Synthesis of 2,3-dihydro-1-phenylbenzo[b]phosphole (1-phenylphosphindane) and its use as a mechanistic test in the asymmetric Appel reaction. Decisive evidence against involvement of pseudorotation in the stereoselecting step.

Damien J. Carr, Jaya Satyanarayana Kudavalli, Katherine Dunne, Helge Müller-Bunz and Declan G. Gilheany*

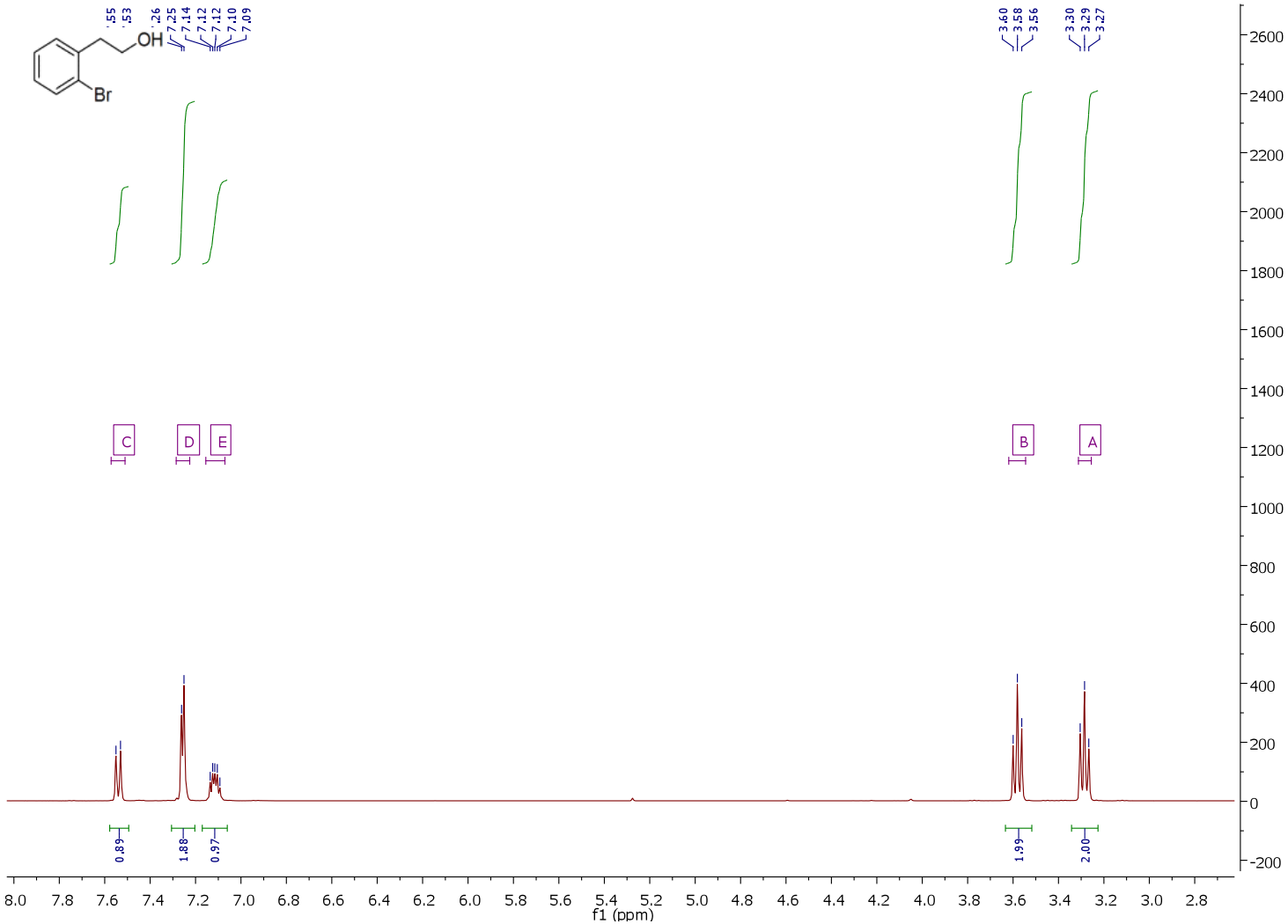
Centre for Synthesis and Chemical Biology, School of Chemistry and Chemical Biology, University College Dublin, Dublin 4, Ireland

Supporting Information

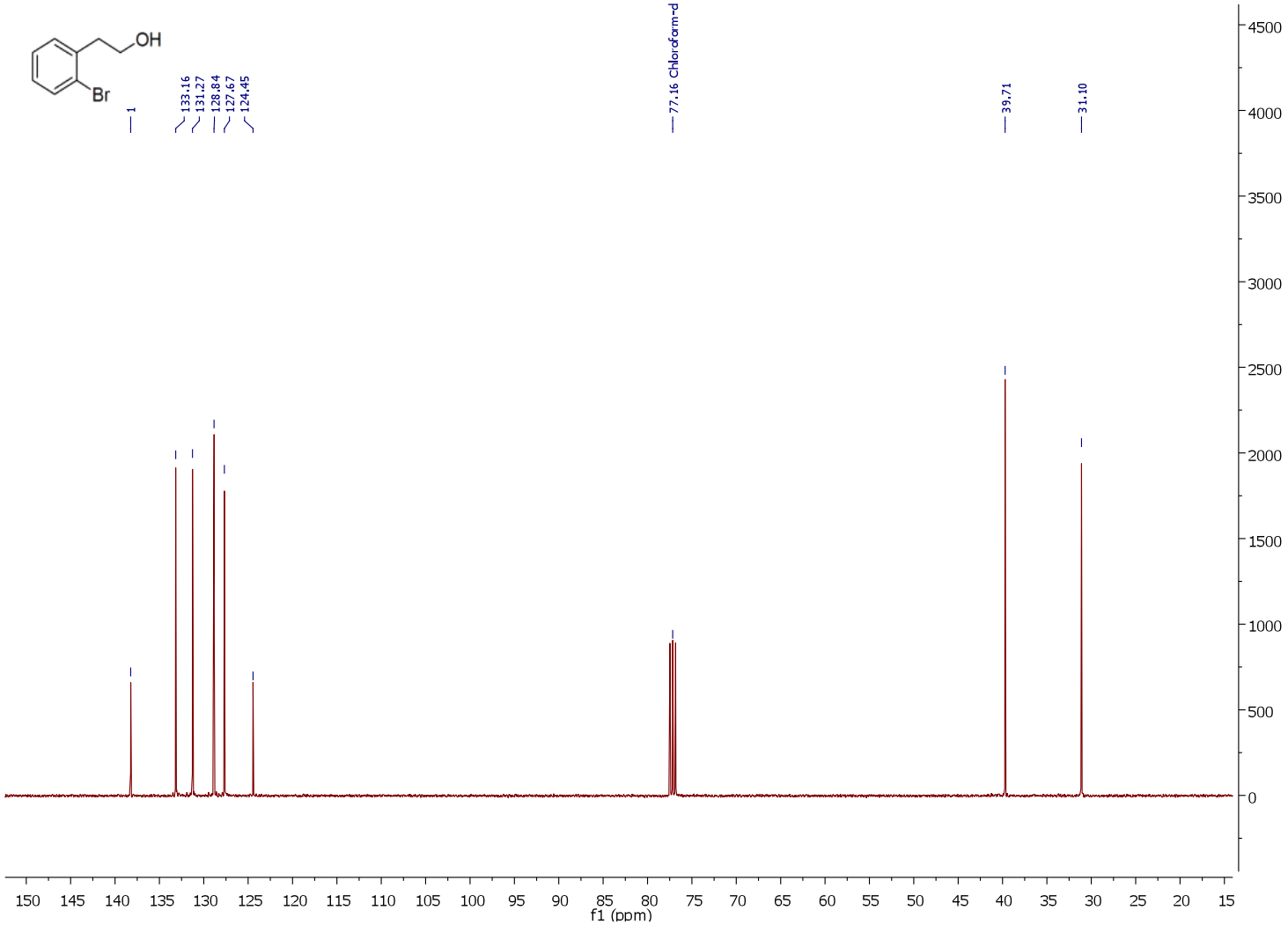
Contents:

^{1}H NMR, ^{13}C NMR and ^{31}P NMR spectra for all isolated compounds	S1-S25
HPLC analysis for Appel reaction procedure	S27-S35
^{31}P NMR spectra for intermediates observed	S37-S41
Crystallography Information	S43-S60

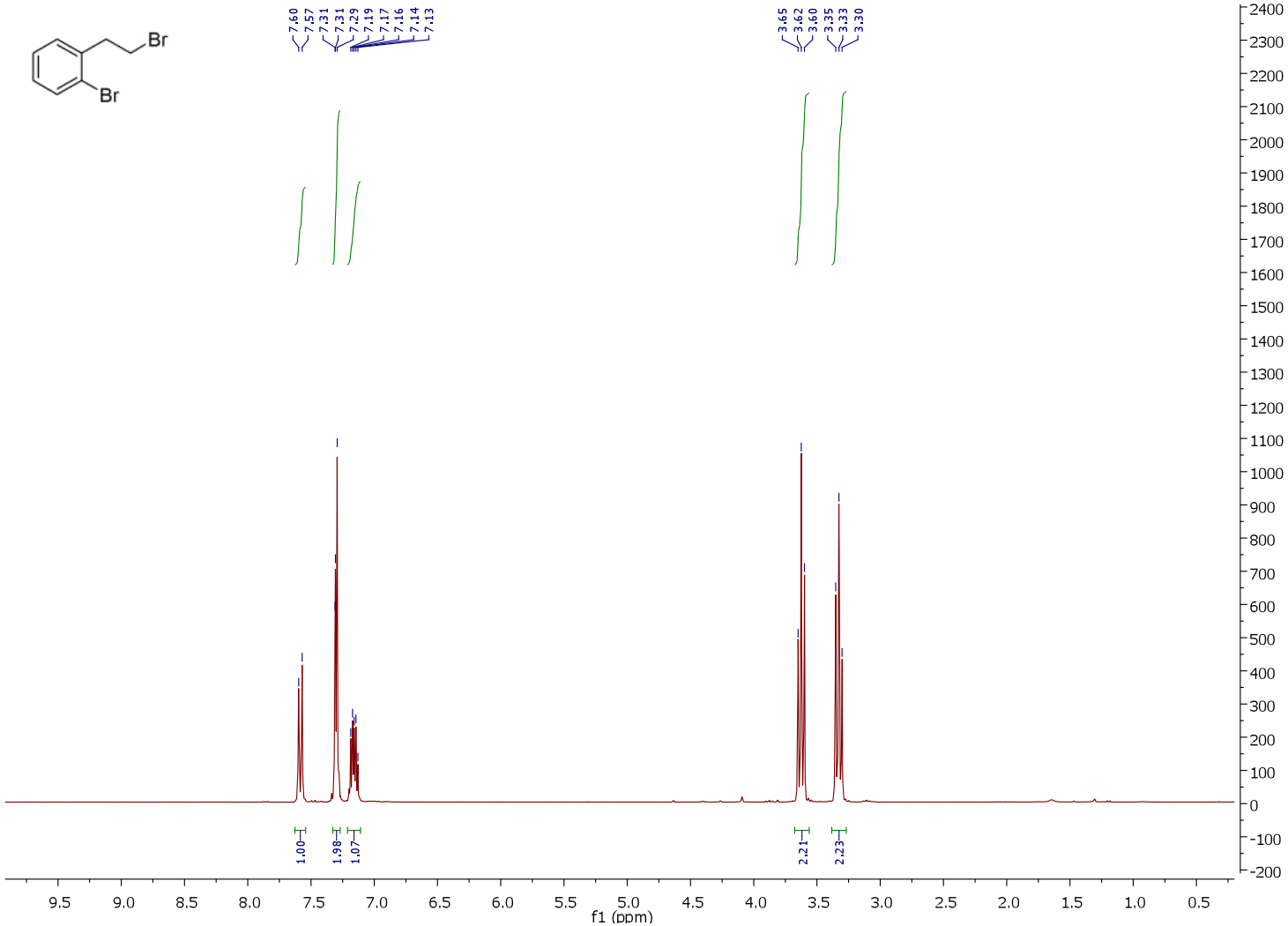
^{1}H NMR 2-(2-Bromophenyl)ethanol



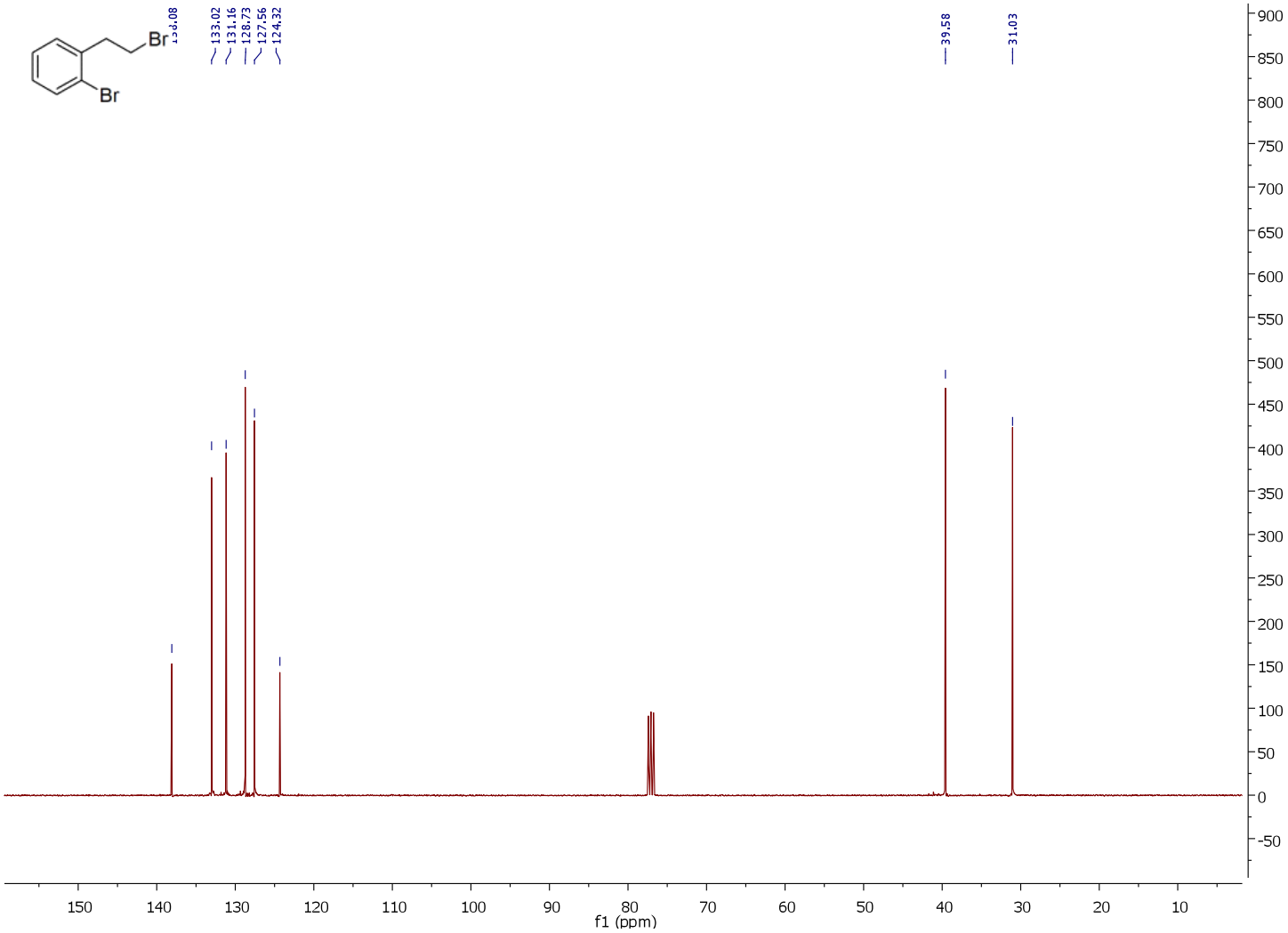
^{13}C NMR 2-(2-Bromophenyl)ethanol



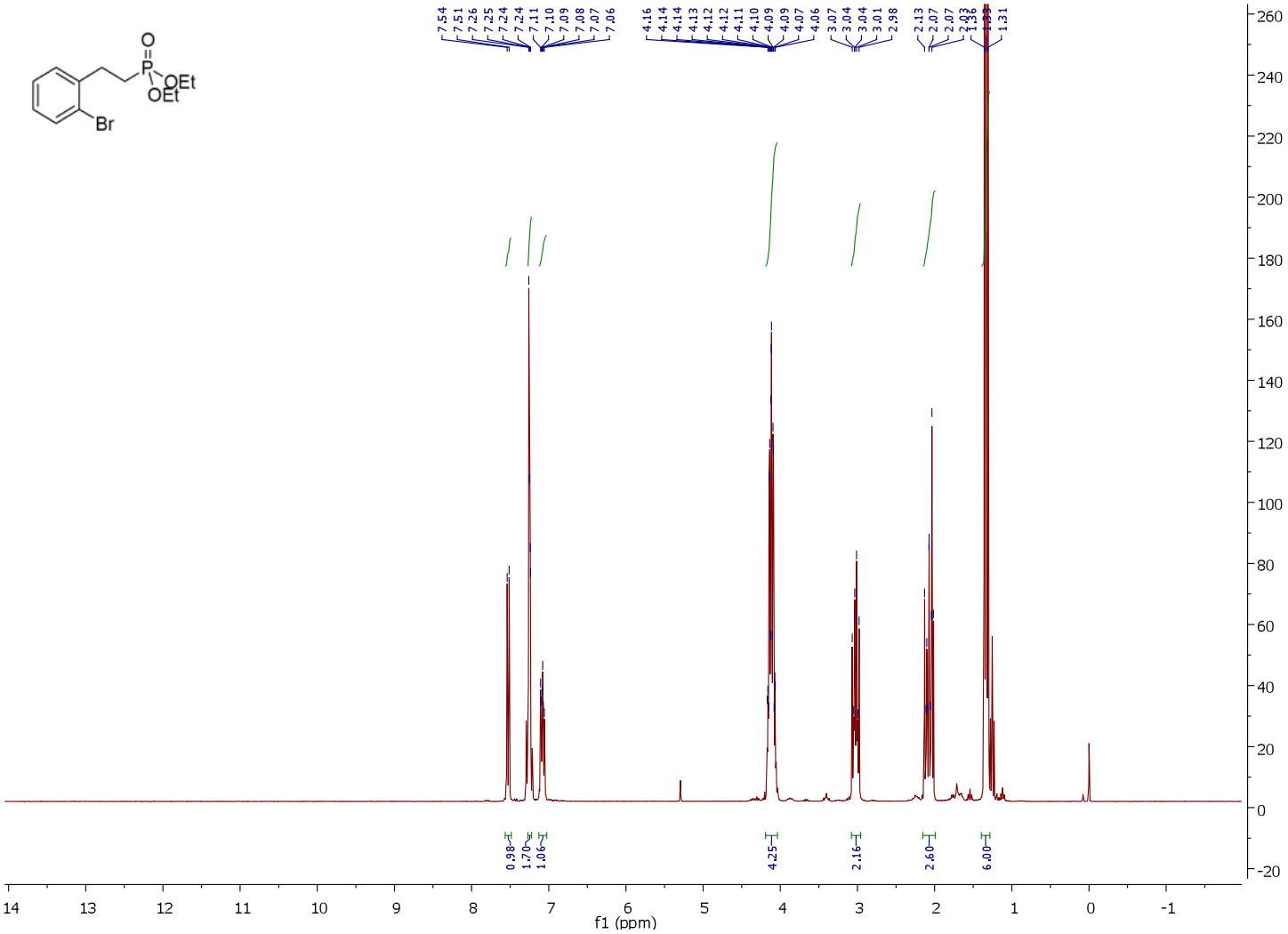
^{1}H NMR (2-Bromophen-1-yl)ethyl bromide (4)



