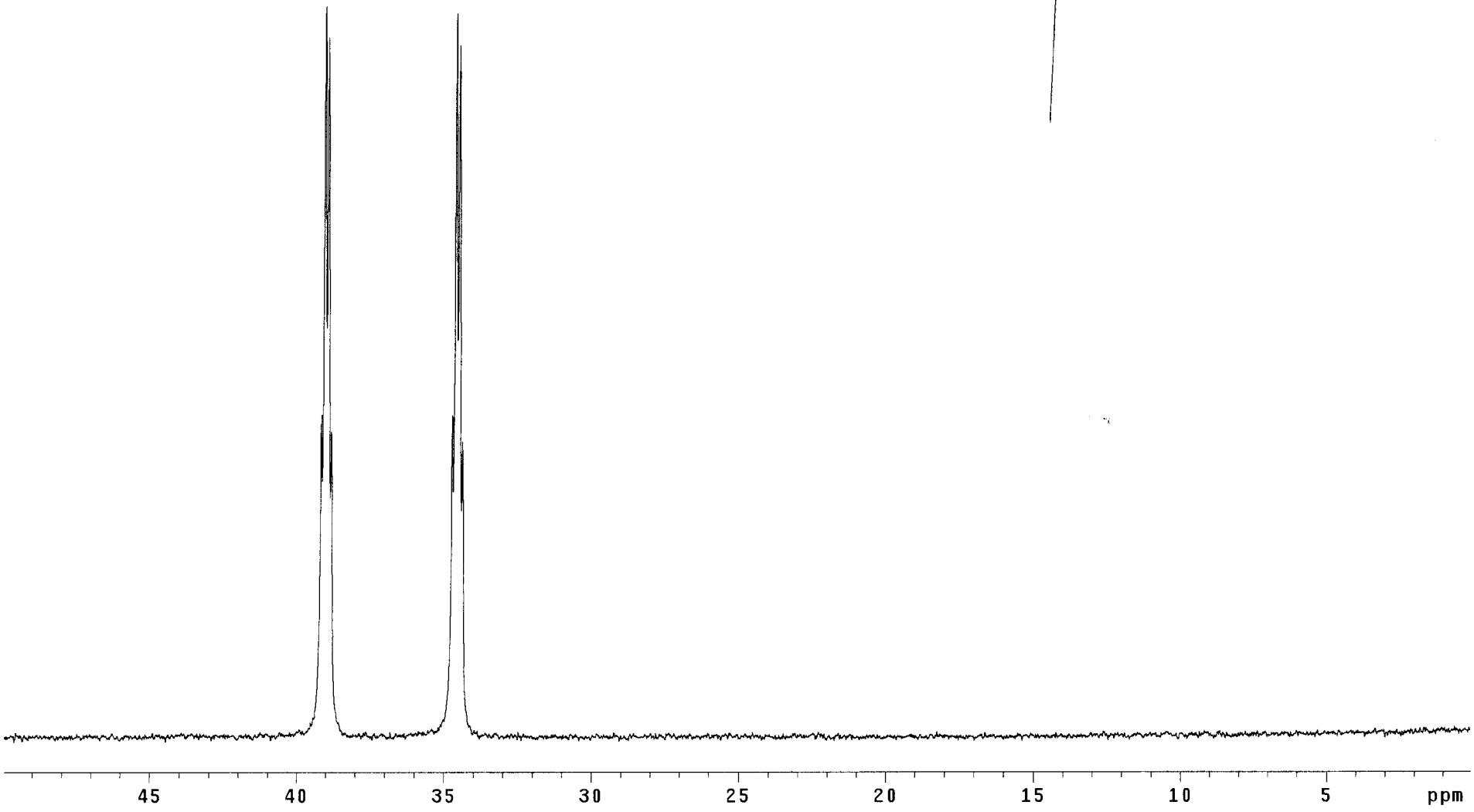


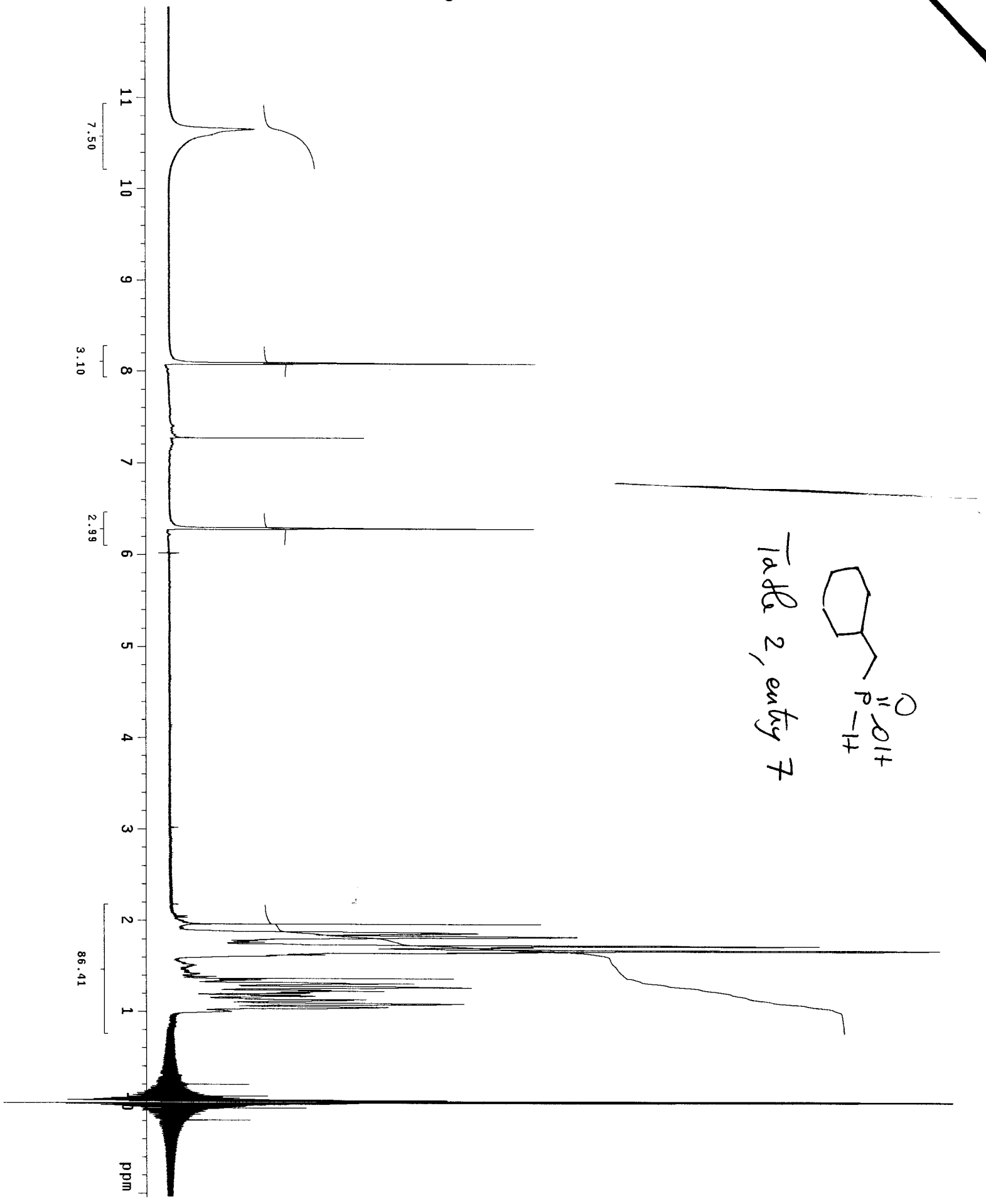
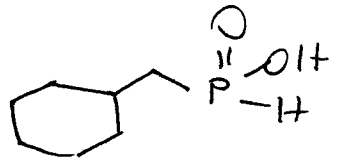


Table 2, entry 7
7.50
3.10
2.99
86.41
ppm

INDEX	FREQUENCY	PPM	HEIGHT
1	5843.917	77.454	68.5
2	5811.960	77.030	67.2
3	5780.002	76.606	67.2
4	2839.042	37.628	30.7
5	2766.777	36.670	10.3
6	2746.048	36.395	30.7
7	2605.838	34.537	73.0
8	2595.761	34.403	69.6
9	2395.091	31.744	32.0
10	1960.640	25.986	93.5
11	1953.731	25.894	82.9
12	0.000	0.000	69.0



