

Supporting Information

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1. General Chemistry. All reactions were conducted in oven- or flame-dried glassware, under nitrogen. ^1H NMR spectra were recorded on a 300-MHz spectrometer. Chemical shifts for ^1H 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 (300 MHz spectrometer) and/or 36 MHz (90 MHz spectrometer), and chemical shifts reported (in parts per million) relative to external 85% phosphoric acid ($\delta = 0.0$ ppm). Radial chromatography was carried out using 2 or 4 mm layers of silica gel 60 PF₂₅₄ containing gypsum. Silica gel (200-300 mesh) 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 .

2. Reagents and Solvents. Stock solution (0.5 M) of concentrated EtOP(O)H_2 in reagent grade CH_3CN were also prepared and used for one month without any decomposition of the acid. Unless otherwise specified, HPLC or reagent grade solvents were used as received. Anhydrous toluene were distilled under N_2 from CaH_2 , anhydrous THF under N_2 from sodium benzophenone ketyl, and used immediately. $i\text{Pr}_2\text{NEt}$ was distilled under N_2 from CaH_2 and stored over activated 4Å molecular sieves.

3. ^{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 of the *H*-phosphinate esters are polar compounds and hydrolytically labile.

(3-Chloropropyl)-*H*-phosphinic acid (Scheme 2, compound 6).¹ The preparation of this compound was conducted as described in the literature.

EtOP(O)H₂. The preparation of this compound was conducted as described in the literature.² In a typical procedure, a solution of the hypophosphorous compound (10 mmol), alkoxy silane ((EtO)₂SiMe₂, 15 mmol), in reagent grade solvent (10 mL) was refluxed for 2 h under N₂. After cooling to room temperature, the mixture was used directly.

2-Bromophenyl-*H*-phosphinic acid (Scheme 3).³ The preparation of this compound was conducted as described in the literature.

Ethyl 2-bromophenyl-*H*-phosphinate (Scheme 3, compound 3). To the 2-bromophenyl-*H*-phosphinic acid (4.42 g, 20 mmol) in toluene (70 mL) was added tetraethyl orthosilicate (1.5 equiv, 6.25 g) under N₂ and the mixture was refluxed for 24 h. The solvent was removed by vacuo and the resulting oil was purified by column chromatography (silica, EtOAc/Hexanes 3:7, v/v) to afford the desired product as a yellow oil (3.2 g, 64%): ¹H NMR (CDCl₃, 300 MHz) δ 7.90-8.05 (m, 1H, *aro* CH), 7.66 (d, J = 589 Hz, 1H, H-P), 7.60-7.65 (m, 1H, *aro* CH), 7.40-7.60 (m, 2H, *aro* CH), 4.00-4.30 (m, 2H, -CH₂-O), 1.39 (t, J = 7 Hz, 3H, CH₃-); ¹³C NMR (CDCl₃, 75.45 MHz) δ 134.9 (d, J = 8 Hz), 134.5 (d, J = 2 Hz), 133.7 (d, J = 8 Hz), 130.0 (d, J = 136 Hz), 127.6 (d, J = 12 Hz), 125.0 (d, J = 8 Hz), 62.9 (d, J_{POC} = 6 Hz), 16.5 (d, J_{POCC} = 6 Hz); ³¹P NMR (CDCl₃, 121.47 MHz) δ 23.3 (d, J = 589 Hz); HRMS (EI⁺) calc for C₈H₁₀BrO₂P 247.9602, found 247.9600.

Ethyl (1,1-diethoxyethyl)-(2-bromobenzyl)phosphinate (Scheme 4).⁴ Neat Ciba-Geigy reagent ethyl (1,1-diethoxyethyl)-*H*-phosphinate (20 mmol, 4.2 g) was placed under vacuum in a dry two-neck flask. Anhydrous THF (70 mL) was then added under nitrogen. The flask was placed at -78 °C and deoxygenated under vacuum for 20 min. The reaction flask was back-filled with nitrogen, LiHMDS (1.0 M in THF, 20 mmol, 20 mL) was added at -78 °C. After 10 min, 2-bromobenzyl bromide (1 equiv, 20 mmol, 5 g).

After addition, the temperature of the solution was slowly allowed to warm to rt. After 3 h at rt, the reaction mixture was quenched with NH₄Cl/brine, extracted with EtOAc (3 x 20 mL), dried and concentrated. The resulting oil was purified by column chromatography (silica, EtOAc/Hexanes 30:70, v/v) to afford the desired product (61%): ¹H NMR (CDCl₃, 300 MHz) δ 7.54 (t, J = 8 Hz, 2H, *aro* CH), 7.20-7.30 (m, 1H, *aro* CH), 7.00-7.15 (m, 1H, *aro* CH), 3.95-4.15 (m, 2H, -CH₂-O-), 3.80-3.90 (m, 1H, -CH₂-O-), 3.60-3.75 (m, 3H, -CH₂-O-), 3.30-3.55 (m, 2H, C-CH₂-P), 1.56 (d, J = 11 Hz, 3H, CH₃-C-P-), 1.10-1.30 (m, 9H, CH₃-); ¹³C NMR (CDCl₃, 75.45 MHz) δ 133.0 (d, J = 2 Hz), 132.3 (d, J = 4 Hz), 131.9 (d, J = 9 Hz), 128.5 (d, J = 3 Hz), 127.5 (d, J = 3 Hz), 125.4 (d, J = 7 Hz), 101.5 (d, J_{PC} = 142 Hz), 62.1 (d, J_{POC} = 7 Hz), 58.6 (d, J_{PCOC} = 5 Hz), 57.9 (d, J_{PCOC} = 7 Hz), 33.3 (d, J_{PC} = 80 Hz), 29.9, 20.6 (d, J_{POCCC} = 12 Hz), 16.7 (d, J_{POCC} = 5 Hz), 15.6 (d, J_{PCOCC} = 21 Hz); ³¹P NMR (CDCl₃, 121.47 MHz) δ 44.4 (s); HRMS (EI⁺) calc for C₁₅H₂₄BrO₄P 379.0674, found 379.0654.

Cinnamyl-*H*-phosphinic acid (Scheme 5, compound 7).⁵ The preparation of this compound was conducted as described in the literature.

Ethyl cinnamyl-*H*-phosphinate (Scheme 5, compound 8). To the cinnamyl-*H*-phosphinic acid (23.6 g, 130 mmol) in toluene (430 mL) was added tetraethyl orthosilicate (1.5 equiv, 40.6 g) under N₂ and the mixture was refluxed for 24 h. The solvent was removed by vacuo and the resulting oil was purified by column chromatography (silica, EtOAc/Hexanes 3:7, v/v) to afford the desired product as a yellow oil (25.1 g, 92%): ¹H NMR (CDCl₃, 300 MHz) δ 7.20-7.40 (m, 5H, *aro* CH), 7.04 (d, J = 545 Hz, 1H, H-P), 6.53 (dd, J = 15, 6 Hz, 1H, -CH=CH-), 6.00-6.20 (m, 1H, -CH=CH-), 4.00-4.30 (m, 2H, -CH₂-O-), 2.79 (dd, J = 18, 8 Hz, 2H, -CH₂-P), 1.36 (t, J = 14, 7 Hz, 3H, CH₃-); ¹³C NMR (CDCl₃, 75.45 MHz) δ 136.6 (d, J = 4 Hz), 136.1 (d, J = 14 Hz), 128.8 (2C), 128.0, 126.4 (2C, d, J = 2 Hz), 116.9 (d, J = 10 Hz), 62.8 (d, J_{POC} = 7 Hz), 34.2 (d, J_{PC} = 90 Hz), 16.5 (d, J_{POCC} = 6 Hz); ³¹P NMR (CDCl₃, 121.47 MHz) δ 36.7 (d, J = 544 Hz); HRMS (EI⁺) calc for C₁₁H₁₅O₂P 210.0810, found 210.0805.

Iodide Heck precursor (Scheme 5, compound 9). Ethyl cinnamyl-*H*-phosphinate (8

mmol, 1.68 g), 2-iodoaniline (1.2 equiv, 9.6 mmol, 2.1 g) and paraformaldehyde (1.2 equiv, 9.6 mmol, 317.1 mg) in toluene (40 mL) were refluxed for 16 h. After cooling to room temperature, the mixture was concentrated and the resulting oil was diluted in EtOAc (60 mL) and washed with brine (1 x 20 mL). The organic layer was dried and concentrated. The resulting oil was purified by column chromatography (silica, EtOAc/Hexanes 6:4, v/v) to afford the desired product as a yellow oil (1.6 g, 46%): ^1H NMR (CDCl_3 , 300 MHz) δ 7.66 (d, $J = 8$ Hz, 1H, *aro* CH), 7.15-7.35 (m, 6H, *aro* CH), 6.40-6.65 (m, 3H, *aro* CH and -CH=CH-), 6.10-6.30 (m, 1H, -CH=CH-), 4.40-4.55 (m, 1H, -NH-), 4.15-4.30 (m, 2H, -CH₂-O-), 3.40-3.65 (m, 2H, -N-CH₂-P), 2.88 (dd, $J = 18, 7$ Hz, 2H, -CH₂-P), 1.38 (t, $J = 14, 7$ Hz, 3H, CH₃-); ^{13}C NMR (CDCl_3 , 75.45 MHz) δ 146.7 (d, $J = 9$ Hz), 139.4, 136.7 (d, $J = 3$ Hz), 135.7 ($J = 13$ Hz), 129.7, 128.8, 128.0, 126.5 (d, $J = 2$ Hz), 120.2 (2C), 118.2 (d, $J = 9$ Hz), 111.4 (2C), 86.2, 61.8 (d, $J_{\text{POC}} = 7$ Hz), 41.6 (d, $J_{\text{PC}} = 100$ Hz), 32.8 (d, $J_{\text{PC}} = 89$ Hz), 17.0 (d, $J_{\text{POCC}} = 5$ Hz); ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 48.7 (s); HRMS (EI^+) calc for $\text{C}_{18}\text{H}_{21}\text{INO}_2\text{P}$ 441.0355, found 441.0355.

Ethyl allyl-*H*-phosphinate (Scheme 6, compound 11).⁶ The preparation of this compound was conducted as described in the literature.

Ethyl allyl-*N*-benzyl(aminomethyl)phosphinate (Scheme 6, compound 12a). To compound **11** (12.5 mmol) in toluene (60 mL) was added 1,3,5-tribenzylhexahydro-1,3,5-triazine (0.4 equiv, 15 mmol, 2.72 g). After 16 h of reflux, the mixture was concentrated and the resulting oil was diluted in EtOAc (60 mL) and washed with brine (1 x 20 mL). The organic layer was dried and concentrated. The resulting oil was purified by column chromatography (silica, EtOAc/Hexanes 3:7, v/v) to afford the desired product as a yellow oil (2.36 g, 57%): ^1H NMR (CDCl_3 , 300 MHz) δ 7.20-7.40 (m, 5H, *aro* CH), 5.60-5.85 (m, 1H, -CH=), 5.05-5.20 (m, 2H, CH₂=), 4.00-4.25 (m, 2H, -CH₂-O-), 3.82 (s, 2H, -CH₂-Ph), (d, $J = \text{Hz}$, 2H, -P-CH₂-N), (dd, $J = \text{Hz}$, 2H, P-CH₂-C), (t, $J = 14, 7$ Hz, 3H, CH₃-); ^{13}C NMR (CDCl_3 , 75.45 MHz) δ 139.5, 128.7, 128.5, 127.9, 127.8, 127.5, 120.5, 120.3, 61.1 (d, $J_{\text{POC}} = 6$ Hz), 55.3 (d, $J_{\text{PCNC}} = 16$ Hz), 45.4 (d, $J_{\text{PC}} = 106$ Hz), 33.1 (d, $J_{\text{PC}} = 88$ Hz), 16.8 (d, $J_{\text{POCC}} = 6$ Hz); ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 51.1 (s).

Ethyl ((N-benzyl)aminomethyl)-cinnamylphosphinate (Scheme 6, compound 12b).

To compound **8** (12.5 mmol) in toluene (60 mL) was added 1,3,5-tribenzylhexahydro-1,3,5-triazine (0.4 equiv, 15 mmol, 2.72 g). After 16 h of reflux, the mixture was concentrated and the resulting oil was diluted in EtOAc (60 mL) and washed with brine (1 x 20 mL). The organic layer was dried and concentrated. The resulting oil was purified by column chromatography (silica, EtOAc/Hexanes 3:7, v/v) to afford the desired product as a yellow oil (2.36 g, 83%): ^1H NMR (CDCl_3 , 300 MHz) δ 7.20-7.35 (m, 10H, *aro* CH), 6.49 (dd, $J = 16, 4$ Hz, 1H, -CH=), 6.00-6.20 (m, 1H, CH=), 4.00-4.20 (m, 2H, -CH₂-O-), 3.84 (s, 2H, -CH₂-Ph), 2.91 (d, $J = 11$ Hz, 2H, -P-CH₂-N), 2.83 (dd, $J = 18, 8$ Hz, 2H, P-CH₂-C), 1.31 (t, $J = 14, 7$ Hz, 3H, CH₃-); ^{13}C NMR (CDCl_3 , 75.45 MHz) δ 139.5, 137.1, 137.0, 135.1, 134.9, 128.8, 128.7, 128.5, 127.8, 127.5, 126.5, 126.4, 119.3, 119.1, 61.2 (d, $J_{\text{POC}} = 7$ Hz), 55.4 (d, $J_{\text{PCNC}} = 17$ Hz), 45.6 (d, $J_{\text{PC}} = 106$ Hz), 32.4 (d, $J_{\text{PC}} = 88$ Hz), 16.9 (d, $J_{\text{POCC}} = 6$ Hz); ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 51.3 (s).

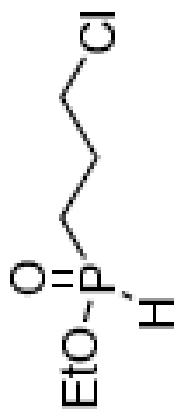
Cyclization attempt from compound 12a. To the ethyl allyl-N-benzyl(aminomethyl)-*H*-phosphinate (1 mmol, 315 mg) in DMSO (20 mL) were added Pd(OAc)₂ (5 mol%, 11.2 mg) and NaOAc (2 equiv, 164.1 mg). The flask was flushed with O₂ and equipped with an O₂ balloon. The reaction mixture was stirred at 80 °C for 60 h. The mixture was then cooled to room temperature, diluted with ether (10 mL) and THF (10 mL) and washed with brine (10 mL). The layers were separated and the aqueous layer was extracted with ether (2 x 10 mL). The combined organic layers were dried and concentrated.

References

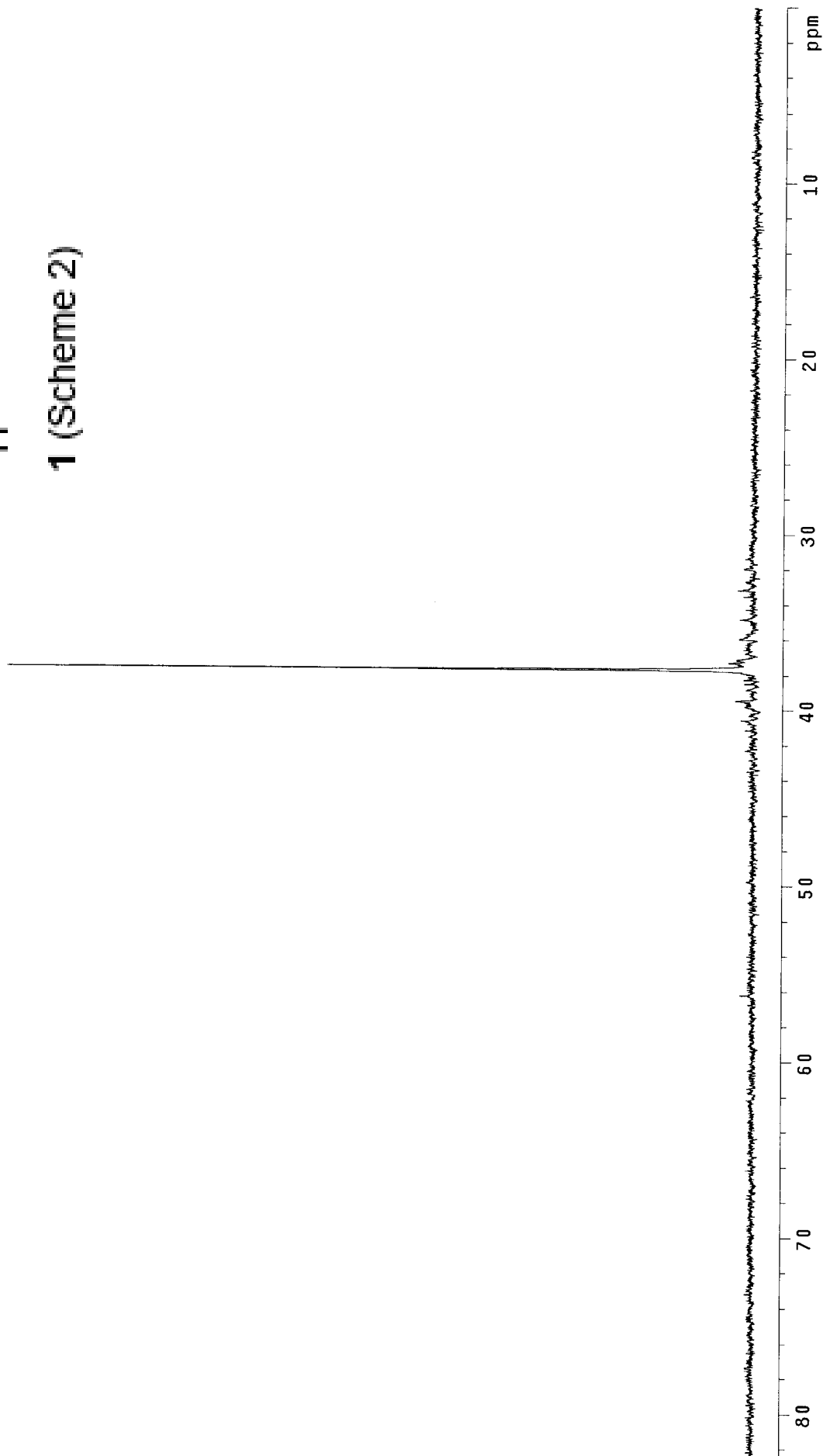
- [1] Deprèle, S.; Montchamp, J.-L. *J. Org. Chem.*, 2001, **66**, 6745.
- [2] Deprèle, S.; Montchamp, J.-L. *J. Organomet. Chem.*, 2002, **643-644**, 154.
- [3] Montchamp, J.-L.; Dumond, Y. R. *J. Am. Chem. Soc.*, 2001, **123**, 510.
- [4] Abrunhosa-Thomas, I.; Sellers, C.E.; Montchamp, J.-L. *J. Org. Chem.*, 2007, **72**,
- [5] Bravo-Altamirano, K.; Montchamp, J.-L. *Org. Lett.*, 2006, **8**, 4169.
- [6] Abrunhosa-Thomas, I.; Ribiere, P.; Adcock, A.C.; Montchamp, J.-L. *Synthesis*, 2006, 325.

INDEX	FREQUENCY	PPM	HEIGHT
1	4579.258	37.701	126.0

³¹P-NMR (¹H decoupled)

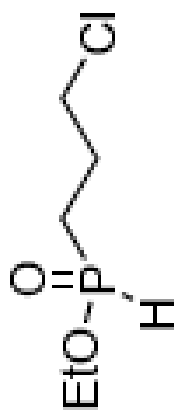


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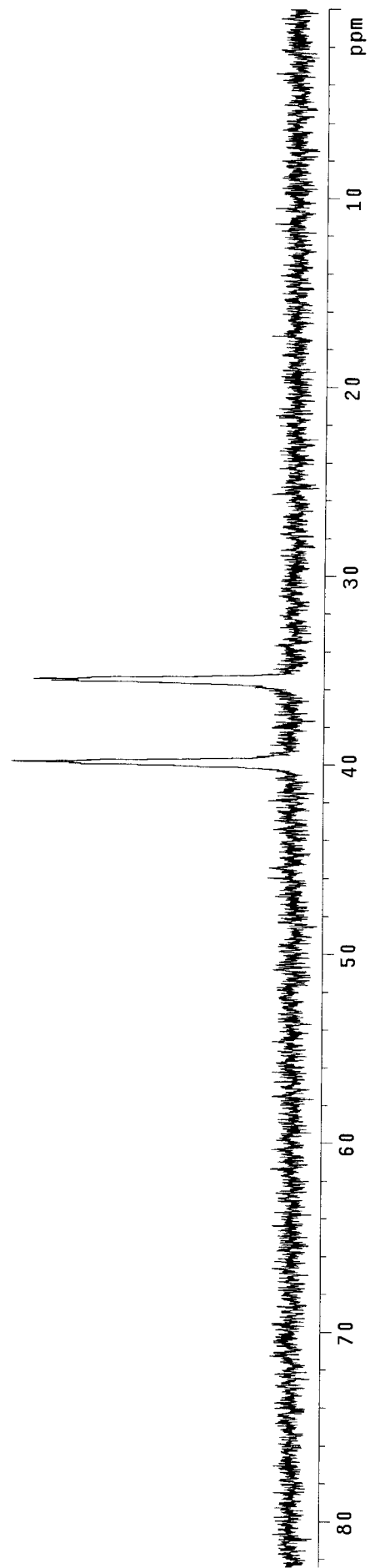


INDEX	FREQUENCY	PPM	HEIGHT
1	4846.888	39.904	43.9
2	4314.881	35.524	40.7

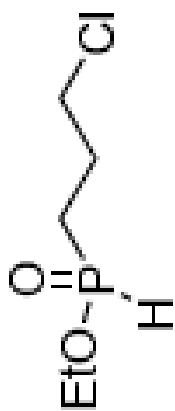
³¹P-NMR (1H coupled)



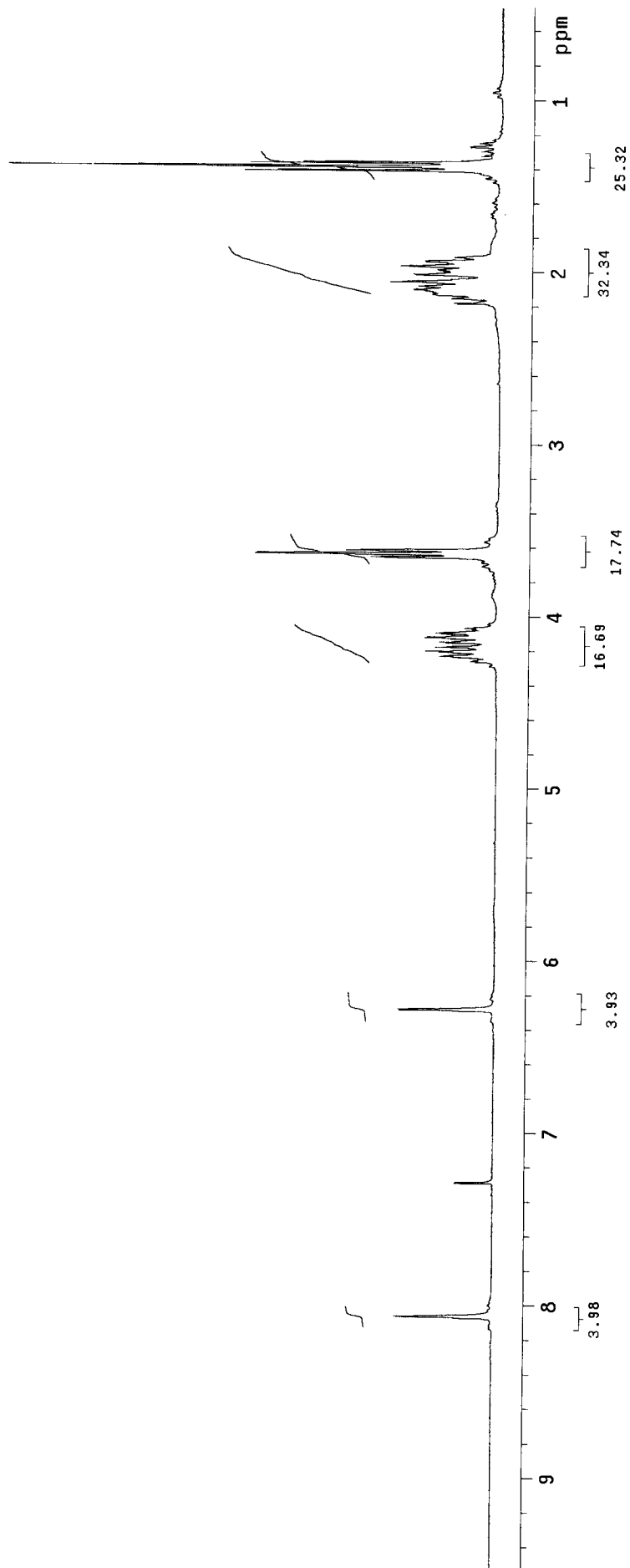
1 (Scheme 2)



¹H-NMR



1 (Scheme 2)

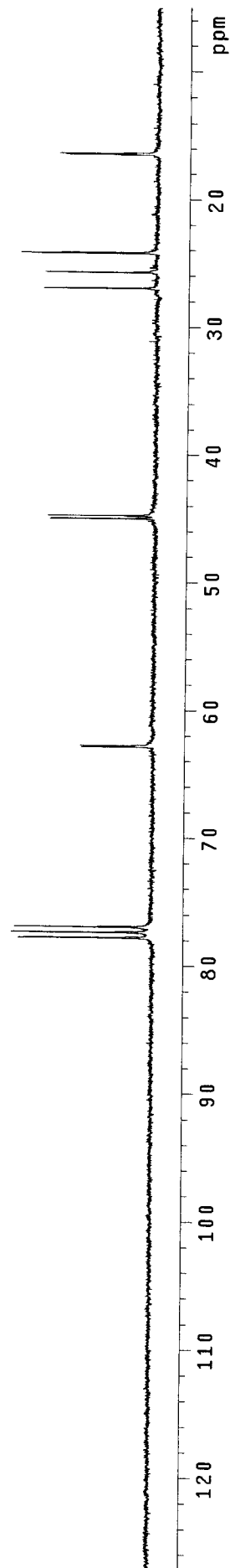


¹³C-NMR

INDEX	FREQUENCY	PPM	HEIGHT
1	5869.954	77.801	20.9
2	5837.997	77.378	22.0
3	5806.040	76.954	21.5
4	4743.970	62.877	11.3
5	4737.061	62.786	11.5
6	3986.015	45.011	16.5
7	3378.453	44.779	16.9
8	2035.679	26.981	17.8
9	1940.960	25.726	17.6
10	1828.678	24.238	20.1
11	1826.663	24.211	21.4
12	1242.798	16.472	14.6
13	1236.752	16.392	15.6



1 (Scheme 2)

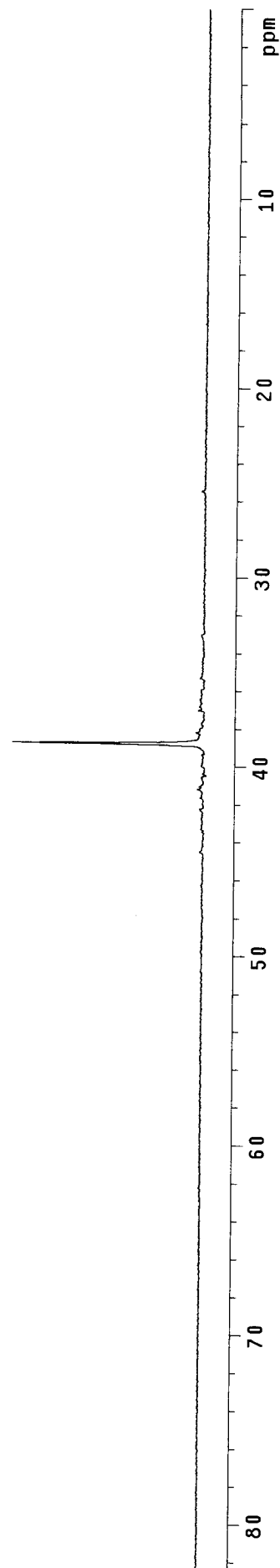


INDEX	FREQUENCY	PPM	HEIGHT
1	4708.998	38.769	29.9

^{31}P -NMR (^1H decoupled)



2 (Equation 2)

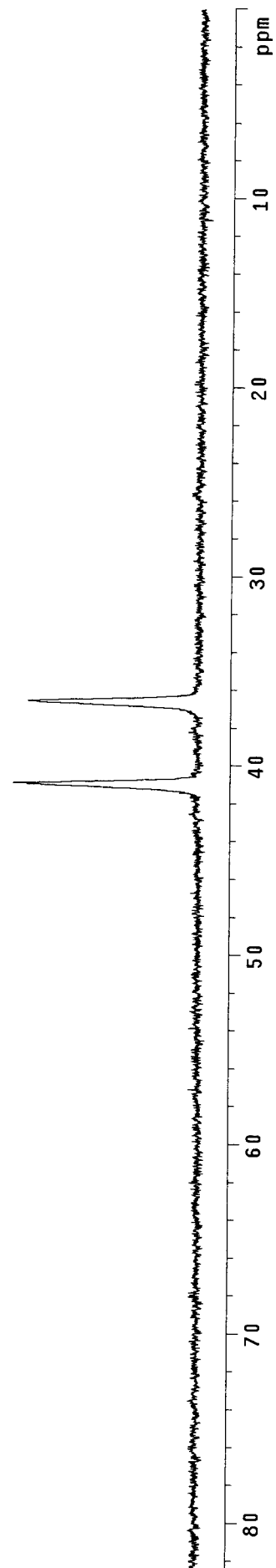


INDEX	FREQUENCY	PPM	HEIGHT
1	4376.231	40.969	29.1
2	4445.846	36.603	26.9

³¹P-NMR (1H coupled)



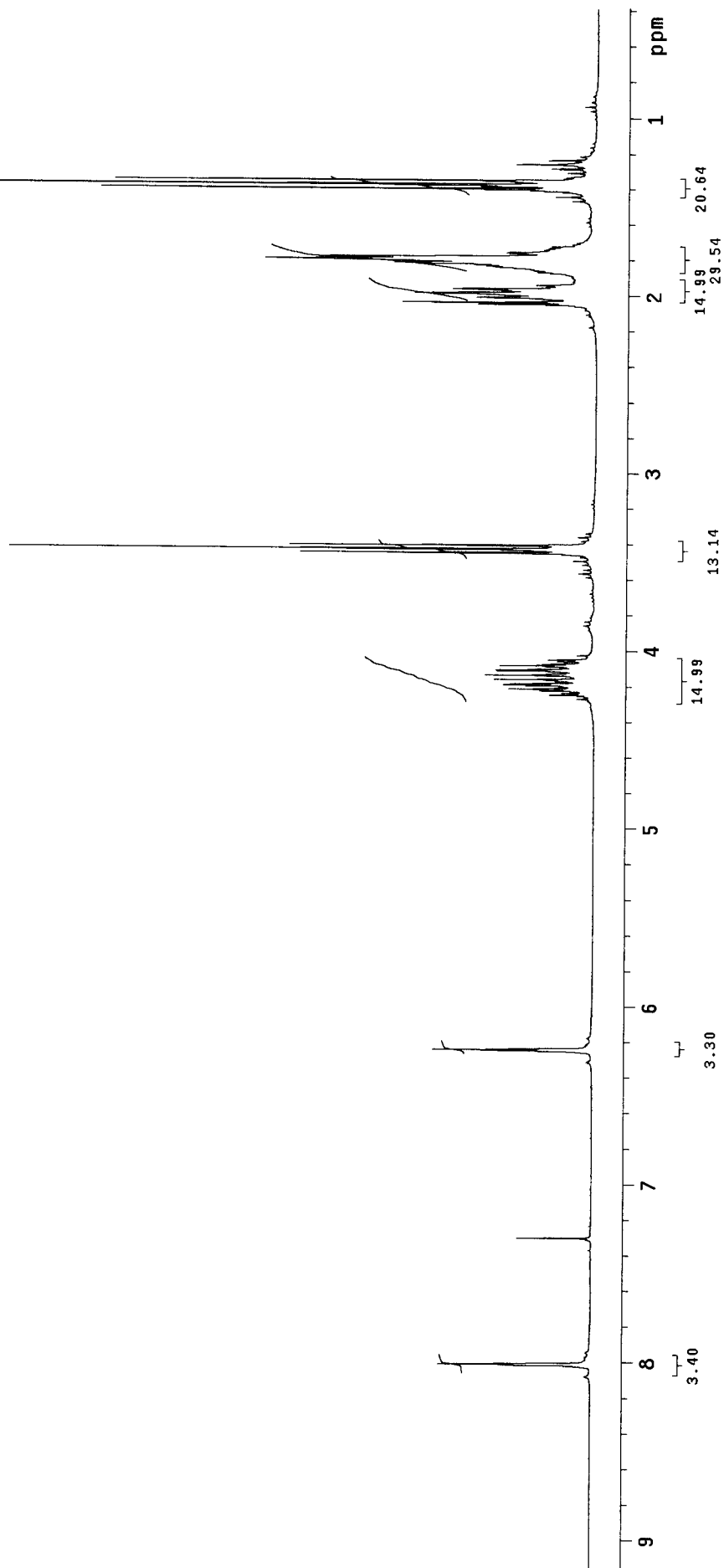
2 (Equation 2)



$^1\text{H-NMR}$



2 (Equation 2)

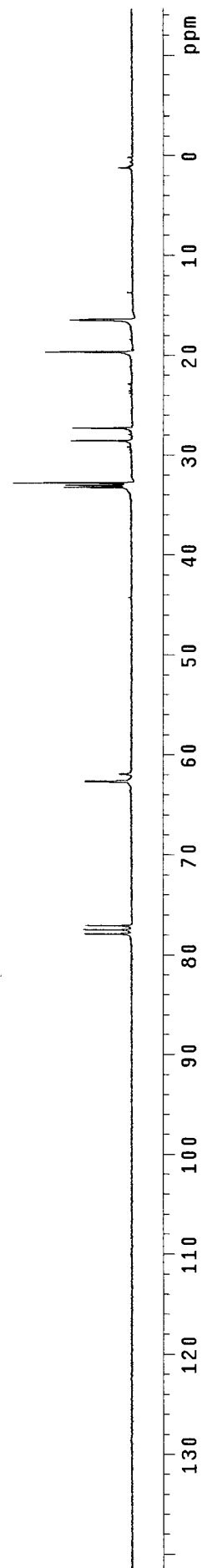


¹³C-NMR

INDEX	FREQUENCY PPM	HEIGHT
1	5877.151	7.4
2	5844.906	7.5
3	5812.949	7.2
4	4730.727	7.3
5	4723.817	7.3
6	2506.111	10.7
7	2490.852	10.4
8	2473.002	18.6
9	2152.279	9.6
10	2058.136	9.3
11	1480.893	13.7
12	1241.934	9.8
13	1236.176	8.2

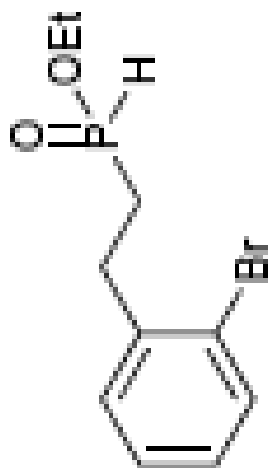


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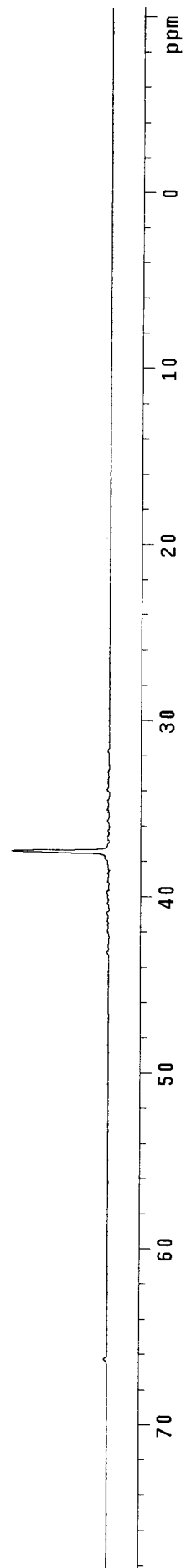


INDEX	FREQUENCY	PPM	HEIGHT
1	4546.619	37.432	15.2

³¹P-NMR (1H decoupled)

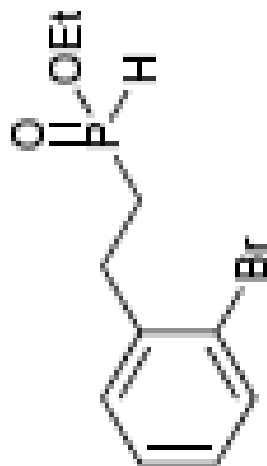


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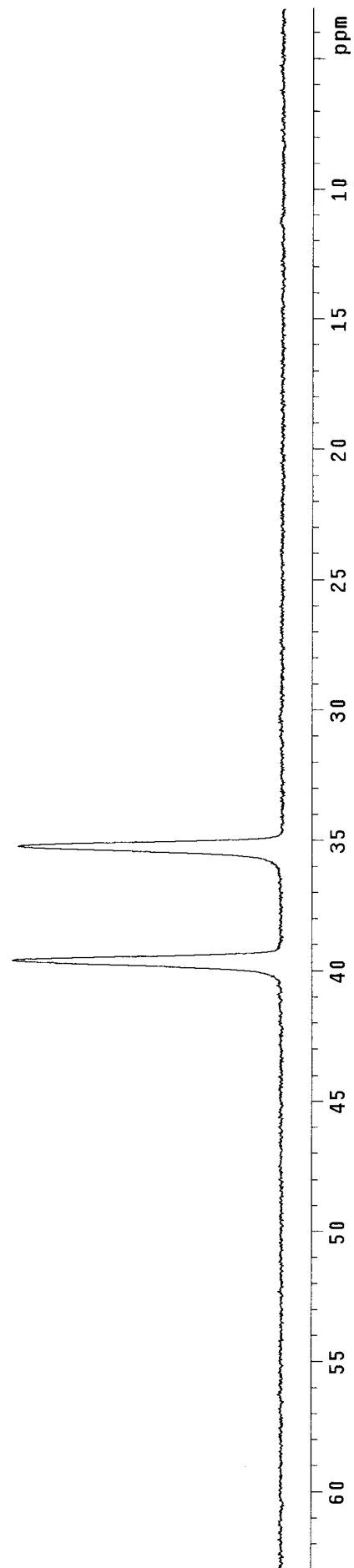


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1	4817.116	39.659	41.9
2	4284.690	35.276	41.1

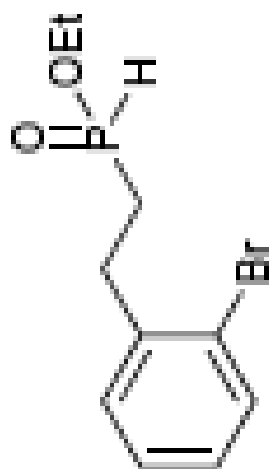
³¹P-NMR (¹H coupled)



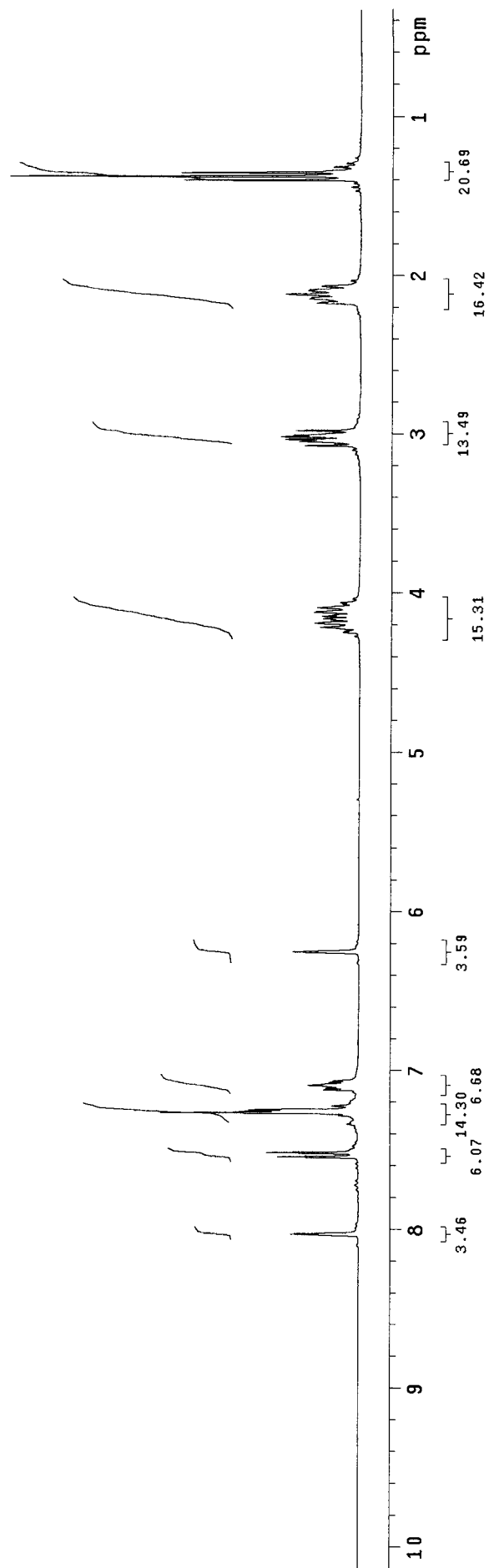
5 (Equation 3)



¹H-NMR

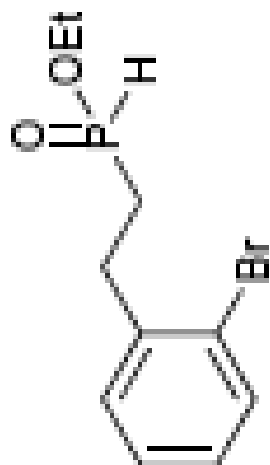


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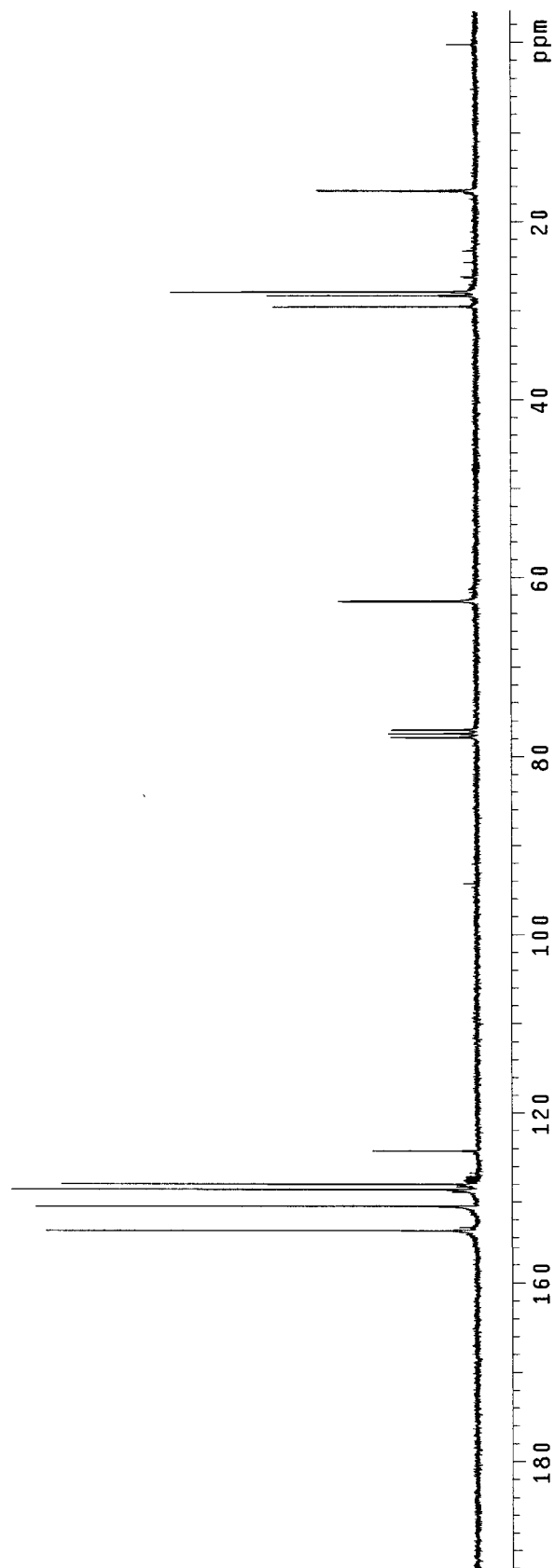


$^{13}\text{C-NMR}$

INDEX	FREQUENCY	PPM
1	10544.326	139.756
2	10528.491	139.547
3	10050.862	133.216
4	9843.861	130.472
5	9702.218	128.595
6	9657.013	127.996
7	9375.157	124.260
8	5875.424	77.874
9	5843.179	77.447
10	5811.222	77.023
11	4732.742	62.729
12	4725.833	62.637
13	2229.437	29.549
14	2137.309	28.328
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16	1247.692	16.537
17	1241.646	16.457

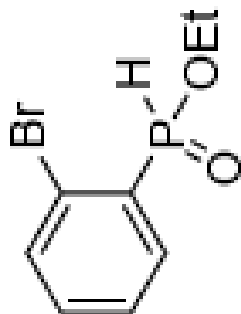


5 (Equation 3)

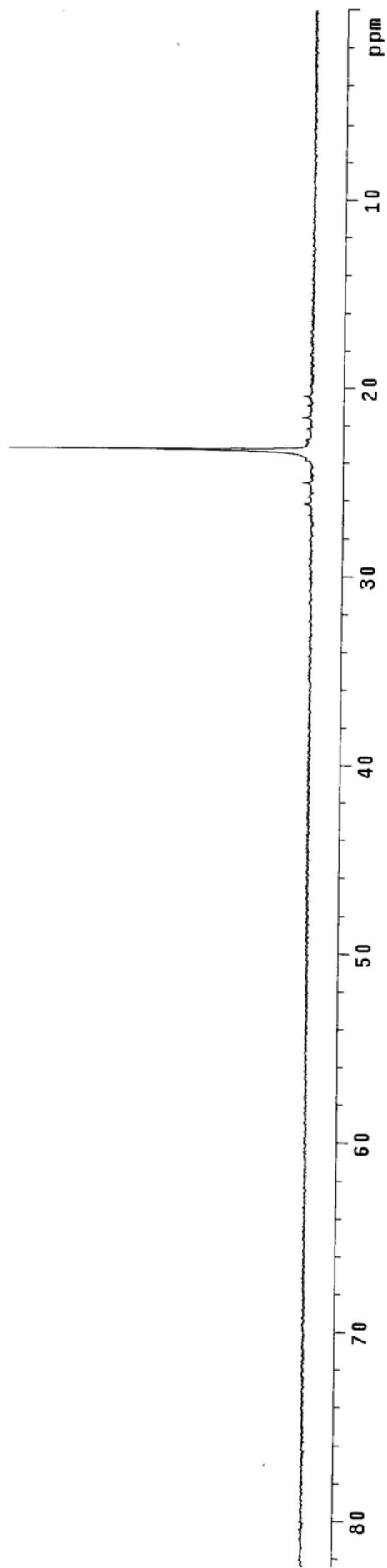


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1	2831.025	23.308
		47.5

³¹P-NMR (¹H decoupled)

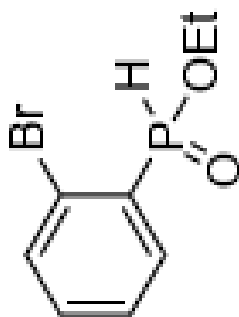


3 (Scheme 3)

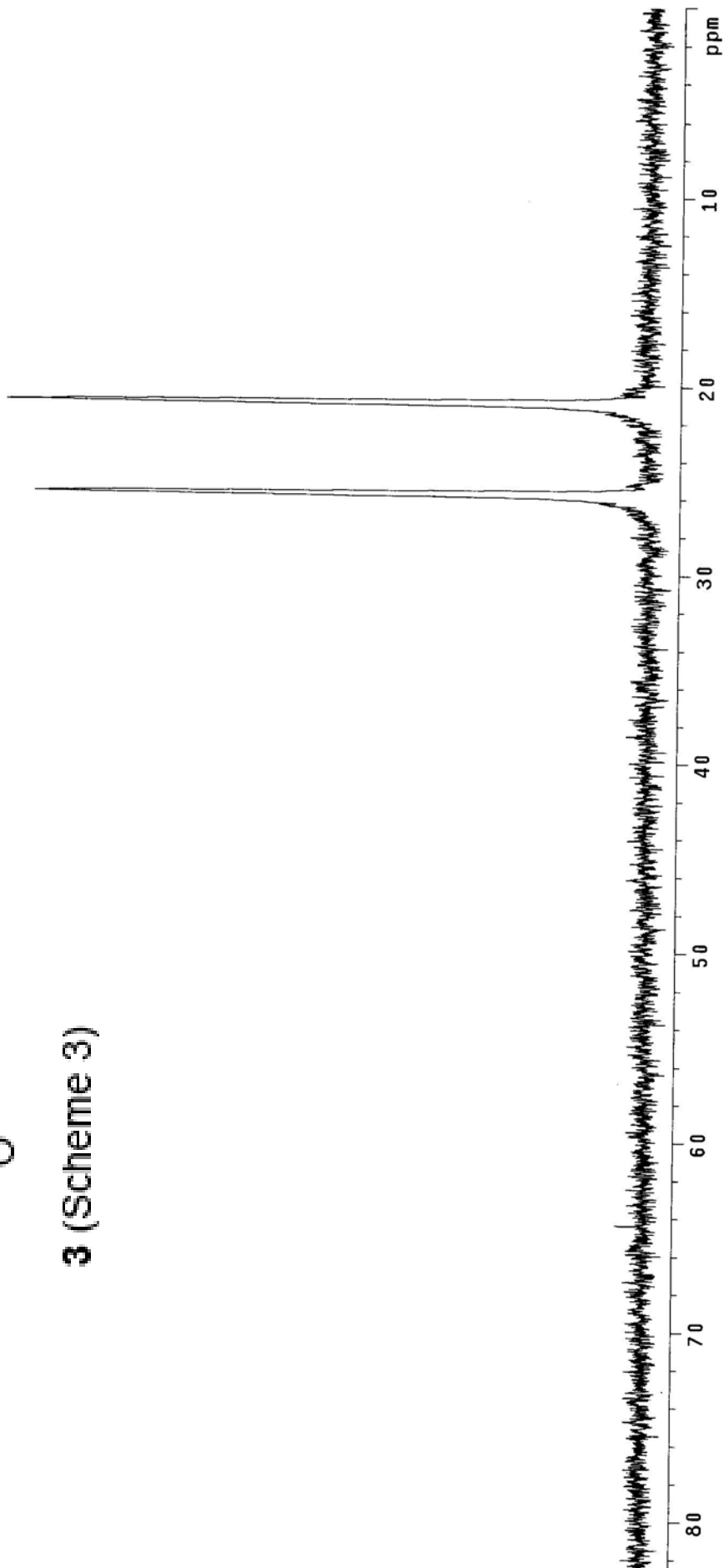


INDEX	FREQUENCY	PPM	HEIGHT
1	3131.713	25.783	96.5
2	2542.169	20.930	101.0

³¹P-NMR (¹H coupled)

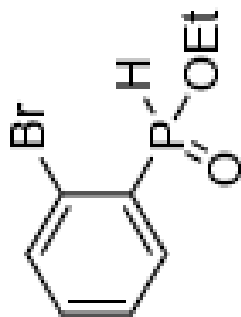


3 (Scheme 3)

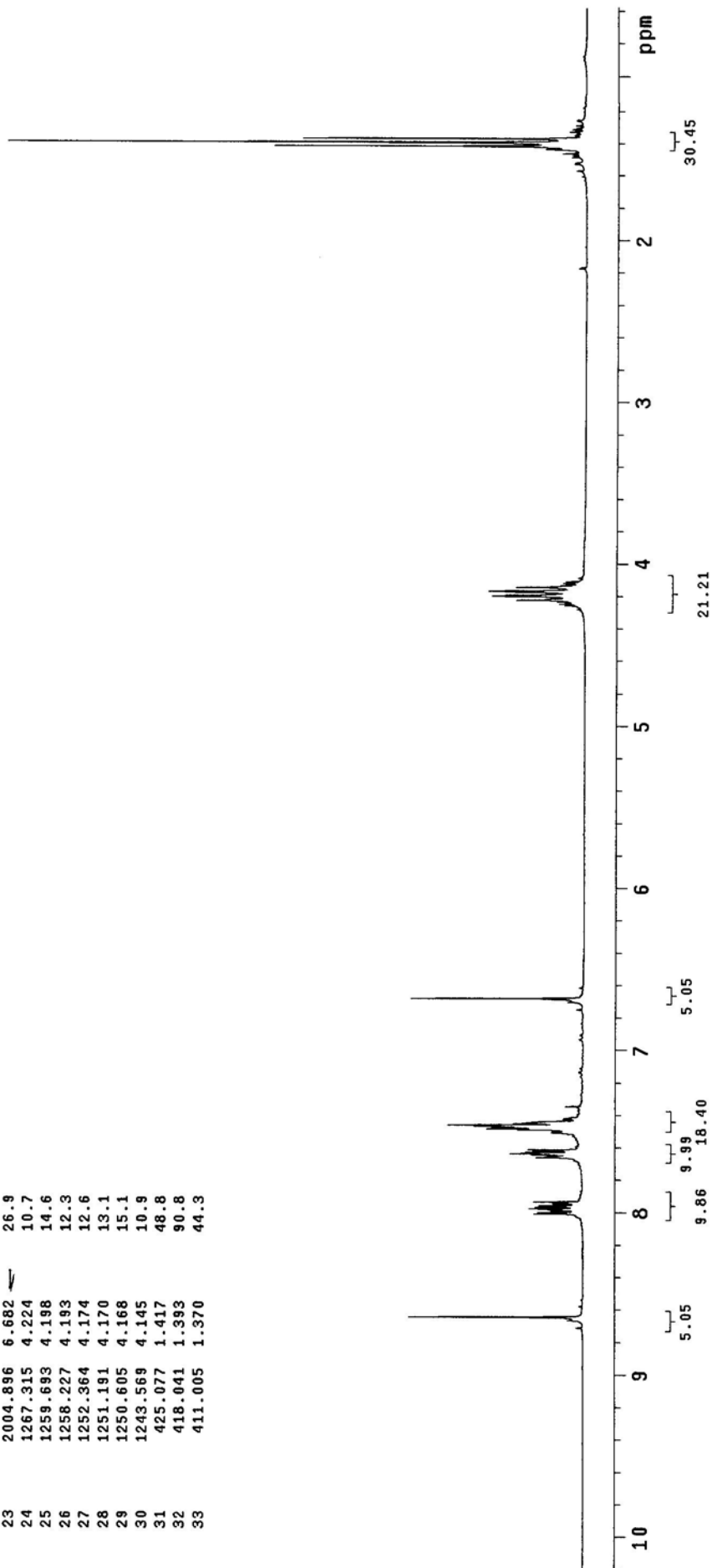


¹H-NMR

INDEX	FREQUENCY PPM	HEIGHT
1	2593.848	27.2
2	2403.003	7.7
3	2393.622	8.5
4	2390.104	7.6
5	2380.723	7.8
6	2293.069	8.2
7	2291.310	11.5
8	2289.258	9.1
9	2286.033	8.0
10	2283.981	8.7
11	2247.044	10.3
12	2245.285	15.2
13	2242.646	14.4
14	2241.474	13.3
15	2240.887	13.6
16	2240.008	17.1
17	2238.542	21.1
18	2235.904	9.3
19	2235.317	11.0
20	2234.731	9.5
21	2234.145	9.6
22	2232.679	7.9
23	2004.896	26.9
24	1267.315	10.7
25	1259.693	14.6
26	1258.227	12.3
27	1252.364	12.6
28	1251.191	13.1
29	1250.605	15.1
30	1243.569	10.9
31	425.077	48.8
32	418.041	90.8
33	411.005	44.3

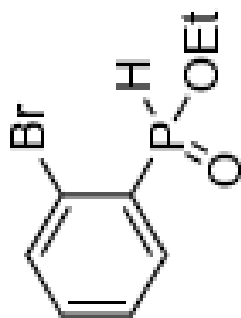


3 (Scheme 3)

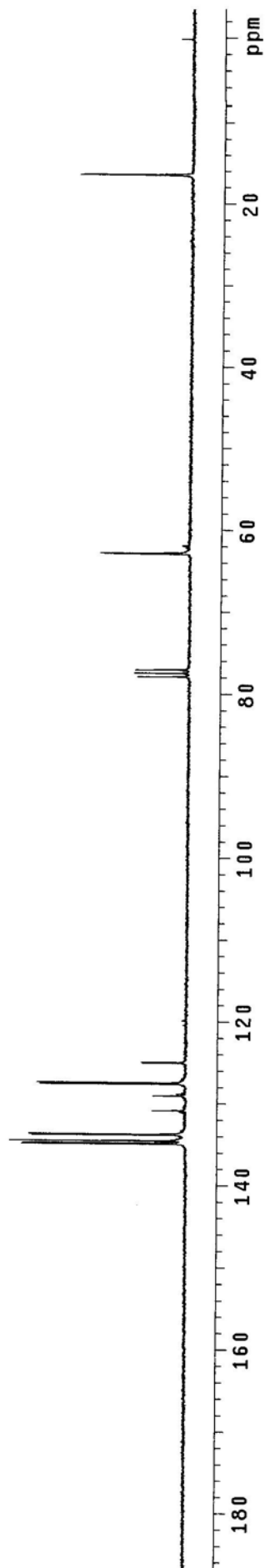


¹³C-NMR

INDEX	FREQUENCY	PPM	HEIGHT
1	10180.418	134.933	25.9
2	10172.069	134.822	25.7
3	10153.355	134.574	26.5
4	10151.340	134.548	27.7
5	10097.502	133.834	24.2
6	10089.153	133.723	24.7
7	9873.803	130.869	5.3
8	9737.337	129.060	5.1
9	9719.199	128.820	1.4
10	9629.950	127.637	23.1
11	9618.434	127.484	23.4
12	9435.904	125.065	6.9
13	9427.843	124.958	7.0
14	5878.303	77.912	8.1
15	5846.058	77.485	8.6
16	5814.101	77.061	8.5
17	4745.986	62.904	13.4
18	4739.364	62.816	14.2
19	1245.677	16.510	17.8
20	1239.055	16.423	17.2
21	13.458	0.178	2.0

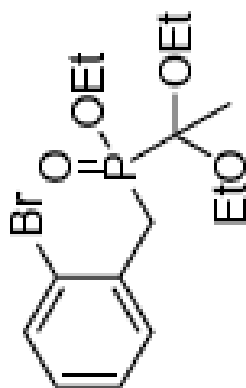


3 (Scheme 3)

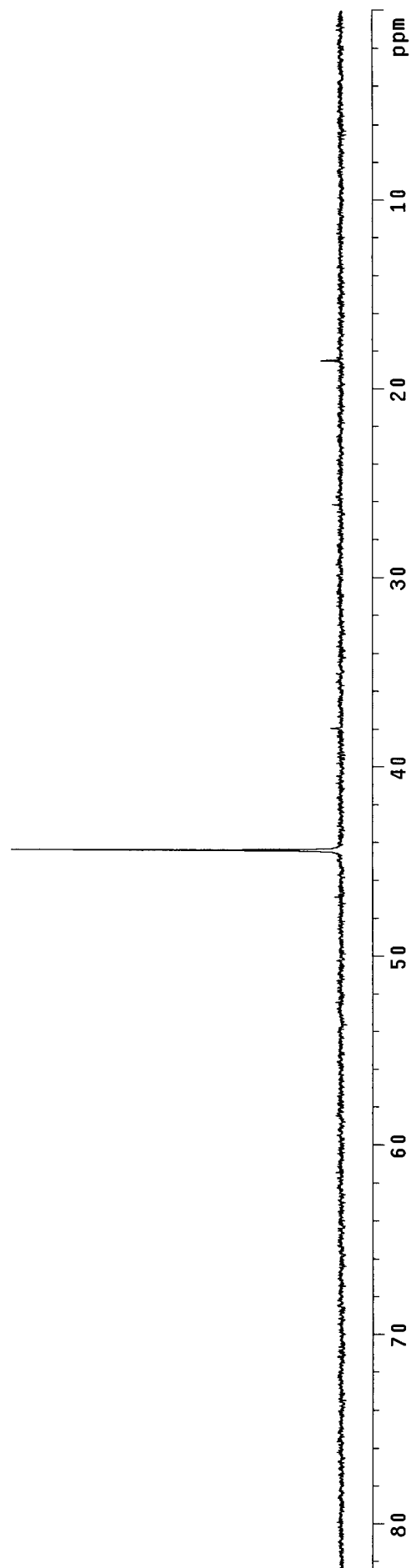


INDEX	FREQUENCY	PPM	HEIGHT
1	5392.380	44.395	51.7

^{31}P -NMR (^1H decoupled)

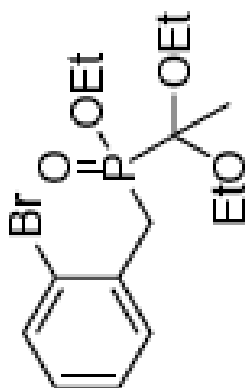


(Scheme 4)

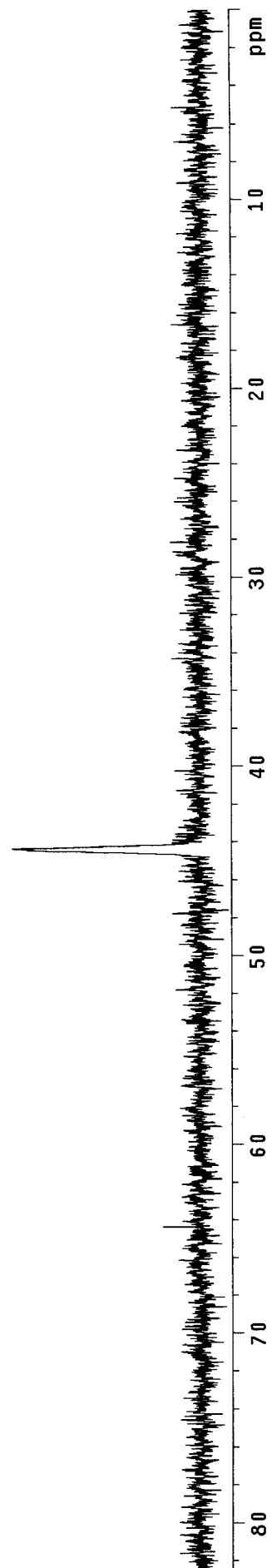


INDEX	FREQUENCY PPM	HEIGHT
1	5389.524	44.372
		29.6

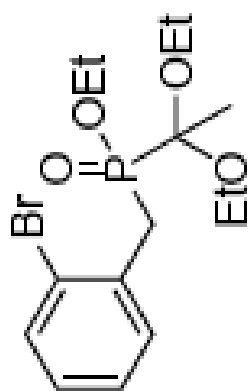
³¹P-NMR (¹H coupled)



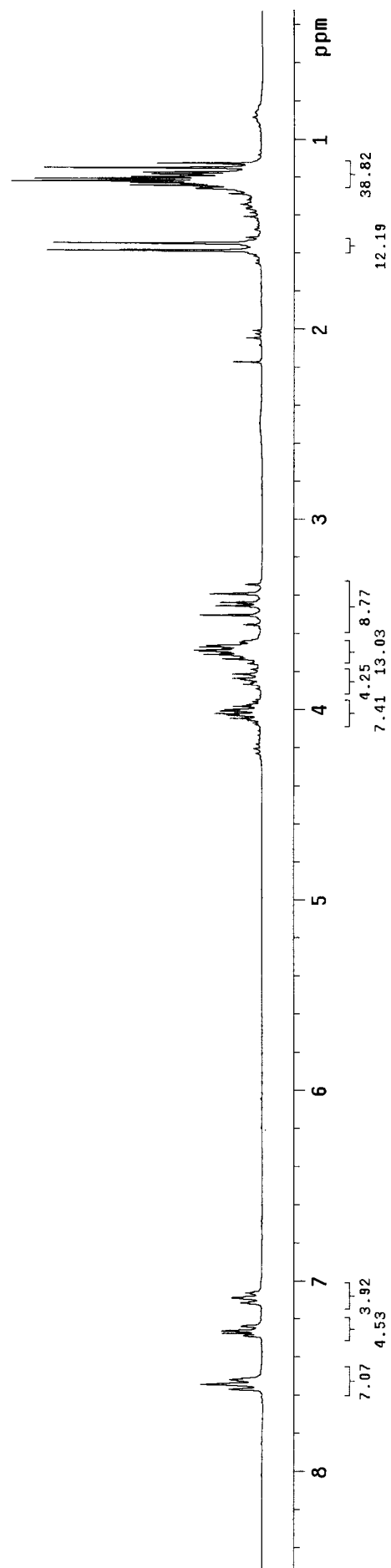
(Scheme 4)



$^1\text{H-NMR}$

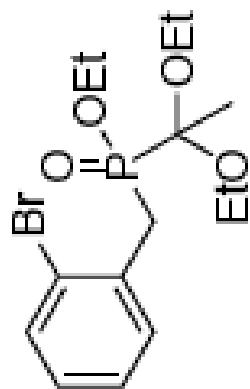


(Scheme 4)

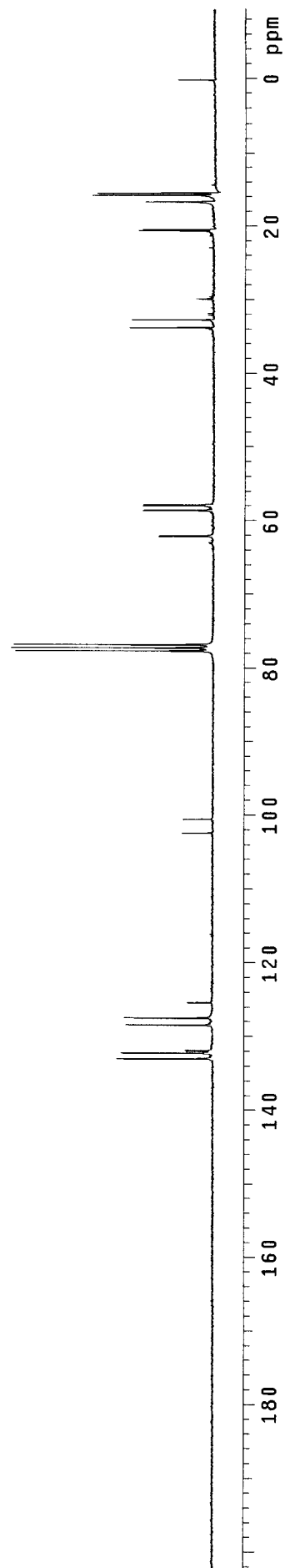


¹³C-NMR

INDEX	FREQUENCY	PPM	HEIGHT
1	10039.922	133.071	14.1
2	10037.619	133.040	15.1
3	9983.493	132.323	14.0
4	9979.175	132.266	14.4
5	9961.037	132.025	4.1
6	9951.536	131.899	4.3
7	9695.303	128.503	13.1
8	9692.424	128.465	13.6
9	9622.176	127.534	13.2
10	9619.585	127.500	13.9
11	9467.285	125.481	3.8
12	9460.088	125.386	3.9
13	7731.814	102.479	4.7
14	7589.591	100.594	4.6
15	5861.317	77.687	31.0
16	5829.360	77.263	31.7
17	5797.403	76.840	31.4
18	4687.830	62.133	8.6
19	4680.344	62.034	8.4
20	4424.975	58.649	11.1
21	4420.081	58.585	10.9
22	4373.153	57.963	11.1
23	4365.668	57.863	11.0
24	2551.599	33.819	13.2
25	2472.426	32.770	12.9
26	2257.364	29.920	2.7
27	1557.475	20.643	11.8
28	1545.095	20.479	11.2
29	1262.375	16.732	10.7
30	1257.193	16.663	9.5
31	1190.112	15.774	19.2
32	1175.429	15.579	3.1
33	1169.095	15.495	18.4
34	16.049	0.213	5.6

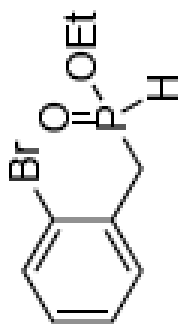


(Scheme 4)

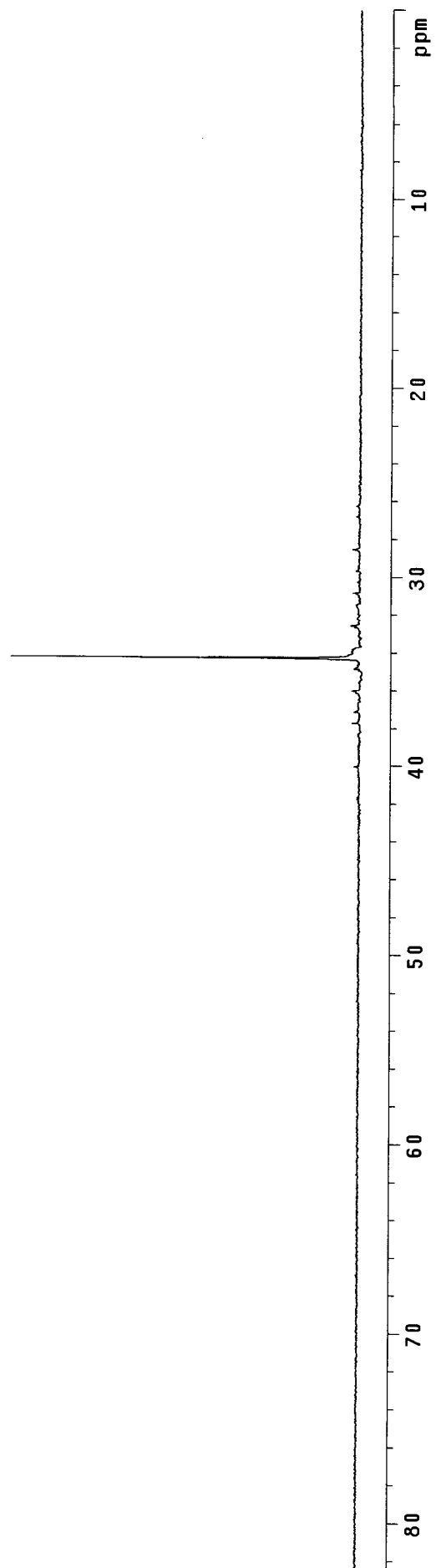


INDEX	FREQUENCY PPM	HEIGHT
1	4163.109	54.5

³¹P-NMR (¹H decoupled)

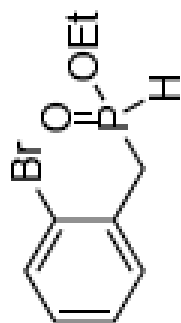


4 (Scheme 4)

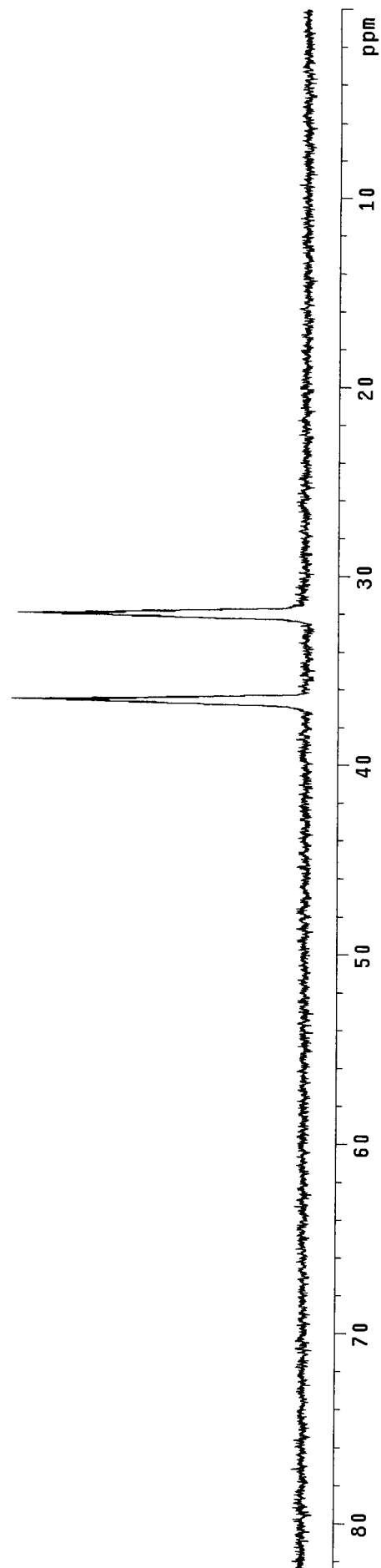


INDEX	FREQUENCY	PPM	HEIGHT
1	4460.533	36.723	27.9
2	4450.333	36.639	37.7
3	4441.358	36.566	46.2
4	4432.382	36.492	33.7
5	4422.182	36.408	22.1
6	3906.892	32.165	27.6
7	3897.100	32.085	38.4
8	3887.717	32.007	45.3
9	3878.741	31.934	34.8
10	3868.541	31.850	25.9

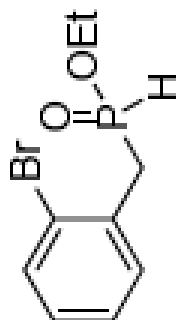
³¹P-NMR (¹H coupled)



4 (Scheme 4)

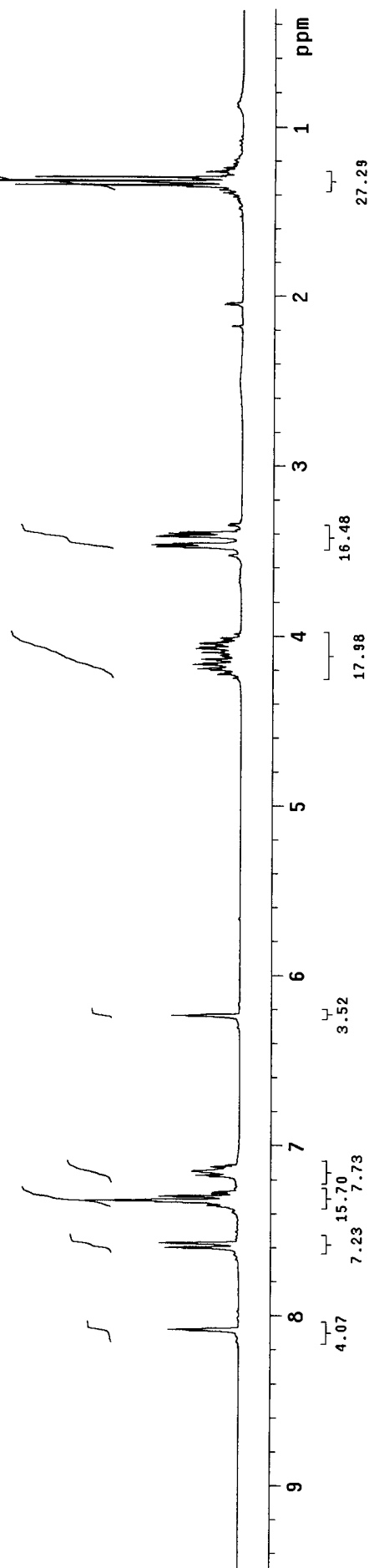


¹H-NMR



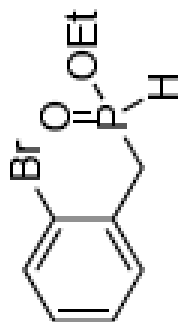
4 (Scheme 4)

INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	2426.162	8.086	8.3	40	1039.532	3.465	14.1
2	2424.696	8.081	11.1	41	1037.187	3.457	10.8
3	2422.937	8.075	6.0	42	1024.875	3.416	13.5
4	2280.463	7.600	11.6	43	1019.598	3.398	11.5
5	2272.548	7.574	12.5	44	1017.253	3.390	9.8
6	2205.709	7.351	5.1	45	405.728	1.352	9.0
7	2203.070	7.342	5.0	46	403.080	1.343	35.6
8	2200.139	7.333	13.2	47	398.693	1.329	15.1
9	2197.793	7.325	24.0	48	396.054	1.320	65.4
10	2196.034	7.319	16.0	49	391.657	1.305	8.0
11	2189.878	7.298	12.6	50	388.725	1.296	32.6
12	2152.647	7.174	6.9	51	378.465	1.261	5.9
13	2147.370	7.157	6.2				
14	2146.198	7.153	7.4				
15	2145.318	7.150	7.5				
16	1873.269	6.243	7.5				
17	1872.683	6.241	8.0				
18	1871.803	6.238	8.4				
19	1871.217	6.236	10.7				
20	1869.458	6.230	6.1				
21	1260.279	4.200	4.8				
22	1258.520	4.194	5.0				
23	1257.348	4.190	6.9				
24	1256.761	4.188	6.0				
25	1251.485	4.171	5.6				
26	1250.312	4.167	7.6				
27	1249.726	4.165	6.7				
28	1248.260	4.160	6.1				
29	1241.224	4.137	6.1				
30	1240.638	4.135	6.2				
31	1229.498	4.098	6.2				
32	1228.912	4.096	6.0				
33	1222.462	4.074	7.1				
34	1221.876	4.072	6.8				
35	1220.703	4.068	6.6				
36	1220.117	4.066	6.6				
37	1213.667	4.045	6.4				
38	1213.081	4.043	6.6				
39	1043.344	3.477	13.3				

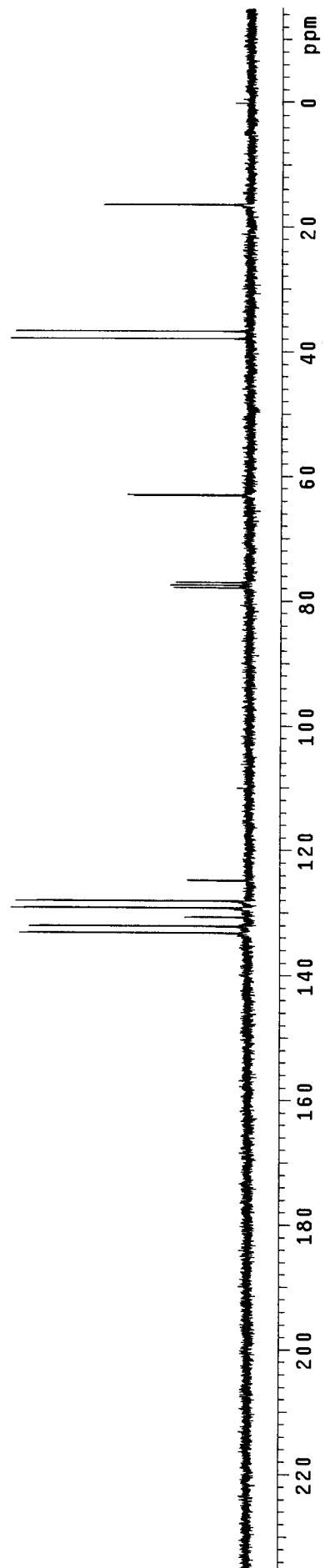


¹³C-NMR

INDEX	FREQUENCY	PPM	HEIGHT
1	10049.710	133.201	35.9
2	10046.256	133.155	34.5
3	9970.250	132.148	34.3
4	9964.492	132.071	32.8
5	9861.423	130.705	9.9
6	9855.937	130.606	8.9
7	9748.565	129.209	37.3
8	9744.535	129.156	35.1
9	9666.225	128.118	36.5
10	9662.771	128.072	33.1
11	9421.797	124.878	9.4
12	9414.311	124.779	9.6
13	5876.000	77.882	12.0
14	5844.043	77.458	12.5
15	5811.798	77.031	11.6
16	4758.365	63.068	18.2
17	4751.456	62.977	19.2
18	2861.093	37.921	37.6
19	2772.132	36.742	36.9
20	1242.222	16.465	23.1
21	1236.176	16.385	22.5



4 (Scheme 4)



INDEX	FREQUENCY	PPM	HEIGHT
1	6265.069	51.580	71.4
2	6147.976	50.616	55.7

³¹P-NMR (¹H decoupled)

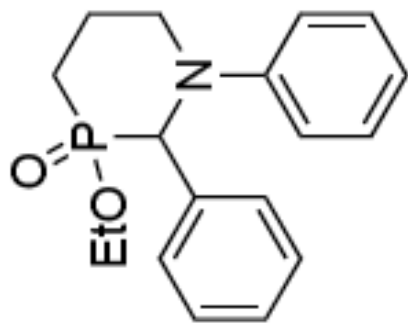
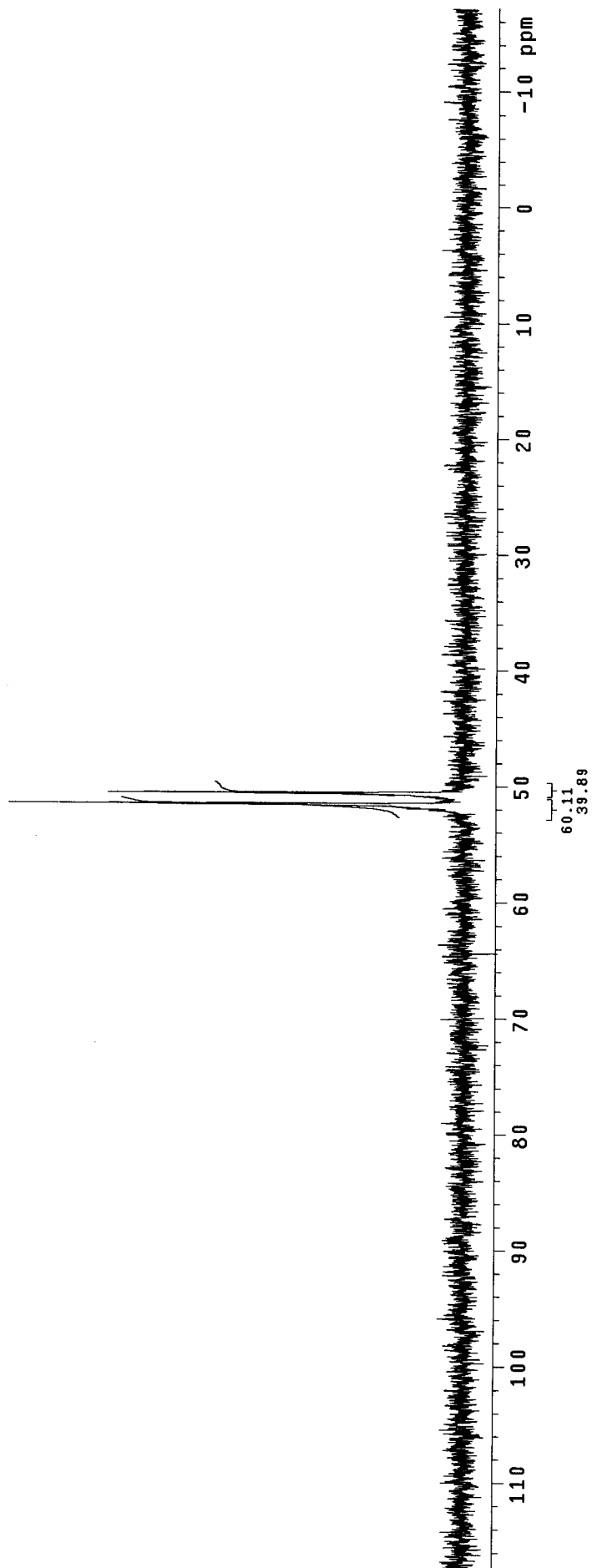


Table 1, entry 1



INDEX	FREQUENCY	PPM	HEIGHT
1	6261.397	51.550	16.6
2	6152.464	50.653	14.8

³¹P-NMR (1H coupled)

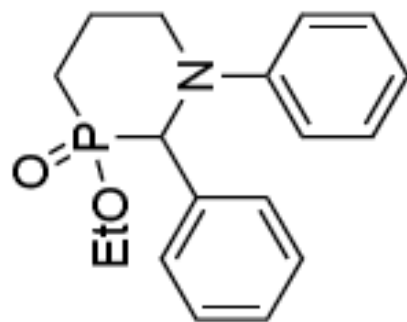
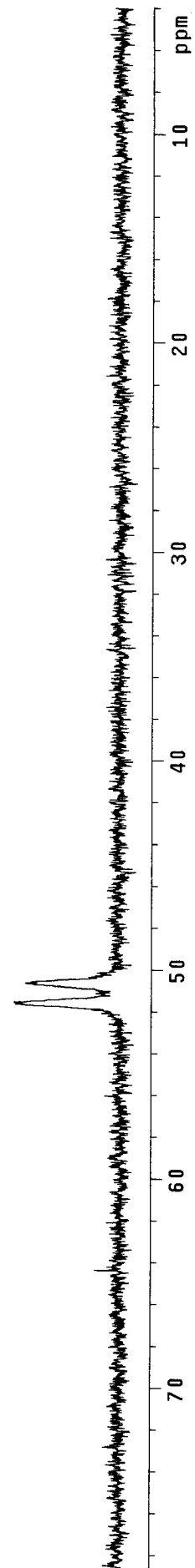


Table 1, entry 1



¹H-NMR

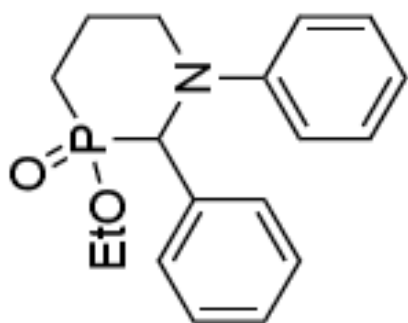
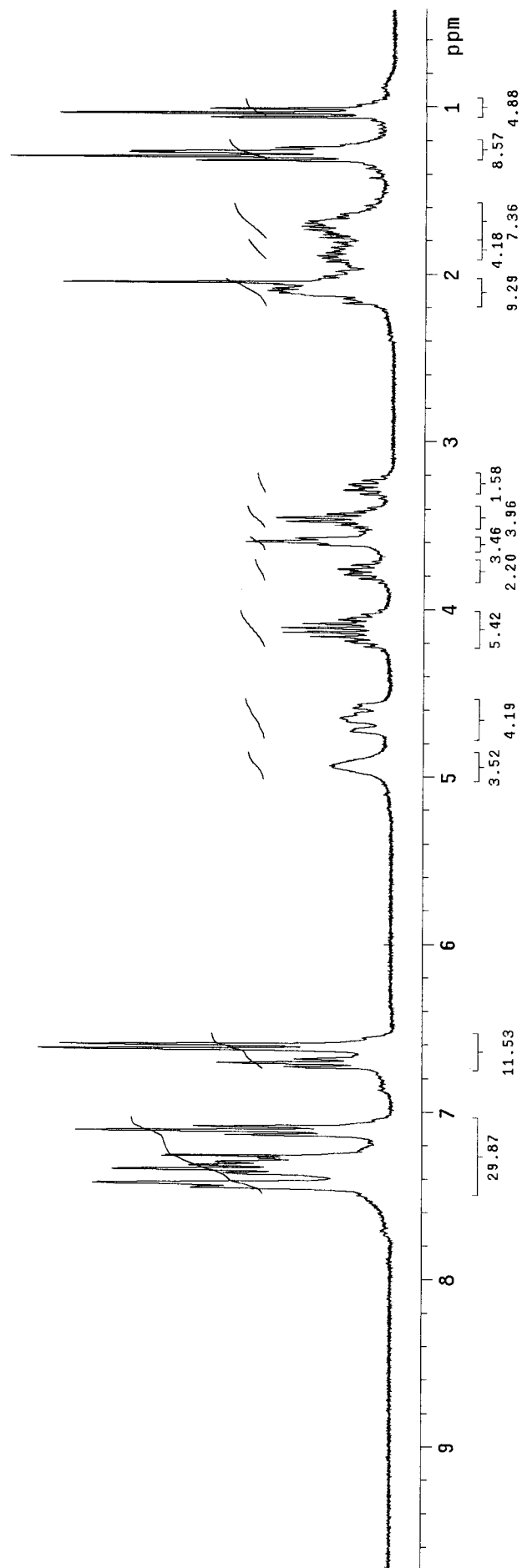


Table 1, entry 1



¹³C-NMR

INDEX	FREQUENCY	PPM	HEIGHT
1	11047.290	146.423	6.7
2	10259.591	135.982	7.6
3	9769.006	129.480	70.2
4	9749.717	128.225	28.7
5	9730.427	128.969	27.6
6	9676.878	128.259	26.3
7	9672.559	128.202	24.3
8	9643.769	127.820	24.5
9	9638.875	127.755	22.7
10	8967.776	118.861	27.8
11	8626.612	114.339	44.7
12	8620.567	114.259	45.7
13	5864.196	77.725	37.9
14	5832.239	77.302	40.6
15	5800.282	76.878	39.1
16	4712.589	62.462	9.3
17	4705.967	62.374	9.6
18	4688.405	62.141	9.1
19	4681.496	62.049	11.1
20	4421.808	58.607	9.0
21	4404.822	58.382	12.1
22	4329.104	57.379	10.5
23	4308.375	57.104	9.4
24	3426.820	45.420	11.3
25	3421.350	45.347	10.7
26	3410.122	45.198	11.7
27	3404.364	45.122	13.1
28	1962.264	26.008	8.8
29	1906.124	25.264	11.1
30	1881.652	24.940	21.7
31	1868.408	24.764	12.2
32	1785.781	23.669	11.4
33	1281.089	16.980	9.4
34	1275.907	16.911	9.5
35	1253.738	16.617	9.0
36	1089.922	14.446	6.4
37	18.352	0.243	7.9

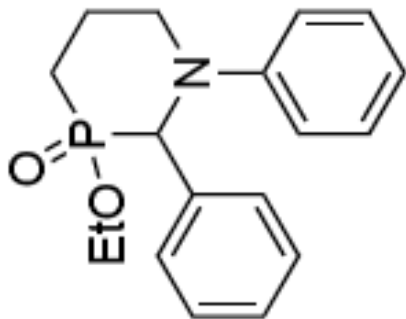
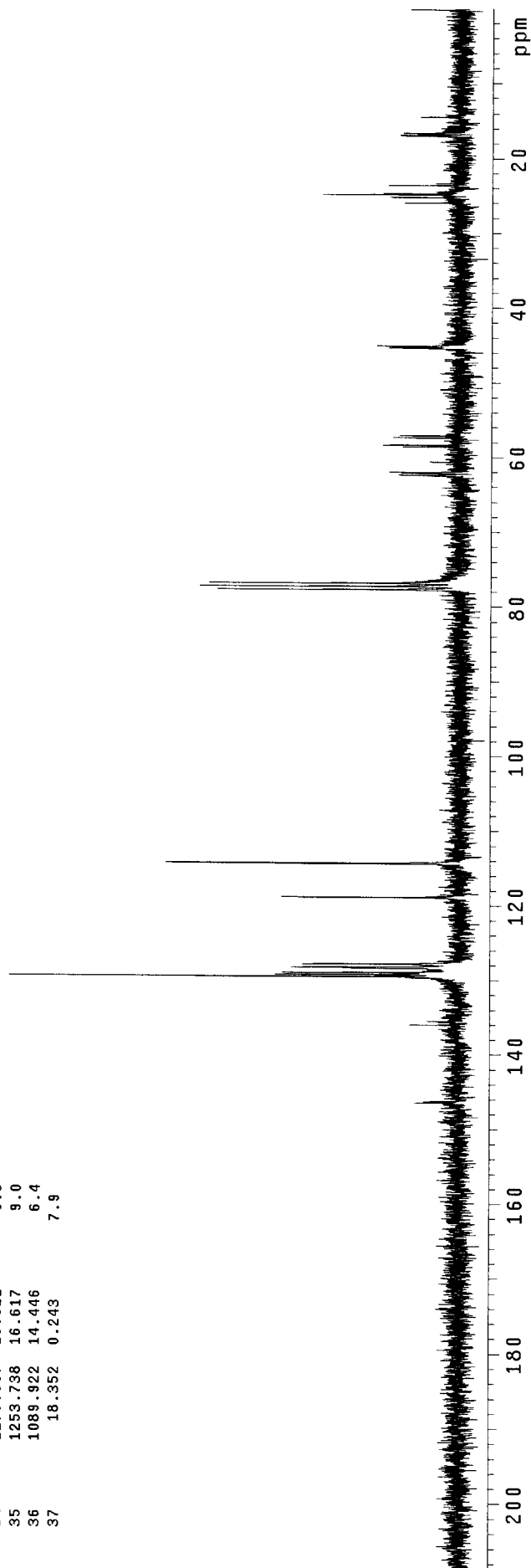


Table 1, entry 1



INDEX	FREQUENCY PPM	HEIGHT
1	5372.389	44.231
		60.0

³¹P-NMR (¹H decoupled)

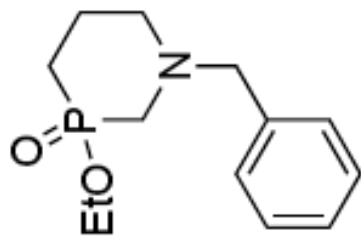
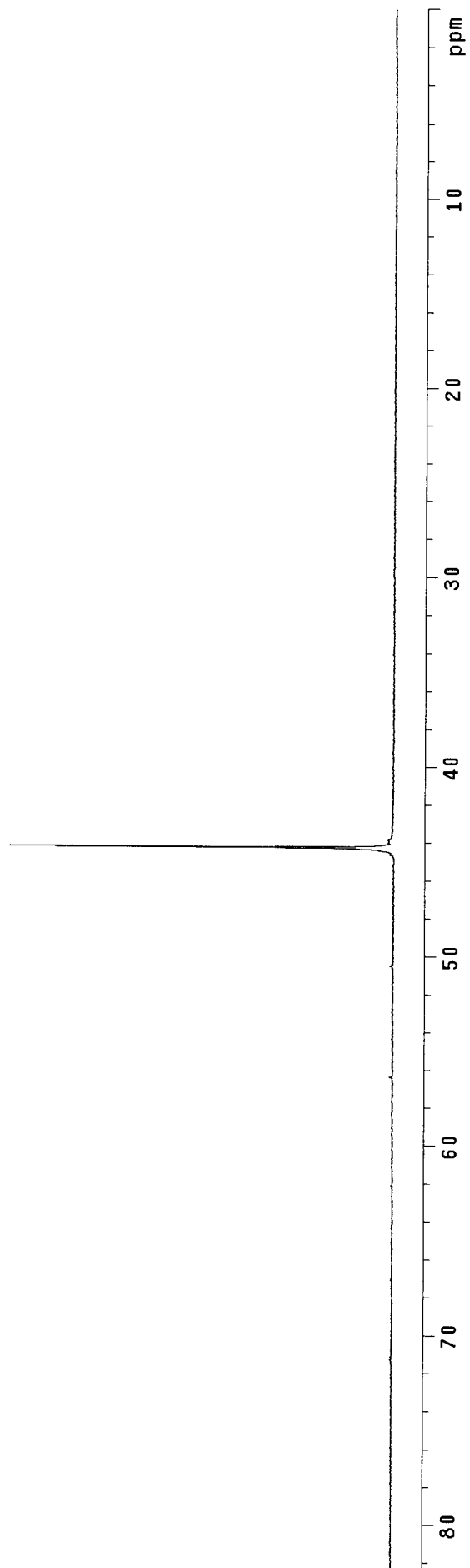


Table 1, entry 2



INDEX	FREQUENCY	PPM	HEIGHT
1	5380.548	44.298	38.6

³¹P-NMR (¹H coupled)

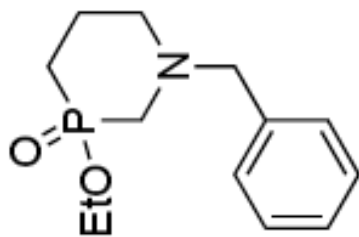
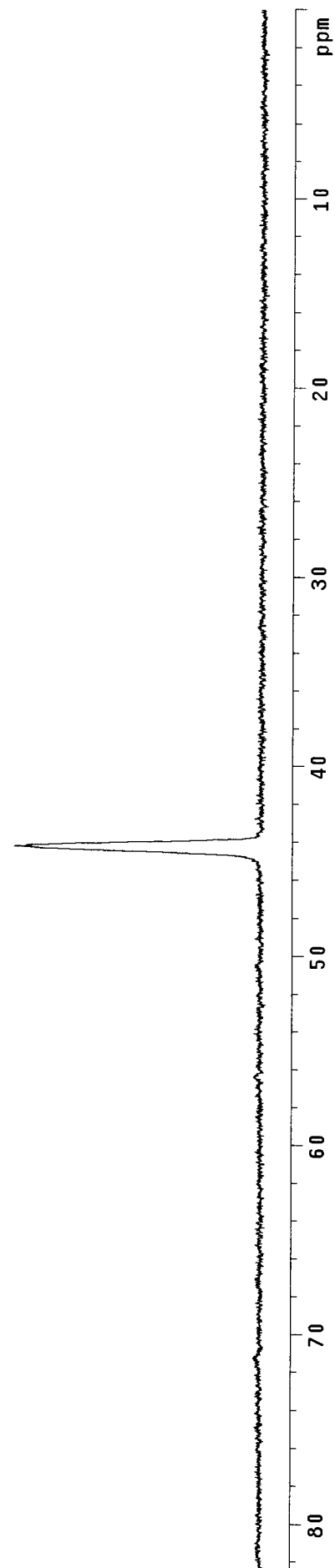


Table 1, entry 2



¹H-NMR

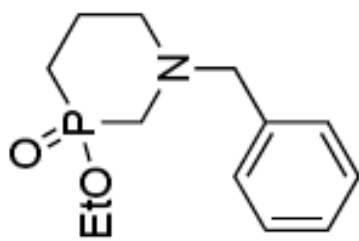
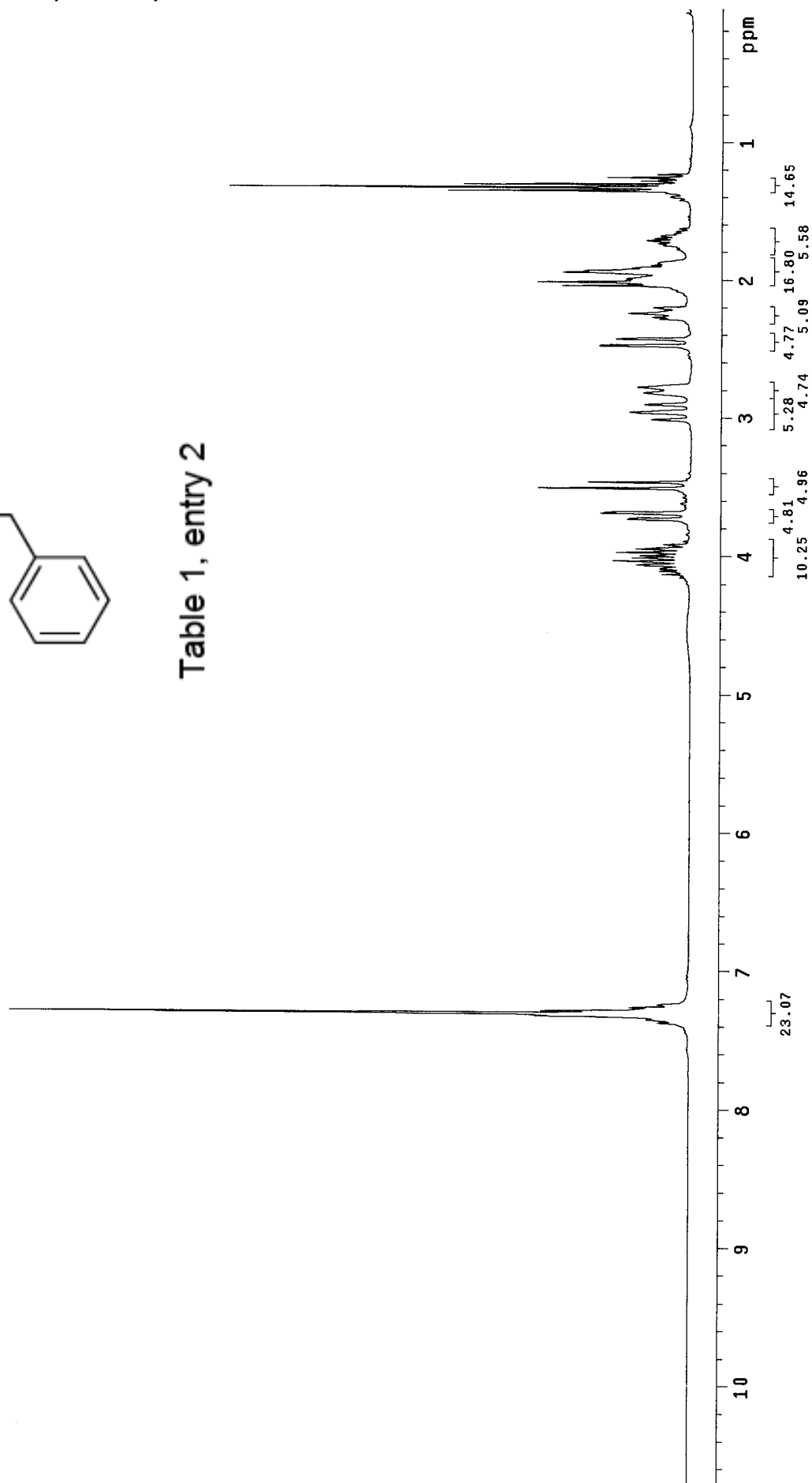


Table 1, entry 2



¹³C-NMR

INDEX	FREQUENCY	PPM	HEIGHT
1	10364.387	137.371	15.3
2	9747.414	129.194	86.2
3	9682.424	128.465	87.2
4	9625.919	127.584	43.5
5	5889.243	78.057	6.0
6	5856.998	77.630	6.6
7	5824.753	77.202	6.4
8	4870.835	64.560	22.1
9	4855.388	64.354	22.6
10	4559.138	60.428	16.9
11	4552.516	60.340	17.2
12	4093.025	54.250	23.0
13	4088.130	54.185	23.9
14	3987.365	52.849	21.1
15	3889.190	51.548	20.8
16	1976.947	26.203	21.2
17	1887.986	25.024	21.2
18	1769.082	23.448	22.3
19	1763.036	23.368	21.6
20	1265.542	16.774	16.5
21	1259.496	16.694	15.7

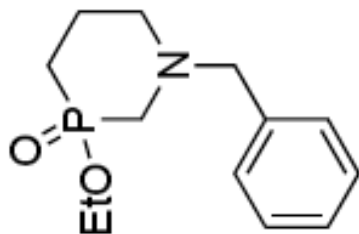
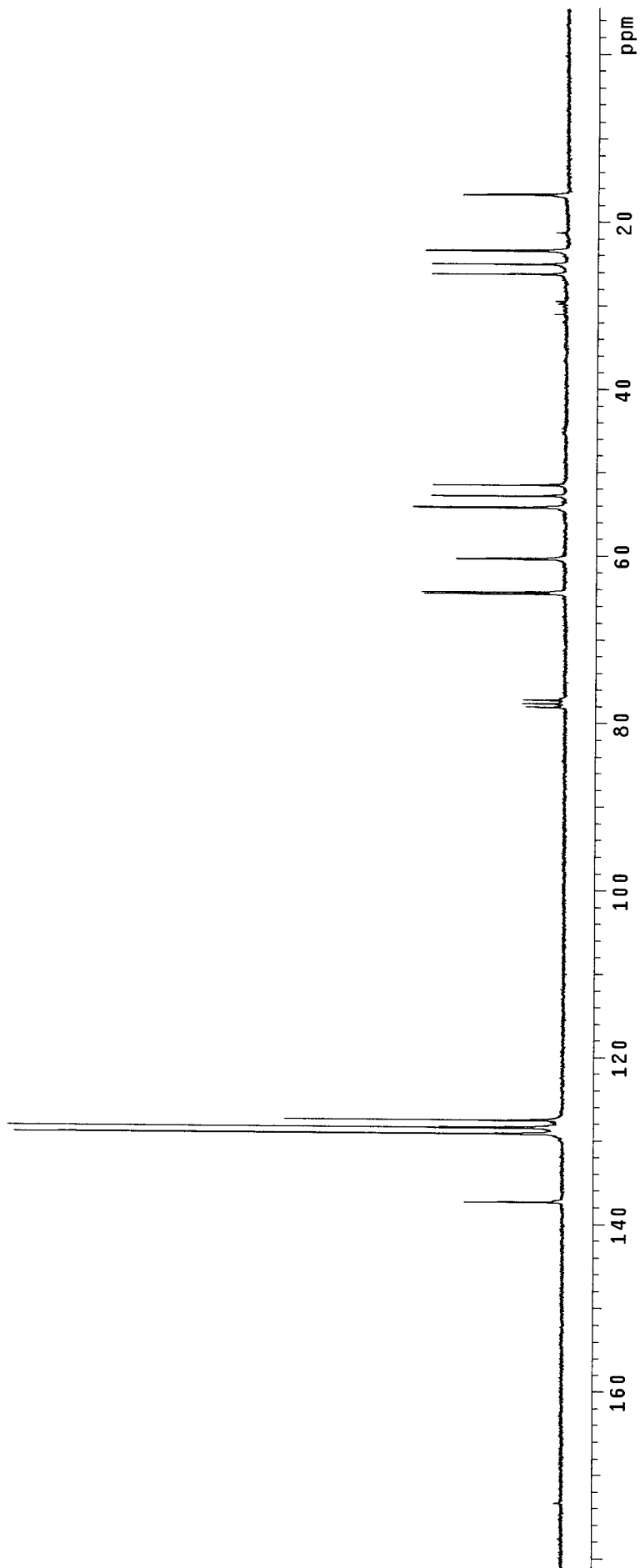


Table 1, entry 2



INDEX	FREQUENCY	PPM	HEIGHT
1	6303.828	51.899	123.1
2	6189.999	50.962	126.0

³¹P-NMR (¹H decoupled)

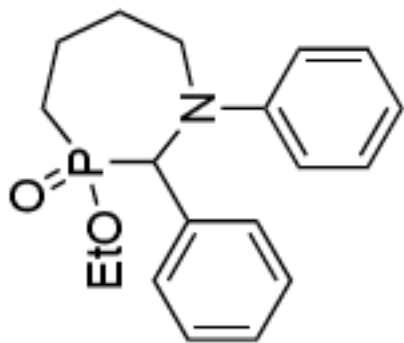
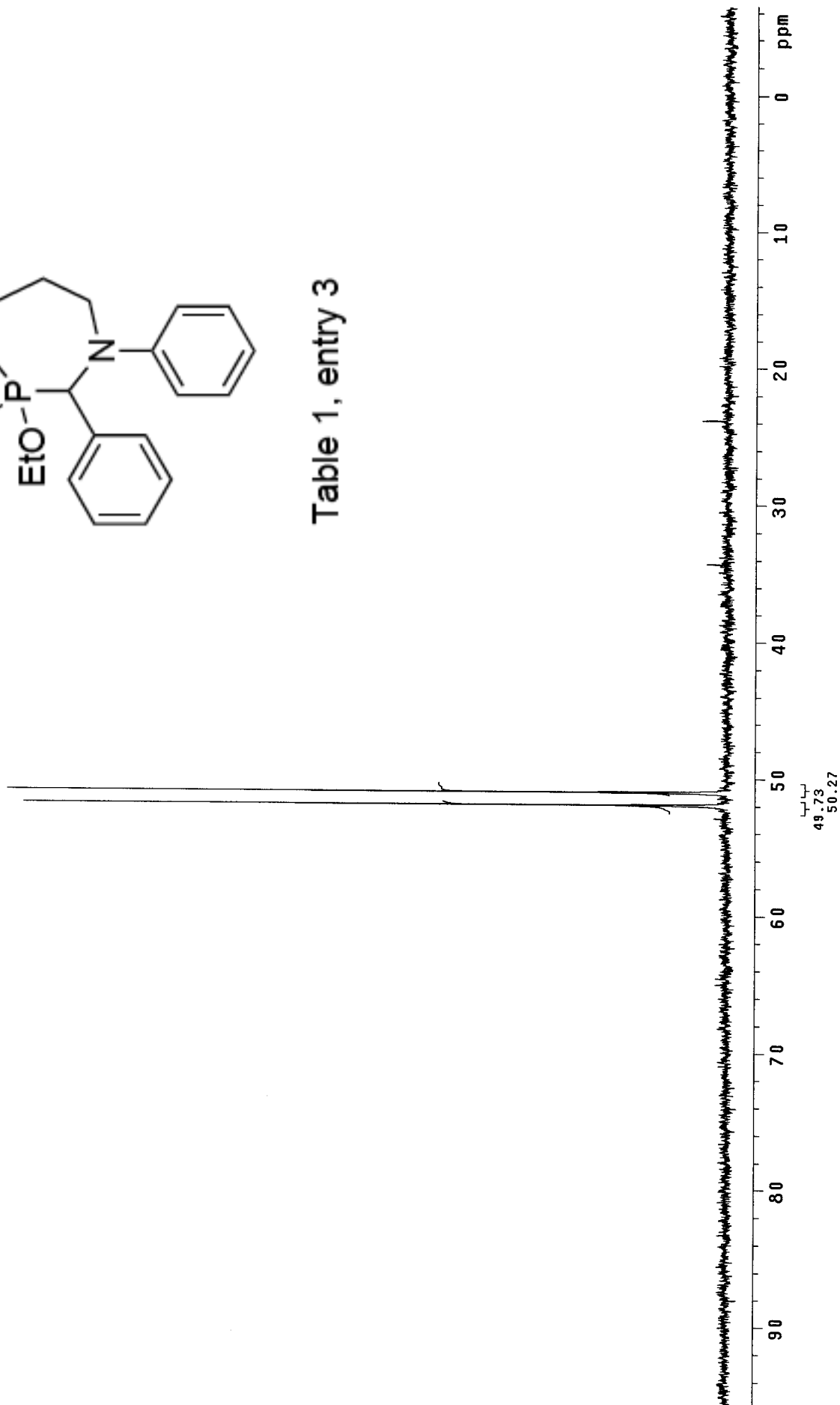


Table 1, entry 3



INDEX	FREQUENCY	PPM	HEIGHT
1	6312.803	51.973	33.3
2	6196.934	51.019	34.4

³¹P-NMR (¹H coupled)

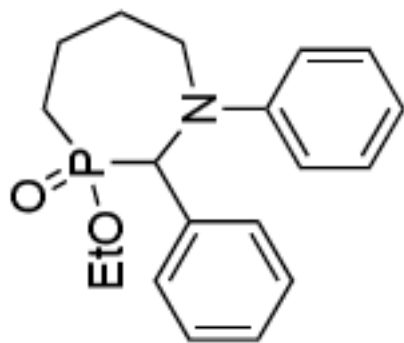
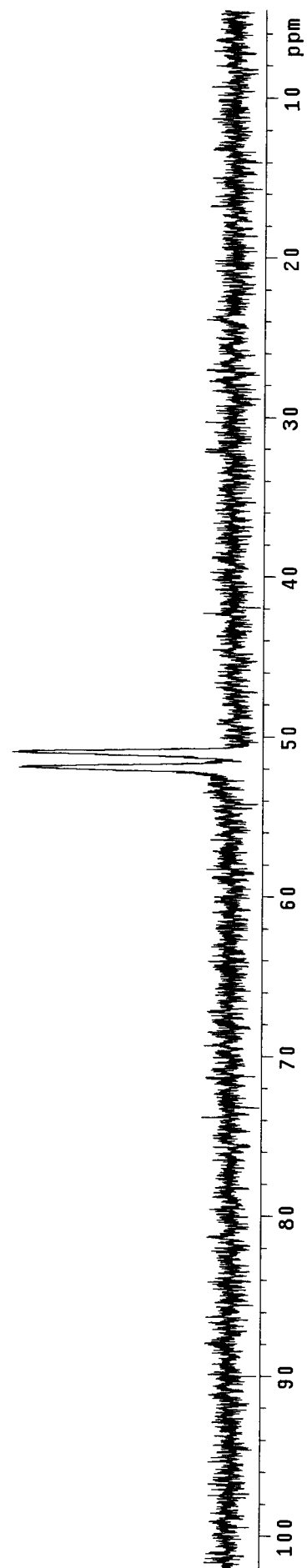


Table 1, entry 3



¹H-NMR

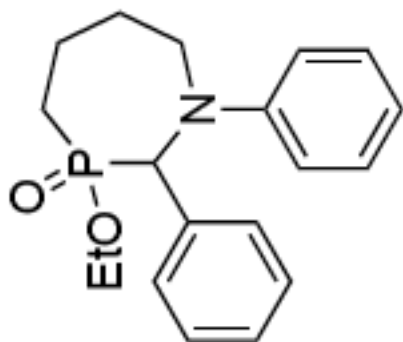
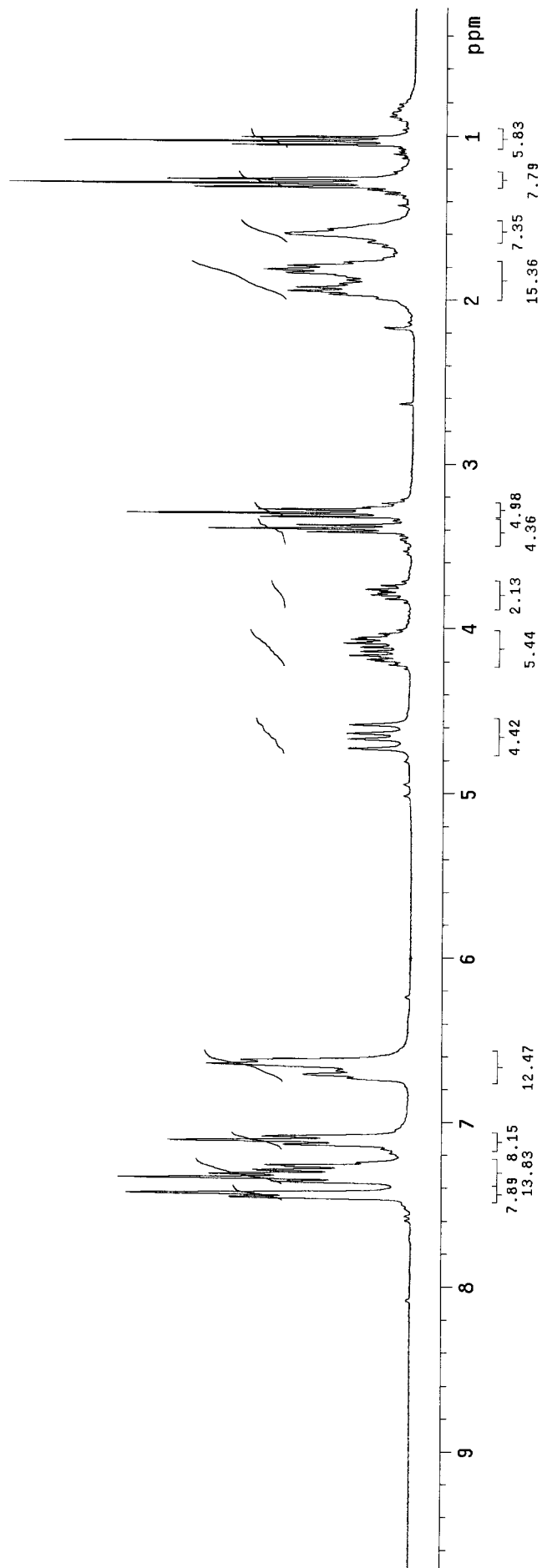


Table 1, entry 3



¹³C-NMR

INDEX	FREQUENCY PPM	HEIGHT	INDEX	FREQUENCY PPM	HEIGHT
1	11039.516	146.320	9.7	1557.187	20.639
2	10267.652	136.089	13.1	1537.321	20.376
3	10231.376	135.608	9.7	1533.003	20.319
4	9767.279	129.457	147.9	1283.680	17.014
5	9746.550	129.183	88.6	1278.488	16.945
6	9744.247	129.152	86.6	1261.800	16.724
7	9727.836	128.935	81.5	1256.617	16.655
8	9725.821	128.908	82.2		
9	9682.924	128.339	89.4		
10	9678.605	128.282	95.6		
11	9648.376	127.881	48.0		
12	9643.769	127.820	46.8		
13	8973.534	118.937	44.8		
14	8632.083	114.411	64.0		
15	8440.916	111.877	5.7		
16	5872.545	77.836	68.0		
17	5840.588	77.412	69.9		
18	5808.343	76.985	67.8		
19	4705.392	62.366	25.5		
20	4698.482	62.275	27.2		
21	4682.647	62.065	24.9		
22	4675.450	61.969	25.0		
23	4419.505	53.577	11.7		
24	4400.216	58.321	10.9		
25	4328.528	57.371	11.8		
26	4305.208	57.062	10.6		
27	2538.644	33.648	41.3		
28	2535.477	33.606	38.1		
29	2523.961	33.453	41.9		
30	2521.369	33.419	41.2		
31	2480.276	33.007	59.6		
32	2483.366	32.915	64.3		
33	2259.379	29.946	4.8		
34	2230.589	29.565	6.7		
35	2075.122	27.504	16.9		
36	1996.813	26.466	18.0		
37	1981.842	26.268	17.4		
38	1801.517	25.203	17.1		
39	1561.793	20.700	29.9		

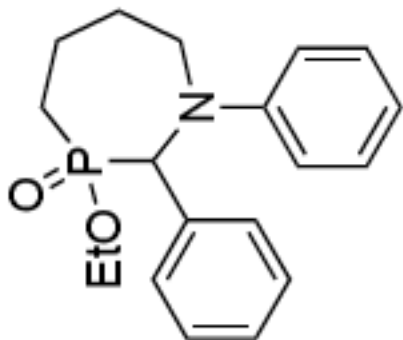
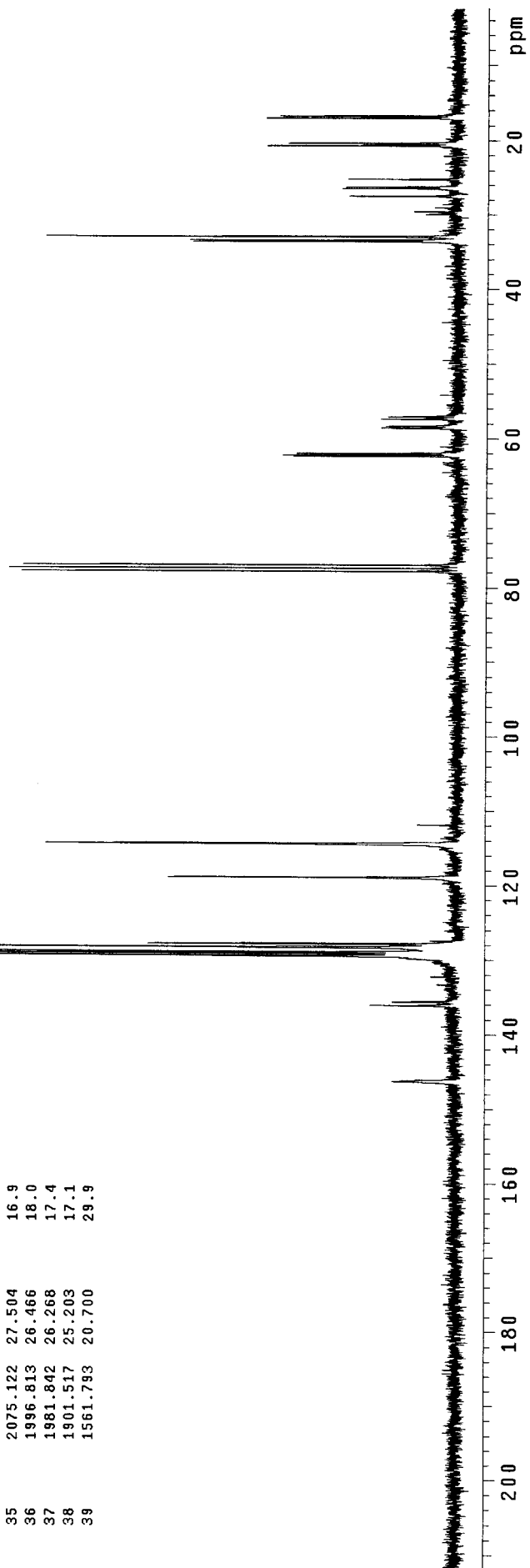


Table 1, entry 3



INDEX	FREQUENCY	PPM	HEIGHT
1	7660.391	63.068	86.1

³¹P-NMR (¹H decoupled)

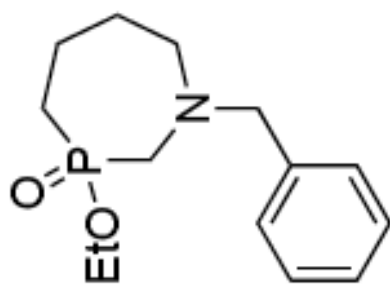
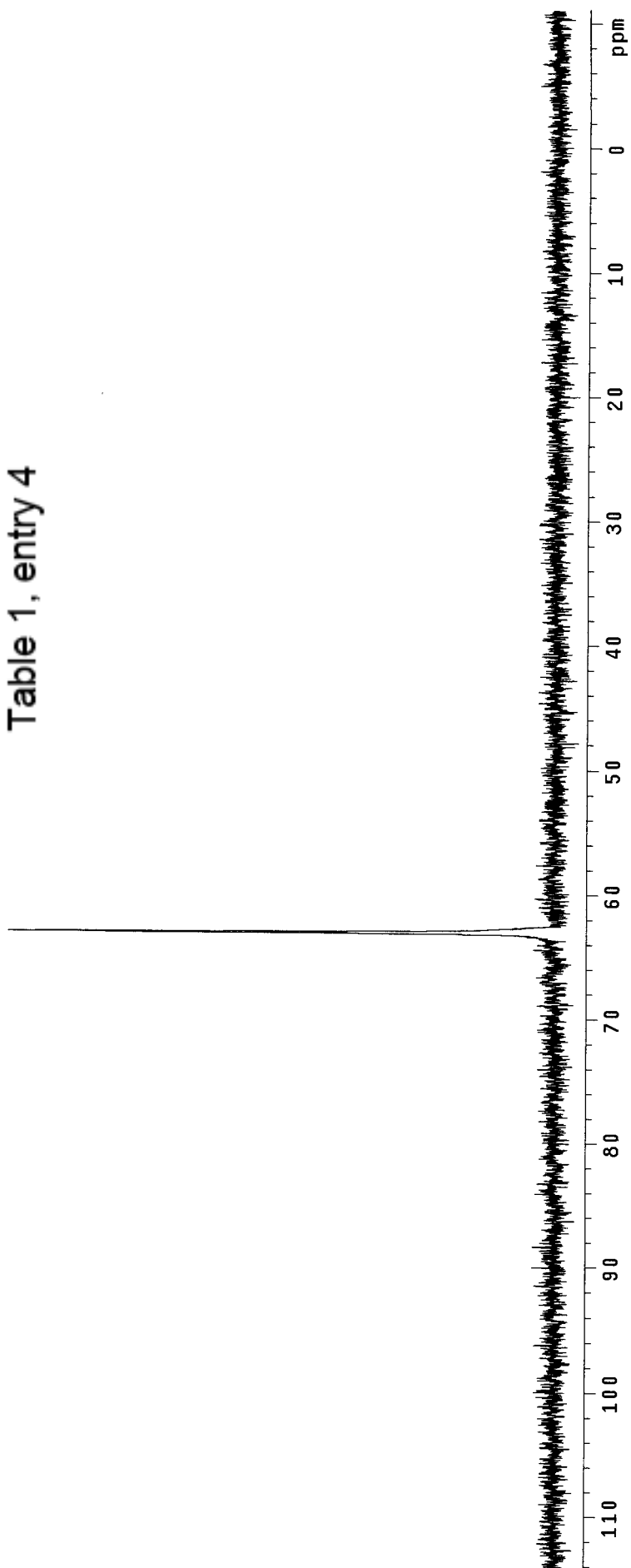


Table 1, entry 4



INDEX	FREQUENCY	PPM	HEIGHT
1	7664.879	63.105	30.3

³¹P-NMR (¹H coupled)

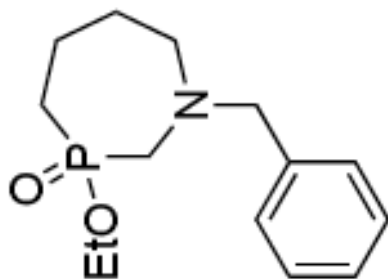
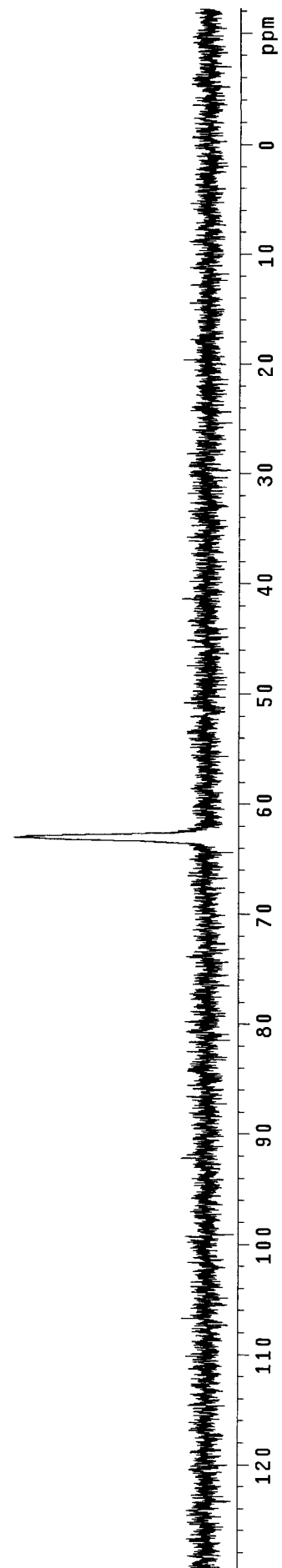


Table 1, entry 4



¹H-NMR

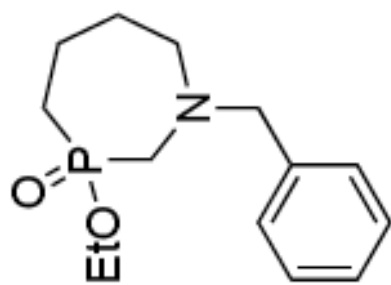
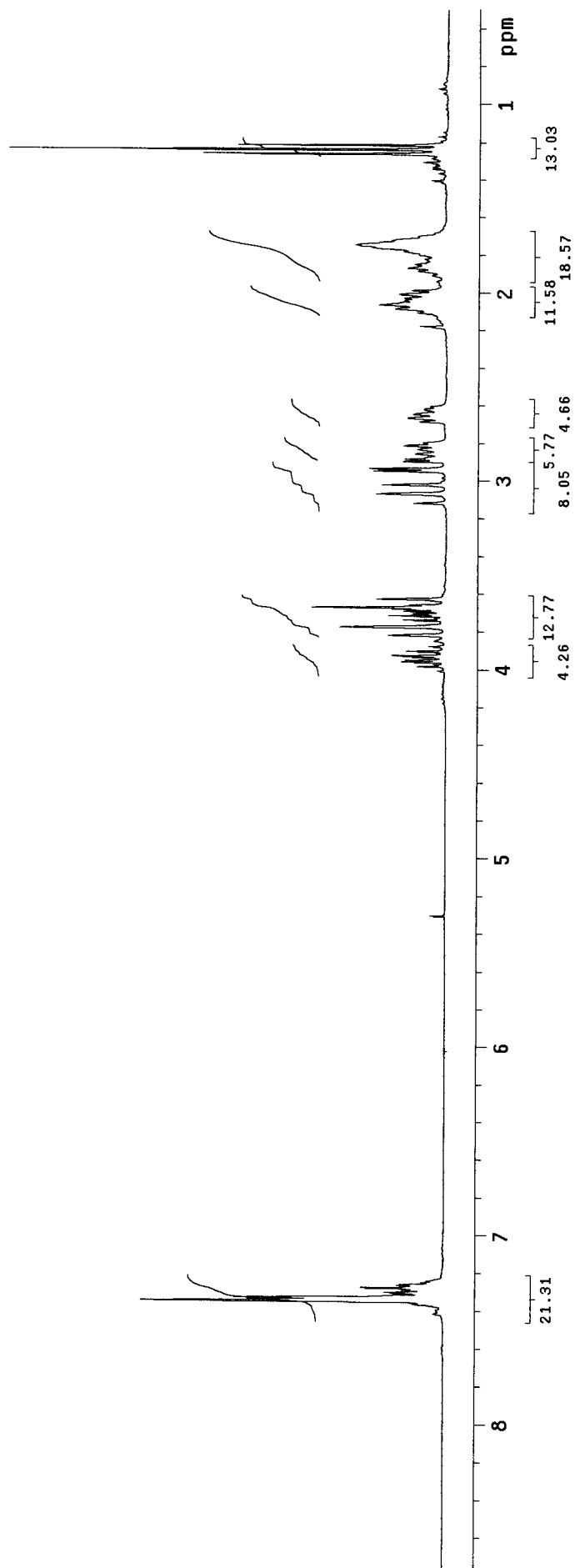


Table 1, entry 4



¹³C-NMR

INDEX	FREQUENCY	PPM	HEIGHT
1	10465.153	138.707	22.5
2	9744.823	129.160	125.3
3	9693.864	128.484	130.9
4	9619.297	127.496	64.0
5	5880.318	77.939	10.6
6	5848.361	77.515	11.5
7	5816.116	77.088	11.8
8	4849.055	64.270	29.2
9	4833.220	64.060	30.2
10	4529.196	60.031	19.5
11	4522.574	59.943	21.4
12	4398.200	58.295	57.6
13	4166.152	55.219	27.7
14	4061.068	53.826	27.8
15	2276.941	30.179	54.3
16	2237.498	29.656	33.7
17	2223.679	29.473	8.1
18	2149.400	28.489	32.5
19	1508.819	19.998	36.9
20	1507.092	19.975	40.6
21	1264.966	16.766	21.0
22	1258.921	16.686	22.5

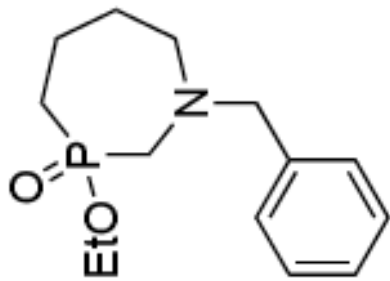
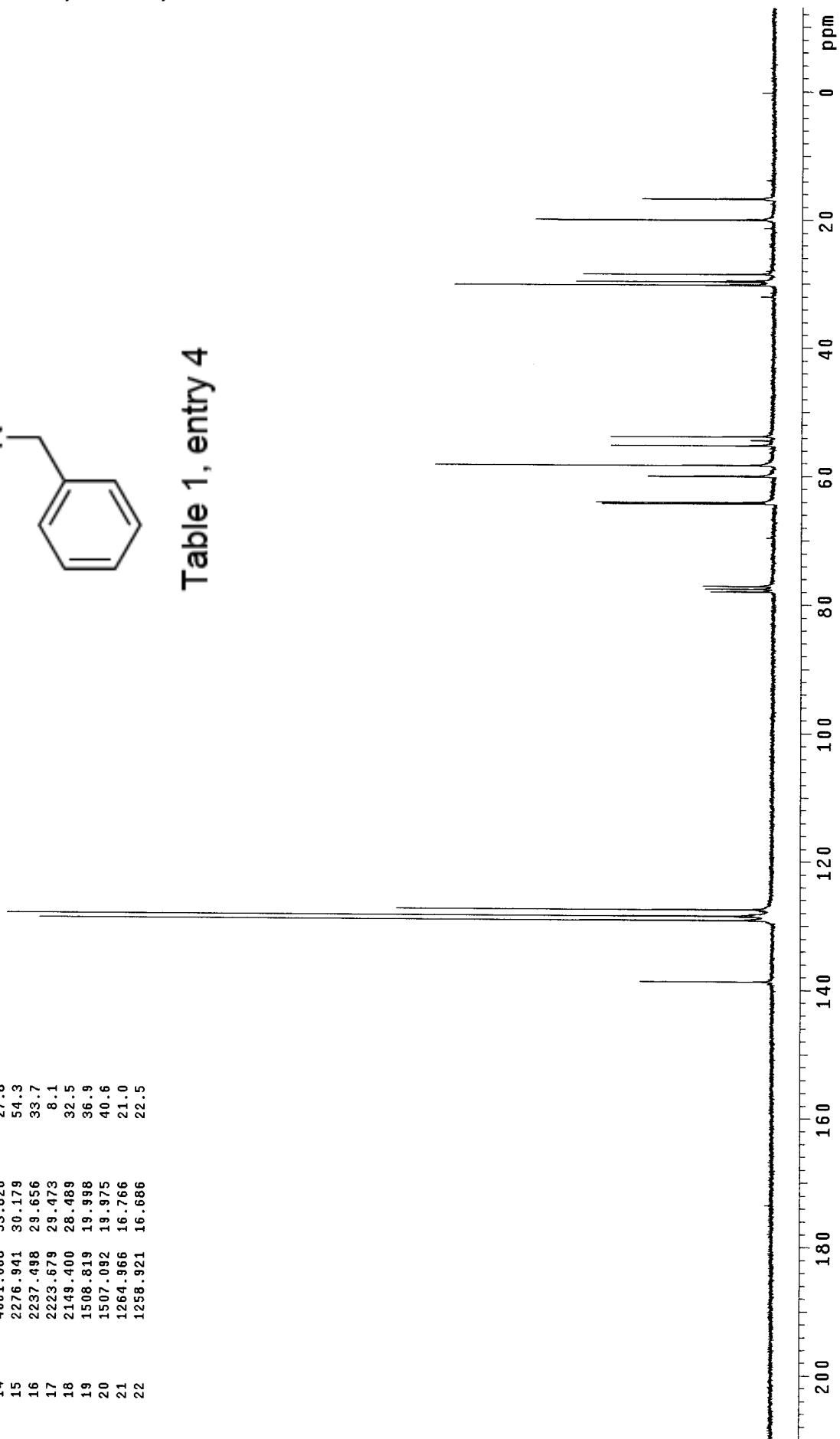


Table 1, entry 4



INDEX	FREQUENCY	PPM	HEIGHT
1	6148.384	50.619	52.3
2	5999.060	49.390	40.8

³¹P-NMR (¹H decoupled)

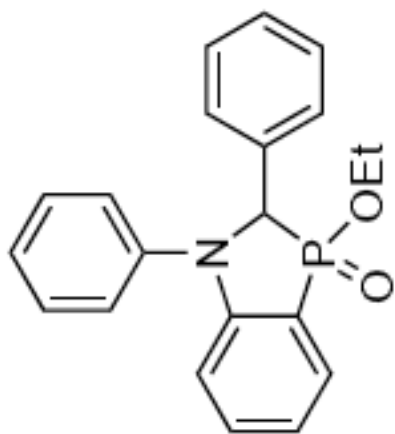
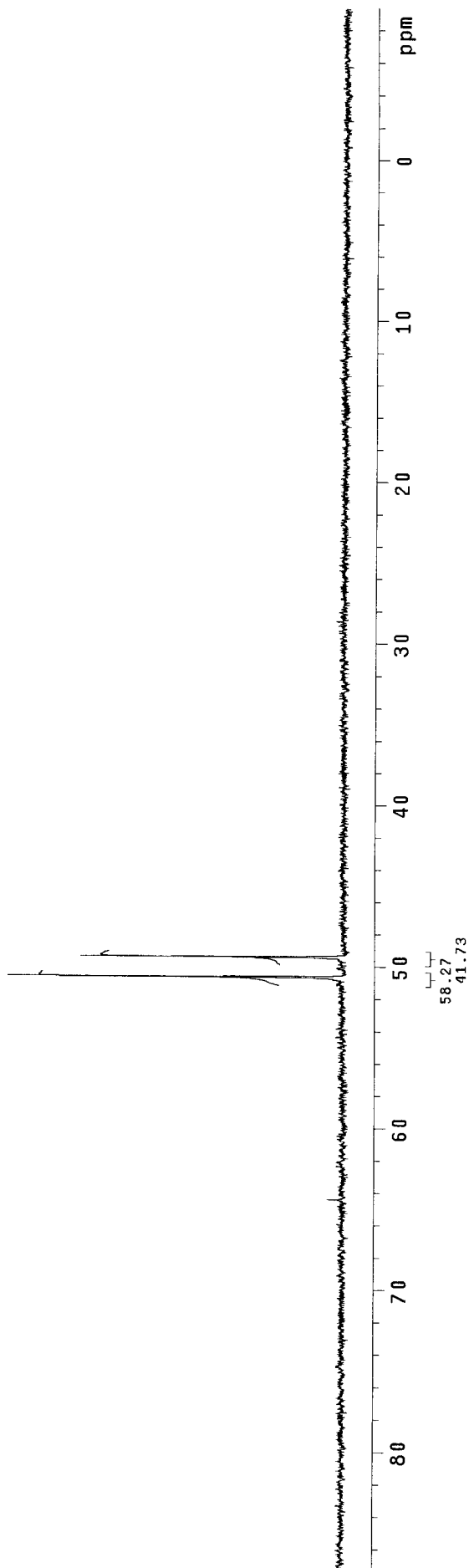


Table 2, entry 1



INDEX	FREQUENCY	PPM	HEIGHT
1	6146.752	50.606	37.1
2	5996.612	49.370	31.2

³¹P-NMR (1H coupled)

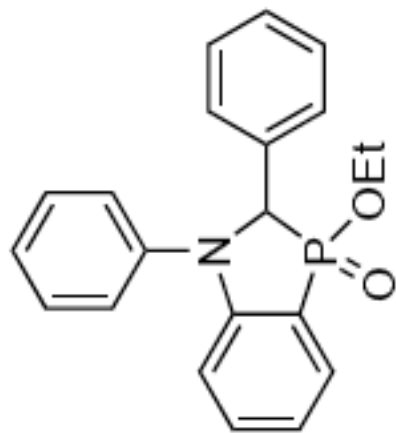
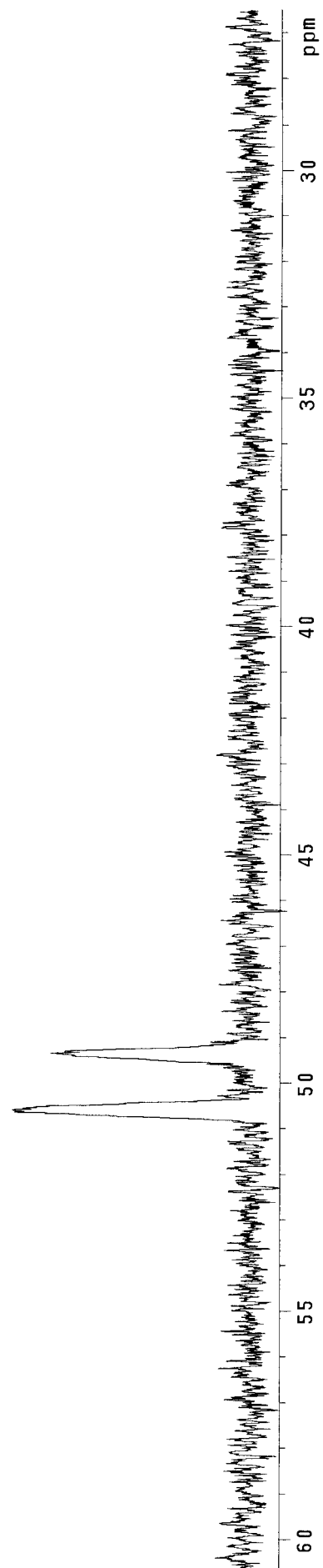


Table 2, entry 1



¹H-NMR

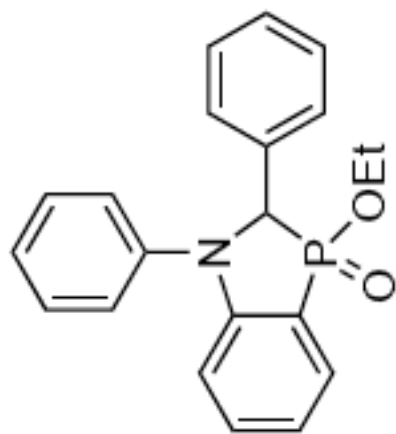
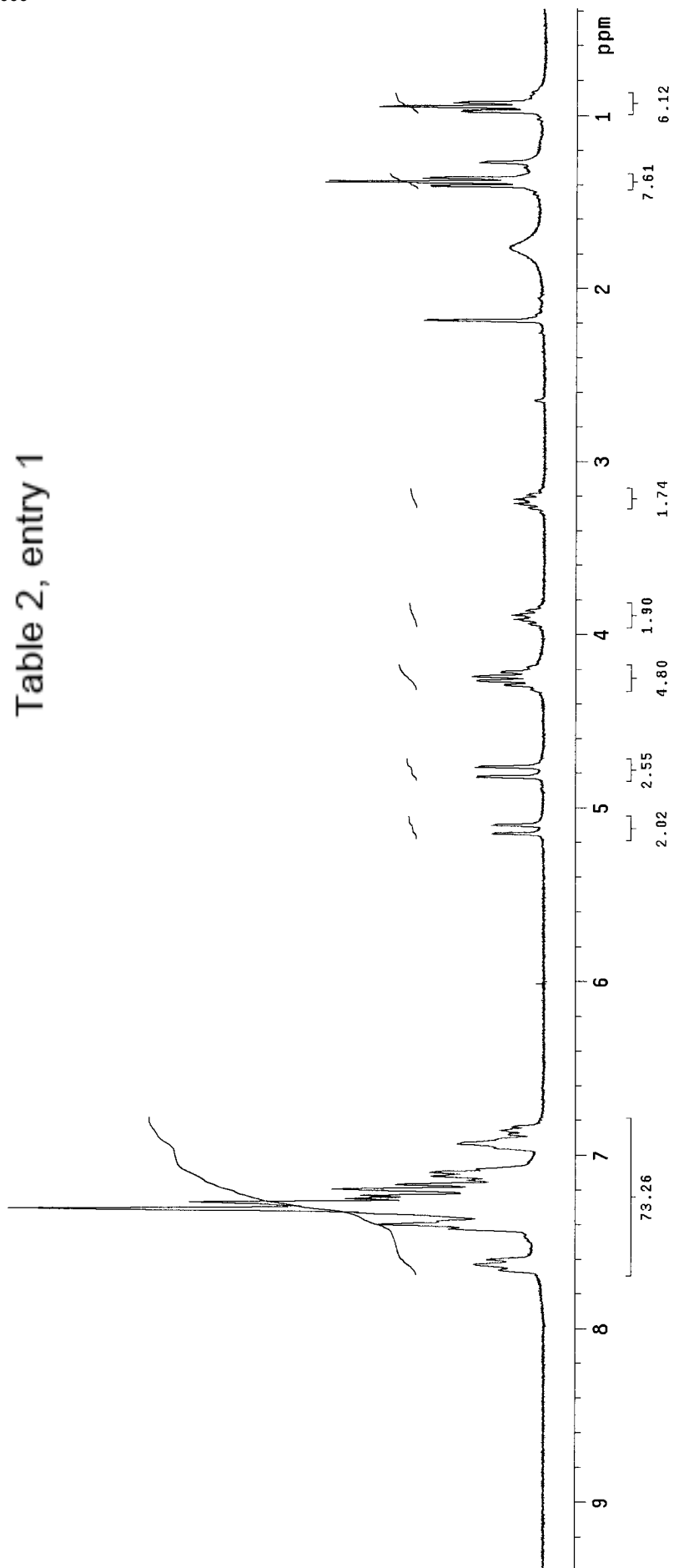


Table 2, entry 1



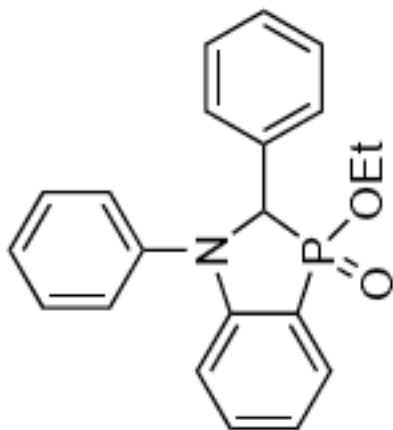
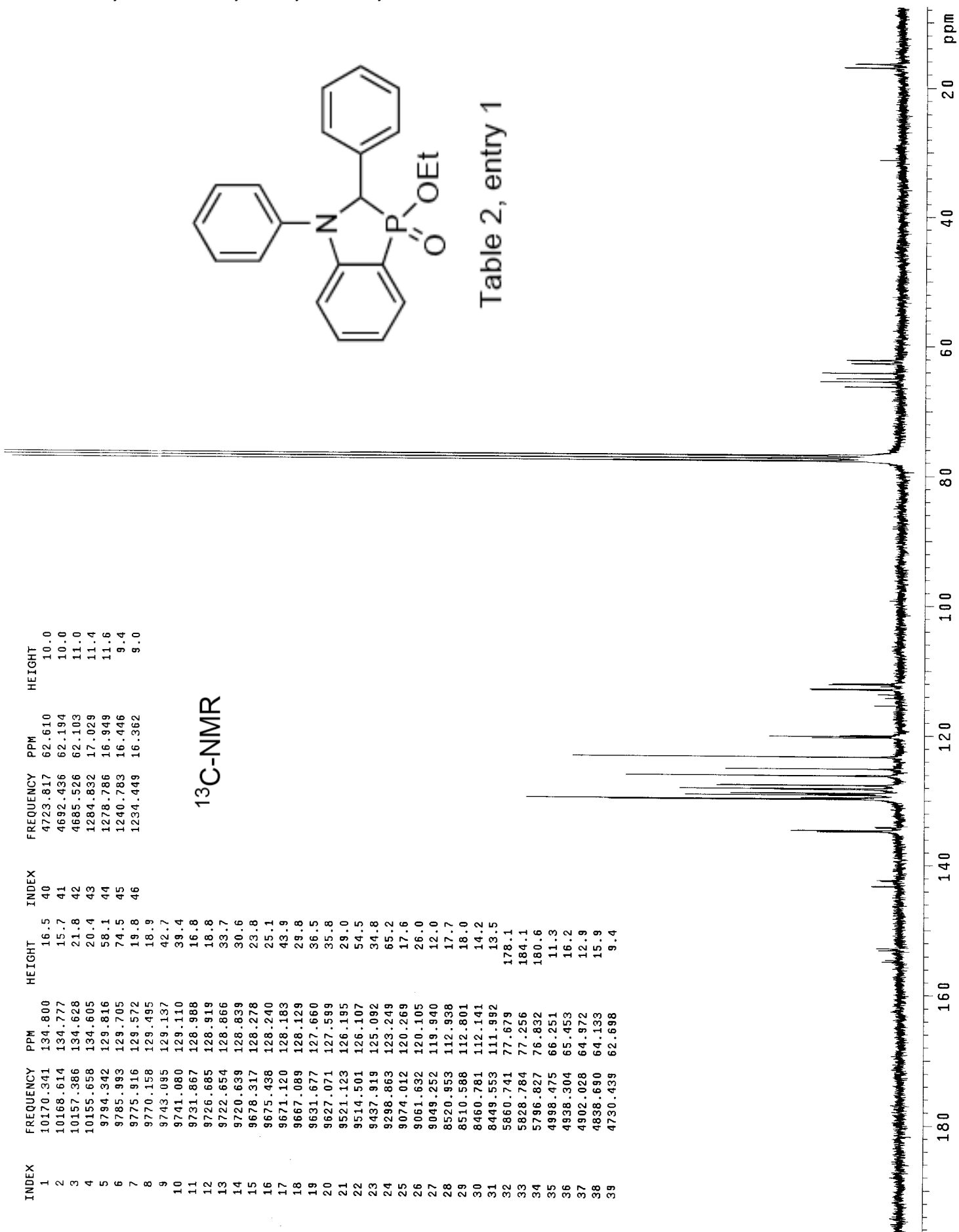


Table 2, entry 1

INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	10170.341	134.800	16.5	40	4723.817	62.610	10.0
2	10168.614	134.777	15.7	41	4692.436	62.194	10.0
3	10157.386	134.628	21.8	42	4685.526	62.103	11.0
4	10155.658	134.605	20.4	43	1284.832	17.029	11.4
5	9784.342	129.816	58.1	44	1278.786	16.949	11.6
6	9785.993	129.705	74.5	45	1240.783	16.446	9.4
7	9775.916	129.572	19.8	46	1234.449	16.362	9.0
8	9770.158	129.495	18.9				
9	9743.095	129.137	42.7				
10	9741.080	129.110	39.4				
11	9731.867	128.988	16.8				
12	9726.685	128.919	18.8				
13	9722.654	128.866	33.7				
14	9720.639	128.839	30.6				
15	9678.317	128.278	23.8				
16	9675.438	128.240	25.1				
17	9671.120	128.183	43.9				
18	9667.089	128.129	29.8				
19	9631.677	127.660	36.5				
20	9627.071	127.599	35.8				
21	9521.123	126.195	29.0				
22	9514.501	126.107	54.5				
23	9437.919	125.092	34.8				
24	9298.863	123.249	65.2				
25	9074.012	120.269	17.6				
26	9061.632	120.105	26.0				
27	9049.252	119.940	12.0				
28	8520.953	112.938	17.7				
29	8510.588	112.801	18.0				
30	8460.781	112.141	14.2				
31	8449.553	111.992	13.5				
32	5860.741	77.679	178.1				
33	5828.784	77.256	184.1				
34	5796.827	76.832	180.6				
35	4998.475	66.251	11.3				
36	4938.304	65.453	16.2				
37	4902.028	64.872	12.9				
38	4838.690	64.133	15.9				
39	4730.439	62.698	9.4				

¹³C-NMR



INDEX	FREQUENCY	PPM	HEIGHT
1	6374.818	52.484	35.4

^{31}P -NMR (^1H decoupled)

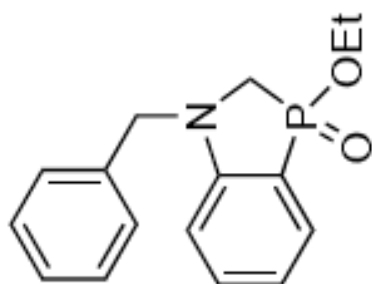
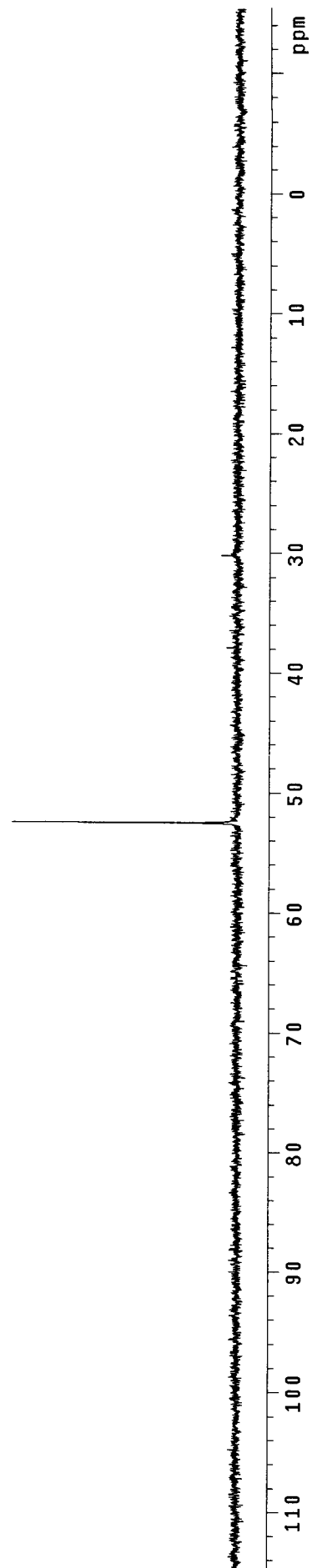


Table 2, entries 2 & 3



INDEX	FREQUENCY	PPM	HEIGHT
1	6374.410	52.480	30.6

³¹P-NMR (1H coupled)

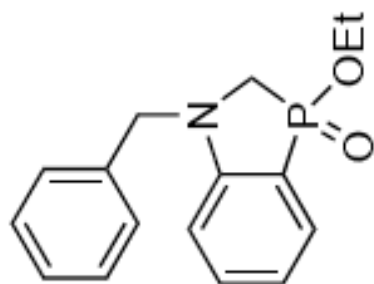
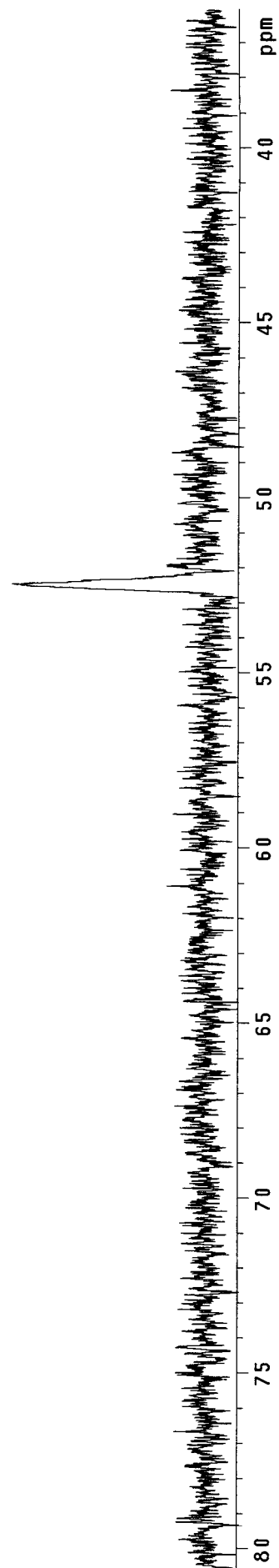


Table 2, entries 2 & 3



¹H-NMR

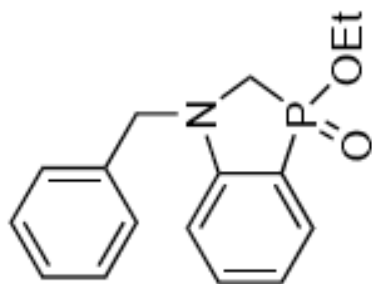
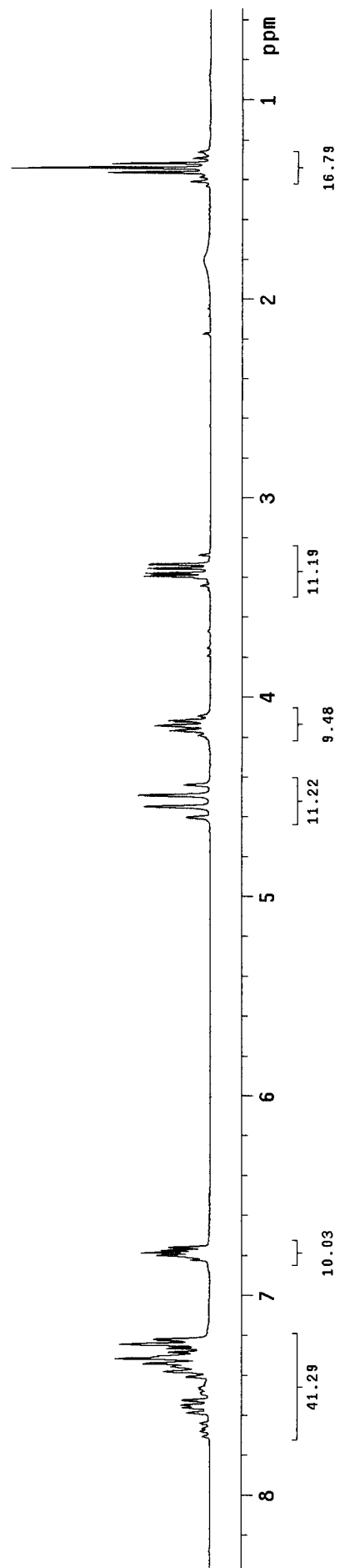


Table 2, entries 2 & 3



¹³C-NMR

INDEX	FREQUENCY PPM	HEIGHT
1	11727.314	6.3
2	11703.706	6.6
3	10318.899	15.6
4	10188.767	27.2
5	10186.752	27.7
6	9987.812	9.2
7	9977.735	10.4
8	9973.129	5.5
9	9739.928	86.4
10	9720.351	12.0
11	9715.169	26.9
12	9709.411	27.3
13	9648.376	48.8
14	9616.131	85.9
15	8899.543	24.3
16	8887.451	24.2
17	8610.490	5.3
18	8479.495	5.5
19	8286.677	24.1
20	8285.161	24.5
21	5869.378	17.3
22	5837.421	18.1
23	5805.176	18.2
24	4674.298	12.4
25	4667.964	13.9
26	3968.651	25.0
27	3957.999	25.6
28	3643.898	24.1
29	3541.693	24.5
30	1271.876	15.7
31	1265.542	15.3

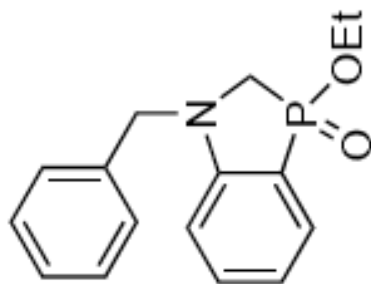
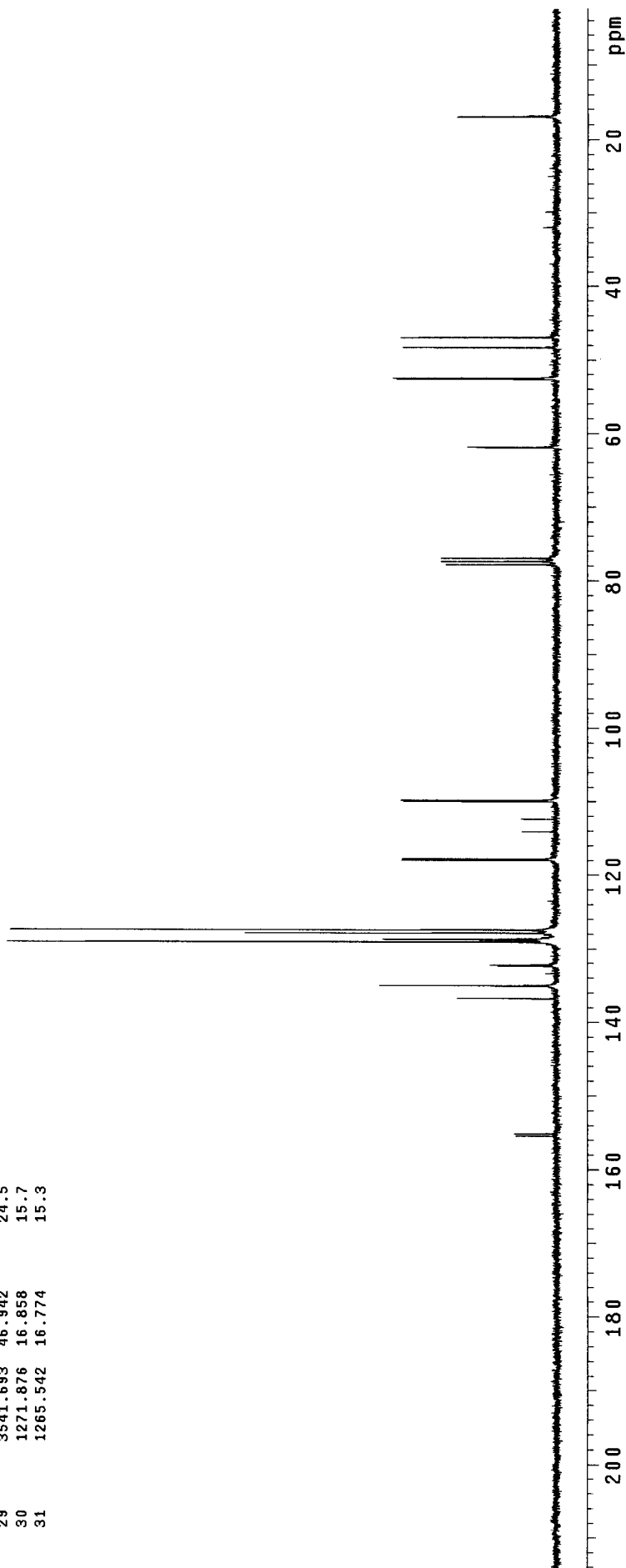


Table 2, entries 2 & 3



INDEX	FREQUENCY	PPM	HEIGHT
1	6202.646	51.066	38.0
2	6049.650	49.807	25.5

^{31}P -NMR (^1H decoupled)

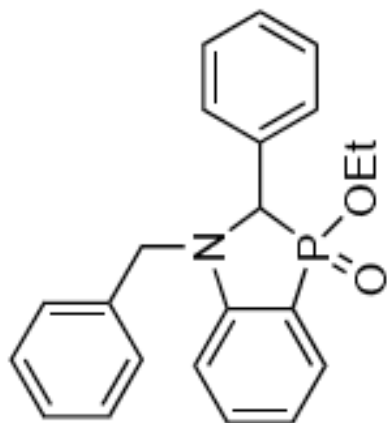
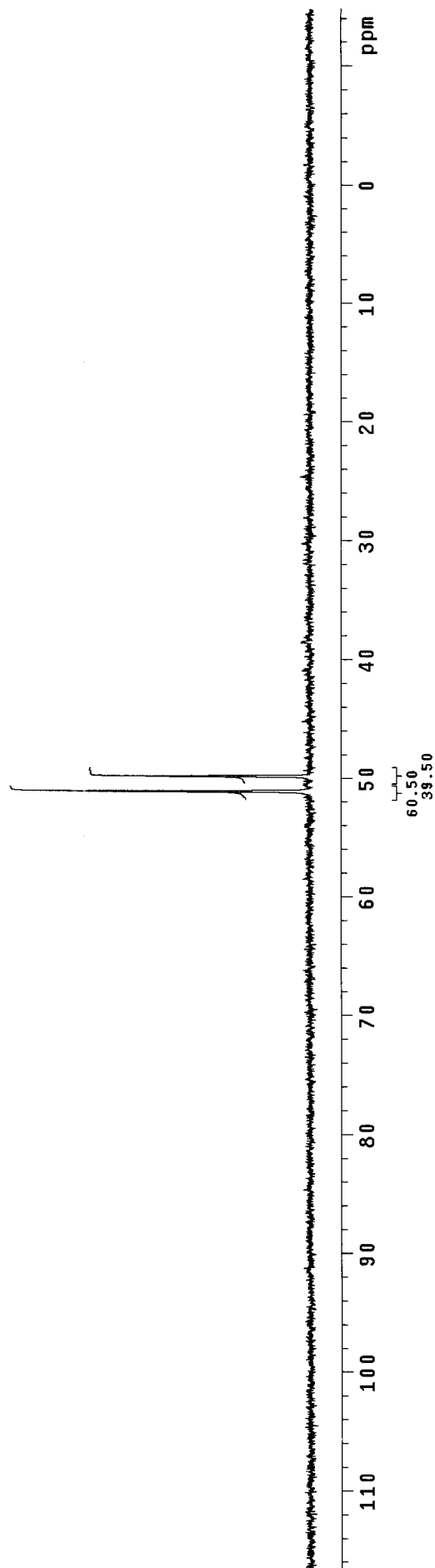


Table 2, entry 4



INDEX	FREQUENCY PPM	HEIGHT
1	6205.502	25.3
2	6051.282	18.1

³¹P-NMR (¹H coupled)

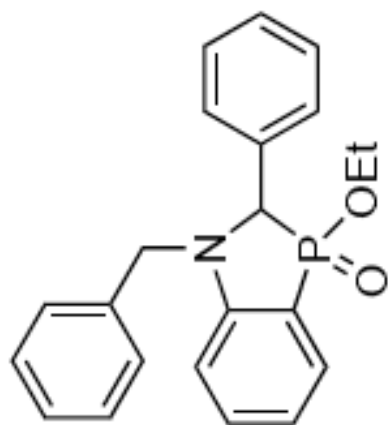
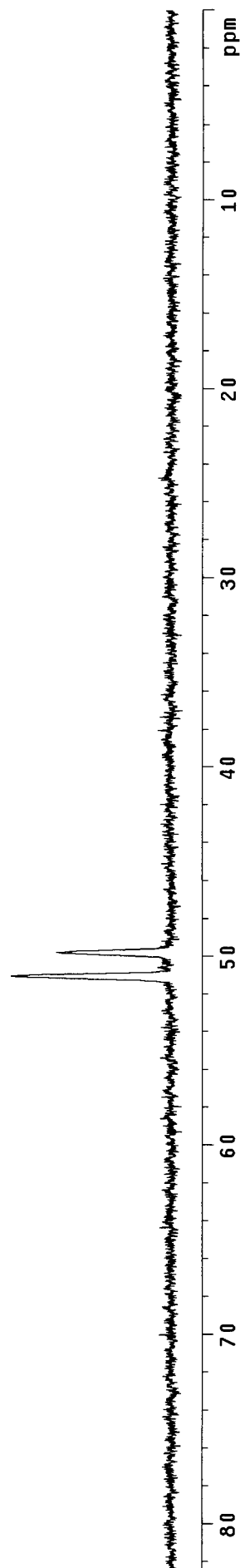


Table 2, entry 4



¹H-NMR

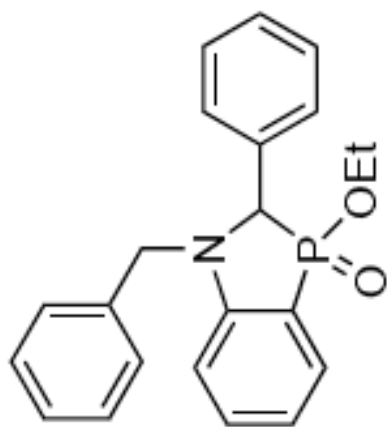
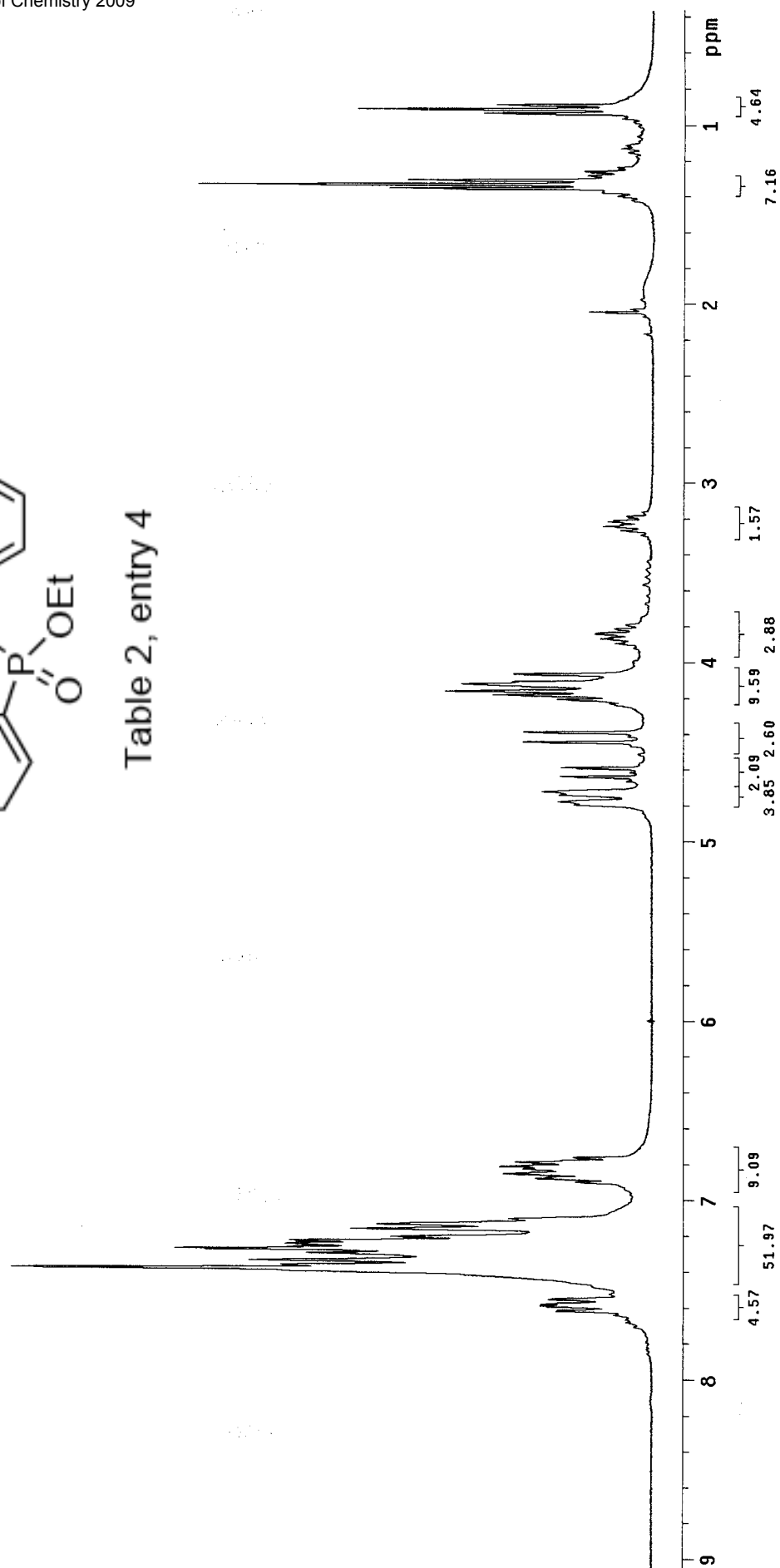


Table 2, entry 4



¹³C-NMR

INDEX	FREQUENCY PPM	HEIGHT	INDEX	FREQUENCY PPM	HEIGHT
1	11724.722	155.402	37	40	9.5
2	11701.978	155.100	41	8264.144	109.534
3	11681.825	154.833	42	8253.204	109.389
4	11659.081	154.532	43	5872.833	77.840
5	10337.324	137.013	44	5840.876	77.416
6	10288.381	136.364	45	5808.631	76.989
7	10201.723	135.215	46	4834.372	64.076
8	10199.707	135.189	47	4736.773	62.782
9	10100.381	133.872	48	4725.257	62.629
10	10096.926	133.826	49	4715.756	62.503
11	10090.592	133.743	50	4708.846	62.412
12	9989.539	132.403	51	4676.026	61.977
13	9979.462	132.270	52	4669.404	61.889
14	9772.748	128.530	53	4623.915	61.286
15	9767.567	129.461	54	3705.509	49.113
16	9753.172	129.270	55	3697.448	49.007
17	9751.444	129.247	56	3688.811	48.892
18	9745.686	129.171	57	1285.408	17.037
19	9743.383	129.141	58	1279.362	16.957
20	9733.307	129.007	59	1241.646	16.457
21	9722.078	128.858	60	1235.313	16.373
22	9709.699	128.694	61	22.971	0.300
23	9700.486	128.572	62		
24	9697.895	128.538	63		
25	9686.091	128.381	64		
26	9681.772	128.324	65		
27	9637.435	127.736	66		
28	9634.268	127.694	67		
29	9629.662	127.633	68		
30	9602.887	127.278	69		
31	8964.897	118.822	70		
32	8952.805	118.662	71		
33	8923.151	118.269	72		
34	8911.059	118.109	73		
35	8562.410	113.488	74		
36	8536.211	113.140	75		
37	8429.112	111.721	76		
38	8406.368	111.420	77		
39	8361.743	110.828	78		

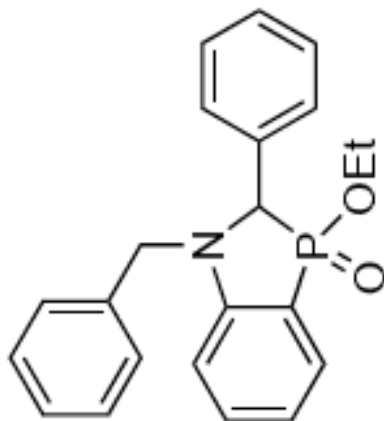
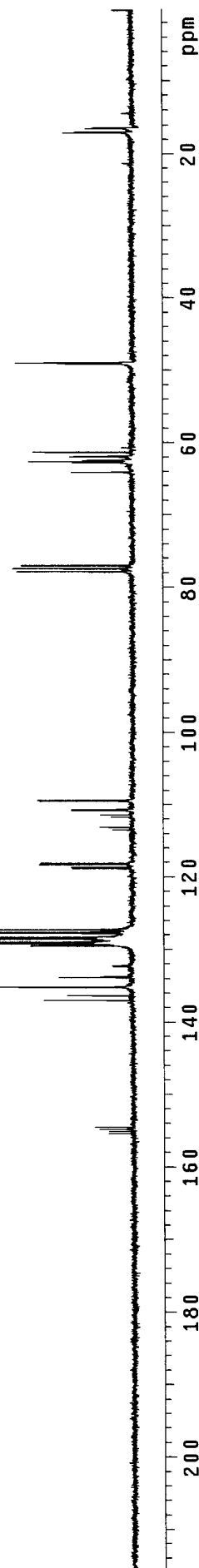


Table 2, entry 4



INDEX	FREQUENCY	PPM	HEIGHT
1	6359.314	52.356	30.6
2	6229.574	51.288	19.3

³¹P-NMR (¹H coupled)

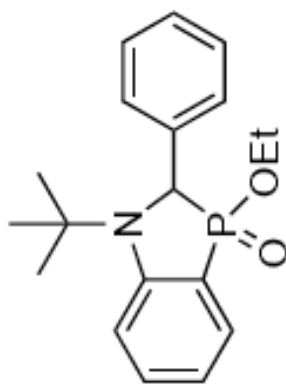
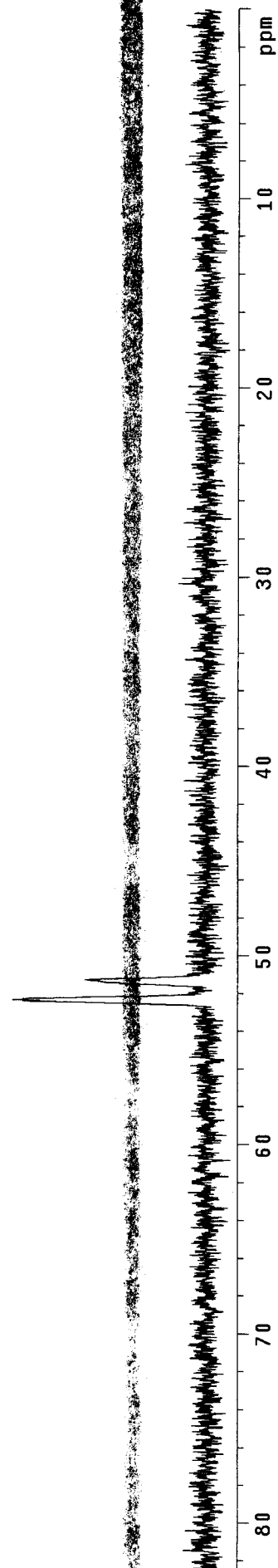


Table 2, entry 5



¹H-NMR

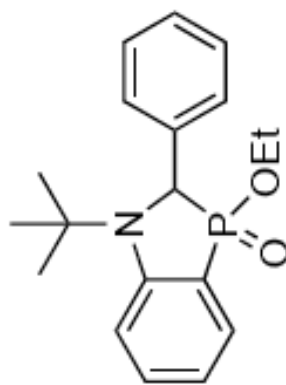
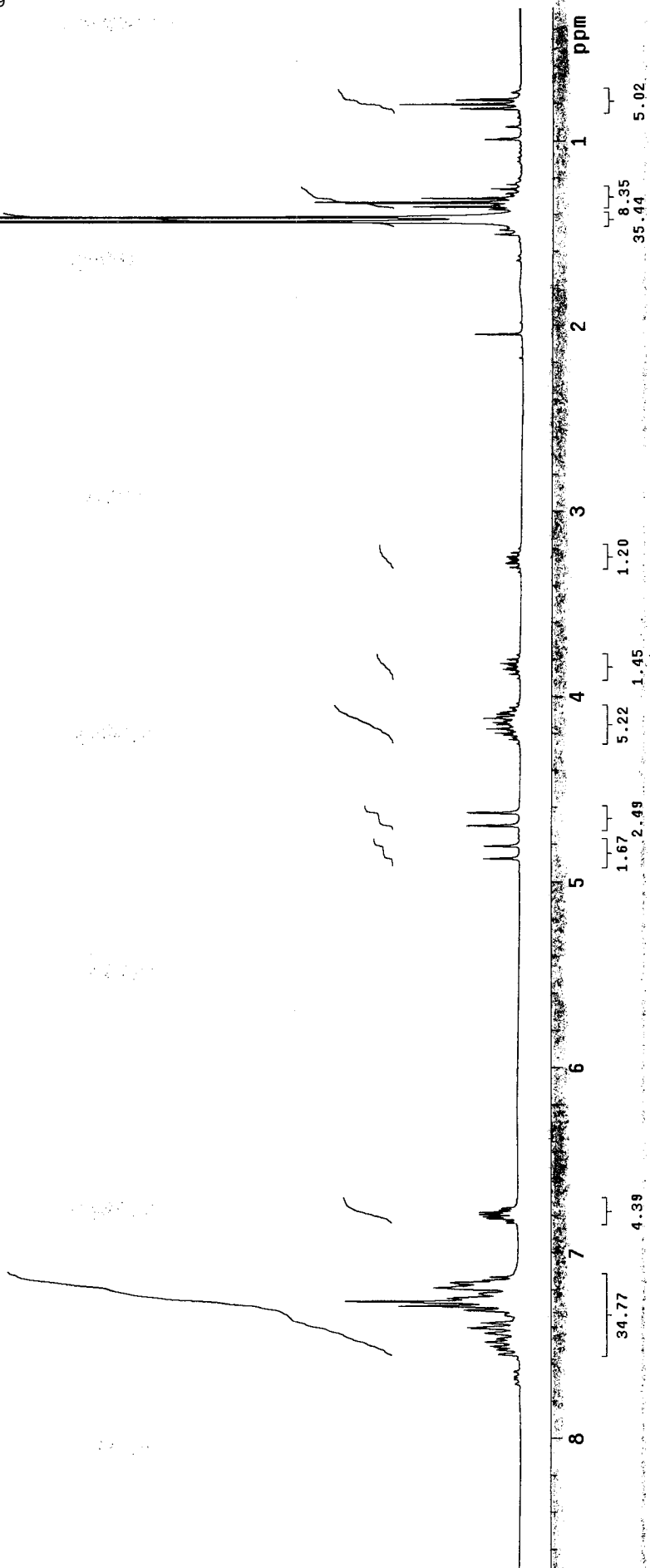


Table 2, entry 5



¹³C-NMR

INDEX	FREQUENCY PPM	HEIGHT	INDEX	FREQUENCY PPM	HEIGHT
1	11687.583	154.909	6.7	40	43.1
2	11662.248	154.574	6.5	41	45.8
3	11637.200	154.242	9.9	42	44.8
4	11613.880	153.932	9.7	43	16.3
5	10501.716	139.192	9.1	44	18.0
6	10487.398	139.134	10.1	45	23.5
7	10343.370	137.093	23.9	46	21.7
8	10124.277	134.189	30.4	47	22.4
9	10121.686	134.155	51.6	48	22.0
10	10119.671	134.128	43.0	49	36.8
11	9987.236	132.373	11.3	50	35.9
12	9977.159	132.239	12.2	51	13.1
13	9815.934	130.102	35.3	52	13.1
14	9809.888	130.022	35.0	53	20.2
15	9760.657	129.370	24.4	54	20.1
16	9755.763	129.305	25.0	55	5.6
17	9736.473	129.049	82.3	56	146.3
18	9734.170	129.018	102.5	57	207.1
19	9731.003	128.976	59.8	58	23.6
20	9719.199	128.820	16.3	59	21.2
21	3707.107	128.660	16.2	60	15.5
22	9644.057	127.824	38.0	61	14.9
23	8641.178	127.786	38.3		
24	9631.101	127.652	26.0		
25	9628.510	127.618	26.5		
26	9579.855	126.973	77.7		
27	9575.824	126.920	76.0		
28	9569.778	126.840	51.2		
29	9565.748	126.786	48.6		
30	8915.954	118.174	22.7		
31	8903.862	118.013	22.4		
32	8884.284	117.754	35.5		
33	8872.193	117.594	34.0		
34	8717.301	115.541	6.4		
35	8666.055	114.861	21.8		
36	8654.539	114.709	21.2		
37	8611.642	114.140	33.7		
38	8601.277	114.003	33.9		
39	8589.185	113.843	8.1		

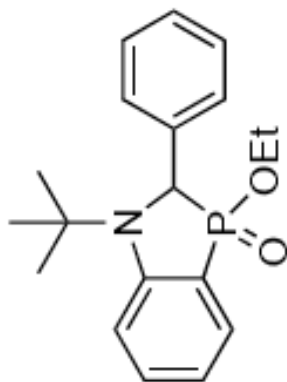
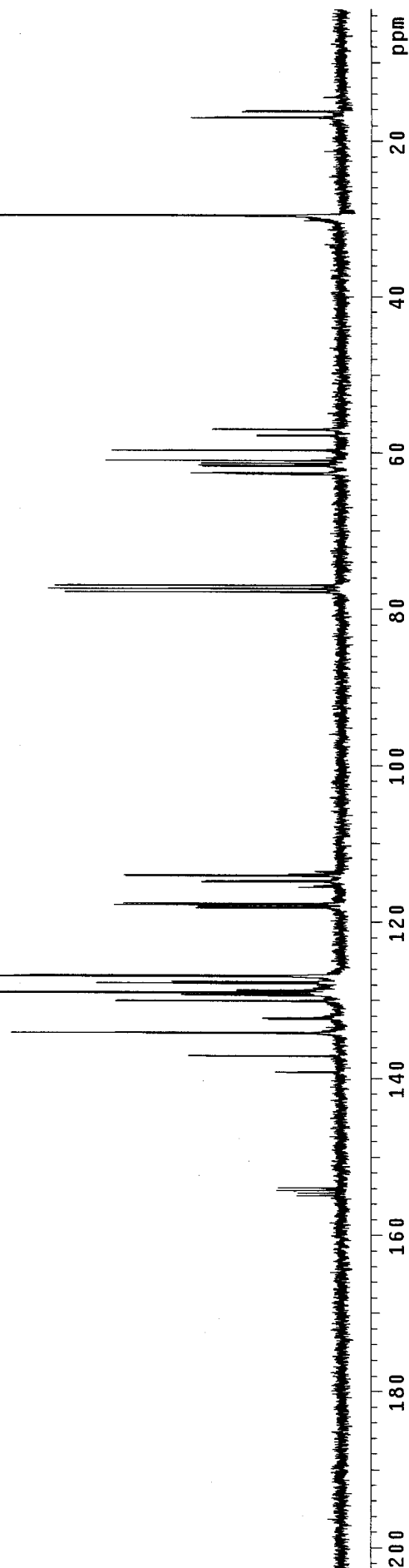


Table 2, entry 5



INDEX	FREQUENCY	PPM	HEIGHT
1	6131.656	50.482	126.0
2	5967.237	49.128	110.8

^{31}P -NMR (^1H decoupled)

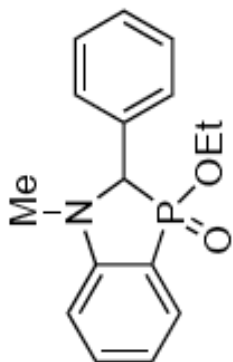
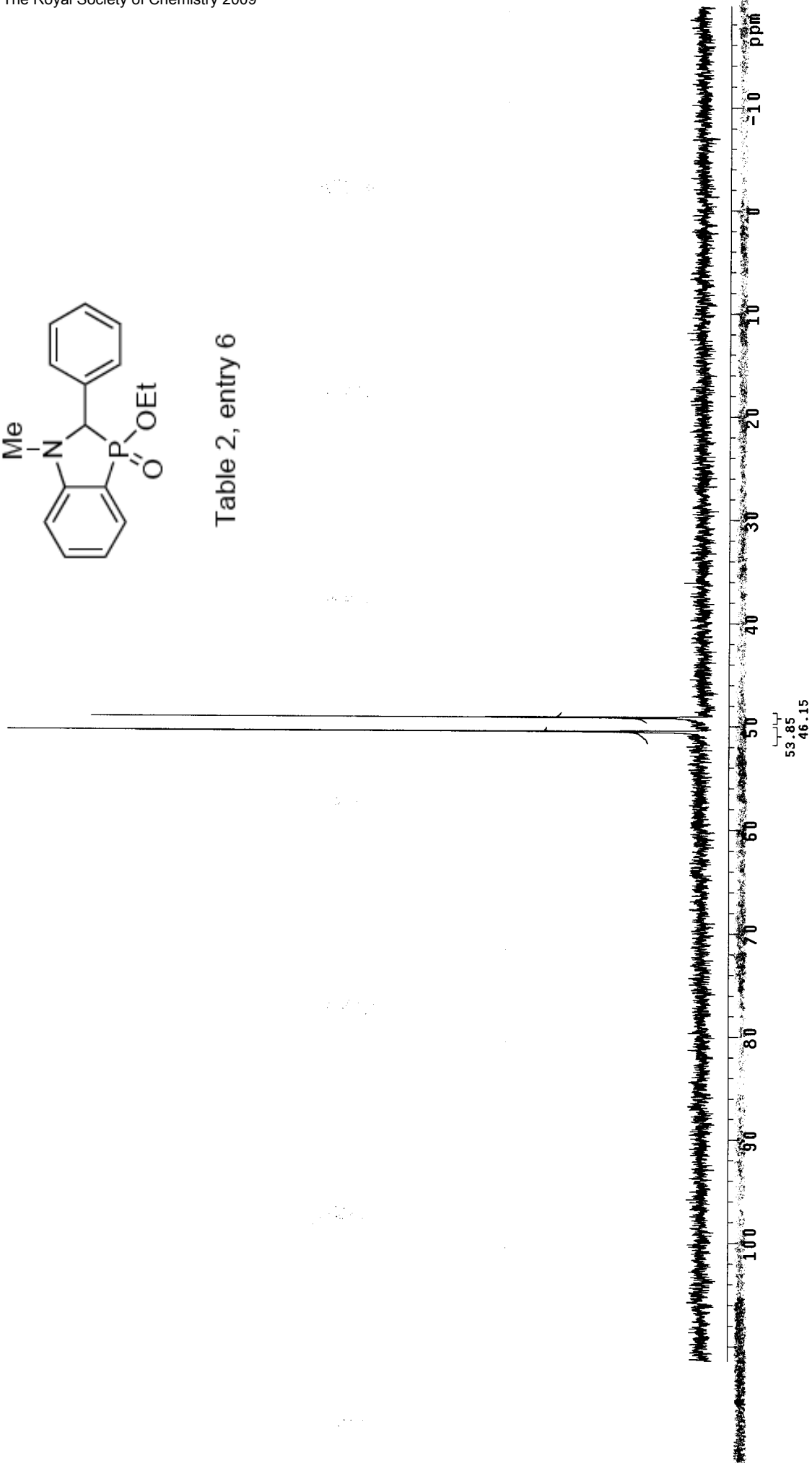


Table 2, entry 6



INDEX	FREQUENCY	PPM	HEIGHT
1	6132.880	50.492	29.9
2	5969.685	49.148	30.7

³¹P-NMR (¹H coupled)

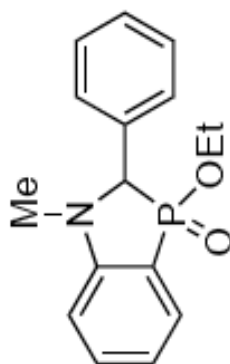
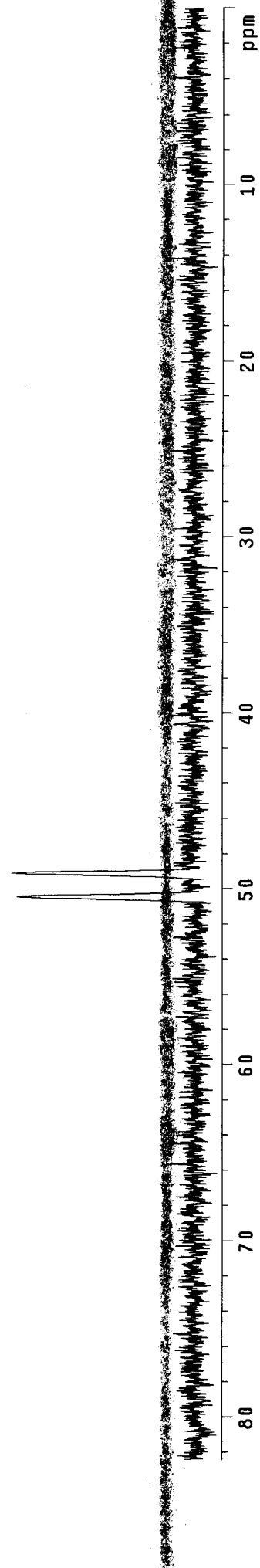


Table 2, entry 6



¹H-NMR

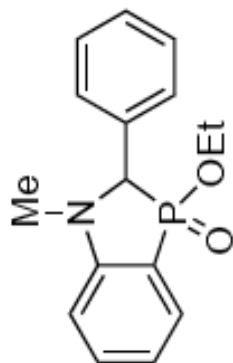
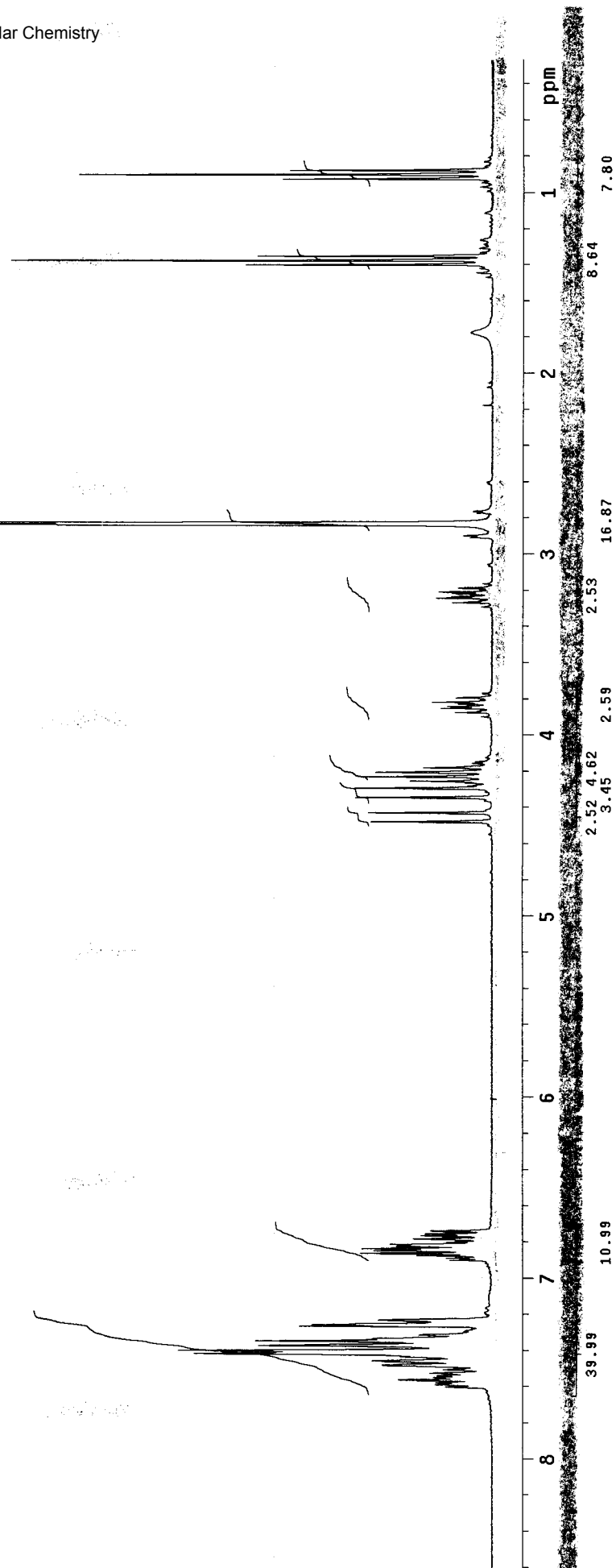


Table 2, entry 6



¹³C-NMR

INDEX	FREQUENCY PPM	HEIGHT	INDEX	FREQUENCY PPM	HEIGHT
1	11795.258	156.336	6.5	40	35.3
2	11772.226	156.031	6.8	41	26.4
3	11733.935	155.524	7.1	42	25.8
4	11710.903	155.218	7.3	43	28.2
5	10190.844	135.177	30.9	44	28.4
6	10196.828	135.151	34.4	45	16.6
7	10194.813	135.124	36.3	46	17.7
8	10192.798	135.097	33.8	47	16.1
9	10113.913	134.052	10.1	48	17.7
10	10107.003	133.960	10.8	49	21.0
11	10103.836	133.918	11.1	50	21.0
12	10099.517	133.861	11.0	51	21.6
13	9747.702	129.198	70.7	52	22.7
14	9745.686	129.171	93.4	53	17.6
15	9740.792	129.106	79.0	54	18.2
16	9738.489	129.076	68.2	55	16.7
17	9727.836	128.935	30.4	56	17.3
18	9722.654	128.866	30.1		
19	9693.000	128.473	34.5		
20	9690.121	128.435	50.7		
21	9686.954	128.393	33.2		
22	9668.241	128.145	61.3		
23	9663.634	128.084	61.5		
24	9647.224	127.866	36.3		
25	9643.193	127.813	36.6		
26	8968.928	118.876	25.0		
27	8956.836	118.716	24.8		
28	8907.317	118.059	28.0		
29	8895.225	117.899	27.5		
30	8572.199	113.617	5.4		
31	8552.046	113.350	6.1		
32	8439.188	111.855	5.4		
33	8422.490	111.633	6.1		
34	8326.907	110.366	24.7		
35	8315.679	110.218	25.4		
36	8239.385	109.206	28.5		
37	8228.444	109.061	28.6		
38	5868.802	77.786	34.0		
39	5836.845	77.363	35.7		

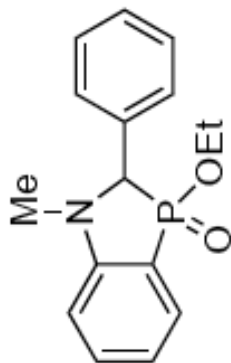
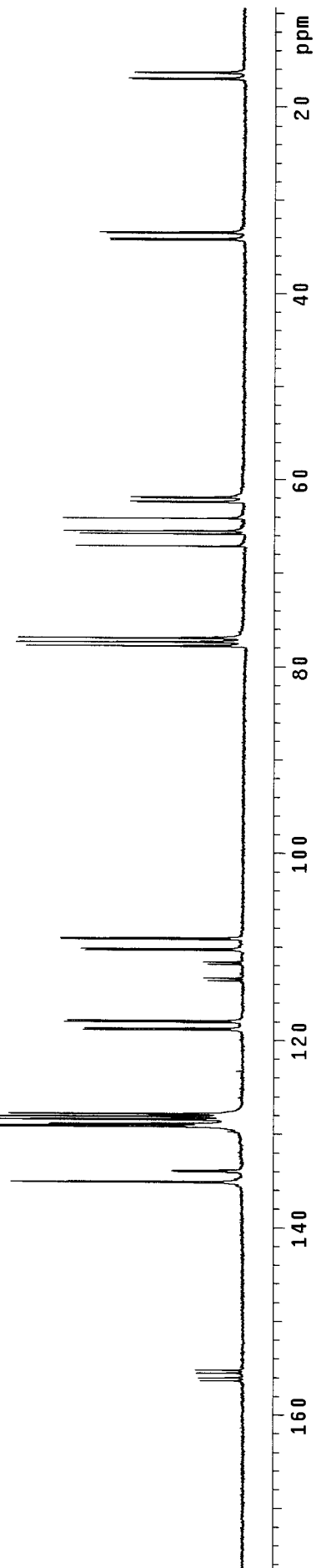


Table 2, entry 6



INDEX	FREQUENCY	PPM	HEIGHT
1	6610.227	54.422	62.4
2	5225.921	43.025	126.0

³¹P-NMR (¹H decoupled)

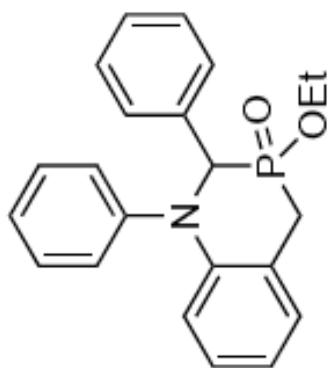
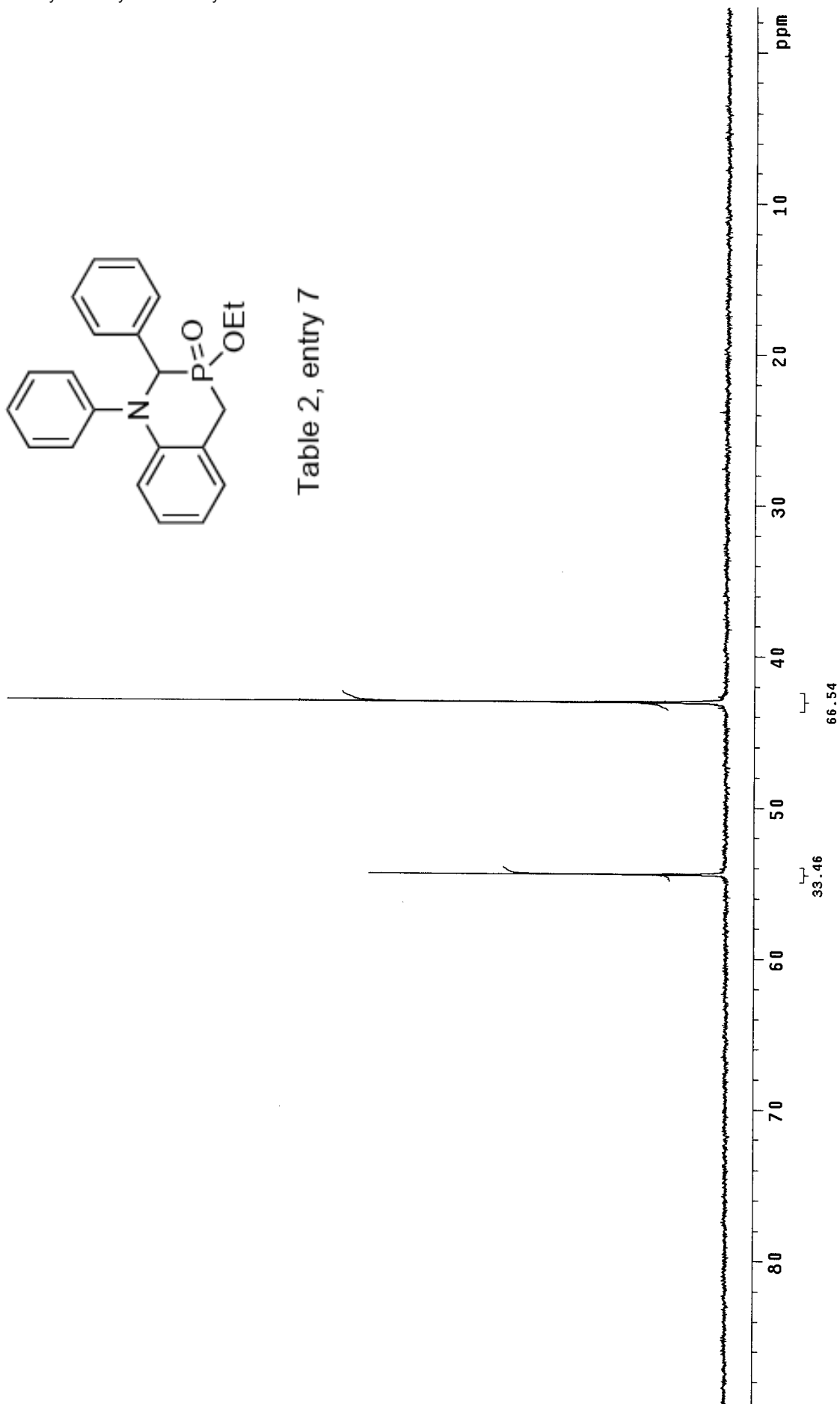


Table 2, entry 7



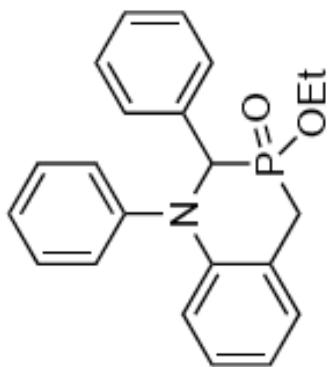
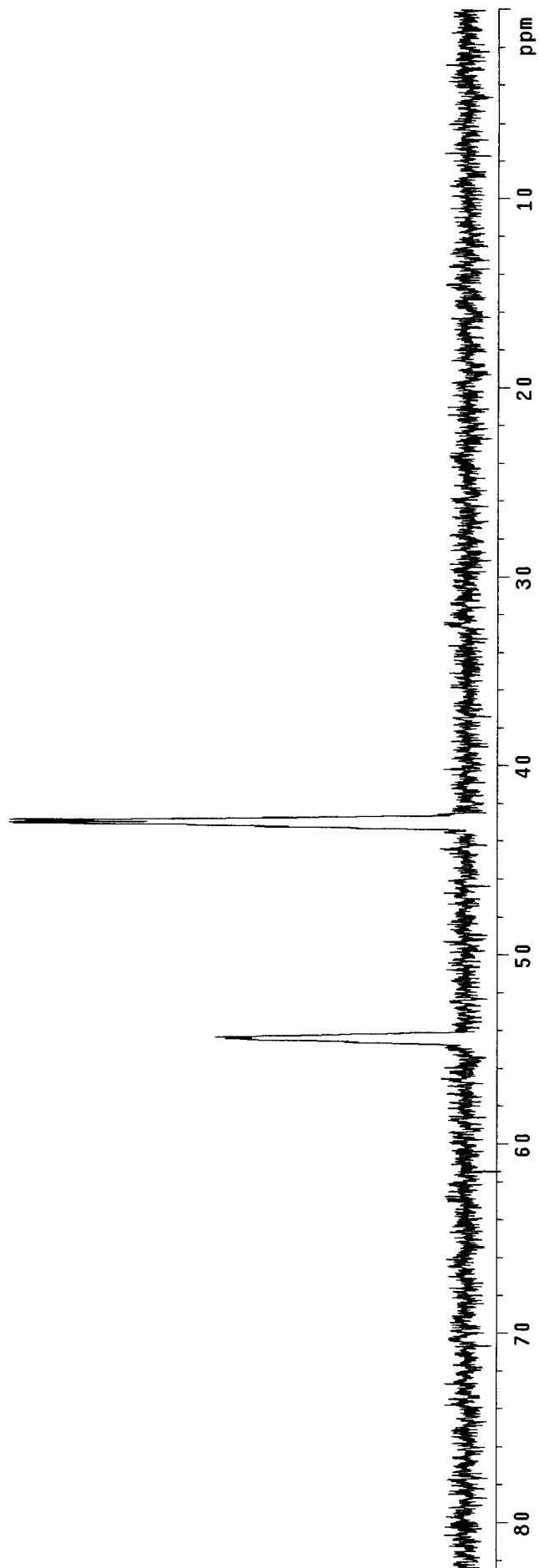


Table 2, entry 7

INDEX	FREQUENCY	PPM	HEIGHT
1	6605.739	54.385	39.2
2	5239.384	43.136	71.0
3	5231.224	43.069	71.6
4	5222.248	42.995	69.0
5	5213.681	42.924	71.5

³¹P-NMR (¹H coupled)



$^1\text{H-NMR}$

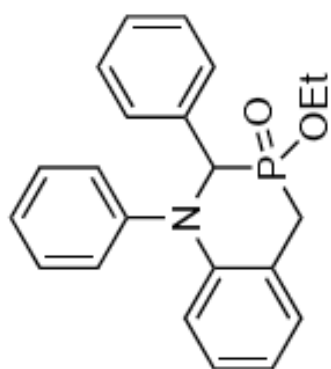
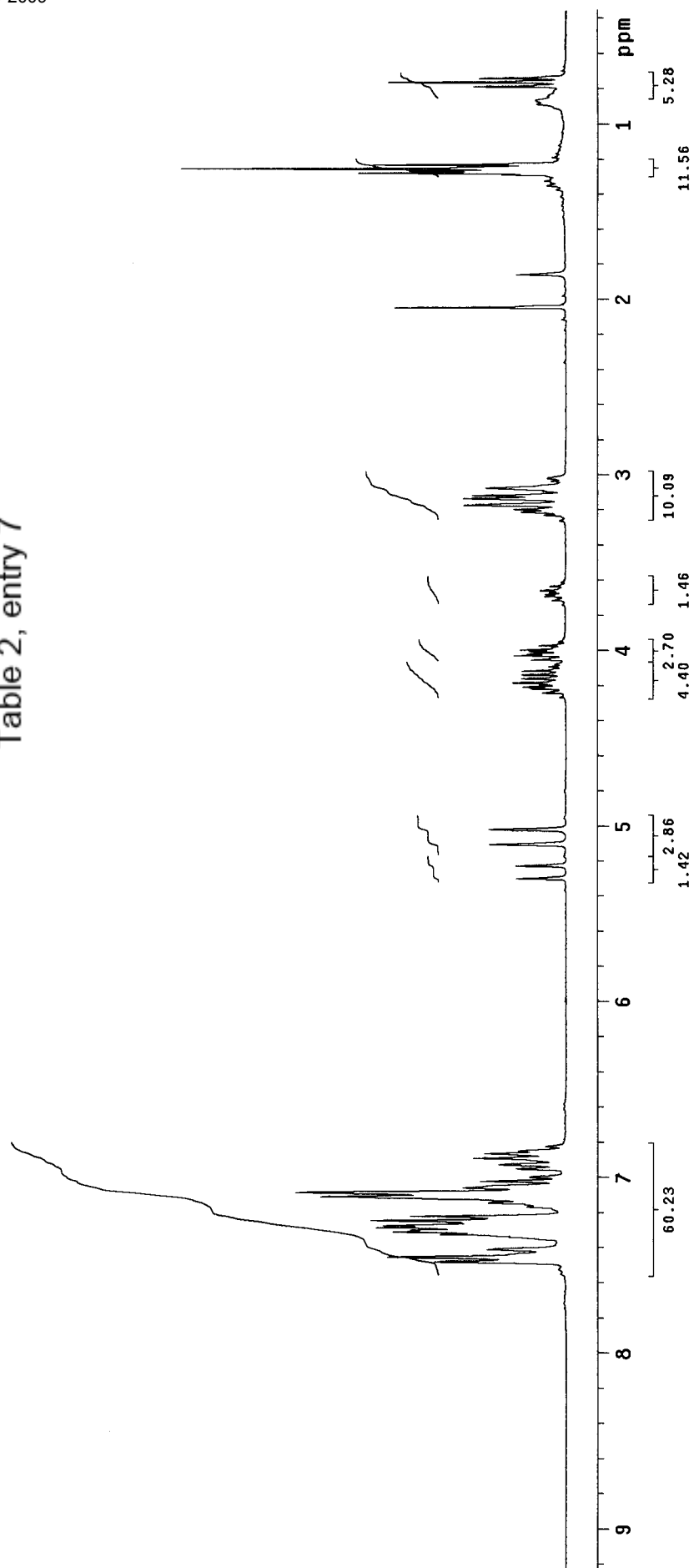


Table 2, entry 7



¹³C-NMR

INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	11262.640	149.277	19.9	40	9175.641	121.616	21.1
2	11258.322	149.220	18.4	41	9169.307	121.532	21.0
3	11210.530	148.586	9.5	42	9125.258	120.948	27.4
4	11204.772	148.510	9.1	43	8850.888	117.311	56.8
5	10811.786	143.301	15.8	44	5883.485	77.981	24.9
6	10802.861	143.183	15.5	45	5851.528	77.557	25.9
7	10443.560	138.421	17.9	46	5819.283	77.130	25.3
8	10310.837	136.662	21.4	47	4817.961	63.858	14.1
9	10305.655	136.593	20.1	48	4784.565	63.415	31.1
10	9986.660	132.365	37.6	49	4722.954	62.599	14.5
11	9974.856	132.209	36.8	50	4693.588	62.210	30.7
12	8877.545	130.919	18.6	51	4660.767	61.775	15.4
13	9870.348	130.823	18.6	52	4653.857	61.683	18.2
14	9785.129	129.694	136.9	53	4650.690	61.641	28.4
15	9749.429	129.221	71.8	54	4644.069	61.553	27.1
16	9746.262	129.179	52.7	55	2421.180	32.091	15.5
17	9744.535	129.156	48.2	56	2348.053	31.122	48.3
18	9731.579	128.984	89.2	57	2337.400	30.980	15.6
19	9729.852	128.961	89.0	58	2229.437	29.549	10.5
20	9698.183	128.541	23.5	59	2199.495	29.153	33.9
21	9696.455	128.519	23.3	60	2113.413	28.012	33.9
22	9677.166	128.263	68.3	61	1266.982	16.793	28.9
23	9647.512	127.870	61.3	62	1261.224	16.716	28.3
24	9645.209	127.839	44.3	63	1237.328	16.400	14.4
25	9606.054	127.320	80.1	64	1232.146	16.331	13.9
26	9601.735	127.263	78.3				
27	9573.233	126.885	39.1				
28	9569.490	126.836	38.7				
29	9536.247	126.660	11.4				
30	9546.170	126.527	10.1				
31	9490.605	125.790	19.6				
32	9488.014	125.756	19.3				
33	9436.192	125.069	31.5				
34	9309.603	123.394	62.1				
35	9231.494	122.356	45.2				
36	9229.479	122.329	45.3				
37	9222.569	122.238	49.9				
38	9221.705	122.226	50.2				
39	9201.552	121.959	127.7				

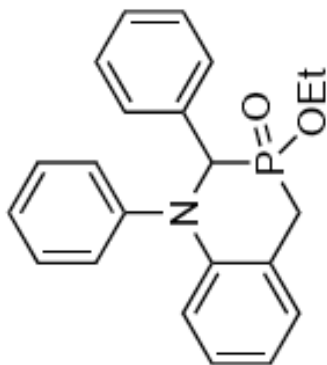
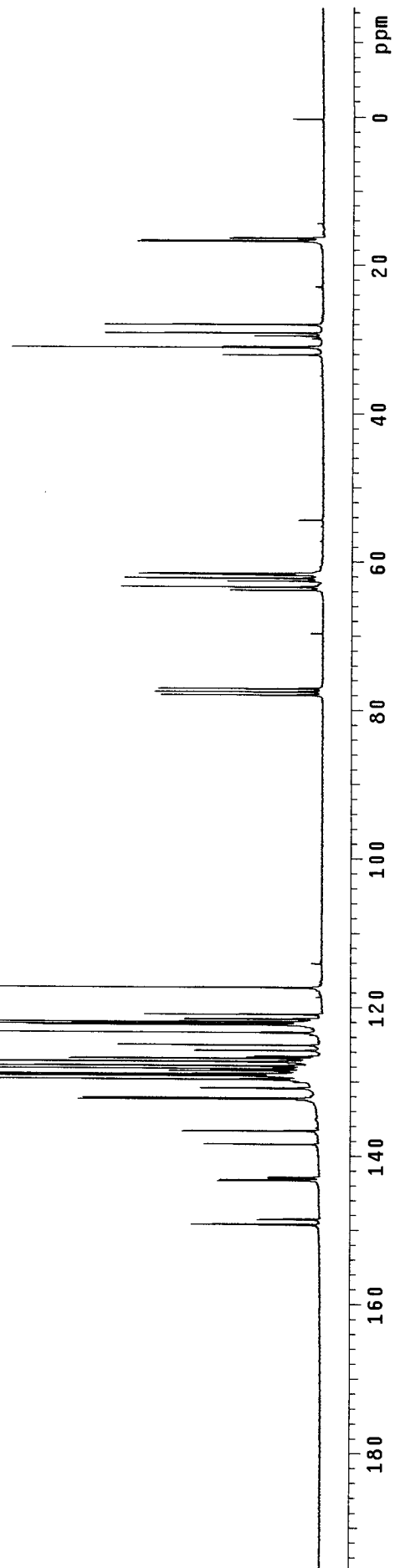


Table 2, entry 7



INDEX	FREQUENCY	PPM	HEIGHT
1	6952.530	57.240	99.9

³¹P-NMR (¹H decoupled)

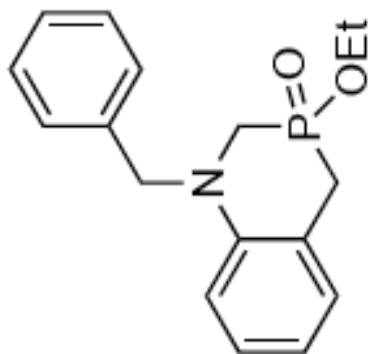
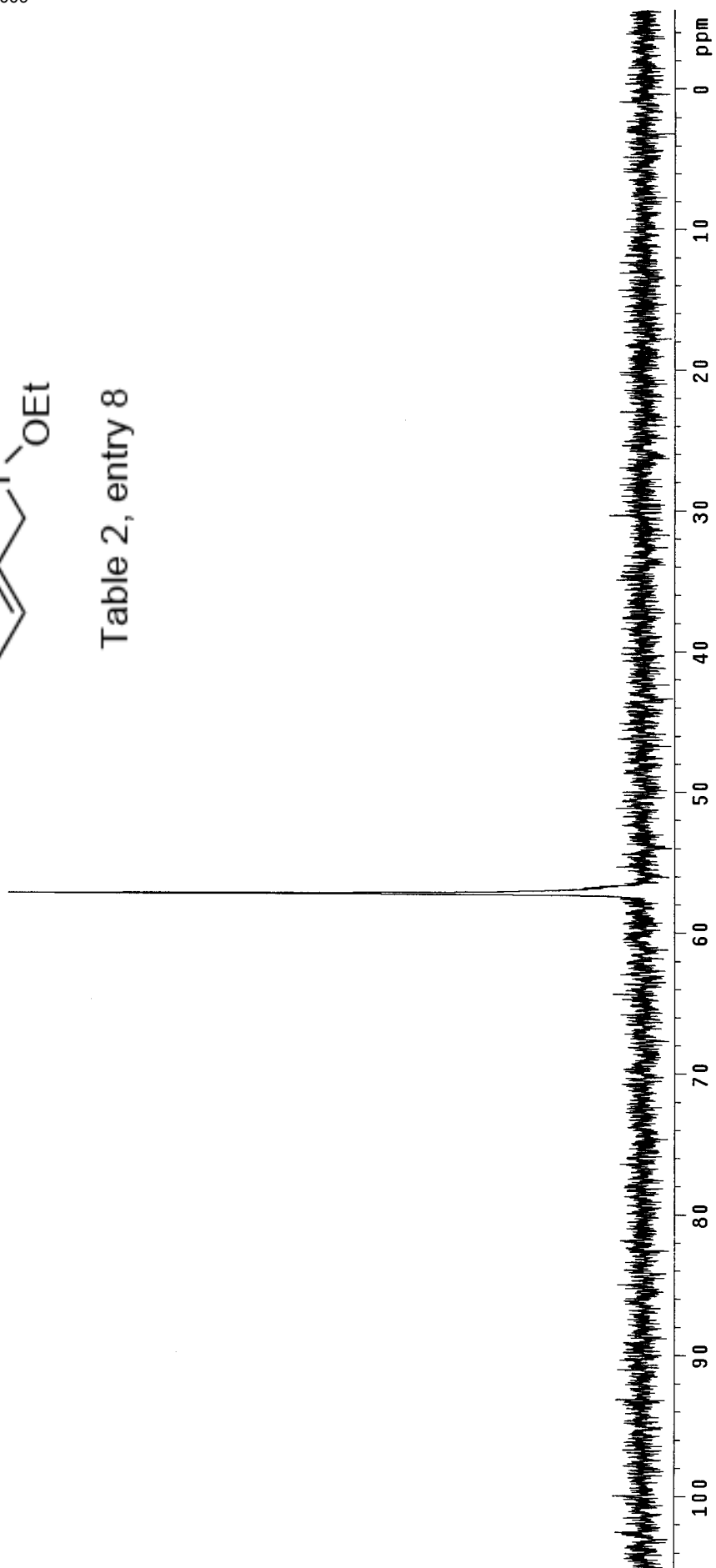


Table 2, entry 8



INDEX	FREQUENCY PPM	HEIGHT
1	6954.162	57.253
		31.8

³¹P-NMR (¹H coupled)

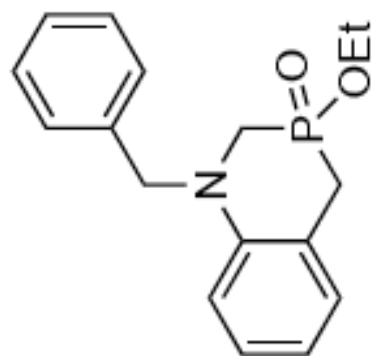
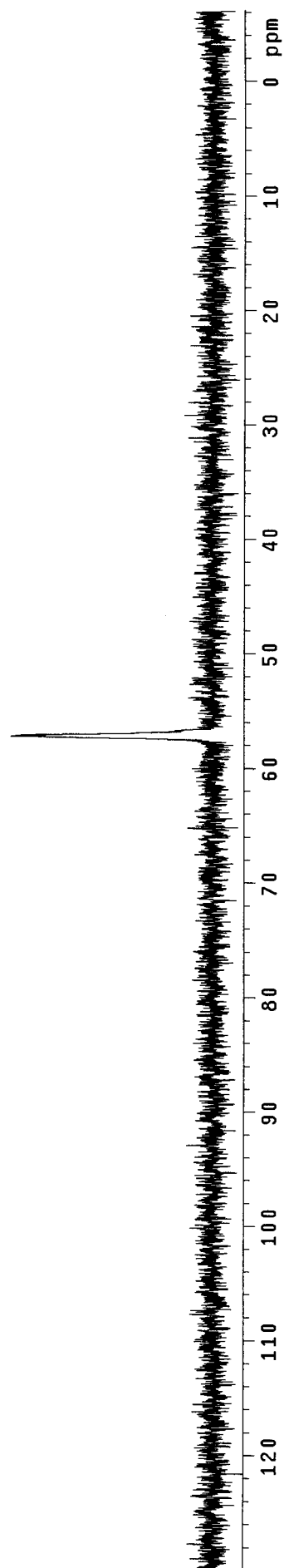


Table 2, entry 8



¹H-NMR

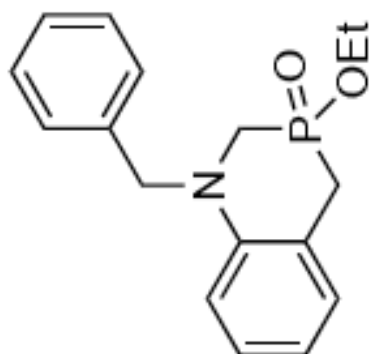
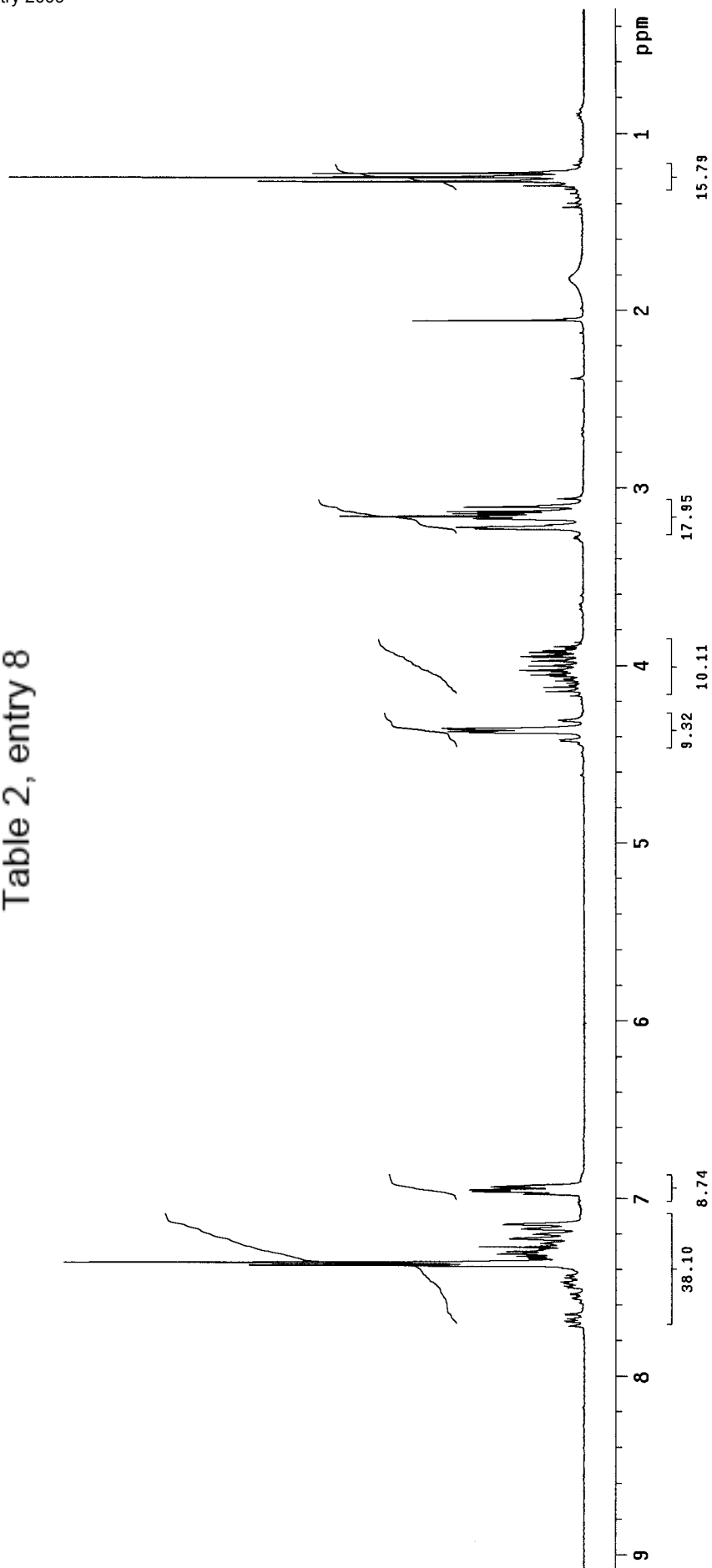


Table 2, entry 8



¹³C-NMR

INDEX	FREQUENCY PPM	HEIGHT
1	11196.423	148.399
2	11192.104	148.342
3	10362.947	137.352
4	9988.387	132.388
5	9978.311	132.254
6	9974.280	132.201
7	9891.652	131.106
8	9881.000	130.965
9	9729.852	128.961
10	9721.215	128.847
11	9709.123	128.686
12	9703.365	128.610
13	9693.864	128.484
14	9690.697	128.442
15	9688.970	128.419
16	9648.951	127.889
17	9286.195	123.081
18	9278.710	122.982
19	9197.234	121.902
20	8751.850	115.999
21	8749.546	115.968
22	5873.697	77.851
23	5841.452	77.424
24	5809.495	77.000
25	4604.914	61.034
26	4588.232	60.947
27	4319.891	57.257
28	4306.072	57.073
29	3699.751	49.037
30	3588.333	47.560
31	2428.089	32.182
32	2340.855	31.026
33	1265.542	16.774
34	1259.784	16.697

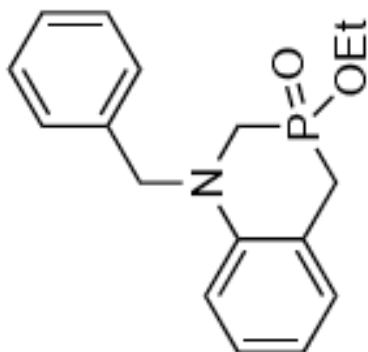
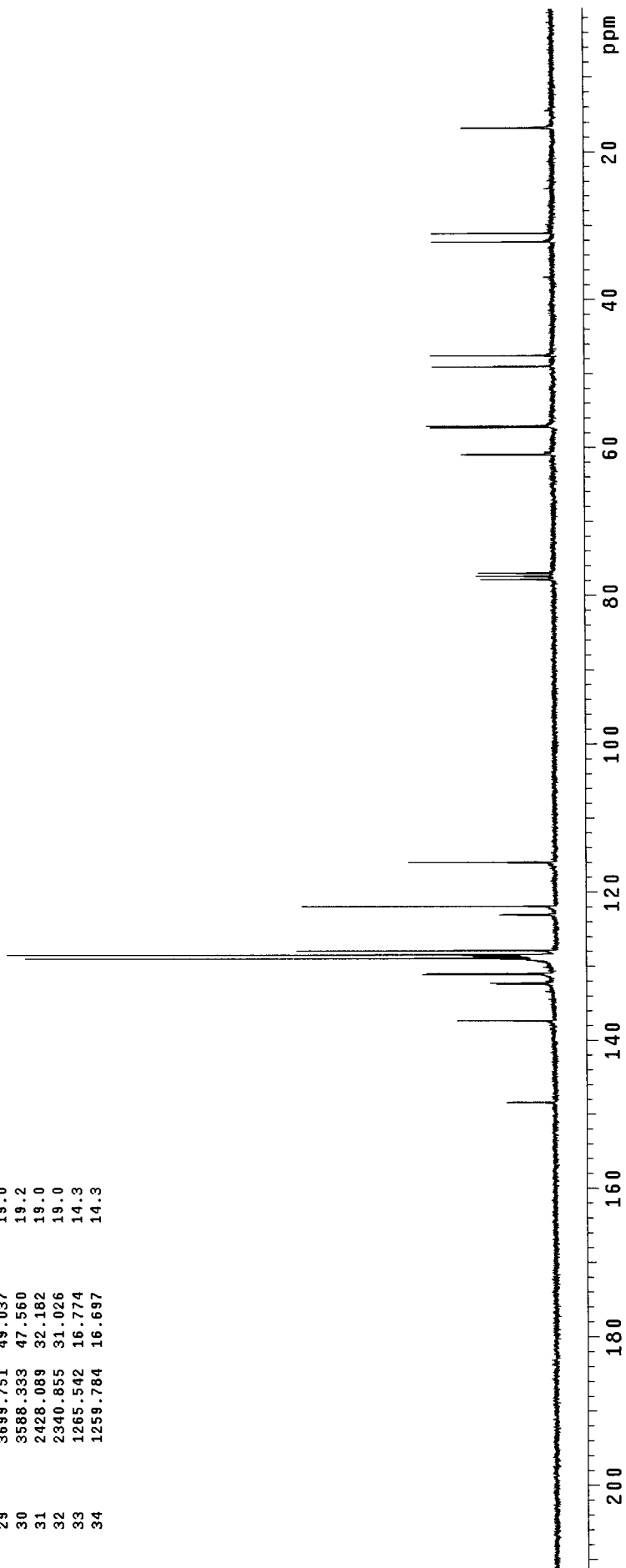


Table 2, entry 8

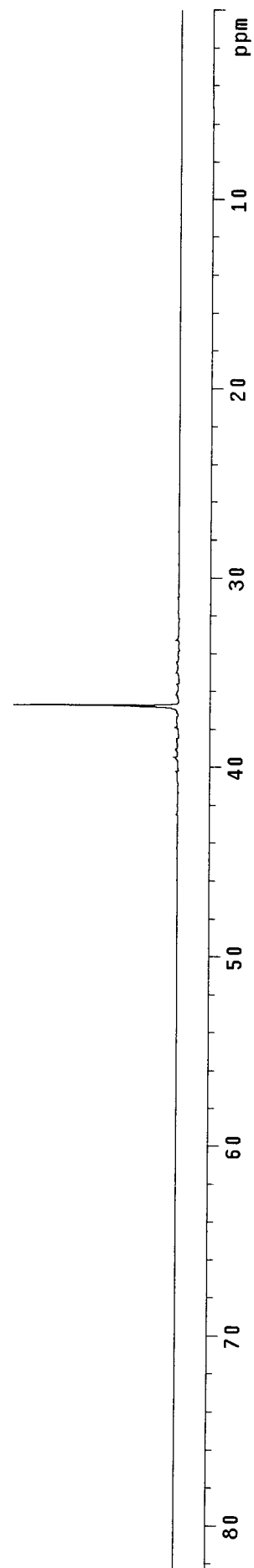


INDEX	FREQUENCY	PPM	HEIGHT
1	4463.389	36.747	25.8

³¹P-NMR (¹H decoupled)



8 (Scheme 5)

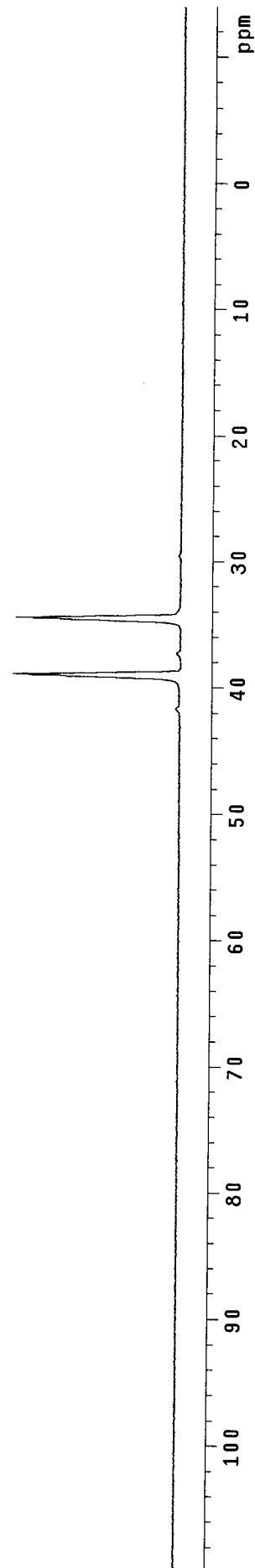


INDEX	FREQUENCY	PPM	HEIGHT
1	4744.901	39.065	23.8
2	4736.742	38.997	26.2
3	4729.398	38.937	20.4
4	4218.596	34.732	12.6
5	4200.644	34.584	23.1
6	4192.892	34.520	25.8
7	4185.549	34.460	20.8

³¹P-NMR (1H coupled)



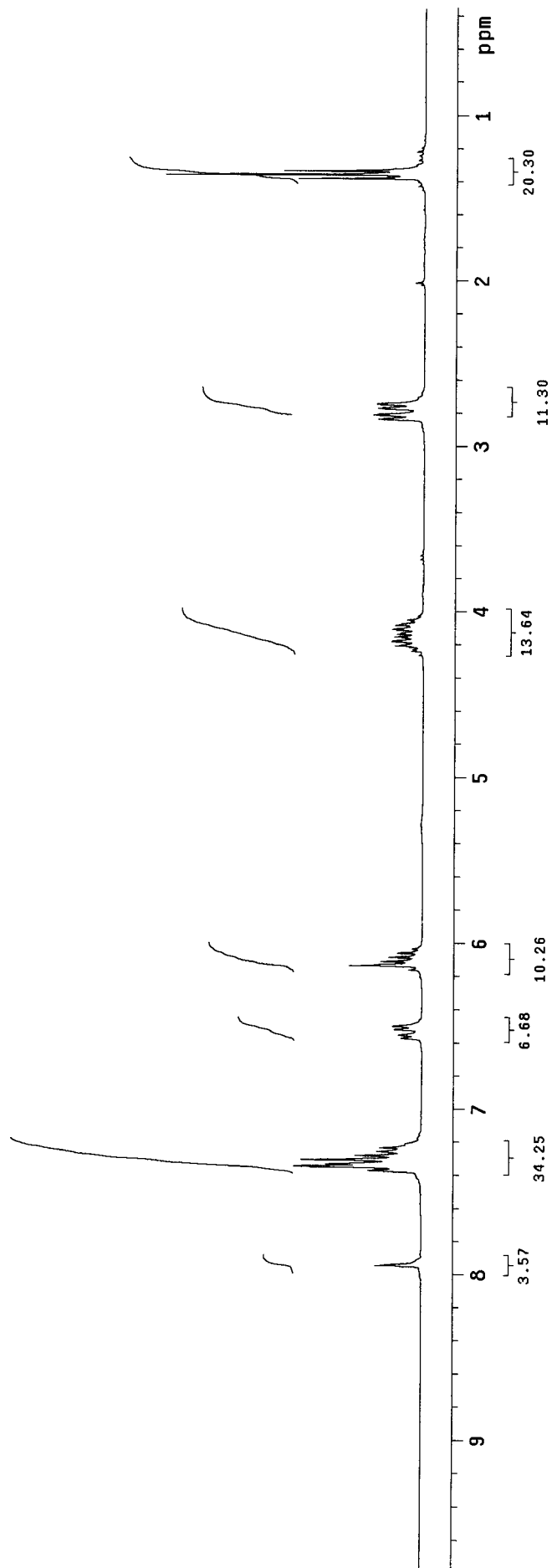
8 (Scheme 5)



¹H-NMR



8 (Scheme 5)

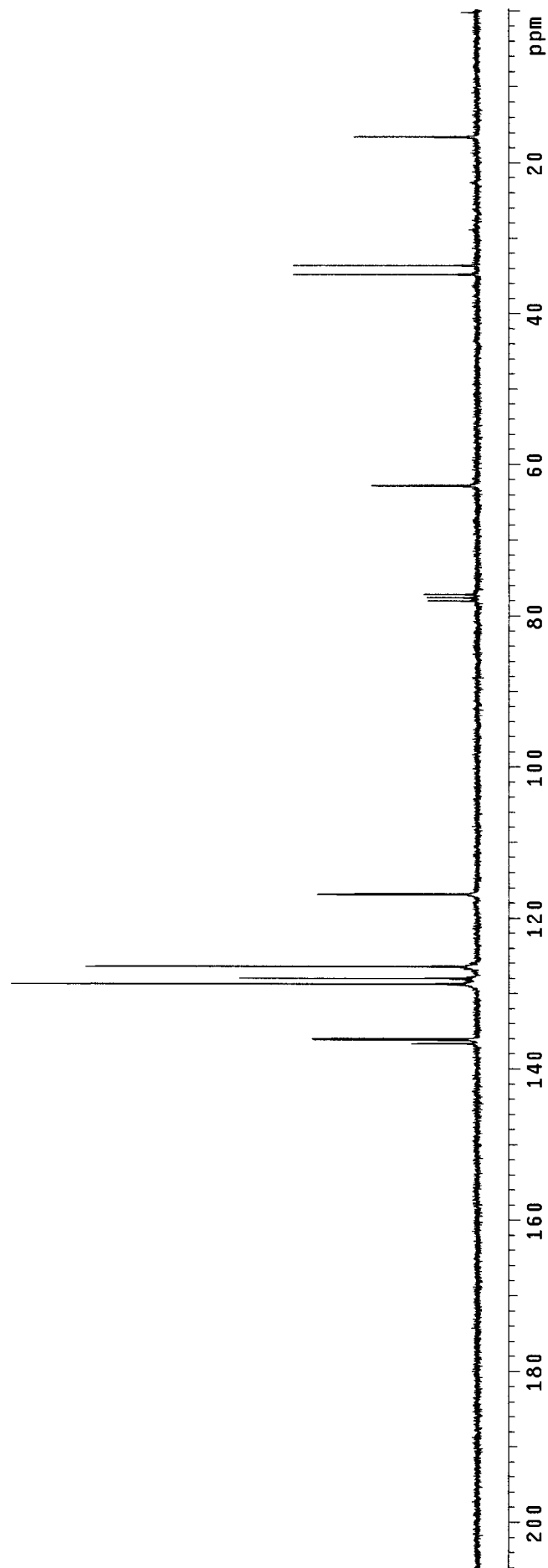


¹³C-NMR

INDEX	FREQUENCY	PPM	HEIGHT
1	10311.125	136.666	10.3
2	10306.807	136.608	9.3
3	10275.713	136.196	25.6
4	10261.318	136.005	25.9
5	9717.760	128.801	72.9
6	9716.896	128.790	71.0
7	9708.547	128.679	6.5
8	9661.043	128.048	37.2
9	9659.892	128.034	35.8
10	9542.140	126.473	61.2
11	9539.837	126.443	59.6
12	8822.673	116.937	25.2
13	8812.597	116.804	24.9
14	5886.076	78.015	7.8
15	5854.119	77.592	7.9
16	5821.874	77.164	8.4
17	4743.107	62.866	16.2
18	4735.909	62.771	16.6
19	2627.605	34.827	28.9
20	2537.204	33.629	28.9
21	1248.844	16.552	19.4
22	1242.798	16.472	19.2

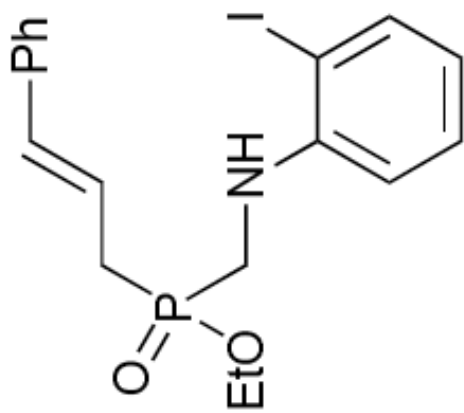


8 (Scheme 5)

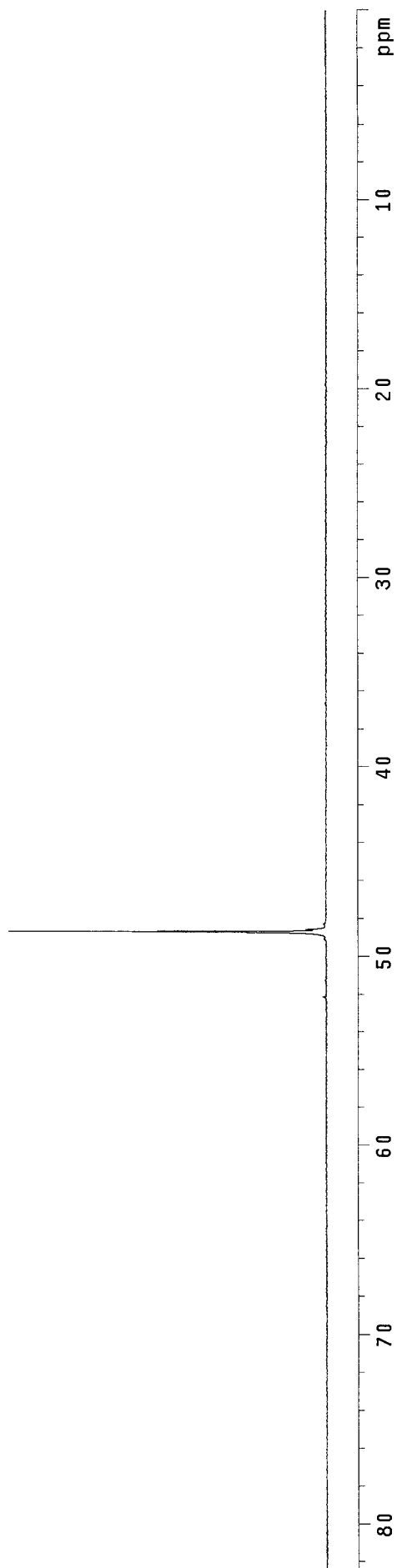


INDEX	FREQUENCY	PPM	HEIGHT
1	5914.198	48.691	49.8

³¹P-NMR (¹H decoupled)

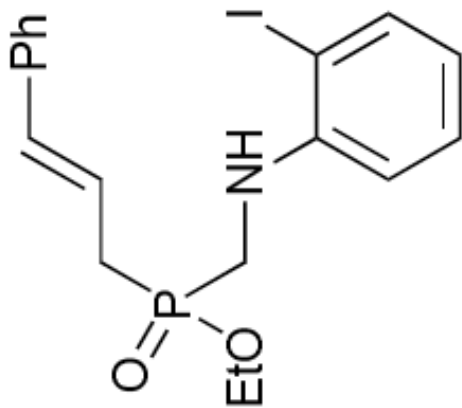


9 (Scheme 5)

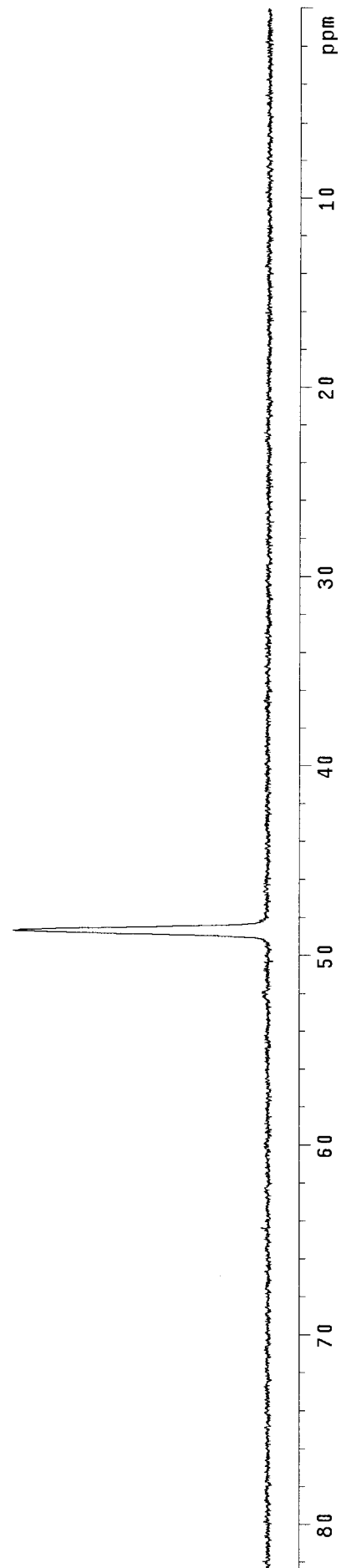


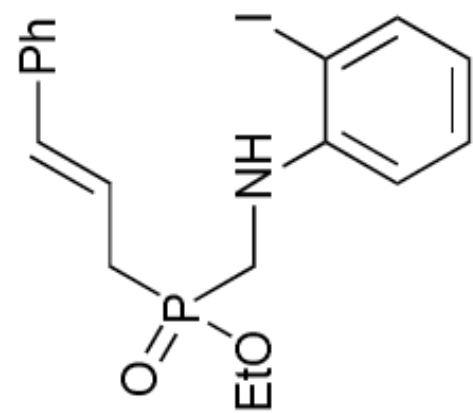
INDEX	FREQUENCY	PPM	HEIGHT
1	5919.094	48.732	39.9
2	5913.382	48.685	39.0

^{31}P -NMR (^1H coupled)



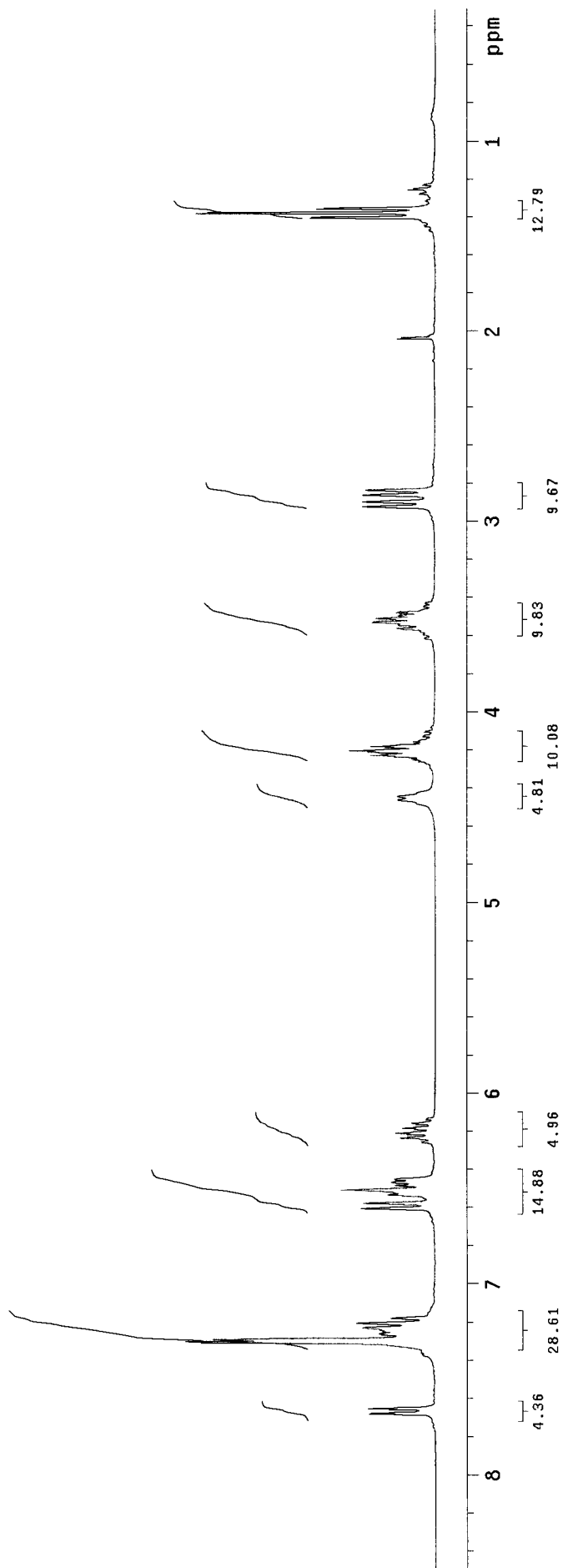
9 (Scheme 5)





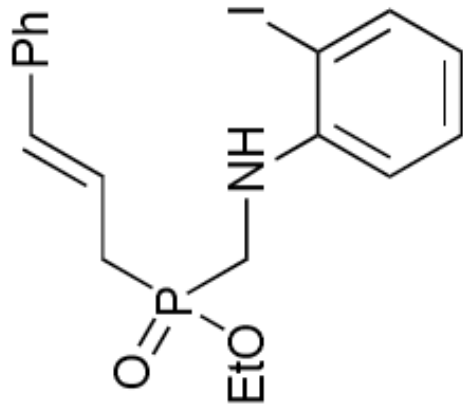
9 (Scheme 5)

¹H-NMR

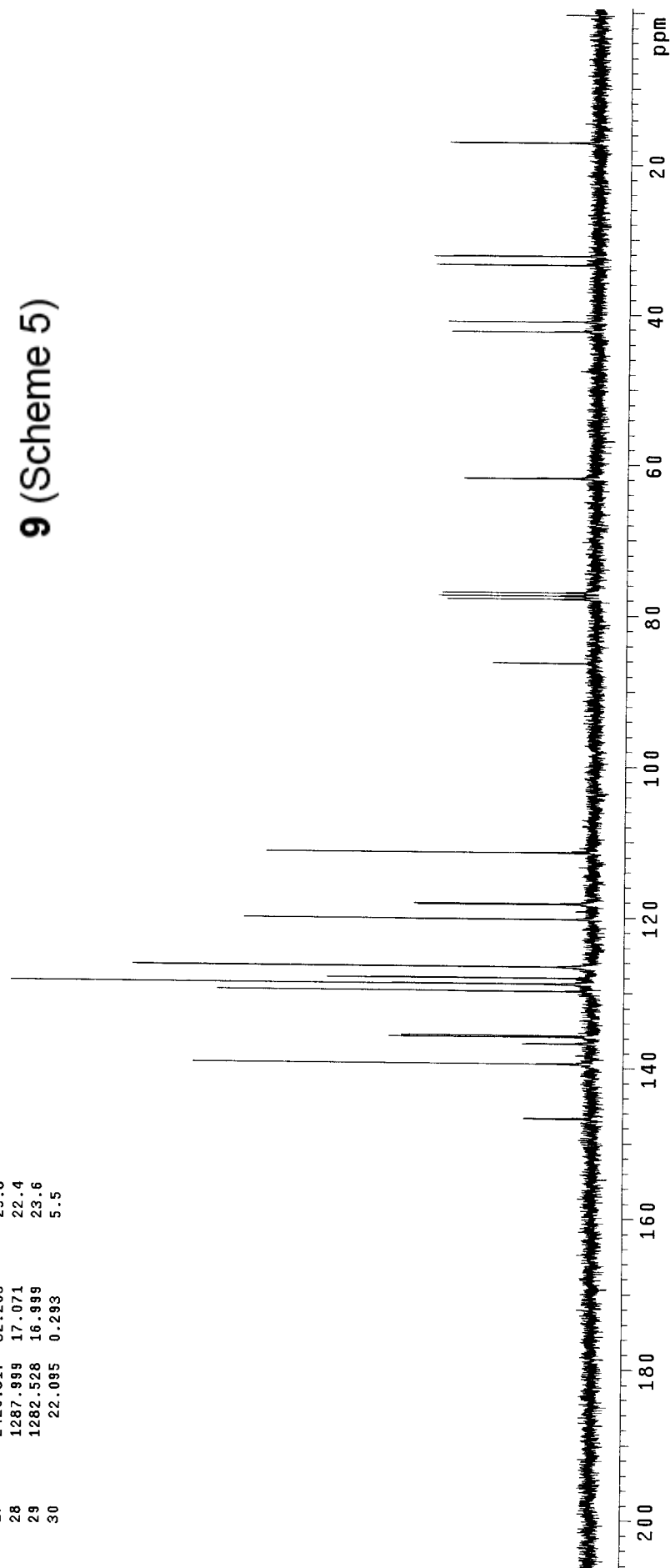


¹³C-NMR

INDEX	FREQUENCY	PPM	HEIGHT
1	11073.777	146.774	10.7
2	11064.276	146.648	10.6
3	10519.566	139.428	62.4
4	10316.883	136.742	10.9
5	10313.716	136.700	10.4
6	10249.226	135.845	31.9
7	10235.983	135.670	30.0
8	9781.175	129.774	58.7
9	9718.911	128.816	91.3
10	9719.487	128.824	91.4
11	9661.043	128.049	41.6
12	9549.337	126.569	71.5
13	9547.322	126.542	72.1
14	9070.845	120.227	54.6
15	8824.879	118.292	27.5
16	8915.378	118.166	28.1
17	8406.080	111.416	51.2
18	6507.656	86.254	16.0
19	5869.954	77.801	23.3
20	5837.997	77.378	24.7
21	5806.040	76.954	24.1
22	4667.676	61.866	20.4
23	4660.767	61.775	20.9
24	3189.013	42.268	23.0
25	3088.536	40.936	23.6
26	2519.354	33.392	25.4
27	2429.817	32.205	25.8
28	1287.999	17.071	22.4
29	1282.528	16.999	23.6
30	22.095	0.293	5.5

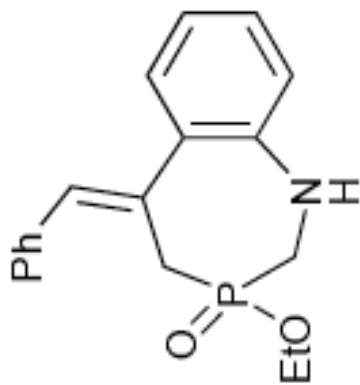


9 (Scheme 5)

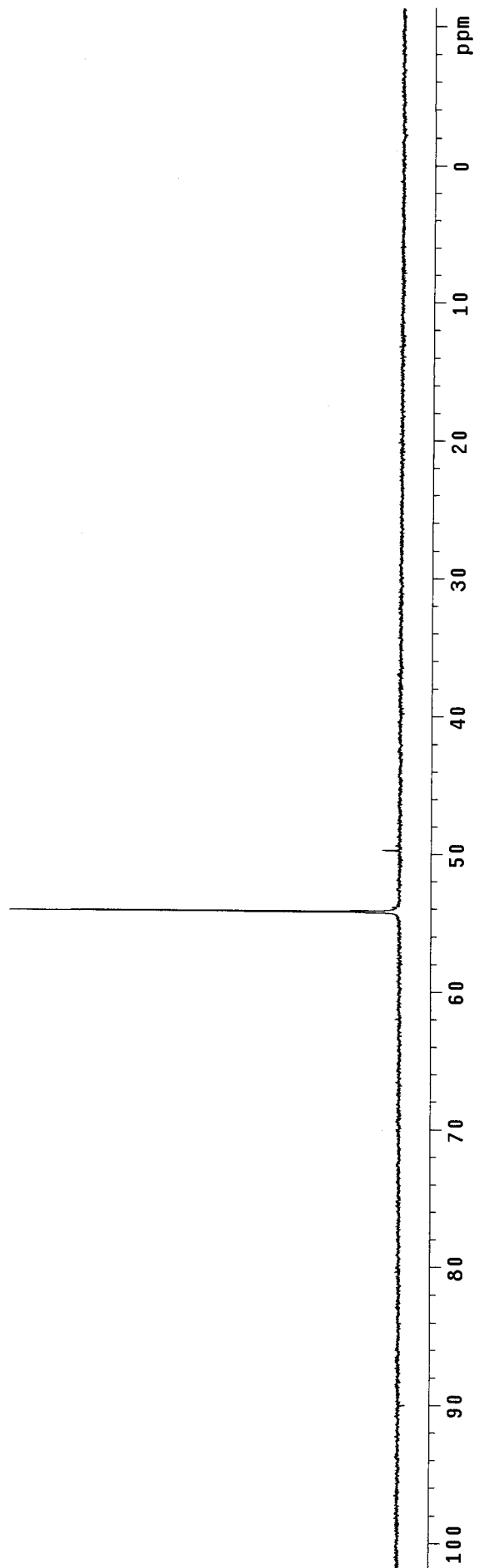


INDEX	FREQUENCY PPM	HEIGHT
1	6580.852	61.2

^{31}P -NMR (^1H decoupled)

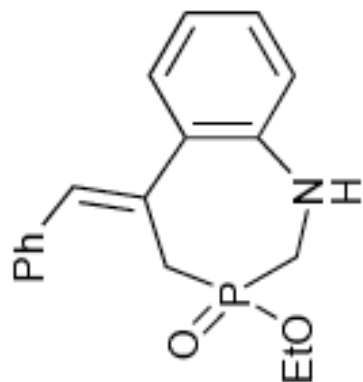


10 (Scheme 5)



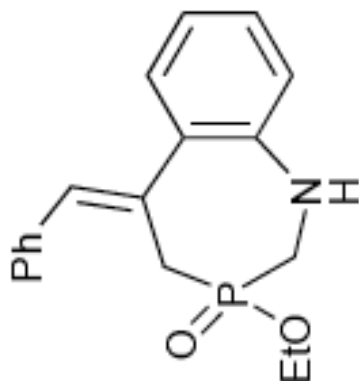
INDEX	FREQUENCY PPM	HEIGHT
1	6580.036	41.9

³¹P-NMR (¹H coupled)



10 (Scheme 5)

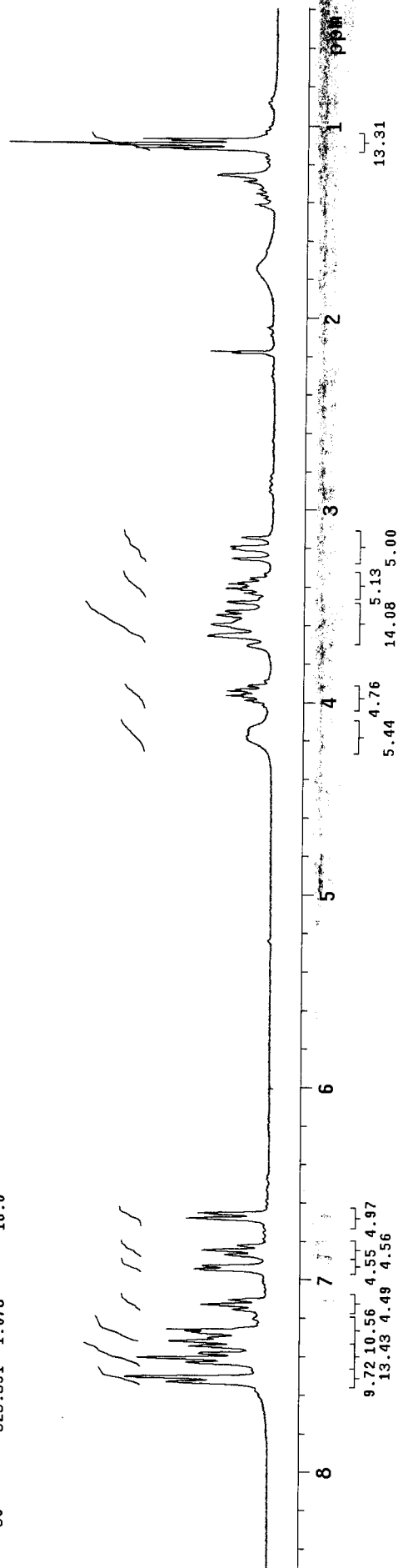




10 (Scheme 5)

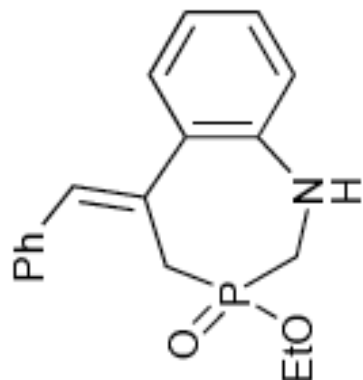
¹H-NMR

INDEX	FREQUENCY PPM	HEIGHT	INDEX	FREQUENCY PPM	HEIGHT
1	2261.115	15.9	40	320.713	1.069
2	2253.493	22.5			21.0
3	2230.627	12.9			
4	2223.005	20.5			
5	2216.555	10.9			
6	2205.122	12.4			
7	2197.500	15.5			
8	2181.930	9.5			
9	2183.722	11.9			
10	2181.670	13.1			
11	2179.031	15.9			
12	2140.042	10.6			
13	2131.833	6.2			
14	2084.635	11.7			
15	2079.651	10.4			
16	2062.062	6.9			
17	2054.733	10.5			
18	2005.190	12.9			
19	1997.274	11.1			
20	1189.922	7.3			
21	1182.886	7.1			
22	1096.405	10.3			
23	1078.815	9.7			
24	1064.451	8.9			
25	1058.294	7.6			
26	1044.223	7.2			
27	1022.823	7.4			
28	1015.787	7.1			
29	976.211	6.5			
30	961.553	6.6			
31	957.742	6.8			
32	654.911	6.7			
33	652.273	10.1			
34	377.292	9.3			
35	337.130	14.7			
36	334.785	22.1			
37	330.387	29.3			
38	327.749	41.8			
39	323.351	16.6			

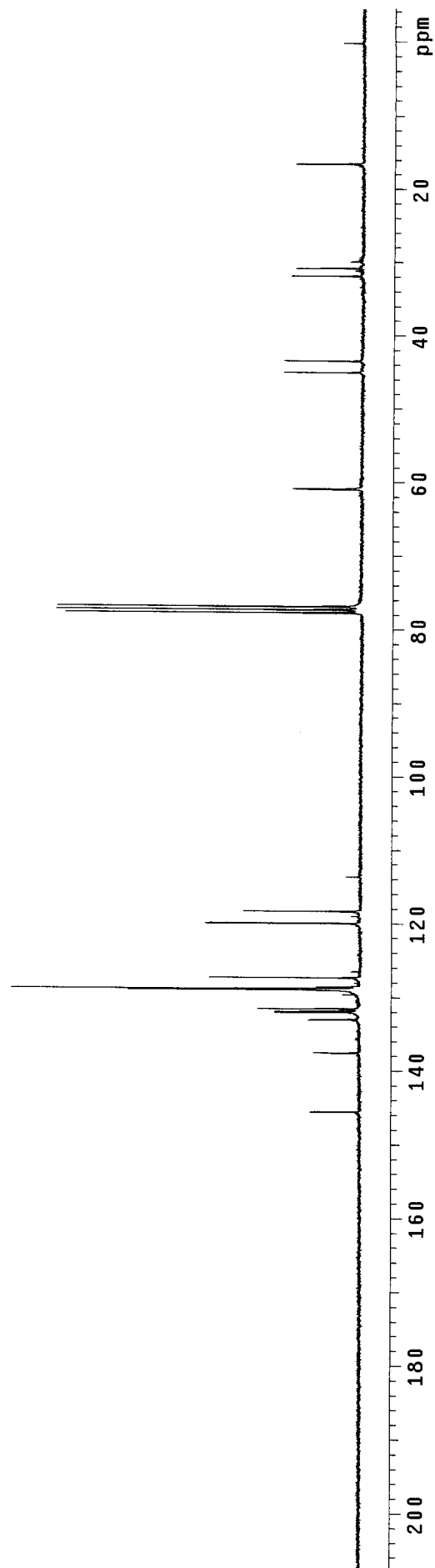


¹³C-NMR

INDEX	FREQUENCY	PPM	HEIGHT
1	10983.376	145.576	7.8
2	10981.072	145.545	7.2
3	10378.782	137.562	7.2
4	10375.615	137.520	7.1
5	10040.786	133.082	8.1
6	10033.300	132.983	7.8
7	9962.476	132.044	13.4
8	9951.248	131.896	13.5
9	9926.201	131.564	16.2
10	9924.185	131.537	14.9
11	9731.003	128.976	36.5
12	9728.988	128.950	36.0
13	9724.382	128.889	29.6
14	9718.336	128.809	54.7
15	9701.349	128.583	6.6
16	9698.758	128.549	6.8
17	9605.766	127.317	23.6
18	9050.116	119.952	24.4
19	8928.046	118.334	18.5
20	8926.318	118.311	17.3
21	5862.756	77.706	46.4
22	5630.799	77.282	47.9
23	5788.842	76.859	47.8
24	4599.156	60.958	10.4
25	4592.246	60.866	10.9
26	3399.470	45.957	12.4
27	3280.854	43.485	12.4
28	2405.921	31.889	11.3
29	2327.899	30.854	10.6
30	1255.754	16.644	10.7
31	1249.708	16.564	10.5

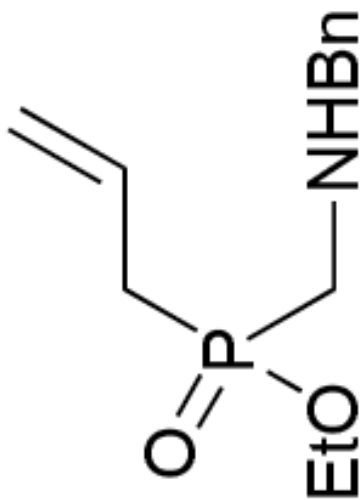


10 (Scheme 5)

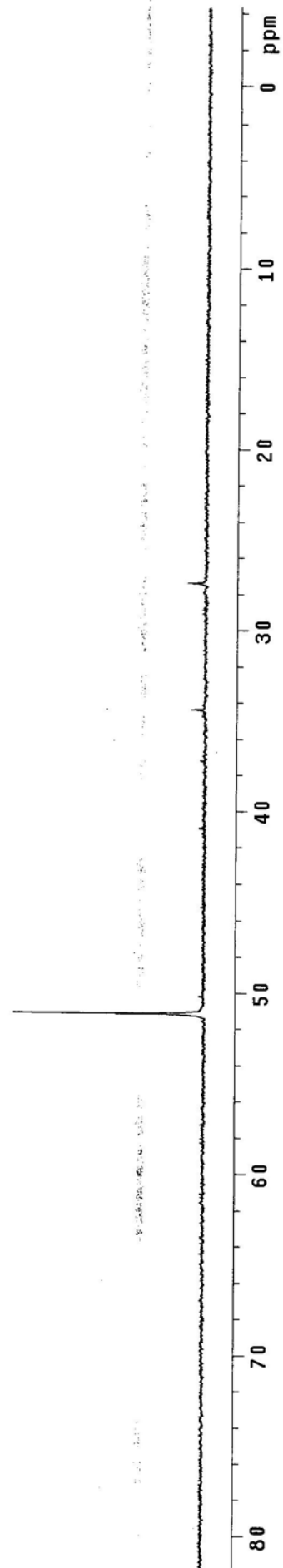


INDEX	FREQUENCY PPM	HEIGHT
1	6208.766	51.117
		30.1

³¹P-NMR (¹H decoupled)

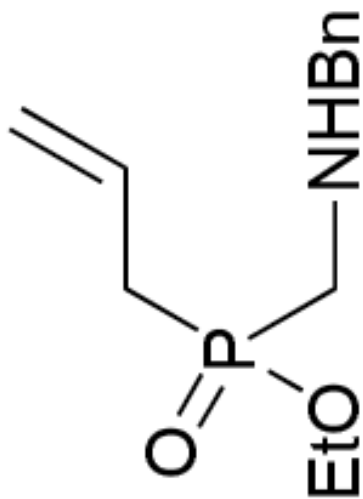


12a (Scheme 6)

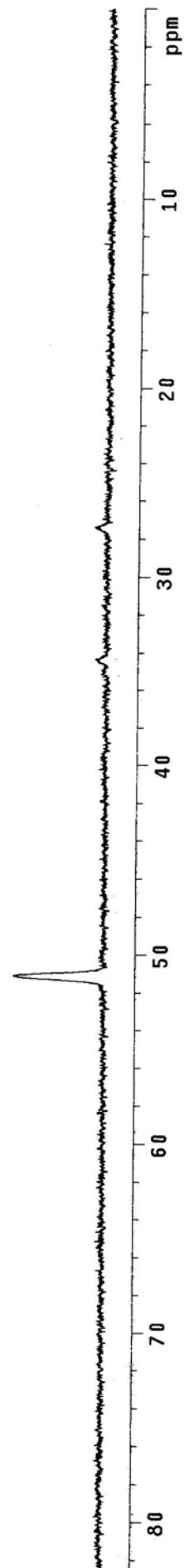


INDEX	FREQUENCY PPM	HEIGHT
1	6214.478	51.164
		14.3

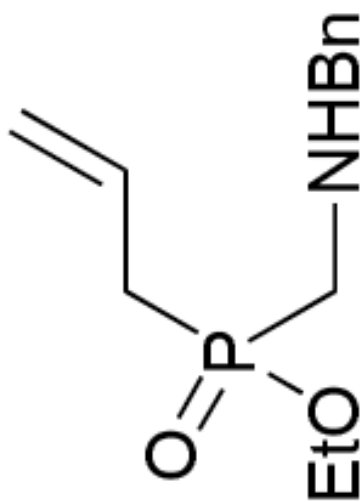
³¹P-NMR (¹H coupled)



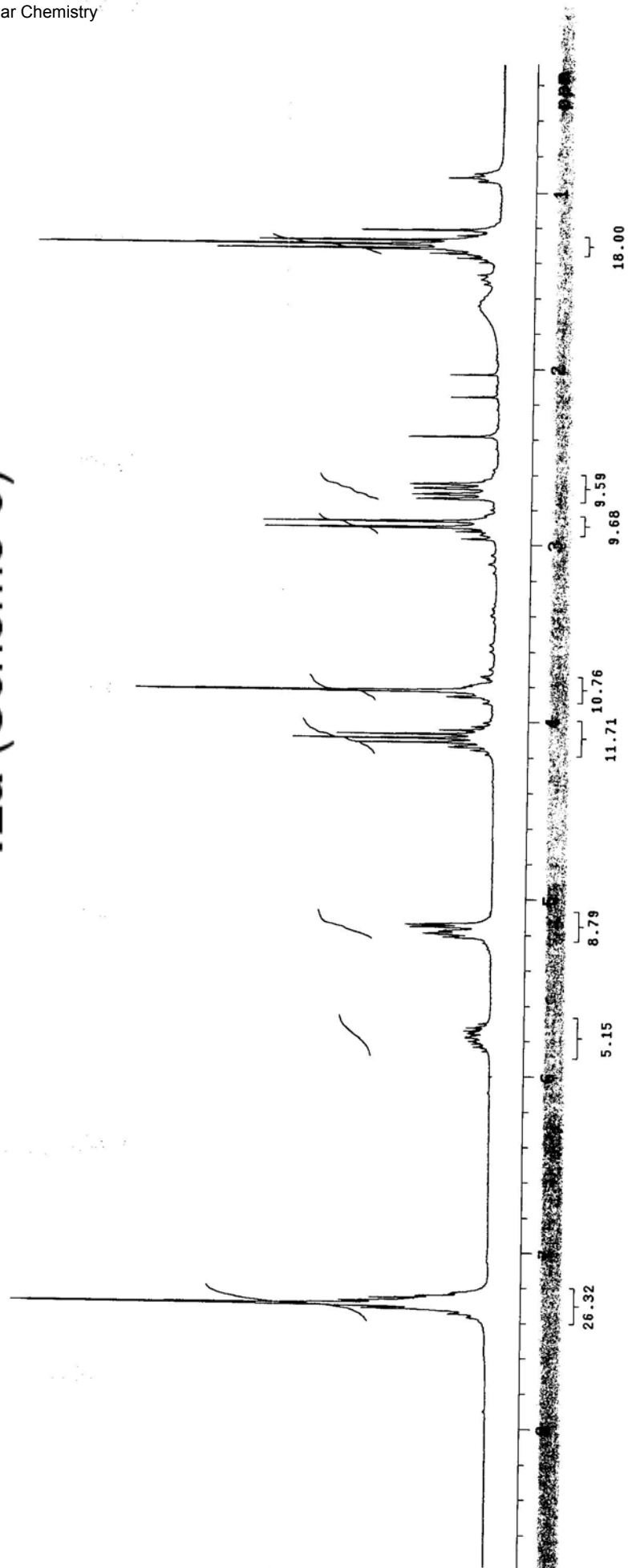
12a (Scheme 6)



$^1\text{H-NMR}$

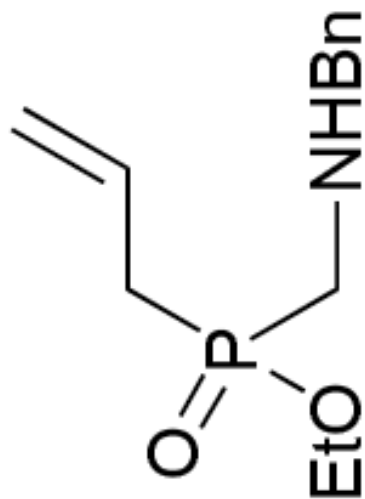


12a (Scheme 6)

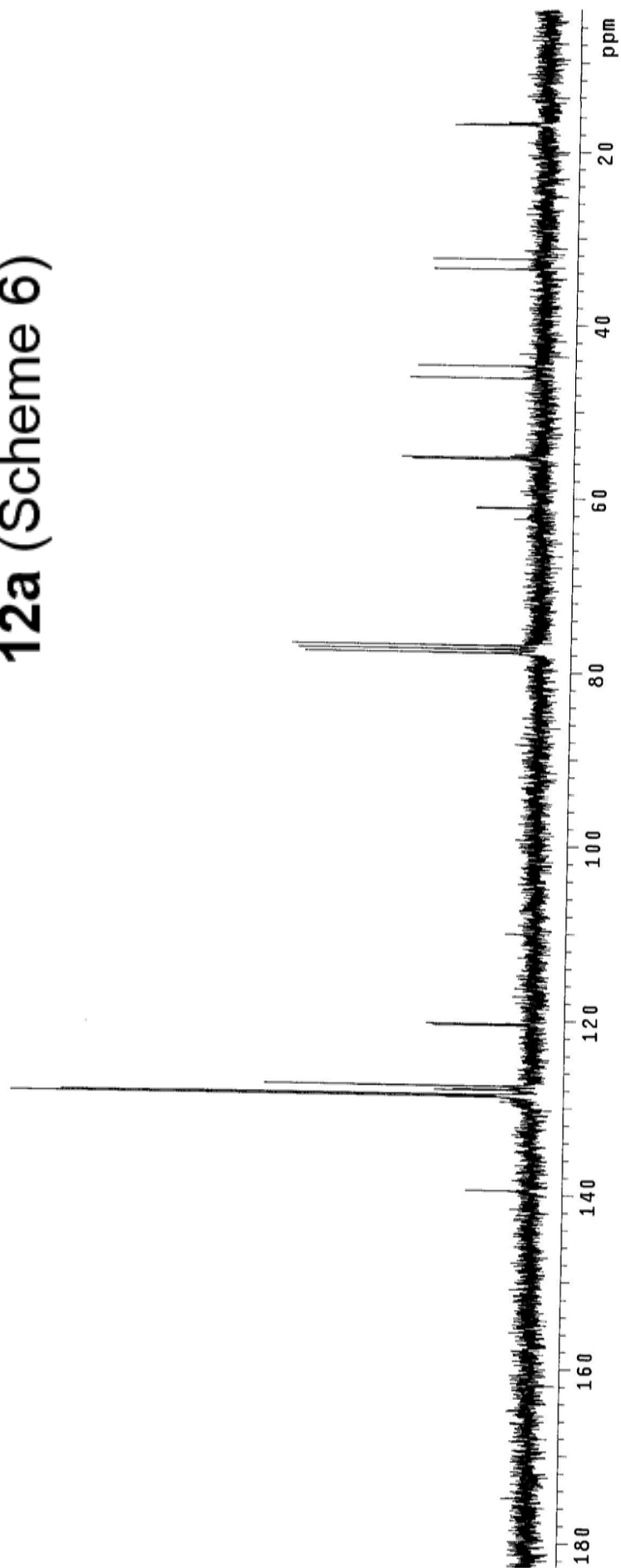


¹³C-NMR

INDEX	FREQUENCY	PPM	HEIGHT
1	10524.172	139.489	10.3
2	9708.547	128.679	82.1
3	9693.288	128.477	74.1
4	9653.270	127.946	15.5
5	9644.057	127.824	12.6
6	9619.010	127.492	42.2
7	9089.846	120.478	15.8
8	9077.466	120.314	17.0
9	5863.332	77.714	36.9
10	5831.375	77.290	38.0
11	5799.130	76.863	38.9
12	4611.248	61.118	10.4
13	4604.626	61.031	10.3
14	4185.441	55.475	20.7
15	4169.031	55.257	22.3
16	3480.658	46.133	21.3
17	3374.710	44.729	20.0
18	2538.068	33.640	17.8
19	2450.258	32.476	17.9
20	1278.498	16.945	14.8
21	1272.740	16.869	13.4
22	1258.921	16.686	6.4

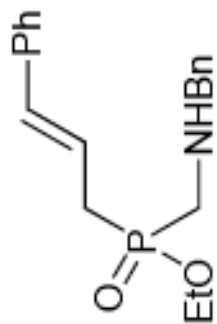


12a (Scheme 6)

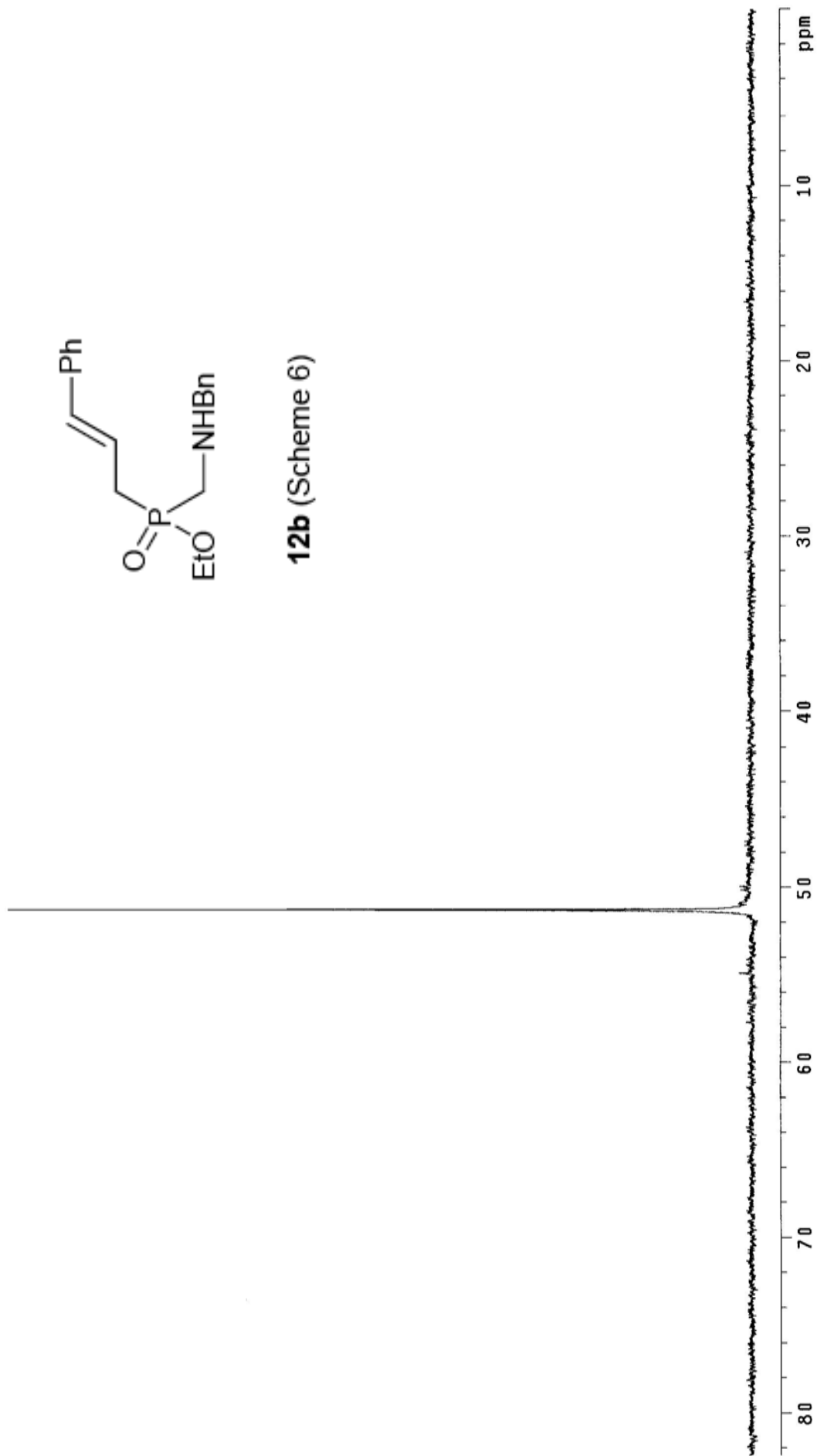


INDEX	FREQUENCY PPM	HEIGHT
1	6229.574	51.288
		126.0

³¹P-NMR (¹H decoupled)

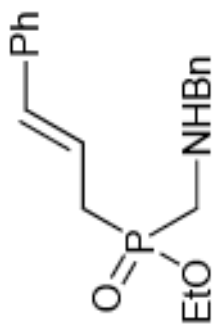


12b (Scheme 6)

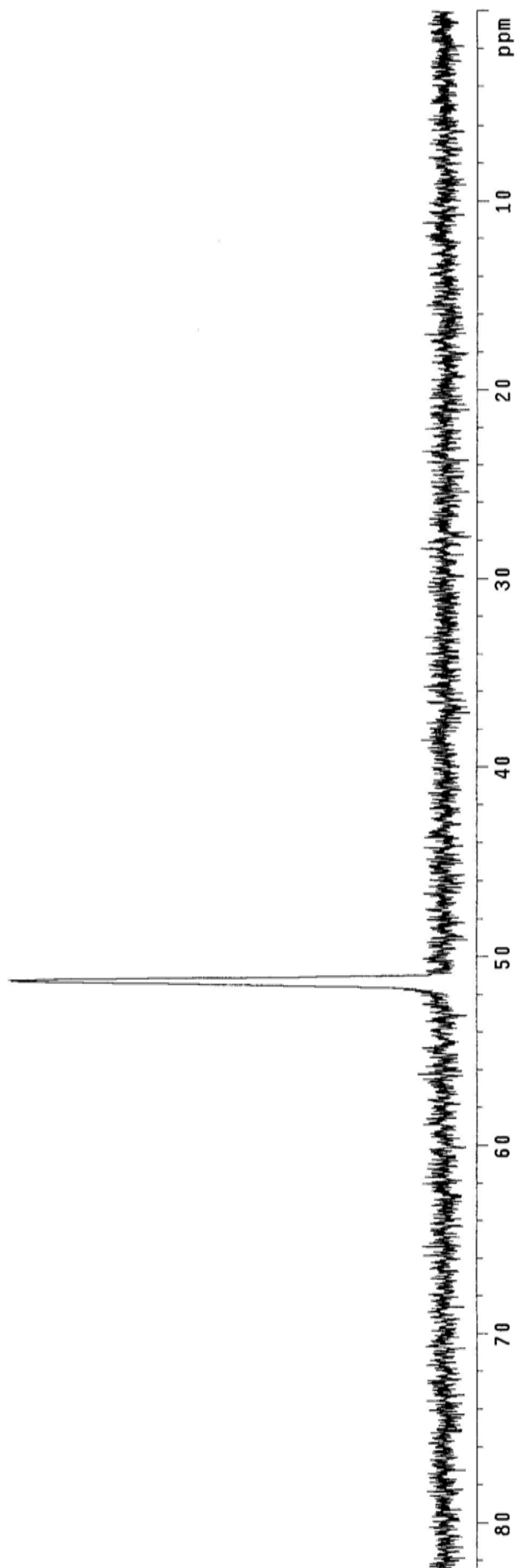


INDEX	FREQUENCY PPM	HEIGHT
1	6231.205	51.301
		68.6

³¹P-NMR (¹H coupled)

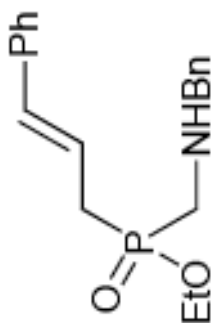


12b (Scheme 6)

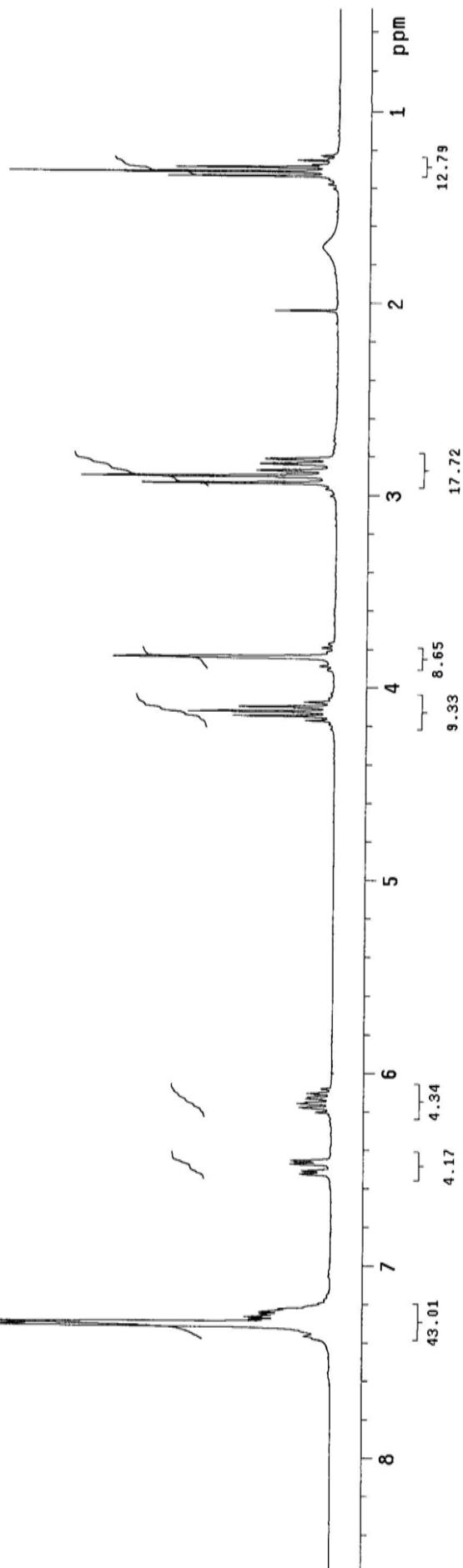


¹H-NMR

INDEX	FREQUENCY PPM	HEIGHT
1	2193.689	66.5
2	2190.464	104.5
3	2184.015	12.9
4	2180.790	13.5
5	2179.031	10.7
6	2177.566	10.1
7	2176.100	11.1
8	2172.875	11.2
9	2168.185	8.3
10	1243.862	16.0
11	1236.827	22.9
12	1229.205	15.0
13	1152.105	34.7
14	880.642	30.5
15	869.795	39.8
16	861.587	12.6
17	851.326	12.0
18	843.704	11.3
19	611.817	9.9
20	400.745	26.7
21	393.709	51.6
22	386.673	25.6

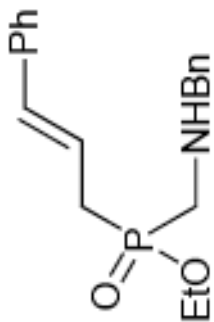


12b (Scheme 6)



¹³C-NMR

INDEX	FREQUENCY	PPM	HEIGHT
1	10525.900	139.512	21.4
2	10340.203	137.051	12.1
3	10336.748	137.005	11.3
4	10192.222	135.090	29.6
5	10179.266	134.918	29.3
6	9716.896	128.790	98.6
7	9710.850	128.709	125.4
8	9696.455	128.519	117.5
9	9645.209	127.839	43.3
10	9620.161	127.507	64.5
11	9541.276	126.462	73.1
12	9539.261	126.435	69.2
13	8998.869	119.273	28.3
14	8989.081	119.143	29.1
15	5865.347	77.740	46.1
16	5833.102	77.313	47.0
17	5801.145	76.889	47.2
18	4618.157	61.210	19.8
19	4611.248	61.118	19.3
20	4188.032	55.509	30.9
21	4171.334	55.288	30.9
22	3493.325	46.301	26.5
23	3387.090	44.893	26.2
24	2487.109	32.965	27.1
25	2399.011	31.797	27.0
26	1283.968	17.018	20.5
27	1278.498	16.945	24.2



12b (Scheme 6)