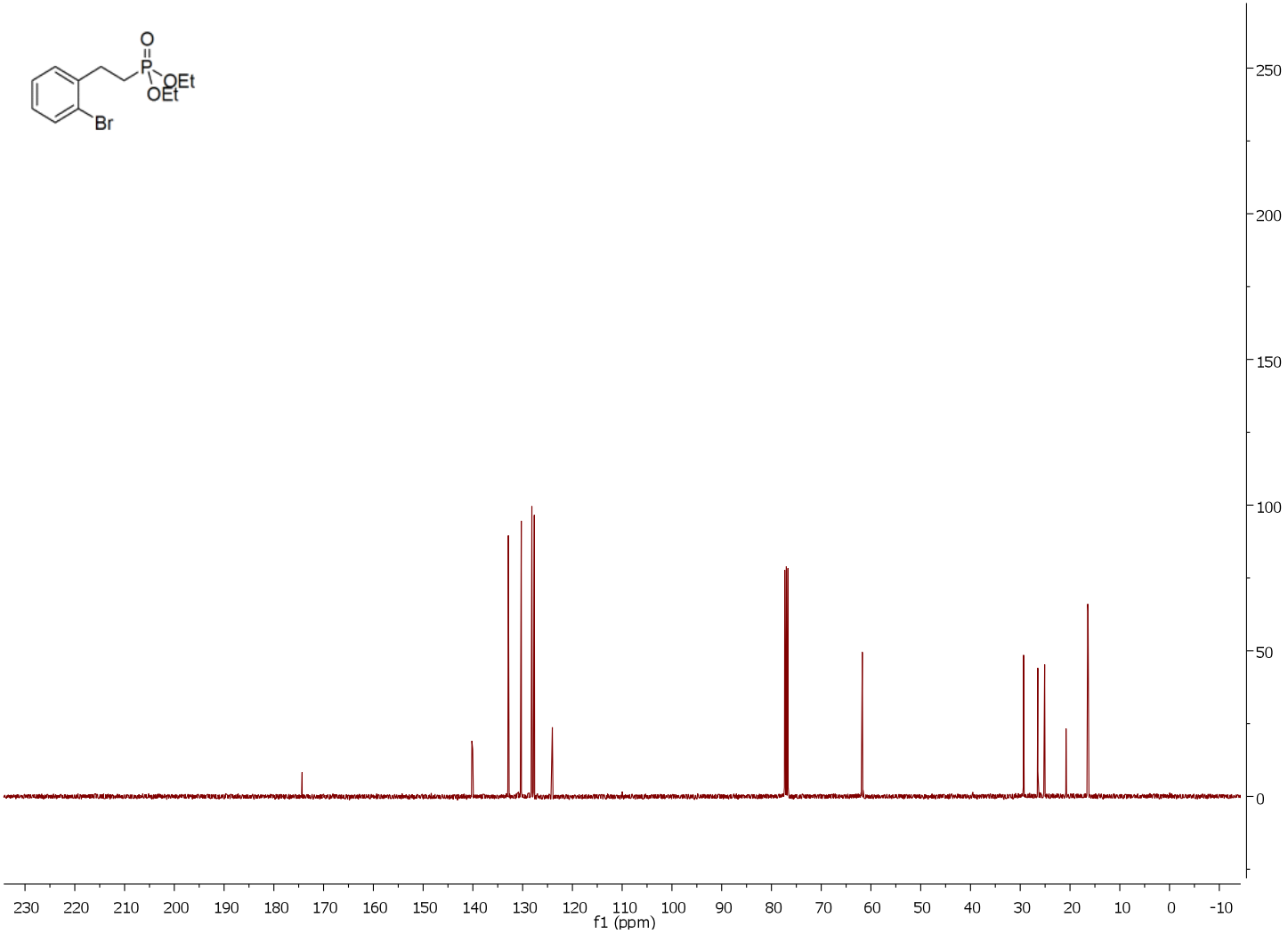
^{13}C NMR (2-Bromophen-1-yl)ethyl bromide (4)



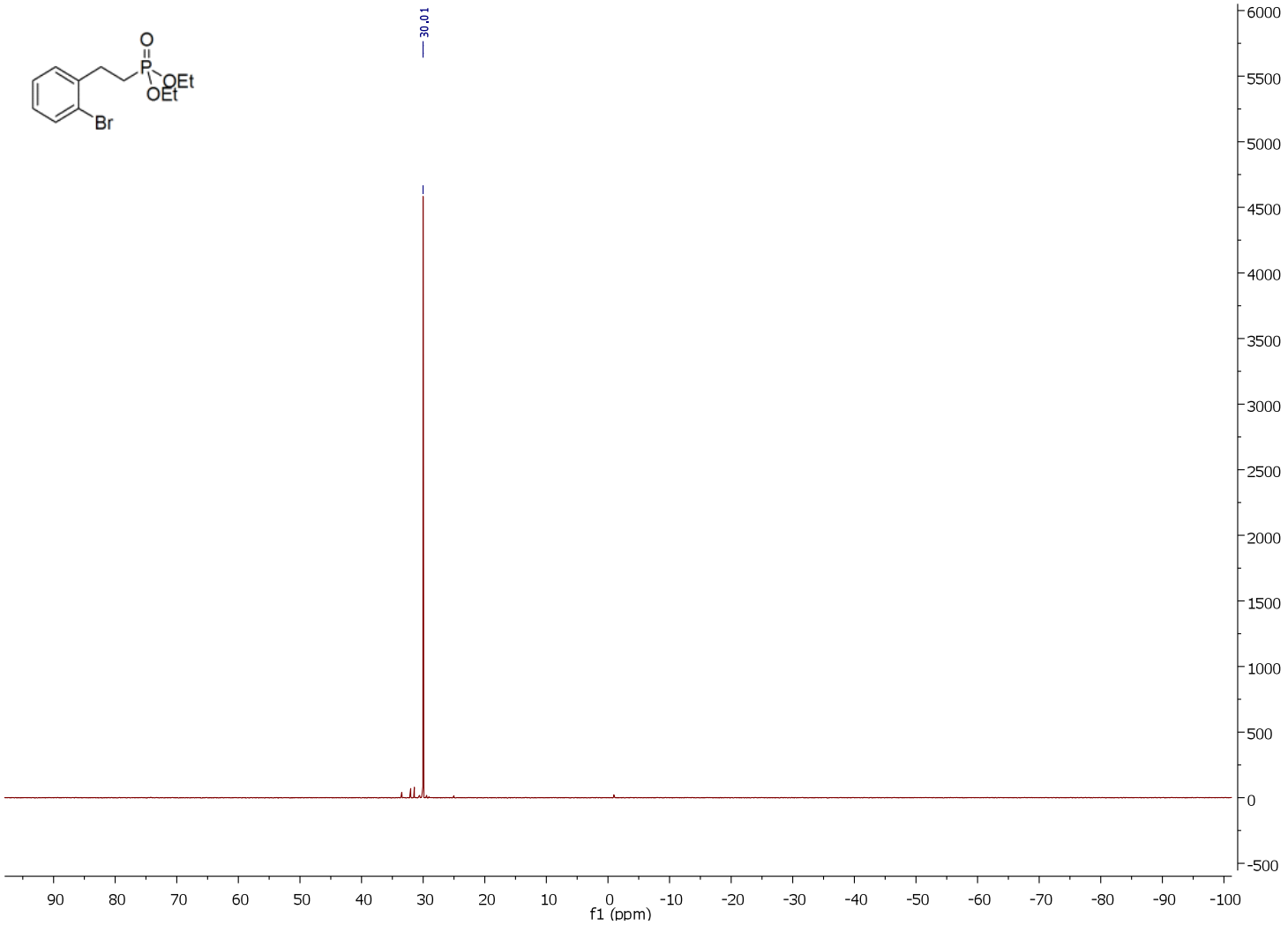
^{1}H NMR Diethyl 2-(2-bromophen-1-yl)ethylphosphonate (5)



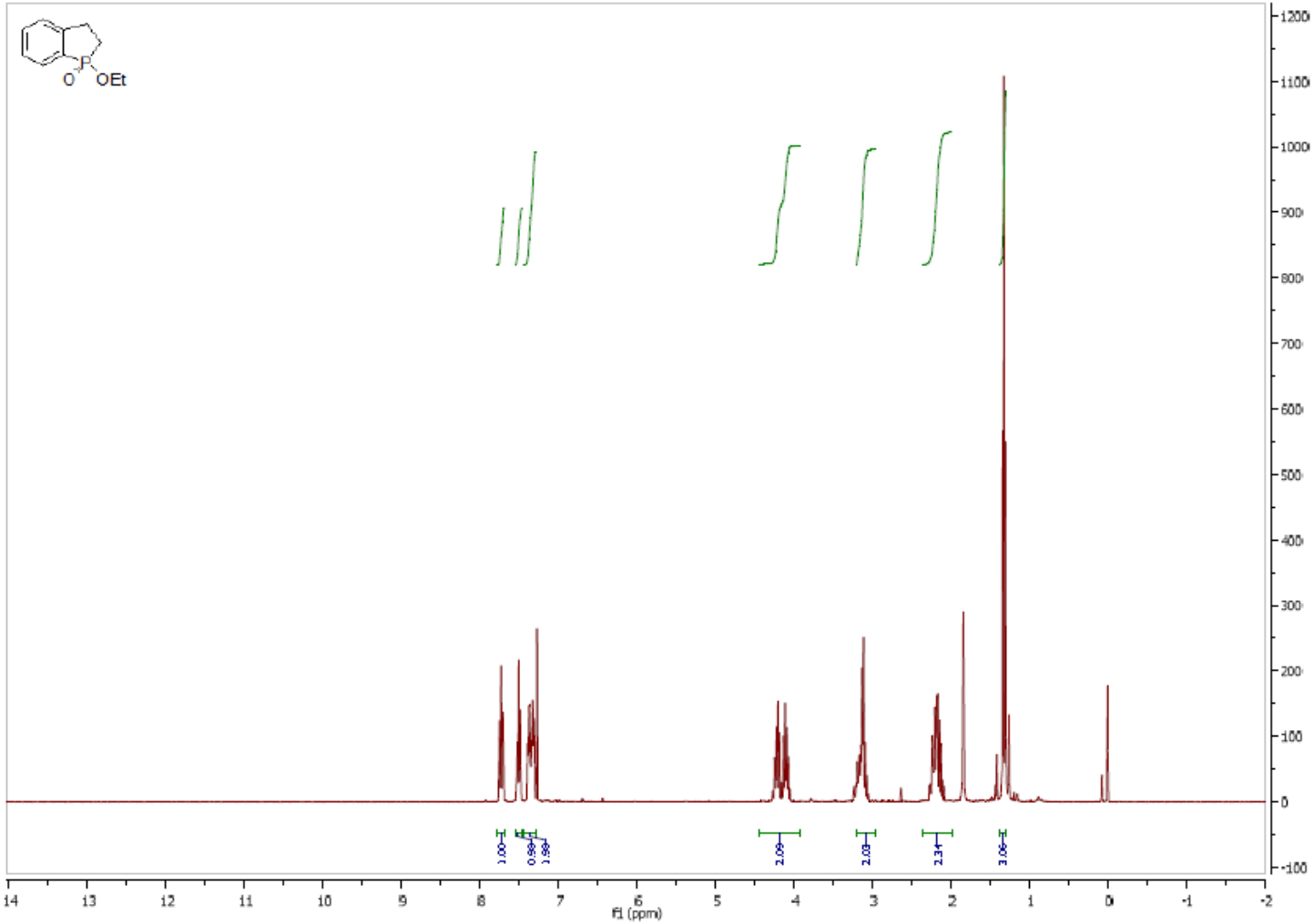
^{13}C NMR Diethyl 2-(2-bromophen-1-yl)ethylphosphonate (5)



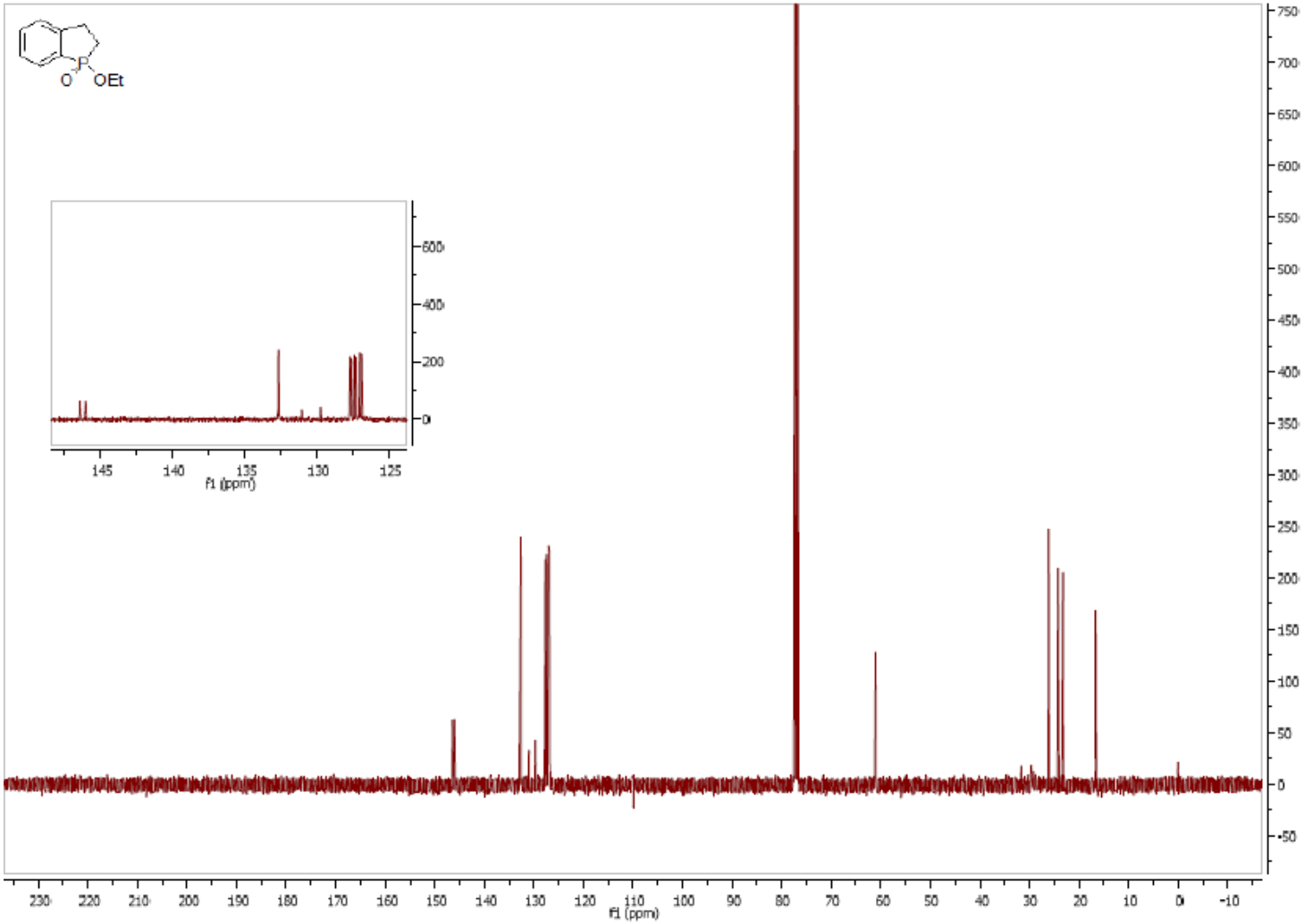
^{31}P NMR Diethyl 2-(2-bromophen-1-yl)ethylphosphonate (5)



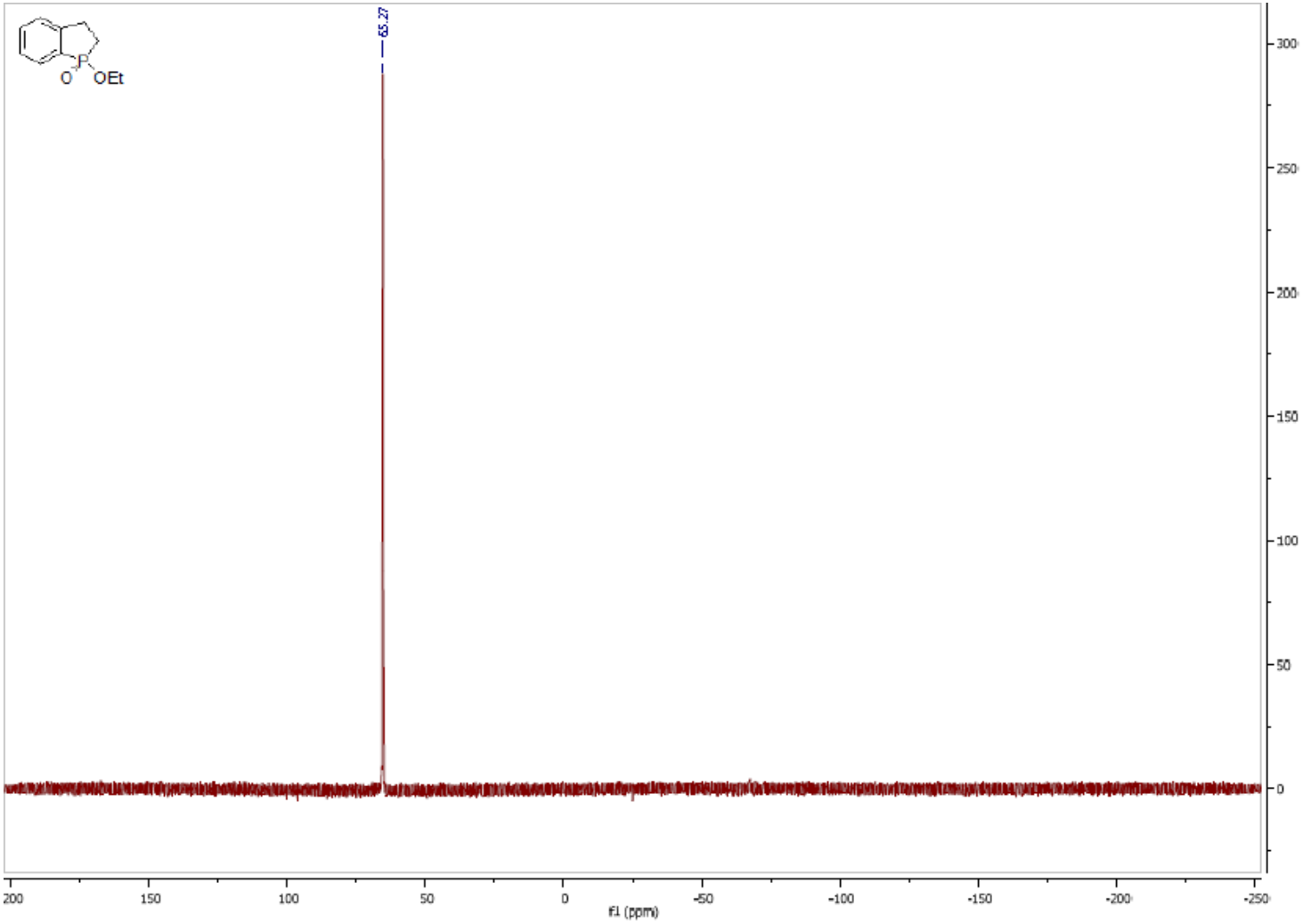
^{1}H NMR 11-ethoxy-2,3-dihydrobenzo[b]phosphole-1-oxide (6)



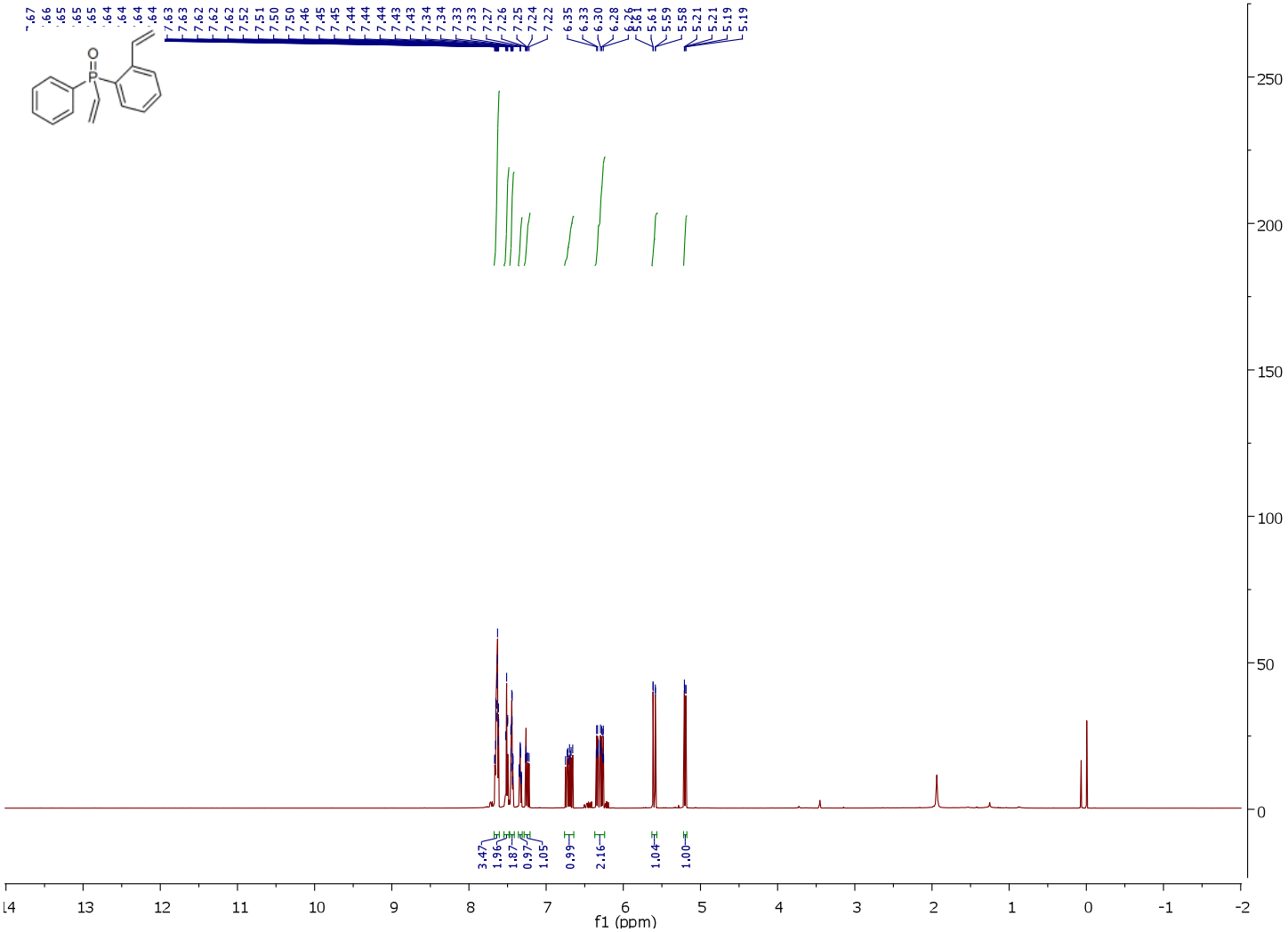
^{13}C NMR 1-ethoxy-2,3-dihydrobenzo[b]phosphole-1-oxide (6)