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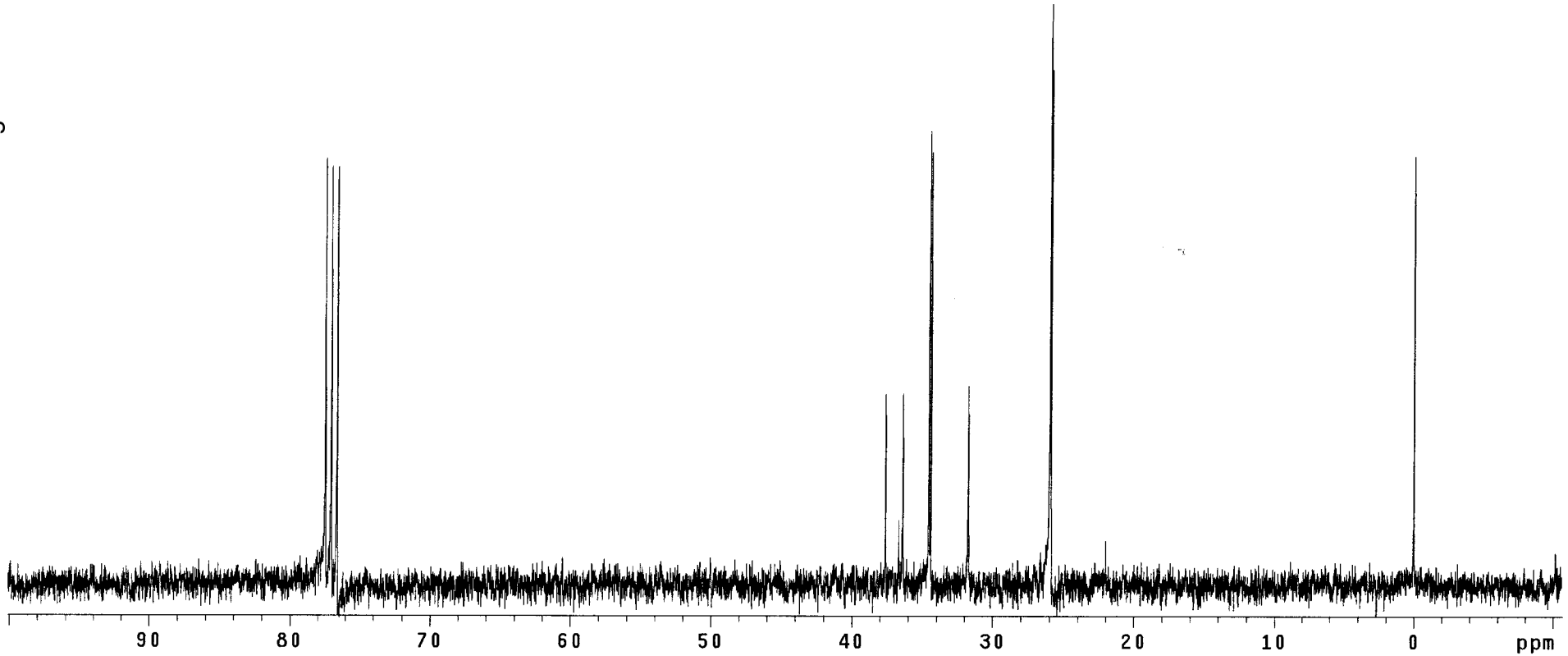
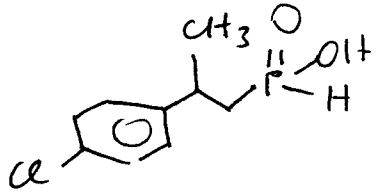


INDEX	FREQUENCY	PPM	HEIGHT
1	5246.840	43.196	7.2
2	5107.005	42.044	6.2
3	5037.088	41.469	8.6
4	4967.541	40.896	11.8
5	4898.363	40.327	11.4
6	4828.076	39.748	126.0
7	4757.789	39.169	11.2
8	4688.611	38.600	12.3
9	4618.694	38.024	8.6
10	4552.106	37.476	4.3
11	4408.942	36.297	7.2



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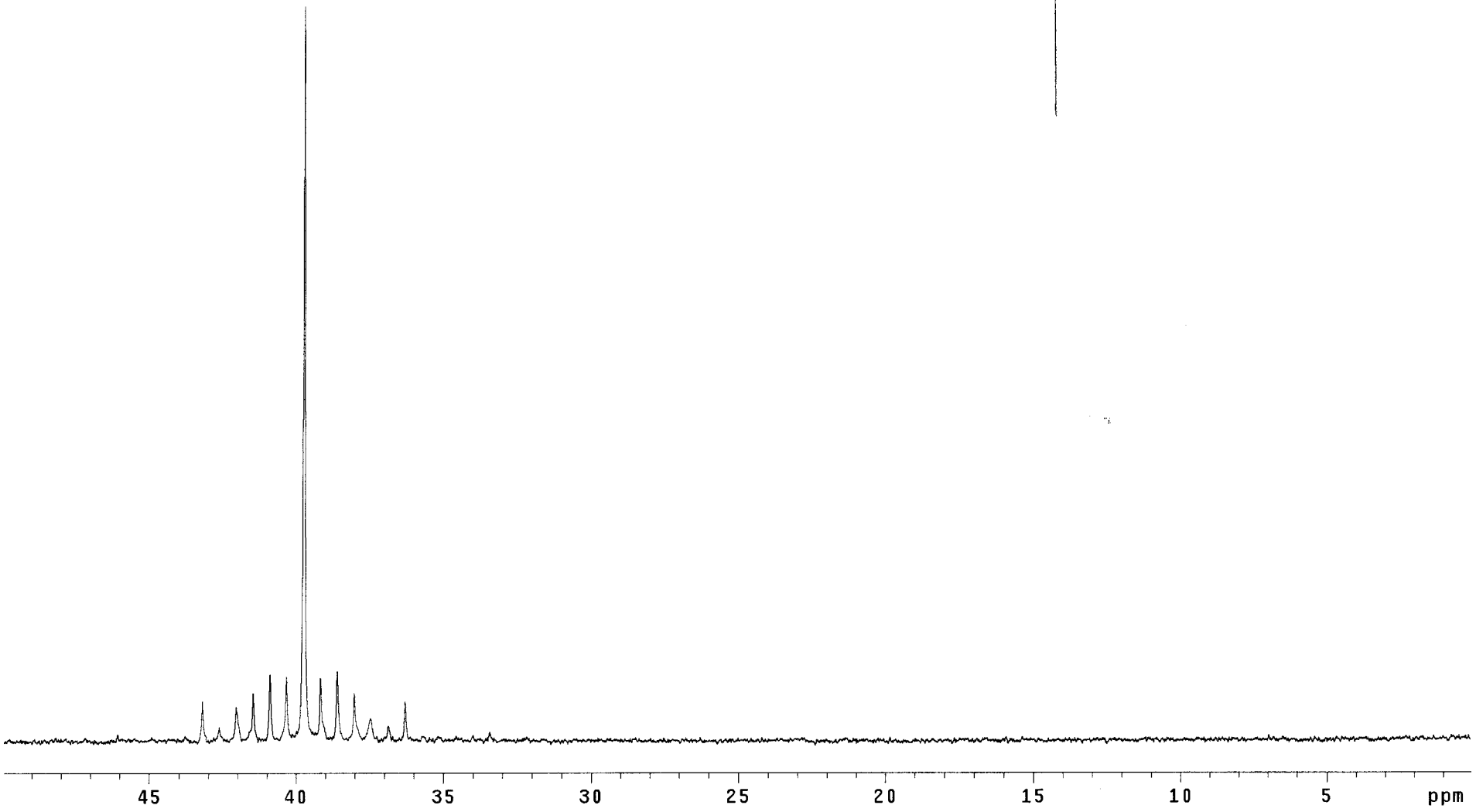
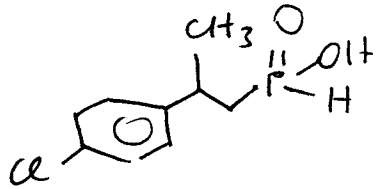


