

Supporting Information

Oxidative cross-coupling of secondary phosphine chalcogenides with amino alcohols and aminophenols: aspects of the reaction chemoselectivity

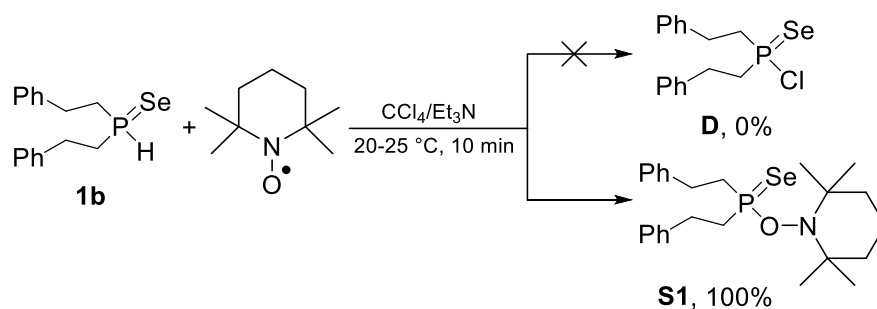
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1. The radical trapping reaction with TEMPO



Scheme S1 The radical trapping reaction of **1b** with TEMPO.

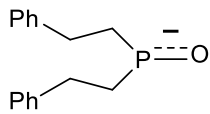
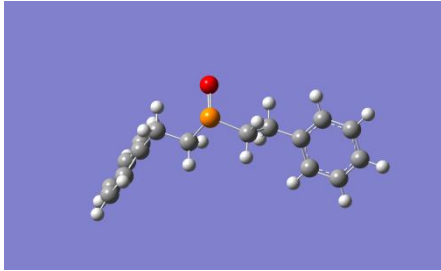
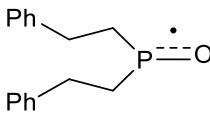
Typical procedure and analytical data.

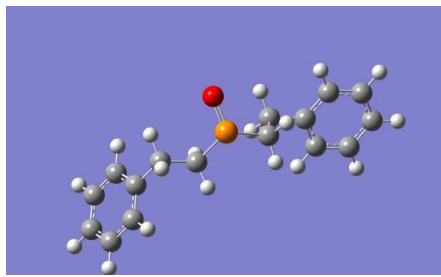
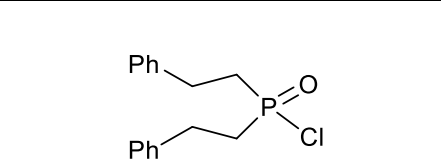
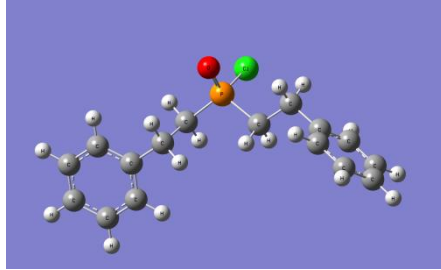
To a solution of secondary phosphine selenide **1b** (321 mg, 1.0 mmol) in CCl₄ (3 mL), Et₃N (101 mg, 1.0 mmol) and TEMPO (156 mg, 1.0 mmol) was added. The mixture was stirred at 20–25 °C for 10 min. After completion of the reaction, the solvent was immediately removed under reduced pressure. The residue was dissolved in CDCl₃ and analyzed by NMR spectroscopy.

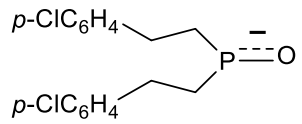

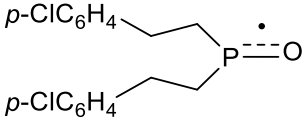
1-[[bis(2-phenylethyl)phosphoroselenoyl]oxy]-2,2,6,6-tetramethylpiperidine (S1). ¹³C{¹H} NMR (100.62 MHz, CDCl₃): δ 15.6 (CH₂CH₂C(Me)₂); 31.0 (d, PhCH₂, ²J_{PC} = 3.6 Hz); 37.9 (d, CH₂P, ¹J_{PC} = 54.2 Hz); 41.4 (CH₂CH₂C(Me)₂); 48.0 (CH₂CH₂C(Me)₂); 62.5 (d, CH₂CH₂C(Me)₂, ³J_{PC} = 2.2 Hz); 127.7 (C_p); 129.3 (C_o); 129.9 (C_m); 141.4 (d, C_i, ³J_{PC} = 15.6 Hz). ¹⁵N NMR (40.56 MHz, CDCl₃): δ -286.3. ³¹P NMR (161.98 MHz, CDCl₃): δ 113.0 (+ d-satellites, ¹J_{PSe} = 787.1 Hz). ⁷⁷Se NMR (76.31 MHz, CDCl₃): δ -217.1 (d, ¹J_{PSe} = 789.8 Hz).

2. Quantum chemical calculations

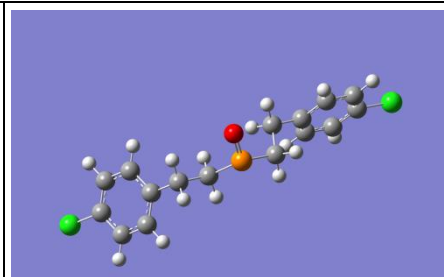
Table S1 Calculated geometry of chalcogenophosphoryl chlorides (**D**) and deprotonated phosphine chalcogenides in anionic (**A**) and radical (**B**) states. B3LYP/6-311++G(d,p)

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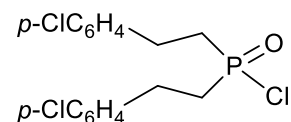
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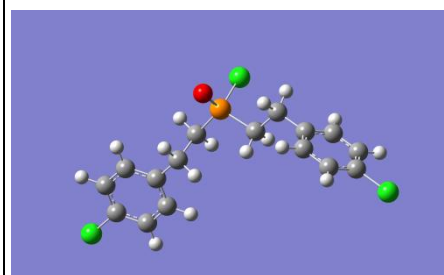
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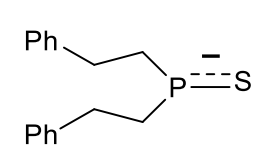
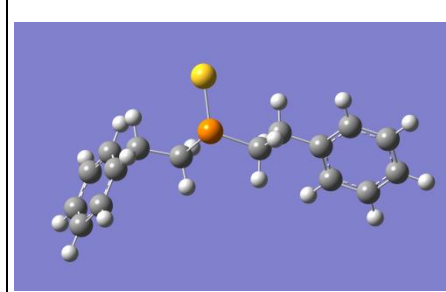
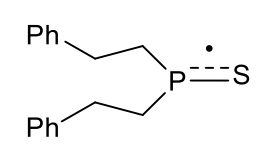


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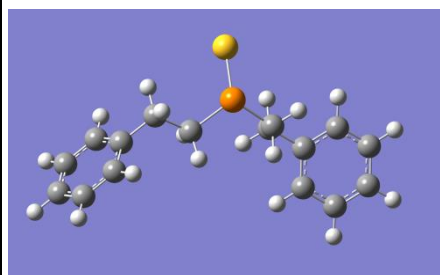
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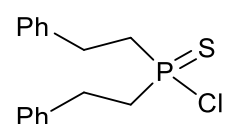
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1	7.127074000	0.524332000	1.226046000	
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1	1.756048000	0.248430000	-0.983272000	
6	2.446383000	1.771125000	0.428990000	
1	1.945186000	2.563616000	-0.135276000	
1	2.267050000	1.967453000	1.492769000	
6	3.924298000	1.809792000	0.133208000	
6	4.425812000	2.372805000	-1.048335000	
6	4.847587000	1.253741000	1.032760000	
6	5.792495000	2.379891000	-1.326532000	
1	3.730900000	2.813156000	-1.757502000	
6	6.213377000	1.258134000	0.760551000	
1	4.479257000	0.817976000	1.956398000	
6	6.695348000	1.820439000	-0.423080000	
1	6.152603000	2.824572000	-2.249336000	
1	6.905697000	0.824828000	1.475843000	
1	7.759432000	1.825252000	-0.635543000	
16	0.171649000	0.642331000	2.806446000	
15	0.062730000	0.080327000	0.799383000	
6	-3.901100000	5.071592000	-0.194248000	
6	-3.251285000	3.991126000	0.399170000	
6	-3.101225000	2.779110000	-0.284501000	
6	-3.624597000	2.676069000	-1.577945000	
6	-4.276180000	3.753819000	-2.175346000	
6	-4.414886000	4.956758000	-1.485189000	
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6	-3.901100000	5.071592000	-0.194248000	
6	-3.251285000	3.991126000	0.399170000	
6	-3.101225000	2.779110000	-0.284501000	
6	-3.624597000	2.676069000	-1.577945000	
6	-4.276180000	3.753819000	-2.175346000	
6	-4.414886000	4.956758000	-1.485189000	

1	-4.012260000	6.000678000	0.353966000
1	-2.862835000	4.086378000	1.408518000
1	-3.528824000	1.739997000	-2.119903000
1	-4.680179000	3.651821000	-3.176594000
1	-4.924383000	5.795218000	-1.946417000
6	-2.365282000	1.618767000	0.350794000
1	-2.536655000	1.614643000	1.430445000
1	-2.760120000	0.674039000	-0.034845000
6	-0.850235000	1.683666000	0.085914000
1	-0.441210000	2.628275000	0.456915000
1	-0.645808000	1.633083000	-0.988792000
6	1.843711000	0.456967000	0.280807000
1	2.375727000	-0.415287000	0.668707000
1	1.824808000	0.362861000	-0.810151000
6	2.569049000	1.754301000	0.705478000
1	2.039615000	2.622157000	0.302628000
1	2.534201000	1.833013000	1.795213000
6	4.005313000	1.774743000	0.230483000
6	4.345776000	2.329968000	-1.008057000
6	5.022402000	1.211747000	1.010202000
6	5.664811000	2.321849000	-1.458104000
1	3.572445000	2.780369000	-1.623171000
6	6.342366000	1.201855000	0.564292000
1	4.779068000	0.784310000	1.978084000
6	6.667895000	1.756187000	-0.672831000
1	5.909439000	2.761704000	-2.418686000
1	7.117349000	0.766264000	1.185284000
1	7.695098000	1.751877000	-1.019438000
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15	0.090872000	0.279689000	0.871493000

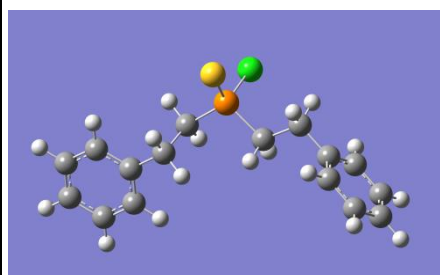
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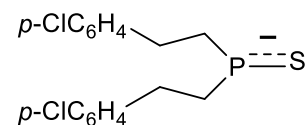
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6	-3.116312000	2.791181000	-0.351505000
6	-3.647573000	2.722198000	-1.643911000
6	-4.305119000	3.815177000	-2.205837000
6	-4.441371000	4.997862000	-1.480929000
1	-4.029477000	5.990666000	0.384375000
1	-2.869449000	4.049648000	1.377186000
1	-3.553512000	1.801771000	-2.212122000
1	-4.715657000	3.740749000	-3.206776000
1	-4.955608000	5.847782000	-1.915091000
6	-2.377519000	1.612403000	0.246043000
1	-2.528016000	1.582106000	1.327811000
1	-2.778527000	0.680058000	-0.159176000
6	-0.867894000	1.683914000	-0.046929000
1	-0.444117000	2.602206000	0.371856000
1	-0.676695000	1.683975000	-1.123854000
6	1.842530000	0.511740000	0.090841000
1	2.341336000	-0.437320000	0.302131000
1	1.807661000	0.629072000	-0.996124000
6	2.597765000	1.678887000	0.759140000
1	2.092469000	2.624190000	0.541057000
1	2.570803000	1.544507000	1.843297000



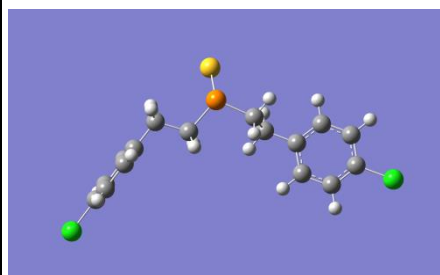
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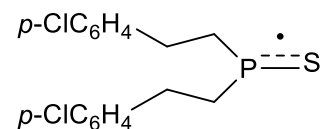
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6	4.387993000	2.573679000	-0.796853000	
6	5.028624000	0.986061000	0.891953000	
6	5.703377000	2.619830000	-1.256010000	
1	3.630989000	3.186158000	-1.277411000	
6	6.344385000	1.029174000	0.435933000	
1	4.773111000	0.353137000	1.736219000	
6	6.685671000	1.845803000	-0.641366000	
1	5.960952000	3.263695000	-2.089648000	
1	7.103732000	0.429425000	0.925283000	
1	7.709711000	1.882341000	-0.995176000	
16	-0.110375000	0.015656000	2.596131000	
15	0.112173000	0.308384000	0.684939000	
17	-0.498771000	-1.315067000	-0.540142000	
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6	-2.961119000	4.001362000	0.347925000	
6	-3.070295000	2.724395000	-0.220729000	
6	-3.883862000	2.586573000	-1.351393000	
6	-4.564663000	3.671770000	-1.903593000	
6	-4.428881000	4.920491000	-1.311428000	
1	-3.540874000	6.078510000	0.267562000	
1	-2.338788000	4.134613000	1.226814000	
1	-3.990163000	1.609174000	-1.811551000	
1	-5.191970000	3.547740000	-2.777870000	
17	-5.290279000	6.309654000	-1.998915000	
6	-2.316286000	1.552861000	0.360610000	
1	-2.338740000	1.601908000	1.454571000	
1	-2.810662000	0.618630000	0.072553000	
6	-0.840627000	1.502307000	-0.068950000	
1	-0.357860000	2.450994000	0.188489000	
1	-0.765906000	1.358279000	-1.155551000	
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1	2.419031000	-0.393856000	0.474588000	
1	1.737950000	0.217061000	-1.028548000	
6	2.431500000	1.769428000	0.353374000	
1	1.949311000	2.543446000	-0.251154000	
1	2.218680000	1.997672000	1.405540000	
6	3.917514000	1.801241000	0.108967000	
6	4.468120000	2.369090000	-1.046835000	
6	4.804430000	1.236565000	1.038833000	
6	5.843576000	2.377518000	-1.278334000	
1	3.808401000	2.818536000	-1.782587000	
6	6.180656000	1.234254000	0.828227000	
1	4.403633000	0.796658000	1.946449000	
6	6.687105000	1.805905000	-0.334531000	
1	6.252639000	2.823466000	-2.176745000	
1	6.852065000	0.798791000	1.558234000	
17	8.438287000	1.811109000	-0.613802000	
16	0.214600000	0.706136000	2.769513000	
15	0.070625000	0.073161000	0.787395000	
6	-3.954056000	5.035393000	-0.158443000	
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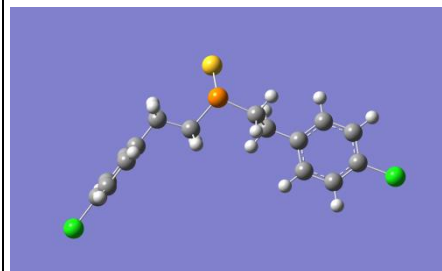
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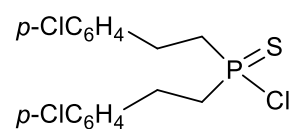
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1	-4.097904000	5.955863000	0.393025000
1	-2.938423000	4.040211000	1.442271000
1	-3.490294000	1.745871000	-2.141017000
1	-4.649368000	3.661506000	-3.188955000
17	-5.258658000	6.281488000	-2.205300000
6	-2.374394000	1.594733000	0.356435000
1	-2.544371000	1.589880000	1.436167000
1	-2.767372000	0.649066000	-0.028617000
6	-0.859844000	1.663846000	0.088658000
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1	-0.655363000	1.596416000	-0.985055000
6	1.838068000	0.450271000	0.300845000
1	2.369496000	-0.424243000	0.684423000
1	1.814007000	0.358349000	-0.790177000
6	2.567860000	1.745011000	0.726541000
1	2.039196000	2.615596000	0.328958000
1	2.536782000	1.820517000	1.816601000
6	4.002461000	1.765368000	0.248270000
6	4.348419000	2.348831000	-0.974772000
6	5.017931000	1.174687000	1.008582000
6	5.663700000	2.345283000	-1.433781000
1	3.582544000	2.823011000	-1.580501000
6	6.338275000	1.160907000	0.566755000
1	4.778605000	0.722964000	1.965973000
6	6.649317000	1.747633000	-0.655771000
1	5.921440000	2.804775000	-2.379496000
1	7.117376000	0.706347000	1.165355000
17	8.314581000	1.739843000	-1.222182000
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15	0.087164000	0.276907000	0.899537000



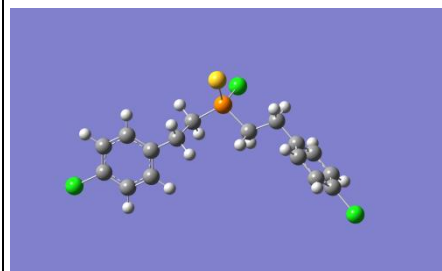
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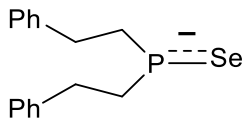
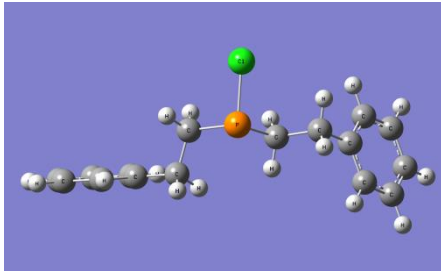


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6	-3.127515000	2.788574000	-0.235949000
6	-3.659040000	2.618089000	-1.518291000
6	-4.325437000	3.653543000	-2.169469000
6	-4.462347000	4.879255000	-1.526490000
1	-4.072792000	6.035185000	0.242966000
1	-2.894827000	4.185560000	1.386311000
1	-3.561381000	1.659418000	-2.017761000
1	-4.738374000	3.509836000	-3.159820000
17	-5.303314000	6.194280000	-2.337967000
6	-2.379153000	1.666260000	0.450296000
1	-2.507500000	1.735279000	1.533046000
1	-2.791308000	0.702809000	0.140429000
6	-0.875606000	1.703544000	0.120801000
1	-0.442284000	2.657630000	0.437249000
1	-0.706215000	1.595017000	-0.954341000
6	1.830642000	0.527728000	0.306700000
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1	1.765570000	0.524309000	-0.785136000

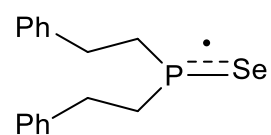


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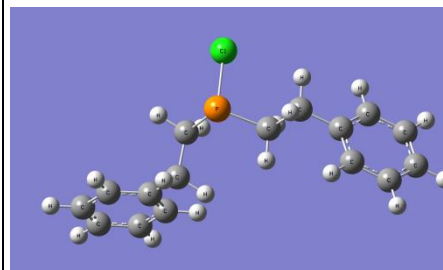


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6	4.034028000	1.762971000	0.305017000	
6	4.366036000	2.422588000	-0.882045000	
6	5.047026000	1.086115000	0.992006000	
6	5.667712000	2.409252000	-1.378130000	
1	3.601363000	2.964243000	-1.429920000	
6	6.353940000	1.061322000	0.511876000	
1	4.817766000	0.574116000	1.921048000	
6	6.652175000	1.724177000	-0.674297000	
1	5.915932000	2.927502000	-2.295555000	
1	7.132299000	0.539218000	1.053557000	
17	8.299907000	1.702406000	-1.288591000	
16	-0.049347000	0.327136000	2.905721000	
15	0.116775000	0.404934000	0.968850000	
17	-0.538302000	-1.333531000	-0.054939000	
6	-3.664455000	5.100653000	-0.177344000	
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6	-3.056172000	2.744053000	-0.263612000	
6	-3.784724000	2.638423000	-1.455199000	
6	-4.441761000	3.739896000	-2.003856000	
6	-4.384223000	4.978633000	-1.367024000	
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1	-5.000454000	3.629546000	-2.928191000	
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6	-2.322524000	1.559516000	0.320678000	
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6	-0.823110000	1.542426000	-0.021676000	
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1	2.413707000	-0.395365000	0.545714000	
1	1.772109000	0.227607000	-0.969172000	
6	2.442216000	1.767537000	0.428263000	
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1	2.267034000	1.974959000	1.490524000	
6	3.919671000	1.806980000	0.127387000	
6	4.416466000	2.362918000	-1.059227000	
6	4.845774000	1.259247000	1.028838000	
6	5.782416000	2.370353000	-1.341452000	
1	3.718822000	2.797371000	-1.769330000	
6	6.210748000	1.264074000	0.752532000	
1	4.480560000	0.830095000	1.956767000	
6	6.688375000	1.818725000	-0.436473000	
1	6.139285000	2.809291000	-2.268203000	
1	6.905859000	0.837458000	1.469082000	
1	7.751902000	1.824053000	-0.651595000	
34	0.155790000	0.638071000	2.958439000	
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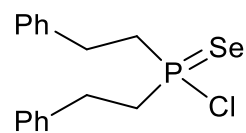
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6	-3.237661000	3.968954000	0.410709000
6	-3.091117000	2.768931000	-0.294667000
6	-3.605283000	2.694360000	-1.593814000
6	-4.244296000	3.788255000	-2.175350000
6	-4.379469000	4.978891000	-1.463539000
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1	-4.641241000	3.708556000	-3.181450000
1	-4.879193000	5.829694000	-1.912733000
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1	-2.767928000	0.656993000	-0.078429000
6	-0.851300000	1.650214000	0.071214000
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1	-0.642294000	1.595756000	-1.002827000
6	1.841199000	0.439255000	0.283459000
1	2.384573000	-0.417865000	0.688790000
1	1.831084000	0.324058000	-0.806051000
6	2.546956000	1.754138000	0.683224000
1	2.009441000	2.606270000	0.258232000
1	2.504363000	1.856088000	1.770863000
6	3.986103000	1.783000000	0.217408000
6	4.331364000	2.331165000	-1.022944000
6	5.001948000	1.233920000	1.008867000
6	5.653853000	2.329938000	-1.463082000
1	3.559020000	2.770591000	-1.647110000
6	6.325219000	1.231036000	0.573031000
1	4.754634000	0.812036000	1.978197000
6	6.655456000	1.778276000	-0.666109000
1	5.902308000	2.764016000	-2.425324000
1	7.099021000	0.806403000	1.203029000
1	7.685187000	1.779479000	-1.005189000
34	-0.062124000	0.275573000	3.008462000
15	0.081560000	0.235391000	0.856655000



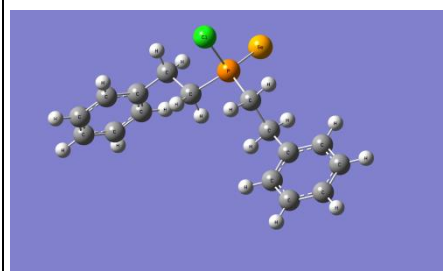
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6	-3.608030000	2.762119000	-1.664374000
6	-4.253341000	3.873234000	-2.204425000
6	-4.415500000	5.027835000	-1.440484000
1	-4.061604000	5.952158000	0.471243000
1	-2.922264000	3.979176000	1.424783000
1	-3.494485000	1.863530000	-2.263184000
1	-4.634575000	3.834782000	-3.218887000
1	-4.920522000	5.891685000	-1.857640000
6	-2.386924000	1.588442000	0.220372000
1	-2.547745000	1.533362000	1.299725000
1	-2.786342000	0.667253000	-0.211323000
6	-0.874519000	1.663719000	-0.055310000
1	-0.459145000	2.583210000	0.368718000
1	-0.670058000	1.663834000	-1.130137000



Charge = 0. Multiplicity = 1



6	1.840543000	0.503494000	0.088892000
1	2.348916000	-0.435441000	0.321220000
1	1.800856000	0.594894000	-1.000464000
6	2.585502000	1.693345000	0.726958000
1	2.076514000	2.628801000	0.476551000
1	2.552809000	1.591769000	1.814299000
6	4.023104000	1.762502000	0.254818000
6	4.369327000	2.492769000	-0.886812000
6	5.031178000	1.075173000	0.939560000
6	5.687948000	2.534448000	-1.336278000
1	3.602153000	3.041059000	-1.425370000
6	6.350721000	1.114265000	0.493350000
1	4.782266000	0.510447000	1.832665000
6	6.683074000	1.843370000	-0.647288000
1	5.938162000	3.110183000	-2.220463000
1	7.119735000	0.579610000	1.039769000
1	7.709877000	1.876864000	-0.993334000
34	-0.126545000	-0.003083000	2.754377000
15	0.109207000	0.292213000	0.686545000
17	-0.490163000	-1.338710000	-0.538878000

Table S2 The energy of the stages of the chalcogenophosphoryl chlorides formation reaction (kcal·mol⁻¹)

Phosphine chalcogenides	1 + Et ₃ N → A + Et ₃ NH ⁺	A + CCl ₄ → B + C	B + C → D + ⁻ CCl ₃
	107.2	12.2	-34.4
	114.0	7.3	-38.0
	108.5	12.8	-37.8
	99.8	14.7	-27.8
	94.5	20.0	-27.6
	95.4	16.5	-24.1

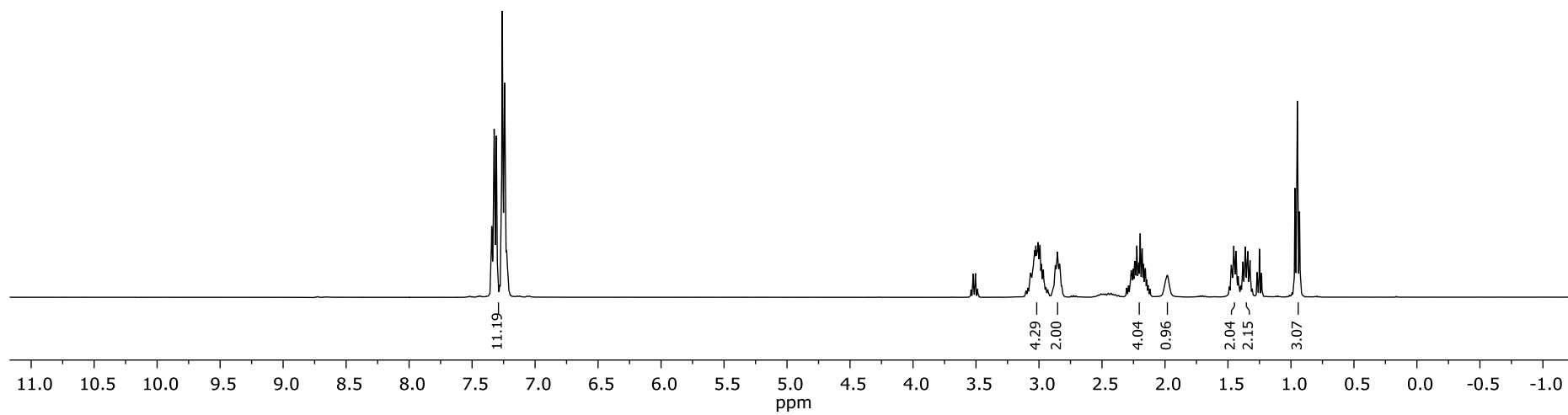
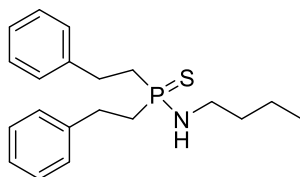
3. NMR Spectra

N-butyl-bis(2-phenylethyl)phosphinothioic amide (2a)

¹H-NMR

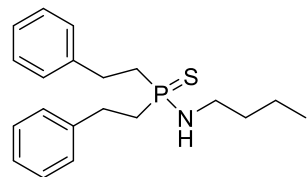
7.34
7.32
7.31
7.26 CDCl₃
7.26
7.24

3.07
3.05
3.04
3.03
3.02
3.01
2.99
2.98
2.97
2.89
2.87
2.85
2.84
2.82
2.27
2.24
2.23
2.22
2.21
2.20
2.18
2.17
2.16
1.98
1.47
1.46
1.45
1.44
1.43
1.42
1.38
1.36
1.34
1.33
0.97
0.95
0.93



***N*-butyl-bis(2-phenylethyl)phosphinothioic amide (2a)**

¹³C_NMR



140.91
140.76

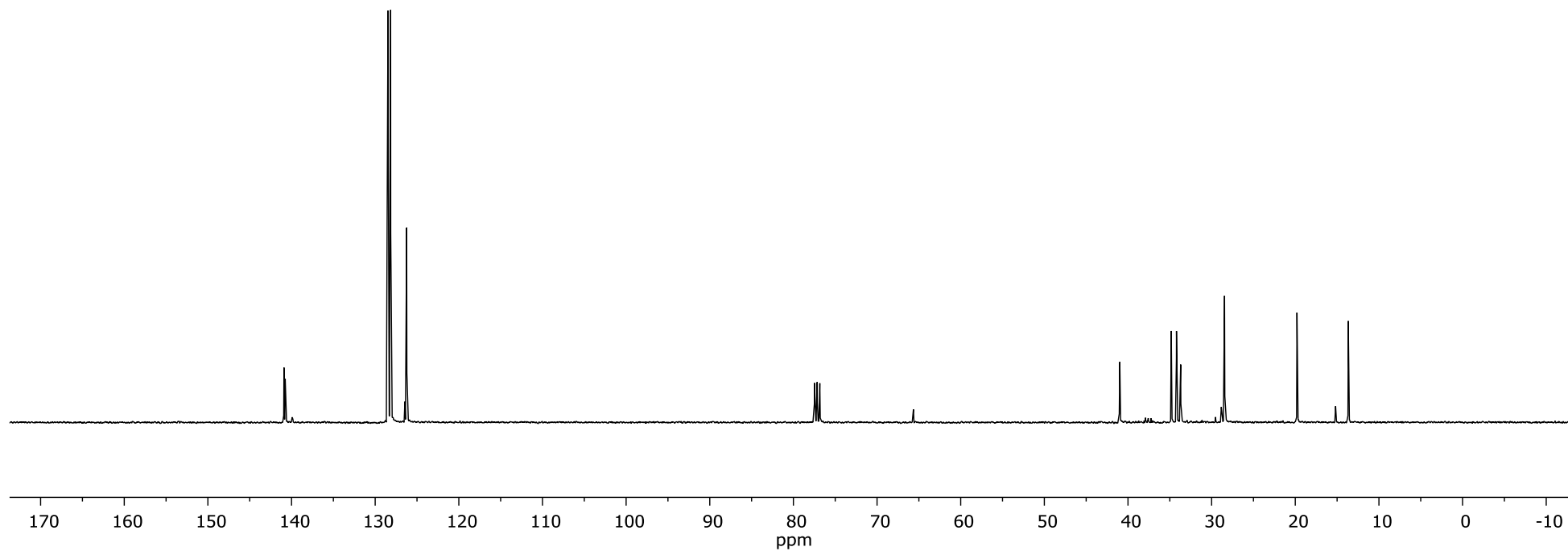
128.49
128.15
126.24

77.48
77.16 CDCl₃
76.84

41.01
40.98
34.83
34.20
33.77
33.70
28.48
28.45

— 19.80

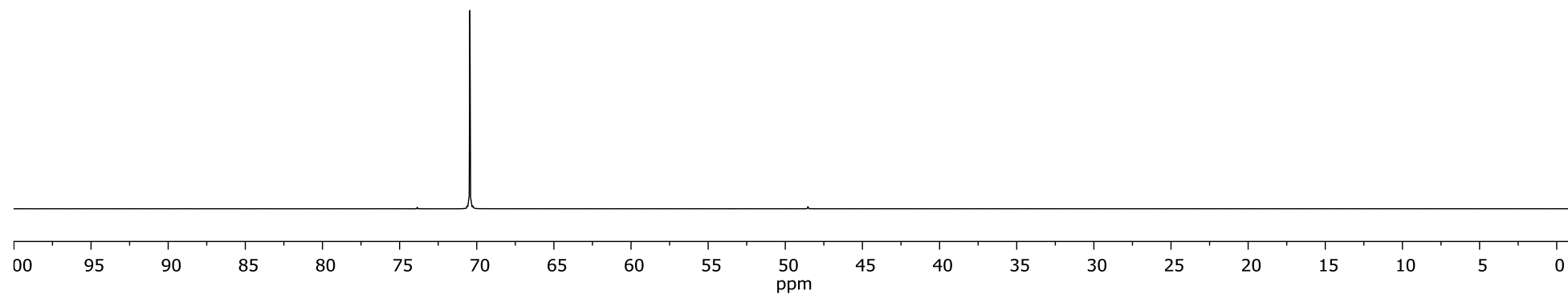
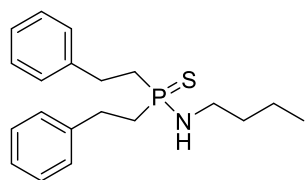
— 13.66



***N*-butyl-bis(2-phenylethyl)phosphinothioic amide (2a)**

31P_NMR

— 70.45

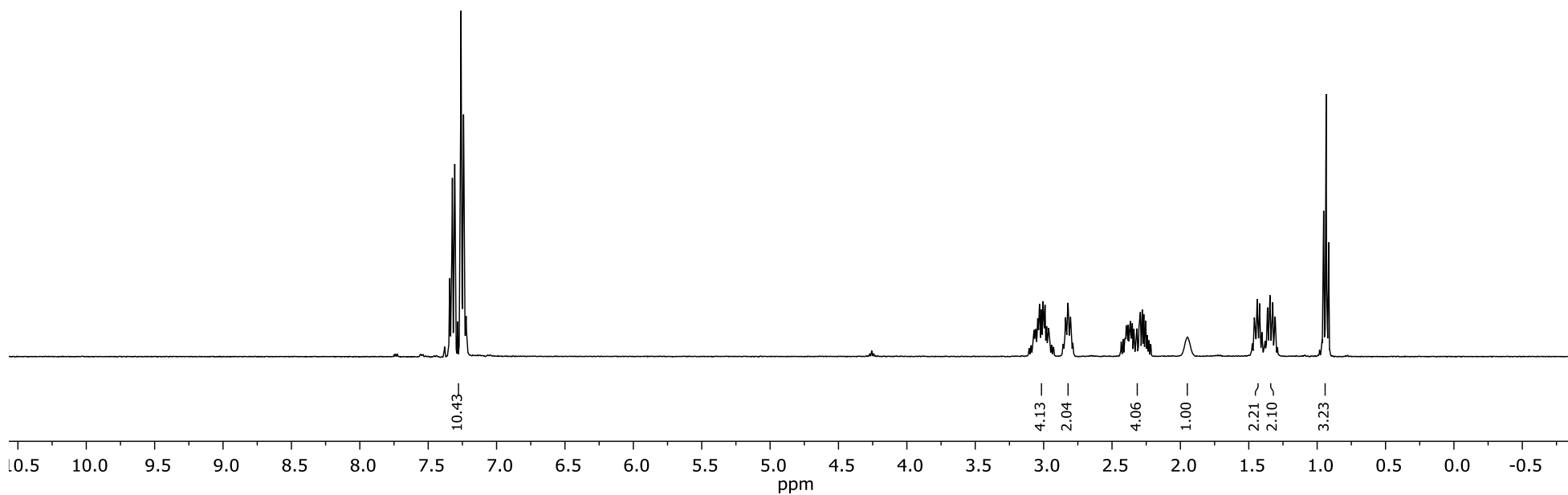
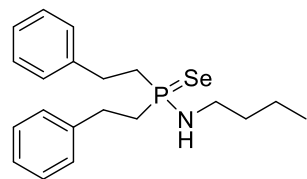


N-butyl-bis(2-phenylethyl)phosphoselenoic amide (2b)

¹H_NMR

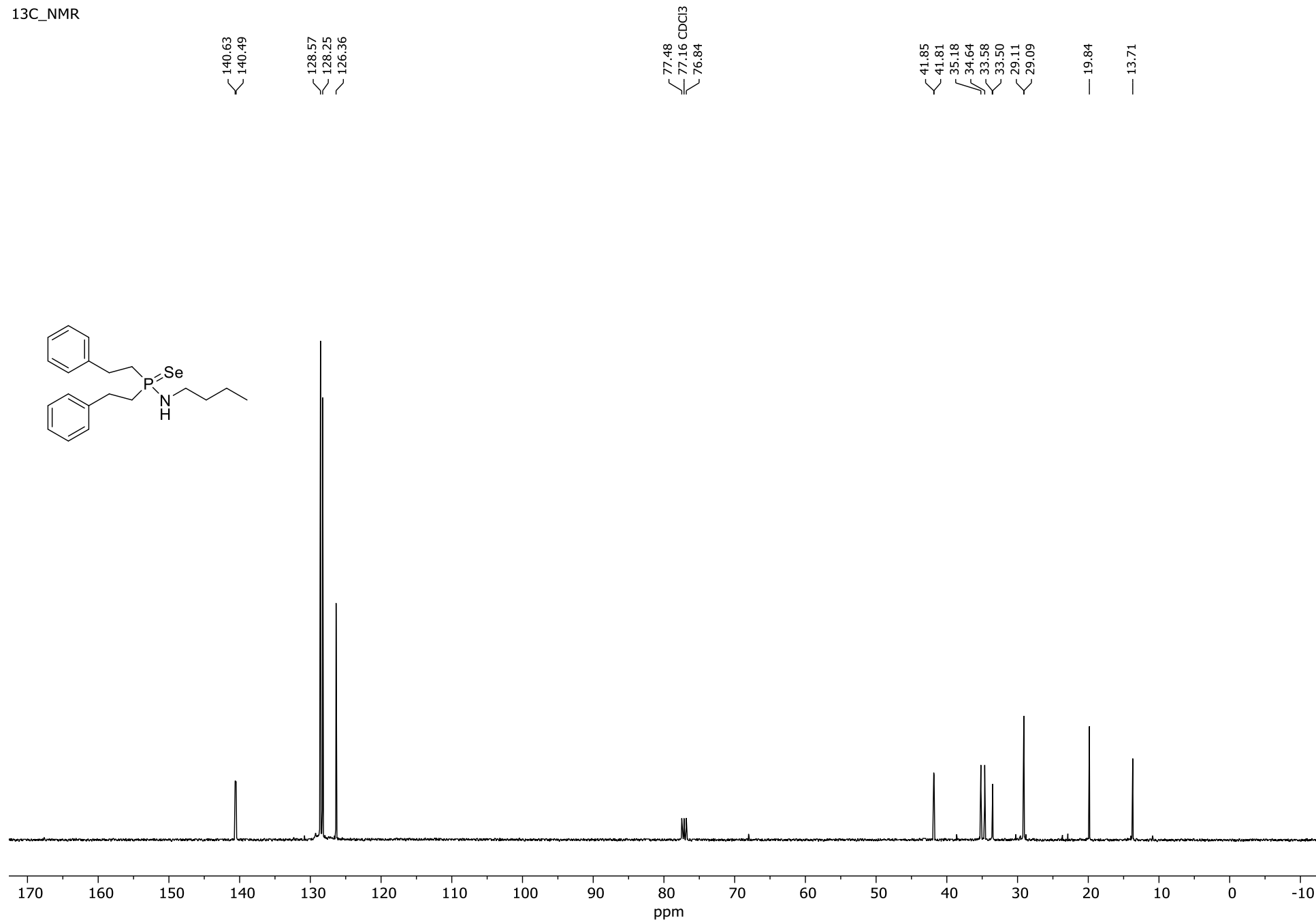
7.34
7.31
7.26 CDCl₃
7.24
7.22

3.09
3.07
3.04
3.03
3.00
2.99
2.98
2.95
2.93
2.86
2.84
2.82
2.80
2.79
2.43
2.42
2.39
2.37
2.35
2.34
2.30
2.29
2.28
2.27
2.25
2.24
2.23
2.22
1.95
1.47
1.46
1.44
1.42
1.40
1.38
1.36
1.34
1.32
1.31
1.29
0.95
0.93
0.92



***N*-butyl-bis(2-phenylethyl)phosphinoselenoic amide (2b)**

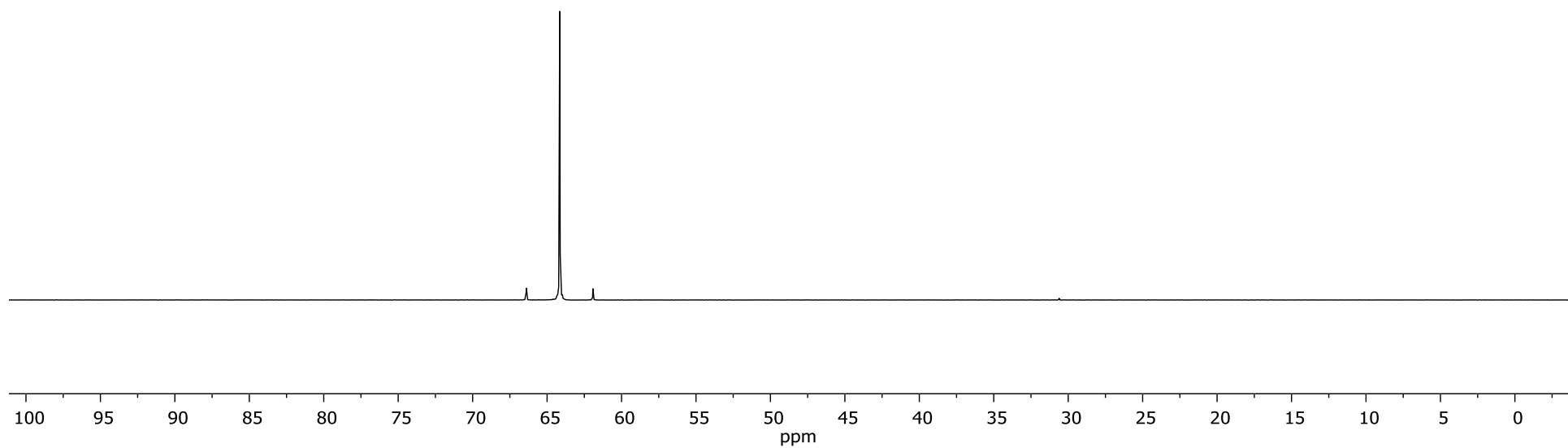
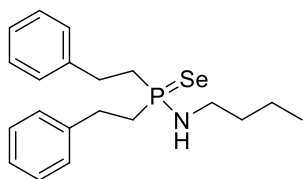
¹³C_NMR



***N*-butyl-bis(2-phenylethyl)phosphinoselenoic amide (2b)**

31P_NMR

66.39
64.15
61.92

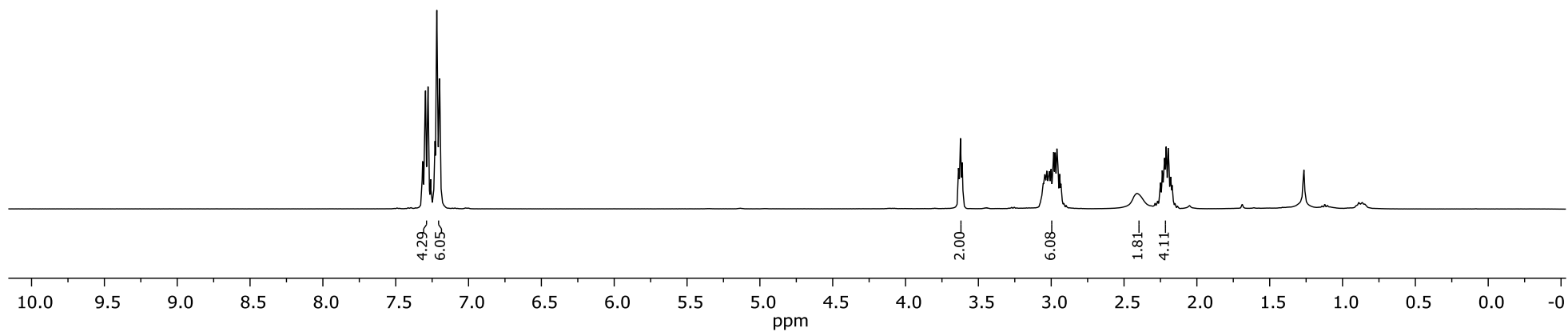
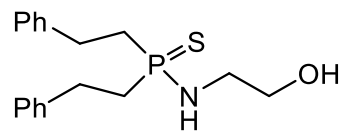


***N*-(2-hydroxyethyl)-bis(2-phenylethyl)phosphinothioic amide (4a)**

¹H_NMR

7.32
7.30
7.28
7.26
7.26 CDCl₃
7.23
7.22
7.20

3.64
3.62
3.61
3.06
3.04
3.03
3.01
3.00
2.98
2.97
2.96
2.94
2.93
2.41
2.25
2.24
2.22
2.21
2.20
2.18
2.17



***N*-(2-hydroxyethyl)-bis(2-phenylethyl)phosphinothioic amide (4a)**

¹³C_NMR

140.90
140.76

128.79
128.41
126.58

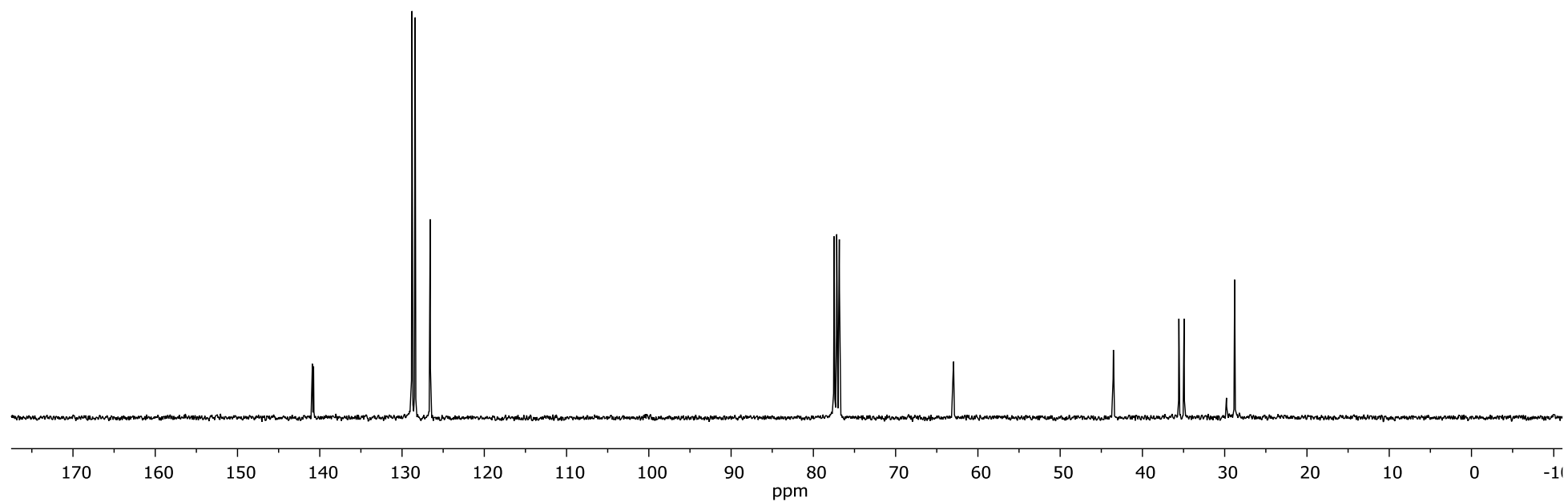
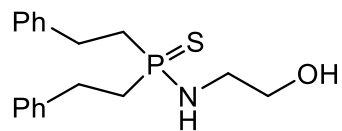
77.48
77.16 CDCl₃
76.84

63.02
62.96

43.55
43.52

35.57
34.94

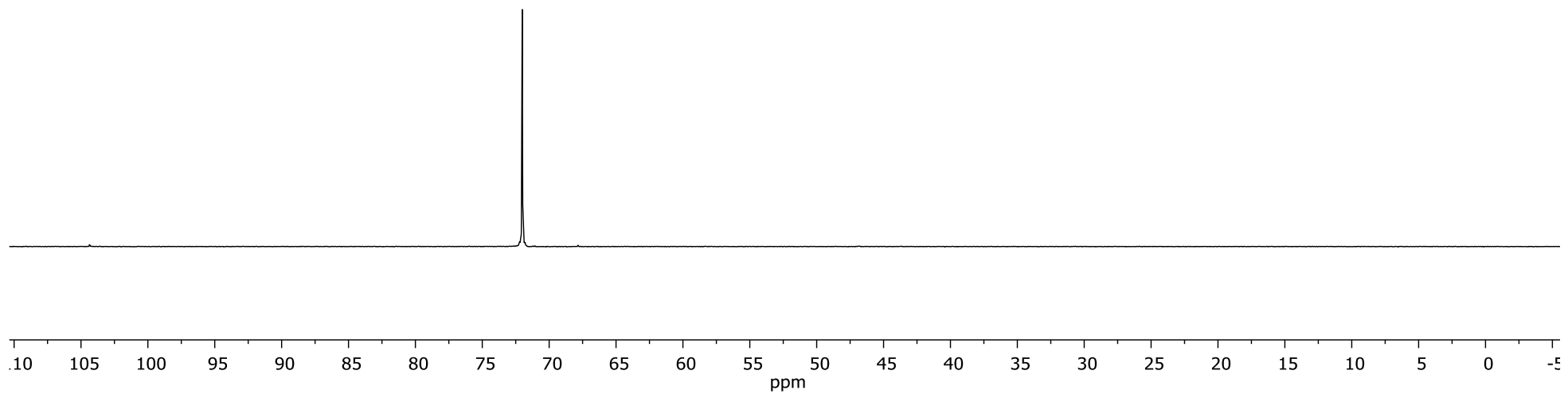
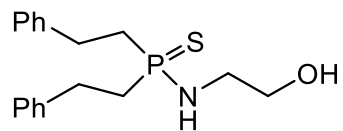
28.81
28.78



***N*-(2-hydroxyethyl)-bis(2-phenylethyl)phosphinothioic amide (4a)**

31P_NMR

— 72.01

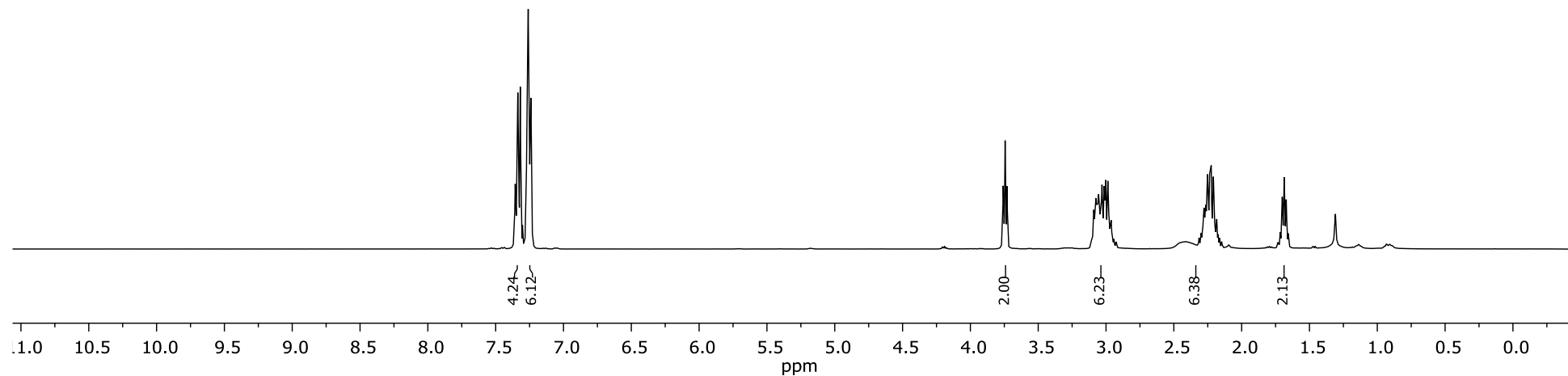
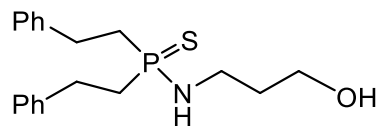


***N*-(3-hydroxypropyl)-bis(2-phenylethyl)phosphinothioic amide (4b)**

¹H_NMR

7.36
7.34
7.32
7.30
7.27
7.26
7.26 CDCl₃
7.24

3.76
3.74
3.73
3.09
3.08
3.07
3.05
3.03
3.01
3.00
2.99
2.96
2.41
2.31
2.30
2.28
2.26
2.25
2.24
2.22
2.21
2.18
1.72
1.70
1.69
1.67
1.66



***N*-(3-hydroxypropyl)-bis(2-phenylethyl)phosphinothioic amide (4b)**

¹³C_NMR

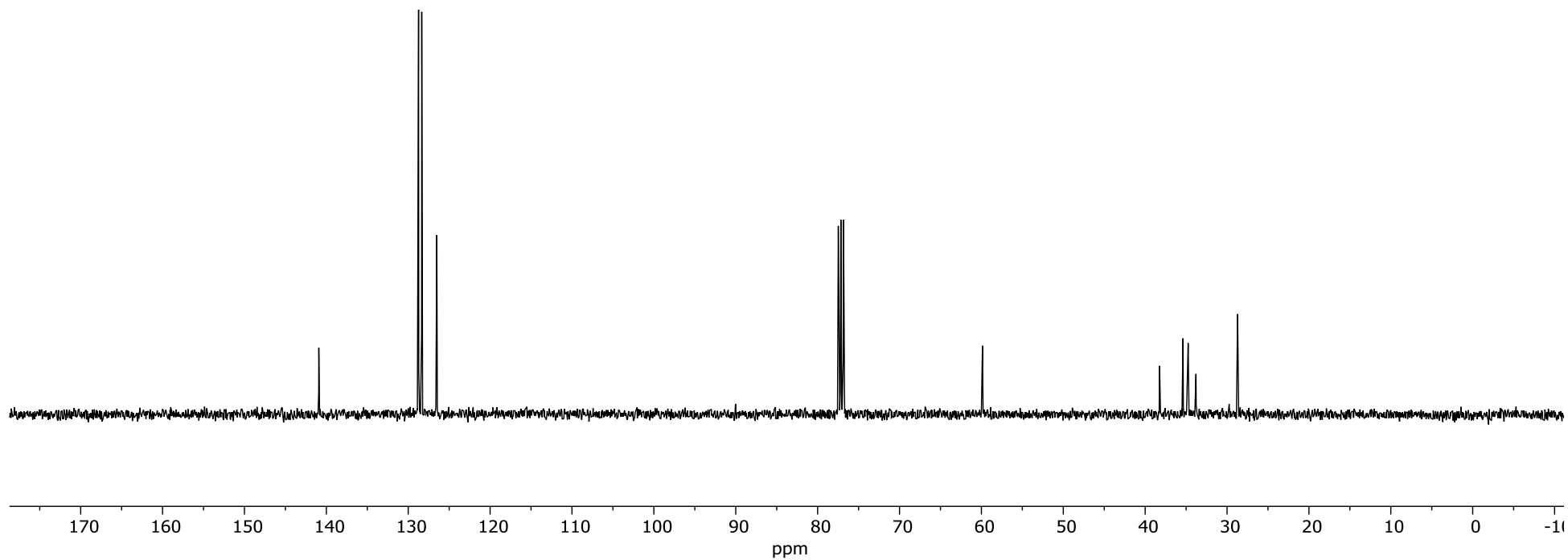
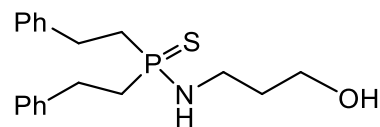
140.92
140.78

128.74
128.35
126.51

77.48
77.16 CDCl₃
76.84

— 59.87

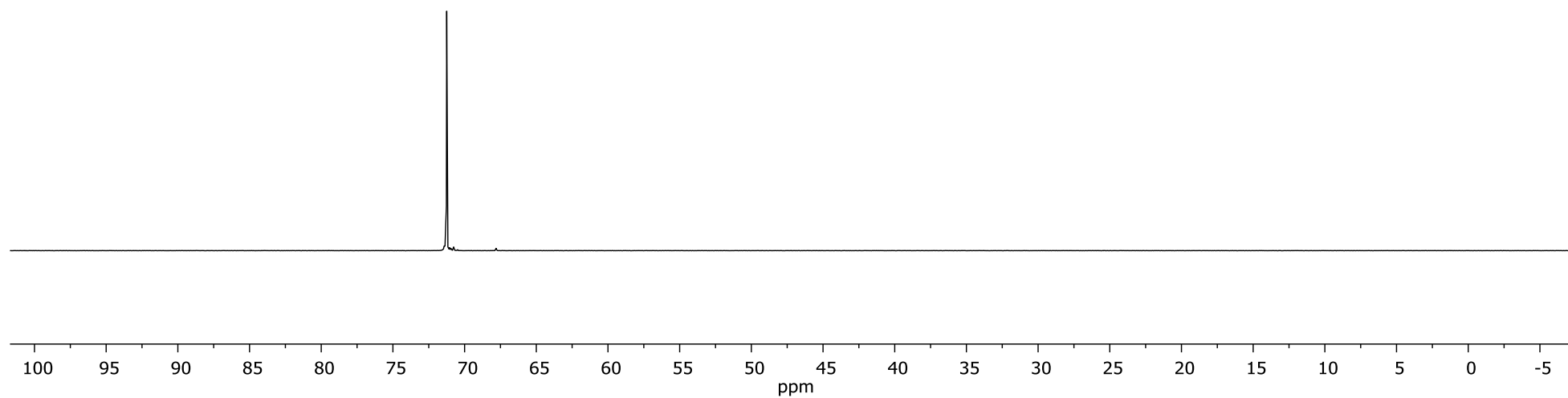
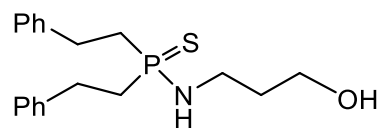
38.24
35.38
34.75
33.86
33.79
28.74
28.71



***N*-(3-hydroxypropyl)-bis(2-phenylethyl)phosphinothioic amide (4b)**

31P_NMR

71.25

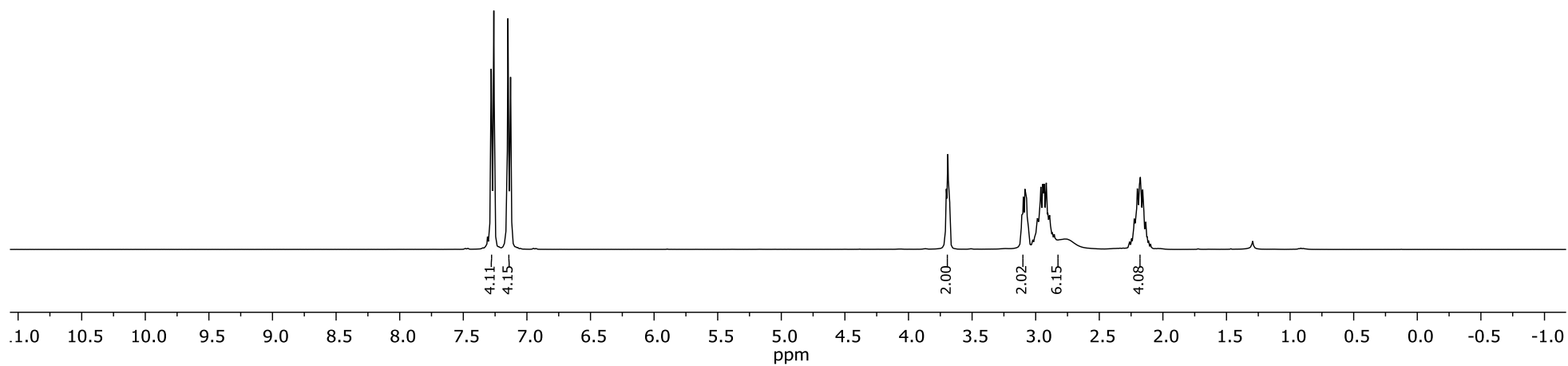
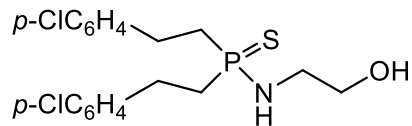


***N*-(2-hydroxyethyl)-bis[2-(4-chlorophenyl)ethyl]phosphinothioic amide (4c)**

1H_NMR

7.28 CDCl₃
7.26
7.15
7.13

3.70
3.69
3.68
3.11
3.10
3.08
3.07
3.06
3.00
2.99
2.98
2.97
2.96
2.94
2.93
2.92
2.90
2.90
2.89
2.88
2.23
2.22
2.21
2.20
2.20
2.19
2.18
2.17
2.16
2.15
2.14
2.13



***N*-(2-hydroxyethyl)-bis[2-(4-chlorophenyl)ethyl]phosphinothioic amide (4c)**

¹³C-NMR

139.14
138.99

132.22
129.65
128.77

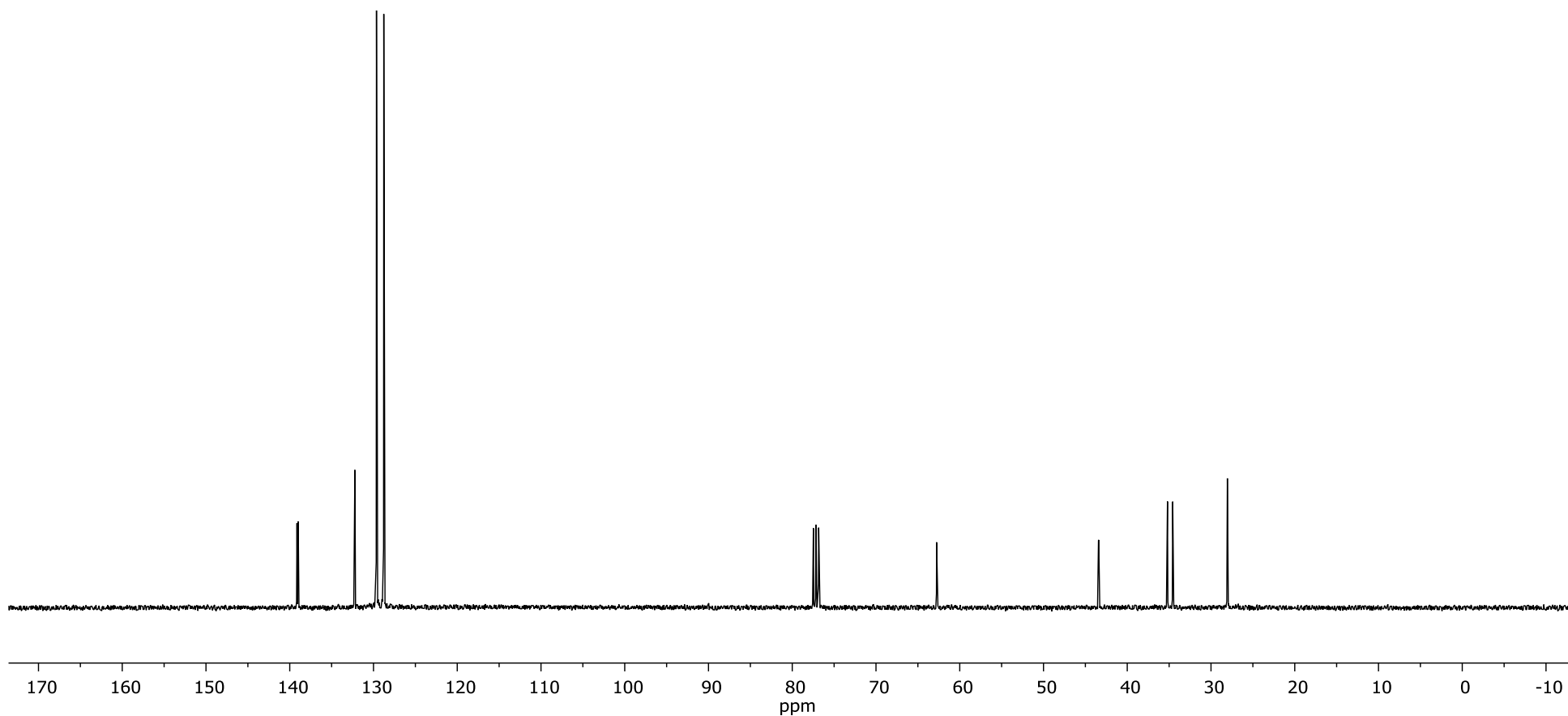
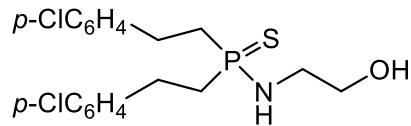
77.48
77.16 CDCl₃
76.84

62.77
62.70

43.43
43.41

35.20
34.57

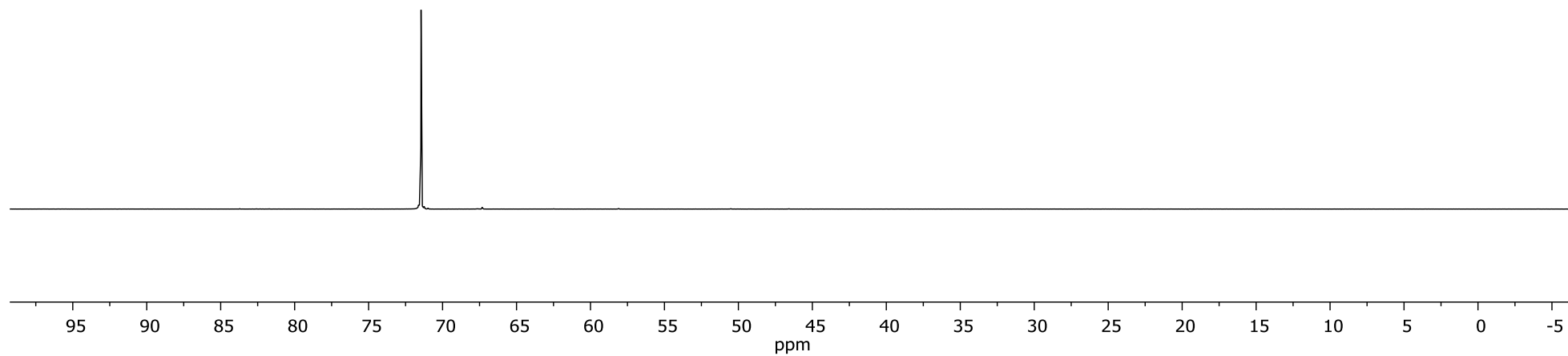
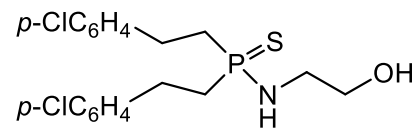
28.05
28.02



***N*-(2-hydroxyethyl)-bis[2-(4-chlorophenyl)ethyl]phosphinothioic amide (4c)**

31P_NMR

— 71.45

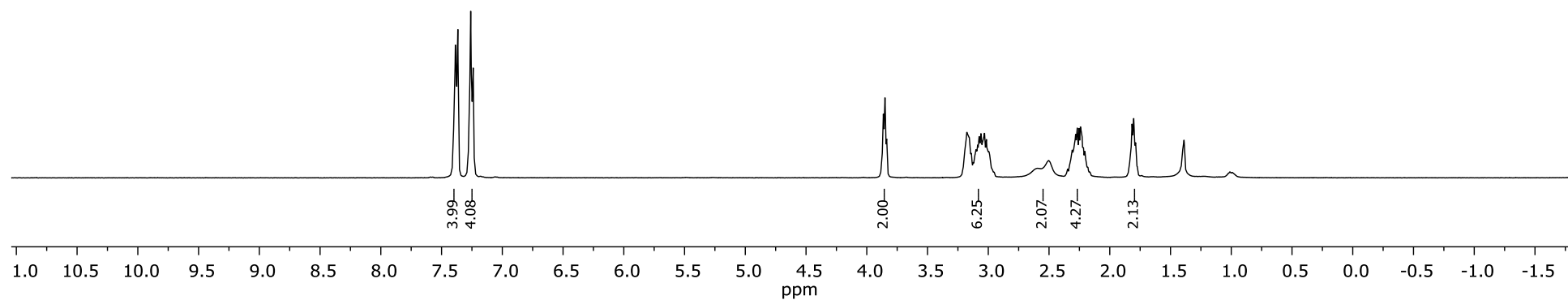
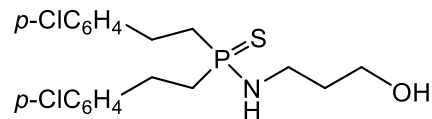


***N*-(3-hydroxypropyl)-bis[2-(4-chlorophenyl)ethyl]phosphinothioic amide (4d)**

1H_NMR

7.39
7.37
7.26 CDCl3
7.24

3.86
3.85
3.84
3.19
3.18
3.16
3.15
3.14
3.11
3.10
3.08
3.06
3.04
3.03
3.02
2.99
2.98
2.97
2.60
2.50
2.33
2.32
2.31
2.29
2.28
2.27
2.25
2.24
2.23
2.20
2.19
2.18
1.84
1.83
1.82
1.80
1.79
1.77



***N*-(3-hydroxypropyl)-bis[2-(4-chlorophenyl)ethyl]phosphinothioic amide (4d)**

¹³C_NMR

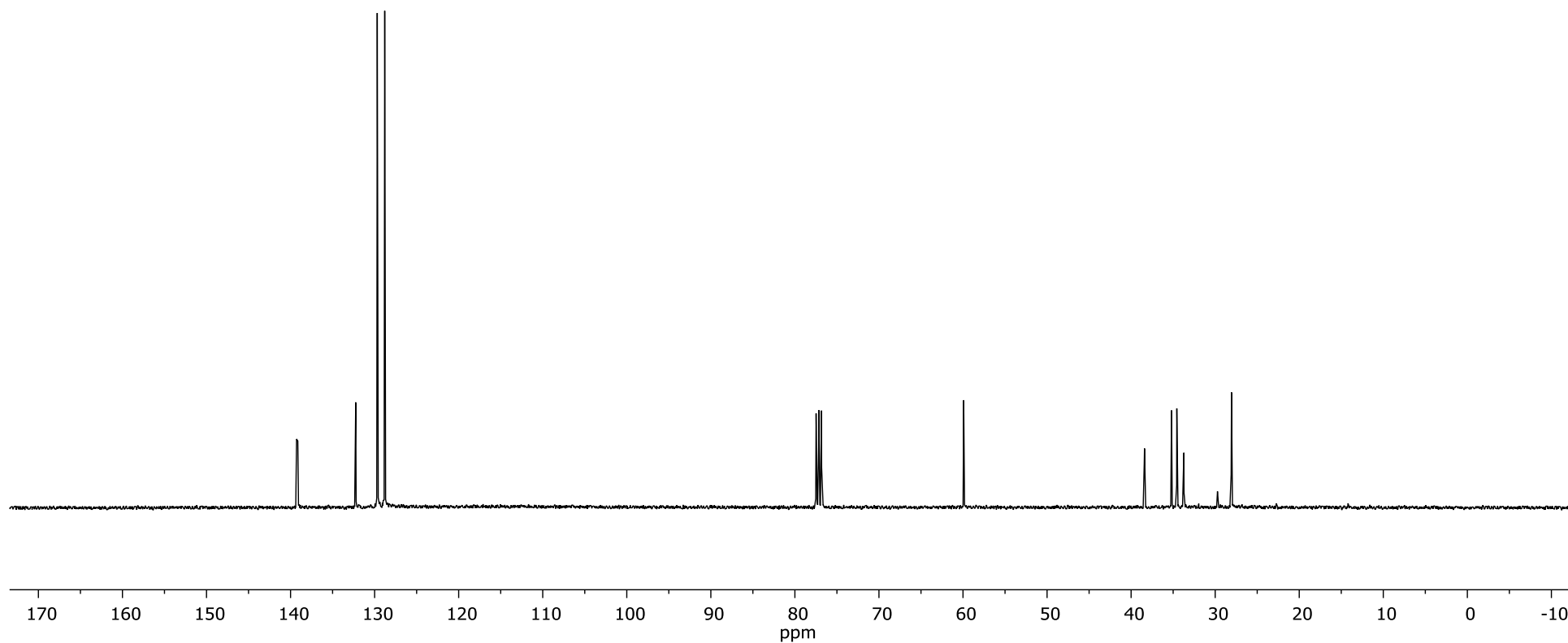
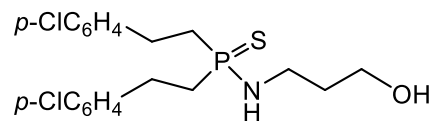
139.29
139.14

132.25
129.69
128.80

77.48
77.16 CDCl₃
76.84

59.92

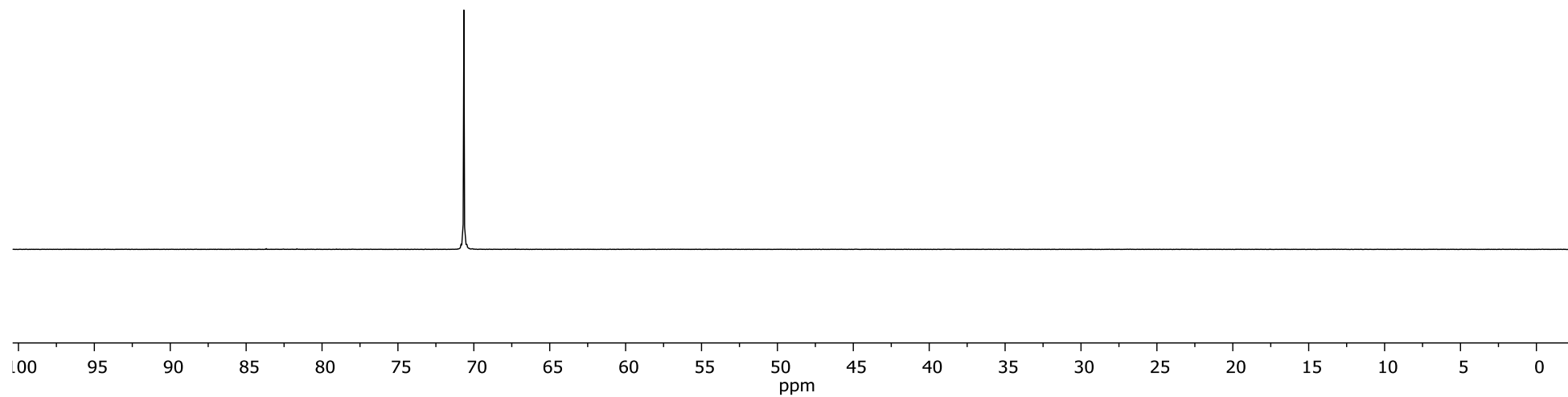
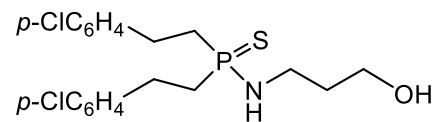
38.43
38.41
35.19
34.56
33.79
33.73
28.08
28.06



***N*-(3-hydroxypropyl)-bis[2-(4-chlorophenyl)ethyl]phosphinothioic amide (4d)**

31P_NMR

70.65

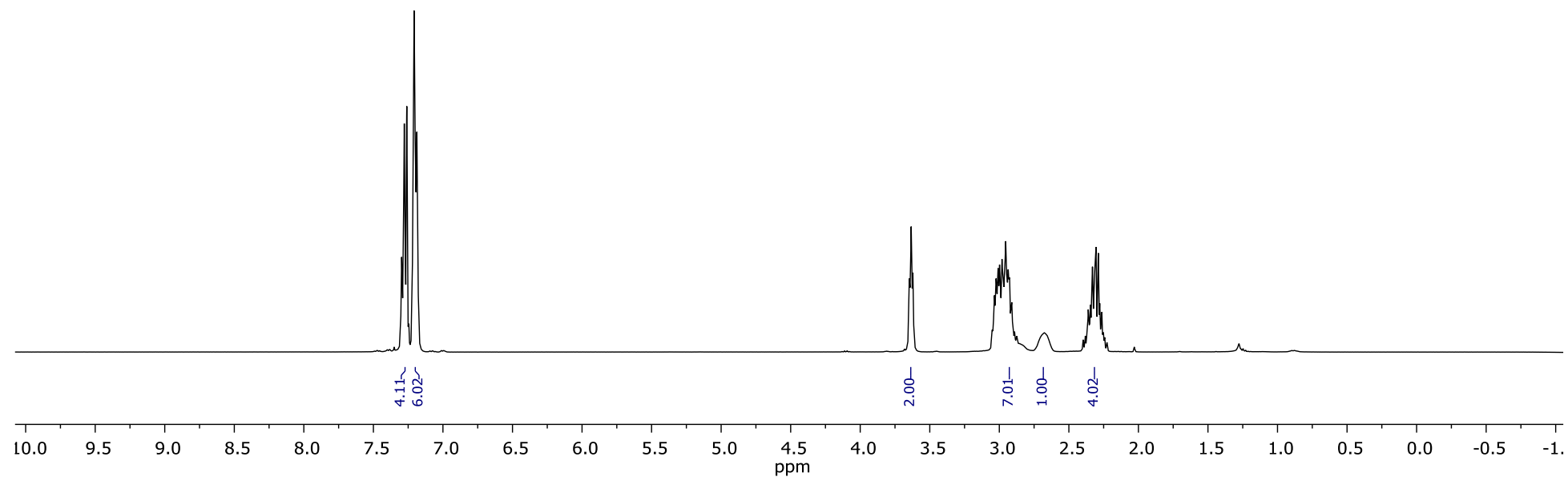
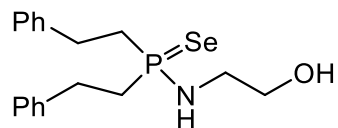


***N*-(2-hydroxyethyl)-bis(2-phenylethyl)phosphinoselenoic amide (4e)**

1H_NMR

7.30
7.28
7.26 CDCl3
7.26
7.21
7.19

3.65
3.63
3.62
3.03
3.02
3.01
2.98
2.95
2.93
2.91
2.88
2.68
2.36
2.34
2.33
2.30
2.29
2.28
2.26



***N*-(2-hydroxyethyl)-bis(2-phenylethyl)phosphinoselenoic amide (4e)**

¹³C_NMR

140.43
140.29

128.62
128.27
126.44

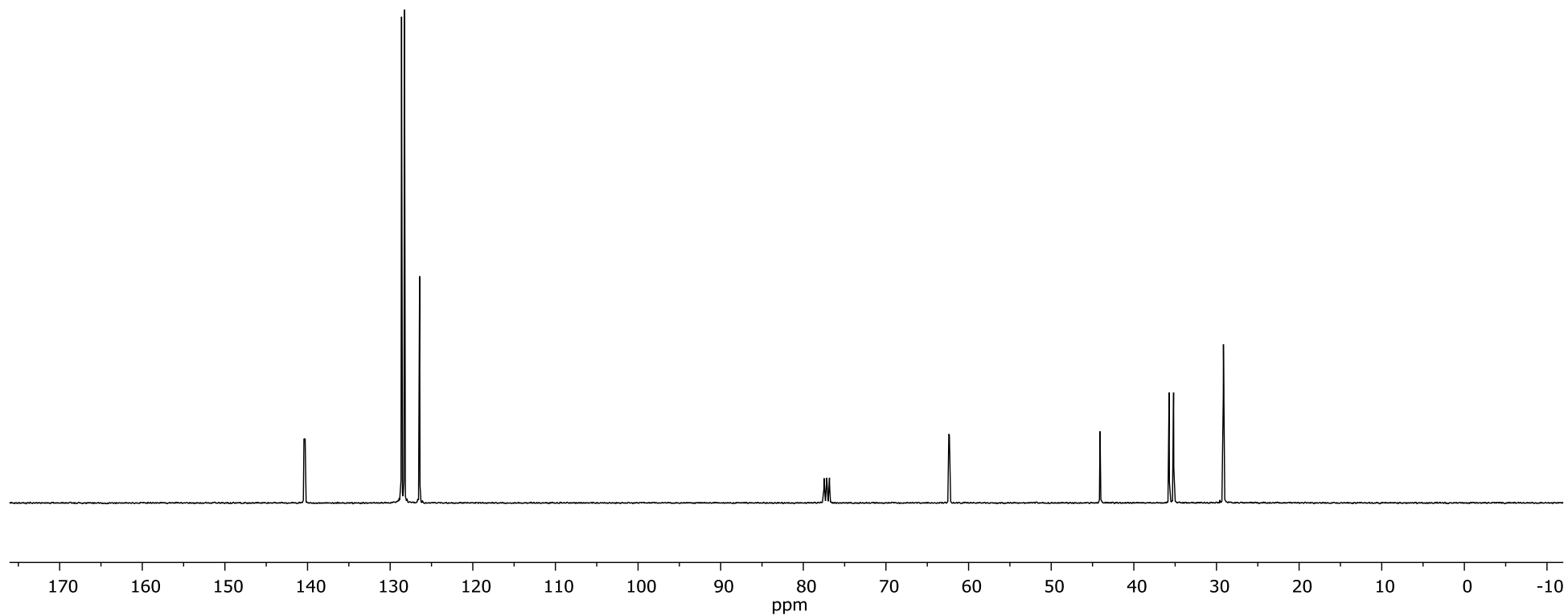
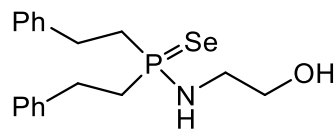
77.48
77.16 CDCl₃
76.84

62.39
62.32

44.09
44.06

35.73
35.19

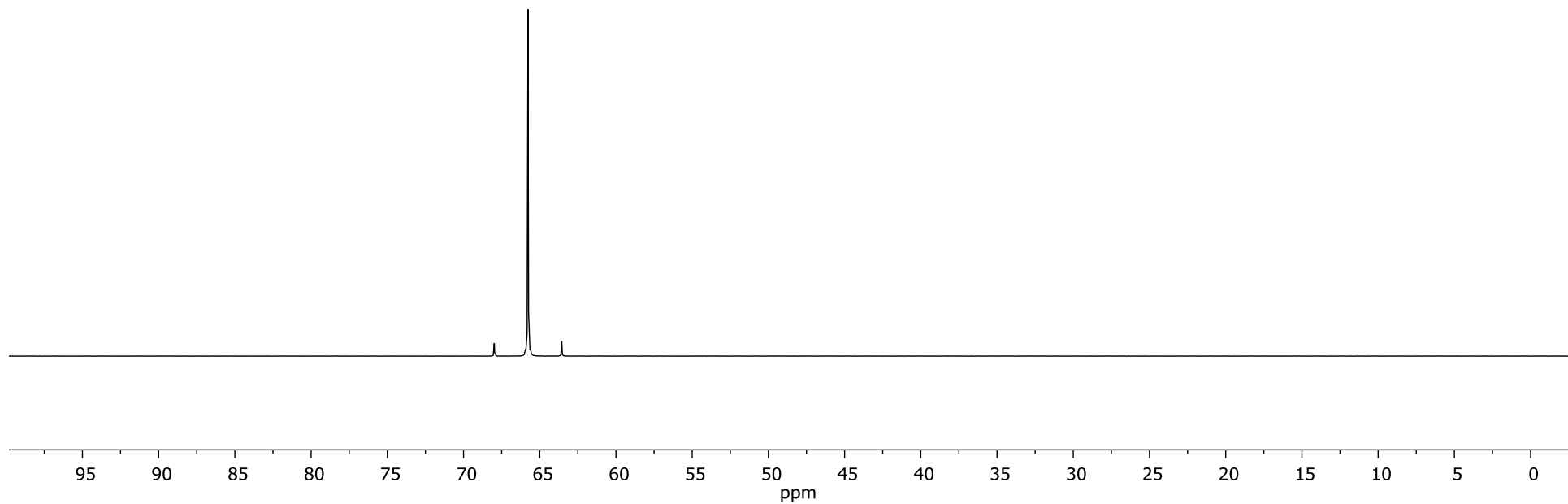
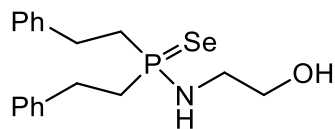
29.16
29.14



***N*-(2-hydroxyethyl)-bis(2-phenylethyl)phosphinoselenoic amide (4e)**

31P_NMR

67.98
65.77
63.56

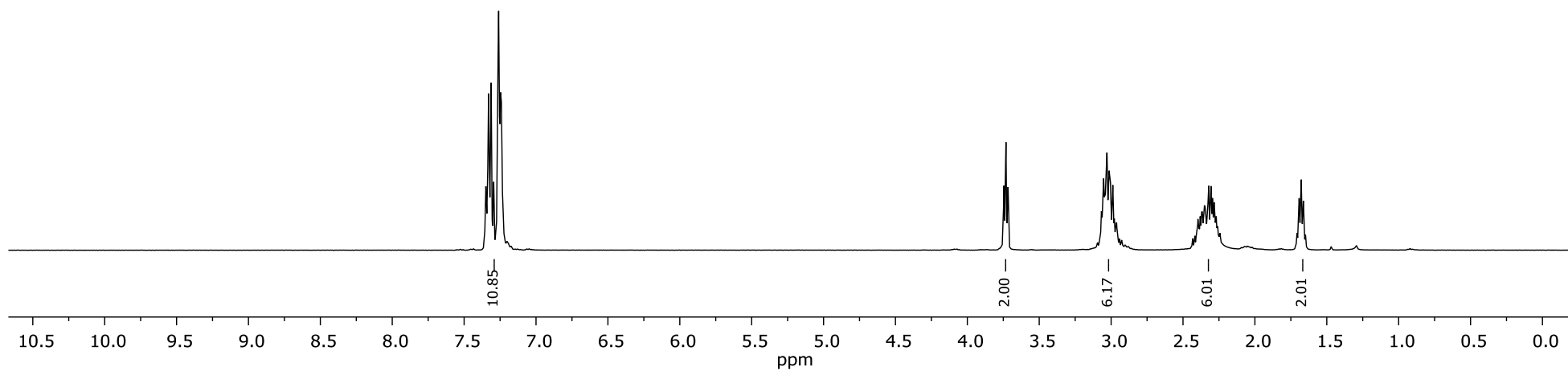
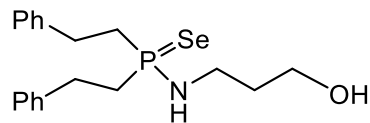


***N*-(3-hydroxypropyl)-bis(2-phenylethyl)phosphinoselenoic amide (4f)**

1H_NMR

7.35
7.33
7.31
7.29
7.26 CDCl3
7.26
7.24

3.75
3.73
3.72
3.07
3.05
3.03
3.01
2.99
2.96
2.42
2.38
2.35
2.32
2.29
2.28
2.24
1.71
1.69
1.68
1.66
1.65



***N*-(3-hydroxypropyl)-bis(2-phenylethyl)phosphinoselenoic amide (4f)**

¹³C_NMR

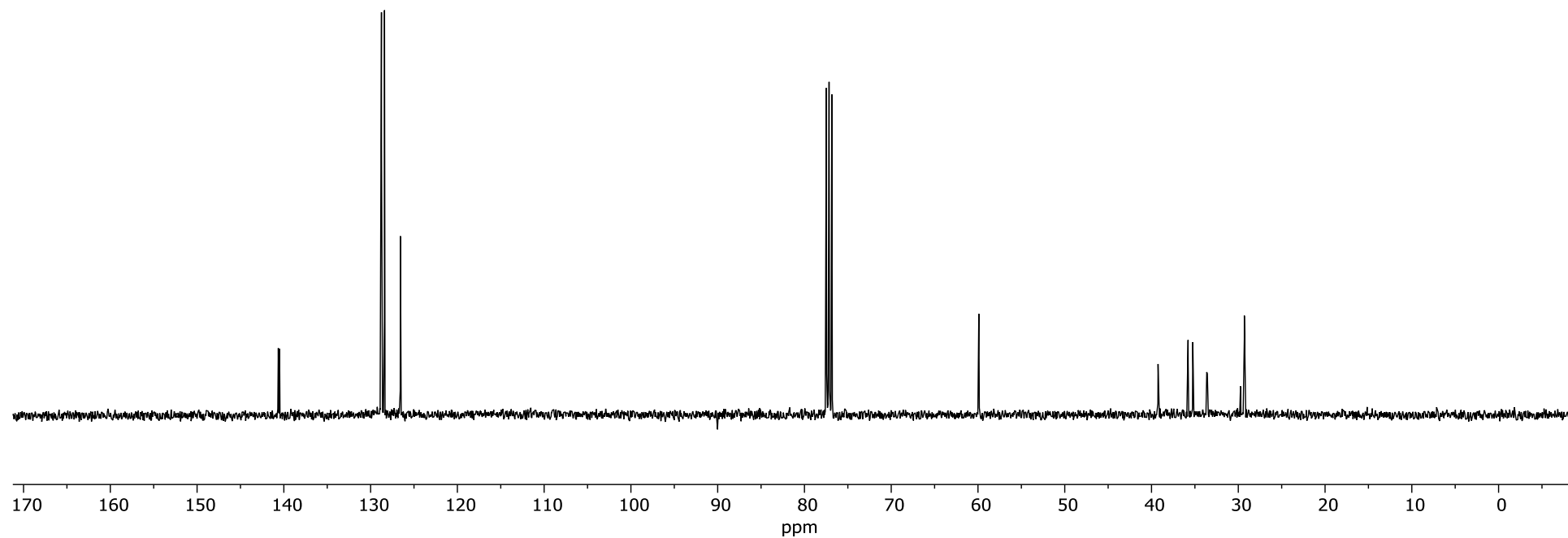
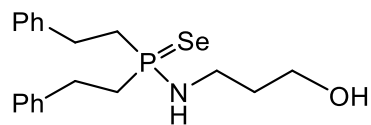
140.64
140.49

128.75
128.40
126.56

77.48
77.16 CDCl₃
76.84

— 59.90

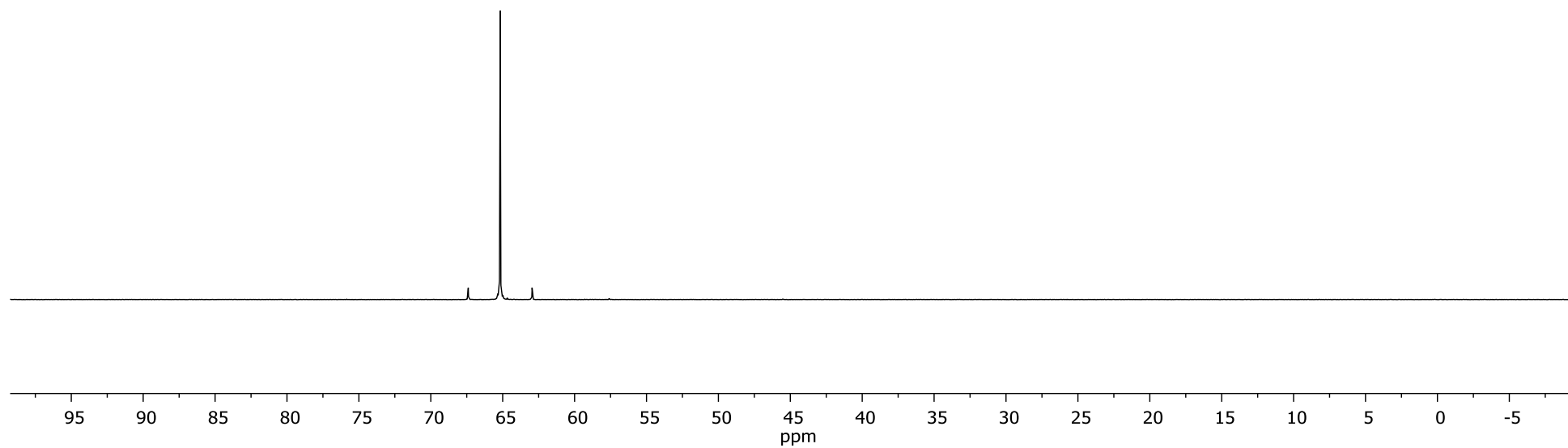
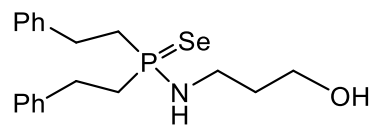
39.24
39.21
35.79
35.24
33.63
33.57
29.30
29.28



***N*-(3-hydroxypropyl)-bis(2-phenylethyl)phosphinoselenoic amide (4f)**

31P_NMR

— 65.17

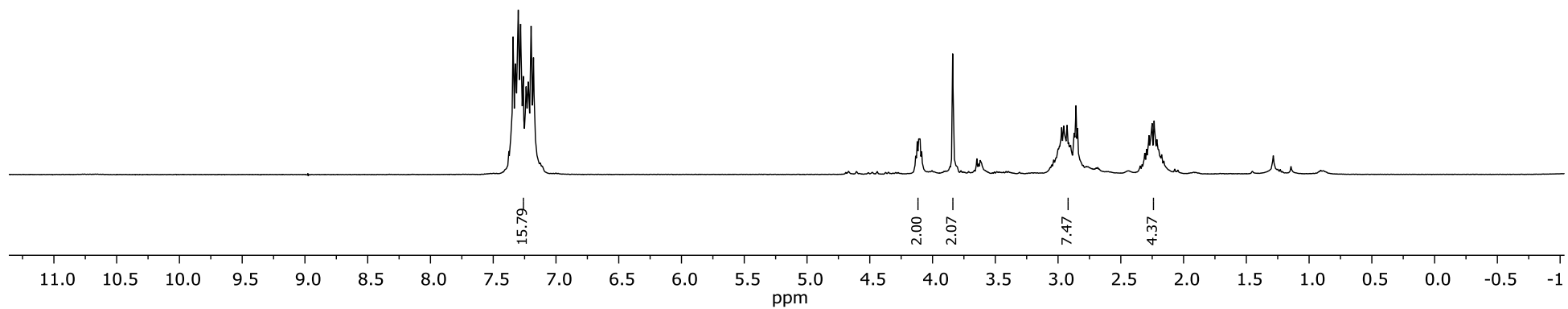
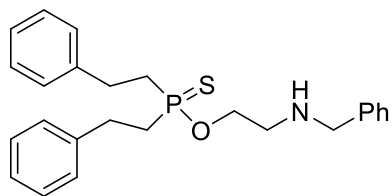


***O*-[2-(benzylamino)ethyl]-bis(2-phenylethyl)phosphinothioate (9a)**

¹H_NMR

7.34
7.32
7.30
7.28
7.26 CDCl₃
7.24
7.22
7.20
7.18

4.13
4.12
4.11
4.10
4.09
3.84
3.02
3.00
2.99
2.97
2.95
2.94
2.93
2.87
2.86
2.84
2.31
2.29
2.28
2.25
2.23
2.21
2.17



***O*-[2-(benzylamino)ethyl]-bis(2-phenylethyl)phosphinothioate (9a)**

¹³C_NMR

140.67
140.52
139.43

128.72
128.58
128.34
128.31
127.30
126.53

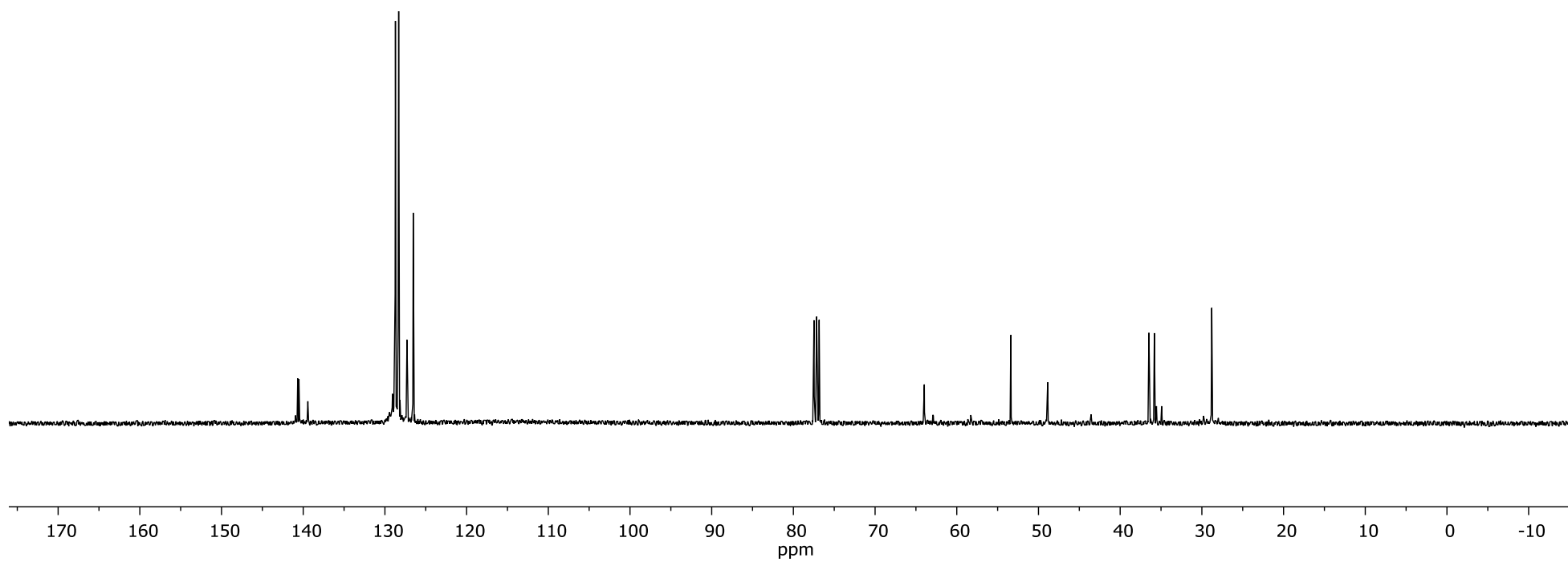
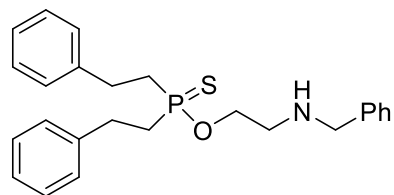
77.48
77.16 CDCl₃
76.84

64.04
63.97

53.38
48.94
48.87

36.48
35.81

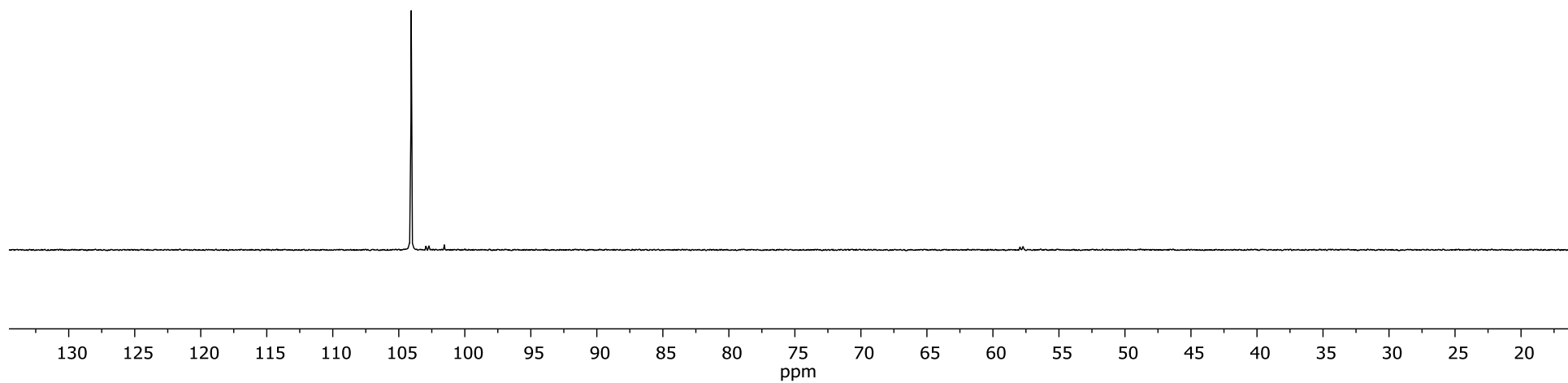
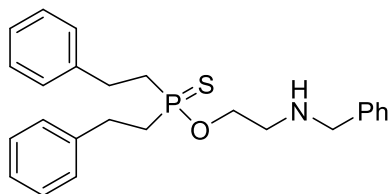
28.82
28.79



***O*-[2-(benzylamino)ethyl]-bis(2-phenylethyl)phosphinothioate (9a)**

31P_NMR

104.06

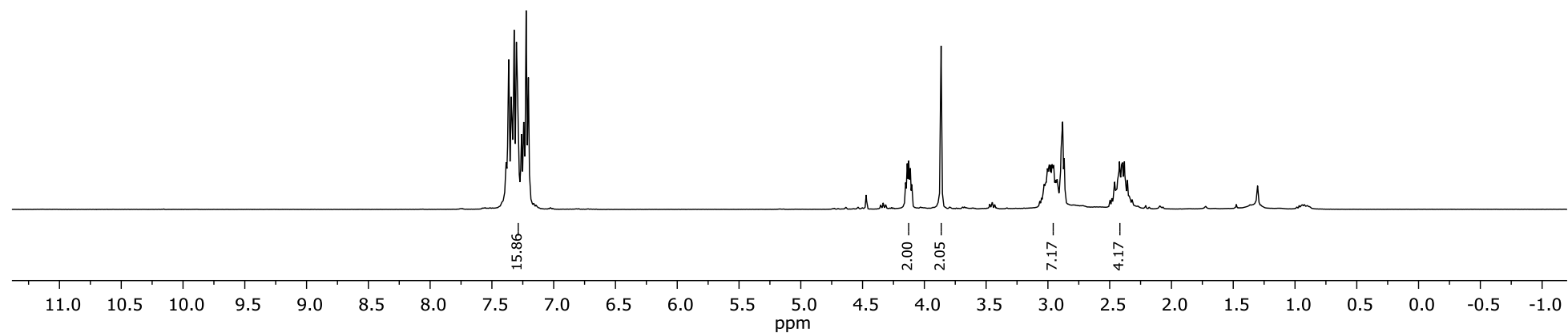
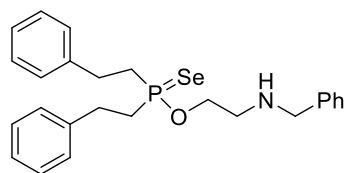


***O*-[2-(benzylamino)ethyl]-bis(2-phenylethyl)phosphoselenoate (9b)**

¹H_NMR

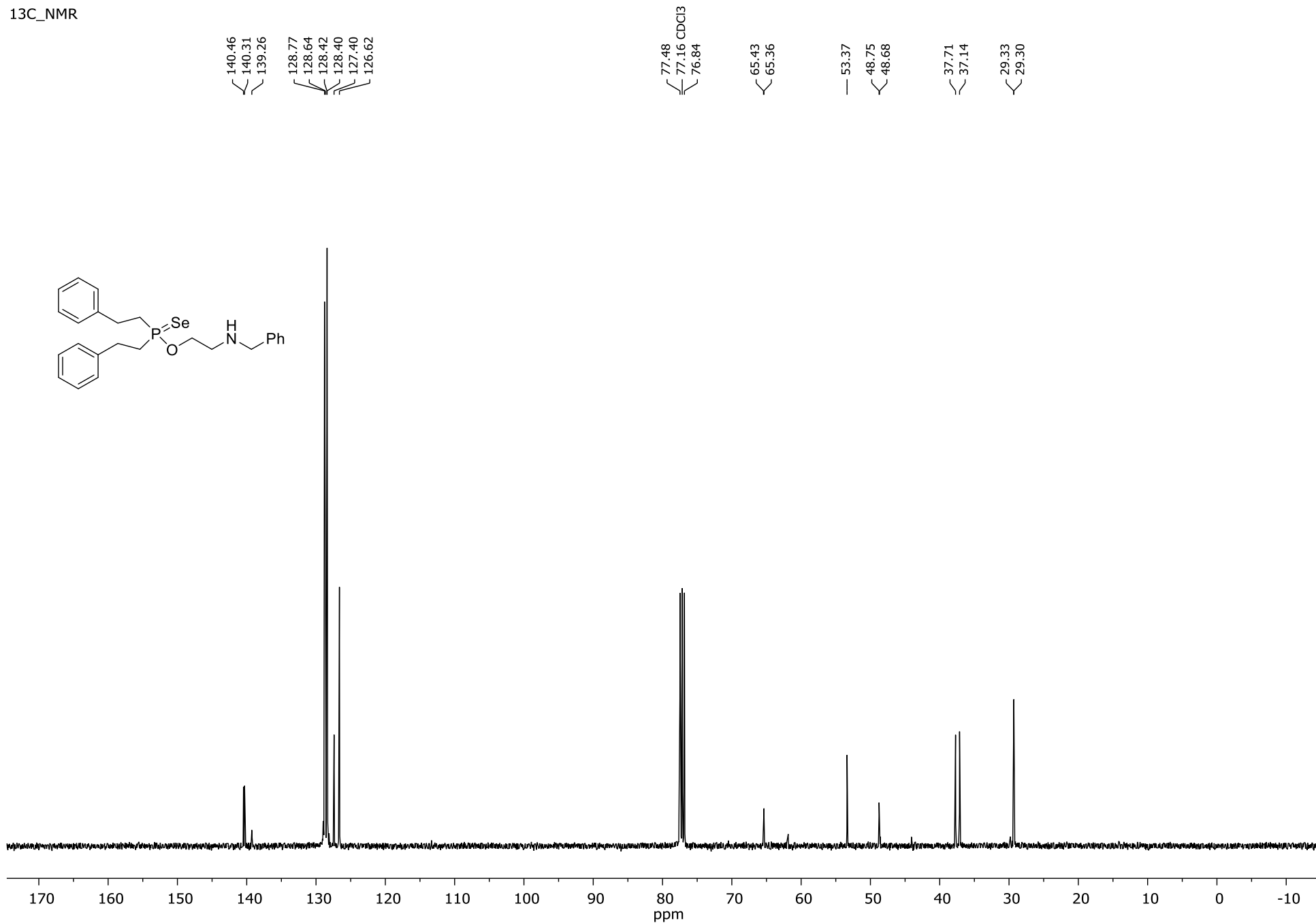
7.38
7.36
7.34
7.32
7.30
7.29
7.26 CDCl₃
7.24
7.22
7.20

4.15
4.14
4.13
4.12
4.10
3.86
3.03
3.00
2.99
2.97
2.95
2.93
2.89
2.88
2.87
2.46
2.45
2.42
2.40
2.38
2.36



***O*-[2-(benzylamino)ethyl]-bis(2-phenylethyl)phosphinoselenoate (9b)**

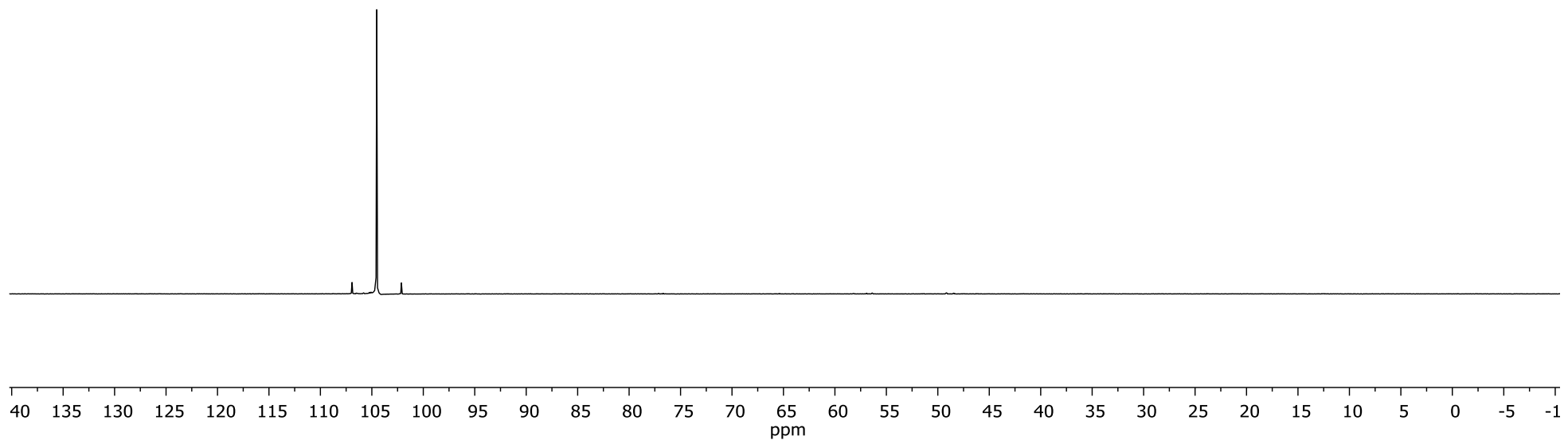
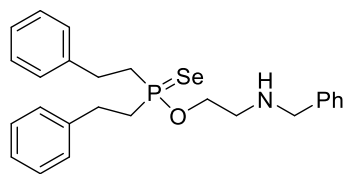
¹³C_NMR



***O*-[2-(benzylamino)ethyl]-bis(2-phenylethyl)phosphinoselenoate (9b)**

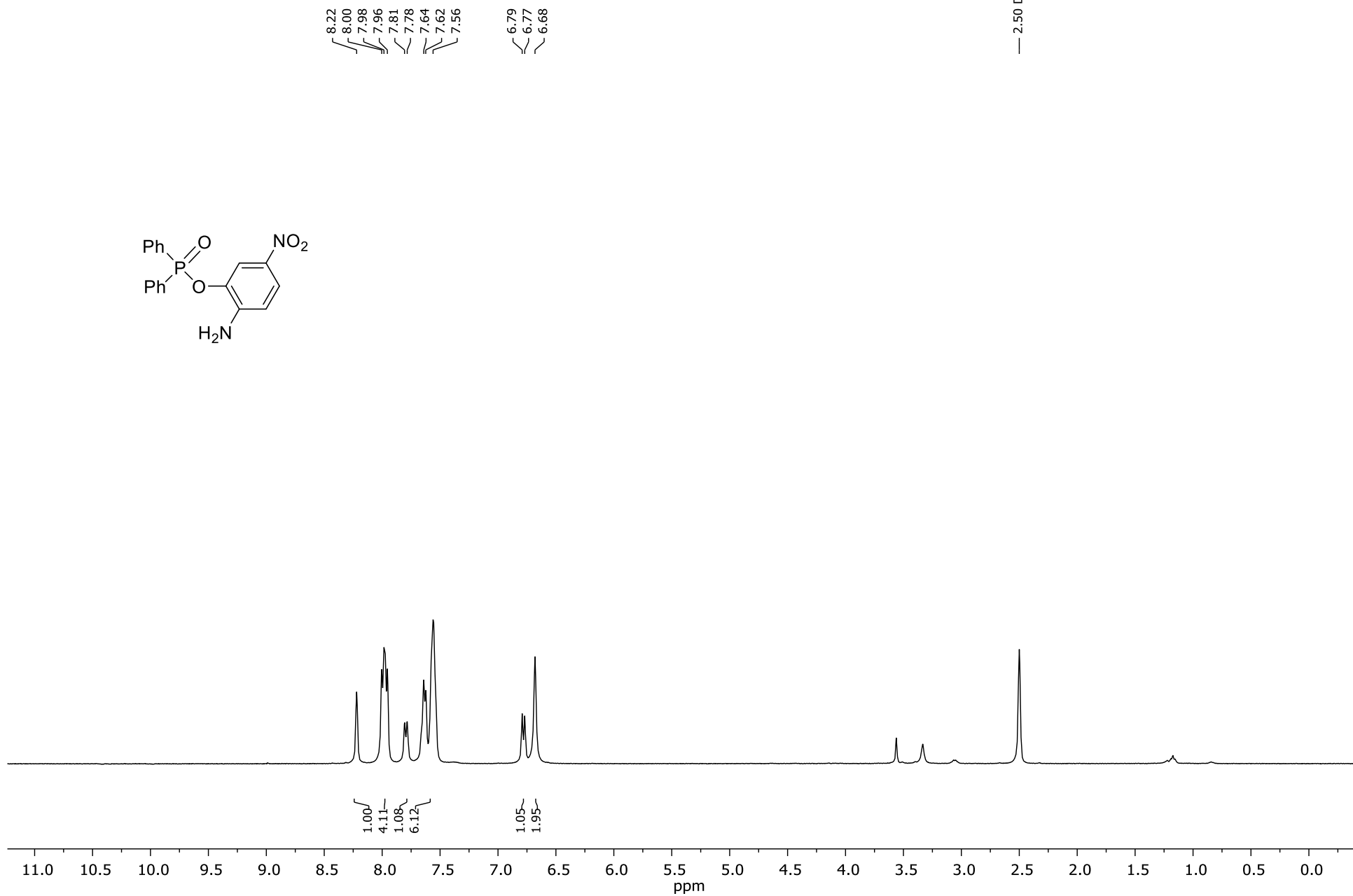
31P_NMR

106.92
104.53
102.13



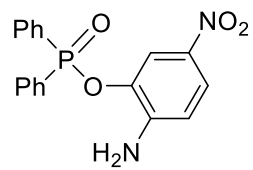
2-amino-5-nitrophenyl diphenylphosphinate (12a)

¹H_NMR



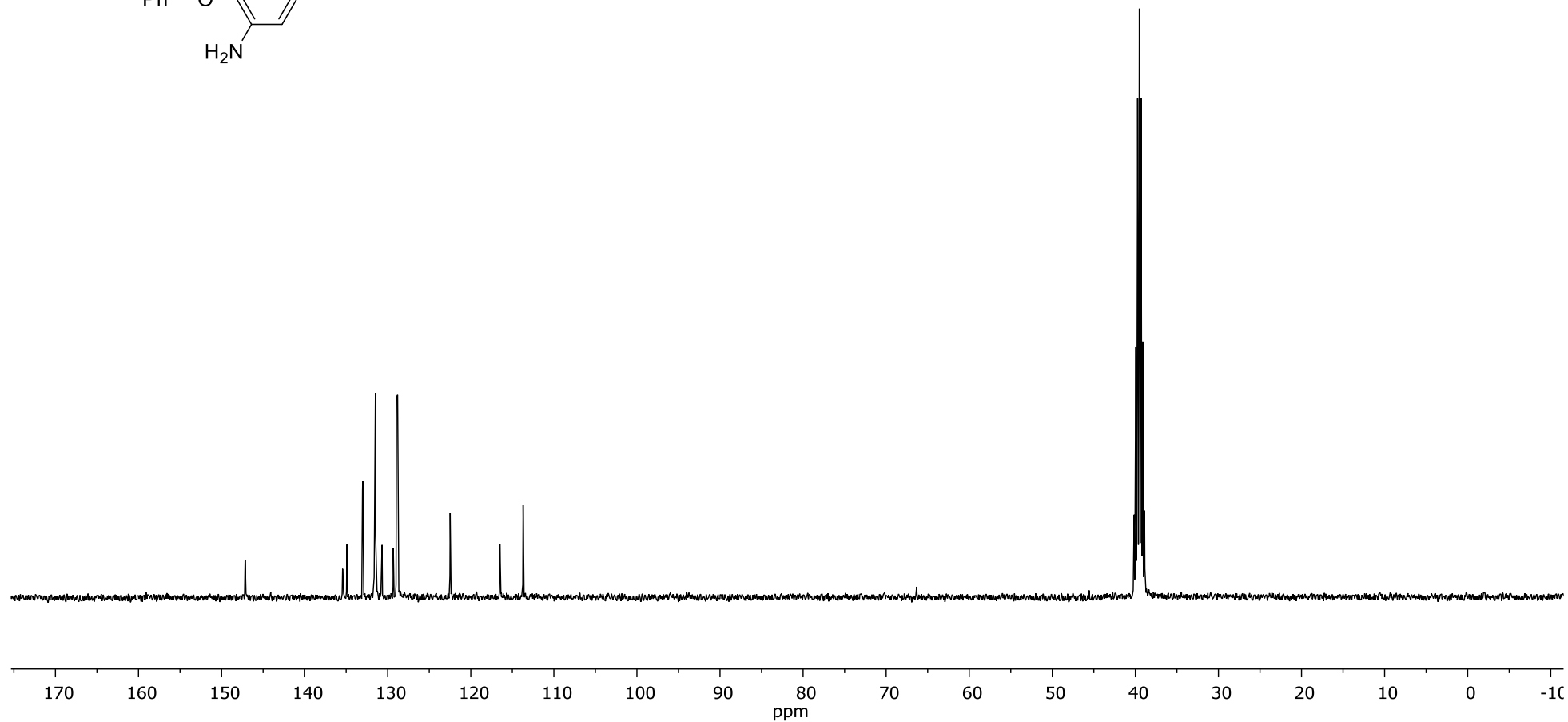
2-amino-5-nitrophenyl diphenylphosphinate (12a)

¹³C-NMR



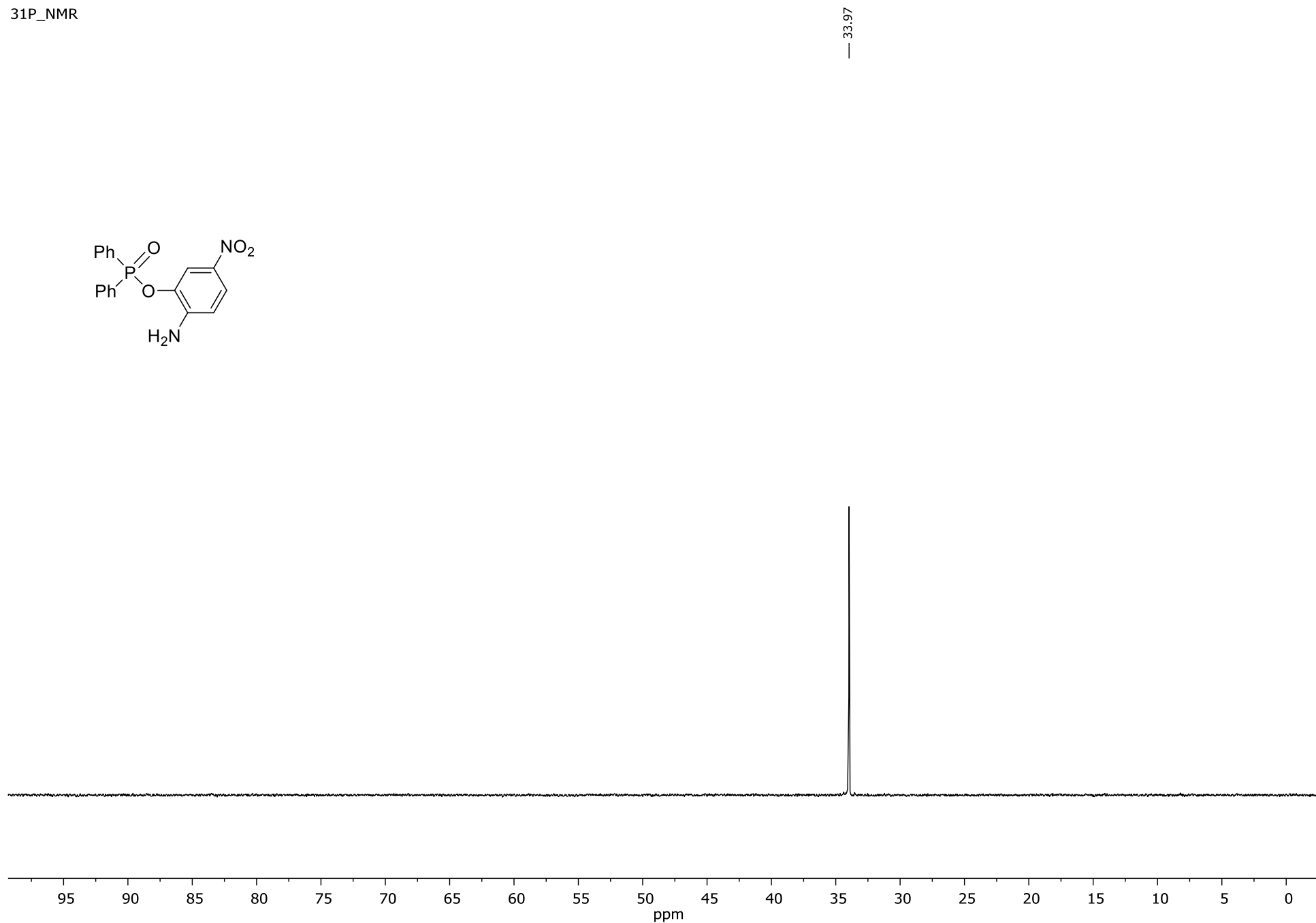
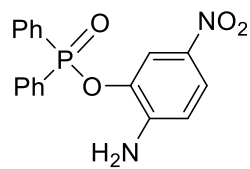
147.16
147.12
135.43
135.35
134.91
133.03
133.01
131.59
131.48
130.68
129.33
128.94
128.81
— 122.48
116.50
116.46
113.70

39.94
39.73
39.52 DMSO-d6
39.31
39.10



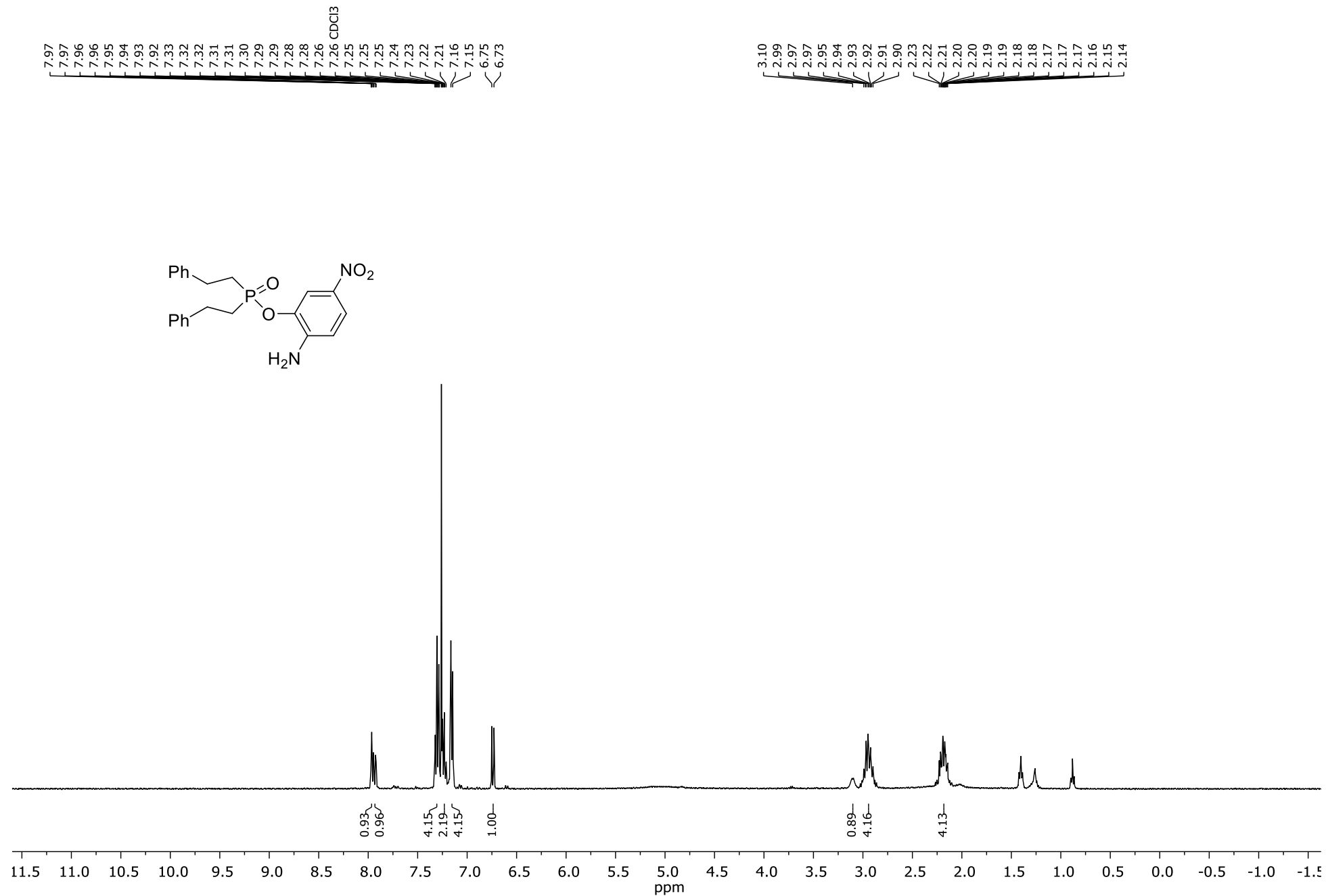
2-amino-5-nitrophenyl diphenylphosphinate (12a)

31P_NMR



2-amino-5-nitrophenyl bis(2-phenylethyl)phosphinate (12b)

¹H_NMR



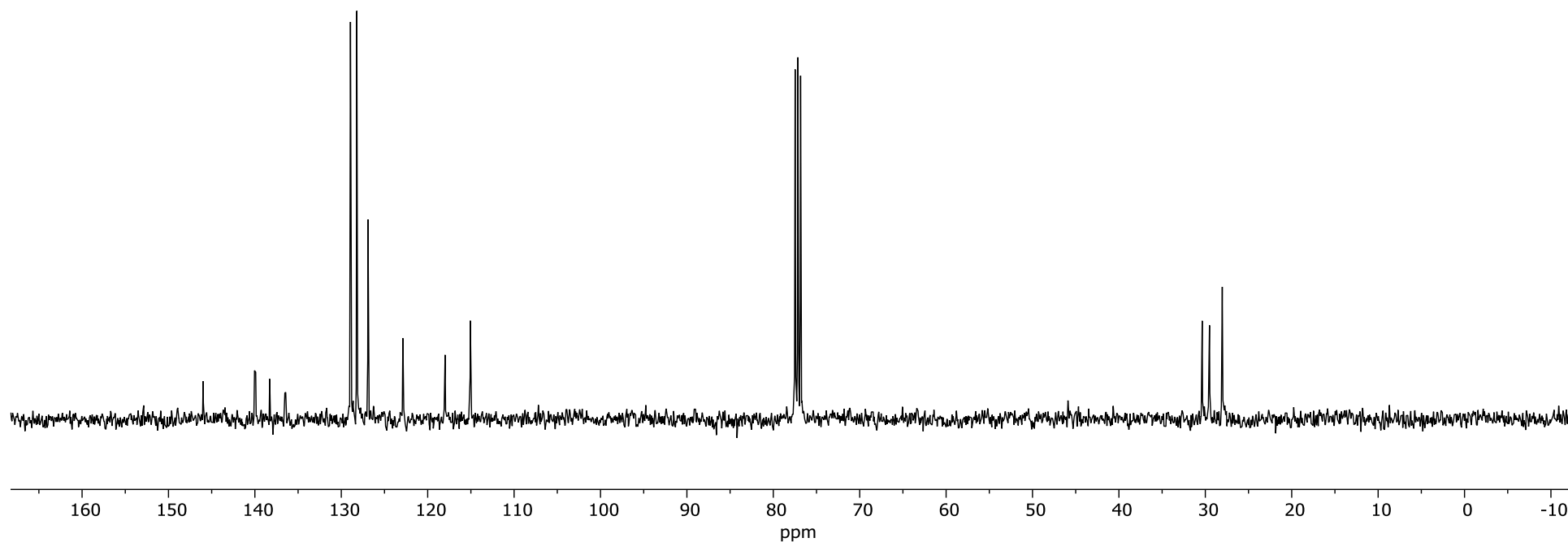
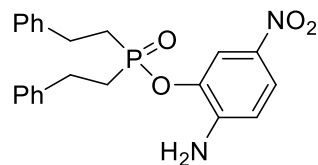
2-amino-5-nitrophenyl bis(2-phenylethyl)phosphinate (12b)

¹³C_NMR

145.99
140.04
139.90
138.29
136.43
128.94
128.20
126.91
122.87
118.00
115.06

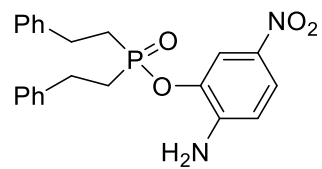
77.48
77.16 CDCl₃
76.84

30.36
29.52
28.05

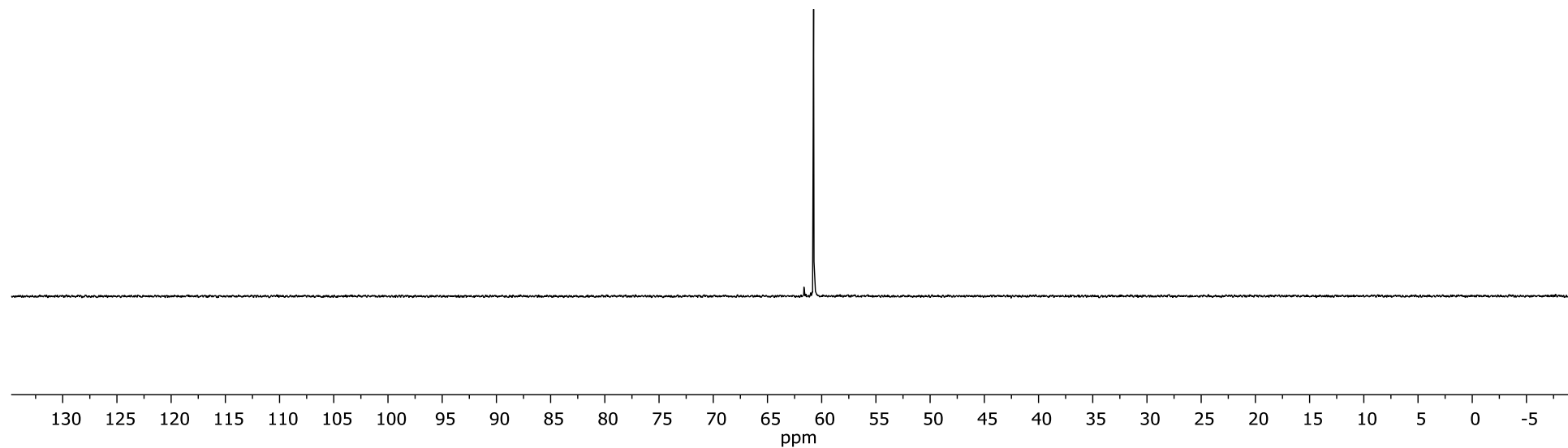


2-amino-5-nitrophenyl bis(2-phenylethyl)phosphinate (12b)

³¹P_NMR

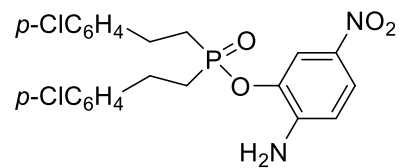


— 60.74



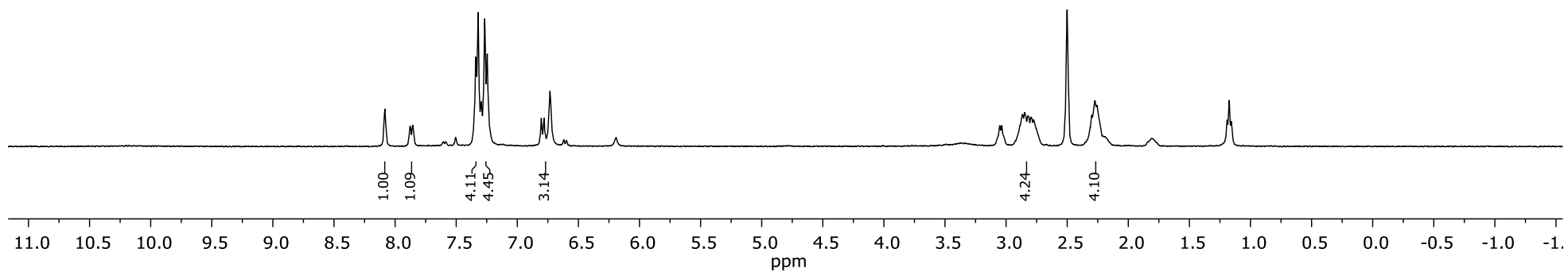
2-amino-5-nitrophenyl bis[2-(4-chlorophenyl)ethyl]phosphinate (12c)

¹H-NMR



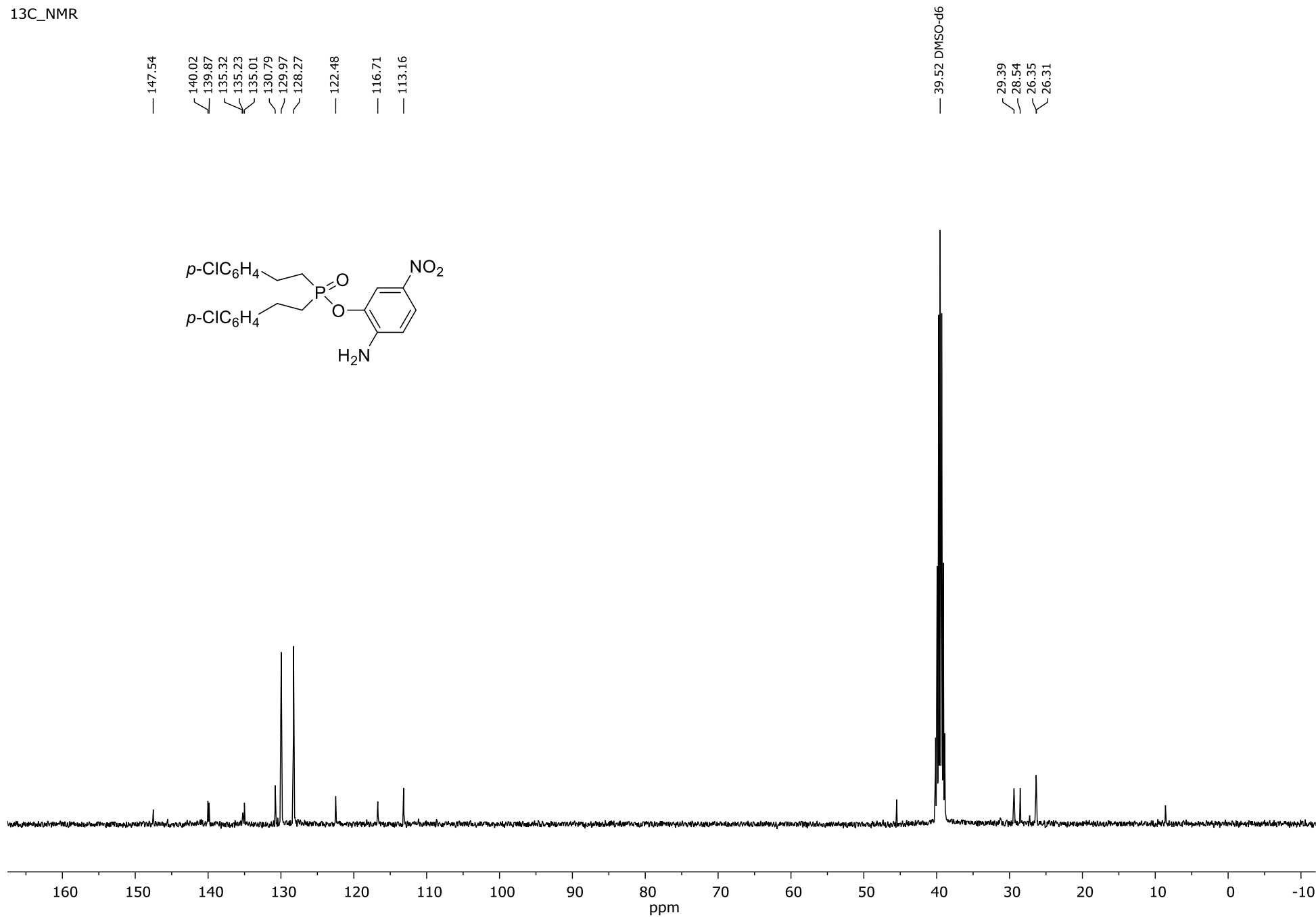
8.08
7.88
7.85
7.34
7.32
7.27
7.25
6.80
6.78
6.73

2.89
2.87
2.86
2.82
2.79
2.77
2.76
2.50 DMSO-d6
2.32
2.30
2.27
2.26
2.24



2-amino-5-nitrophenyl bis[2-(4-chlorophenyl)ethyl]phosphinate (12c)

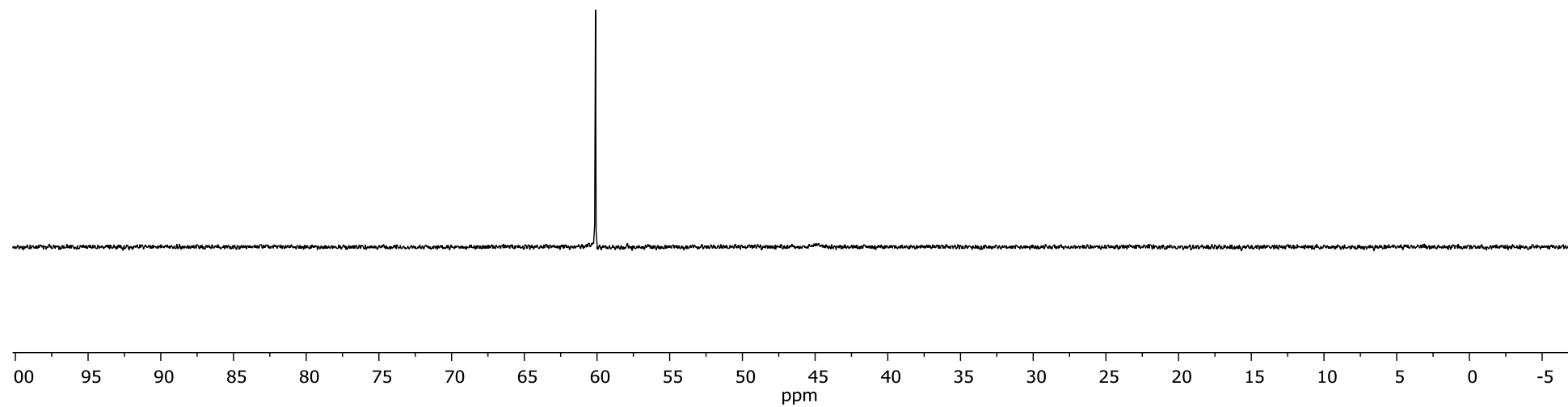
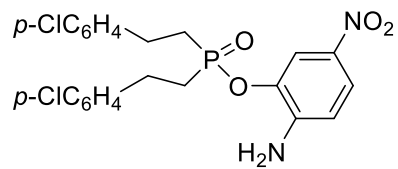
¹³C_NMR



2-amino-5-nitrophenyl bis[2-(4-chlorophenyl)ethyl]phosphinate (12c)

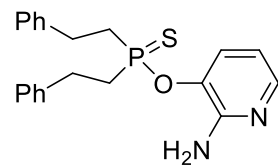
³¹P-NMR

60.10



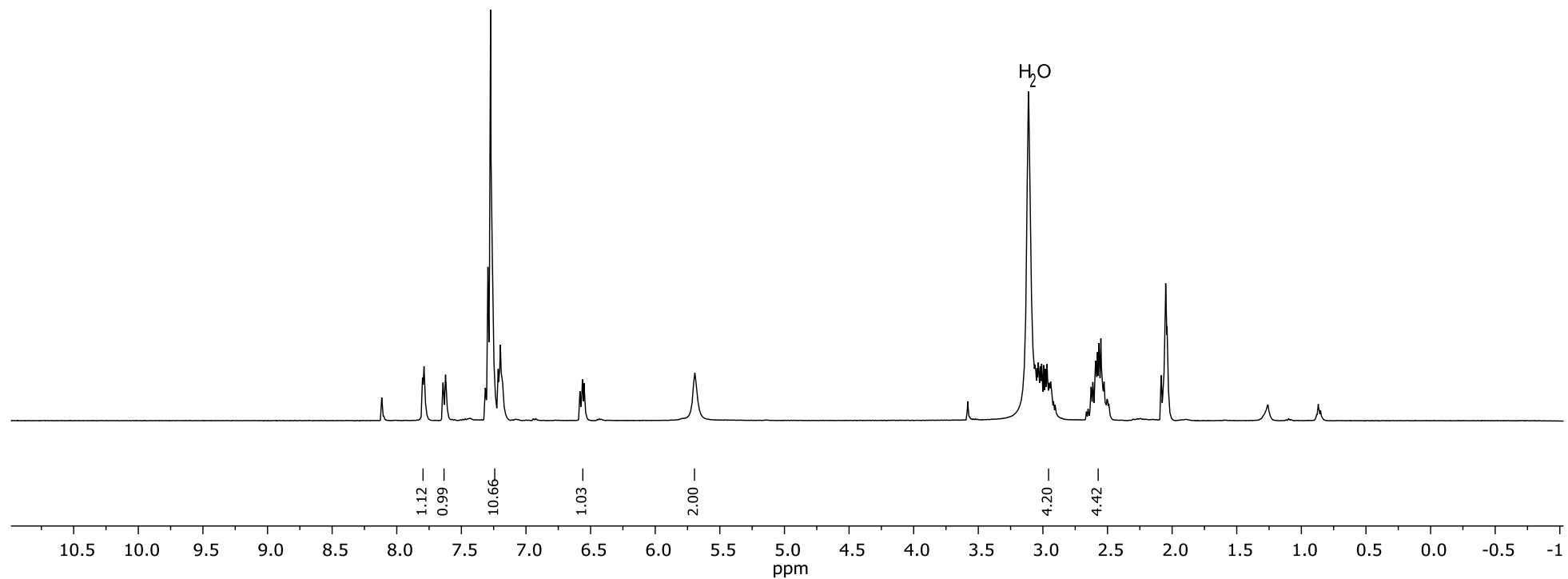
***O*-(2-aminopyridin-3-yl) bis(2-phenylethyl)phosphinothioate (12d)**

¹H_NMR



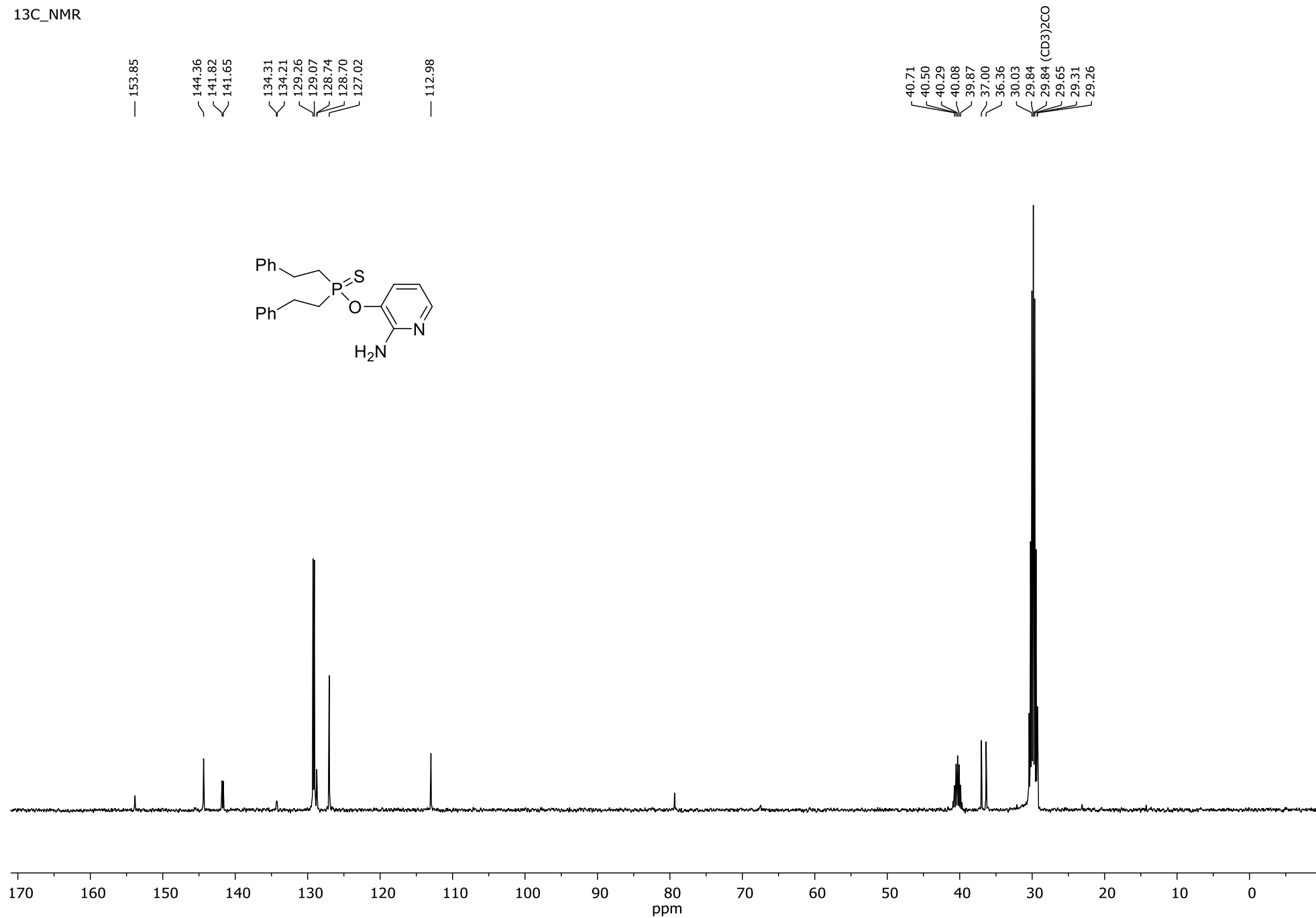
8.12
7.80
7.79
7.64
7.62
7.31
7.29
7.28
7.26
7.22
7.20
7.18
6.58
6.57
6.56
6.55
5.69

3.11
3.05
3.02
2.98
2.95
2.93
2.63
2.59
2.58
2.55
2.54
2.53
2.05 (CD₃)₂CO
2.04



***O*-(2-aminopyridin-3-yl) bis(2-phenylethyl)phosphinothioate (12d)**

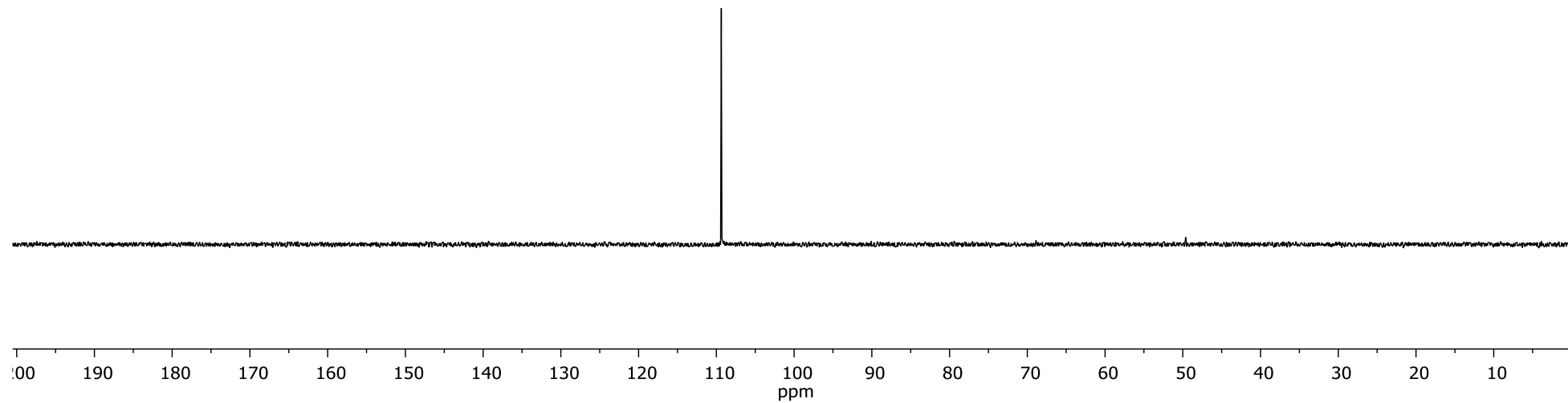
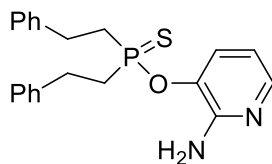
¹³C_NMR



***O*-(2-aminopyridin-3-yl) bis(2-phenylethyl)phosphinothioate (12d)**

³¹P-NMR

— 109.36



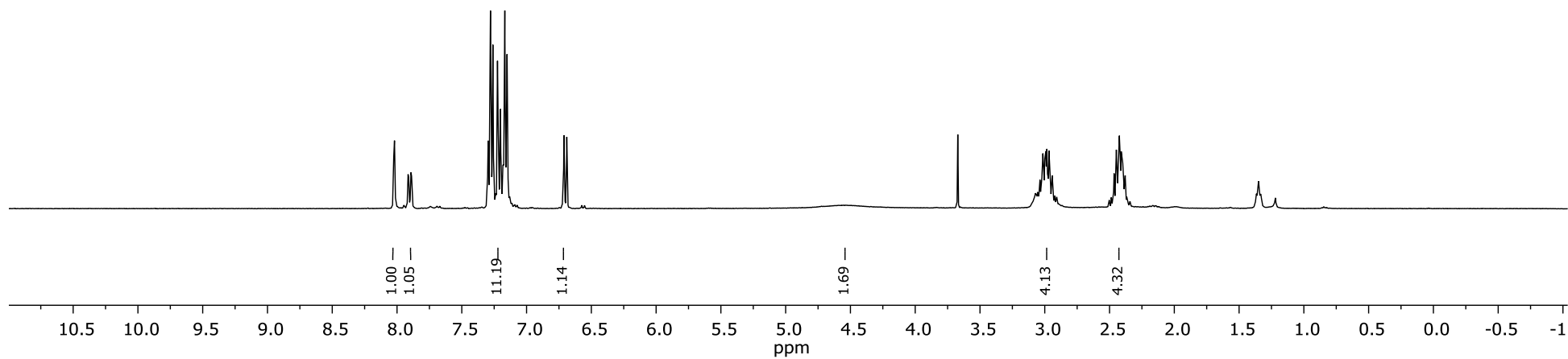
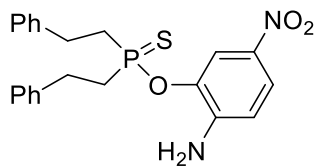
***O*-(2-amino-5-nitrophenyl) bis(2-phenylethyl)phosphinothioate (12e)**

1H_NMR

8.03
8.02
8.02
7.91
7.91
7.89
7.89
7.30
7.28
7.26 CDCl3
7.26
7.22
7.22
7.20
7.17
7.15
7.15
6.71
6.69

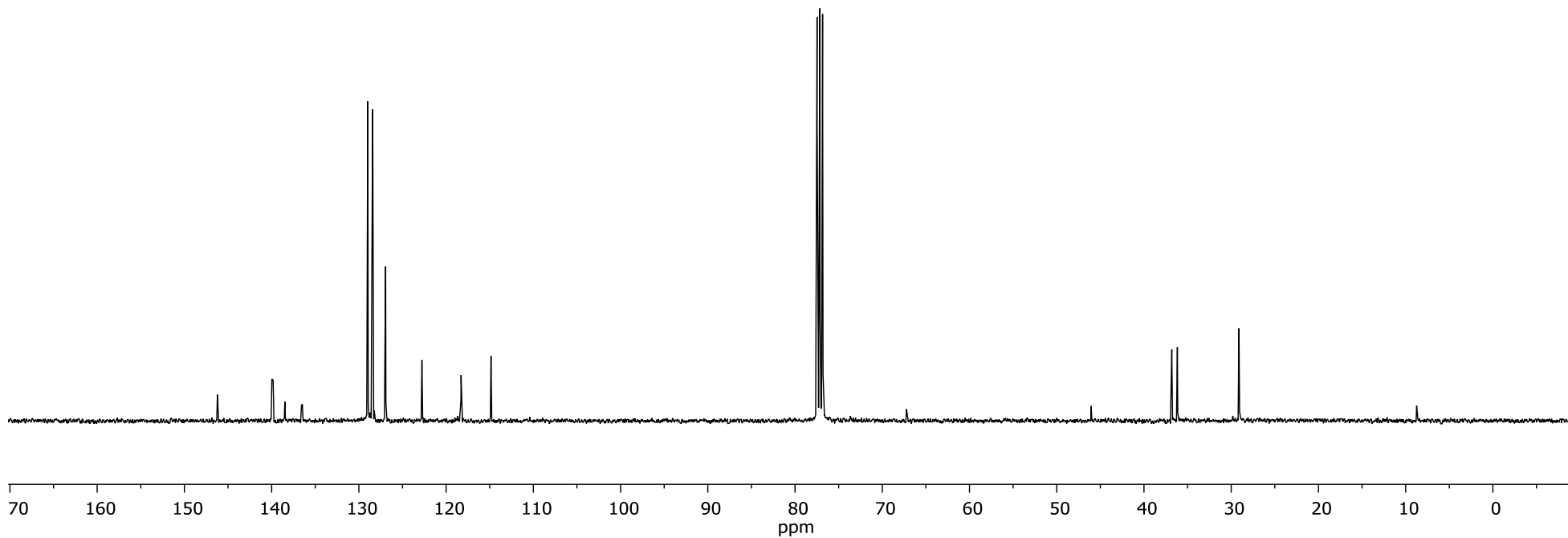
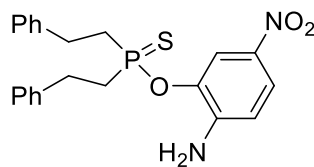
— 4.57

3.04
3.01
3.00
2.99
2.98
2.97
2.94
2.46
2.45
2.45
2.42
2.40
2.39
2.38



***O*-(2-amino-5-nitrophenyl) bis(2-phenylethyl)phosphinothioate (12e)**

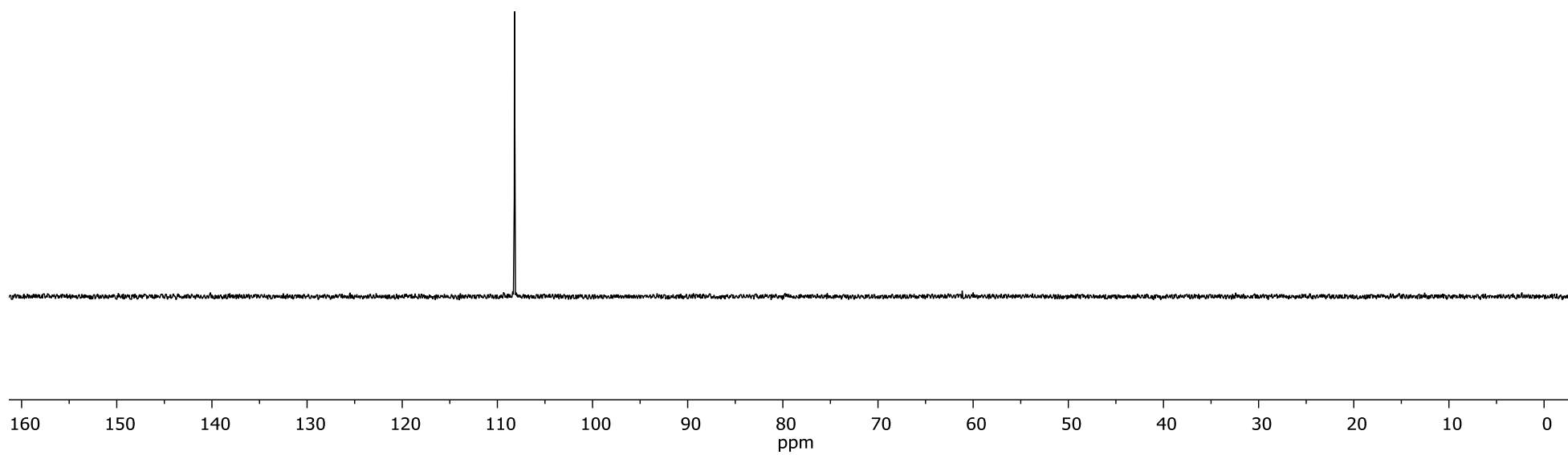
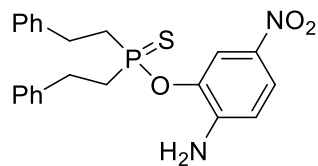
¹³C_NMR



***O*-(2-amino-5-nitrophenyl) bis(2-phenylethyl)phosphinothioate (12e)**

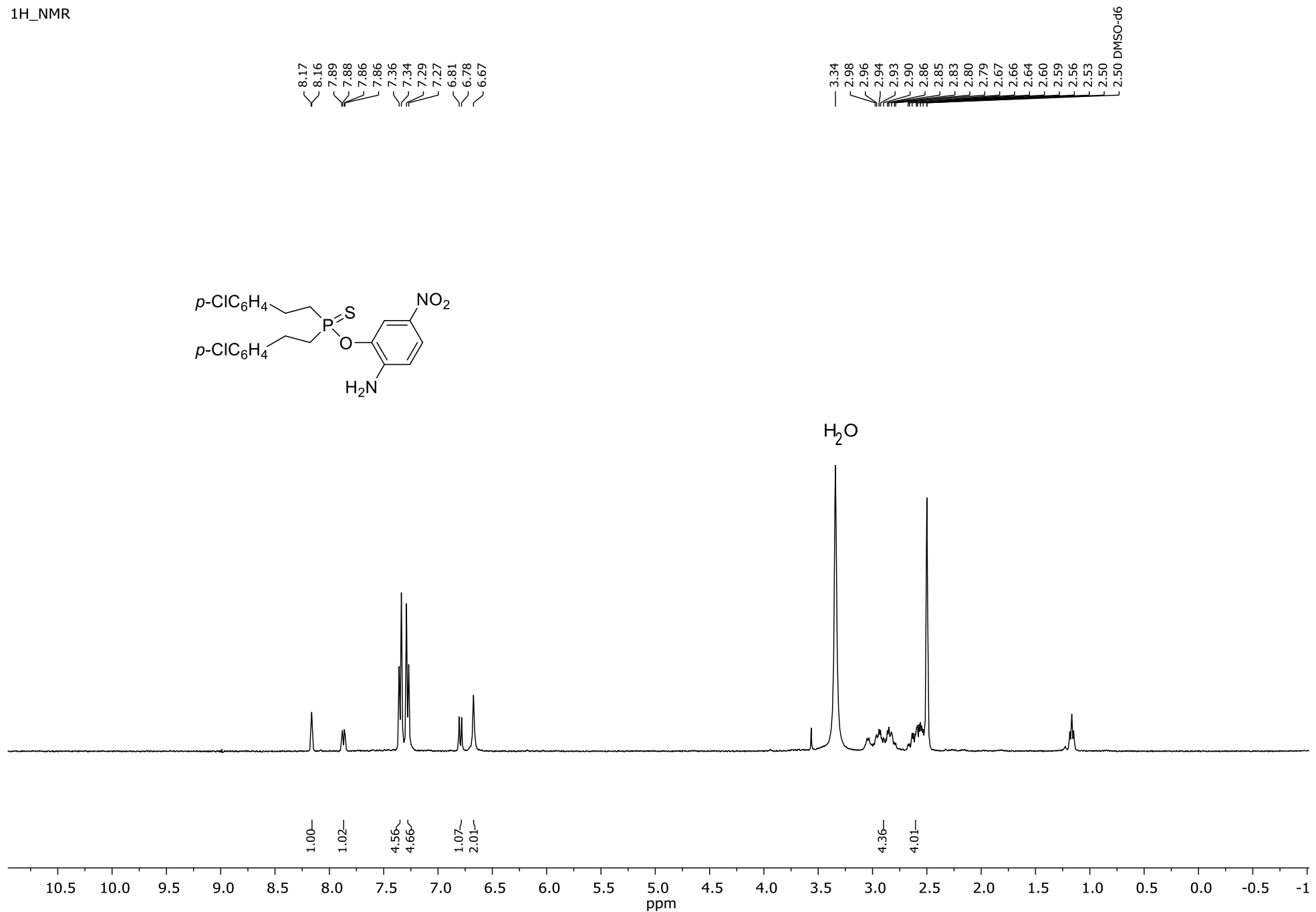
³¹P_NMR

— 108.21



***O*-(2-amino-5-nitrophenyl) bis[2-(4-chlorophenyl)ethyl]phosphinothioate (12f)**

1H_NMR



***O*-[2-amino-5-nitrophenyl] bis[2-(4-chlorophenyl)ethyl]phosphinothioate (12f)**

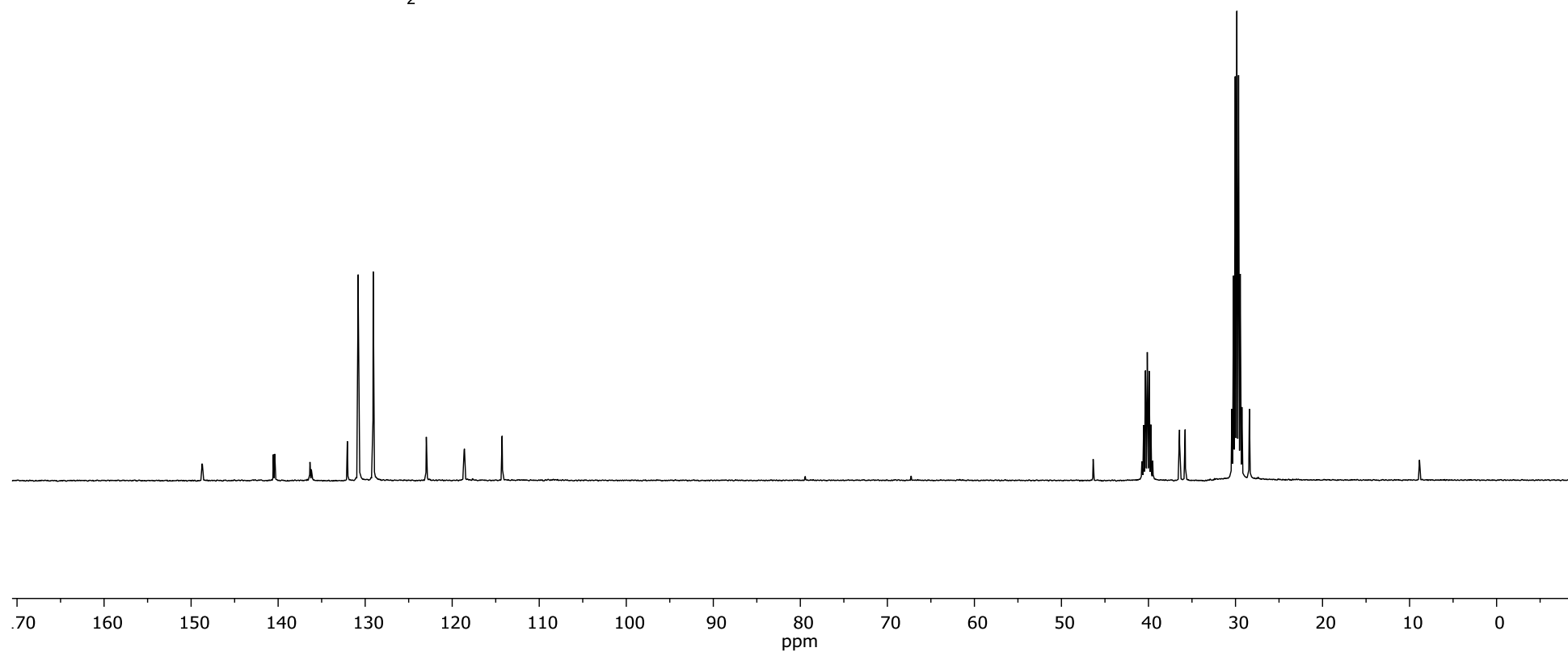
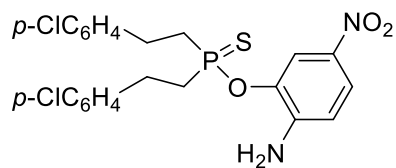
¹³C_NMR

148.74
148.71

140.55
140.39
136.34
136.19
136.09
132.05
130.80
129.08

122.97
118.63
118.59
114.26

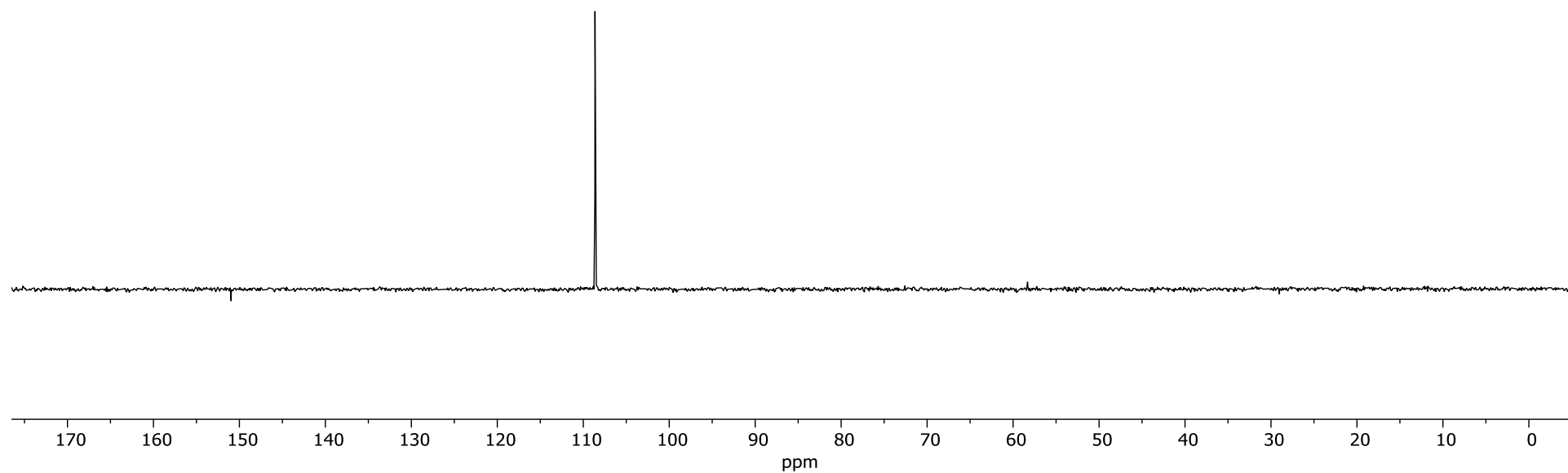
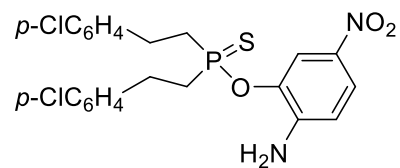
40.55
40.34
40.13
39.93
39.72
36.43
35.78
30.42
30.23
30.03
29.84
29.84 (CD₃)₂CO
29.65
29.46
29.26
28.42
28.40



***O*-(2-amino-5-nitrophenyl) bis[2-(4-chlorophenyl)ethyl]phosphinothioate (12f)**

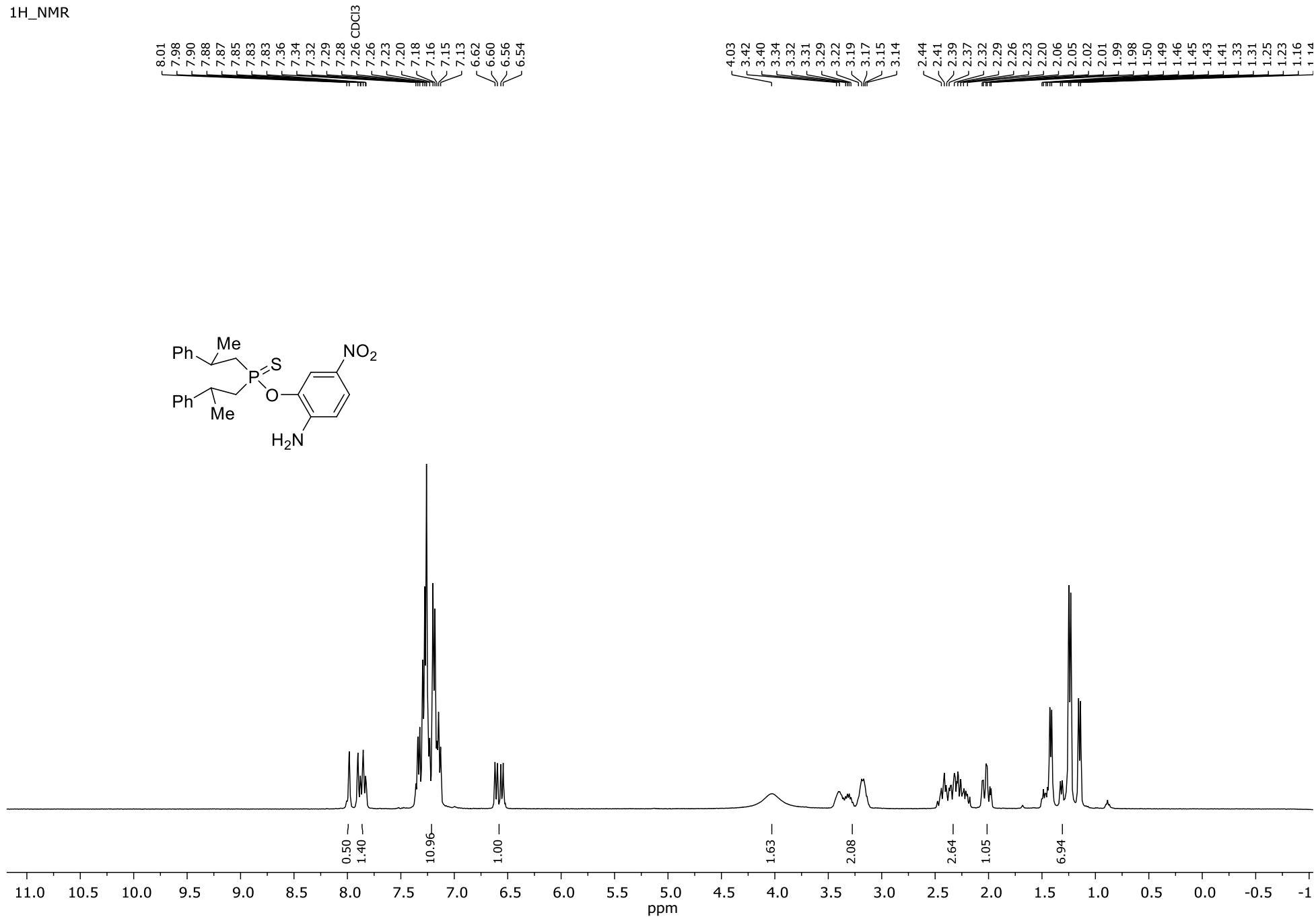
31P_NMR

— 108.63



***O*-(2-amino-5-nitrophenyl) bis(2-phenylpropyl)phosphinothioate (12g)**

¹H_NMR



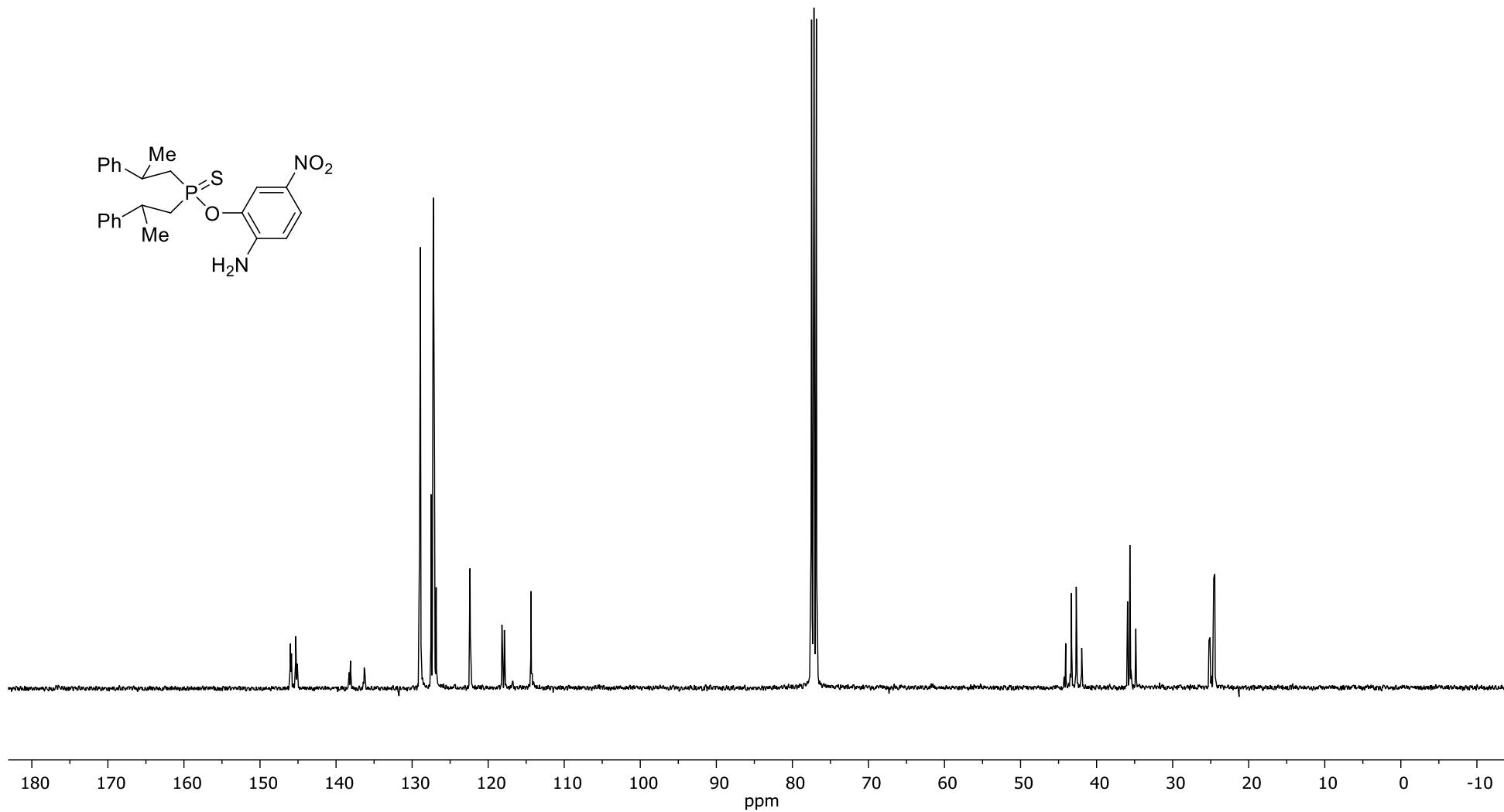
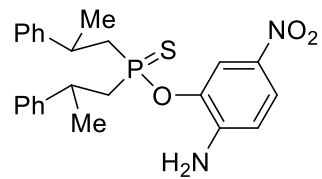
O-(2-amino-5-nitrophenyl) bis(2-phenylpropyl)phosphinothioate (12g)

¹³C_NMR

146.01
145.95
145.90
145.86
145.30
145.24
145.14
145.10
138.25
138.10
136.40
136.30
136.22
136.12
129.01
128.91
128.87
127.50
127.30
127.22
127.05
126.83
122.39
122.37
118.19
118.15
117.87
117.83
114.36
114.24
114.03

77.48
77.16 CDCl₃
76.84

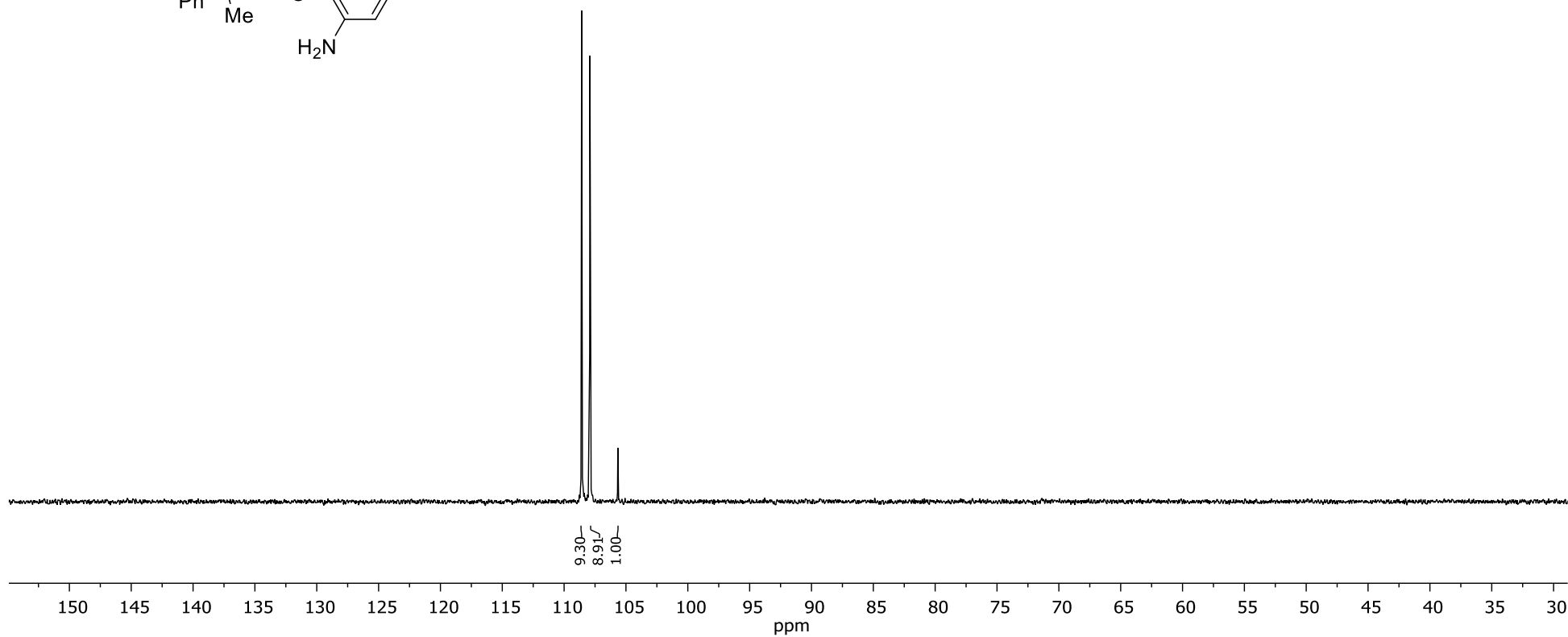
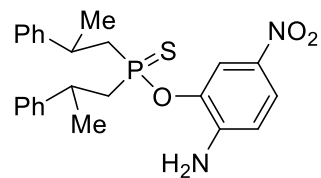
44.28
44.06
43.63
43.44
43.30
42.67
42.62
41.96
35.93
35.62
35.60
35.47
35.44
34.84
34.80
25.23
25.09
24.93
24.64
24.60
24.49
24.46



***O*-(2-amino-5-nitrophenyl) bis(2-phenylpropyl)phosphinothioate (12g)**

31P_NMR

108.58
107.92
105.65

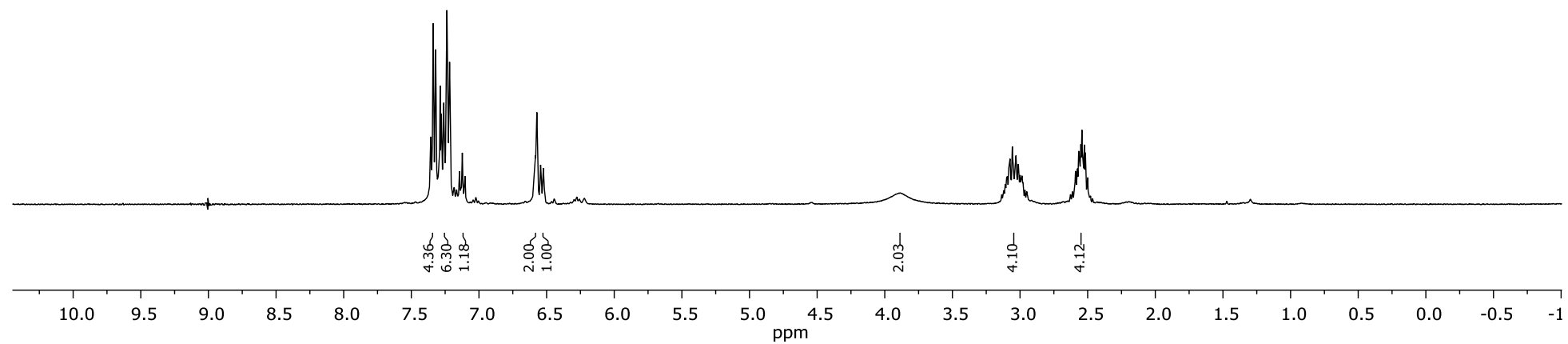
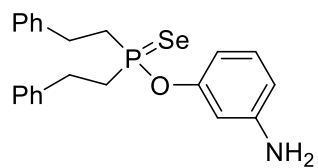


***O*-(3-aminophenyl) bis(2-phenylethyl)phosphinoselenoate (12h)**

1H_NMR

7.36
7.34
7.32
7.29
7.28
7.26 CDCl3
7.24
7.22
7.14
7.12
7.10
6.59
6.57
6.54
6.52

3.89
3.14
3.11
3.09
3.07
3.06
3.03
3.01
2.98
2.95
2.61
2.59
2.57
2.54
2.52
2.50
2.49



***O*-(3-aminophenyl) bis(2-phenylethyl)phosphinoselenoate (12h)**

¹³C_NMR

151.92
151.82
147.80

140.36
140.20

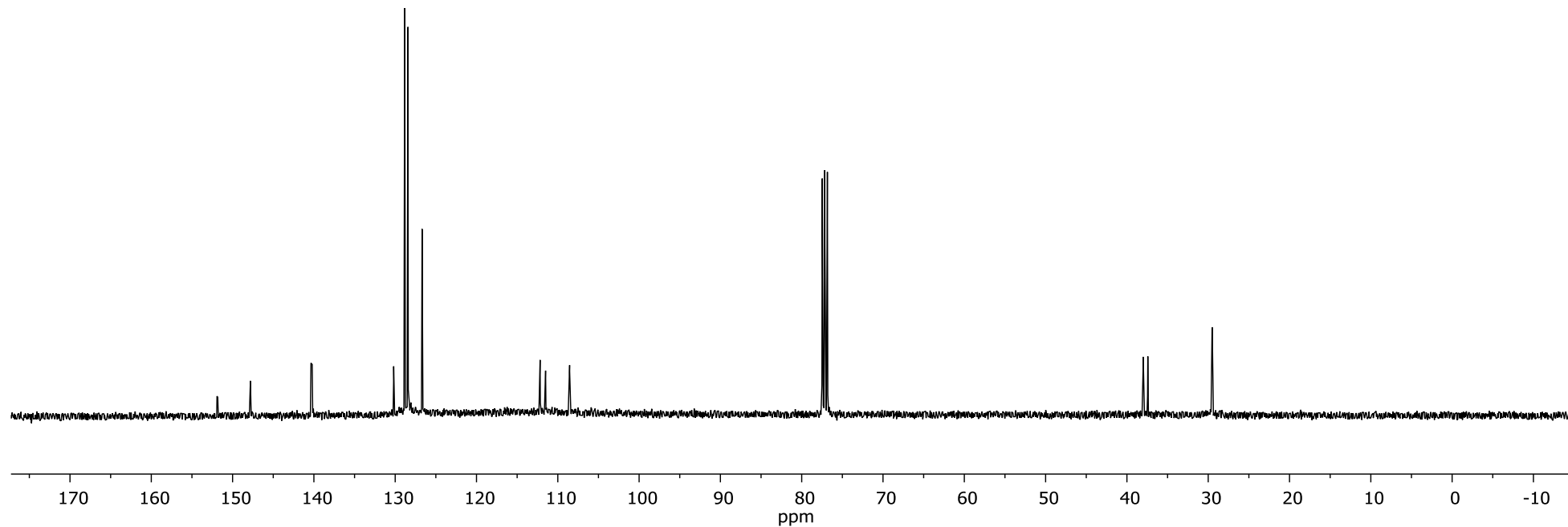
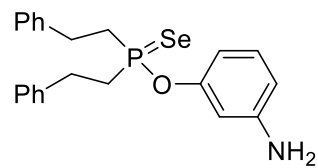
130.18
128.83
128.44
126.69

112.18
111.55
111.50
108.59
108.55

77.48
77.16 CDCl₃
76.84

37.99
37.43

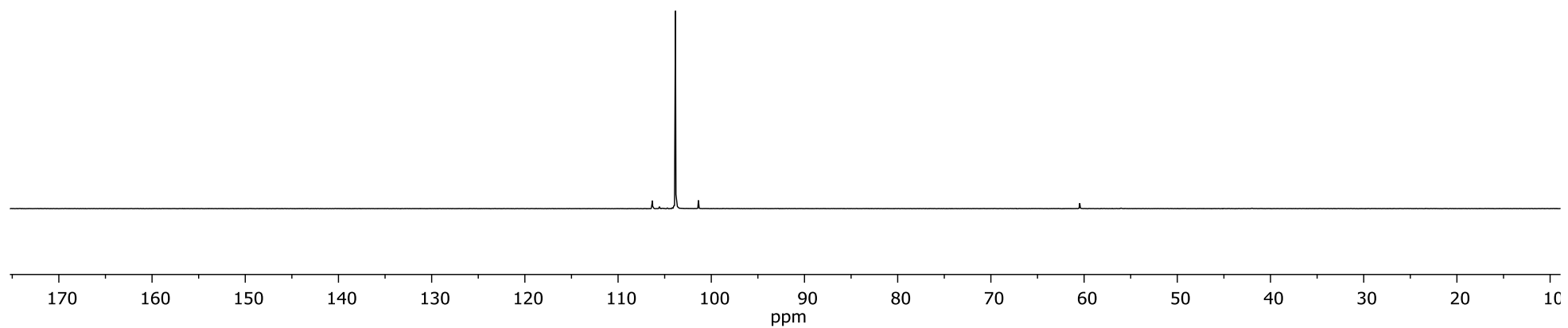
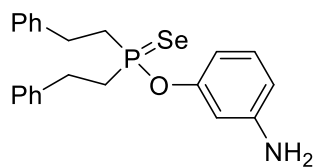
29.53
29.50



***O*-(3-aminophenyl) bis(2-phenylethyl)phosphinoselenoate (12h)**

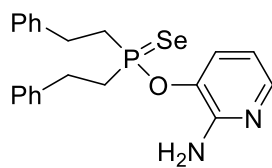
³¹P_NMR

106.31
103.84
101.38



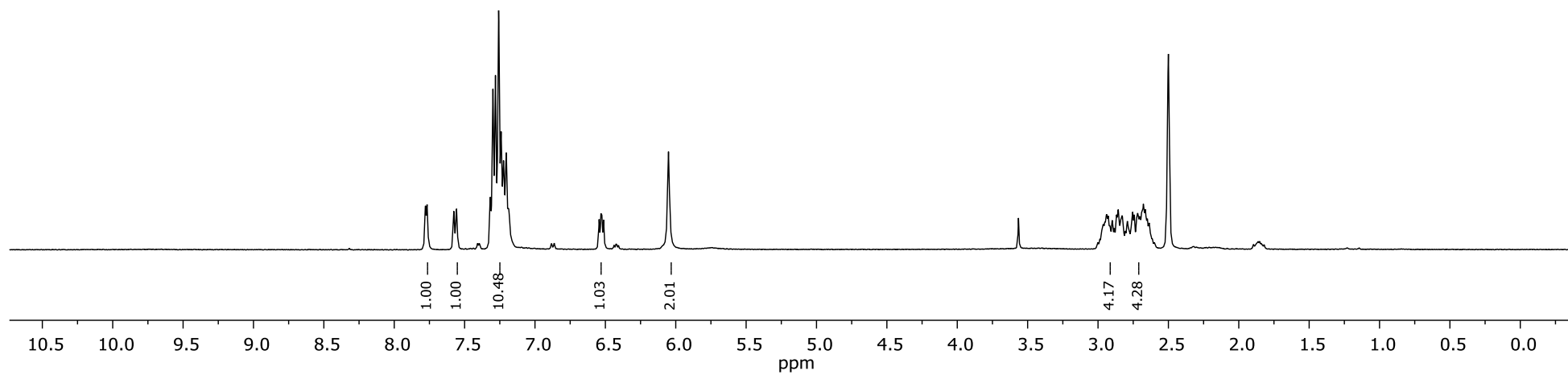
***O*-(2-aminopyridin-3-yl) bis(2-phenylethyl)phosphinoselenoate (12i)**

¹H-NMR



7.78
7.77
7.58
7.56
7.32
7.30
7.28
7.26
7.24
7.22
7.20
7.19
6.54
6.53
6.52
6.51
— 6.05

3.00
2.96
2.93
2.91
2.86
2.83
2.79
2.75
2.74
2.72
2.71
2.68
2.66
2.65
2.64
2.50 DMSO-d6

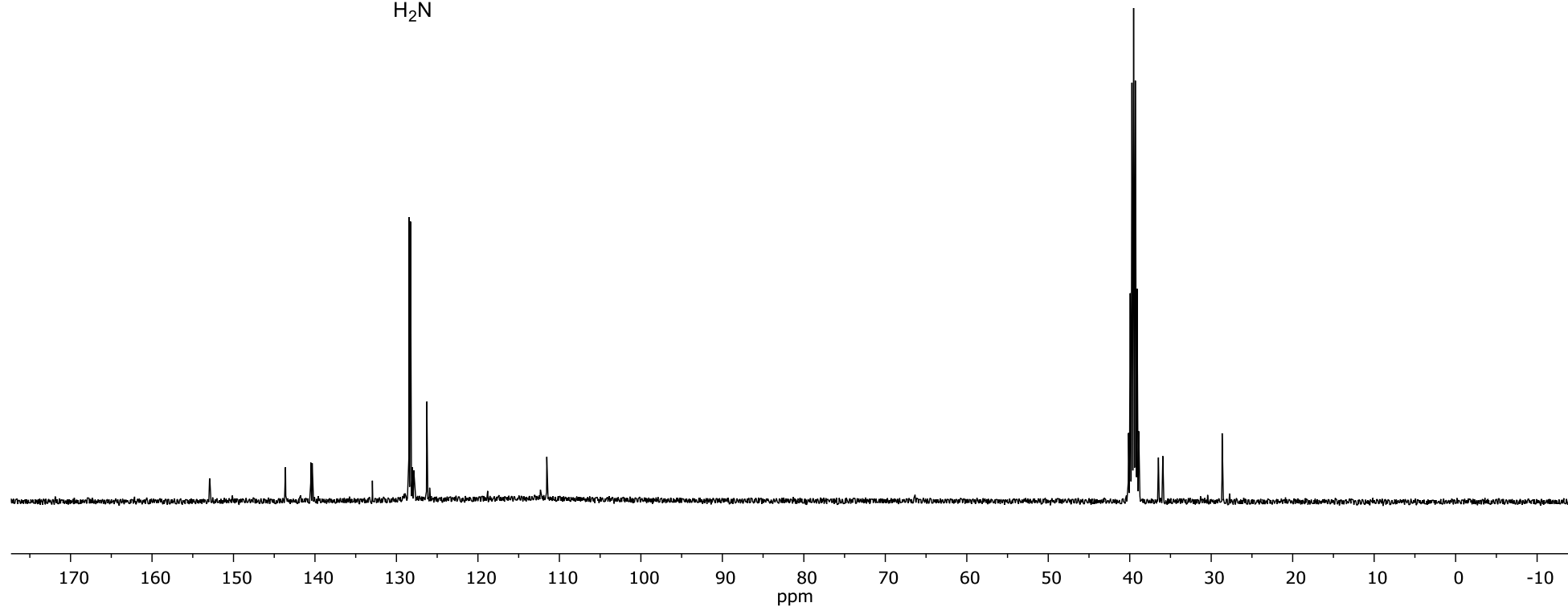
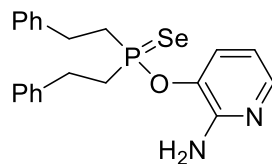


***O*-(2-aminopyridin-3-yl) bis(2-phenylethyl)phosphinoselenoate (12i)**

¹³C-NMR

— 152.94
— 143.62
— 140.51
— 140.34
— 132.95
— 132.85
— 128.46
— 128.27
— 127.88
— 127.83
— 126.27
— 111.55

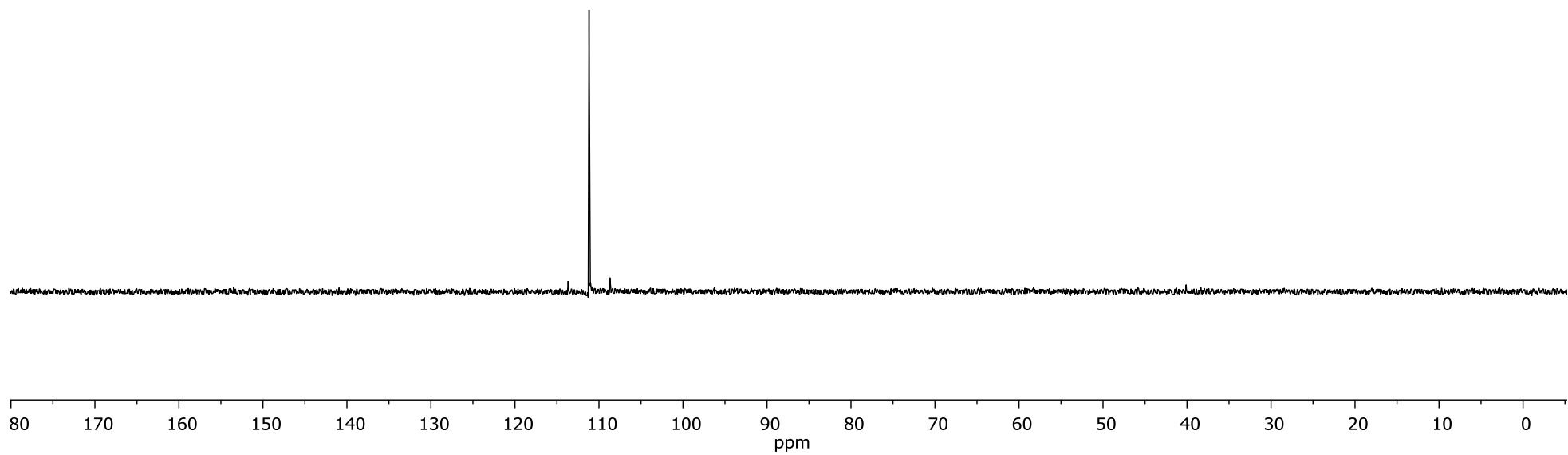
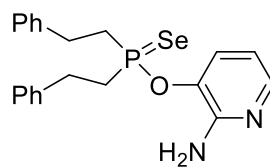
39.94
39.73
39.52 DMSO-d6
39.31
39.10
36.48
35.94
28.66
28.64



***O*-(2-aminopyridin-3-yl) bis(2-phenylethyl)phosphinoselenoate (12i)**

31P_NMR

113.67
111.18
108.70

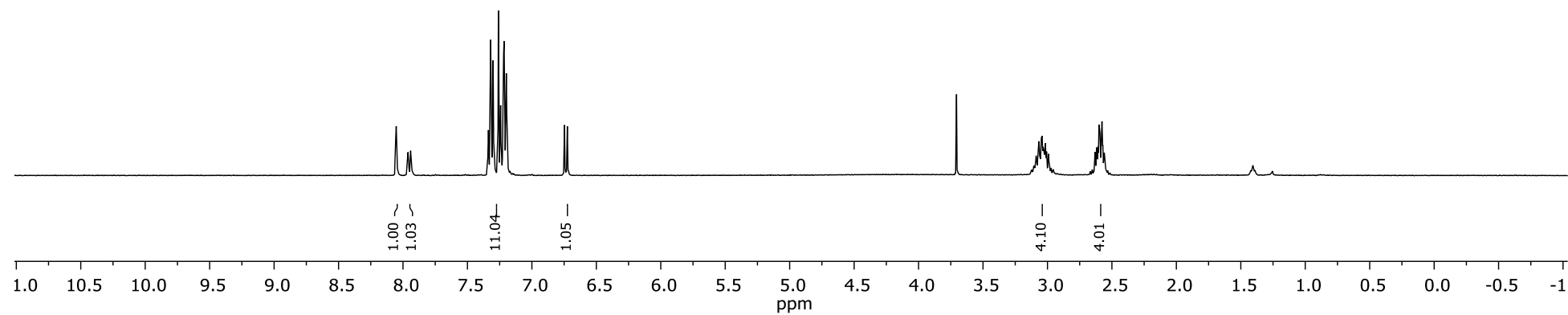
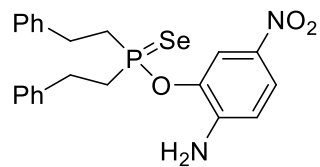


***O*-(2-amino-5-nitrophenyl) bis(2-phenylethyl)phosphinoselenoate (12j)**

1H_NMR

8.06
8.05
8.05
7.96
7.96
7.96
7.94
7.94
7.93
7.93
7.34
7.32
7.30
7.26 CDCl3
7.24
7.22
7.22
7.20
6.75
6.72

3.29
3.09
3.07
3.07
3.05
3.04
3.02
3.02
3.01
2.99
2.63
2.62
2.61
2.61
2.60
2.59
2.58
2.57
2.56
2.55



***O*-(2-amino-5-nitrophenyl) bis(2-phenylethyl)phosphinoselenoate (12j)**

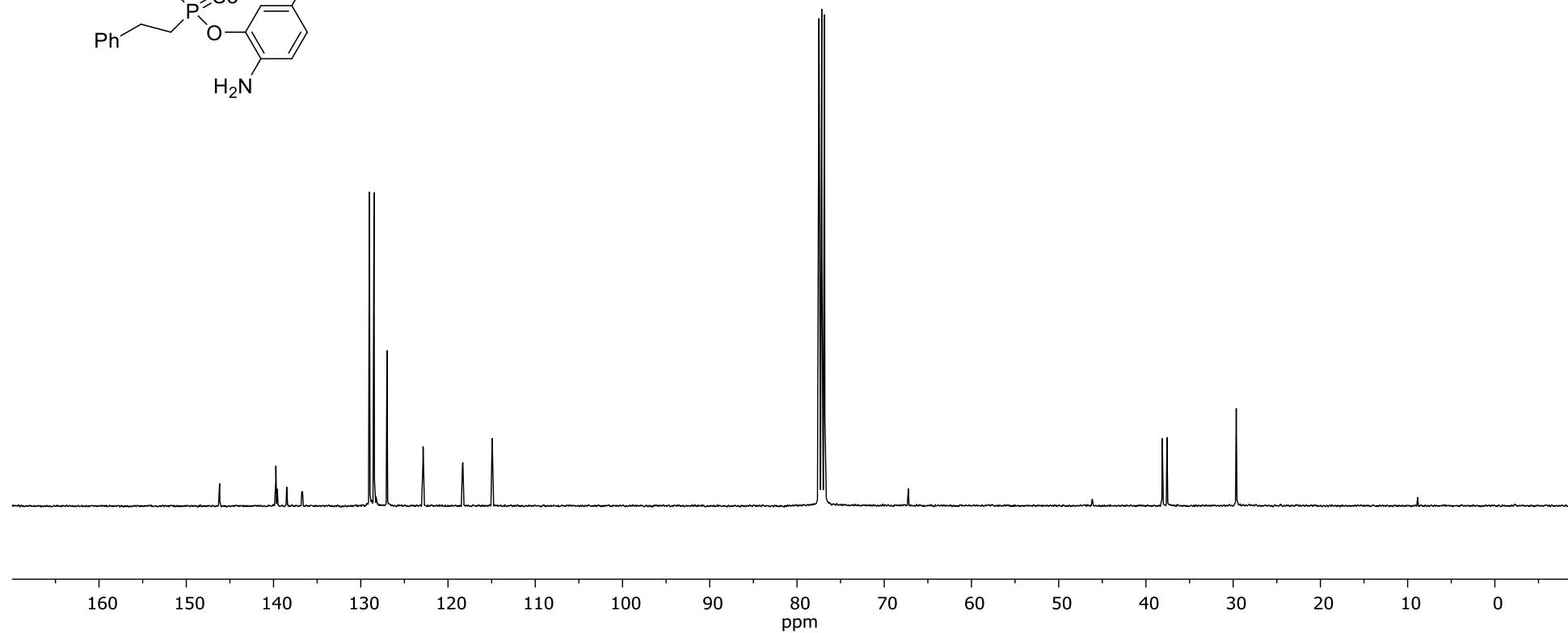
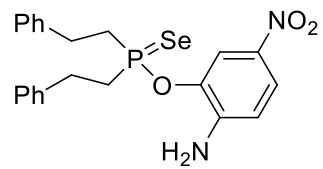
¹³C_NMR

146.21
146.18
139.73
139.58
138.49
136.77
136.67
129.01
128.48
127.00
— 122.86
118.34
118.30
114.94

77.48
77.16 CDCl₃
77.16
76.84

38.12
37.59

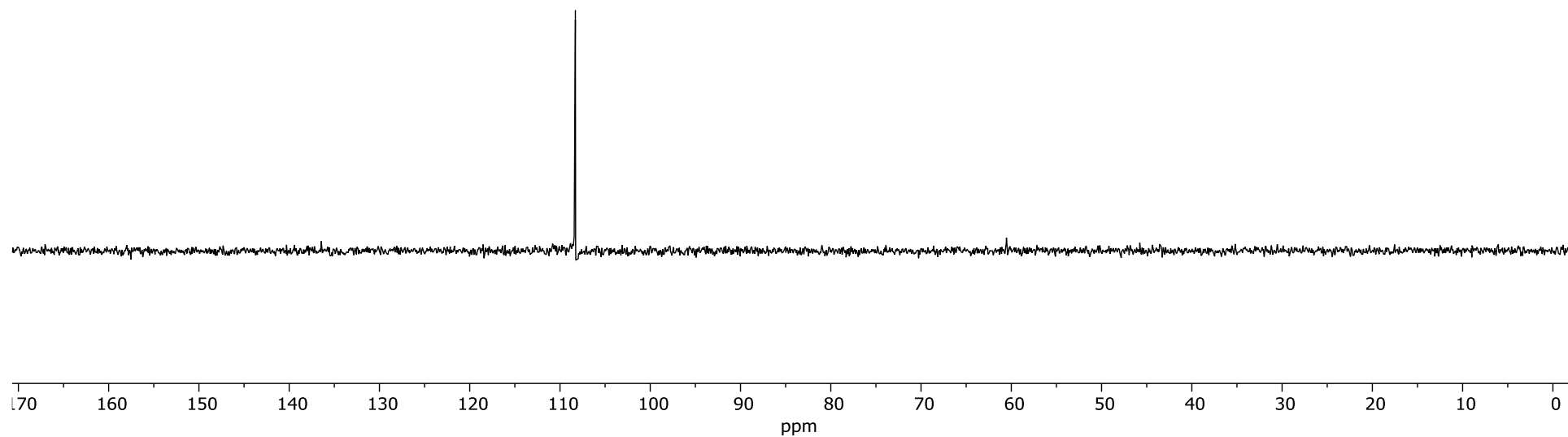
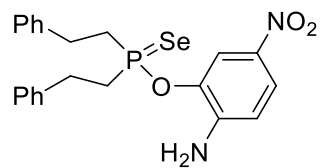
29.67
29.65



***O*-(2-amino-5-nitrophenyl) bis(2-phenylethyl)phosphinoselenoate (12j)**

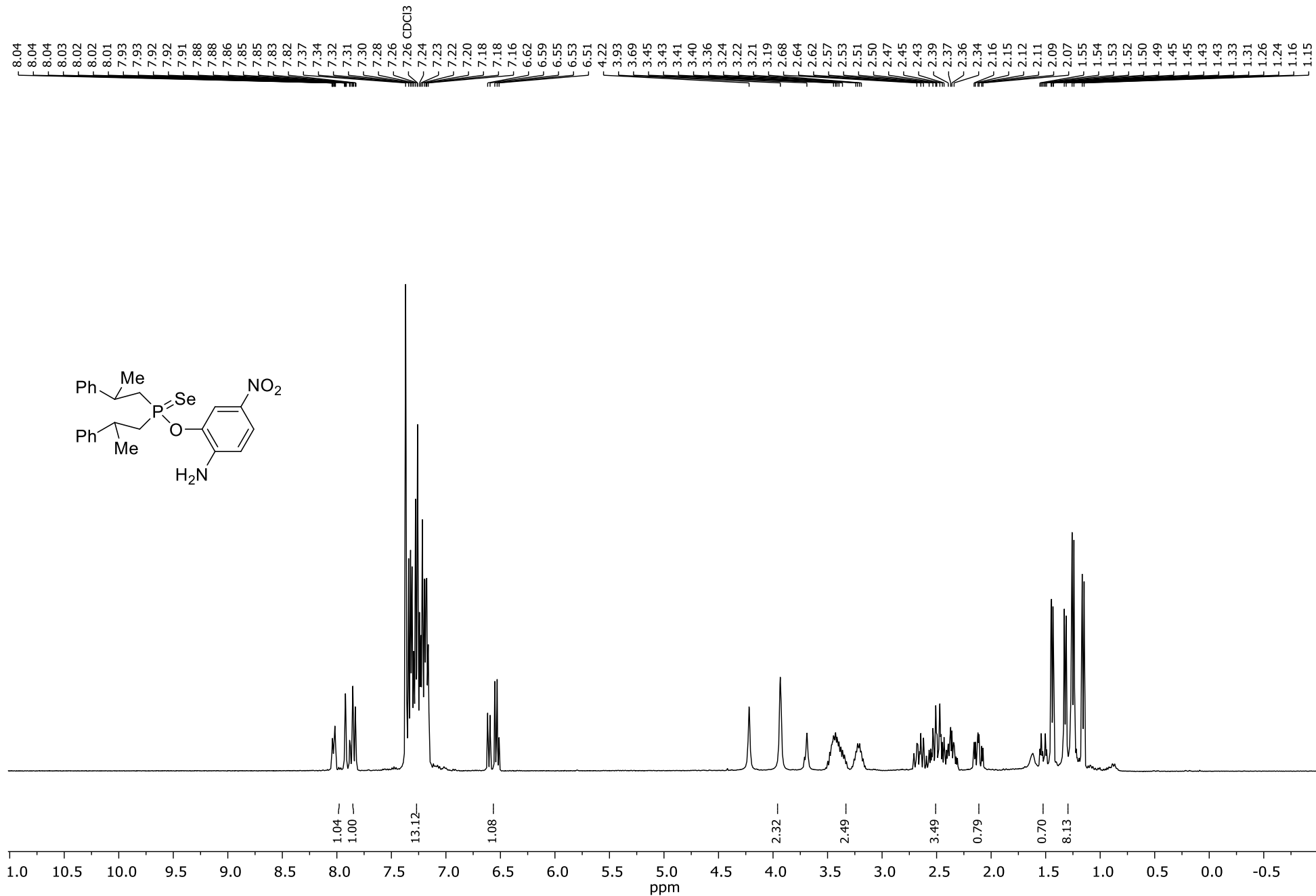
31P_NMR

— 108.29



O-(2-amino-5-nitrophenyl) bis(2-phenylpropyl)phosphinoselenoate (12k)

1H_NMR



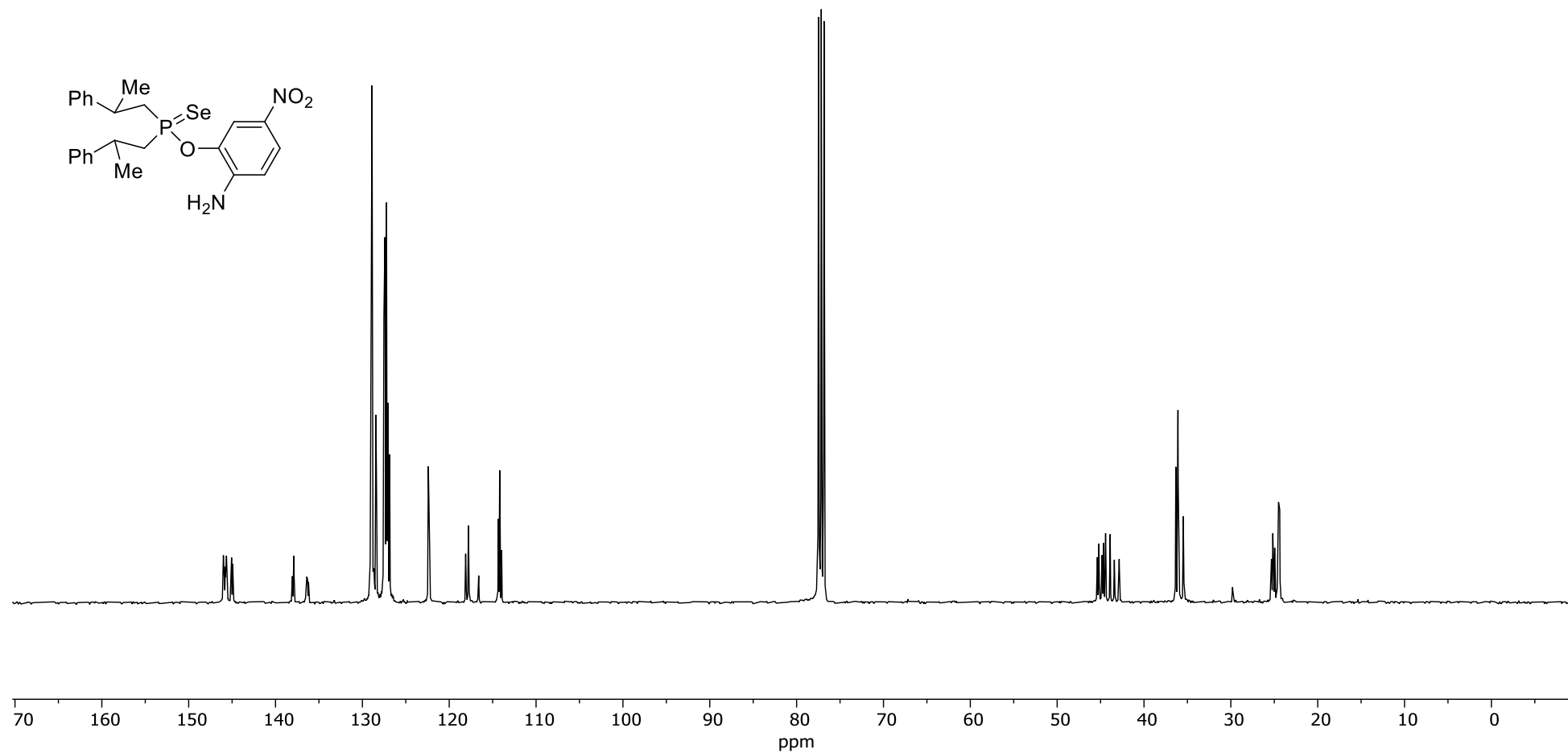
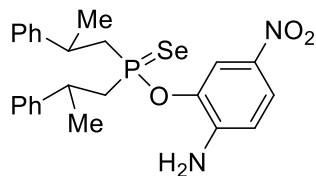
***O*-(2-amino-5-nitrophenyl) bis(2-phenylpropyl)phosphinoselenoate (12k)**

¹³C_NMR

146.03
146.00
145.91
145.88
145.79
145.74
145.65
145.59
145.12
145.06
144.94
144.90
138.06
137.89
136.50
136.40
136.31
136.21
129.06
128.98
128.89
128.44
127.52
127.40
127.38
127.22
127.11
127.06
126.86
122.41
122.24
118.11
118.07
117.79
117.75
116.65
116.60
114.35
114.19
113.97

77.48
77.16 CDCl₃
76.84

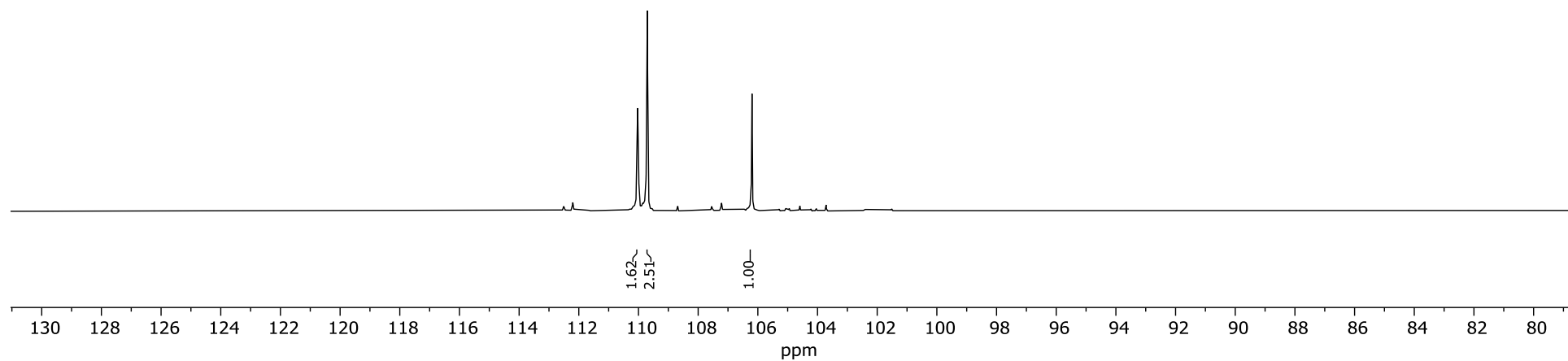
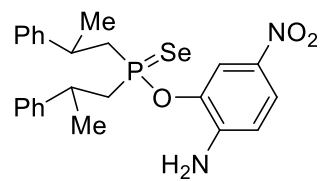
45.37
45.19
44.83
44.67
44.42
43.90
43.42
42.86
36.34
36.09
36.05
35.48
35.44
25.31
25.16
25.09
24.95
24.61
24.51
24.46
24.37



***O*-(2-amino-5-nitrophenyl) bis(2-phenylpropyl)phosphinoselenoate (12k)**

31P_NMR

110.03
109.71
106.20



1-[[bis(2-phenylethyl)phosphoroselenoyl]oxy]-2,2,6,6-tetramethylpiperidine (S1).

¹³C_NMR

141.49
141.34

129.92
129.34
127.69

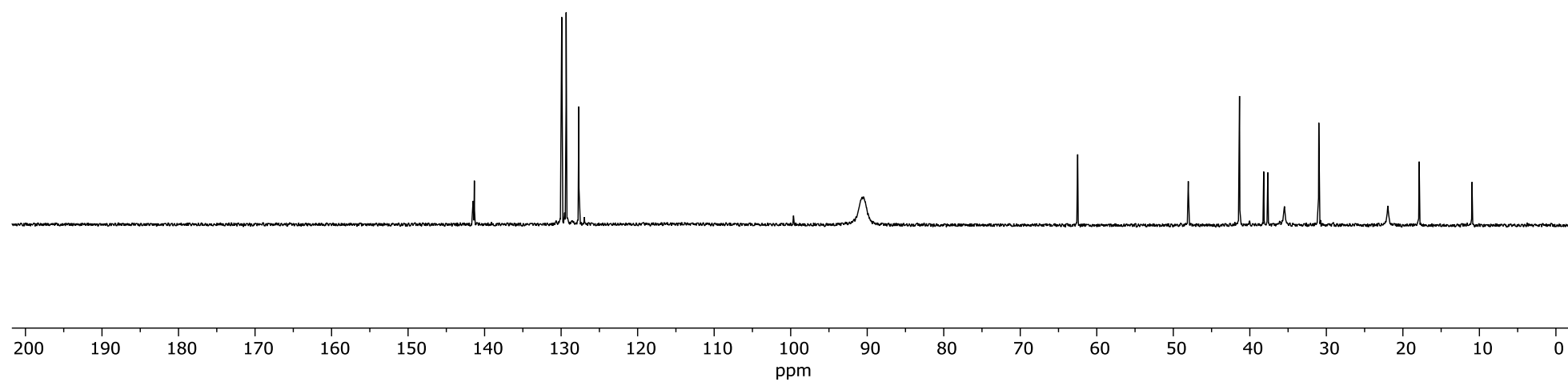
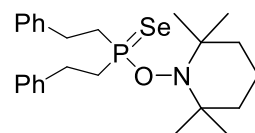
62.50
62.48

48.04

41.35
38.19
37.65

30.99

15.60
11.68



1-[[bis(2-phenylethyl)phosphoroselenoyl]oxy]-2,2,6,6-tetramethylpiperidine (S1).

³¹P-NMR

115.40
112.96
110.53