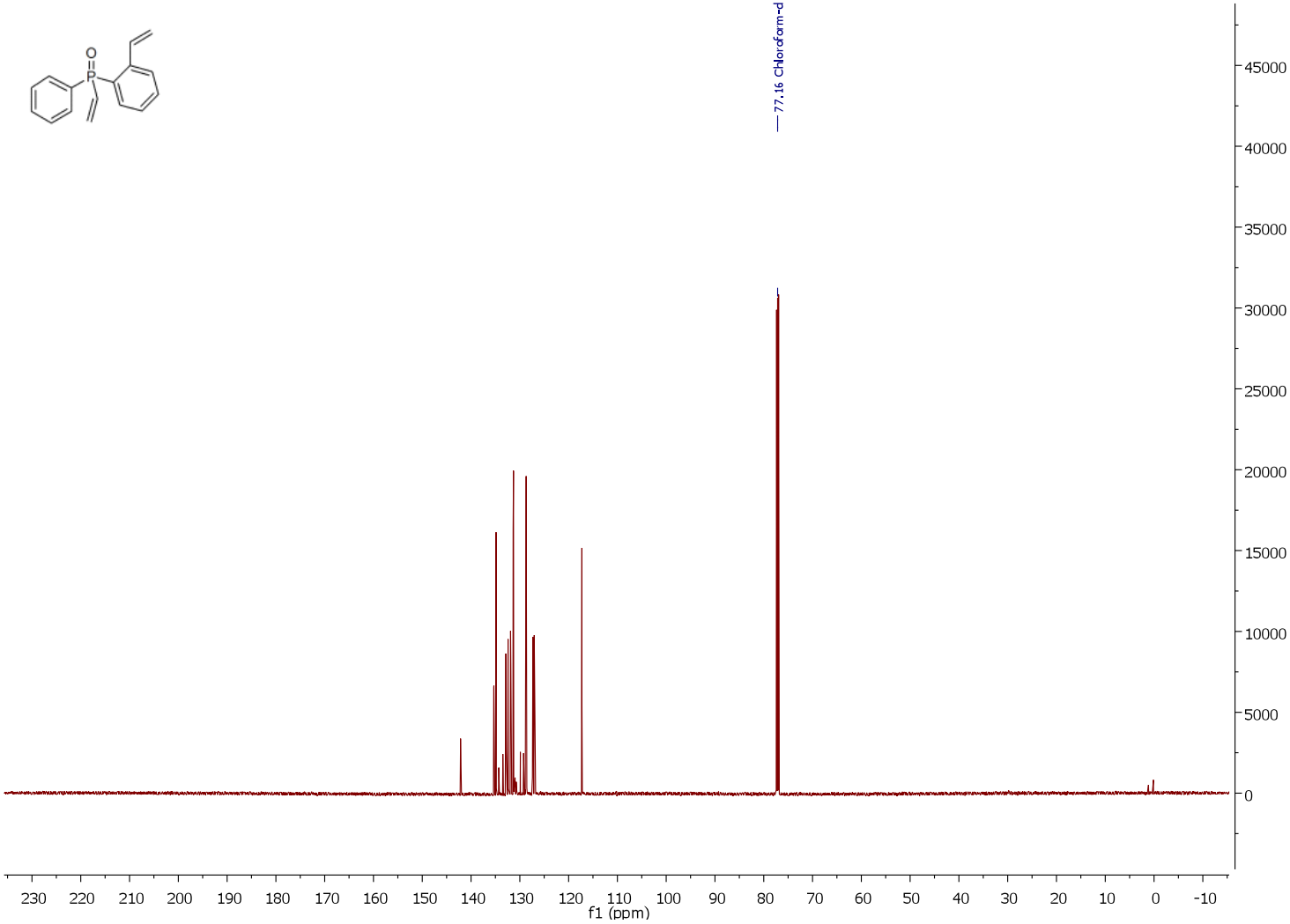
^{31}P NMR 1-ethoxy-2,3-dihydrobenzo[b]phosphole-1-oxide (6)

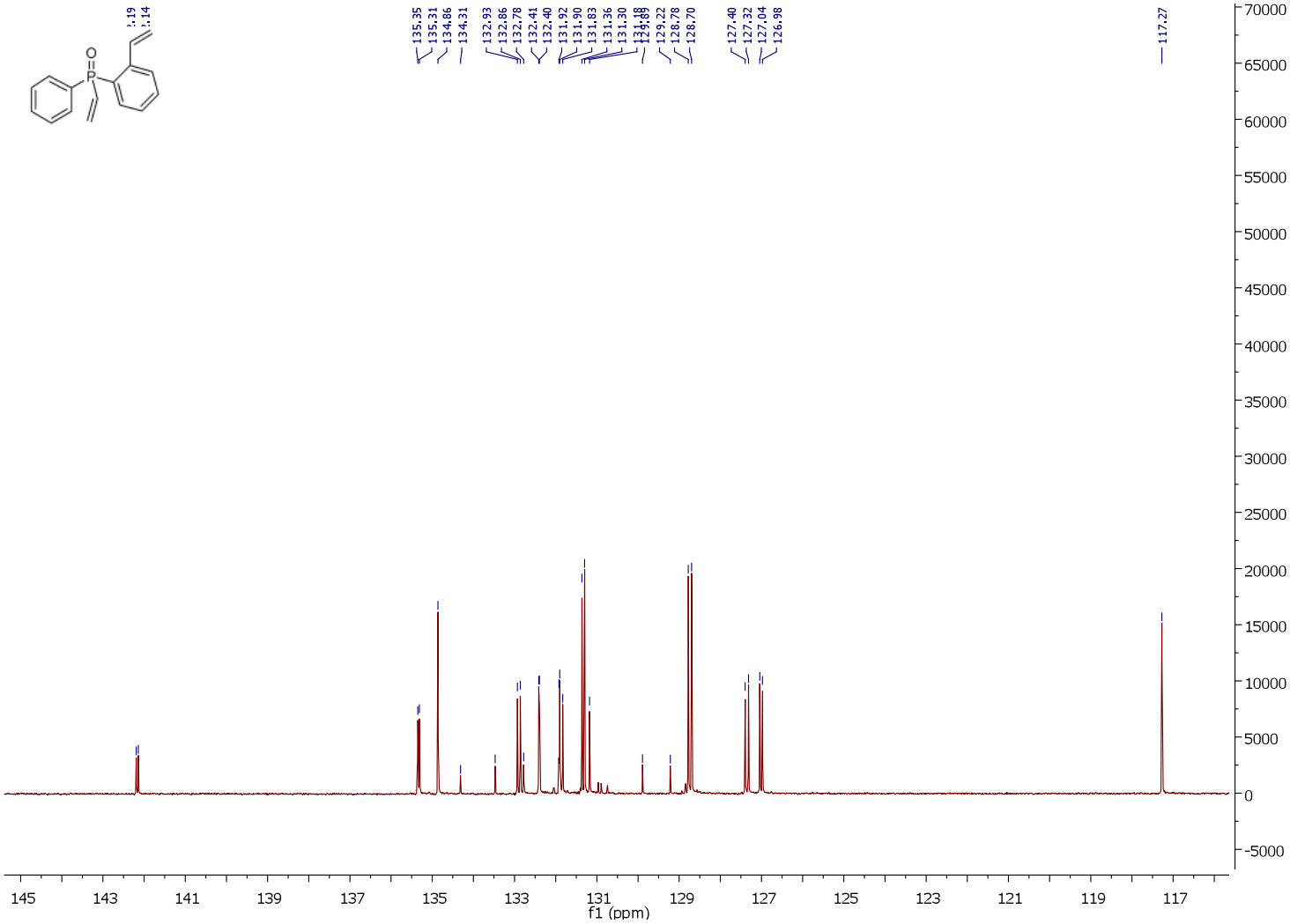


^{1}H NMR Phenyl(*o*-vinylphenyl)(vinyl)phosphine Oxide (7)

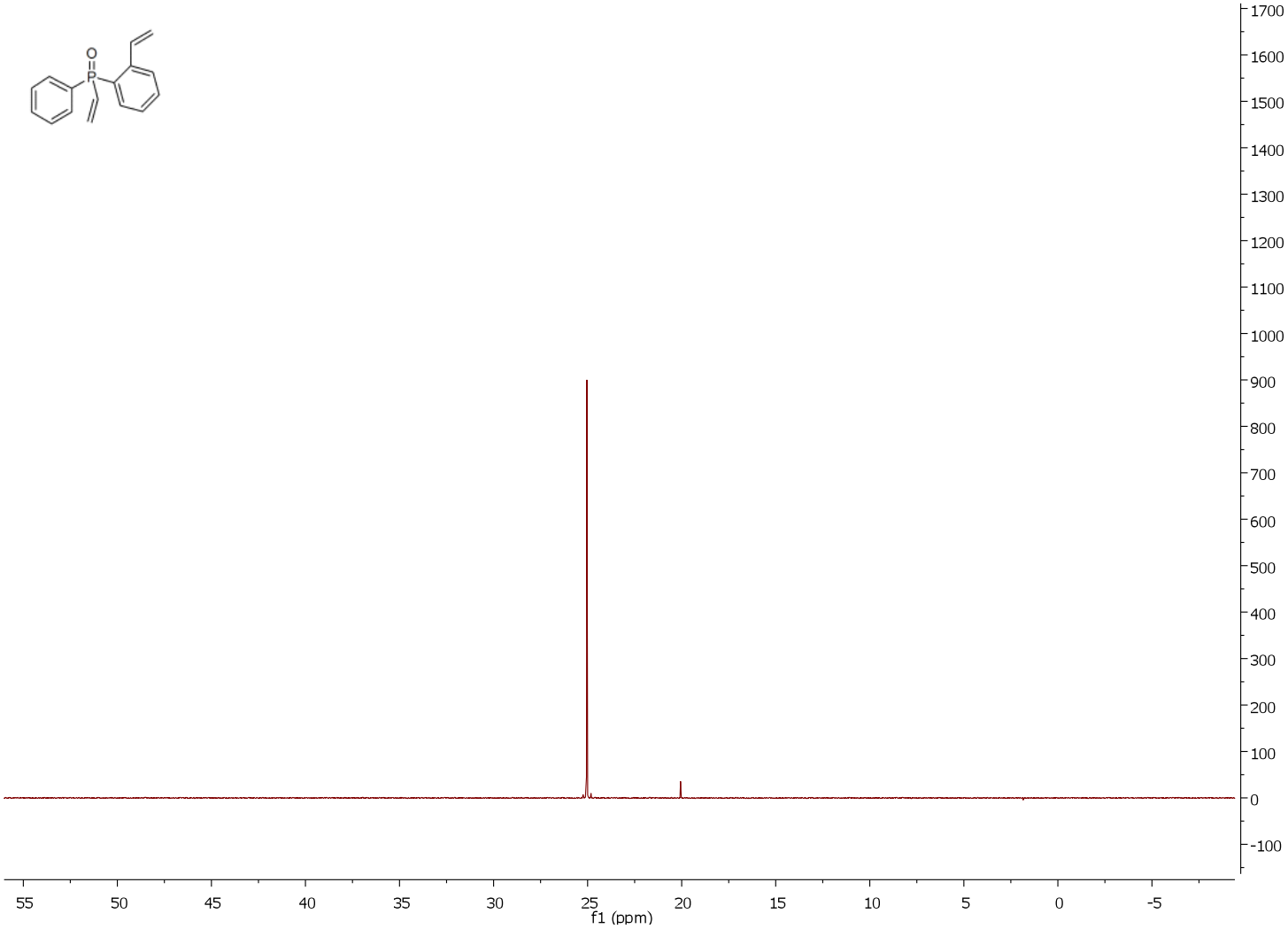


^{13}C NMR Phenyl(*o*-vinylphenyl)(vinyl)phosphine Oxide (7)

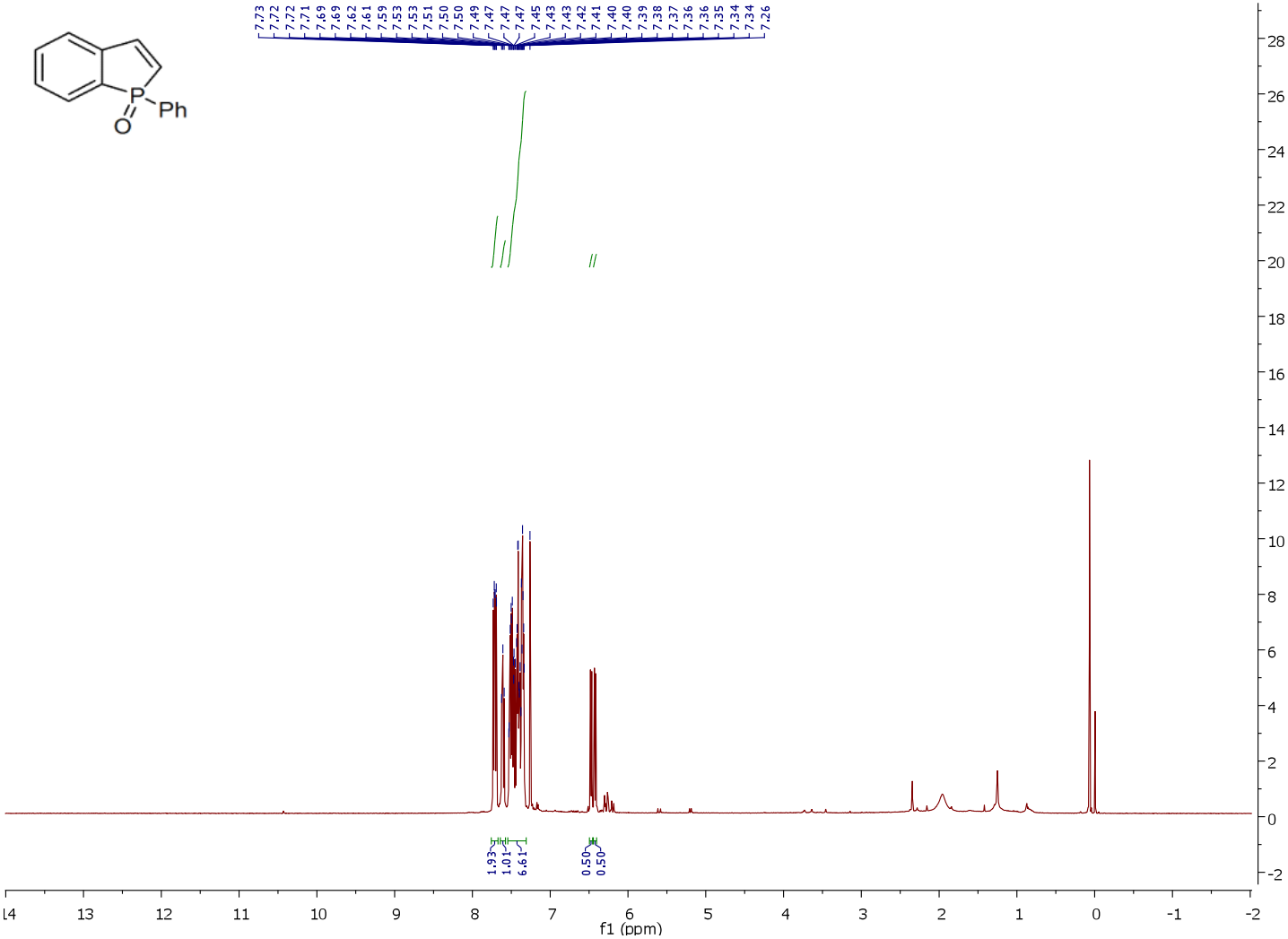



135.35
135.31
134.86
134.31
132.93
132.86
132.78
132.41
132.40
131.92
131.90
131.83
131.36
131.30
129.22
128.78
128.70
127.40
127.32
127.04
126.98
117.27
f1 (ppm)

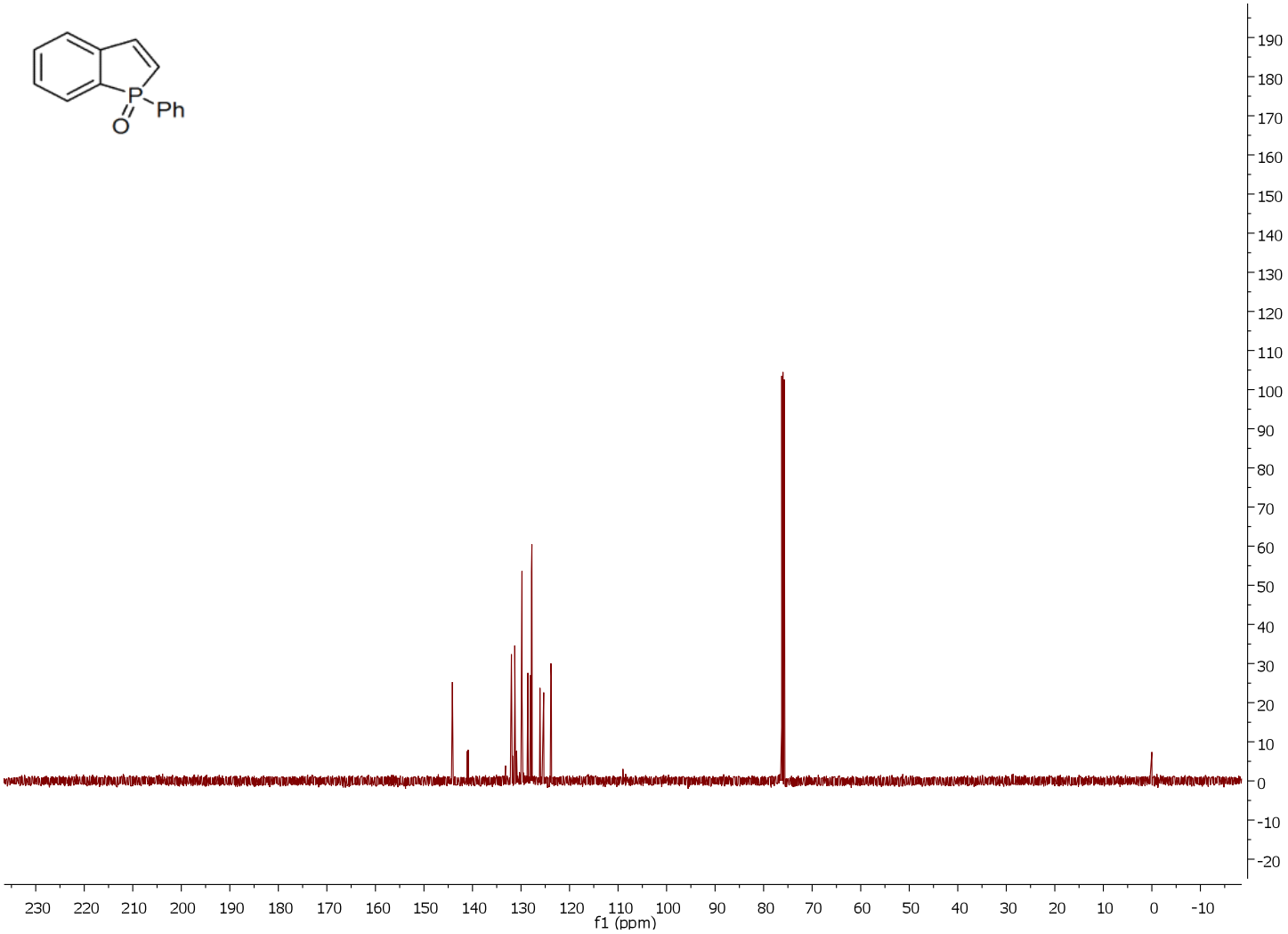
^{31}P NMR Phenyl(*o*-vinylphenyl)(vinyl)phosphine Oxide (7)

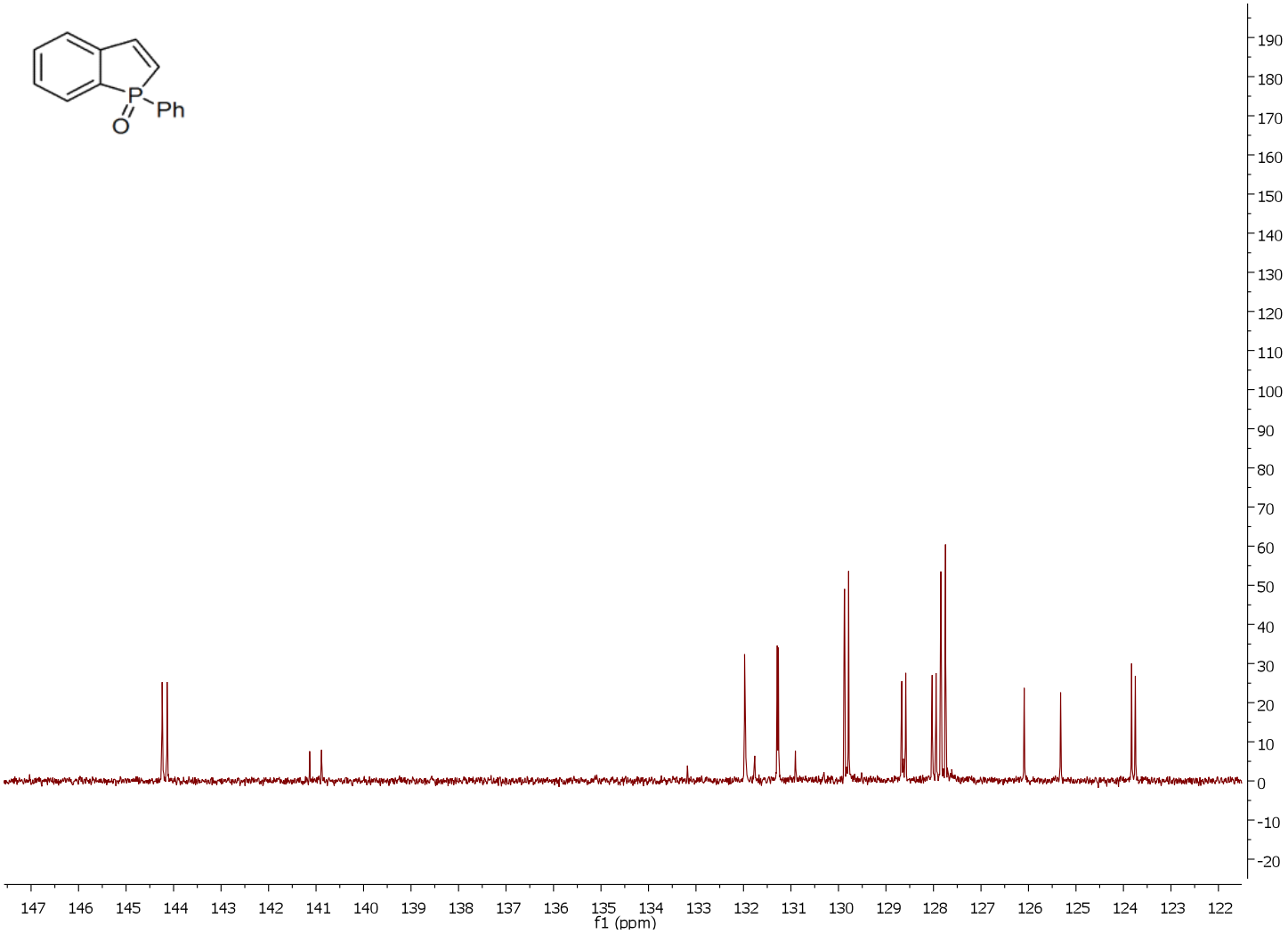


^{1}H NMR 1-Phenylbenzo[b]phosphole-1-oxide (8):