INDEX	FREQUENCY	PPM	HEIGHT
1	5125.132	42.194	54.6
2	5110.335	42.072	125.2
3	5098.127	41.971	123.3
4	5082.960	41.846	60.6
5	4576.152	37.674	58.4
6	4560.615	37.546	125.3
7	4548.777	37.449	126.0
8	4533.610	37.324	60.8



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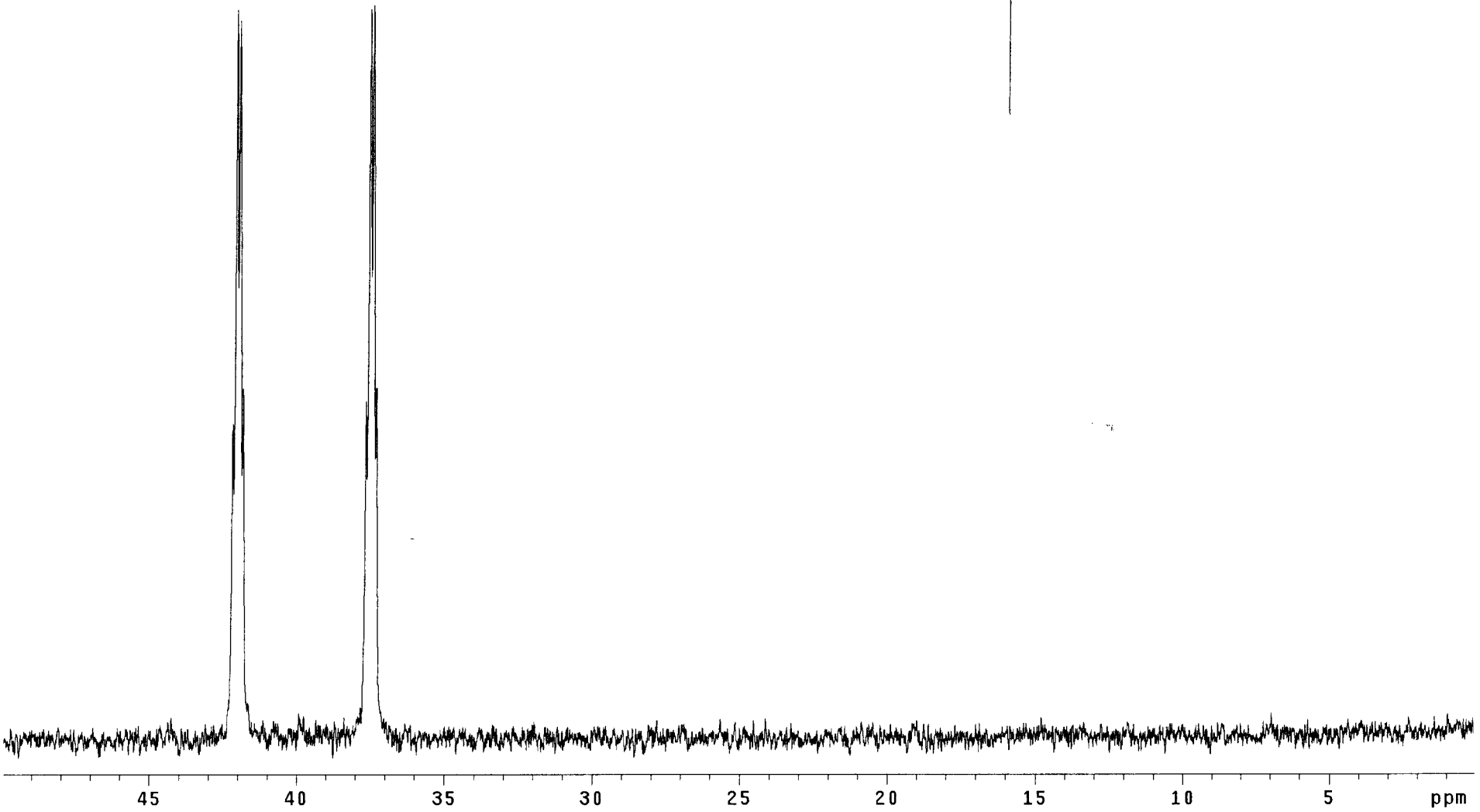
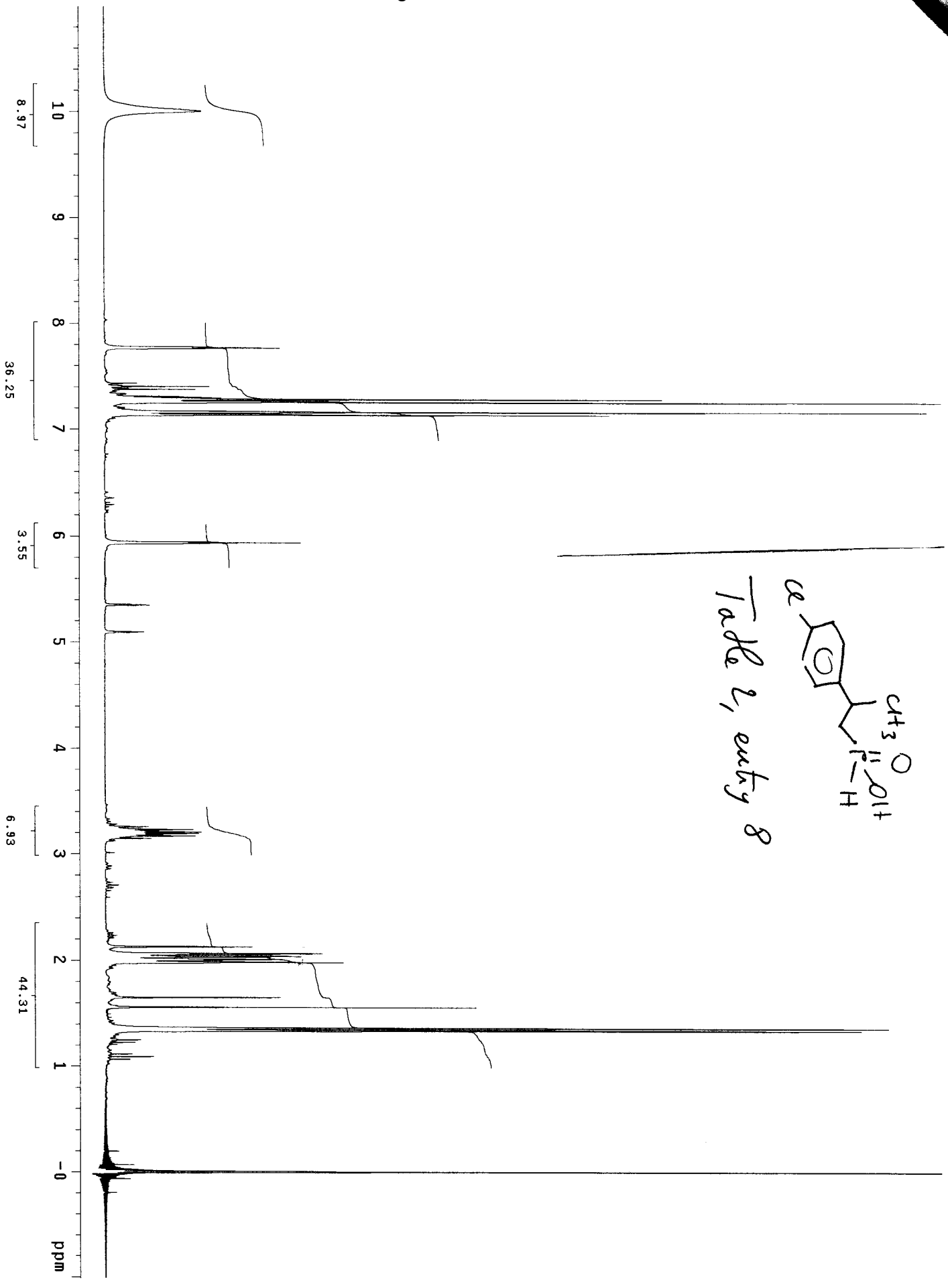
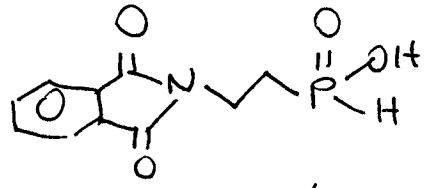