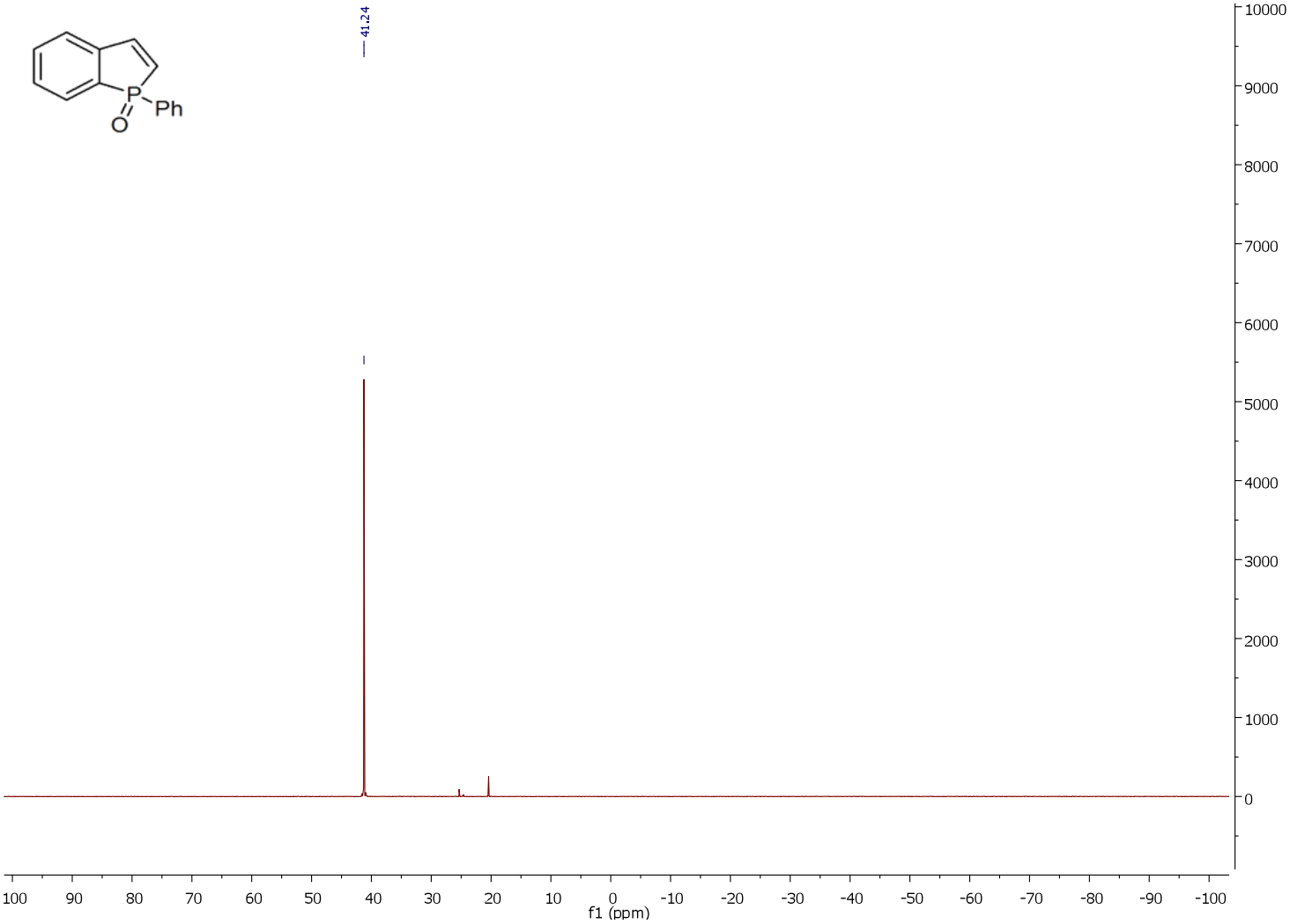


^{13}C NMR 1-Phenylbenzo[b]phosphole-1-oxide (8):

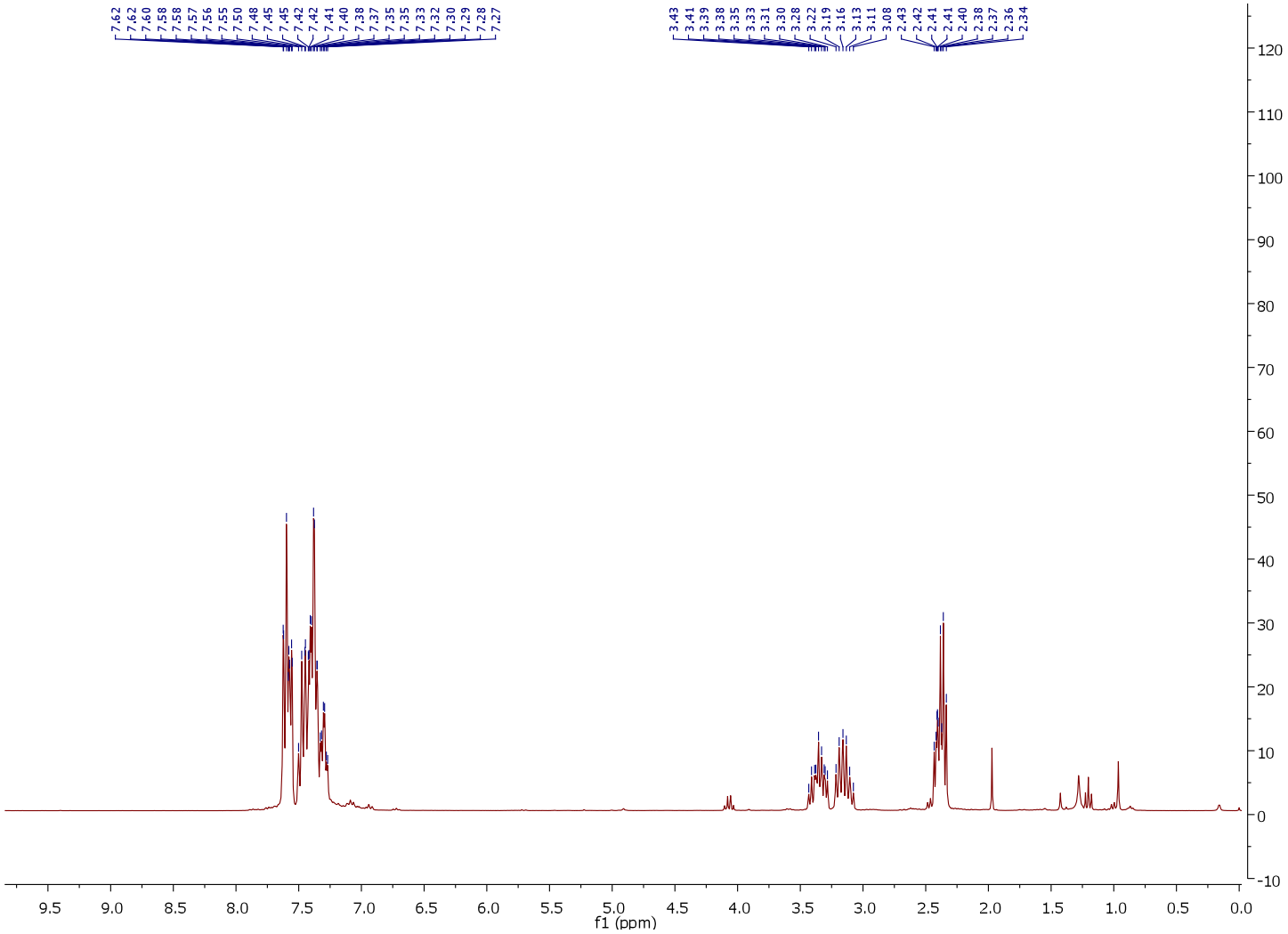



P
Ph
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
147 146 145 144 143 142 141 140 139 138 137 136 135 134 133 132 131 130 129 128 127 126 125 124 123 122
f1 (ppm)

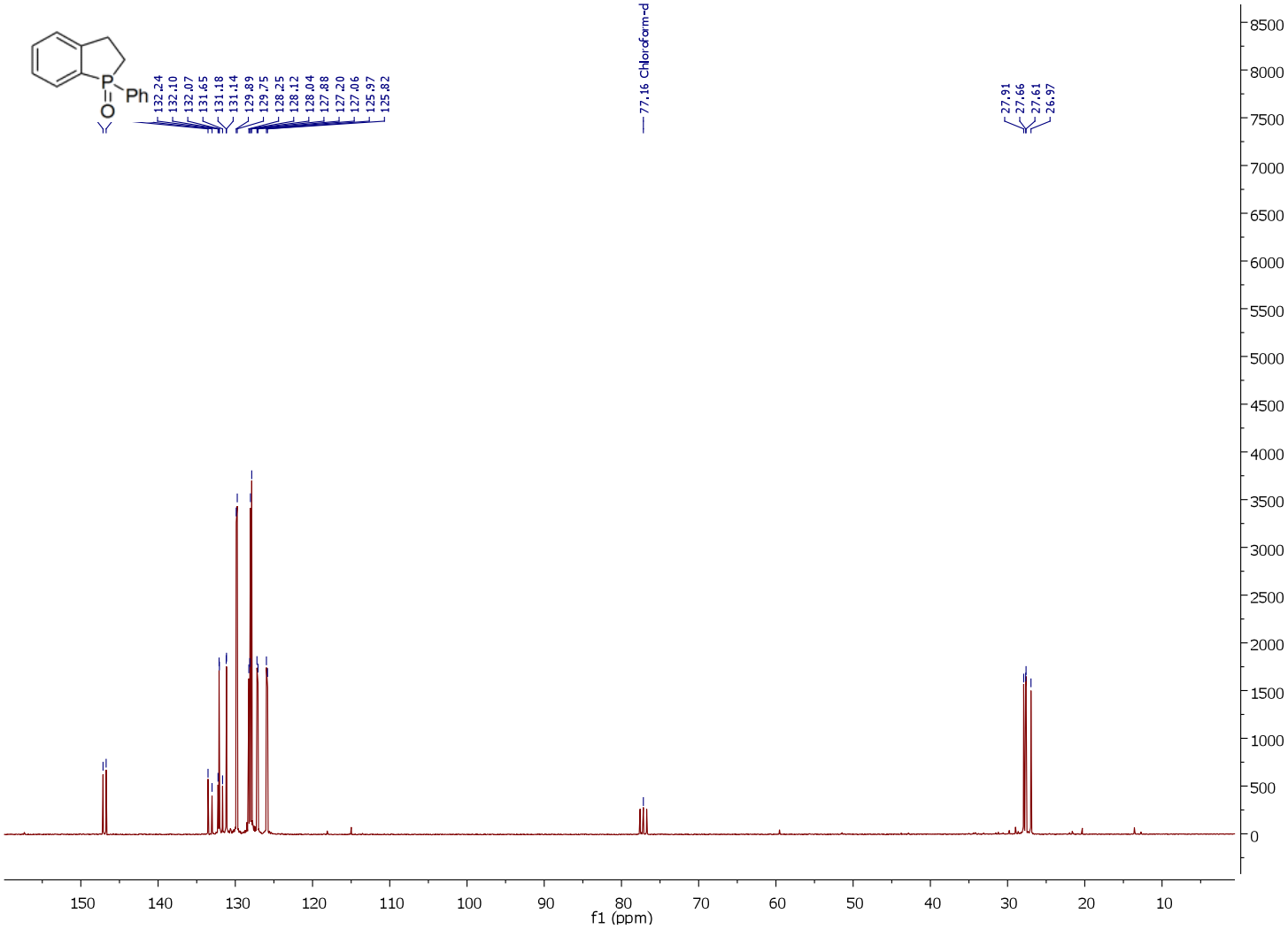
^{31}P NMR 1-Phenylbenzo[b]phosphole-1-oxide (8):



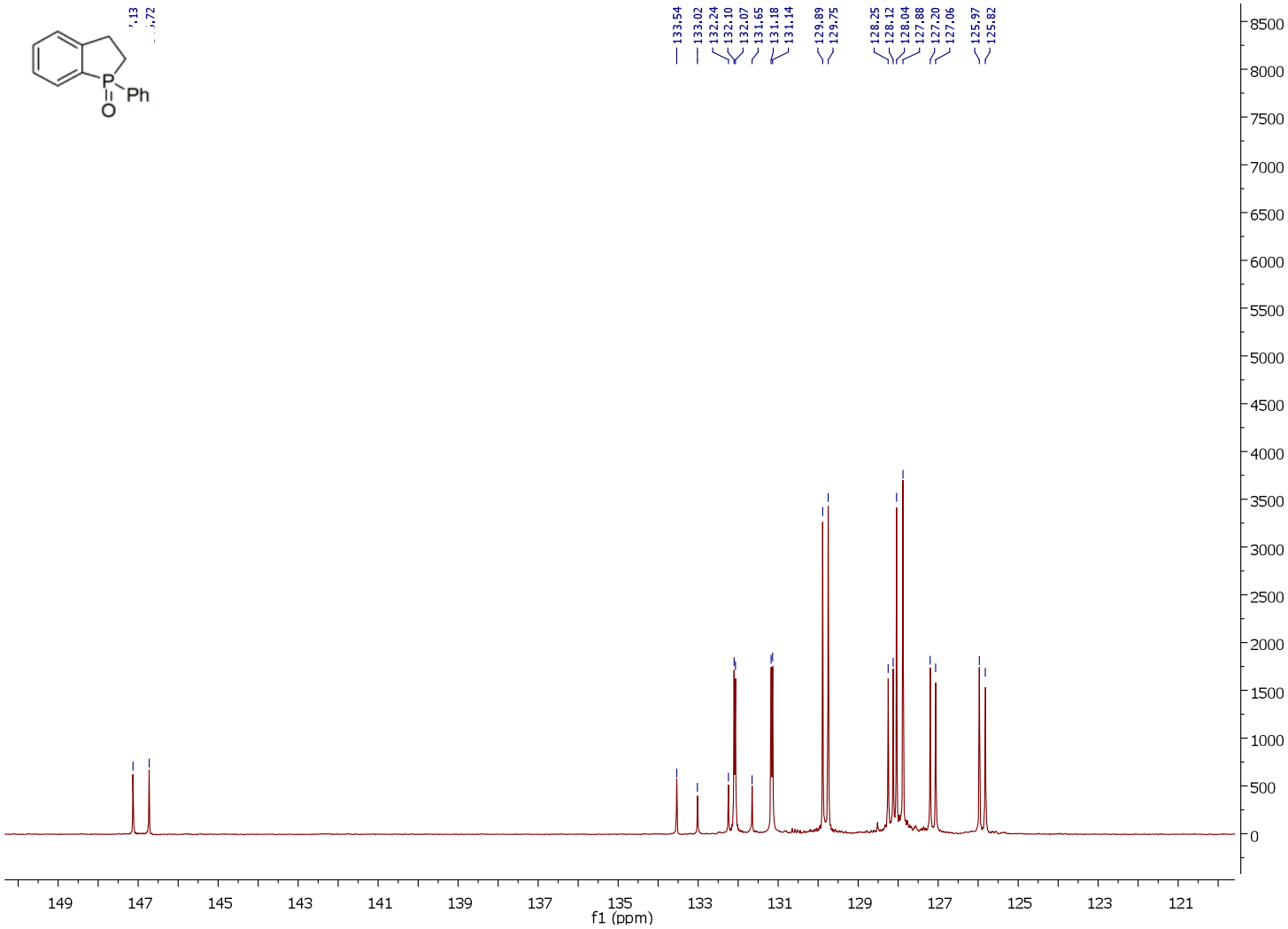
^{1}H NMR 1-Phenyl-2,3-dihydrobenzo[b]phosphole-1-oxide (oxo-1)



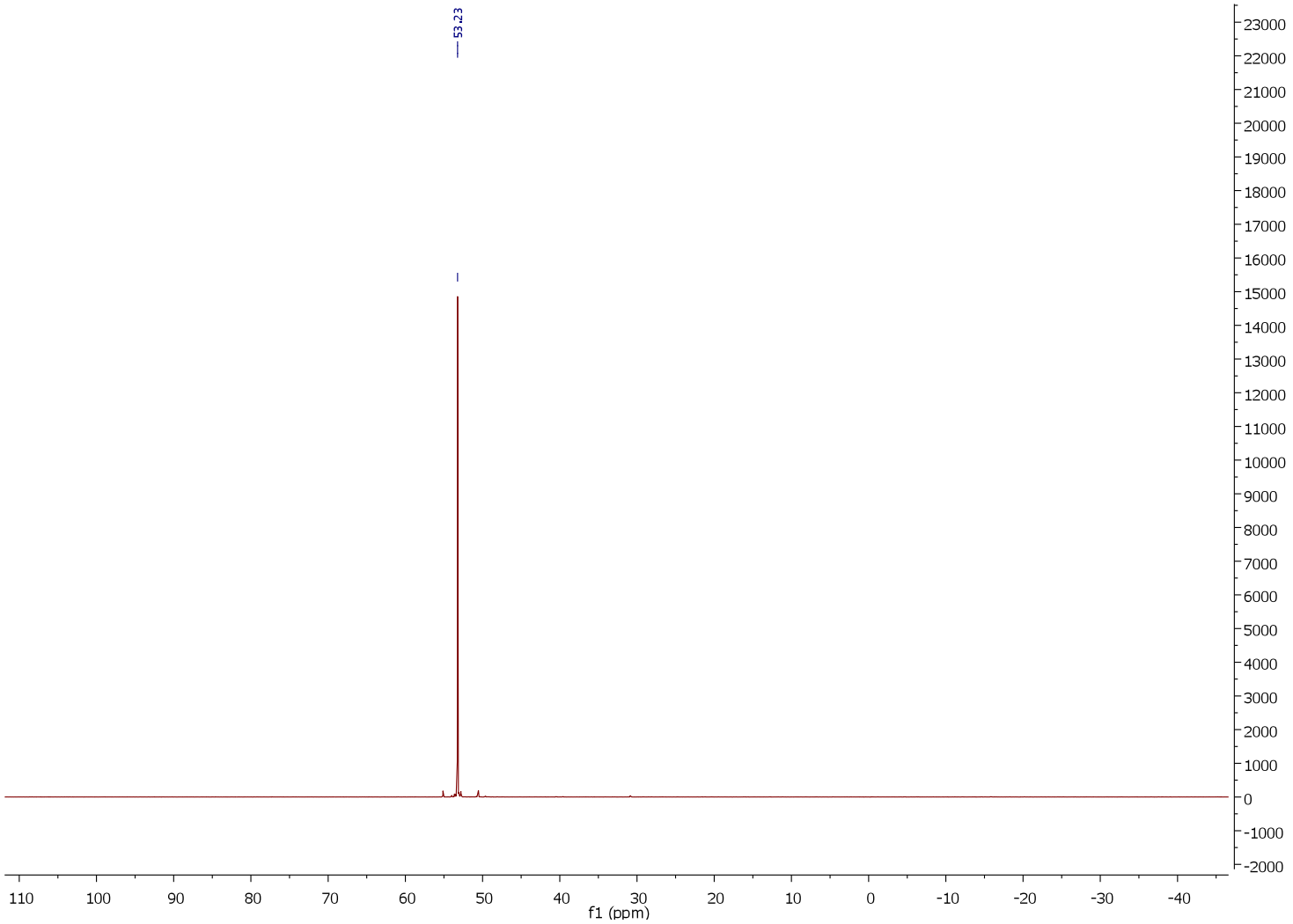
^{13}C NMR 1-Phenyl-2,3-dihydrobenzo[b]phosphole-1-oxide (oxo-1)



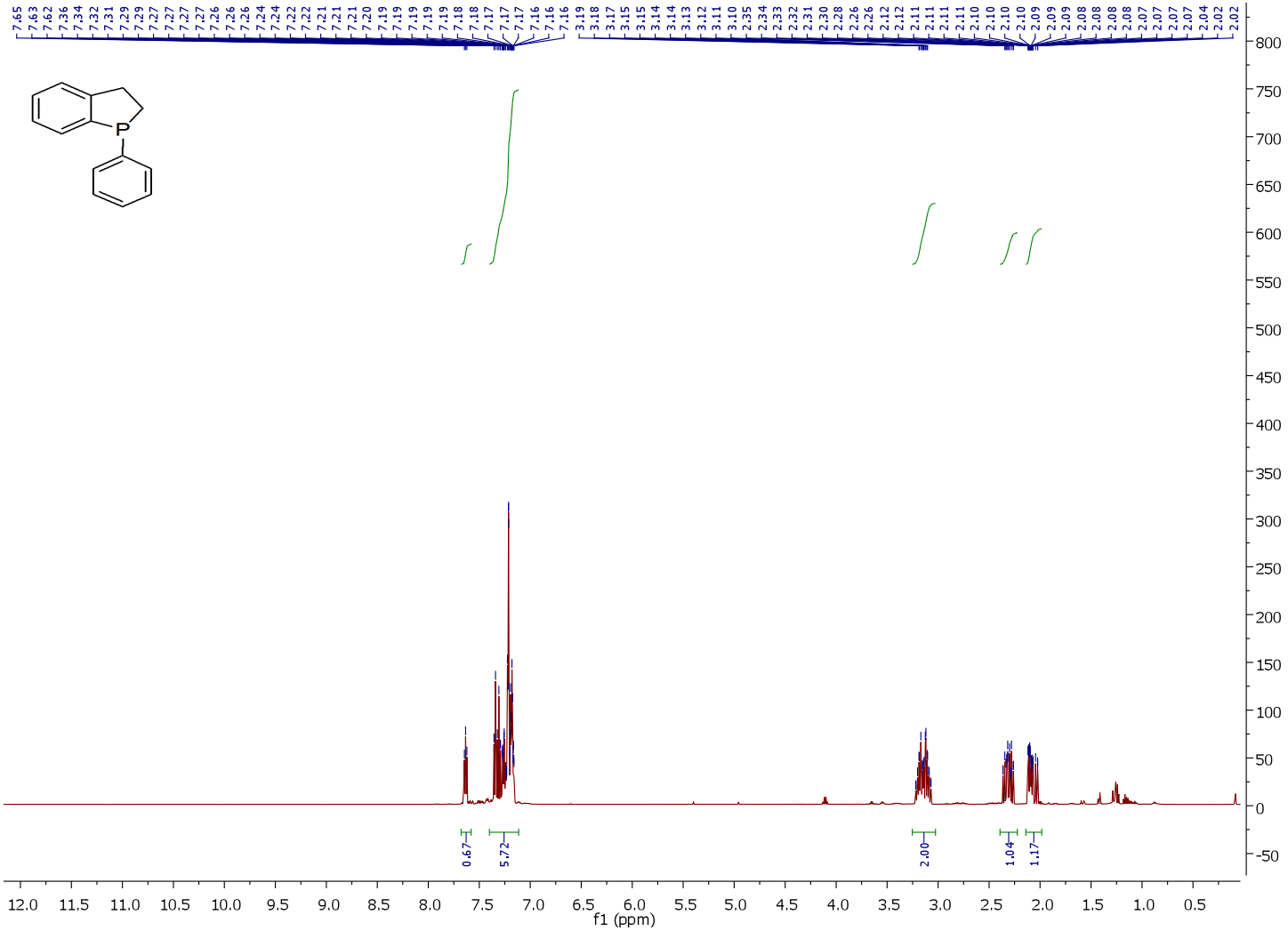
^{13}C NMR 1-Phenyl-2,3-dihydrobenzo[b]phosphole-1-oxide (oxo-1)



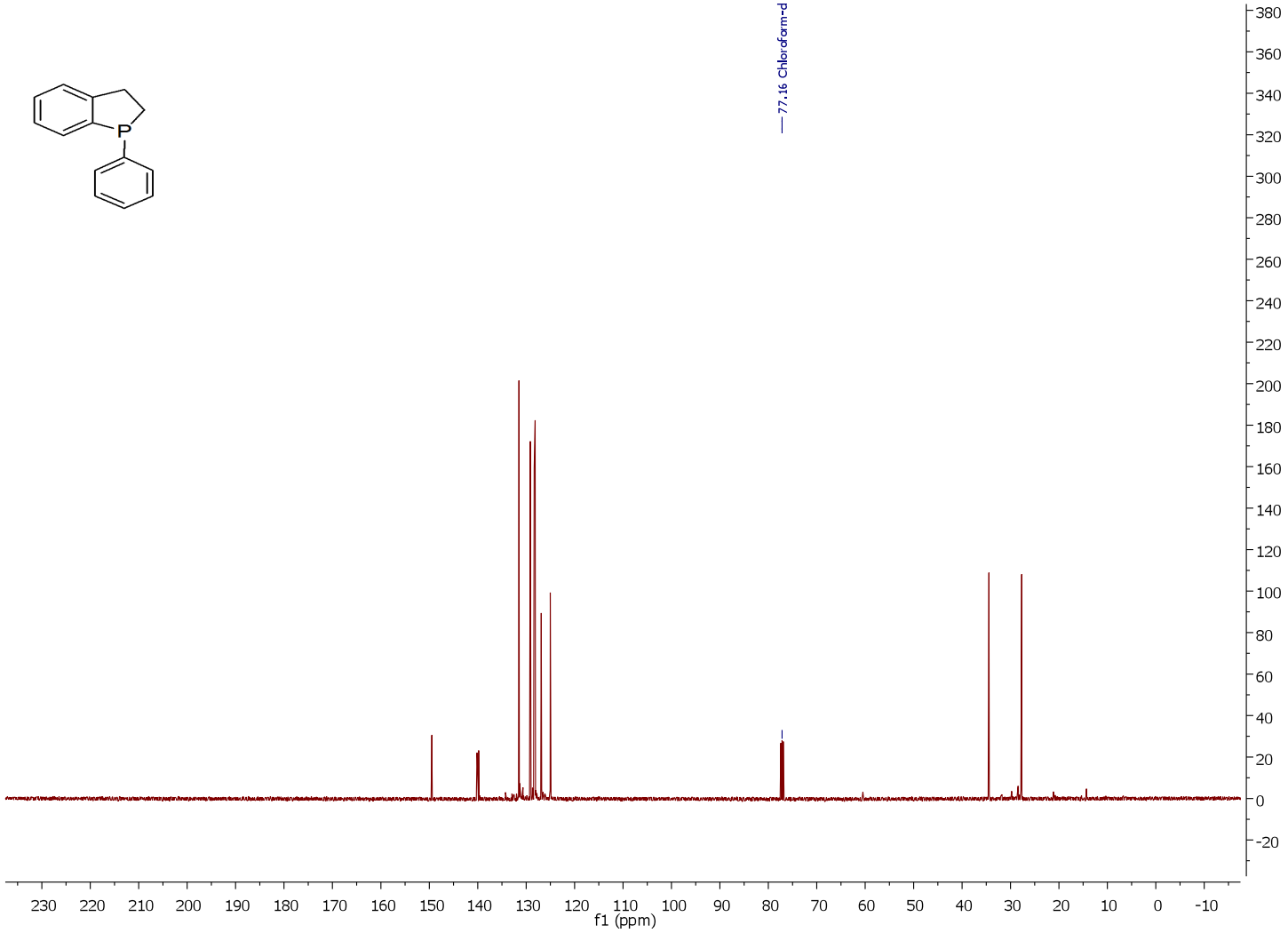
^{31}P NMR 1-Phenyl-2,3-dihydrobenzo[b]phosphole-1-oxide (oxo-1)



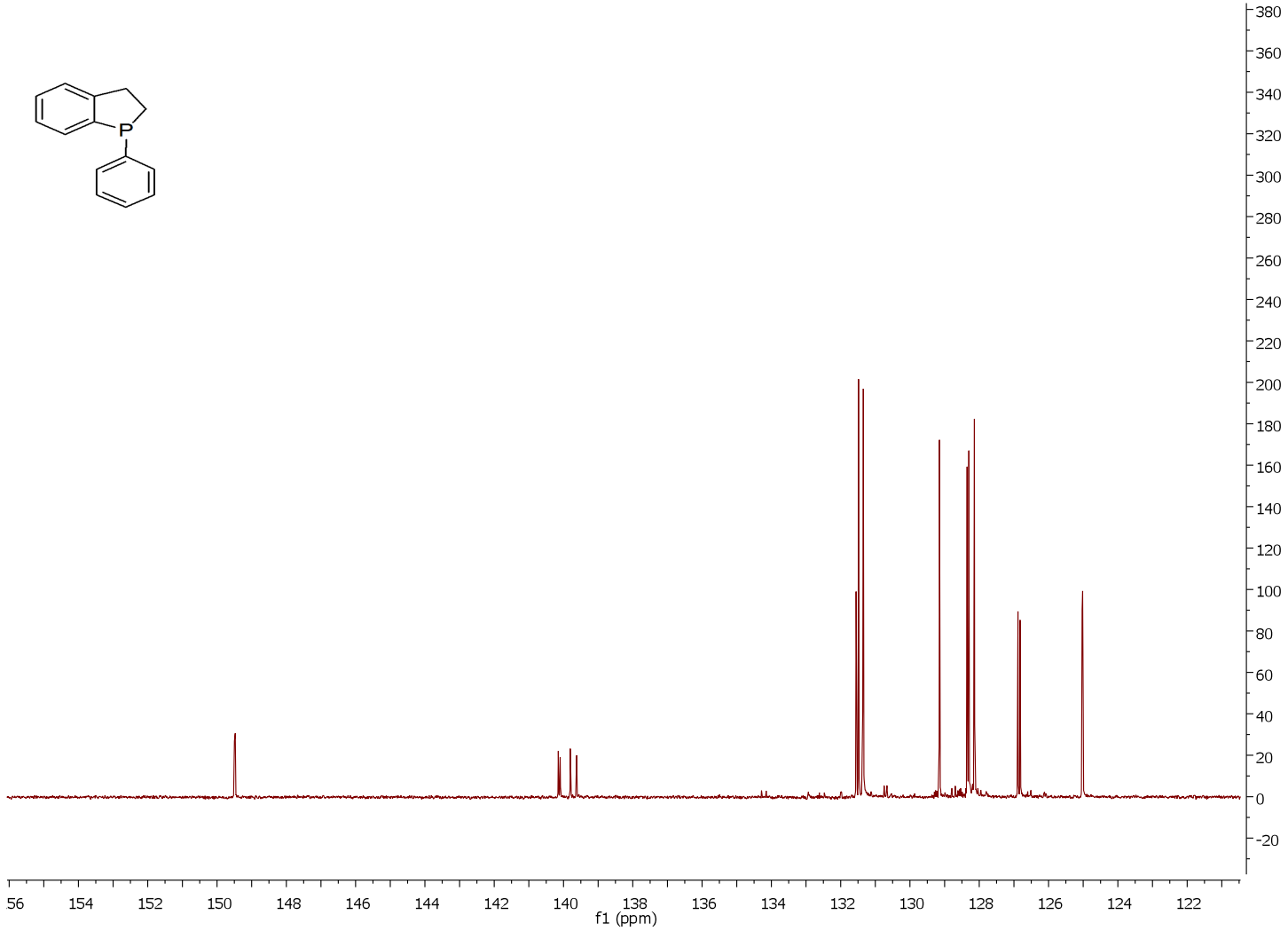
^{1}H NMR 1-Phenyl-2,3,dihydrobenzo[b]phosphole (1)



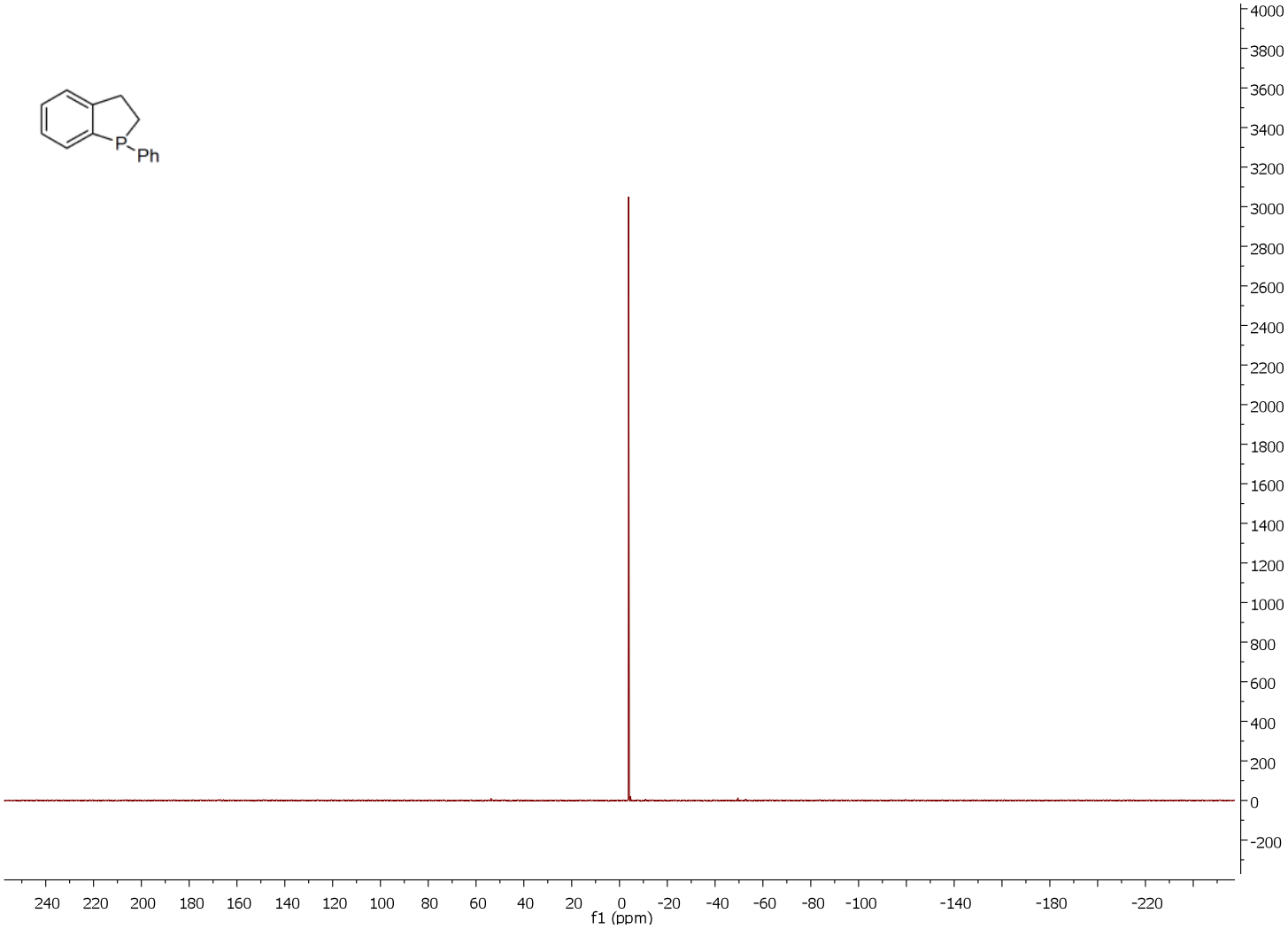
^{13}C NMR 1-Phenyl-2,3,dihydrobenzo[b]phosphole (1)



Expansion ^{13}C NMR 1-Phenyl-2,3,dihydrobenzo[b]phosphole (1)

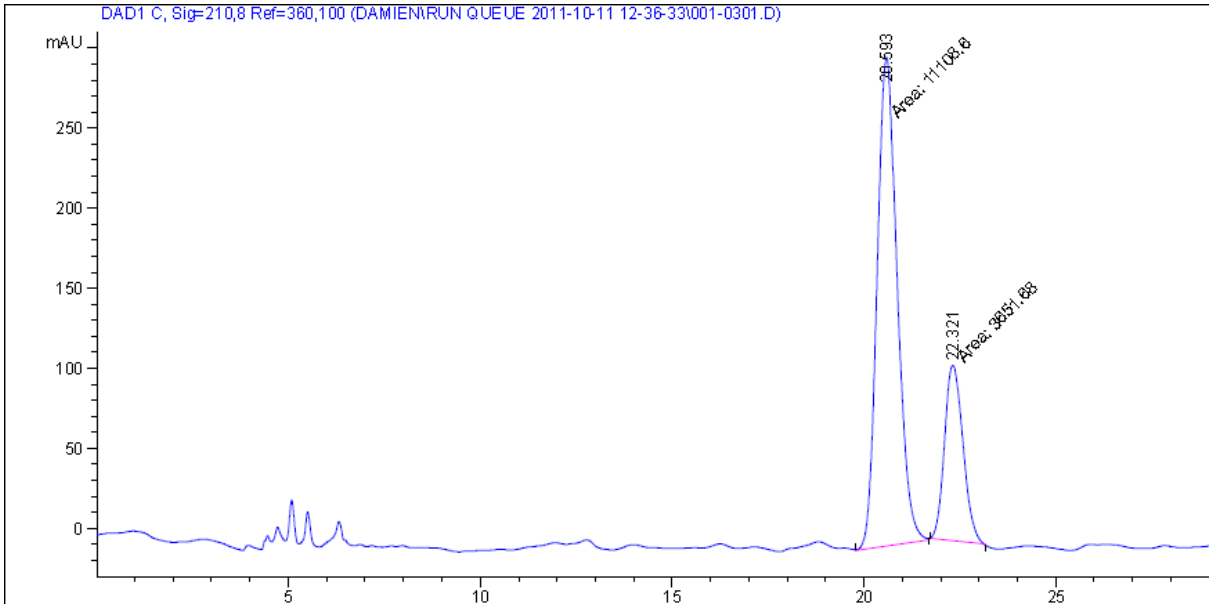


^{31}P NMR 1-Phenyl-2,3,dihydrobenzo[b]phosphole (1)



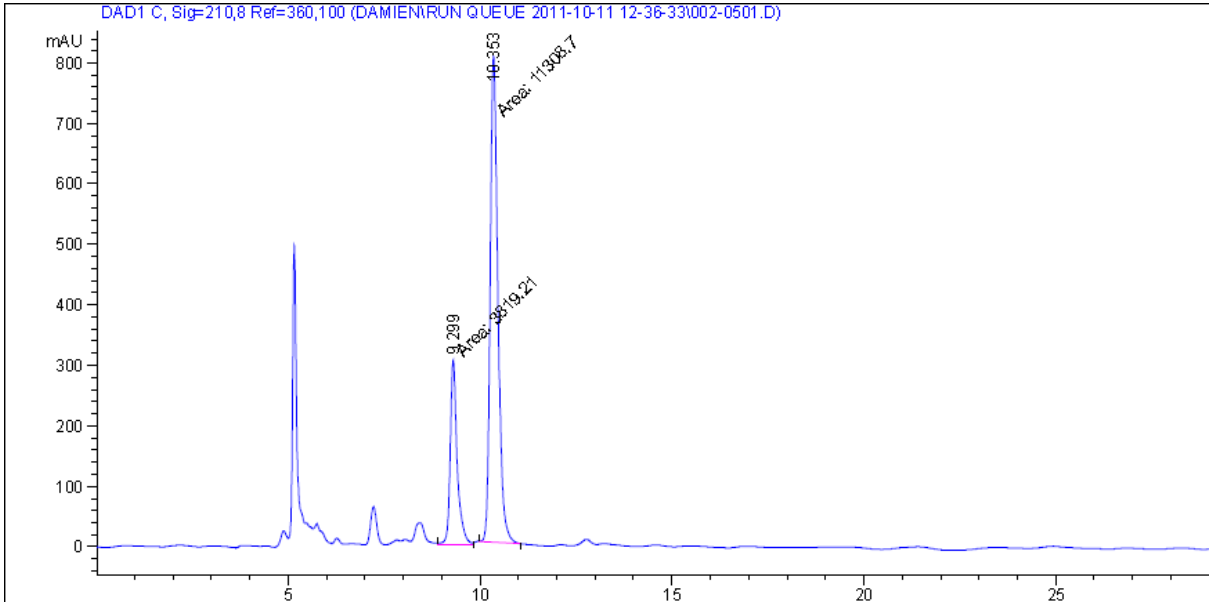
HPLC Traces for Table 1:

Entry 1: Phosphine 1 at 0 °C



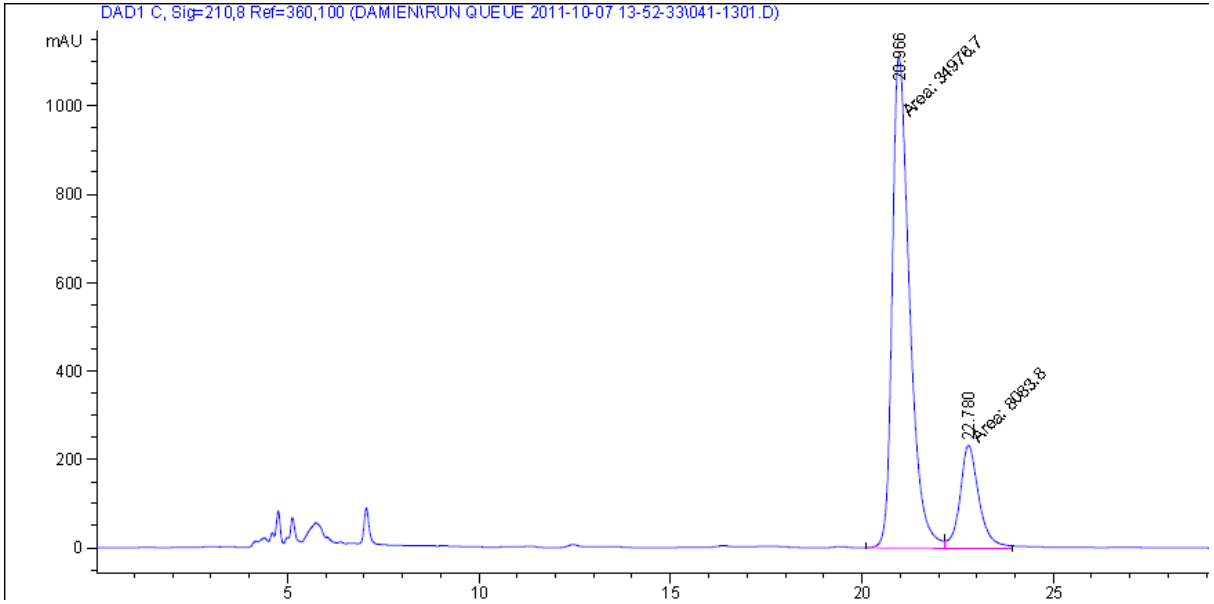
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.593	MM	0.6069	1.11086e4	305.05478	75.2601
2	22.321	MM	0.5564	3651.68018	109.38067	24.7399