8.97
36.25
3.55
6.93
44.31
10
9
8
7
6
5
4
3
2
1
0
ppm
CH3 O Olt
P
H
a
Table 2, entry 8

INDEX	FREQUENCY	PPM	HEIGHT
1	3330.379	27.418	101.0



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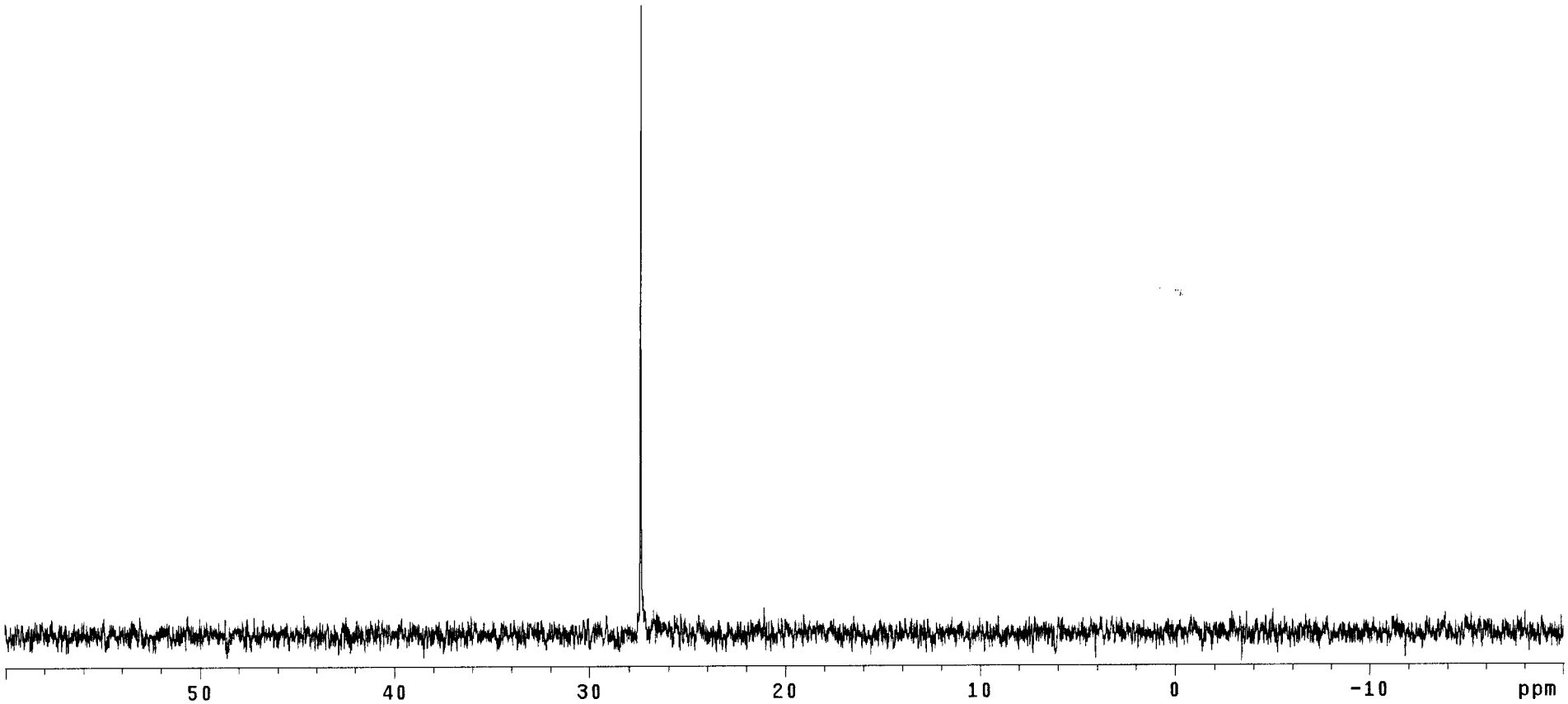
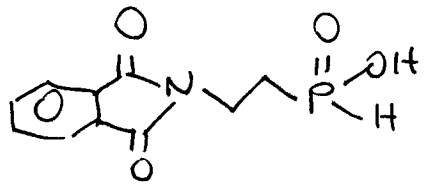


INDEX	FREQUENCY	PPM	HEIGHT
1	3595.830	29.603	17.4
2	3063.497	25.221	21.2



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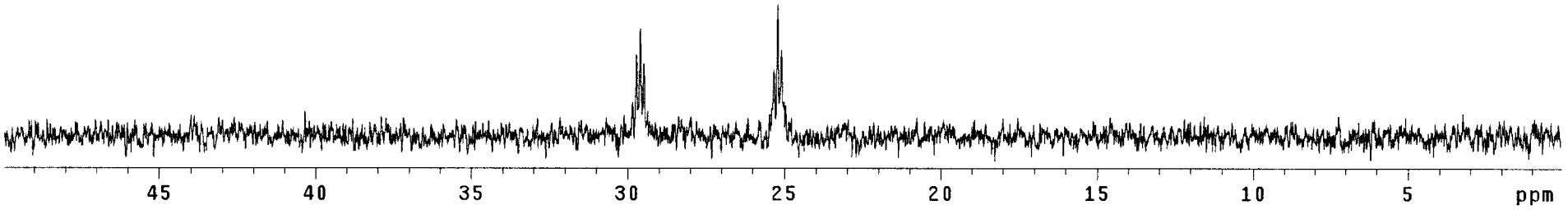
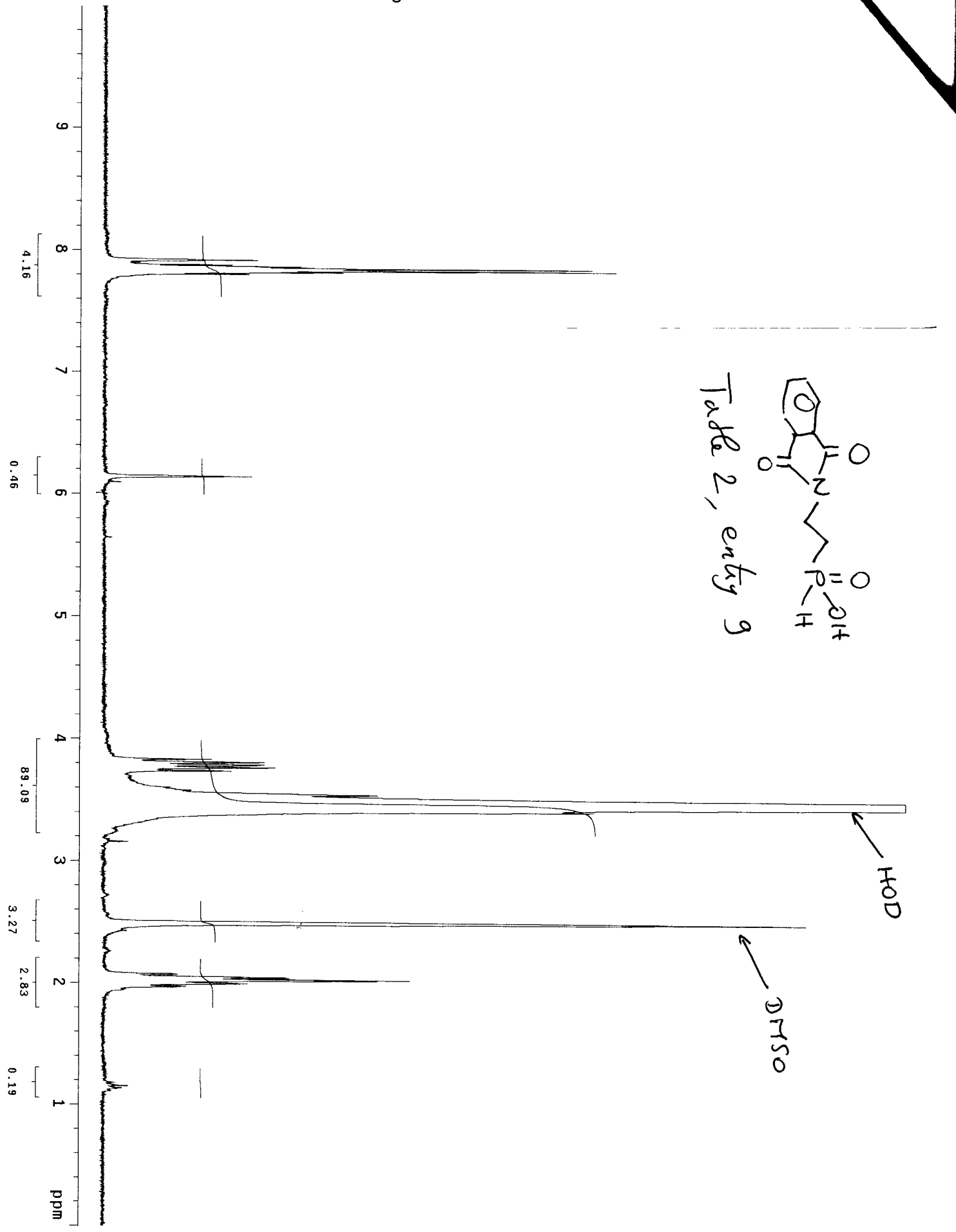



HOD
DMSO
Table 2, entry 9
ppm
4.16
0.46
89.09
3.27
2.83
0.19

INDEX	FREQUENCY	PPM	HEIGHT
1	12698.672	168.304	23.5
2	10190.722	135.064	38.5
3	9988.036	132.378	27.7
4	9334.778	123.720	40.6
5	3088.367	40.932	53.8
6	3067.350	40.654	166.8
7	3046.333	40.375	329.4
8	3025.316	40.096	383.3
9	3004.299	39.818	324.5
10	2983.570	39.543	159.9
11	2962.264	39.261	50.9
12	2369.754	31.408	14.1
13	2273.305	30.130	7.8
14	2184.630	28.954	9.7

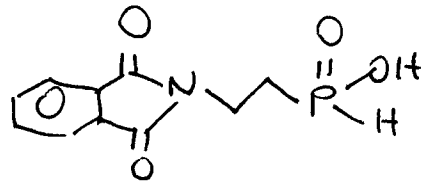
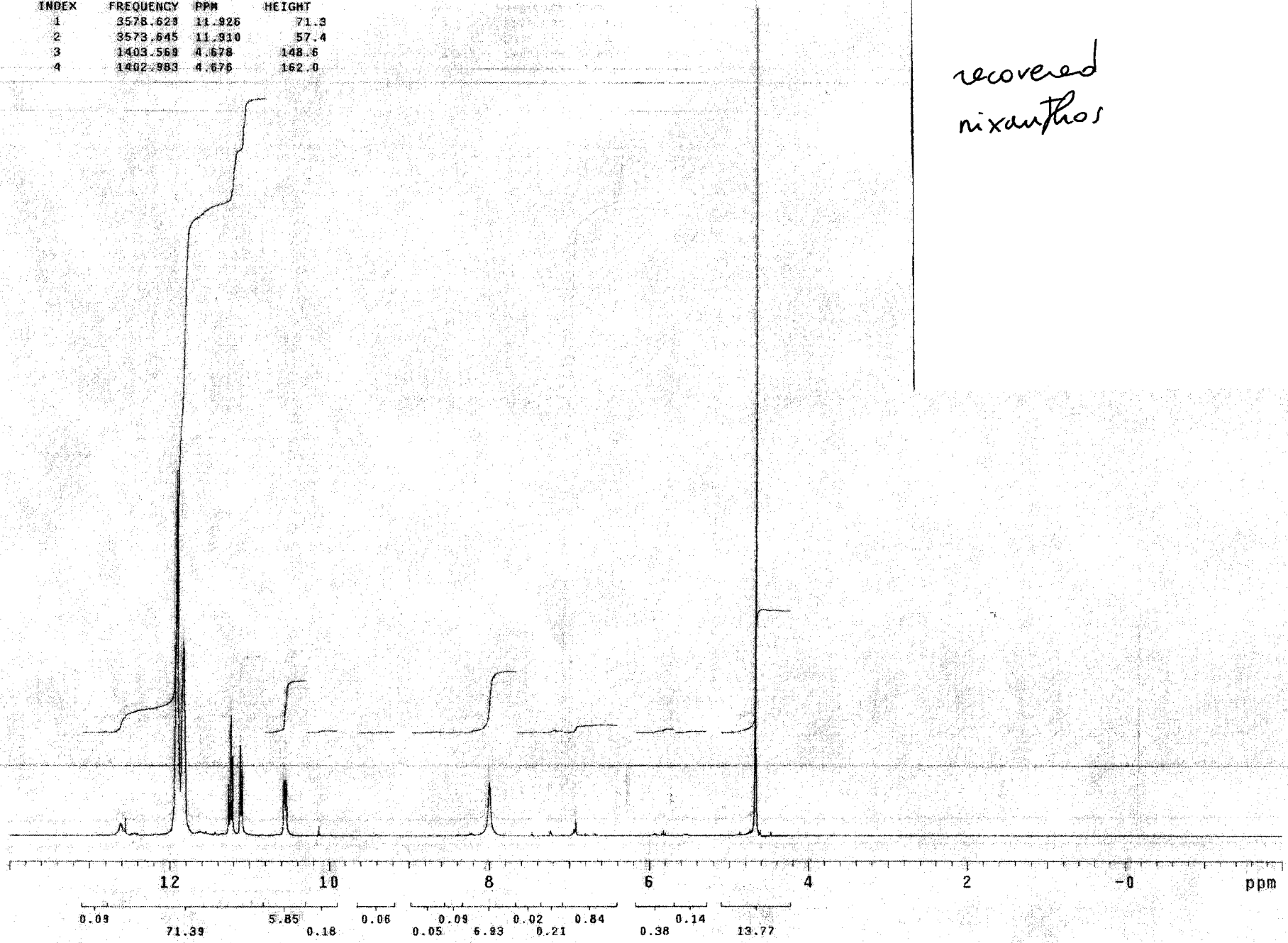


Table 2, entry 9

INDEX	FREQUENCY	PPM	HEIGHT
1	3578.628	11.926	71.3
2	3573.645	11.910	57.4
3	1403.569	4.678	148.6
4	1402.983	4.676	162.0

recovered
nixanthos



INDEX	FREQUENCY	PPM	HEIGHT
1	-2241.649	-18.455	126.0

recovered
nixanthos