Entry 1: Phosphine 2 at 0 °C



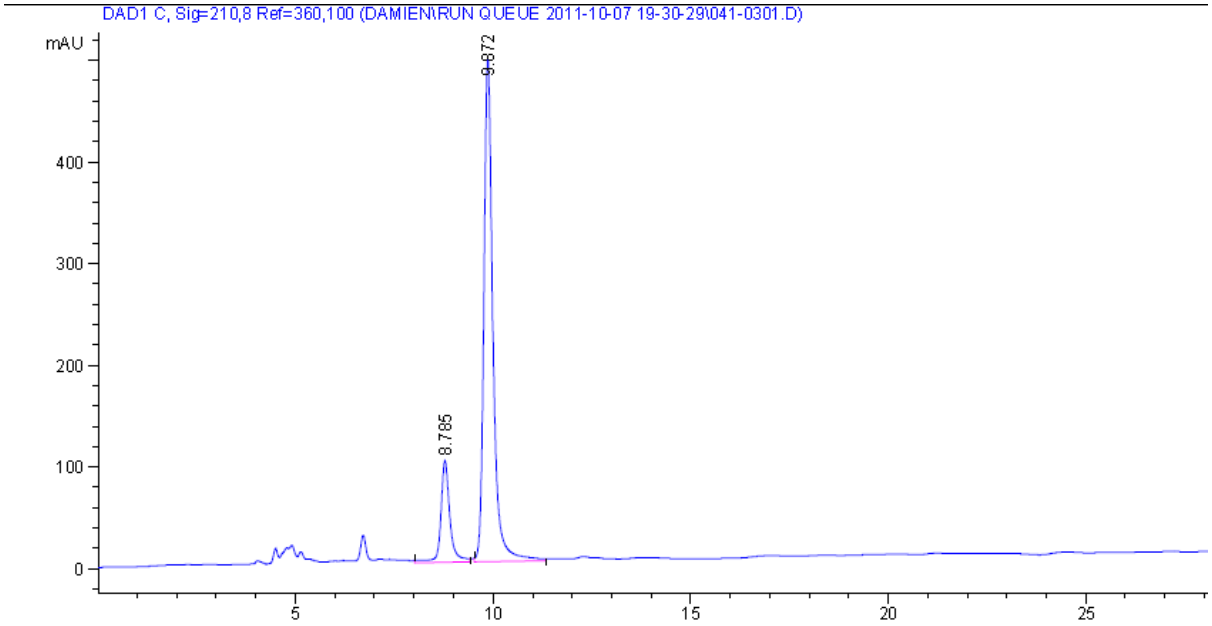
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.299	MM	0.2090	3819.20703	304.56198	25.2461
2	10.353	MM	0.2344	1.13087e4	804.21619	74.7539

Entry 2: Phosphine 1 at -44 °C



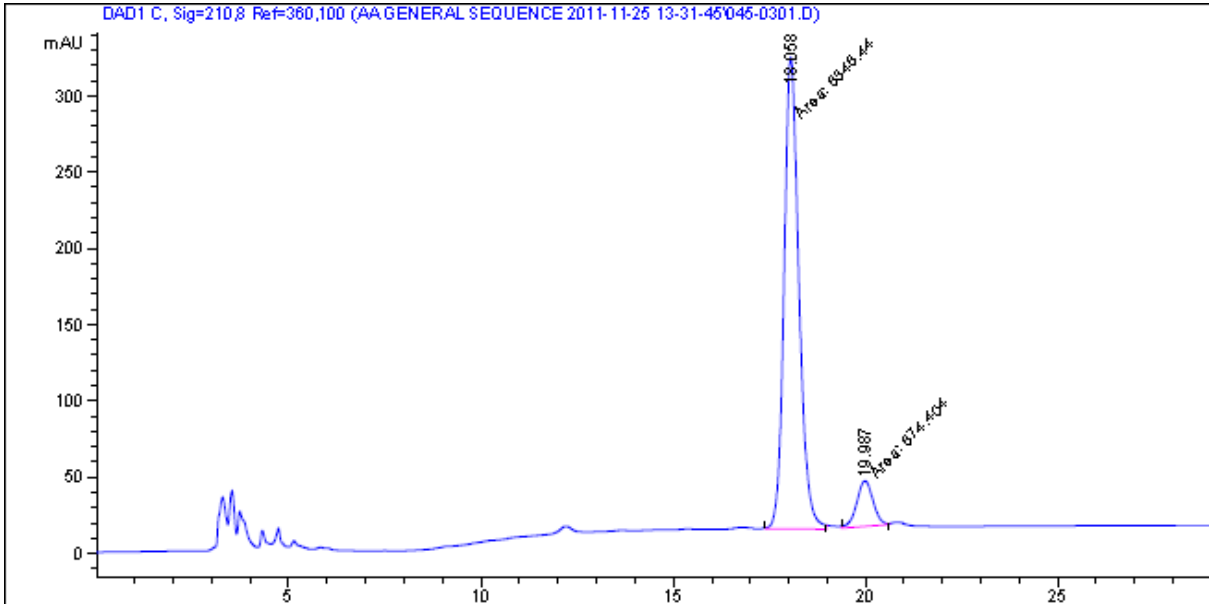
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.966	MM	0.5236	3.49767e4	1113.41882	81.2269
2	22.780	MM	0.5773	8083.79736	233.39604	18.7731

Entry 2: Phosphine 2 at -44 °C



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.785	BB	0.2370	1624.24207	100.27585	17.0574
2	9.872	BB	0.2383	7897.96680	494.70871	82.9426

Entry 3: Phosphine 1 at -80 °C



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.058	MM	0.4505	8348.44434	308.88425	90.5192
2	19.987	MM	0.4893	874.40393	29.78119	9.4808

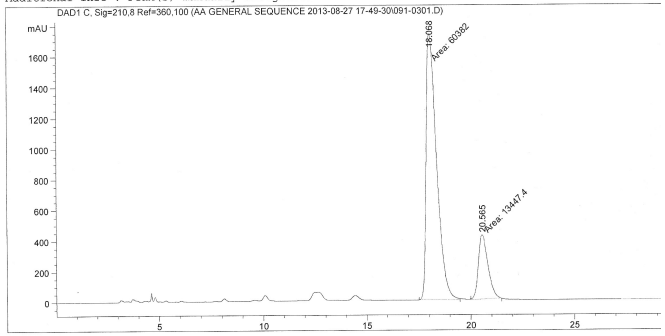
Control: Phosphine 1 at -80 °C (-)-menthol

Data File C:\CHEM32\1\DATA\AA GENERAL SEQUENCE 2013-08-27 17-49-30\091-0301.D
Sample Name: DC429b_dil

==========

Acq. Operator : Seq. Line : 3
Acq. Instrument : HPLC1 Location : Vial 91
Injection Date : 8/27/2013 6:03:43 PM Inj : 1
Inj Volume : 5.000 µl
Acq. Method : C:\CHEM32\1\DATA\AA GENERAL SEQUENCE 2013-08-27 17-49-30\ISO_90_10_30MIN_1MLMIN.M
Last changed : 11/4/2011 10:19:41 AM
Analysis Method : C:\CHEM32\1\DATA\AA GENERAL SEQUENCE 2013-08-27 17-49-30\091-0301.D\DA.M (ISO_90_10_30MIN_1MLMIN.M, From Data File)
Last changed : 11/4/2011 10:19:41 AM
Method Info : Isocratic at 90/10 heptane/EtOH for 30min at 1ml/min

Additional Info : Peak(s) manually integrated



==========

Area Percent Report

==========

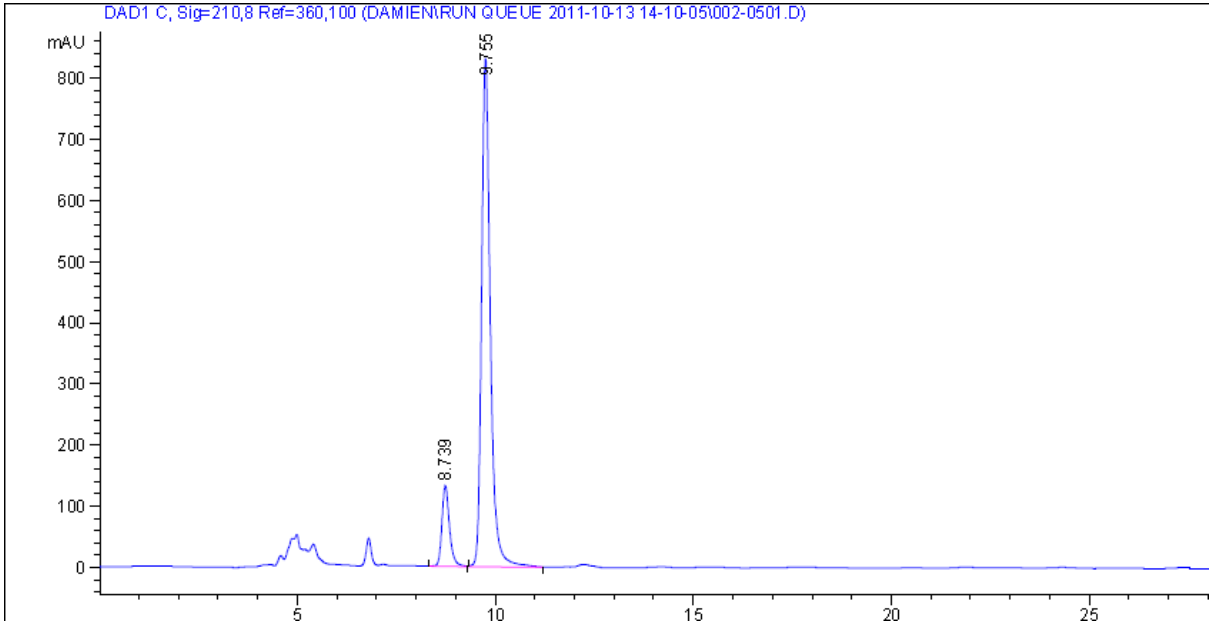
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.068	MM	0.5816	6.03820e4	1730.43127	81.7858
2	20.565	MM	0.5348	1.34474e4	419.05539	18.2142

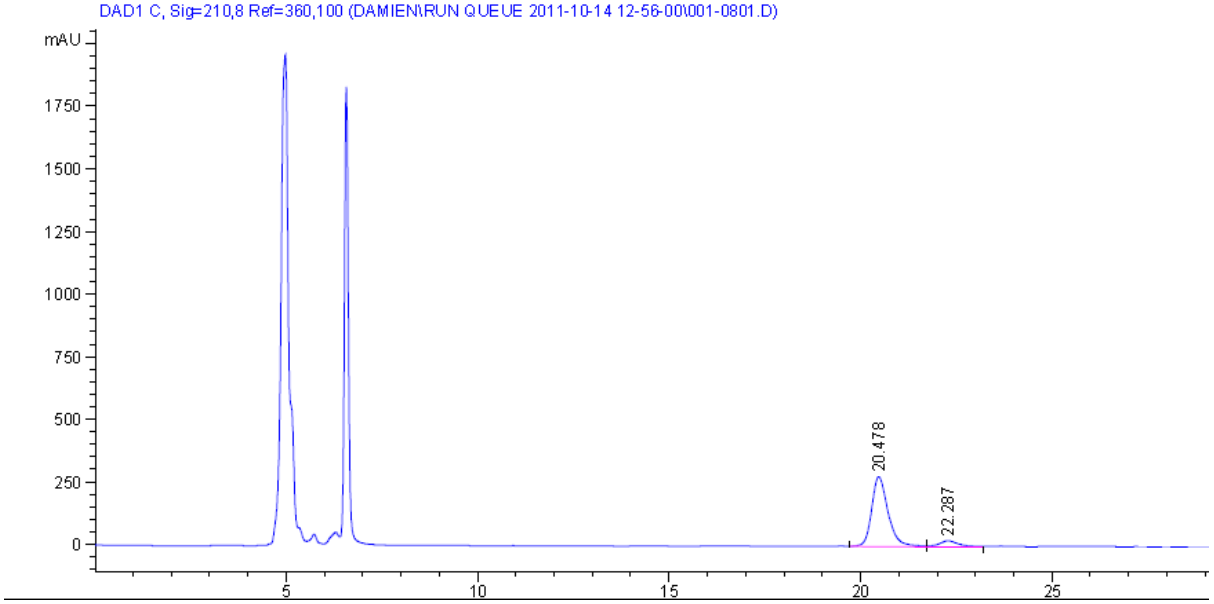
HPLC1 8/27/2013 6:35:36 PM Page 1 of 2

Entry 3: Phosphine 2 at -80 °C



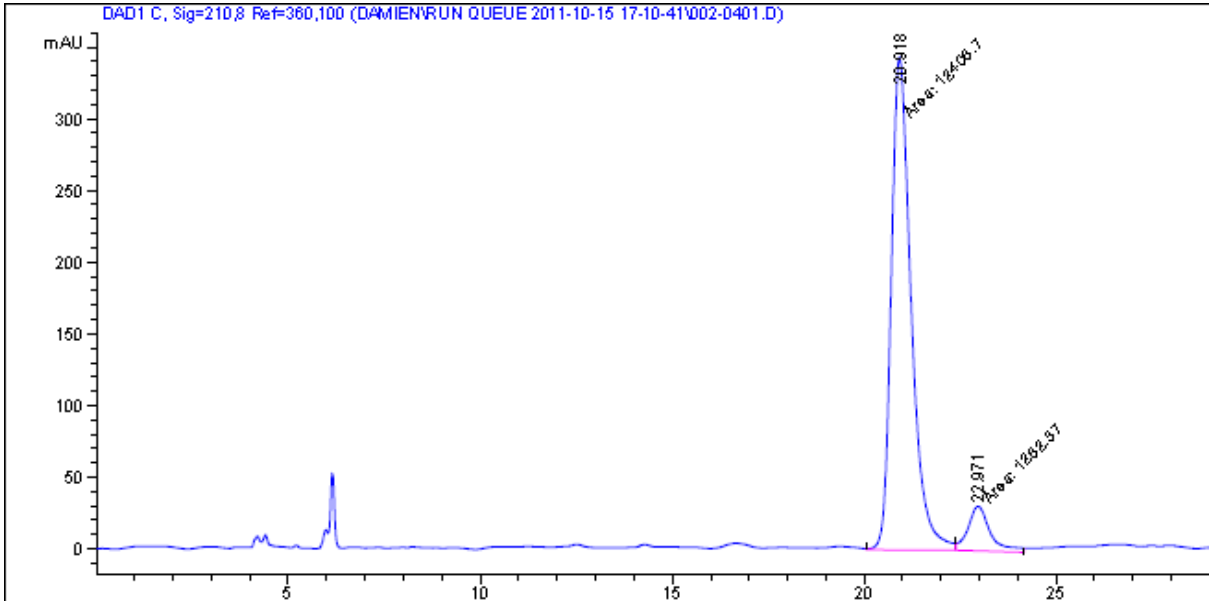
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.739	BB	0.2042	1765.17432	131.81430	12.0489
2	9.755	BB	0.2327	1.28849e4	831.84174	87.9511

Entry 4: Phosphine 1 at -80 °C (-)-8-Phenylmenthol



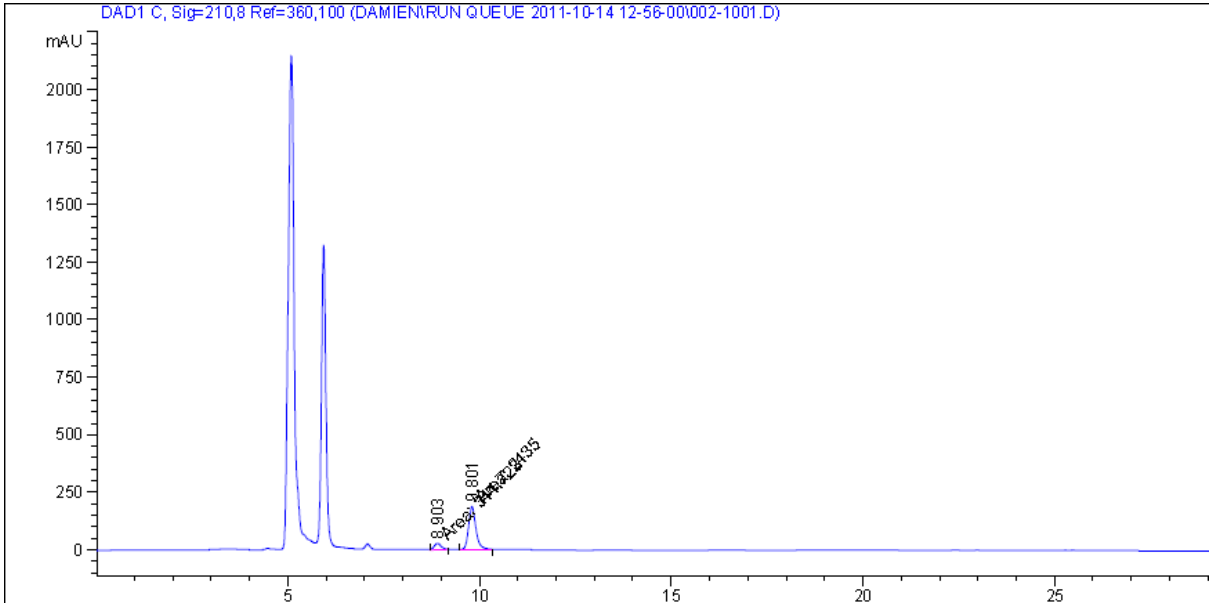
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.478	VV	0.4583	8389.41895	279.38412	90.9907
2	22.287	VV	0.5219	830.66254	23.64412	9.0093

Entry 4: Phosphine 1 at -80 °C (after purification)



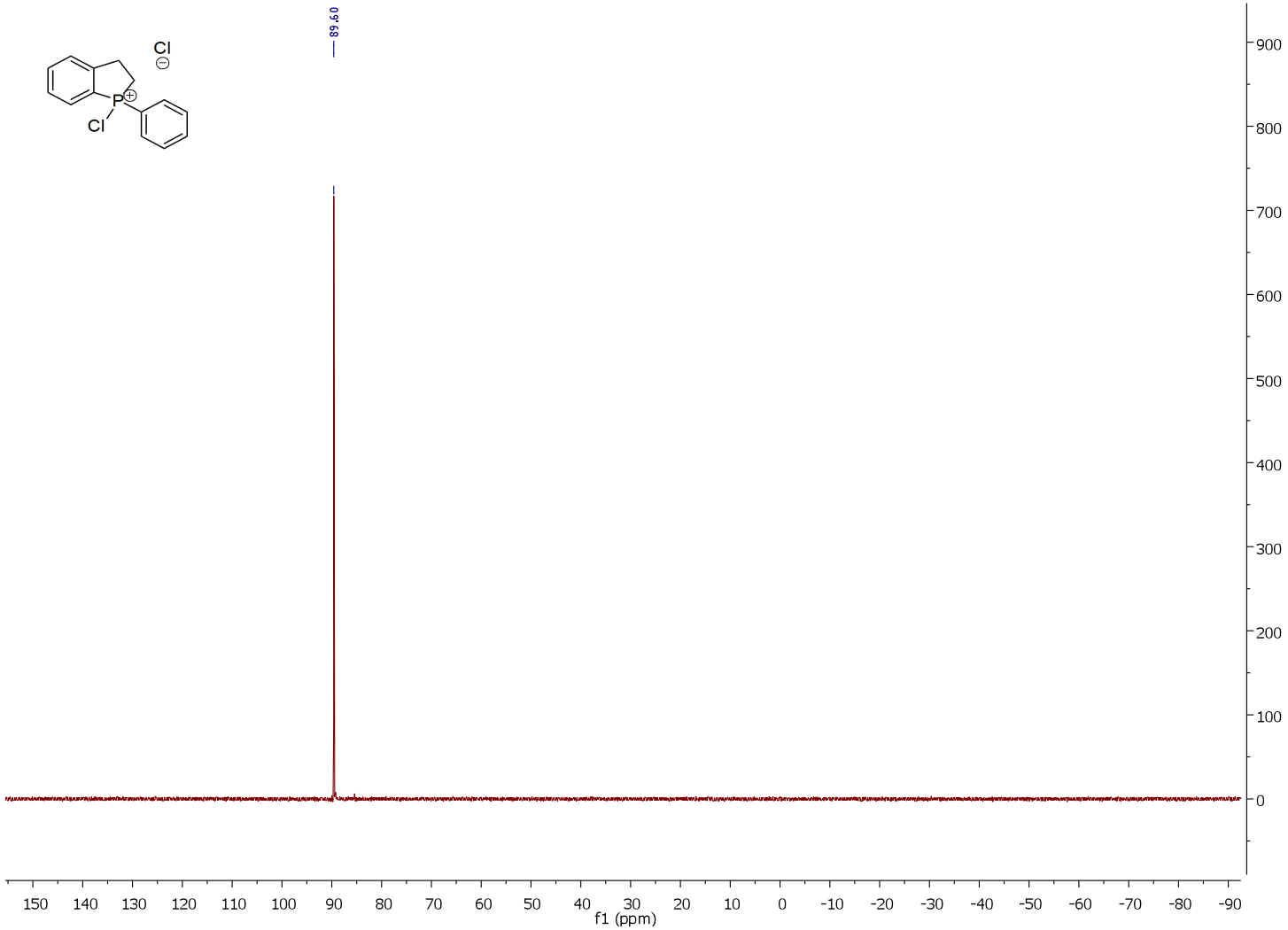
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.918	MM	0.6014	1.24087e4	343.90021	90.7661
2	22.971	MM	0.6736	1262.36682	31.23292	9.2339

Entry 4: Phosphine 2 at -80 °C (-)-8-Phenylmenthol

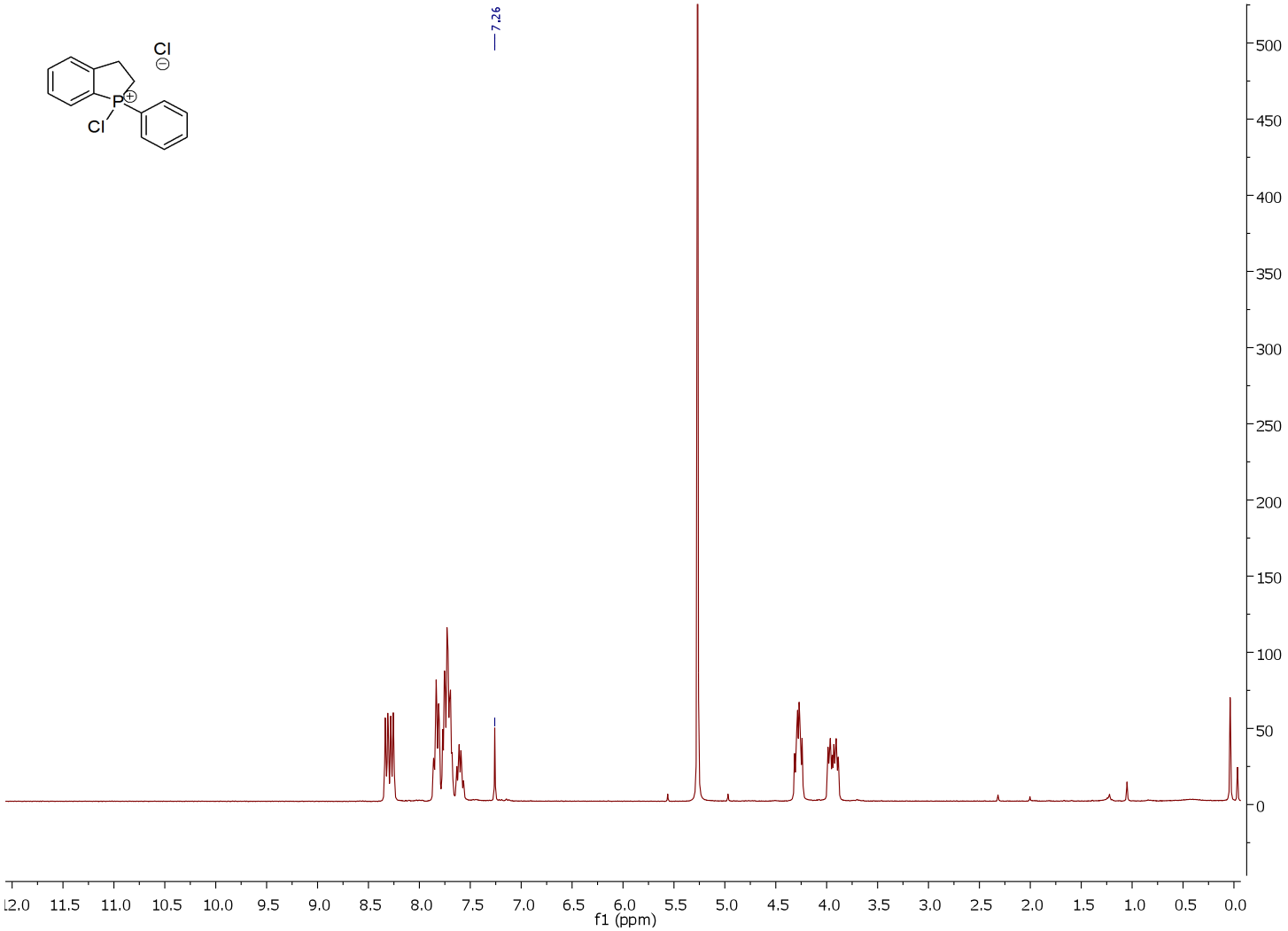


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.903	MM	0.2081	344.72330	27.60316	12.4014
2	9.801	MM	0.2171	2434.99854	186.93300	87.5986

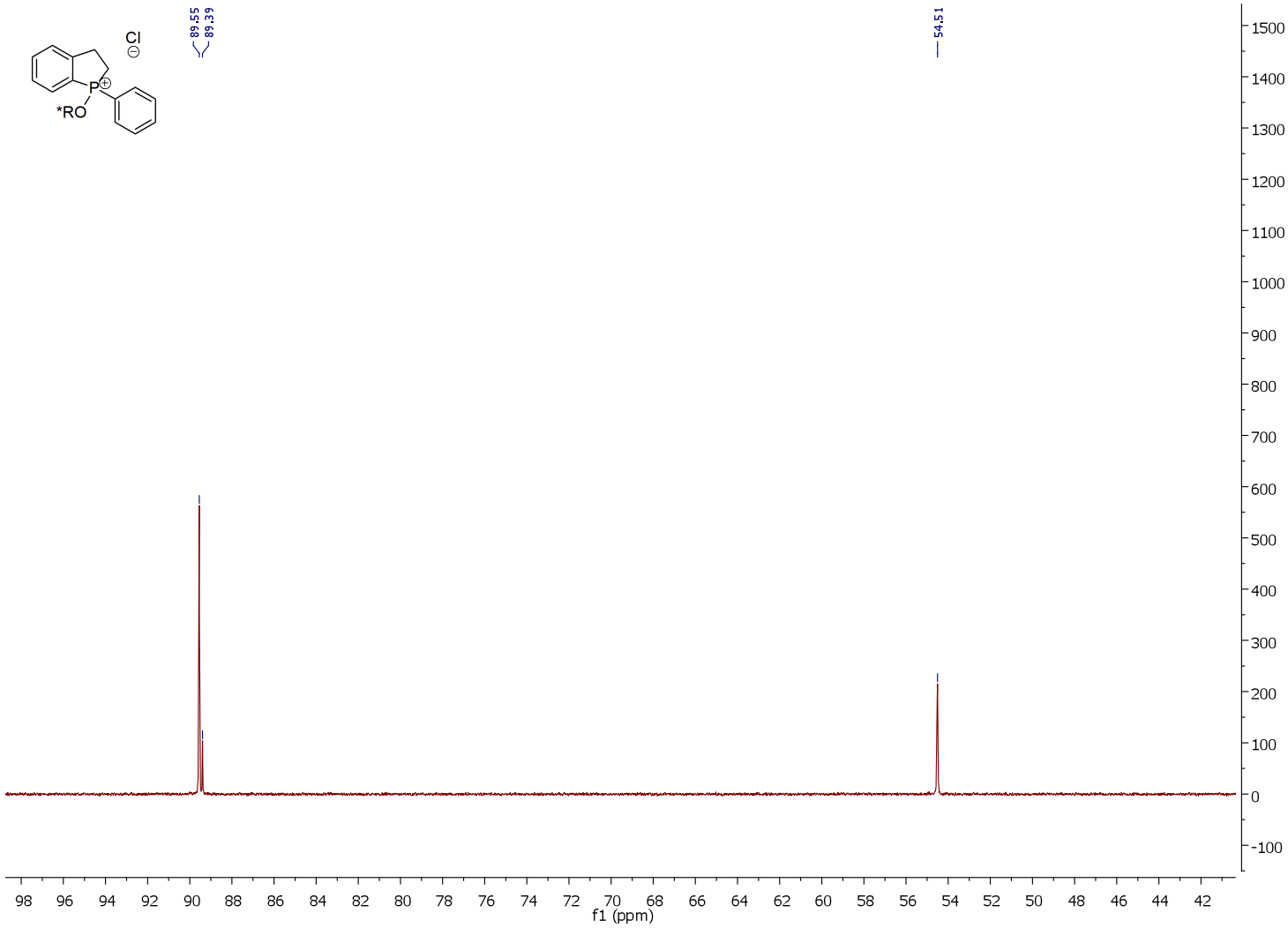
^{31}P NMR of Chlorophosphonium Salt (CPS) derived from 1-Phenyl-2,3,dihydrobenzo[b]phosphole



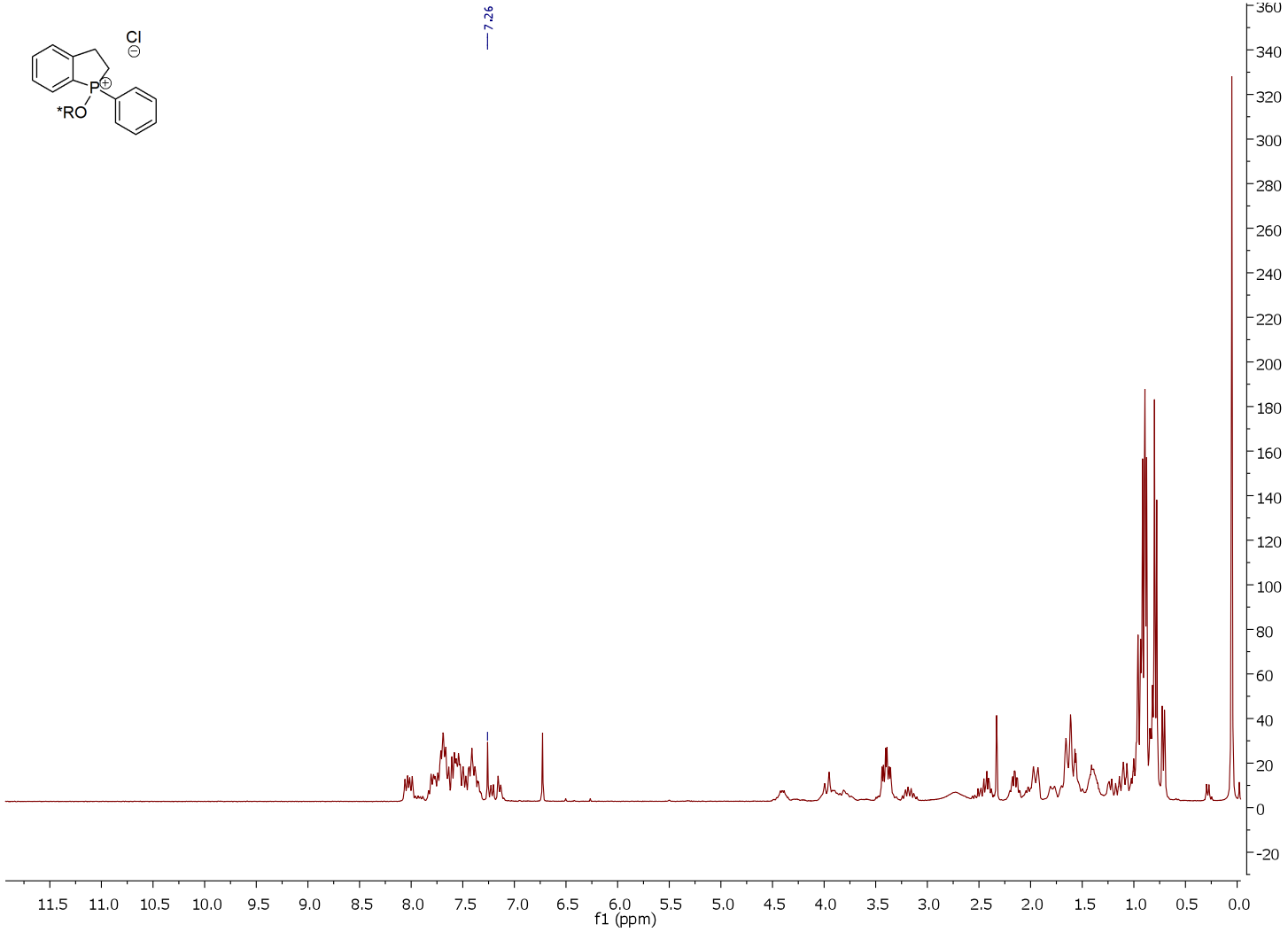
^{1}H NMR of Chlorophosphonium Salt (CPS) derived from 1-Phenyl-2,3,dihydrobenzo[b]phosphole



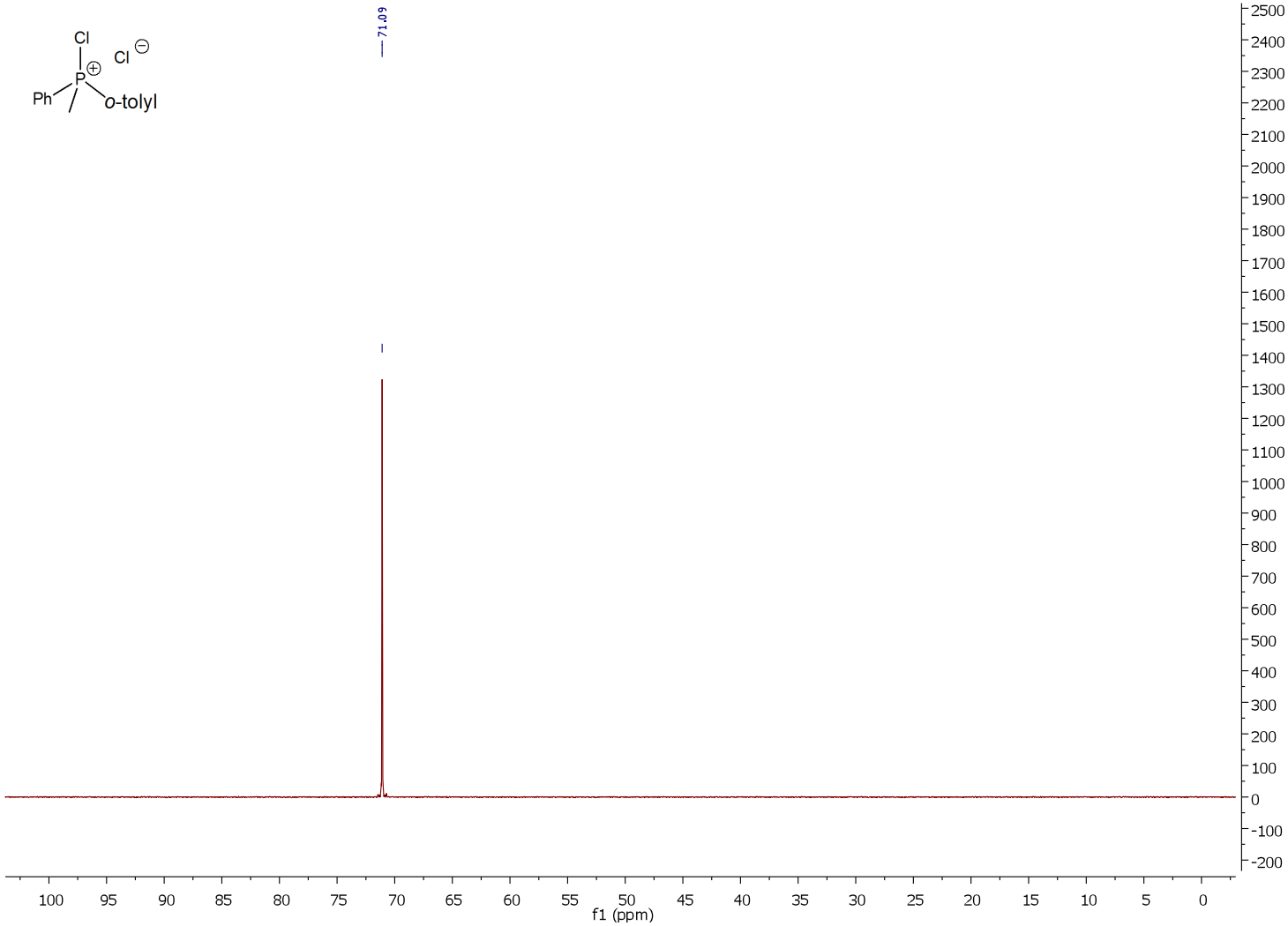
^{31}P NMR of Alkoxyphosphonium Salt (DAPS) with oxide formation (54 ppm) derived from 1-Phenyl-2,3,dihydrobenzo[b]phosphole, HCA and (-)-8-Phenylmenthol



^{1}H NMR of Alkoxyphosphonium Salt (DAPS) derived from 1-Phenyl-2,3,dihydrobenzo[b]phosphole, HCA and (-)-8-Phenylmenthol



^{31}P NMR of Chlorophosphonium Salt (CPS) derived from methylphenyl(o-tolyl)phosphine



^{31}P NMR of Alkoxyphosphonium Salt (DAPS) derived from methylphenyl(o-tolyl)phosphine, HCA and (-)-menthol

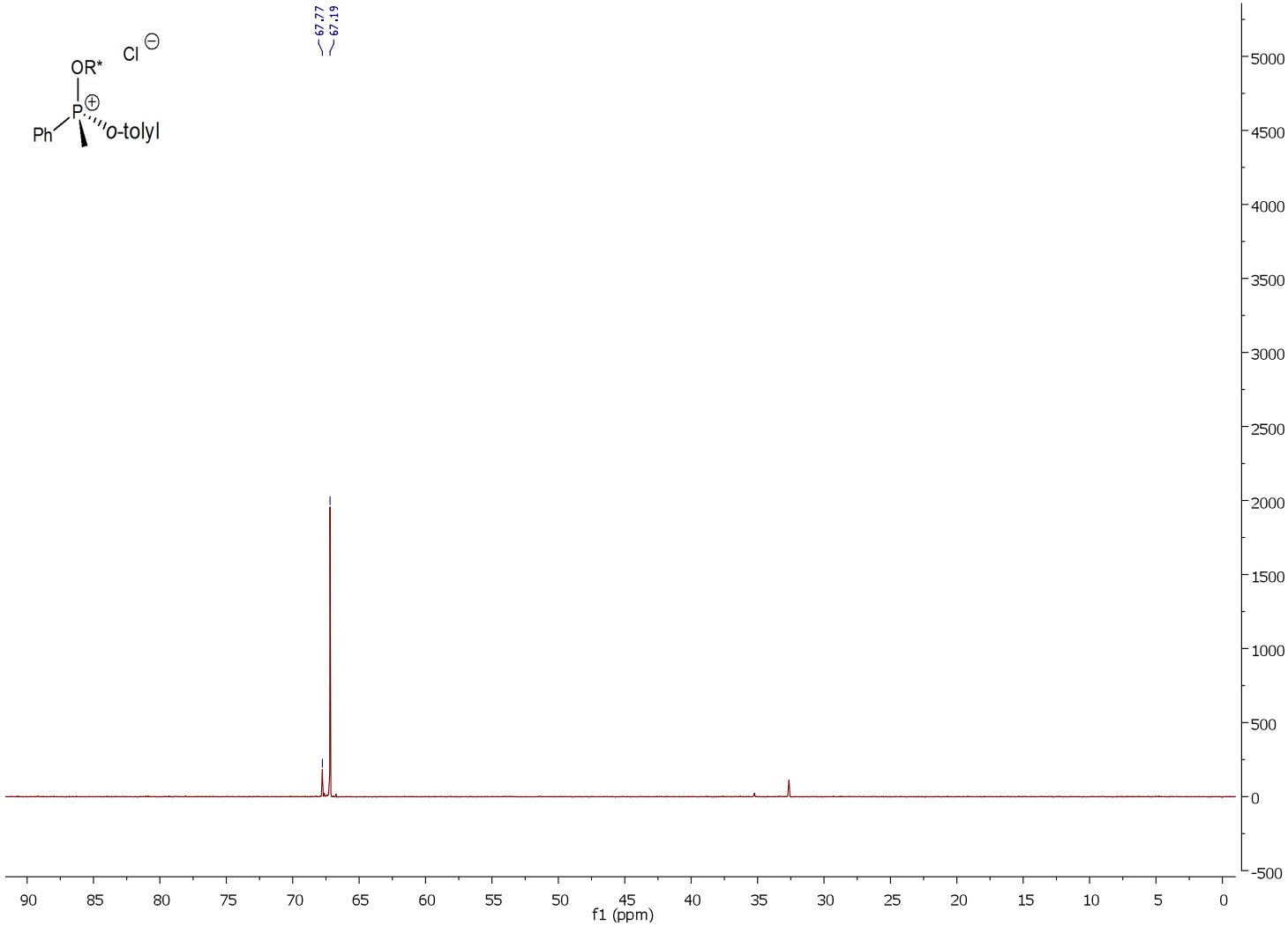
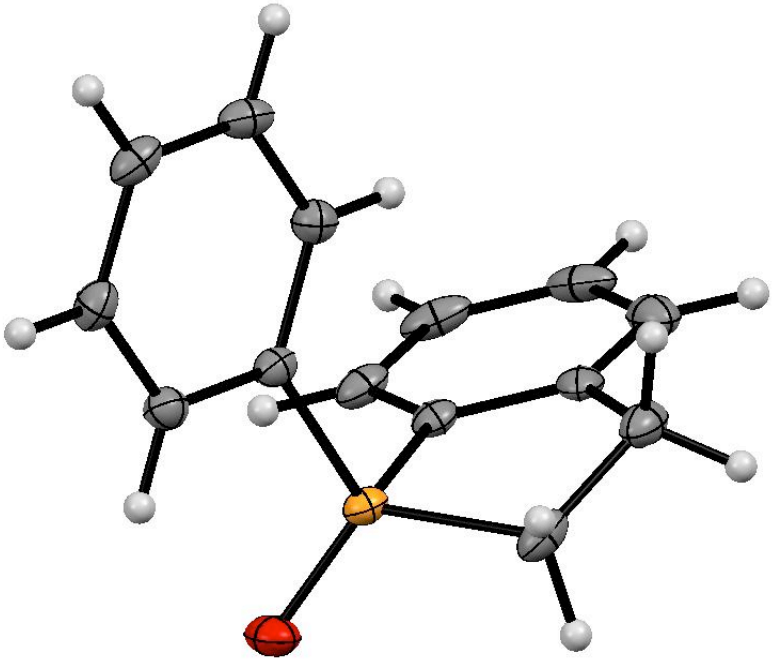


Figure 2. ORTEP diagram: (*R*)-2,3-dihydro-1-phenylbenzo[b]phosphole-1-oxide (oxo-1)



Crystallography Data For (*R*)-^{31}P NMR 1-Phenyl-2,3-dihydrobenzo[b]phosphole-1-oxide (oxo-1)

Table 1. Crystal data and structure refinement for DC264(mason).

Identification code	gil69	
Empirical formula	C14 H13 O P	
Formula weight	228.21	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 21	
Unit cell dimensions	a = 8.3822(2) Å	α= 90°.
	b = 10.6016(2) Å	β= 90.375(2)°.
	c = 12.8429(3) Å	γ = 90°.
Volume	1141.26(4) Å^3	
Z	4	
Density (calculated)	1.328 Mg/m^3	
Absorption coefficient	0.214 mm^{-1}	
F(000)	480	
Crystal size	0.3315 x 0.2919 x 0.2121 mm^3	
Theta range for data collection	2.89 to 29.58°.	
Index ranges	-11<=h<=11, -13<=k<=14, -17<=l<=16	
Reflections collected	12742	
Independent reflections	5291 [R(int) = 0.0296]	
Completeness to theta = 27.00°	99.3 %	
Absorption correction	Analytical	

Max. and min. transmission	0.971 and 0.955
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5291 / 1 / 289
Goodness-of-fit on F^2	1.062
Final R indices [I>2sigma(I)]	R1 = 0.0314, wR2 = 0.0680
R indices (all data)	R1 = 0.0344, wR2 = 0.0704
Absolute structure parameter	-0.01(6)
Largest diff. peak and hole	0.223 and -0.223 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for DC264(mason). U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
P(1)	2732(1)	6349(1)	4051(1)	14(1)
O(1)	4012(1)	7032(1)	4632(1)	21(1)
C(1)	3195(2)	4911(2)	3372(1)	14(1)
C(2)	2516(2)	4926(2)	2373(1)	16(1)
C(8)	1958(2)	7134(2)	2888(1)	18(1)
C(9)	1081(2)	5997(2)	4905(1)	14(1)
C(3)	2702(2)	3873(2)	1738(1)	19(1)
C(4)	3537(2)	2832(2)	2098(1)	20(1)
C(5)	4232(2)	2837(2)	3090(1)	19(1)
C(6)	4060(2)	3875(2)	3731(1)	17(1)
C(7)	1596(2)	6099(2)	2083(1)	20(1)
C(10)	928(2)	6712(2)	5817(1)	18(1)
C(11)	-323(2)	6484(2)	6492(1)	21(1)
C(12)	-1430(2)	5548(2)	6269(1)	21(1)
C(13)	-1283(2)	4830(2)	5373(1)	21(1)
C(14)	-25(2)	5047(2)	4695(1)	18(1)
P(2)	5959(1)	9235(1)	2010(1)	16(1)
O(2)	4441(1)	9847(1)	1676(1)	23(1)
C(15)	6190(2)	7582(2)	1763(1)	16(1)
C(16)	6844(2)	6979(2)	2635(1)	19(1)

C(21)	7219(2)	7832(2)	3553(1)	23(1)
C(22)	6296(2)	9072(2)	3397(1)	21(1)
C(17)	7088(2)	5683(2)	2597(2)	26(1)
C(18)	6711(2)	5019(2)	1698(2)	29(1)
C(19)	6061(2)	5625(2)	830(2)	26(1)
C(20)	5798(2)	6918(2)	863(1)	21(1)
C(23)	7684(2)	10006(2)	1449(1)	16(1)
C(24)	9137(2)	9388(2)	1332(1)	19(1)
C(25)	10450(2)	10020(2)	926(1)	21(1)
C(26)	10295(2)	11270(2)	623(1)	21(1)
C(27)	8854(2)	11896(2)	734(1)	21(1)
C(28)	7548(2)	11263(2)	1153(1)	19(1)

Table 3. Bond lengths [Å] and angles [°] for DC264(mason).

P(1)-O(1)	1.4903(12)
P(1)-C(1)	1.7989(17)
P(1)-C(9)	1.8098(17)
P(1)-C(8)	1.8253(17)
C(1)-C(6)	1.393(2)
C(1)-C(2)	1.401(2)
C(2)-C(3)	1.392(2)
C(2)-C(7)	1.509(2)
C(8)-C(7)	1.537(2)
C(8)-H(8A)	0.9900
C(8)-H(8B)	0.9900
C(9)-C(14)	1.394(2)
C(9)-C(10)	1.402(2)
C(3)-C(4)	1.385(3)
C(3)-H(3)	0.9500
C(4)-C(5)	1.398(3)
C(4)-H(4)	0.9500
C(5)-C(6)	1.383(2)
C(5)-H(5)	0.9500
C(6)-H(6)	0.9500
C(7)-H(7A)	0.9900
C(7)-H(7B)	0.9900
C(10)-C(11)	1.387(2)

C(10)-H(10)	0.9500
C(11)-C(12)	1.388(3)
C(11)-H(11)	0.9500
C(12)-C(13)	1.386(3)
C(12)-H(12)	0.9500
C(13)-C(14)	1.391(2)
C(13)-H(13)	0.9500
C(14)-H(14)	0.9500
P(2)-O(2)	1.4890(13)
P(2)-C(15)	1.7916(18)
P(2)-C(22)	1.8099(17)
P(2)-C(23)	1.8145(17)
C(15)-C(20)	1.391(2)
C(15)-C(16)	1.399(2)
C(16)-C(17)	1.390(3)
C(16)-C(21)	1.518(3)
C(21)-C(22)	1.538(3)
C(21)-H(21A)	0.9900
C(21)-H(21B)	0.9900
C(22)-H(22A)	0.9900
C(22)-H(22B)	0.9900
C(17)-C(18)	1.387(3)
C(17)-H(17)	0.9500
C(18)-C(19)	1.394(3)
C(18)-H(18)	0.9500

C(19)-C(20)	1.389(3)
C(19)-H(19)	0.9500
C(20)-H(20)	0.9500
C(23)-C(28)	1.390(3)
C(23)-C(24)	1.391(2)
C(24)-C(25)	1.392(2)
C(24)-H(24)	0.9500
C(25)-C(26)	1.388(3)
C(25)-H(25)	0.9500
C(26)-C(27)	1.386(3)
C(26)-H(26)	0.9500
C(27)-C(28)	1.396(2)
C(27)-H(27)	0.9500
C(28)-H(28)	0.9500

O(1)-P(1)-C(1)	119.80(8)
O(1)-P(1)-C(9)	110.33(7)
C(1)-P(1)-C(9)	106.70(7)
O(1)-P(1)-C(8)	116.00(8)
C(1)-P(1)-C(8)	93.84(8)
C(9)-P(1)-C(8)	108.71(8)
C(6)-C(1)-C(2)	121.28(15)
C(6)-C(1)-P(1)	128.43(12)
C(2)-C(1)-P(1)	110.28(13)
C(3)-C(2)-C(1)	118.78(16)

C(3)-C(2)-C(7)	125.13(15)
C(1)-C(2)-C(7)	116.07(15)
C(7)-C(8)-P(1)	106.99(12)
C(7)-C(8)-H(8A)	110.3
P(1)-C(8)-H(8A)	110.3
C(7)-C(8)-H(8B)	110.3
P(1)-C(8)-H(8B)	110.3
H(8A)-C(8)-H(8B)	108.6
C(14)-C(9)-C(10)	119.17(15)
C(14)-C(9)-P(1)	122.83(12)
C(10)-C(9)-P(1)	118.00(13)
C(4)-C(3)-C(2)	120.17(16)
C(4)-C(3)-H(3)	119.9
C(2)-C(3)-H(3)	119.9
C(3)-C(4)-C(5)	120.49(16)
C(3)-C(4)-H(4)	119.8
C(5)-C(4)-H(4)	119.8
C(6)-C(5)-C(4)	120.10(17)
C(6)-C(5)-H(5)	120.0
C(4)-C(5)-H(5)	120.0
C(5)-C(6)-C(1)	119.17(16)
C(5)-C(6)-H(6)	120.4
C(1)-C(6)-H(6)	120.4
C(2)-C(7)-C(8)	108.90(14)
C(2)-C(7)-H(7A)	109.9

C(8)-C(7)-H(7A)	109.9
C(2)-C(7)-H(7B)	109.9
C(8)-C(7)-H(7B)	109.9
H(7A)-C(7)-H(7B)	108.3
C(11)-C(10)-C(9)	120.17(16)
C(11)-C(10)-H(10)	119.9
C(9)-C(10)-H(10)	119.9
C(10)-C(11)-C(12)	120.20(16)
C(10)-C(11)-H(11)	119.9
C(12)-C(11)-H(11)	119.9
C(13)-C(12)-C(11)	120.07(16)
C(13)-C(12)-H(12)	120.0
C(11)-C(12)-H(12)	120.0
C(12)-C(13)-C(14)	120.05(17)
C(12)-C(13)-H(13)	120.0
C(14)-C(13)-H(13)	120.0
C(13)-C(14)-C(9)	120.33(16)
C(13)-C(14)-H(14)	119.8
C(9)-C(14)-H(14)	119.8
O(2)-P(2)-C(15)	117.92(8)
O(2)-P(2)-C(22)	116.93(8)
C(15)-P(2)-C(22)	93.69(8)
O(2)-P(2)-C(23)	111.81(8)
C(15)-P(2)-C(23)	106.43(8)
C(22)-P(2)-C(23)	108.30(8)

C(20)-C(15)-C(16)	121.54(17)
C(20)-C(15)-P(2)	128.11(14)
C(16)-C(15)-P(2)	110.35(13)
C(17)-C(16)-C(15)	118.74(18)
C(17)-C(16)-C(21)	125.91(18)
C(15)-C(16)-C(21)	115.35(16)
C(16)-C(21)-C(22)	107.82(14)
C(16)-C(21)-H(21A)	110.1
C(22)-C(21)-H(21A)	110.1
C(16)-C(21)-H(21B)	110.1
C(22)-C(21)-H(21B)	110.1
H(21A)-C(21)-H(21B)	108.5
C(21)-C(22)-P(2)	106.62(12)
C(21)-C(22)-H(22A)	110.4
P(2)-C(22)-H(22A)	110.4
C(21)-C(22)-H(22B)	110.4
P(2)-C(22)-H(22B)	110.4
H(22A)-C(22)-H(22B)	108.6
C(18)-C(17)-C(16)	119.9(2)
C(18)-C(17)-H(17)	120.1
C(16)-C(17)-H(17)	120.1
C(17)-C(18)-C(19)	121.15(19)
C(17)-C(18)-H(18)	119.4
C(19)-C(18)-H(18)	119.4
C(20)-C(19)-C(18)	119.48(19)

C(20)-C(19)-H(19)	120.3
C(18)-C(19)-H(19)	120.3
C(19)-C(20)-C(15)	119.21(19)
C(19)-C(20)-H(20)	120.4
C(15)-C(20)-H(20)	120.4
C(28)-C(23)-C(24)	119.48(16)
C(28)-C(23)-P(2)	118.41(13)
C(24)-C(23)-P(2)	122.10(13)
C(23)-C(24)-C(25)	120.59(17)
C(23)-C(24)-H(24)	119.7
C(25)-C(24)-H(24)	119.7
C(26)-C(25)-C(24)	119.41(17)
C(26)-C(25)-H(25)	120.3
C(24)-C(25)-H(25)	120.3
C(27)-C(26)-C(25)	120.60(16)
C(27)-C(26)-H(26)	119.7
C(25)-C(26)-H(26)	119.7
C(26)-C(27)-C(28)	119.68(17)
C(26)-C(27)-H(27)	120.2
C(28)-C(27)-H(27)	120.2
C(23)-C(28)-C(27)	120.23(16)
C(23)-C(28)-H(28)	119.9
C(27)-C(28)-H(28)	119.9

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x 10^3$) for DC264(mason). The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
P(1)	13(1)	15(1)	14(1)	-1(1)	2(1)	0(1)
O(1)	17(1)	25(1)	21(1)	-5(1)	2(1)	-4(1)
C(1)	12(1)	17(1)	13(1)	0(1)	4(1)	-1(1)
C(2)	11(1)	20(1)	16(1)	2(1)	2(1)	-3(1)
C(8)	19(1)	18(1)	17(1)	3(1)	3(1)	1(1)
C(9)	14(1)	14(1)	14(1)	3(1)	0(1)	3(1)
C(3)	18(1)	24(1)	14(1)	-2(1)	2(1)	-5(1)
C(4)	18(1)	21(1)	22(1)	-4(1)	6(1)	-4(1)
C(5)	16(1)	17(1)	24(1)	1(1)	5(1)	1(1)
C(6)	14(1)	22(1)	15(1)	1(1)	2(1)	-1(1)
C(7)	18(1)	25(1)	18(1)	1(1)	-2(1)	2(1)
C(10)	17(1)	19(1)	16(1)	0(1)	-1(1)	1(1)
C(11)	26(1)	24(1)	15(1)	-2(1)	4(1)	5(1)
C(12)	20(1)	23(1)	21(1)	5(1)	7(1)	3(1)
C(13)	17(1)	19(1)	27(1)	2(1)	4(1)	-1(1)
C(14)	17(1)	20(1)	18(1)	-1(1)	2(1)	1(1)
P(2)	13(1)	18(1)	16(1)	-2(1)	3(1)	-1(1)
O(2)	14(1)	25(1)	31(1)	1(1)	3(1)	2(1)
C(15)	12(1)	18(1)	18(1)	0(1)	4(1)	-4(1)
C(16)	10(1)	26(1)	20(1)	3(1)	3(1)	0(1)

C(21)	16(1)	35(1)	17(1)	1(1)	1(1)	-3(1)
C(22)	22(1)	26(1)	15(1)	-3(1)	4(1)	-9(1)
C(17)	18(1)	25(1)	35(1)	8(1)	7(1)	3(1)
C(18)	20(1)	20(1)	47(1)	-4(1)	17(1)	0(1)
C(19)	21(1)	28(1)	30(1)	-13(1)	14(1)	-10(1)
C(20)	16(1)	28(1)	18(1)	-4(1)	5(1)	-6(1)
C(23)	14(1)	20(1)	14(1)	-2(1)	1(1)	-2(1)
C(24)	17(1)	19(1)	19(1)	-1(1)	1(1)	0(1)
C(25)	15(1)	27(1)	21(1)	-7(1)	3(1)	-1(1)
C(26)	20(1)	26(1)	17(1)	-5(1)	4(1)	-8(1)
C(27)	25(1)	20(1)	19(1)	2(1)	3(1)	-3(1)
C(28)	17(1)	21(1)	17(1)	-1(1)	1(1)	1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^{-3}) for DC264(mason).

	x	y	z	U(eq)
H(8A)	975	7609	3054	22
H(8B)	2758	7732	2612	22
H(3)	2255	3869	1057	22
H(4)	3638	2108	1666	24
H(5)	4824	2126	3324	23
H(6)	4525	3882	4407	20
H(7A)	438	5915	2071	24
H(7B)	1911	6387	1380	24
H(10)	1683	7354	5972	21
H(11)	-422	6970	7110	26
H(12)	-2290	5399	6731	25
H(13)	-2042	4189	5221	25
H(14)	82	4545	4087	22
H(21A)	8379	8002	3591	27
H(21B)	6890	7423	4211	27
H(22A)	6925	9794	3665	25
H(22B)	5267	9043	3770	25
H(17)	7512	5253	3186	31
H(18)	6899	4136	1673	35

H(19)	5800	5158	221	32
H(20)	5357	7344	277	25
H(24)	9233	8528	1530	22
H(25)	11444	9598	859	25
H(26)	11184	11700	337	25
H(27)	8757	12753	525	25
H(28)	6563	11692	1237	22

Table 6. Torsion angles [°] for DC264(mason).

O(1)-P(1)-C(1)-C(6)	47.16(17)
C(9)-P(1)-C(1)-C(6)	-78.98(16)
C(8)-P(1)-C(1)-C(6)	170.18(15)
O(1)-P(1)-C(1)-C(2)	-134.25(11)
C(9)-P(1)-C(1)-C(2)	99.61(12)
C(8)-P(1)-C(1)-C(2)	-11.23(13)
C(6)-C(1)-C(2)-C(3)	0.7(2)
P(1)-C(1)-C(2)-C(3)	-177.99(12)
C(6)-C(1)-C(2)-C(7)	179.42(15)
P(1)-C(1)-C(2)-C(7)	0.71(18)
O(1)-P(1)-C(8)-C(7)	143.85(11)
C(1)-P(1)-C(8)-C(7)	17.91(13)
C(9)-P(1)-C(8)-C(7)	-91.17(12)
O(1)-P(1)-C(9)-C(14)	-158.85(13)
C(1)-P(1)-C(9)-C(14)	-27.20(16)
C(8)-P(1)-C(9)-C(14)	72.91(15)
O(1)-P(1)-C(9)-C(10)	20.69(15)
C(1)-P(1)-C(9)-C(10)	152.33(13)
C(8)-P(1)-C(9)-C(10)	-107.56(14)
C(1)-C(2)-C(3)-C(4)	0.4(2)
C(7)-C(2)-C(3)-C(4)	-178.18(16)
C(2)-C(3)-C(4)-C(5)	-1.5(3)
C(3)-C(4)-C(5)-C(6)	1.5(3)

C(4)-C(5)-C(6)-C(1)	-0.4(3)
C(2)-C(1)-C(6)-C(5)	-0.7(2)
P(1)-C(1)-C(6)-C(5)	177.72(13)
C(3)-C(2)-C(7)-C(8)	-168.49(15)
C(1)-C(2)-C(7)-C(8)	12.9(2)
P(1)-C(8)-C(7)-C(2)	-19.82(16)
C(14)-C(9)-C(10)-C(11)	-0.9(2)
P(1)-C(9)-C(10)-C(11)	179.56(13)
C(9)-C(10)-C(11)-C(12)	0.0(3)
C(10)-C(11)-C(12)-C(13)	0.5(3)
C(11)-C(12)-C(13)-C(14)	0.0(3)
C(12)-C(13)-C(14)-C(9)	-0.9(3)
C(10)-C(9)-C(14)-C(13)	1.4(2)
P(1)-C(9)-C(14)-C(13)	-179.11(14)
O(2)-P(2)-C(15)-C(20)	43.51(18)
C(22)-P(2)-C(15)-C(20)	166.72(16)
C(23)-P(2)-C(15)-C(20)	-82.97(17)
O(2)-P(2)-C(15)-C(16)	-136.11(12)
C(22)-P(2)-C(15)-C(16)	-12.89(13)
C(23)-P(2)-C(15)-C(16)	97.41(13)
C(20)-C(15)-C(16)-C(17)	-0.7(3)
P(2)-C(15)-C(16)-C(17)	178.94(14)
C(20)-C(15)-C(16)-C(21)	179.54(15)
P(2)-C(15)-C(16)-C(21)	-0.81(18)
C(17)-C(16)-C(21)-C(22)	-162.36(18)

C(15)-C(16)-C(21)-C(22)	17.4(2)
C(16)-C(21)-C(22)-P(2)	-25.16(16)
O(2)-P(2)-C(22)-C(21)	146.07(11)
C(15)-P(2)-C(22)-C(21)	22.08(13)
C(23)-P(2)-C(22)-C(21)	-86.57(13)
C(15)-C(16)-C(17)-C(18)	1.2(3)
C(21)-C(16)-C(17)-C(18)	-179.08(16)
C(16)-C(17)-C(18)-C(19)	-1.1(3)
C(17)-C(18)-C(19)-C(20)	0.6(3)
C(18)-C(19)-C(20)-C(15)	0.0(3)
C(16)-C(15)-C(20)-C(19)	0.1(3)
P(2)-C(15)-C(20)-C(19)	-179.45(14)
O(2)-P(2)-C(23)-C(28)	24.76(16)
C(15)-P(2)-C(23)-C(28)	154.83(13)
C(22)-P(2)-C(23)-C(28)	-105.48(14)
O(2)-P(2)-C(23)-C(24)	-156.85(13)
C(15)-P(2)-C(23)-C(24)	-26.77(16)
C(22)-P(2)-C(23)-C(24)	72.91(16)
C(28)-C(23)-C(24)-C(25)	0.3(2)
P(2)-C(23)-C(24)-C(25)	-178.08(13)
C(23)-C(24)-C(25)-C(26)	-1.0(3)
C(24)-C(25)-C(26)-C(27)	0.9(3)
C(25)-C(26)-C(27)-C(28)	-0.1(3)
C(24)-C(23)-C(28)-C(27)	0.6(2)
P(2)-C(23)-C(28)-C(27)	179.00(13)

C(26)-C(27)-C(28)-C(23)	-0.7(3)

Symmetry transformations used to generate equivalent atoms: