Supporting Information for:

A Ni-PyBisulidine Complex for the Asymmetric Hydrophosphonylation of Aldehydes

Youmao Zeng,[a‡] Ping Deng,[a‡] Shixiong Zhang,[a] Hong Yan,[a] Guojuan Liang,[a] Yan Xiong[b] and Hui Zhou*[a]

hzhou@cqmu.edu.cn

[a] *School of Pharmaceutical Science, Chongqing Medical University, Chongqing, China*

[b] *School of Chemistry and Chemical Engineering, Chongqing University, Chongqing, China*

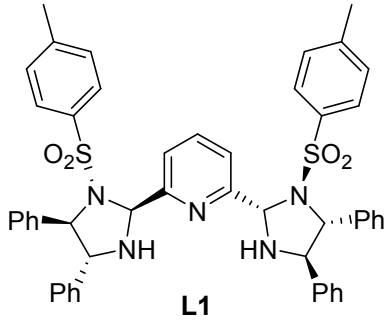
Contents:

1. General information

All reagents were obtained from Adamas, Aladin, TCI, or Acros etc. without further purification unless otherwise noted. High resolution mass spectra were measured on commercial instruments. NMR spectra were recorded on commercial instruments and operating at 500 MHz for ^{1}H NMR and 125 MHz for ^{13}C NMR. Chemical shifts were reported in ppm from tetramethylsilane with the solvent resonance as the internal standard ($CDCl_3$, δ = 7.26) in ^{1}H NMR spectra and Chemical shifts were reported in ppm from the tetramethylsilane with the solvent resonance as internal standard ($CDCl_3$, δ = 77.0) in ^{13}C NMR spectra. Spectra are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz), integration, and assignment. The enantiomeric excess (*ee*) was determined by HPLC analysis. Analytical HPLC was performed on a Shimadzu liquid chromatography (LC-16), using a chiral DAICEL CHIRALCEL OD-H or CHIRALPAK AS-H or CHIRALPAK AD-H column at 210 nm. Optical rotations were measured on a commercial polarimeter and are reported as follows: $[\alpha]_D^T$ (c = g/100 mL, solvent).

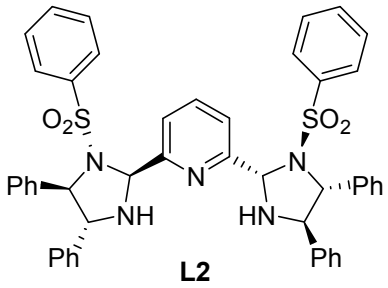
2. The preparation and characterization of the ligands

Ligands **L1**-**L4** were prepared according to the literature.[1]



L1

^{1}HNMR (500MHz, $CDCl_3$): δ 8.02-7.96 (m, 3H), 7.53 (d, *J* = 8.2 Hz, 4H), 7.25-7.17 (m, 4H), 7.16-7.10 (m, 16H), 7.00 (d, *J* = 7.2 Hz, 4H), 5.83 (s, 2H), 4.71 (d, *J* = 5.4 Hz, 2H), 4.26 (d, *J* = 5.4 Hz, 2H), 2.39 (s, 6H).

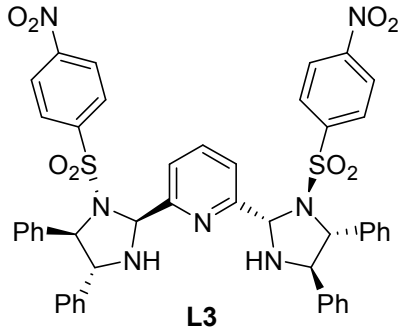


L2

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.99-7.98 (m, 2H), 7.65-7.63 (m, 3H), 7.52-7.49 (m, 2H), 7.35 (t, *J* = 7.9 Hz, 4H), 7.18-7.09 (m, 16H), 6.98-6.97 (m, 4H), 5.88 (s, 2H), 4.71 (d, *J* = 5.8 Hz, 2H), 4.26 (d, *J* = 5.8 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 158.14, 139.51, 139.01, 138.22, 138.08, 128.99, 128.62, 128.37, 127.85, 127.61, 127.10, 126.79, 123.99, 78.30, 71.64, 69.49. HRMS (ESI): *m/z* calcd for $C_{47}H_{41}N_5NaO_4S_2$ $[M + Na]^+$: 826.24922, found

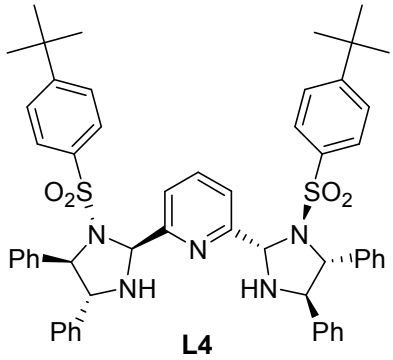
[1] A. Gonzalez-de-Castro, C. M. Robertson, J. Xiao, *J. Am. Chem. Soc.*, **2014**, *136*, 8350-8360.

826.24686.



L3

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.02-7.94 (m, 7H), 7.50 (d, *J* = 8.2 Hz, 4H), 7.20-7.11 (m, 20H), 5.75 (s, 2H), 4.96 (s, 2H), 4.45 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 156.32, 156.07, 149.97, 143.28, 139.37, 139.25, 138.34, 128.84, 128.53, 128.33, 128.16, 125.83, 79.32, 70.67, 68.97. HRMS (ESI): *m/z* calcd for $C_{47}H_{39}N_7NaO_8S_2$ $[M + Na]^+$: 916.21937, found 916.21896.



L4

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.01-7.99 (m, 2H), 7.61 (d, *J* = 8.5 Hz, 4H), 7.37 (d, *J* = 8.6 Hz, 4H), 7.18-7.13 (m, 7H), 7.10-7.06 (m, 10H), 6.96-6.94 (m, 4H), 5.93 (s, 2H), 4.67 (d, *J* = 6.1 Hz, 2H), 4.25 (d, *J* = 6.0 Hz, 2H), 1.34 (s, 18H).

3. General procedures for the preparation of the catalyst

Method A (in situ): The mixture of $Ni(OAc)_2{\cdot}4H_2O$ (0.02 mmol) and ligand (0.02 mmol) was stirred in a certain solvent (0.5 mL) at 35 °C for 1 h.

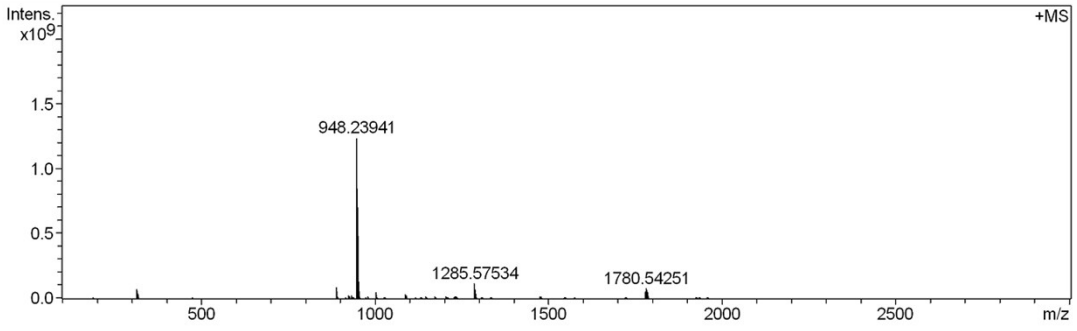
Method B (pre-prepared): The mixture of the **L1** (0.4 mmol) and $Ni(OAc)_2{\cdot}4H_2O$ (0.4 mmol) was stirred in methanol (20 mL) at 80 °C for 4 h. After cooling down to room temperature, the precipitate was collected by filtration. Then, the solid was washed with MeOH and dried under reduced pressure to afford the Ni-**L1** complex as a green solid, 0.36g, 83% yield.

4. ESI-HRMS analysis of Ni-L1 complex and the proposed structure of the complex

In the ESI-HRMS chart of Ni-**L1** complex (Figure S1), the peak corresponding to Ni/L1 was detected. The peak of m/z = 948.23941 was assigned to [**L1** + $Ni(OAc)_2$ –HOAc + H]$^+$ (Calcd. For $C_{51}H_{48}N_5NiO_6S_2$: 948.23940). On the basis of the related structure of Fe-PyBisulidine complex (*J. Am. Chem. Soc.*, **2014**, *136*, 8350-8360) and the geometry of **L1** optimized using Chem3D at MM2 level (Figure S2), we speculated that the ligand could coordinate to the Ni via pyridine nitrogen and secondary amine nitrogens. According to the ESI-HRMS analysis of the complex and the balance of total charge, one proton should removed (Figure S3).

Acquisition Parameter

Polarity	Positive	n/a	n/a	No. of Laser Shots	200
n/a	n/a	No. of Cell Fills	1	Laser Power	20.0 Ip
Broadband Low Mass	150.5 m/z	n/a	n/a	n/a	n/a
Broadband High Mass	3000.0 m/z	n/a	n/a	n/a	n/a
Acquisition Mode	Single MS	n/a	n/a		
Pulse Program	basic	n/a	n/a	Calibration Date	Mon Jul 25 11:54:52 2016
Source Accumulation	0.010 sec	n/a	n/a	Data Acquisition Size	1048576
Ion Accumulation Time	0.020 sec	n/a	n/a	Apodization	Sine-Bell Multiplication
Flight Time to Acq. Cell	0.002 sec				



Meas. m/z	#	Formula	Score	m/z	err [ppm]	Mean err [ppm]	mSig ma	rdb	e⁻ Conf	N-R ule
948.23941	1	C 51 H 48 N 5 Ni O 6 S 2	100.00	948.23940	-0.01	0.38	94.2	30.5	even	ok

Figure S1. The ESI-HRMS chart of Ni-**L1**

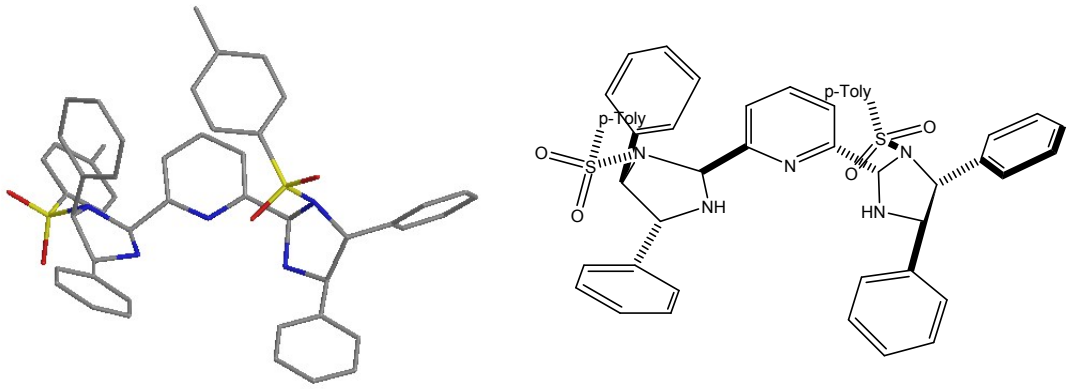
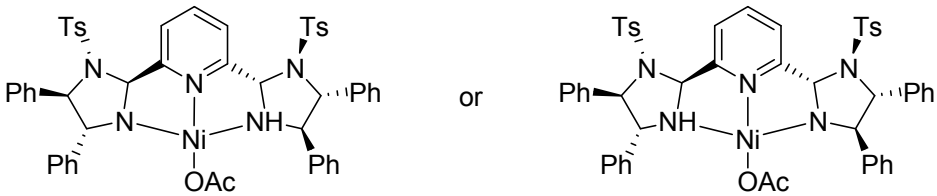


Figure S2. The geometry of **L1** optimized using Chen3D (8.0) at MM2 level

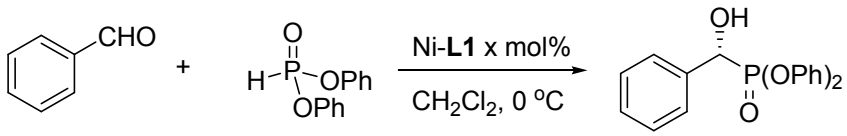


Calcd. For $C_{51}H_{48}N_5NiO_6S_2$ $[M + H]^+$: 948.23940, Found: 948.23941

Figure S3. The proposed structure of Ni-**L1** complex

5. The effects of the catalyst loading and the concentration of benzaldehyde.

Table S1 The effects of the catalyst loading and the concentration of aldehyde. [a]



1a **2a**

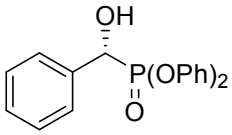
Entry	Time (h)	The loading of Ni-**L1** (mol %)	The concentration of benzaldehyde (M)	Yield (%)[h]	ee (%)[i]
1[b]	17	10	0.1	44	93
2[c]	15	10	0.2	90	93
3[d]	15	10	0.4	98	93
4[d]	17	5	0.4	74	93
5[e]	15	2	1	2	5
6[f]	15	1	2	5	5
7[g]	39	5	0.5	24	85

[a] The catalyst was prepared in *situ*. [b] The reaction was carried out: aldehyde (0.1 mmol), diphenylphosphite (0.1 mmol), 1.0 mL CH_2Cl_2. [c] The reaction was carried out: aldehyde (0.2 mmol), diphenylphosphite (0.2 mmol), 1.0 mL CH_2Cl_2. [d] The reaction was carried out: aldehyde (0.2 mmol), diphenylphosphite (0.2 mmol), 0.5 mL CH_2Cl_2. [e] The reaction was carried out: aldehyde (1.0 mmol), diphenylphosphite (1.0 mmol), 1.0 mL CH_2Cl_2. [f] The reaction was carried out: aldehyde (2.0 mmol), diphenylphosphite (2.0 mmol), 1.0 mL CH_2Cl_2. [g] The reaction was carried out: aldehyde (0.5 mmol), diphenylphosphite (0.5 mmol), 1.0 mL CH_2Cl_2. [h] Isolated yield. [i] Enantiomeric excesses were determined by HPLC analysis.

6. General Procedure for Catalytic Asymmetric Reaction

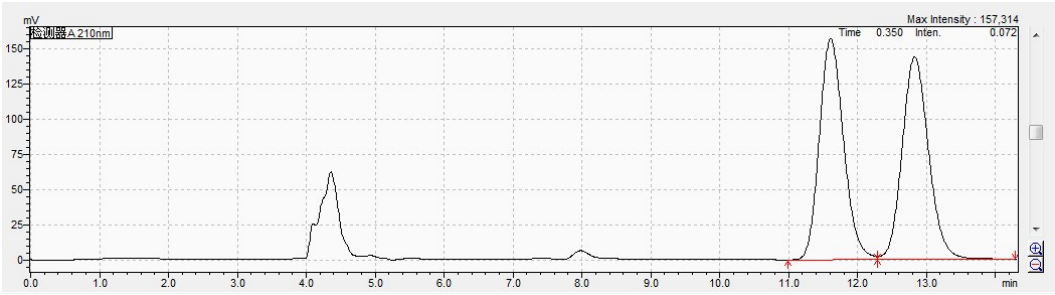
The mixture of diphenylphosphite (0.2 mmol) and the chiral complex (0.02 mmol, 10 mol%) was stirred in CH_2Cl_2 (0.5 mL) at -10 °C for 30 min followed by the addition of the aldehyde (0.2 mmol). The stirring was continued for the time indicated in Table 3 at -10 °C. The residue was purified by silica gel flash column chromatography (petroleum ether / AcOEt, 2:1) to afford the products.

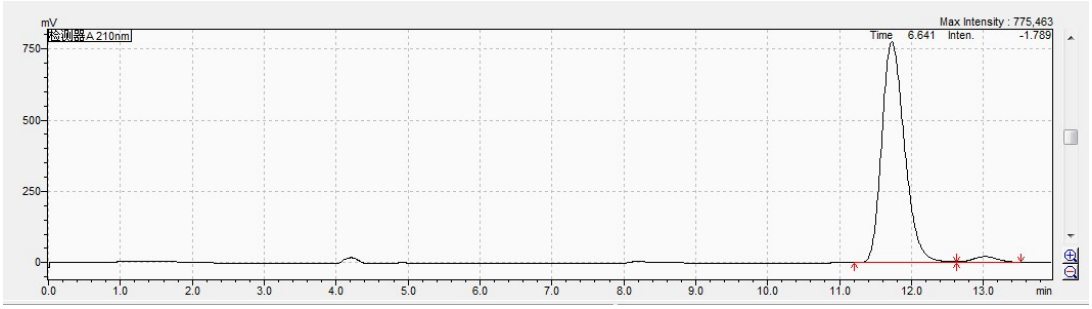
7. Characterization of products



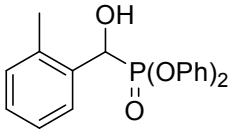
2a

White solid, 62.6 mg, 92% yield, 95% *ee*, ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.57-7.59 (m, 2H), 7.39-7.23 (m, 8H), 7.16-7.13 (m, 2H), 7.06-7.0 (m, 4H), 5.34 (d, J = 8.5 Hz, 1H), 3.54 (brs, 1H). $[\alpha]_D^{25}$ = +38.9 (c 0.80, $CHCl_3$) [lit.[2] $[\alpha]_D^{24}$ = -36.5 (c 1.0, $CHCl_3$) for *S* enantiomer in 91% *ee*]. HPLC (CHIRALCEL OD-H column, hexane / 2-propanol = 90 / 10, flow 0.7 mL/min, detection at 210 nm) t_r (major) = 11.7 min and t_r (minor) = 13.0 min.





Peak#	Retention Time	Area	Area%
1	11.728	17050904	97.334
2	13.022	467056	2.666
Total		17517960	100.000

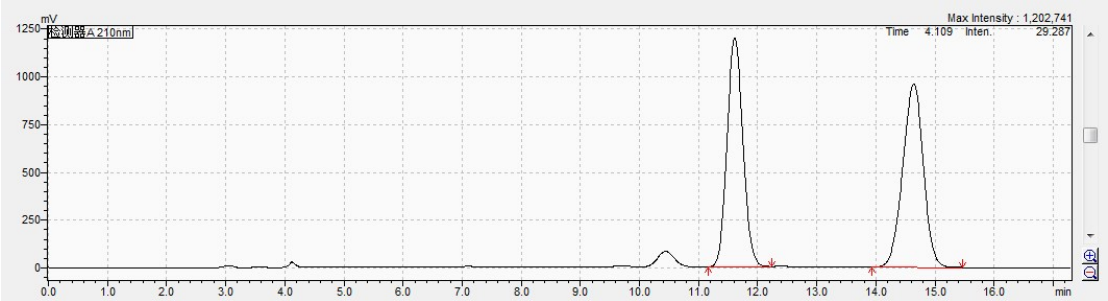


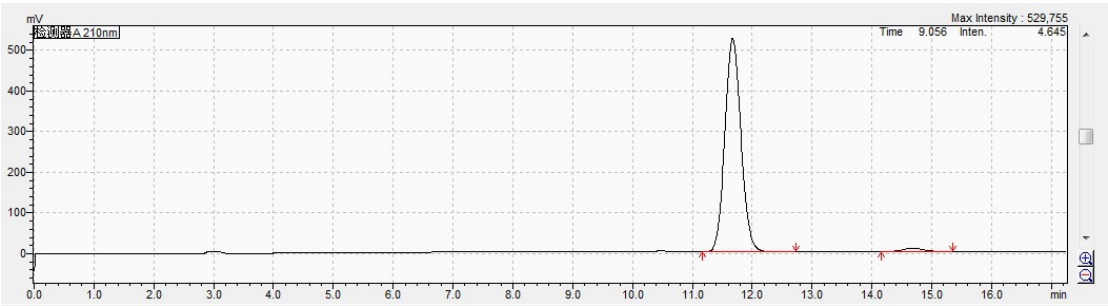
2b

White solid, 65.8 mg, 93% yield, 96% ee, ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.78 (brs, 1H), 7.29-7.22 (m, 8H), 7.18-7.13 (m, 2H), 7.09-7.07 (m, 2H), 7.00-6.99 (m, 2H), 5.63 (d, J = 9.4 Hz, 1H), 3.77 (brs, 1H), 2.45(s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 150.3 (d, J = 10.1 Hz), 136.2 (d, J = 8.0 Hz), 133.9, 130.5, 129.7 (d, J = 5.9 Hz), 128.6 (d, J = 2.1 Hz), 127.6 (d, J = 4.2 Hz), 126.4 (d, J = 1.8 Hz), 125.2 (d, J = 9.3 Hz), 120.6 (d, J = 3.7 Hz), 120.5 (d, J = 3.6 Hz), 67.2 (d, J = 161.8 Hz), 19.6. HRMS (ESI): m/z calcd for $C_{20}H_{19}NaO_4P$ $[M + Na]^+$: 377.09132, found 377.09042. $[\alpha]_D^{25}$ =

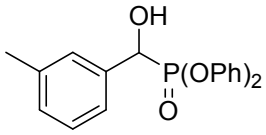
[2] S. Hirashima, R. Arai, K. Nakashima, N. Kawai, J. Kondo, Y. Koseki, T. Miura, *Adv. Synth. Catal.*, **2015**, *357*, 3863-3867.

+63.3 (c 0.41, $CHCl_3$). HPLC (CHIRALPAK AD-H column, hexane / 2-propanol = 80/20, flow 1mL/min, detection at 210 nm) t_r (major) = 11.7 min and t_r (minor) =14.7 min.



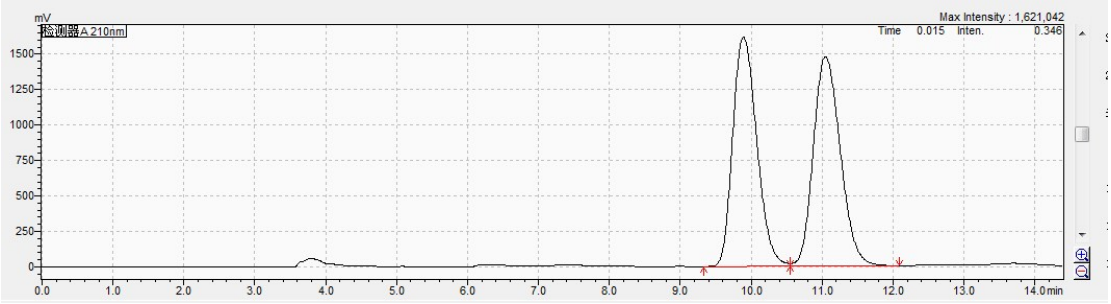


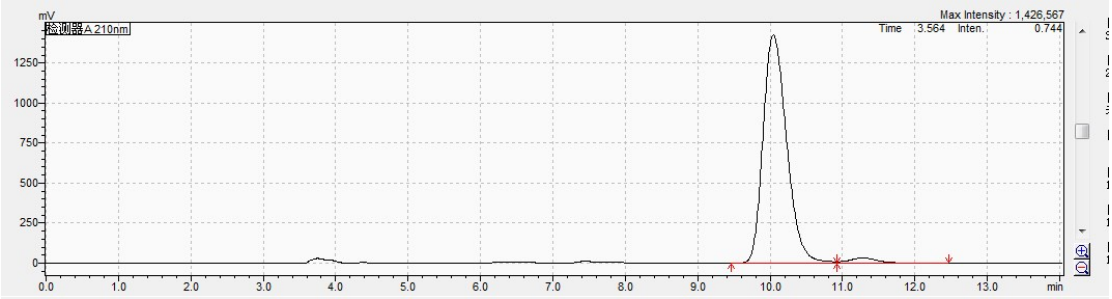
Peak#	Retention Time	Area	Area%
1	11.673	9810696	98.024
2	14.681	197744	1.976
Total		10008440	100.000



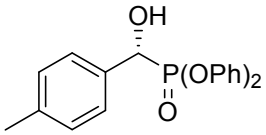
2c

White solid, 71mg, 99% yield, 95% ee, ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30-7.24 (m, 2H), 7.20-7.11 (m, 5H), 7.08-7.00 (m, 3H), 7.00-6.95 (m, 2H), 6.94-6.89 (m, 2H), 5.14 (d, *J* = 9.2 Hz, 1H), 4.64 (brs, 1H), 2.24 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 149.3 (d, *J* = 5.6 Hz), 149.2 (d, *J* = 6.2 Hz), 137.0 (d, *J* = 1.7 Hz), 134.4, 128.6 (d, *J* = 10.0 Hz), 128.2 (d, *J* = 2.8 Hz), 127.3 (d, *J* = 0.9 Hz), 127.1 (d, *J* = 6.2 Hz), 124.1 (d, *J* = 11.3 Hz), 123.6 (d, *J* = 6.2 Hz), 119.6 (d, *J* = 4.0 Hz), 119.5 (d, *J* = 4.0 Hz), 69.6 (d, *J* = 160.2 Hz), 20.4. HRMS (ESI): m/z calcd for $C_{20}H_{19}NaO_4P$ $[M + Na]^+$: 377.09132, found 377.09030. $[\alpha]_D^{25}$ = +26.5 (c 0.32, $CHCl_3$). HPLC (CHIRALCEL OD-H column, hexane / 2-propanol = 90/10, flow 0.8 mL/min, detection at 210 nm) t_r (major) = 10.0 min and t_r (minor) =11.2 min.



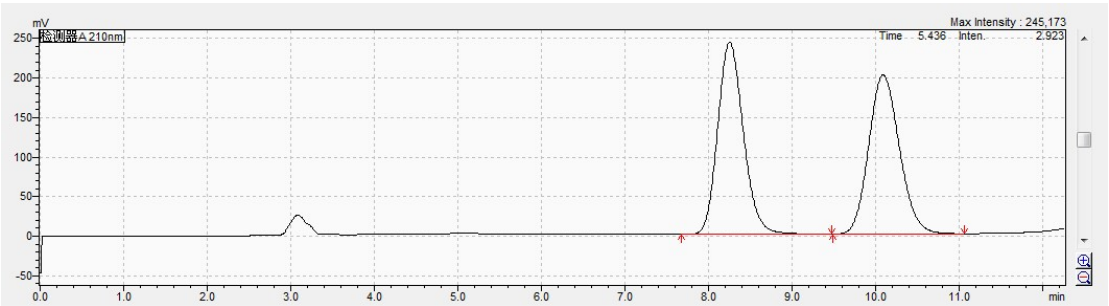


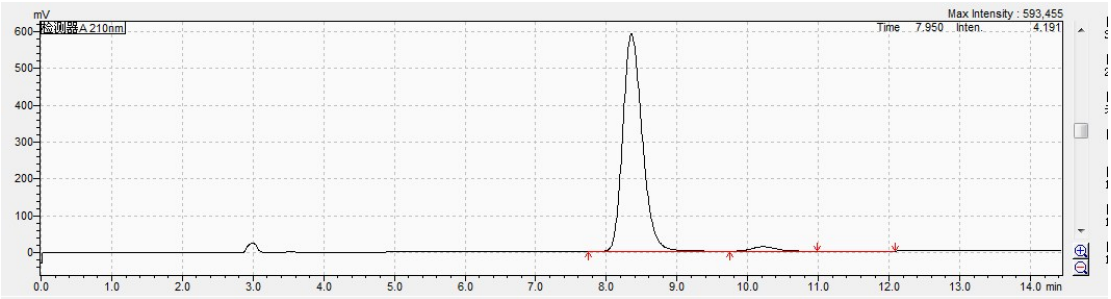
Peak#	Retention Time	Area	Area%
1	10.044	32196171	97.464
2	11.280	837720	2.536
Total		33033891	100.000



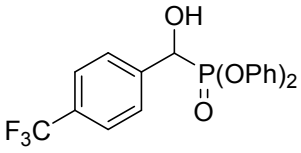
2d

White solid, 71 mg, 99% yield, 95% ee, 1H NMR (500 MHz, $CDCl_3$) δ 7.55-7.45 (m, 2H), 7.35-7.24 (m, 4H), 7.24-7.13 (m, 4H), 7.12-7.09 (m, 2H), 7.07-7.01 (m, 2H), 5.28 (d, J = 8.0 Hz, 1H), 4.16 (brs, 1H), 2.39 (s, 3H). $[\alpha]_D^{25}$ = +40.9 (c 0.57, $CHCl_3$) [lit.[2] $[\alpha]_D^{25}$ = -38.0 (c 1.0, $CHCl_3$) for *S* enantiomer in 91% *ee*]. HPLC (CHIRALCEL OD-H column, hexane / 2-propanol = 90/10, flow 1 mL/min, detection at 210nm) t_r (major) =8.4 min and t_r (minor) =10.2 min.



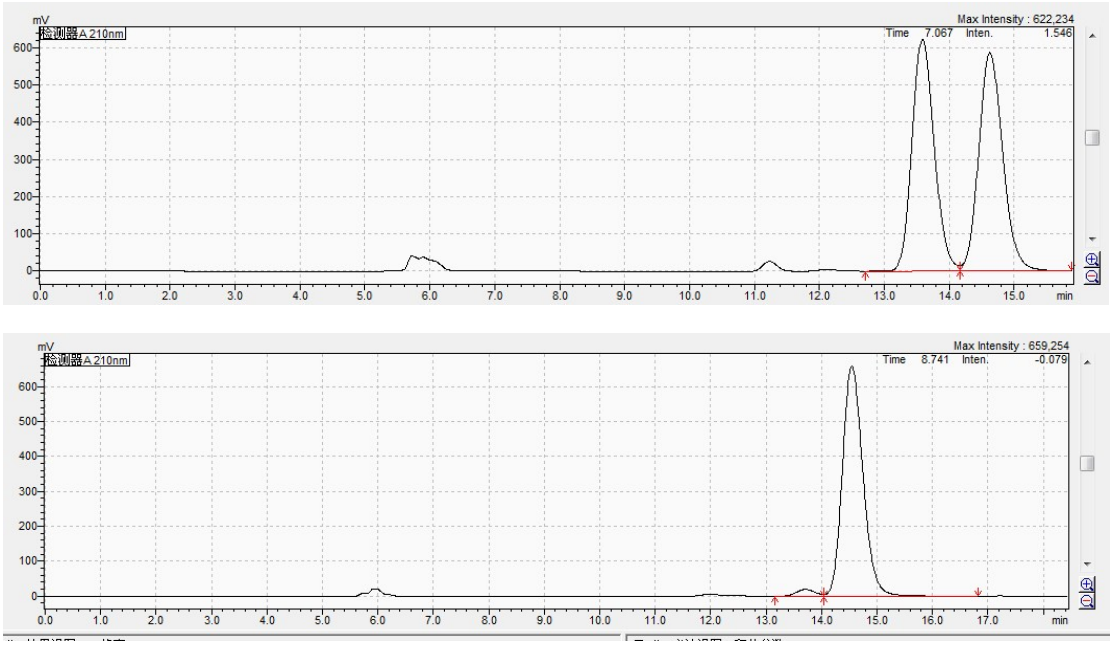


Peak#	Retention Time	Area	Area%
1	8.355	11355306	97.590
2	10.218	280468	2.410
Total		11635774	100.000



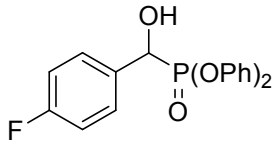
2e

White solid, 77.5 mg, 95% yield, 95% ee, ^{1}H NMR (500 MHz, $CDCl_3$) δ7.69-7.55 (m, 4H), 7.32-7.22 (m, 4H), 7.20-7.12 (m, 2H), 7.10-7.05 (m, 2H), 7.03-6.95 (m, 2H), 5.27 (d, J = 10.4 Hz, 1H), 5.21 (brs, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 150.2 (d, J = 6.2 Hz), 150.1 (d, J = 6.6 Hz), 139.8, 129.8 (d, J = 10.1 Hz), 127.7 (d, J = 5.9 Hz), 125.6, 125.5, 125.3 (qui, J = 3.3 Hz), 120.7 (d, J = 4.1 Hz), 120.5 (d, J = 4.1 Hz), 70.1 (d, J = 159.6 Hz). HRMS (ESI): m/z calcd for $C_{20}H_{16}F_3NaO_4P$ $[M + Na]^+$: 431.06305, found 431.06167. $[\alpha]_D^{25}$ = +32.6 (c 0.41, $CHCl_3$). HPLC (CHIRALCEL OD-H column, hexane / 2-propanol = 90/10, flow 0.5 mL/min, detection at 210 nm) t_r (minor) =13.7 min and t_r (major) =14.5 min.



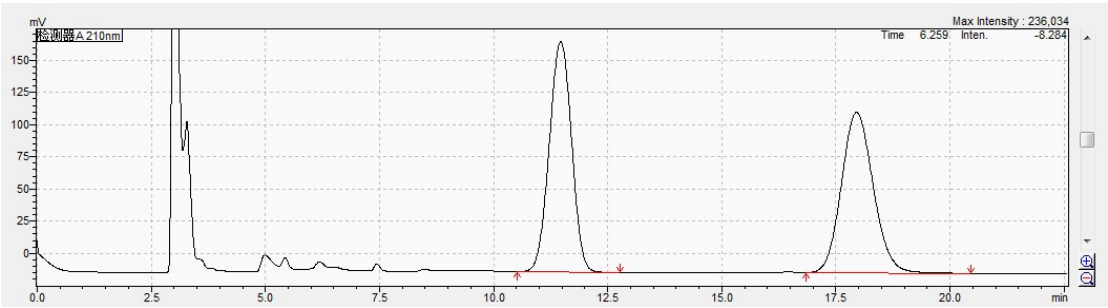
Peak#	Retention Time	Area	Area%

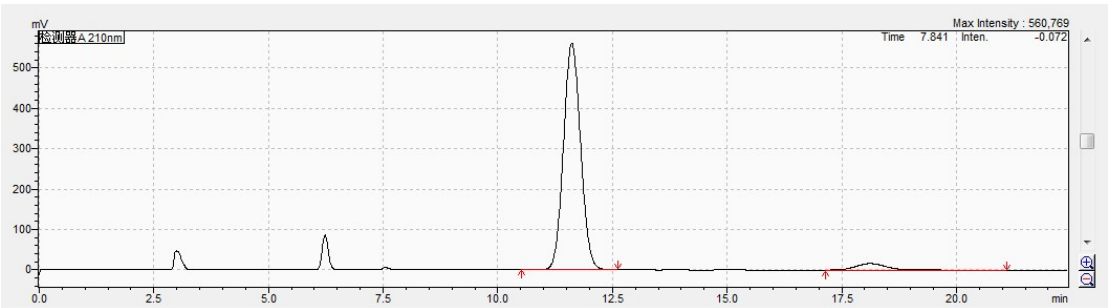
1	13.710	453161	2.601
2	14.547	16971678	97.399
Total		17424839	100.000



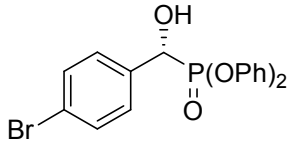
2f

White solid, 66.6 mg, 93% yield, 90% ee, ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.56-7.43 (m, 2H), 7.30-7.20 (m, 4H), 7.19-7.11 (m, 2H), 7.11-7.05 (m, 2H), 7.05-6.96 (m, 4H), 5.19 (d, *J* = 9.0 Hz, 1H), 5.02 (brs, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 161.5 (d, *J* = 3.5 Hz), 159.5 (d, *J* = 3.7 Hz), 148.0 (d, *J* = 1.9 Hz), 147.9 (d, *J* = 2.3 Hz), 129.2 (m), 127.4, (d, *J* = 10.0 Hz), 127.1 (d, *J* = 6.4 Hz), 127.0 (d, *J* = 6.4 Hz), 123.1 (d, *J* = 12.8 Hz), 118.4 (d, *J* = 4.0 Hz), 118.3 (d, *J* = 4.0 Hz), 113.2 (d, *J* = 2.4 Hz), 113.0 (d, *J* = 2.4 Hz), 67.7 (d, *J* = 161.3 Hz). HRMS (ESI): m/z calcd for $C_{19}H_{16}FNaO_4P$ $[M + Na]^+$: 381.06624, found 381.06607. $[\alpha]_D^{25}$ = +30.3 (c 0.35, $CHCl_3$). HPLC (CHIRALPAK AS-H column, hexane / 2-propanol = 80/20, flow 1 mL/min, detection at 210 nm) t_r (major) =11.6 min and t_r (minor) = 18.1 min.





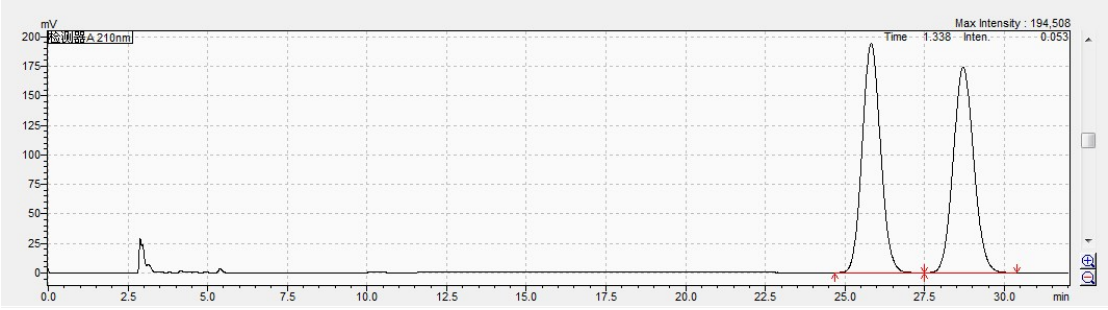
Peak#	Retention Time	Area	Area%
1	11.613	14589556	95.025
2	18.118	763829	4.975
Total		15353385	100.000

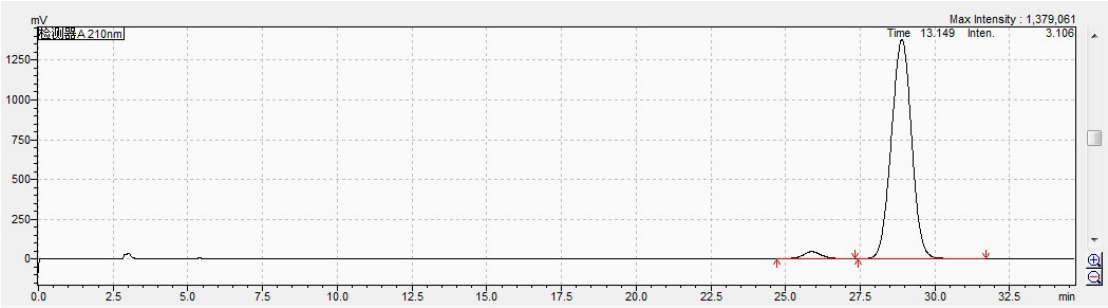


2g

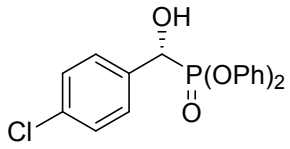
White solid, 78.6 mg, 94% yield, 95%ee, ^{1}H NMR (500 MHz, $CDCl_3$)δ 7.50-7.43 (m, 2H), 7.42-7.36 (m, 2H), 7.30-7.22 (m, 4H), 7.20-7.11 (m, 2H), 7.09-7.04 (m, 2H), 7.02-6.96 (m, 2H), 5.15 (d, *J* =

9.5 Hz, 1H), 5.08 (brs, 1H). $[\alpha]_D^{25}$ = +35.9 (c 0.46, $CHCl_3$) [lit.[2] $[\alpha]_D^{24}$ = -45.8 (c 1.0, $CHCl_3$) for *S* enantiomer in 97% *ee*]. HPLC (CHIRALPAK AD-H column, hexane / 2-propanol = 90/10, flow 1 mL/min, detection at 210nm) t_r (minor) = 25.9 min and t_r (major) = 28.9 min.



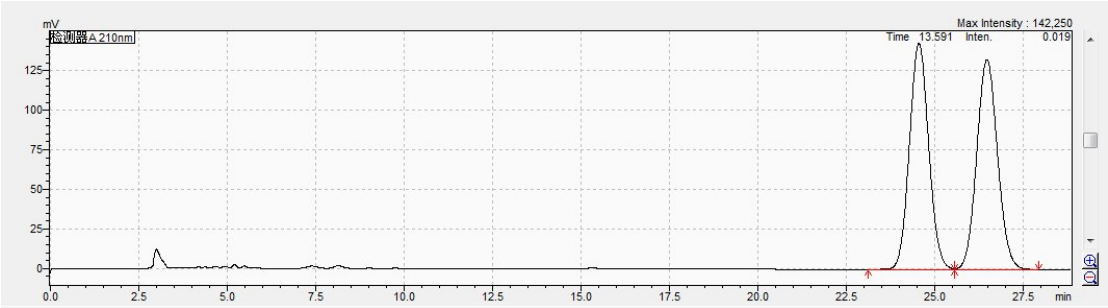


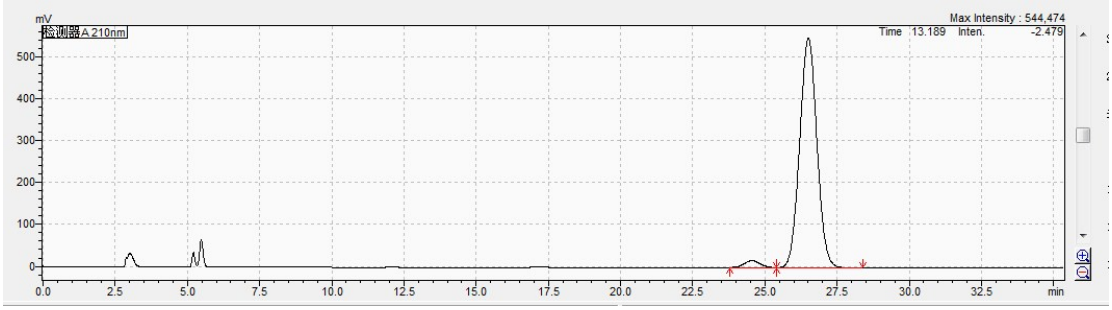
Peak#	Retention Time	Area	Area%
1	25.881	1751599	2.643
2	28.895	64533802	97.357
Total		66285401	100.000



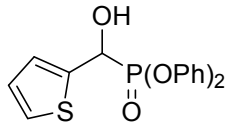
2h

White solid, 73.3 mg, 98% yield, 95% ee, ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.55-7.43 (m, 2H), 7.41-7.24 (m, 7H), 7.24-7.15 (m, 2H), 7.13-7.00 (m, 4H), 5.34-5.26 (m, 1H), 5.25-5.19 (m, 1H). $[\alpha]_D^{25}$ = +39.8 (c 0.42, $CHCl_3$) [lit.[2] $[\alpha]_D^{20}$ = -48.3 (c 1.0, $CHCl_3$) for *S* enantiomer in 93% *ee*].HPLC (CHIRALPAK AD-H column, hexane / 2-propanol = 90/10, flow 1 mL/min, detection at 210 nm) t_r (minor) = 24.5 min and t_r (major) = 26.5 min.



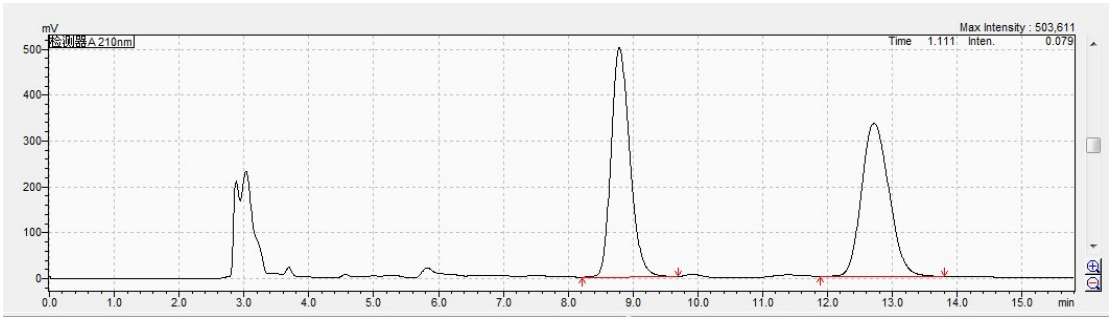


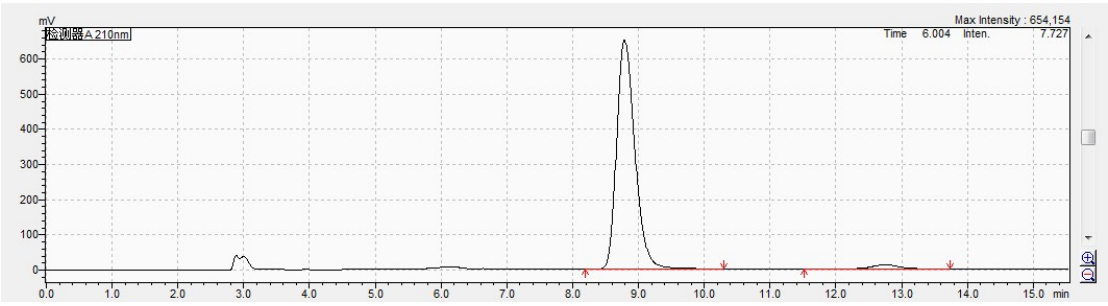
Peak#	Retention Time	Area	Area%
1	24.549	606217	2.602
2	26.508	22696022	97.398
Total		23302239	100.000



2i

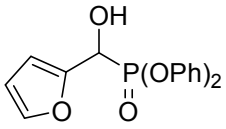
White solid, 69.2 mg, 99% yield, 95% ee, ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36-7.34 (m, 1H), 7.33-7.23 (m, 7H), 7.19-7.15 (m, 2H), 7.15-7.11 (m, 2H), 7.09-7.04 (m, 2H), 7.03-7.00 (m, 1H), 5.51 (d, *J* = 9.7 Hz, 1H), 3.95 (brs, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 150.8 (d, *J* = 11.6 Hz), 138.4, 130.3 (d, *J* = 7.6 Hz), 127.7, 127.1 (d, *J* = 3.2 Hz), 126.0 (d, *J* = 10.6 Hz), 121.2 (d, *J* = 3.9 Hz), 121.1 (d, *J* = 3.7 Hz), 116.5, 67.3 (d, *J* = 169.0 Hz). HRMS (ESI): m/z calcd for $C_{17}H_{15}NaO_4PS$ $[M + Na]^+$: 369.03209, found 369.03203. $[\alpha]_D^{25}$ = +19.9 (c 0.41, $CHCl_3$). HPLC (CHIRALCEL OD-H column, hexane / 2-propanol = 90/10, flow 1 mL/min, detection at 210 nm) t_r (major) = 8.8 min and t_r (minor) = 12.8 min.





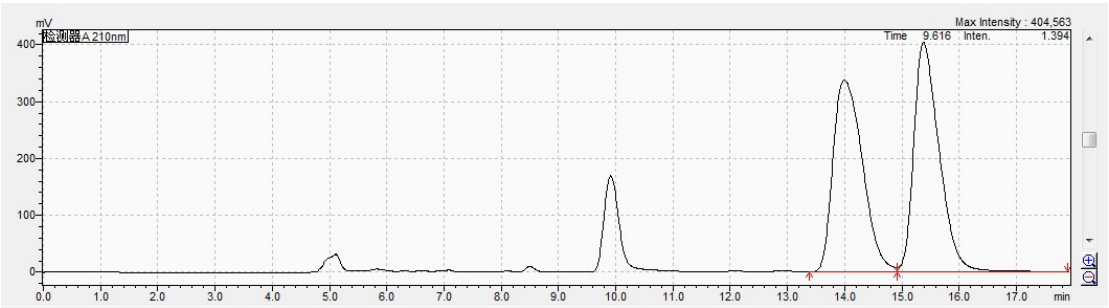
Peak#	Retention Time	Area	Area%

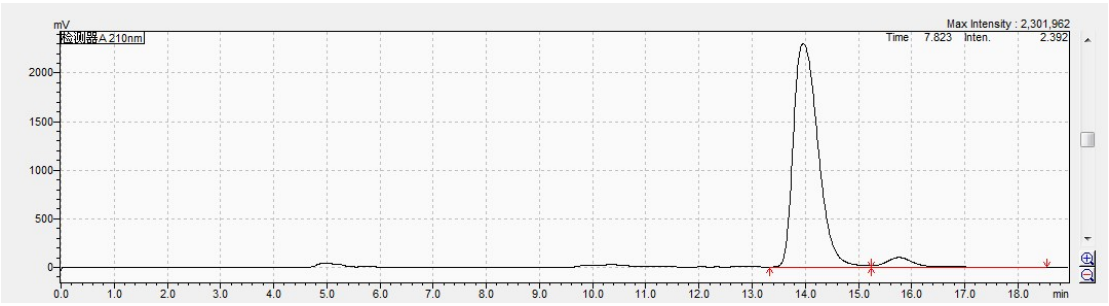
1	8.789	12548503	97.560
2	12.753	313840	2.440
Total		12862343	100.000



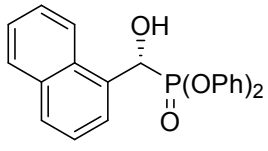
2j

White solid, 64.7 mg, 91% ee, 98% yield, ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46 (s, 1H), 7.38-7.26 (m, 4H), 7.25-7.13 (m, 4H), 7.13-7.05 (m, 2H), 6.65-6.55 (s, 1H), 6.46-6.34 (s, 1H), 5.33 (d, *J* = 12.3 Hz, 1H), 4.16 (brs, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 150.2 (d, *J* = 9.3 Hz), 148.7, 143.3, 129.7 (d, *J* = 10.5 Hz), 125.4 (d, *J* = 14.3 Hz), 120.7 (d, *J* = 3.6 Hz), 120.6 (d, *J* = 3.7 Hz), 110.9, 110.4, (d, *J* = 6.7 Hz), 64.5 (d, *J* = 168.9 Hz). HRMS (ESI): m/z calcd for $C_{17}H_{15}NaO_5P$ [M + Na]$^{+}$: 353.05493, found 353.05419. $[\alpha]_D^{25}$ = +13.8 (c 0.33, $CHCl_3$). HPLC (CHIRALCEL OD-H column, hexane / 2-propanol = 90/10, flow 0.6 mL/min, detection at 210nm) t_r (major) =14.0 min and t_r (minor) = 15.8 min.





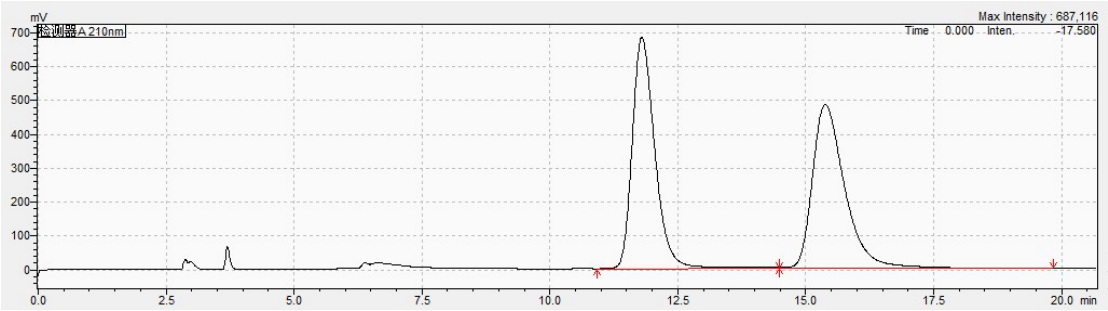
Peak#	Retention Time	Area	Area%
1	13.963	74005617	95.608
2	15.762	3399664	4.392
Total		77405281	100.000

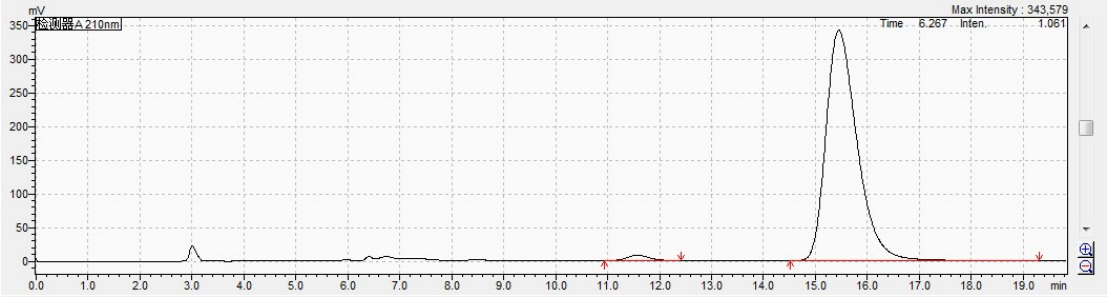


2k

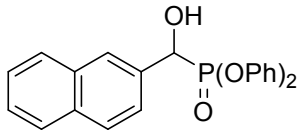
White solid, 70.2 mg, 90% yield, 97% ee, ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.18 (d, *J* = 7.7, 1H), 8.00 (brs, 1H), 7.96-7.84 (m, 2H), 7.62-7.47 (m, 3H), 7.33-7.24 (m, 3H), 7.22-7.13 (m, 3H), 7.11-7.04 (m, 3H), 6.92 (d, *J*

= 7.0 Hz, 2H), 6.22 (d, *J* = 9.7 Hz, 1H), 3.95 (brs, 1H). $[\alpha]_D^{25}$ = +98.9 (c 0.39, $CHCl_3$) [lit.[2] $[\alpha]_D^{22}$ = -131.2 (c 1.0, $CHCl_3$) for *S* enantiomer in 93% *ee*].HPLC (CHIRALCEL OD-H column, hexane / 2-propanol = 90/10, flow 1 mL/min, detection at 210 nm) t_r (minor) =11.6 min and t_r (major) =15.5 min.



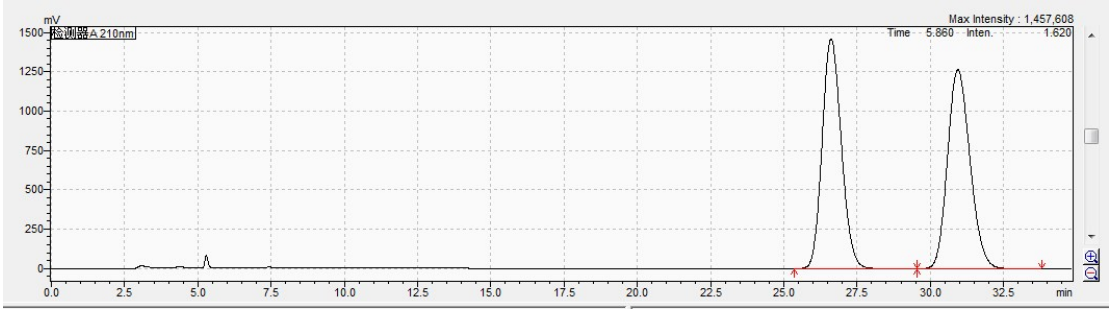


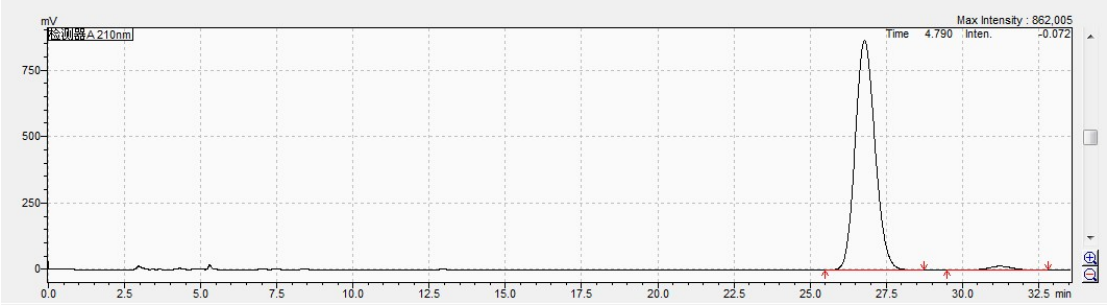
Peak#	Retention Time	Area	Area%
1	11.575	227560	1.572
2	15.452	14247005	98.428
Total		14474565	100.000



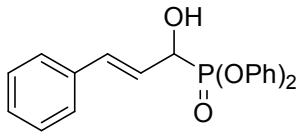
2l

White solid, 78 mg, 99% yield, 96% ee, ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.03 (s, 1H), 7.90-7.78 (m, 3H), 7.73-7.65 (m, 1H), 7.55-7.44 (m, 2H), 7.30-7.17 (m, 5H), 7.17-6.98 (m, 6H), 5.49 (d, *J* = 8.7 Hz, 1H), 3.89 (brs, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 150.3 (d, *J* = 7.7 Hz), 133.4, 133.1, 132.8, 129.7 (d, *J* = 8.6 Hz), 128.3 (d, *J* = 8.1 Hz), 127.7, 126.8 (d, *J* = 8.2 Hz), 126.4 (d, *J* = 13.2 Hz), 125.3 (d, *J* = 10.8 Hz), 124.9 (d, *J* = 4.2 Hz), 120.6 (d, *J* = 3.9 Hz), 120.5 (d, *J* = 3.7 Hz), 70.9 (d, *J* = 160.0 Hz). HRMS (ESI): m/z calcd for $C_{23}H_{19}NaO_4P$ $[M + Na]^+$: 413.09132, found 413.09020. $[\alpha]_D^{25}$ = +45.2 (c 0.38, $CHCl_3$). HPLC (CHIRALPAK AD-H column, hexane / 2-propanol = 80/20, flow 1mL/min, detection at 210 nm) t_r (major) = 26.8 min and t_r (minor) = 31.2 min.



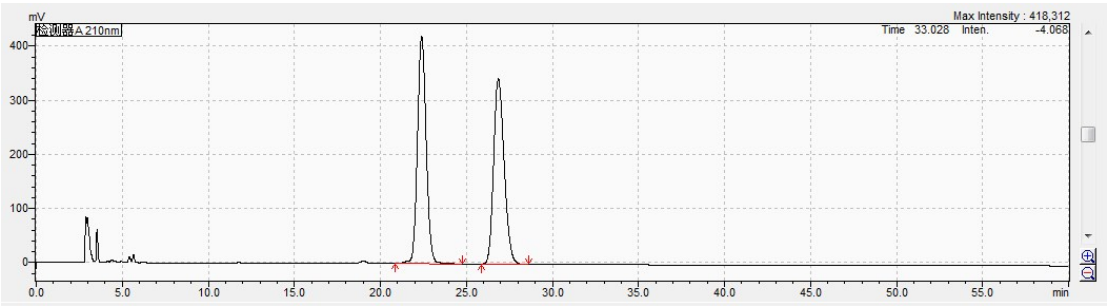


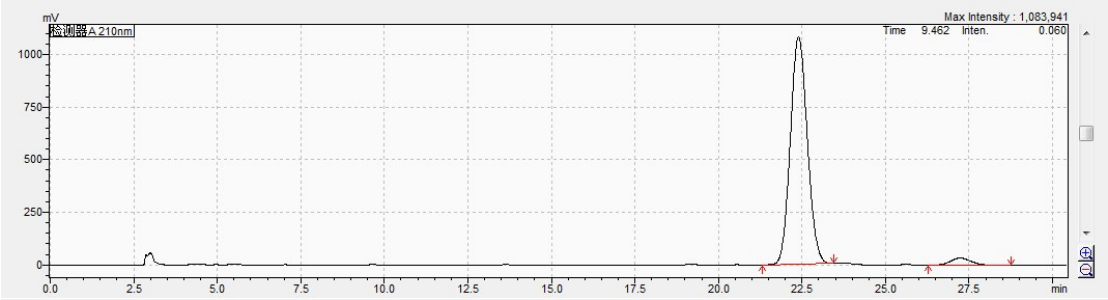
Peak#	Retention Time	Area	Area%
1	26.789	39011588	98.125
2	31.235	745444	1.875
Total		39757032	100.000



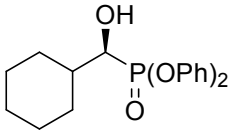
2m

White solid, 66.6 mg, 91% yield, 93% ee, ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36-7.22 (m, 11H), 7.22-7.11 (m, 7H), 6.88-6.79 (m, 1H), 6.45-6.35 (m, 1H), 4.97-4.86 (m, 1H), 4.54 (t, *J* = 6.2 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 150.4 (d, *J* = 5.0 Hz), 136.2 (d, *J* = 2.9 Hz), 133.9 (d, *J* = 14.1 Hz), 129.8 (d, *J* = 5.7 Hz), 128.7, 128.2, 126.9 (d, *J* = 1.8 Hz), 125.4 (d, *J* = 10.9 Hz), 120.9 (d, *J* = 4.1 Hz), 120.8 (d, *J* = 4.1 Hz), 69.5 (d, *J* = 161.5 Hz). HRMS (ESI): m/z calcd for $C_{21}H_{19}NaO_4P$ $[M + Na]^+$: 389.09132, found 389.09106. $[\alpha]_D^{25}$ = +29.3 (c 0.35, $CHCl_3$). HPLC (CHIRALPAK AD-H column, hexane / 2-propanol = 90/10, flow 1 mL/min, detection at 210 nm) t_r (major) = 22.4 min and t_r (minor) = 27.2 min.



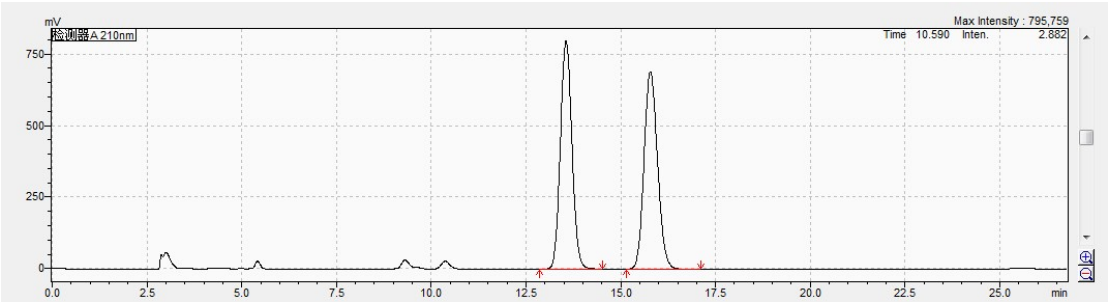


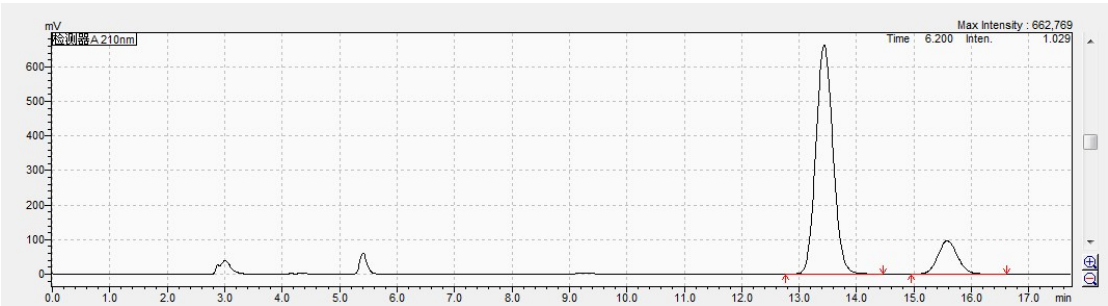
Peak#	Retention Time	Area	Area%
1	22.399	38213753	96.427
2	27.233	1415901	3.573
Total		39629654	100.000



2n

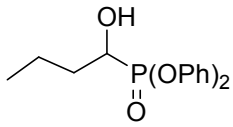
White solid, 69.2 mg, 99% yield, 72% ee, ^{1} H NMR (500 MHz, $CDCl^{3}$) δ 7.34-7.26 (m, 4H), 7.22-7.11 (m, 6H), 3.95 (t, *J* = 5.6, 1H), 3.49 (brs, 1H), 2.17-2.05 (m, 1H), 2.02-1.93 (m, 1H), 1.90-1.82 (m, 1H), 1.82-1.72 (m, 2H), 1.71-1.60 (m, 1H), 1.43-1.11 (m, 6H). $[\alpha]_D^{25}$ = +1.4 (c 0.42, $CHCl_3$) [lit.[2] $[\alpha]_D^{21}$ = +0.7 (c 1.0, $CHCl_3$) for *S* enantiomer in 66% *ee*]. HPLC (CHIRALPAK AD-H column, hexane / 2-propanol = 90/10, flow 1 mL/min, detection at 210 nm) t_r (major) = 13.4 min and t_r (minor) = 15.6 min.





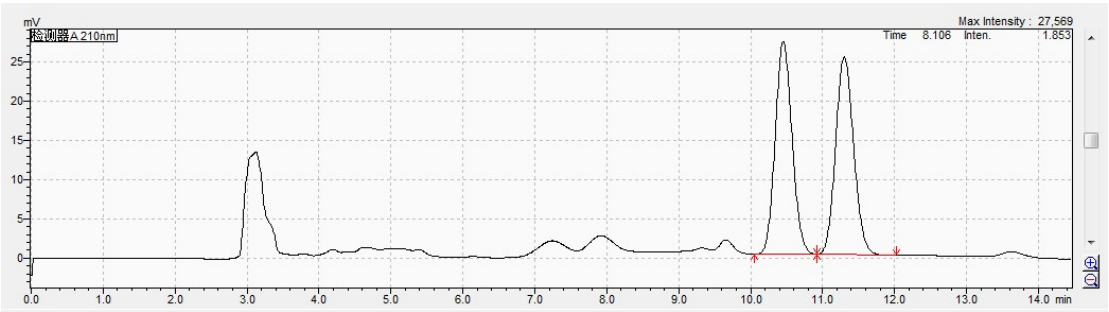
Peak#	Retention Time	Area	Area%

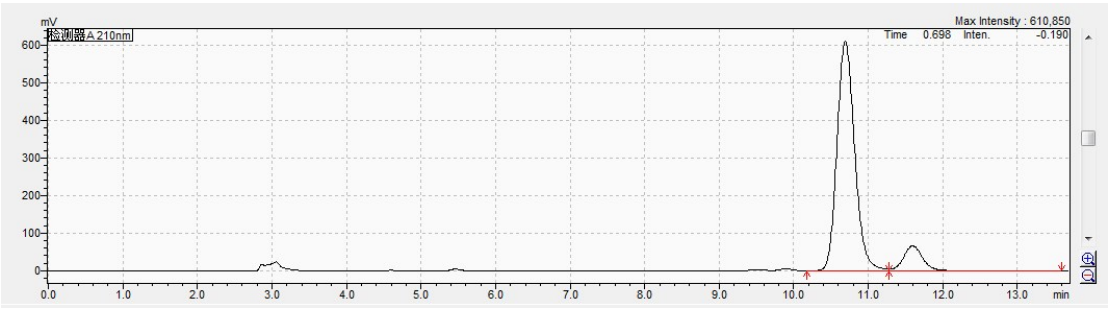
1	13.435	13703285	85.891
2	15.578	2250921	14.109
Total		15954206	100.000



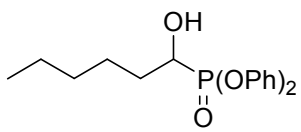
2o

Colorless oil, 53.2 mg, 87% yield, 79% ee, ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38-7.28 (m, 4H), 7.23-7.12 (m, 6H), 4.24-4.17 (m, 1H), 2.87 (brs, 1H), 2.02-1.84 (m, 2H), 1.57-1.35 (m, 2H), 0.98 (t, J = 7.4 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 150.3 (d, J = 9.9 Hz), 129.8, 125.3 (d, J = 3.8 Hz), 120.7 (t, J = 4.1 Hz), 67.6 (d, J = 158.9 Hz), 33.3, 18.9 (d, J = 14.2 Hz), 13.7. HRMS (ESI): m/z calcd for $C_{16}H_{19}NaO_4P$ $[M + Na]^+$: 329.09132, found 329.09133. $[\alpha]_D^{25}$ = -7.0 (c 1.48, $CHCl_3$). HPLC (CHIRALPAK AD-H column, hexane / 2-propanol = 90/10, flow 1 mL/min, detection at 210nm) t_r (major) = 10.692 min and t_r (minor) = 11.594 min.





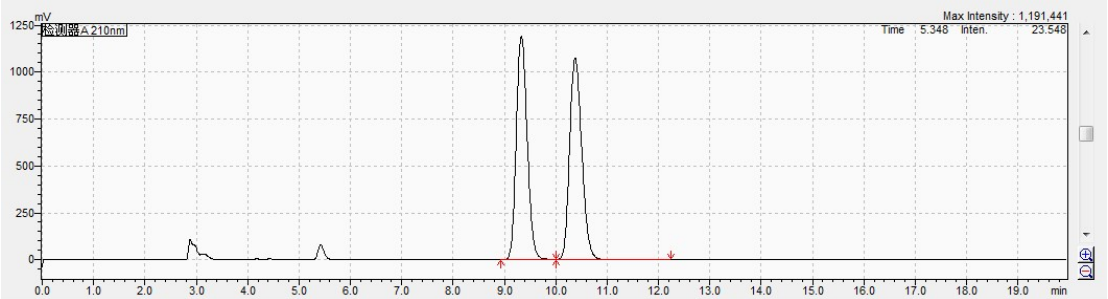
Peak#	Retention Time	Area	Area%
1	10.692	10041464	89.385
2	11.594	1192463	10.615
Total		11233927	100.000

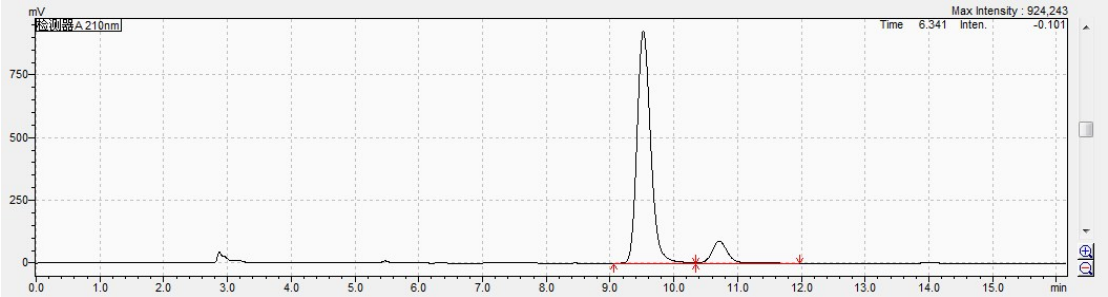


2p

White solid, 66.8 mg, 99% yield, 81% ee, ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33-7.26 (m, 4H), 7.21-7.12 (m, 6H), 4.16-4.10 (m, 1H), 3.88 (brs, 1H), 1.94-1.87 (m, 2H), 1.42-1.26 (m, 6H), 0.88 (t, J = 6.9 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 150.4 (d, J = 10.6 Hz),

129.8, 125.2 (d, J = 5.8 Hz), 120.8 (d, J = 4.0 Hz), 120.7 (d, J = 4.1 Hz), 67.8 (d, J = 158.7 Hz), 31.5, 31.3 (d, J = 0.8 Hz), 25.4 (d, J = 13.9 Hz), 22.5, 14.1. HRMS (ESI): m/z calcd for $C_{18}H_{23}NaO_4P$ $[M + Na]^+$: 357.12262, found 357.12250. $[\alpha]_D^{25}$ = -10.1 (c 0.40, $CHCl_3$). HPLC (CHIRALPAK AD-H column, hexane / 2-propanol = 90/10, flow 1 mL/min, detection at 210nm) t_r (major) = 9.5 min and t_r (minor) = 10.7 min.

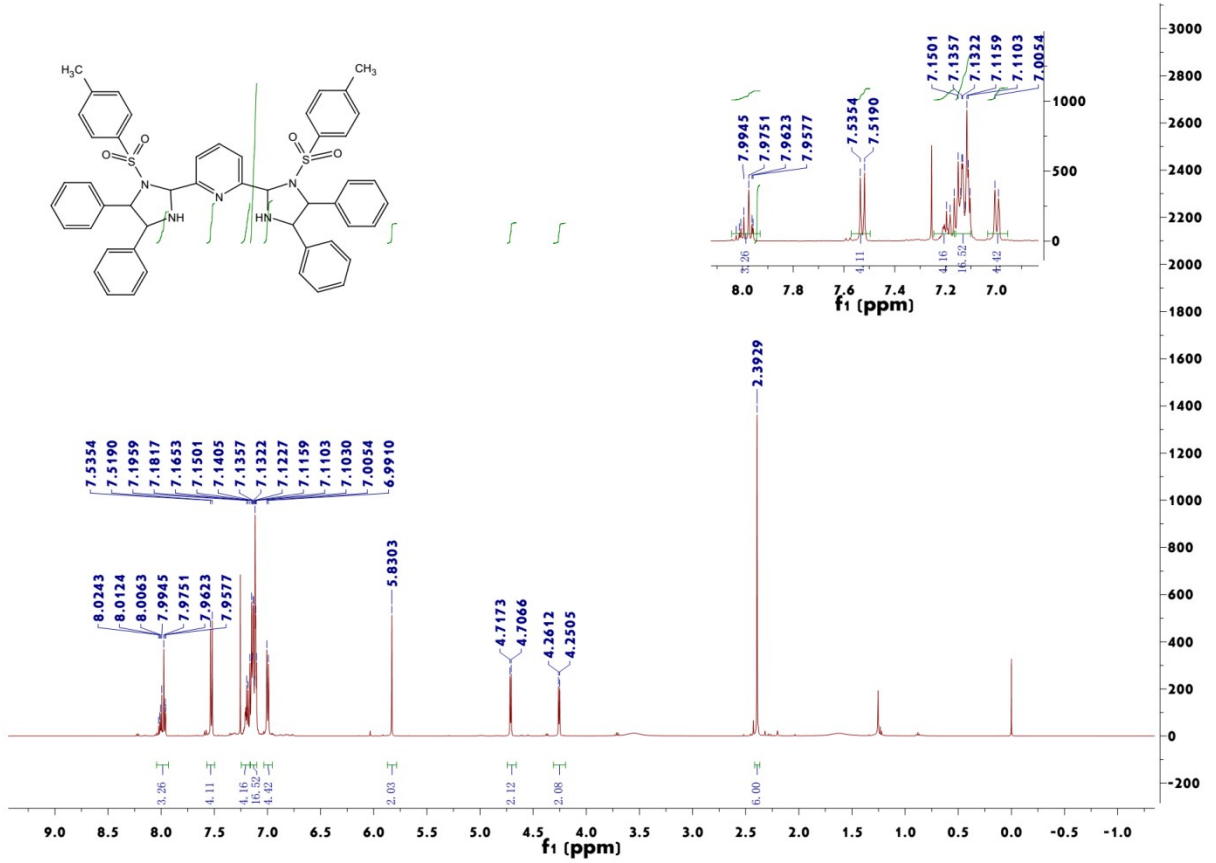




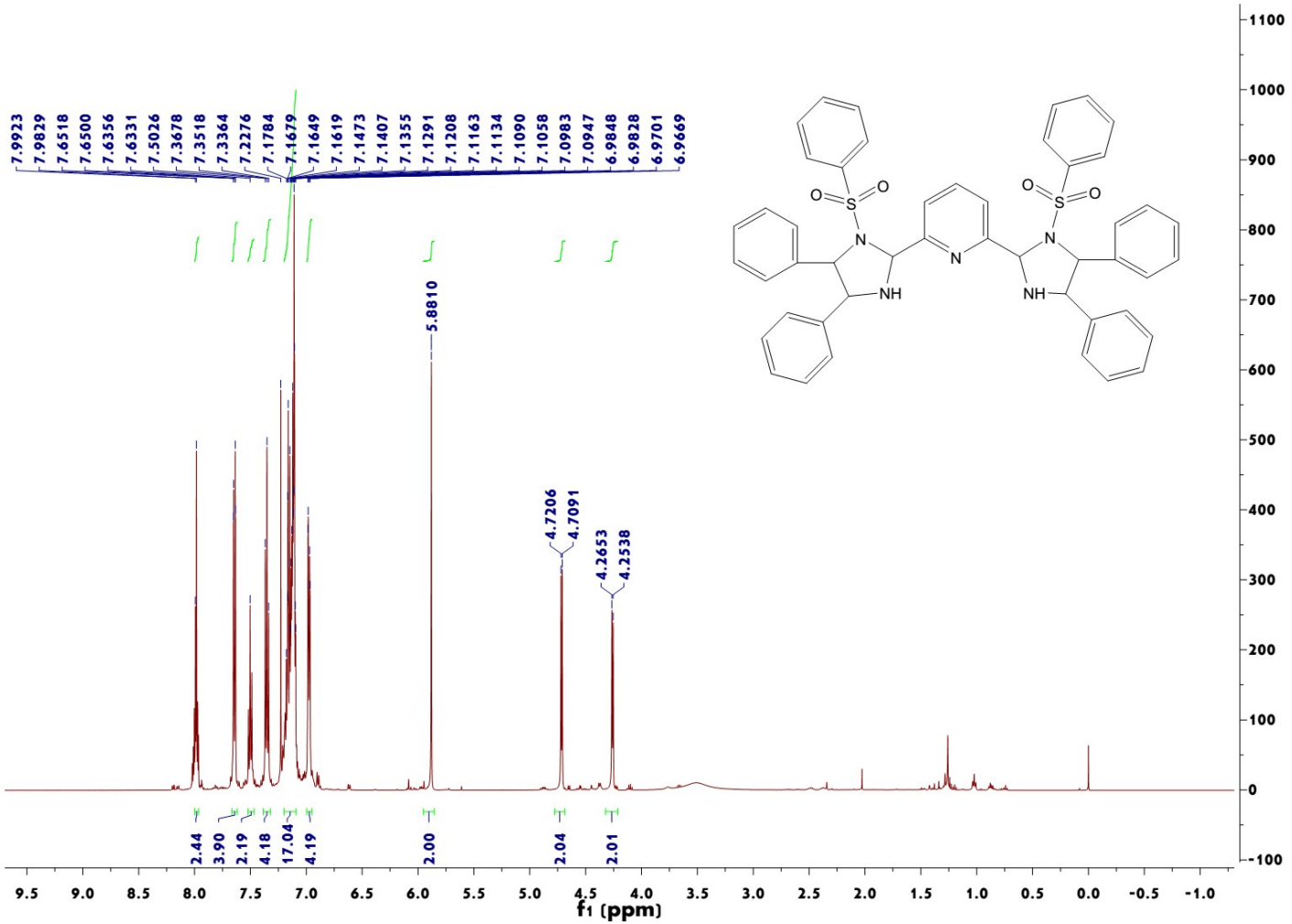
Peak#	Retention Time	Area	Area%
1	9.523	13857672	90.325
2	10.711	1484417	9.675
Total		15342089	100.000

8. Copy of NMR spectra.

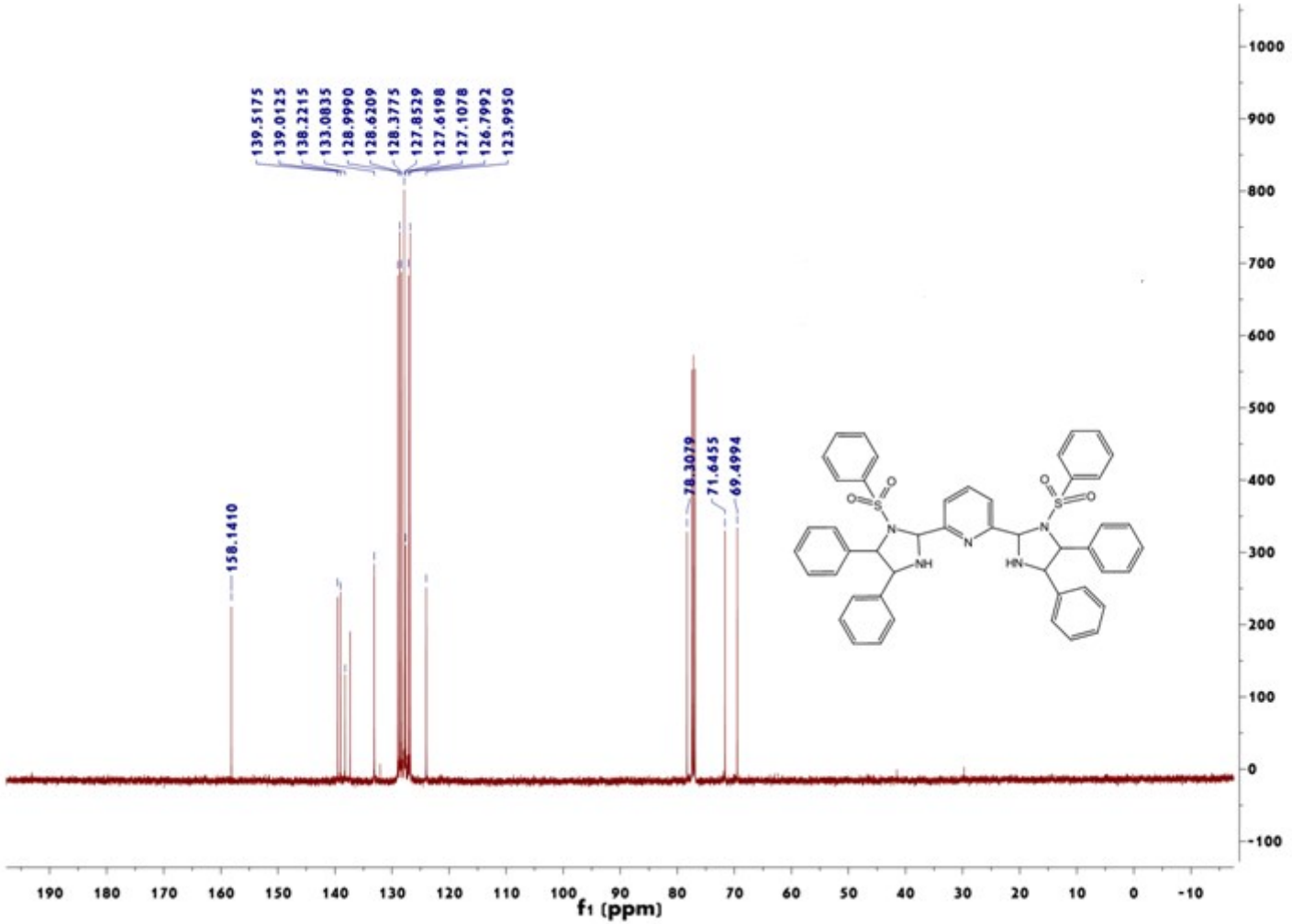
L1



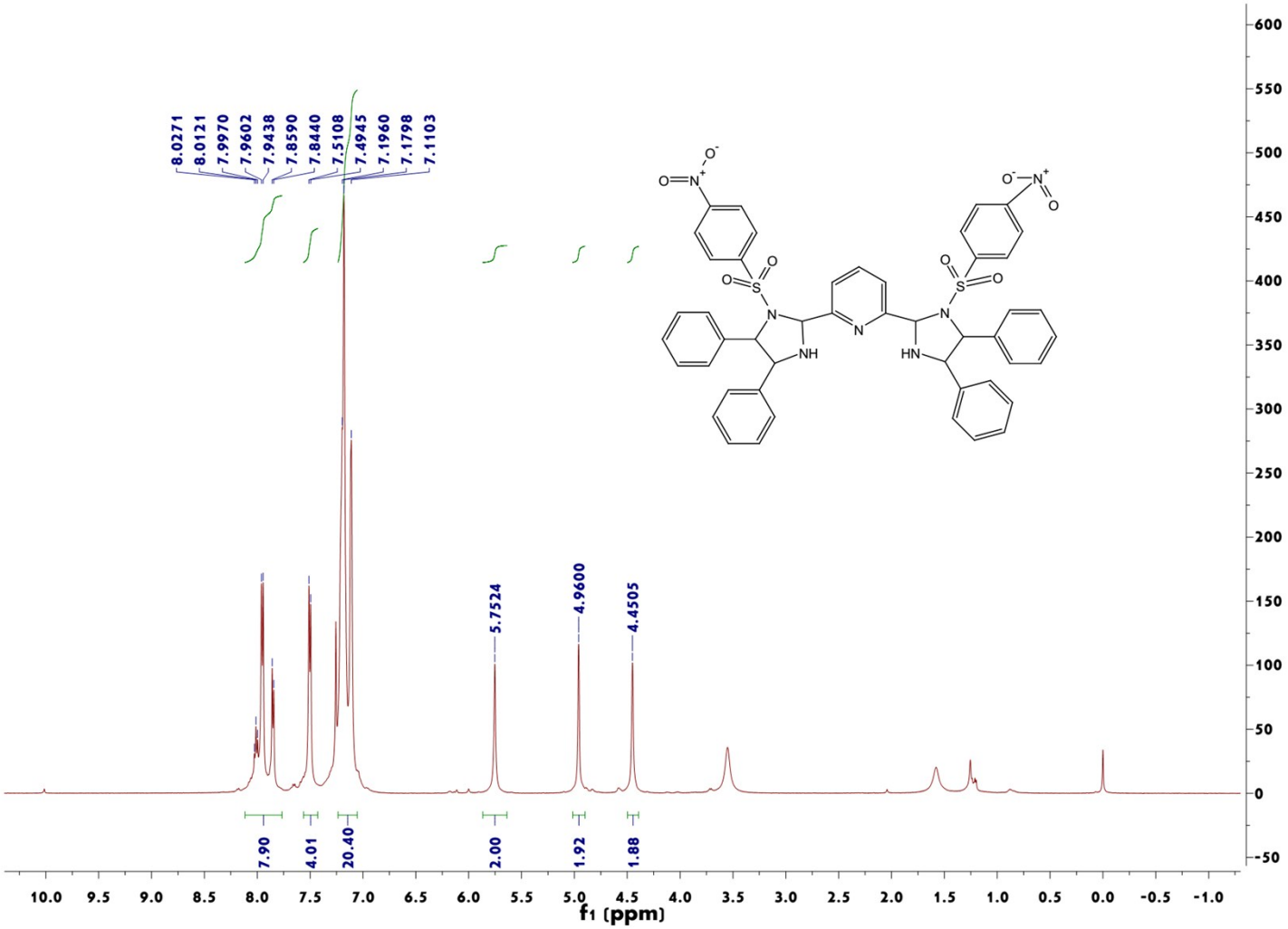
L2


7.9923
7.9829
7.6518
7.6500
7.6356
7.6331
7.5026
7.3678
7.3518
7.3364
7.2276
7.1784
7.1679
7.1649
7.1619
7.1473
7.1407
7.1355
7.1291
7.1208
7.1163
7.1134
7.1090
7.1058
7.0983
7.0947
6.9848
6.9828
6.9701
6.9669
5.8810
4.7206
4.7091
4.2653
4.2538
2.44
3.90
2.19
4.18
17.04
4.19
2.00
2.04
2.01
f1 (ppm)

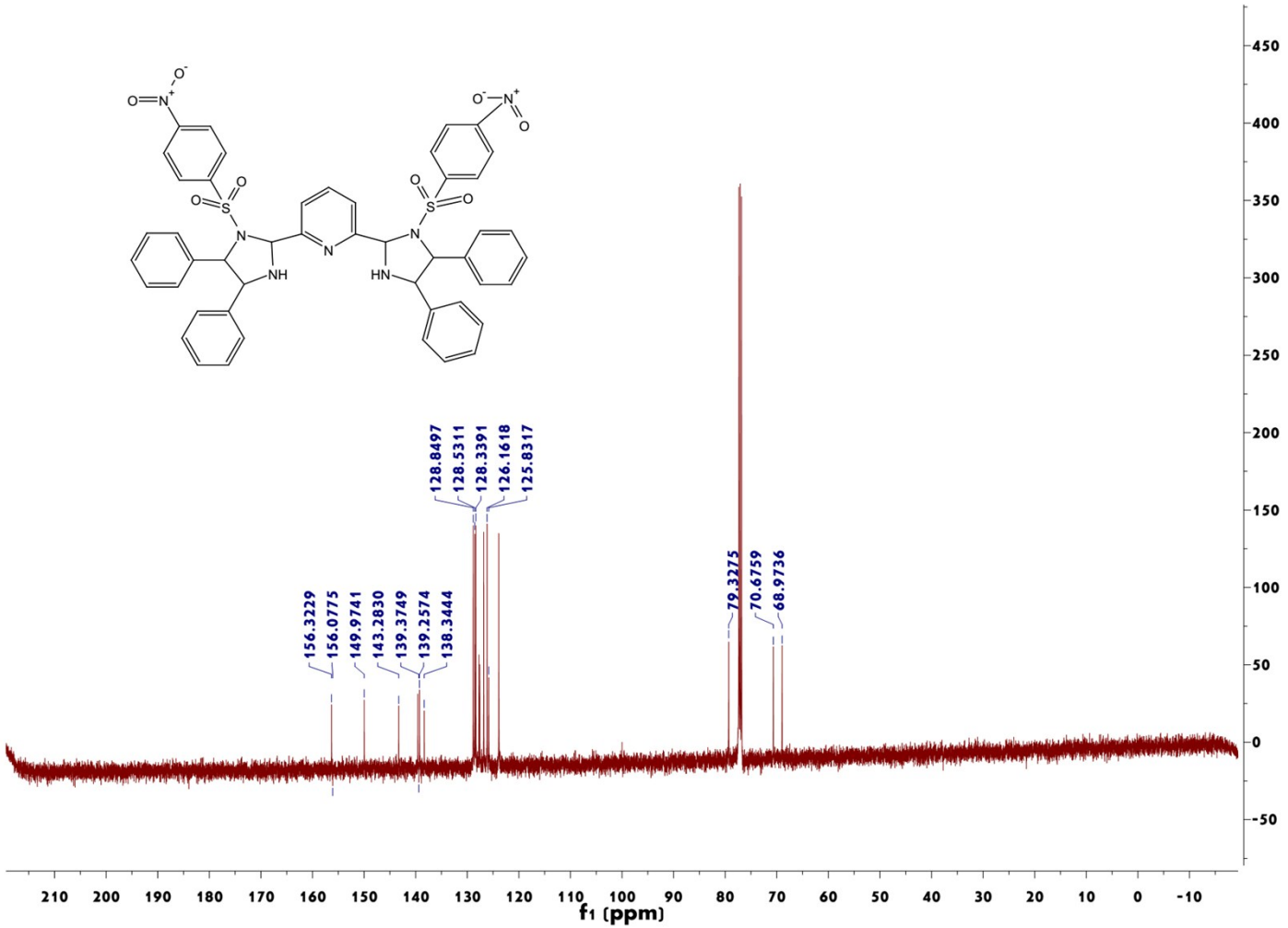
L2


139.5175
139.0125
138.2215
133.0835
128.9990
128.6209
128.3775
127.8529
127.6198
127.1078
126.7992
123.9950
158.1410
78.3079
71.6455
69.4994
1000
900
800
700
600
500
400
300
200
100
0
-100
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

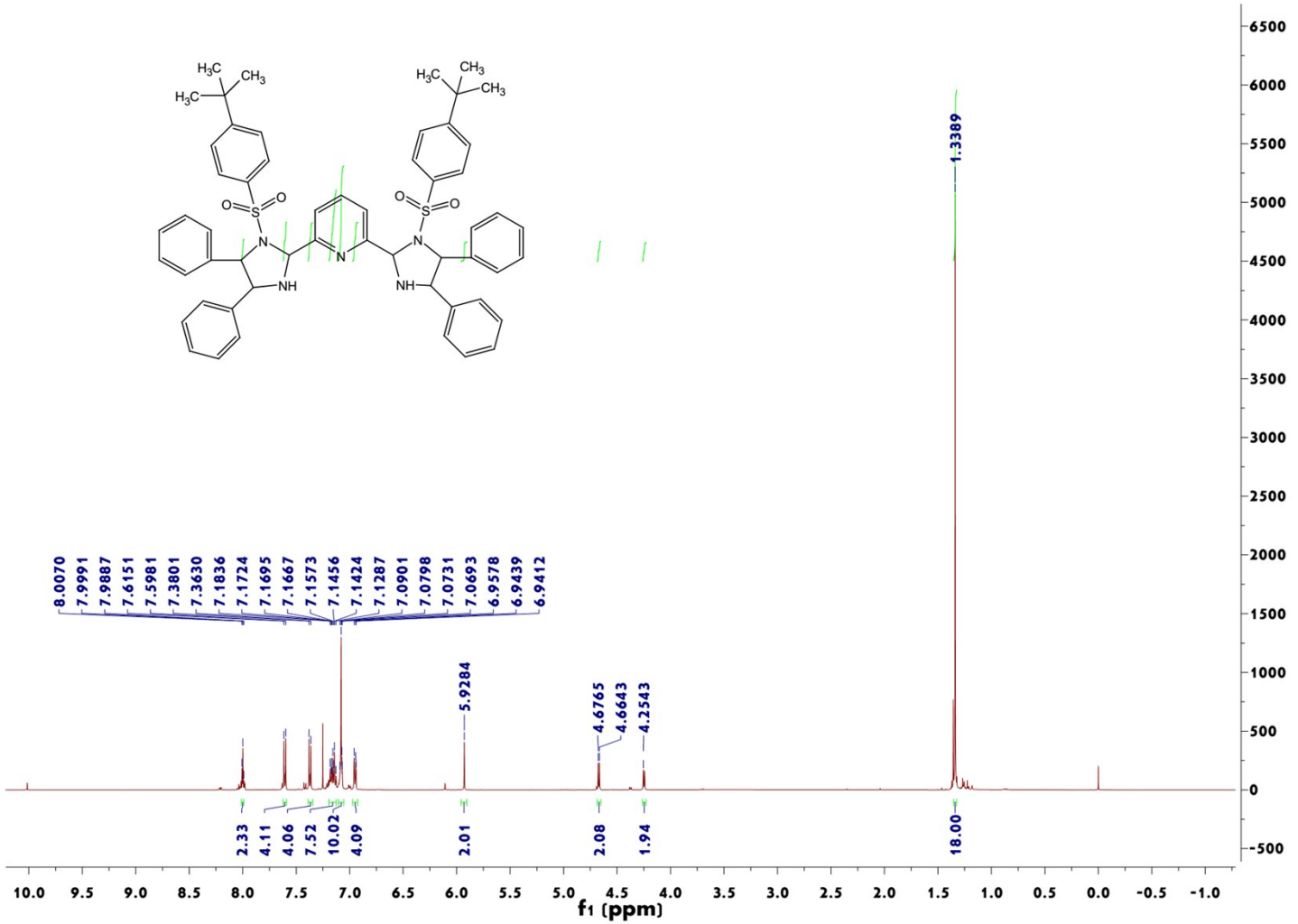
L3


8.0271
8.0121
7.9970
7.9602
7.9438
7.8590
7.8440
7.5108
7.4945
7.1960
7.1798
7.1103
5.7524
4.9600
4.4505
7.90
4.01
20.40
2.00
1.92
1.88
f1 (ppm)

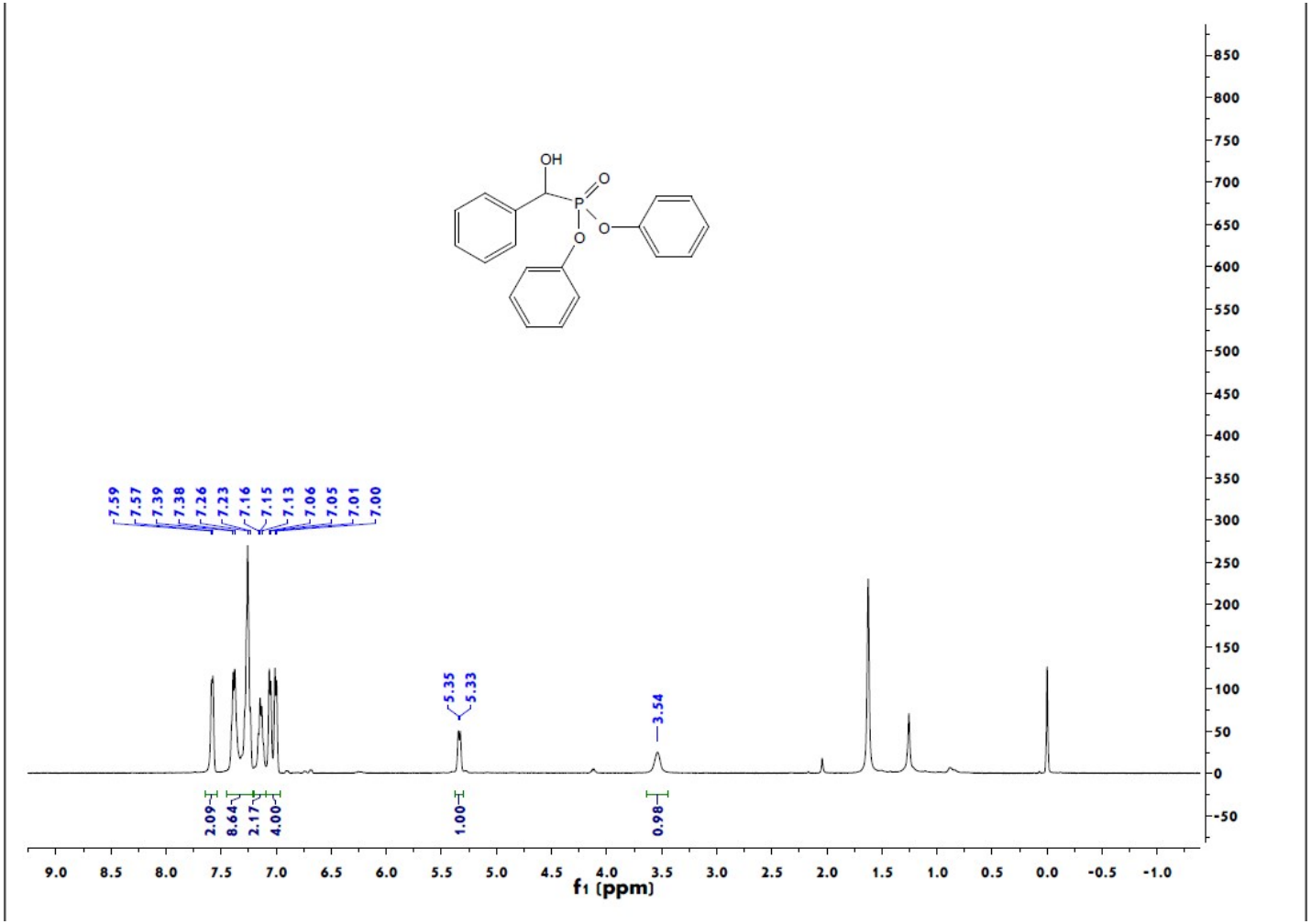
L3


156.3229
156.0775
149.9741
143.2830
139.3749
139.2574
138.3444
128.8497
128.5311
128.3391
126.1618
125.8317
79.3275
70.6759
68.9736
f1 (ppm)

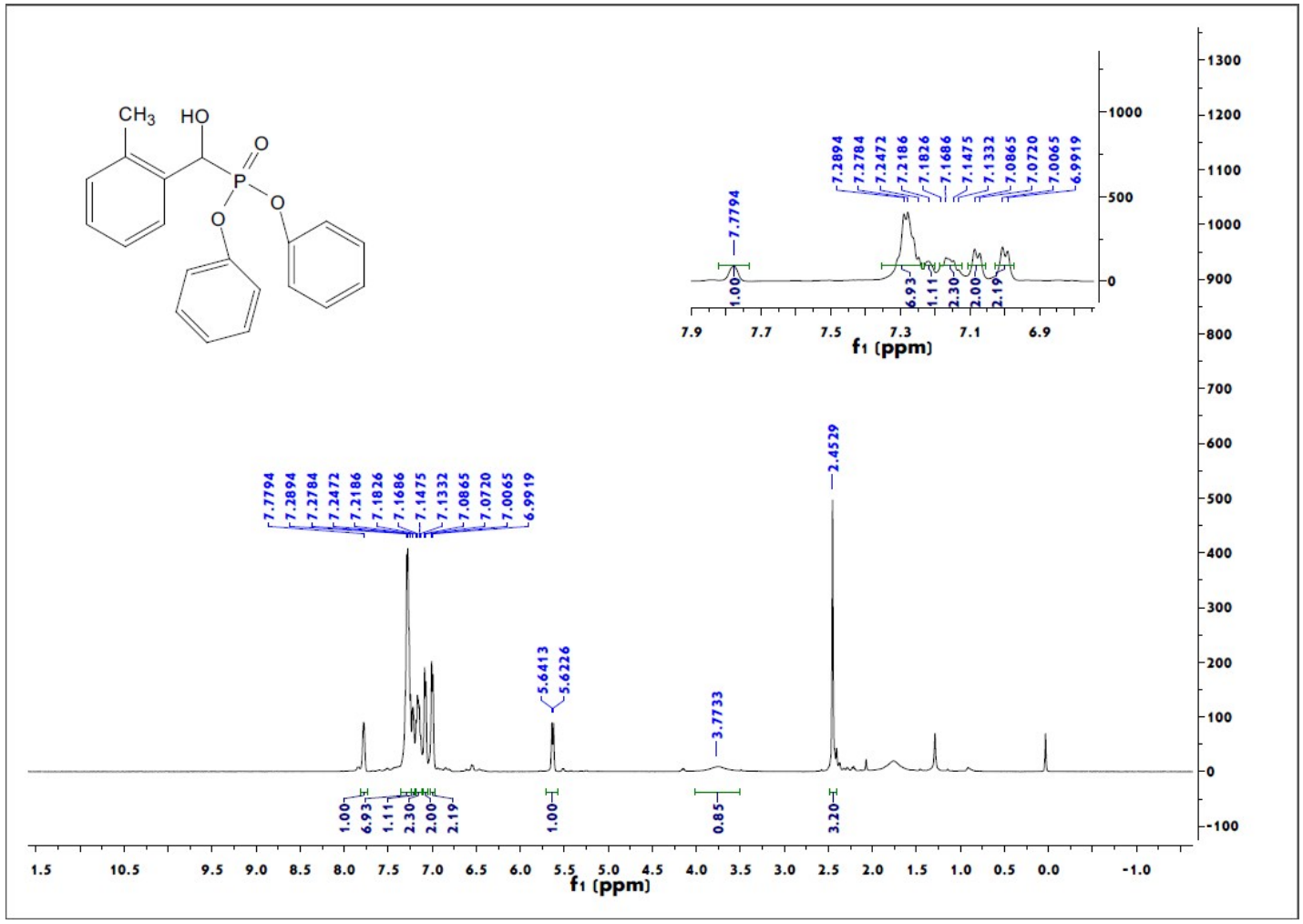
L4


8.0070
7.9991
7.9887
7.6151
7.5981
7.3801
7.3630
7.1836
7.1724
7.1695
7.1667
7.1573
7.1456
7.1424
7.1287
7.0901
7.0798
7.0731
7.0693
6.9578
6.9439
6.9412
5.9284
4.6765
4.6643
4.2543
1.3389
2.33
4.11
4.06
7.52
10.02
4.09
2.01
2.08
1.94
18.00
f1 (ppm)

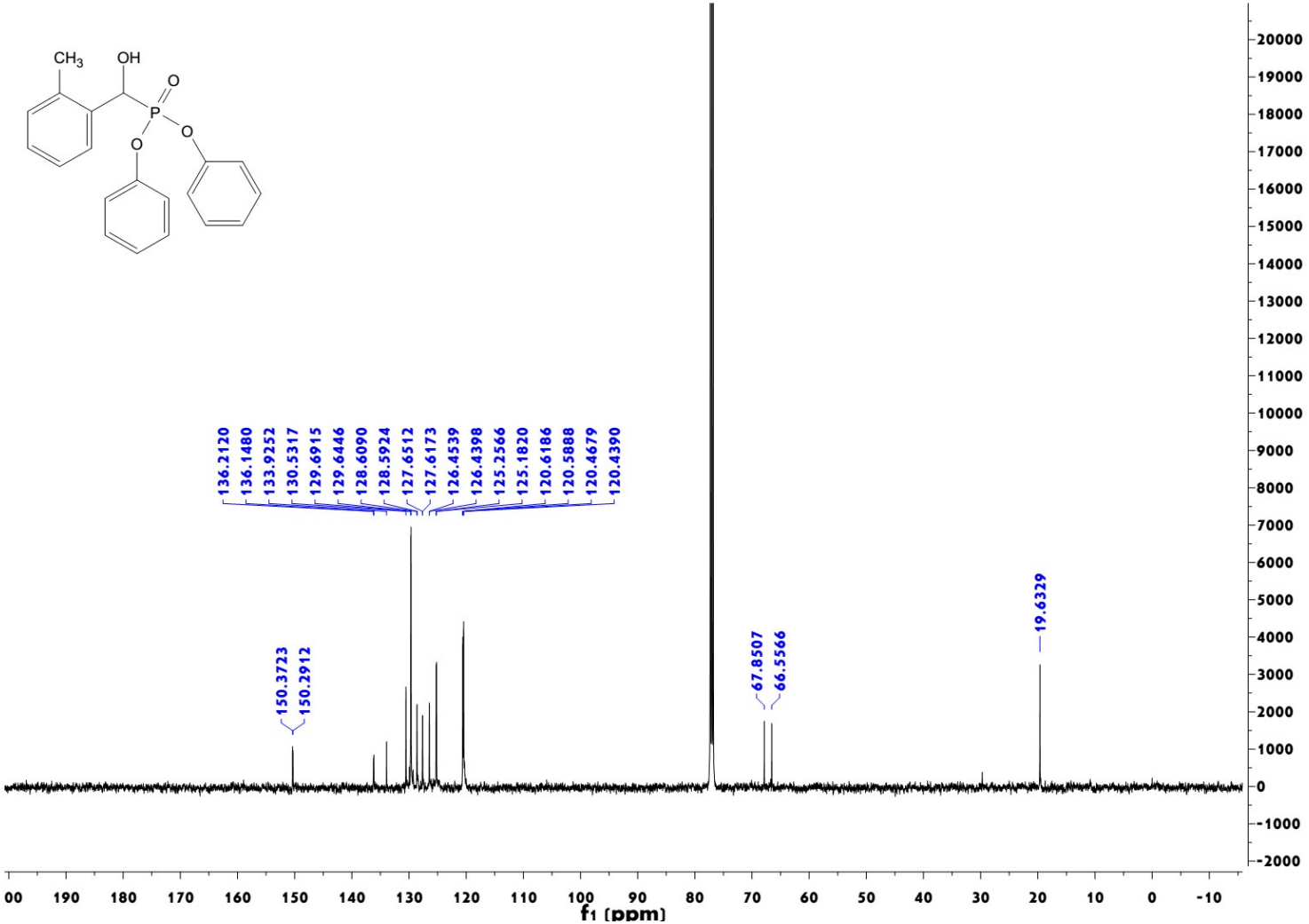
2a

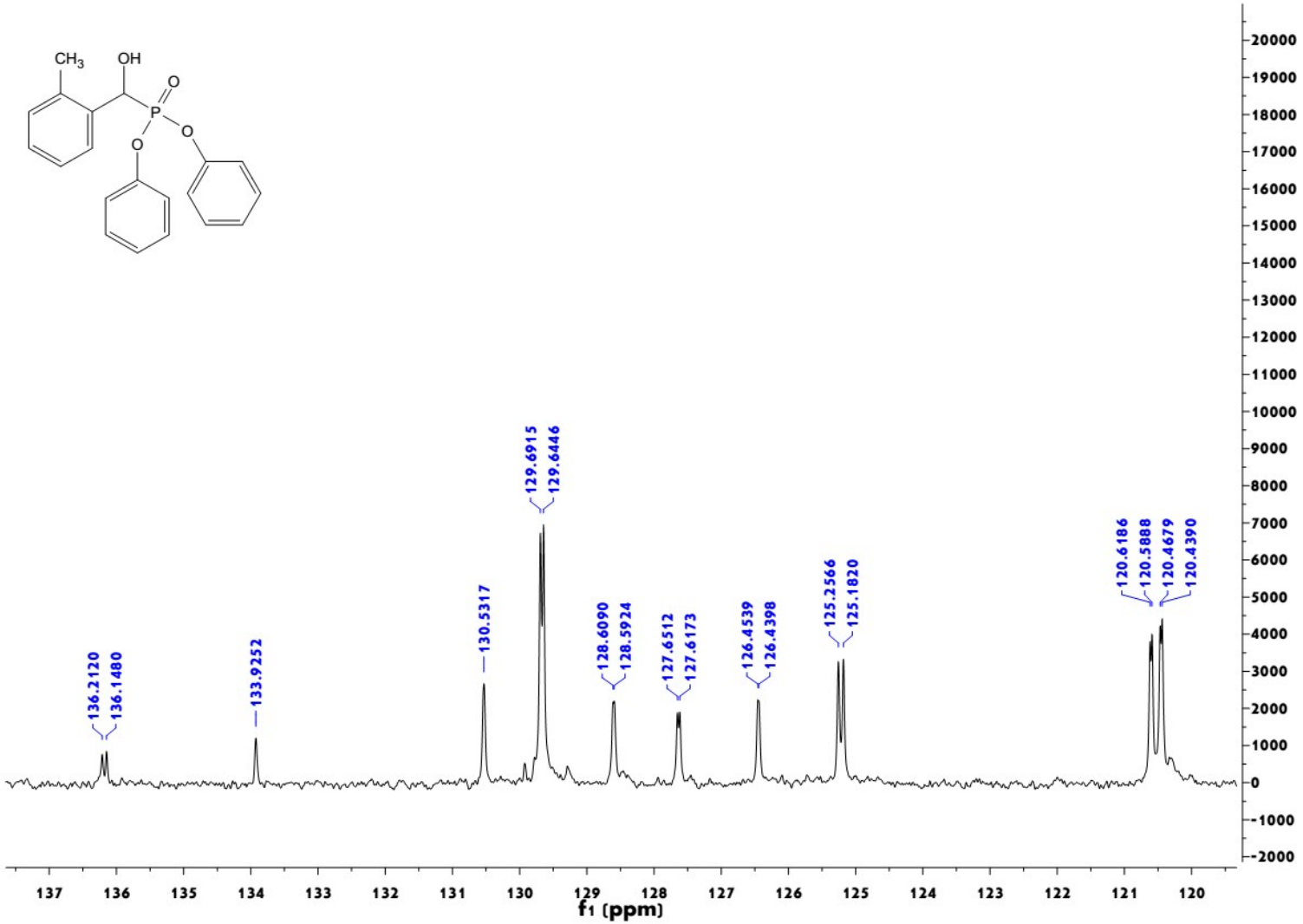

OH
O
P
O
O
7.59
7.57
7.39
7.38
7.26
7.23
7.16
7.15
7.13
7.06
7.05
7.01
7.00
5.35
5.33
3.54
2.09
8.64
2.17
4.00
1.00
0.98
f1 (ppm)

2b

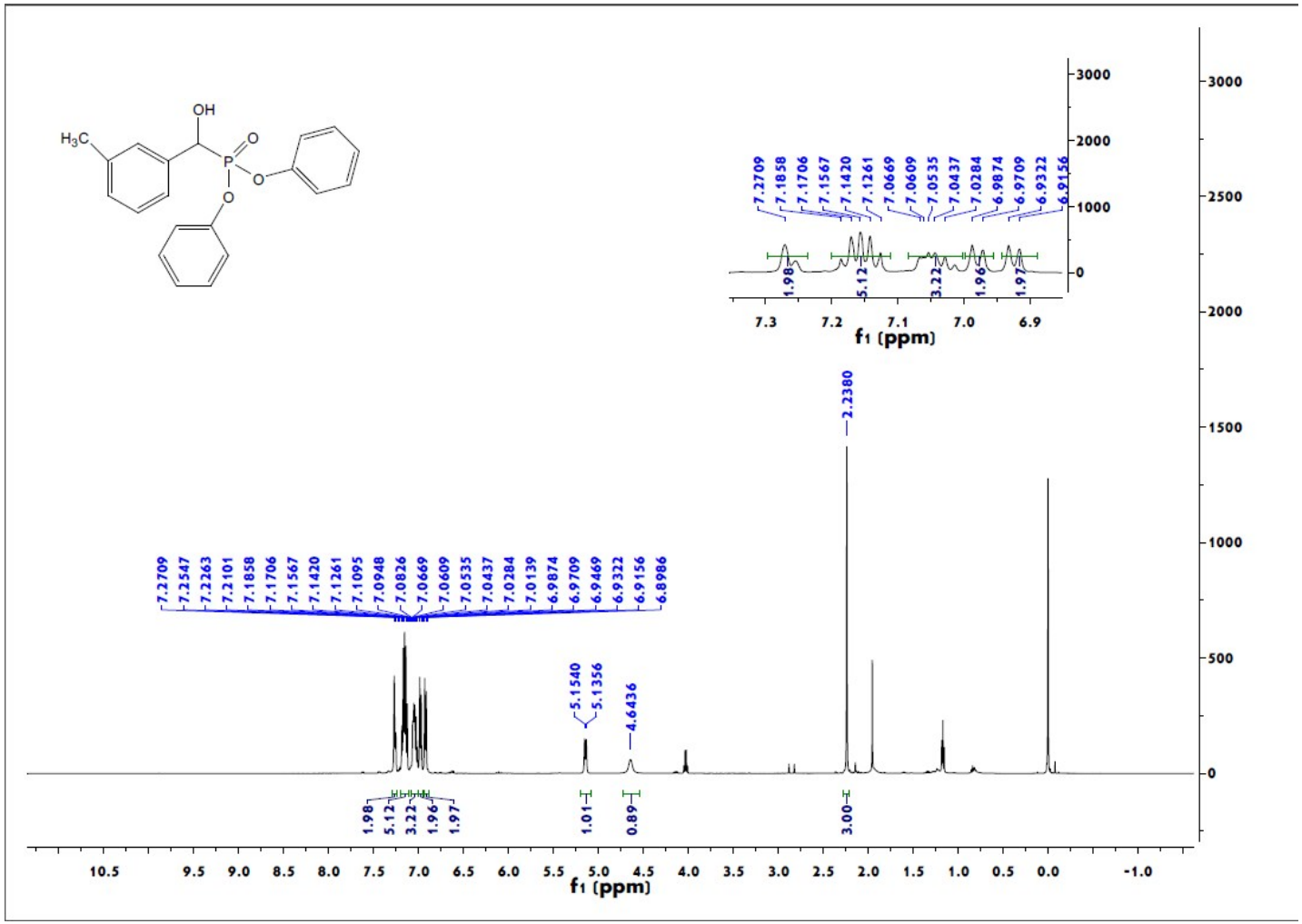

CH3
HO
P
O
O
O
7.7794
7.2894
7.2784
7.2472
7.2186
7.1826
7.1686
7.1475
7.1332
7.0865
7.0720
7.0065
6.9919
1.00
6.93
1.11
2.30
2.00
2.19
f1 (ppm)
5.6413
5.6226
3.7733
2.4529
0.85
3.20

2b

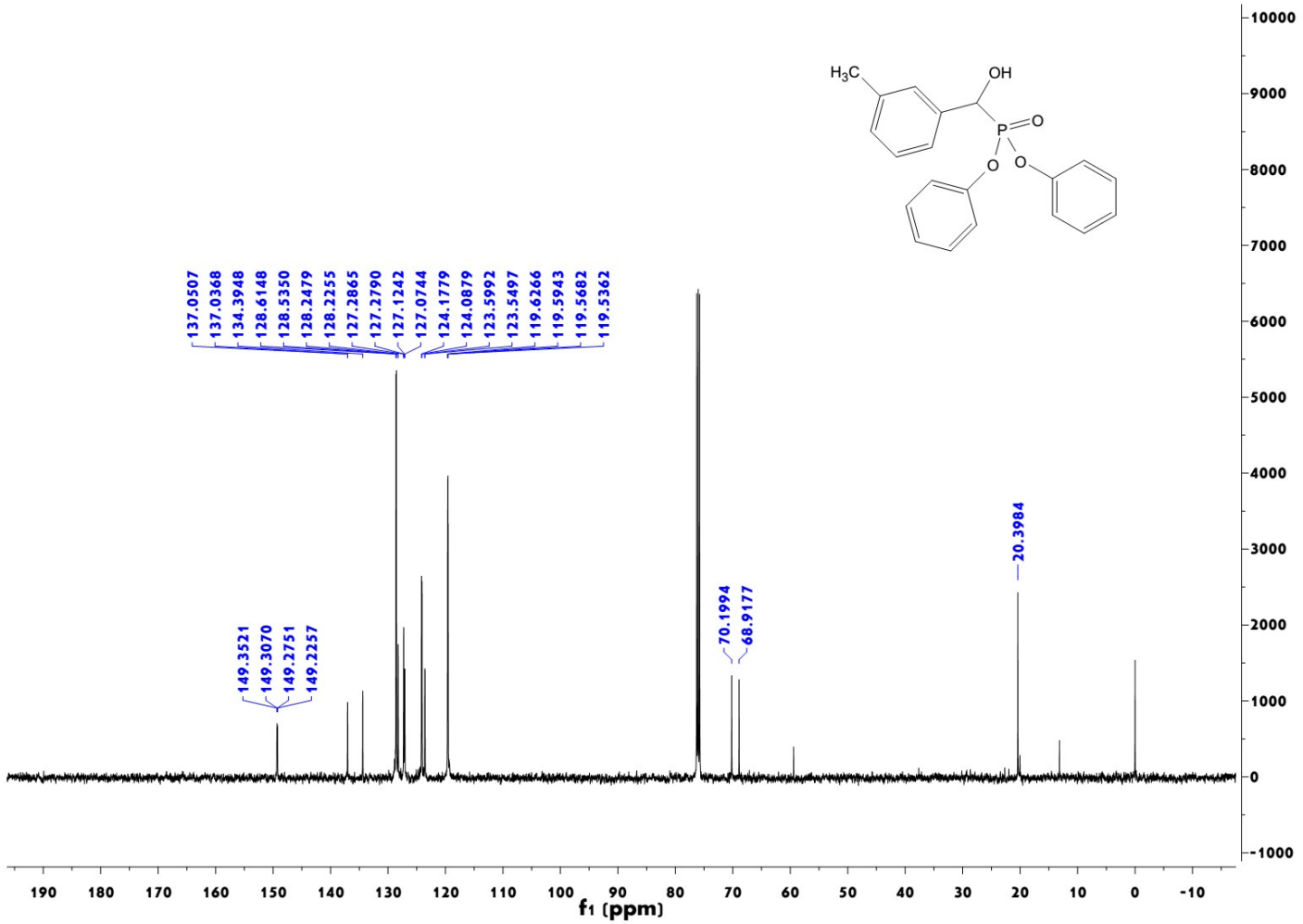

CH3
OH
O
P
O
O
O
136.2120
136.1480
133.9252
130.5317
129.6915
129.6446
128.6090
128.5924
127.6512
127.6173
126.4539
126.4398
125.2566
125.1820
120.6186
120.5888
120.4679
120.4390
150.3723
150.2912
67.8507
66.5566
19.6329
f1 (ppm)

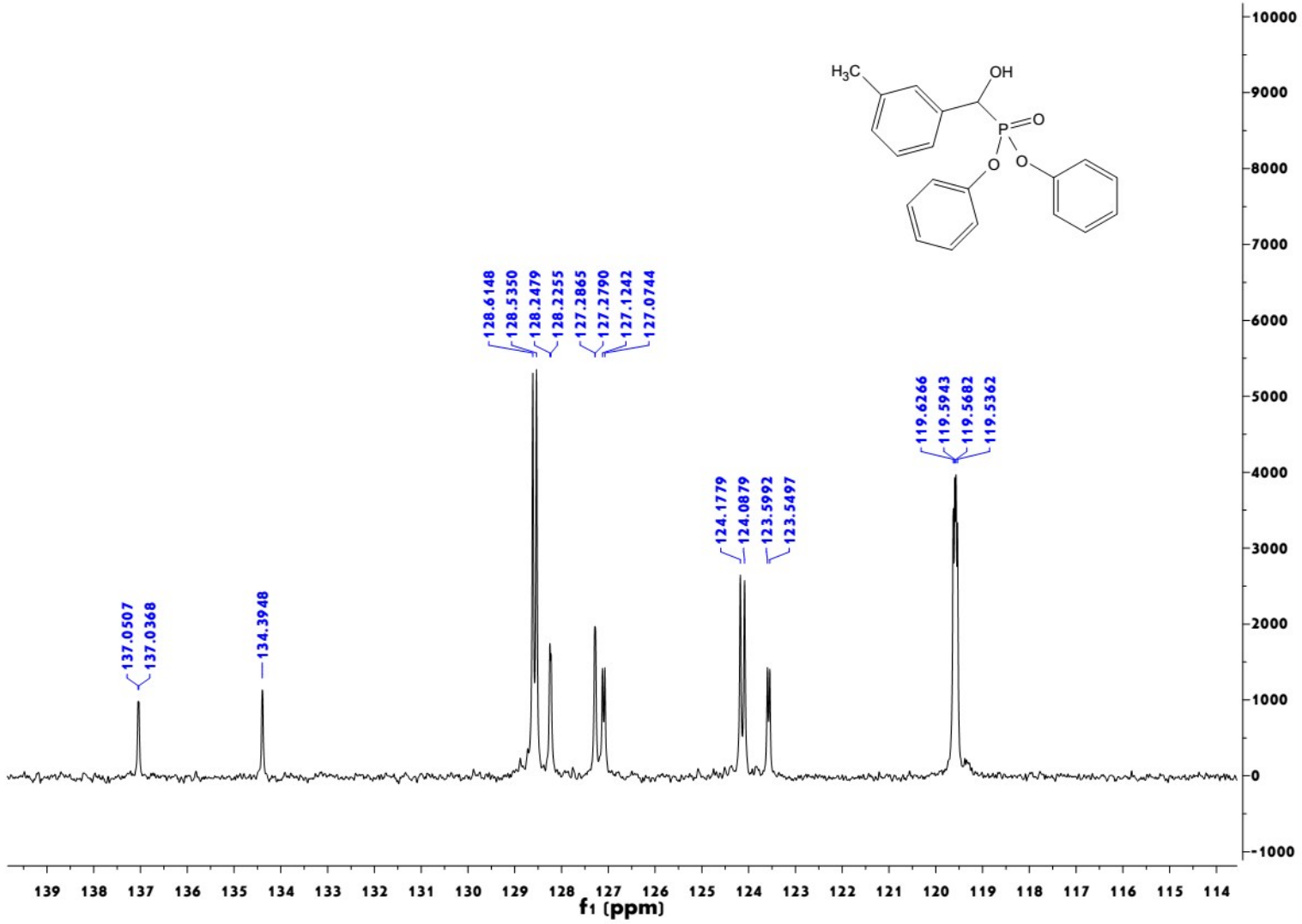

CH3
OH
O
P
O
O
O
136.2120
136.1480
133.9252
130.5317
129.6915
129.6446
128.6090
128.5924
127.6512
127.6173
126.4539
126.4398
125.2566
125.1820
120.6186
120.5888
120.4679
120.4390
f1 (ppm)

2c

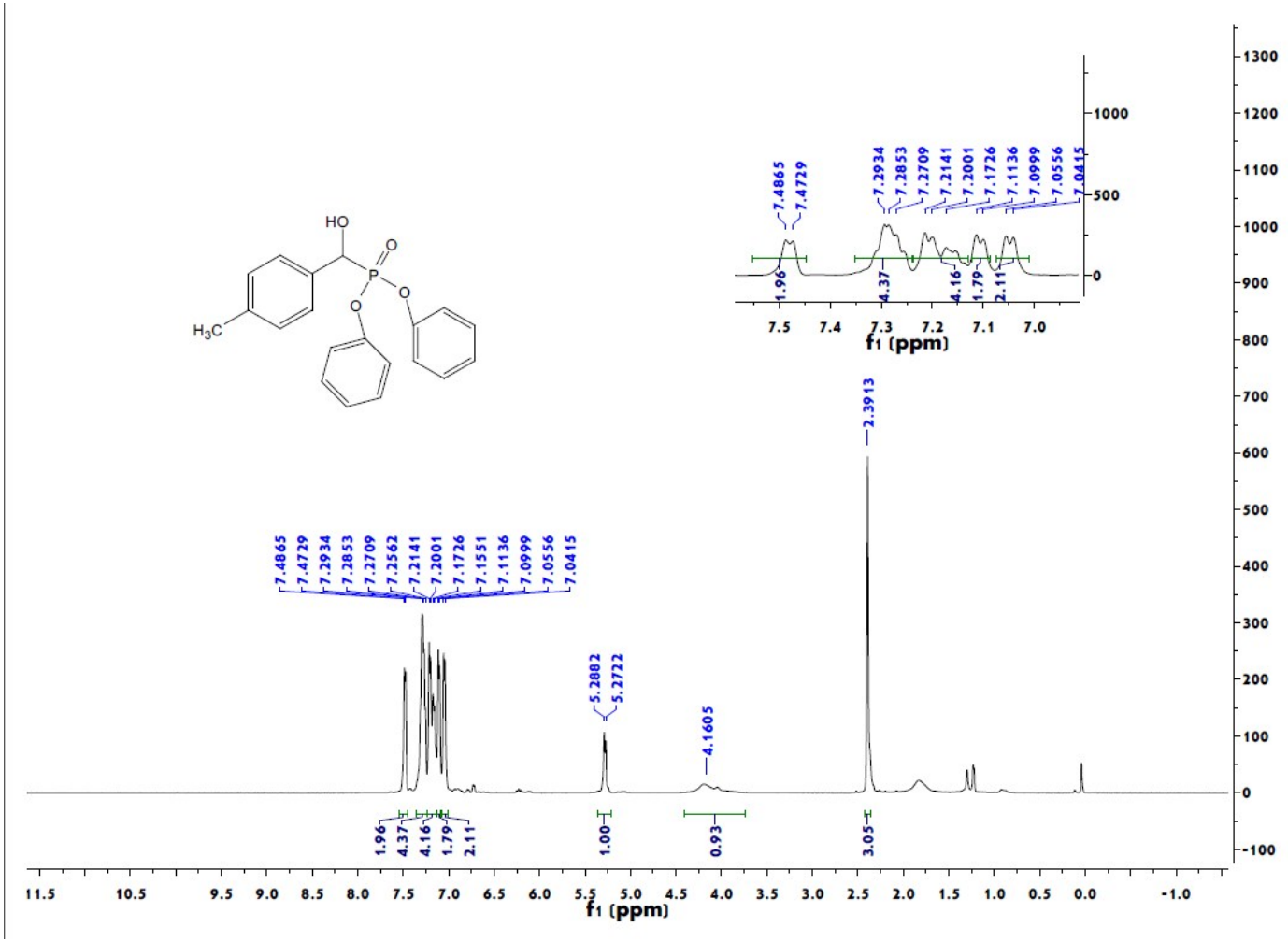

OH
H3C
O
P
O
O
7.2709
7.1858
7.1706
7.1567
7.1420
7.1261
7.0669
7.0609
7.0535
7.0437
7.0284
6.9874
6.9709
6.9322
6.9156
1.98
5.12
3.22
1.96
1.97
f1 (ppm)
2.2380
5.1540
5.1356
4.6436
1.01
0.89
3.00

2c

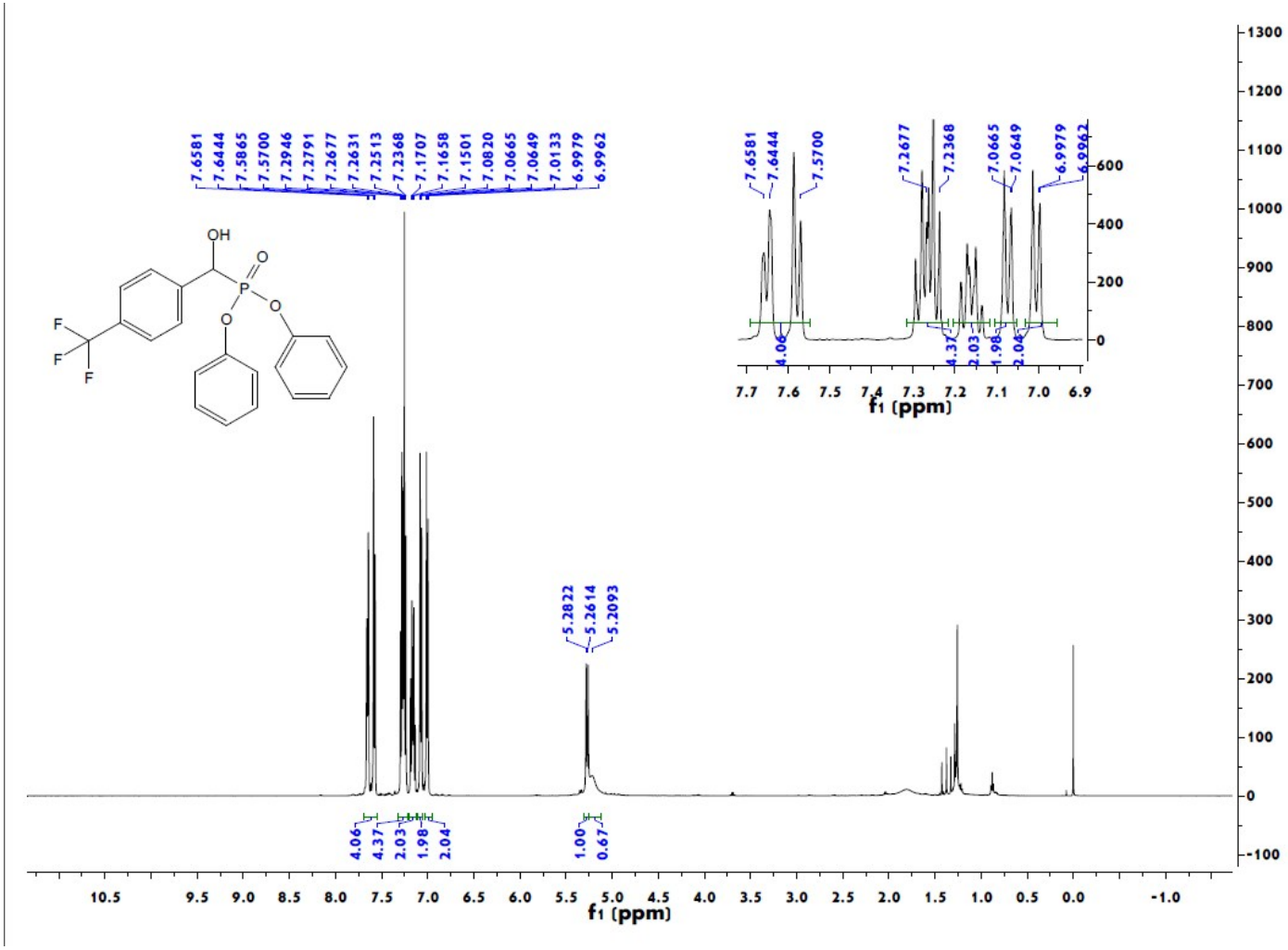

137.0507
137.0368
134.3948
128.6148
128.5350
128.2479
128.2255
127.2865
127.2790
127.1242
127.0744
124.1779
124.0879
123.5992
123.5497
119.6266
119.5943
119.5682
119.5362
149.3521
149.3070
149.2751
149.2257
70.1994
68.9177
20.3984
H3C
OH
P
O
O
O
f1 (ppm)


H3C
OH
P
O
O
O
128.6148
128.5350
128.2479
128.2255
127.2865
127.2790
127.1242
127.0744
124.1779
124.0879
123.5992
123.5497
119.6266
119.5943
119.5682
119.5362
137.0507
137.0368
134.3948
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
139 138 137 136 135 134 133 132 131 130 129 128 127 126 125 124 123 122 121 120 119 118 117 116 115 114
f1 (ppm)

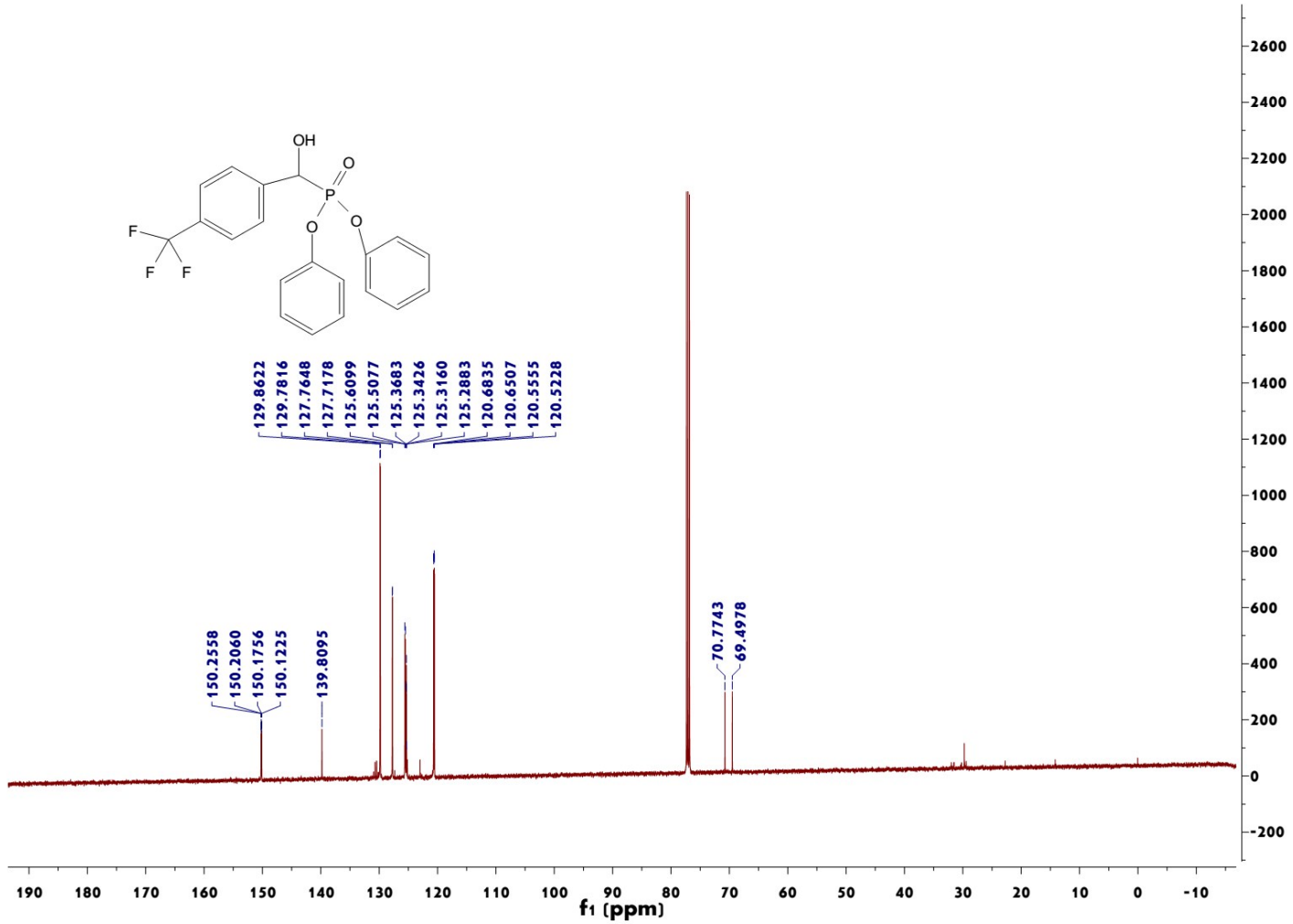
2d

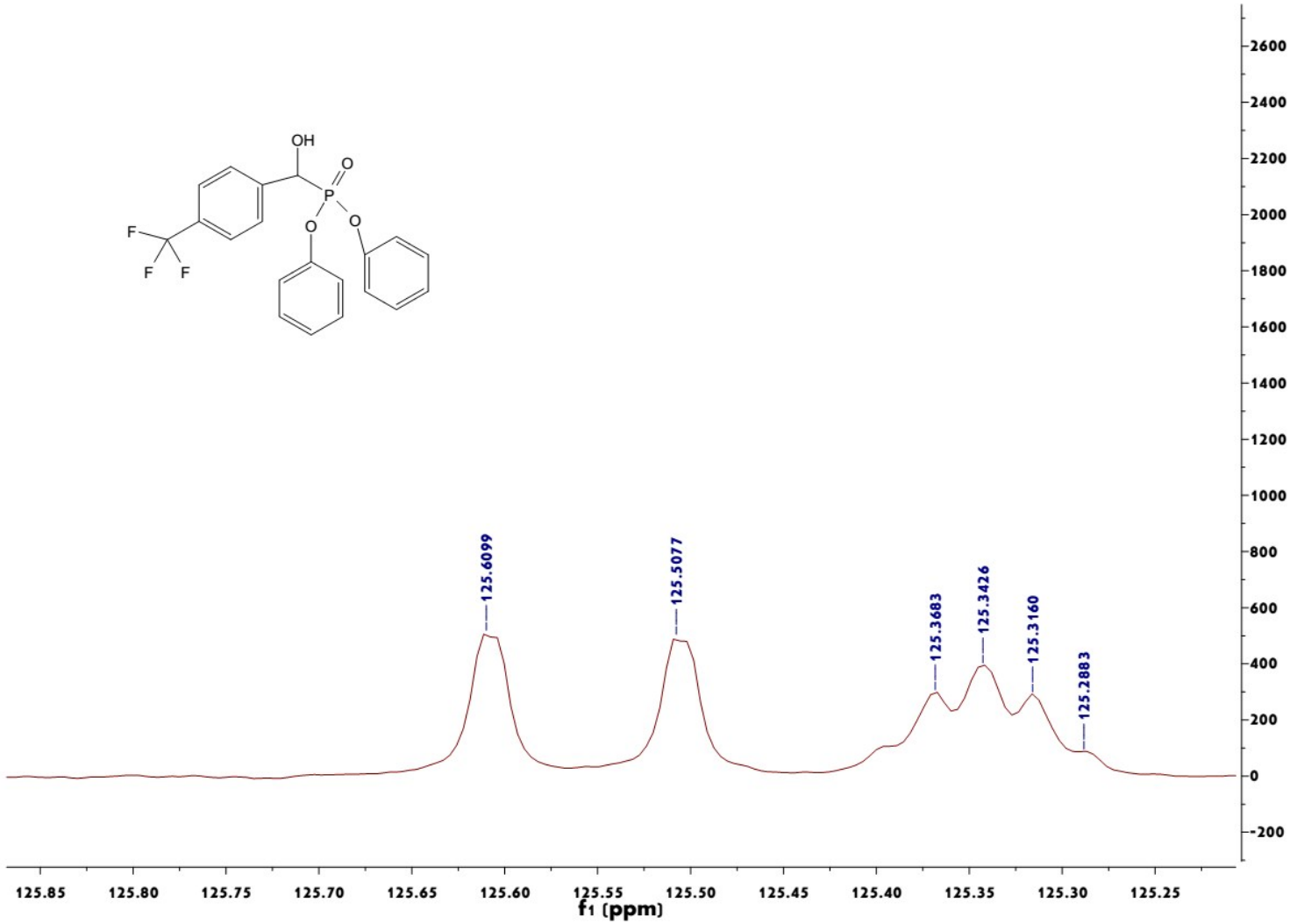

HO
O
P
O
O
O
H3C
7.4865
7.4729
7.2934
7.2853
7.2709
7.2562
7.2141
7.2001
7.1726
7.1551
7.1136
7.0999
7.0556
7.0415
5.2882
5.2722
4.1605
2.3913
1.96
4.37
4.16
1.79
2.11
1.00
0.93
3.05
f1 (ppm)

2e

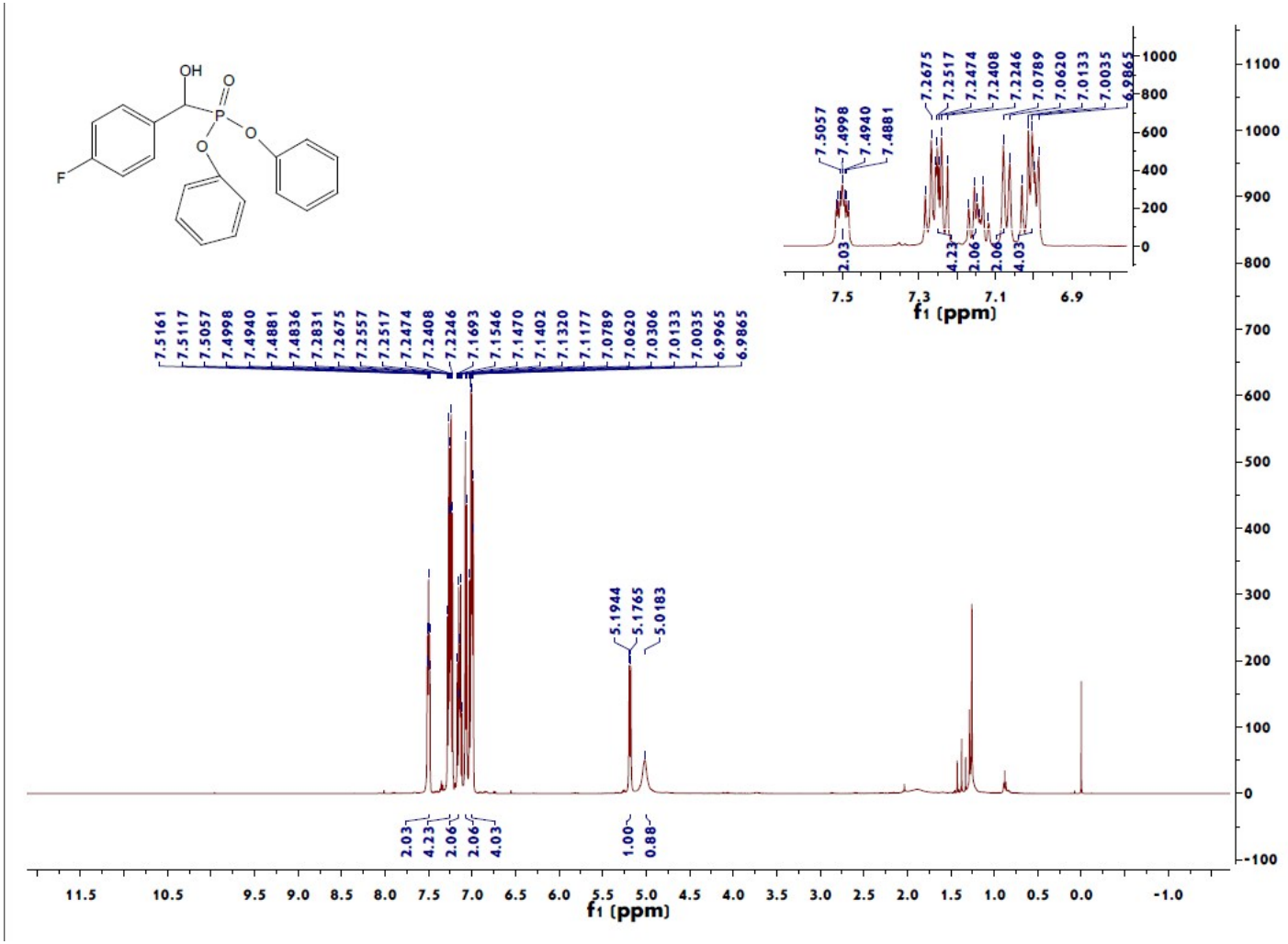

7.6581
7.6444
7.5865
7.5700
7.2946
7.2791
7.2677
7.2631
7.2513
7.2368
7.1707
7.1658
7.1501
7.0820
7.0665
7.0649
7.0133
6.9979
6.9962
OH
O
P
O
O
F
F
F
5.2822
5.2614
5.2093
4.06
4.37
2.03
1.98
2.04
1.00
0.67
f1 (ppm)

2e

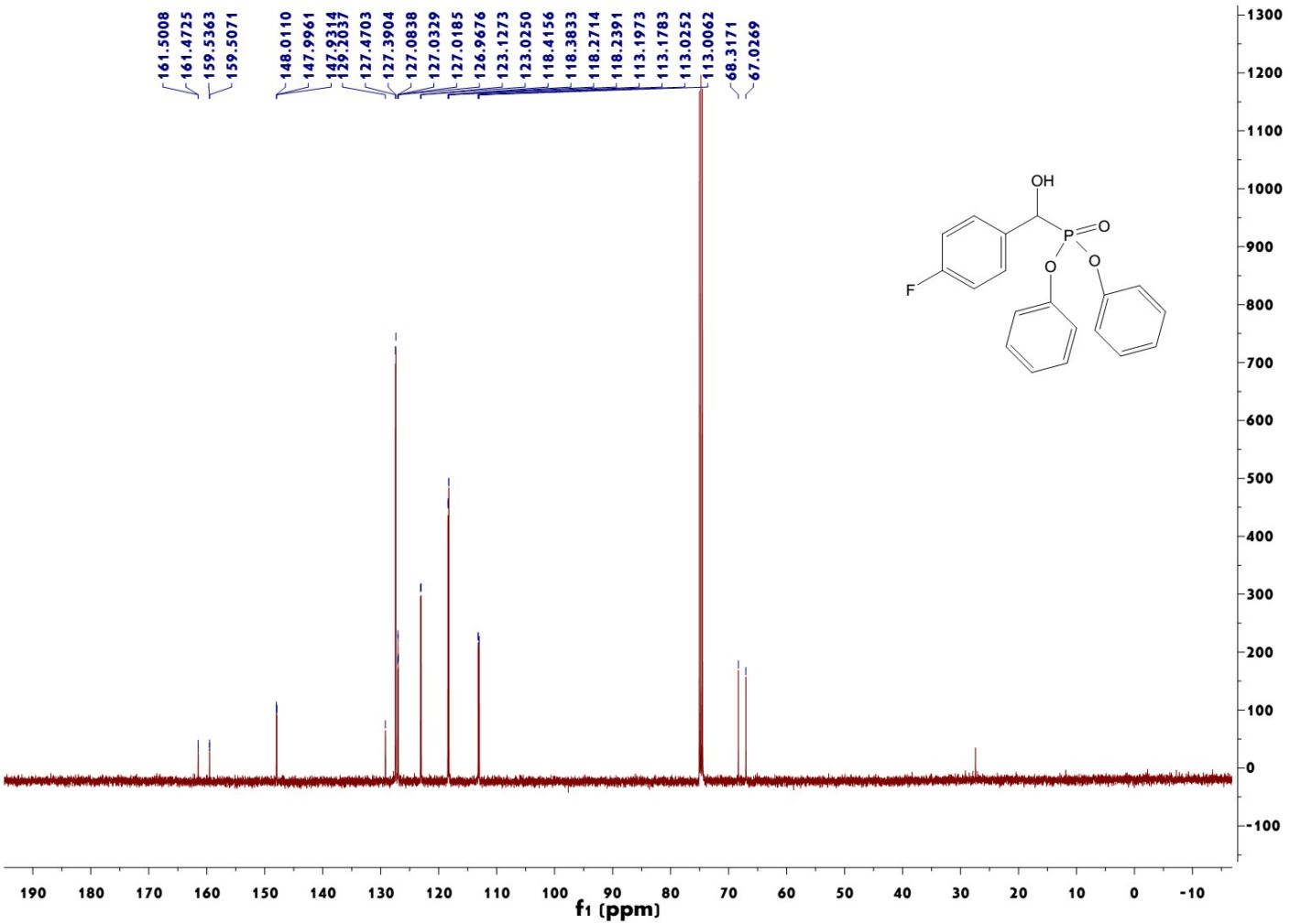

OH
O
P
O
O
F
F
F
129.8622
129.7816
127.7648
127.7178
125.6099
125.5077
125.3683
125.3426
125.3160
125.2883
120.6835
120.6507
120.5555
120.5228
150.2558
150.2060
150.1756
150.1225
139.8095
70.7743
69.4978
f1 (ppm)

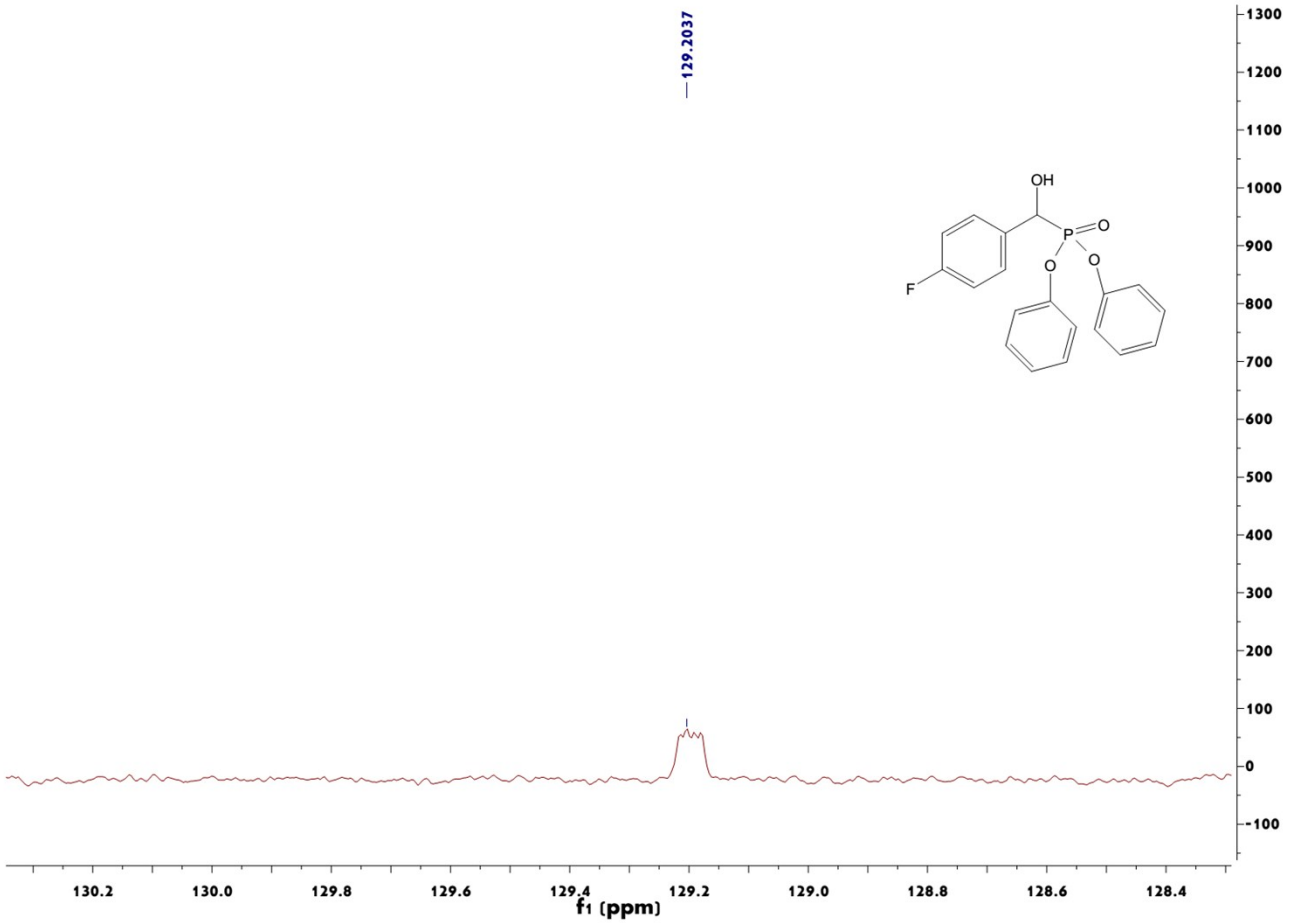

OH
O
P
O
O
F
F
F
125.6099
125.5077
125.3683
125.3426
125.3160
125.2883
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
125.85
125.80
125.75
125.70
125.65
125.60
125.55
125.50
125.45
125.40
125.35
125.30
125.25
f1 (ppm)

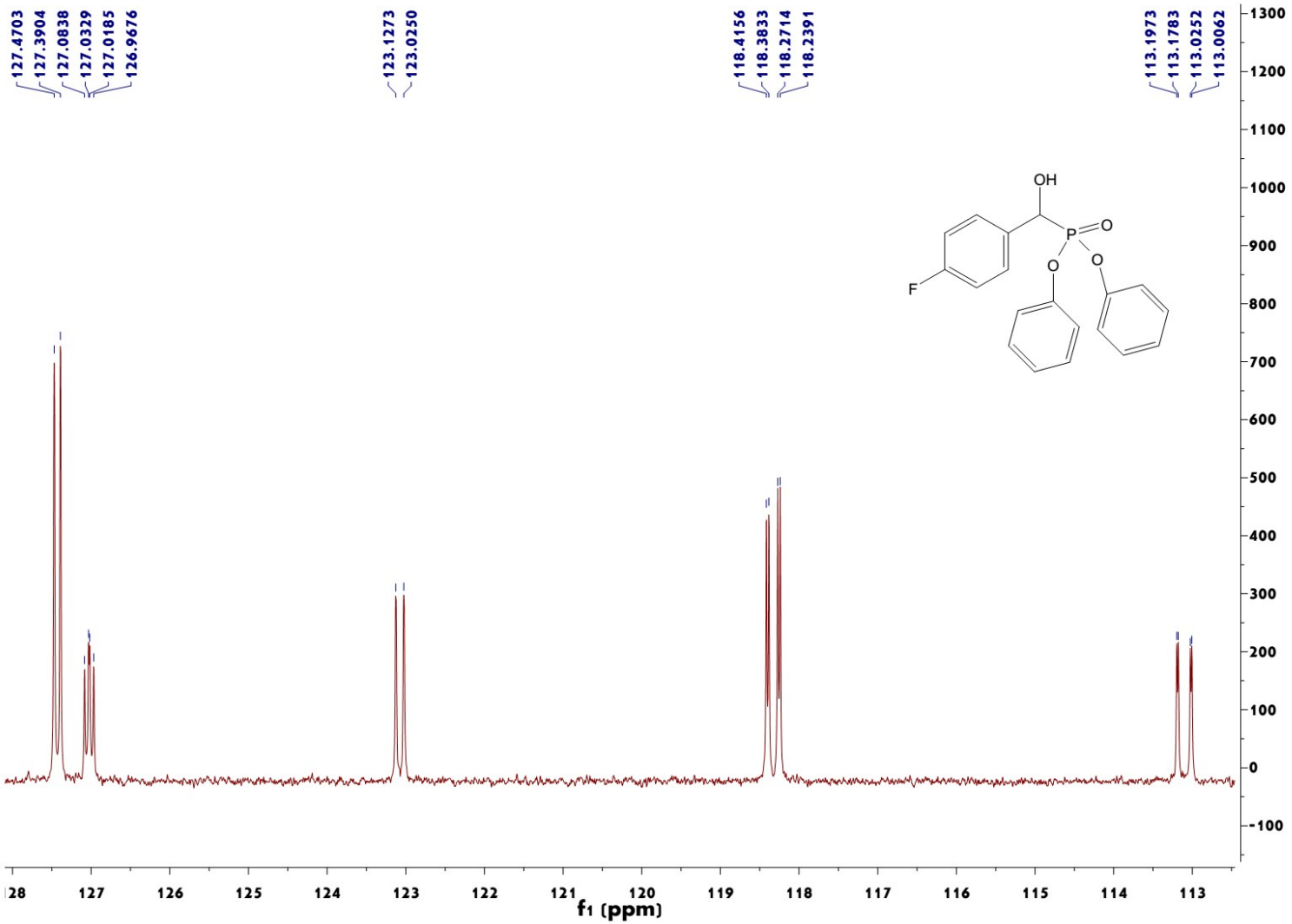
2f


OH
O
P
O
O
F
7.5161
7.5117
7.5057
7.4998
7.4940
7.4881
7.4836
7.2831
7.2675
7.2557
7.2517
7.2474
7.2408
7.2246
7.1693
7.1546
7.1470
7.1402
7.1320
7.1177
7.0789
7.0620
7.0306
7.0133
7.0035
6.9965
6.9865
5.1944
5.1765
5.0183
2.03
4.23
2.06
2.06
4.03
1.00
0.88
f1 (ppm)

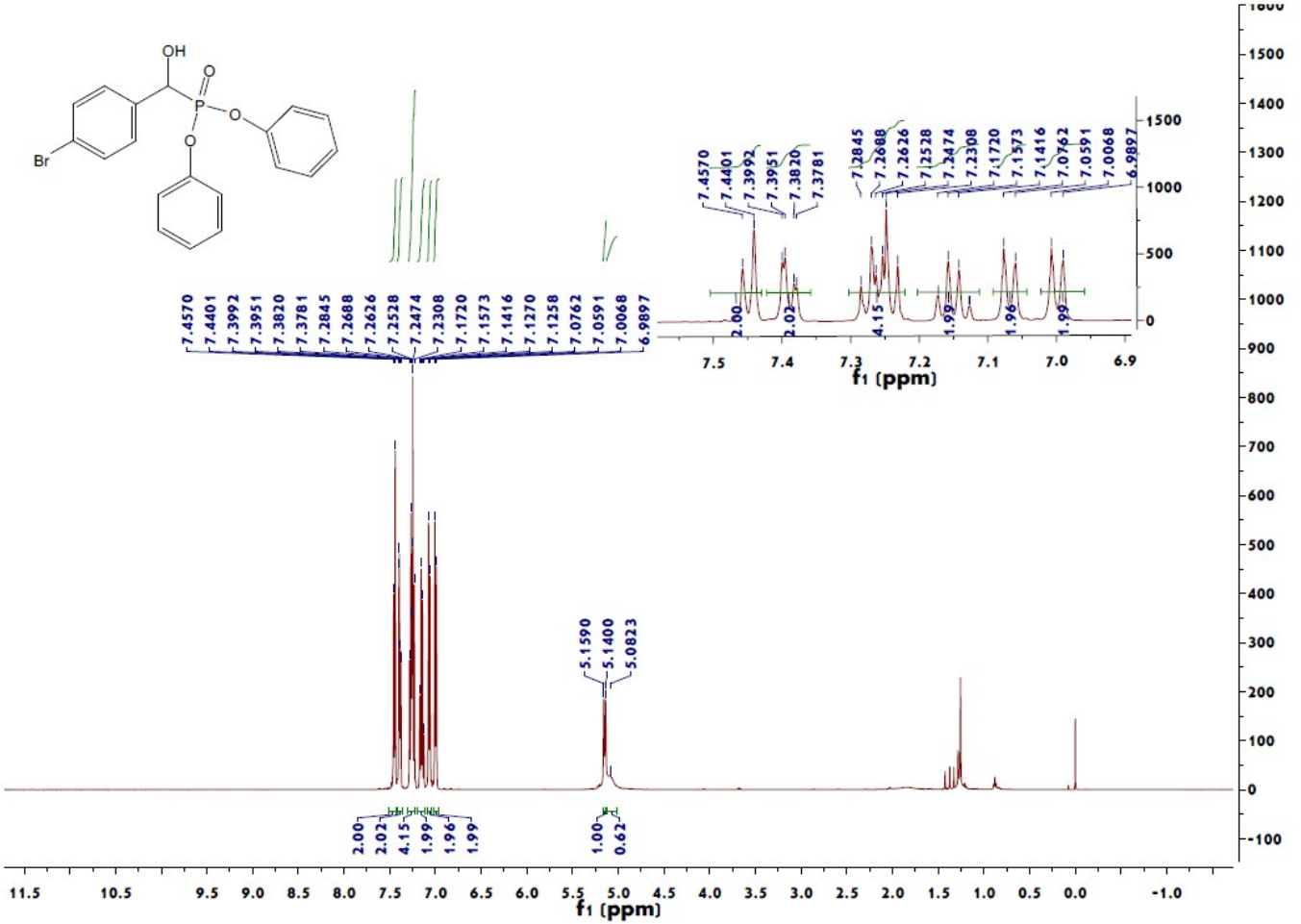
2f


161.5008
161.4725
159.5363
159.5071
148.0110
147.9961
147.8314
129.2037
127.4703
127.3904
127.0838
127.0329
127.0185
126.9676
123.1273
123.0250
118.4156
118.3833
118.2714
118.2391
113.1973
113.1783
113.0252
113.0062
68.3171
67.0269
OH
P
O
O
O
F
f1 (ppm)

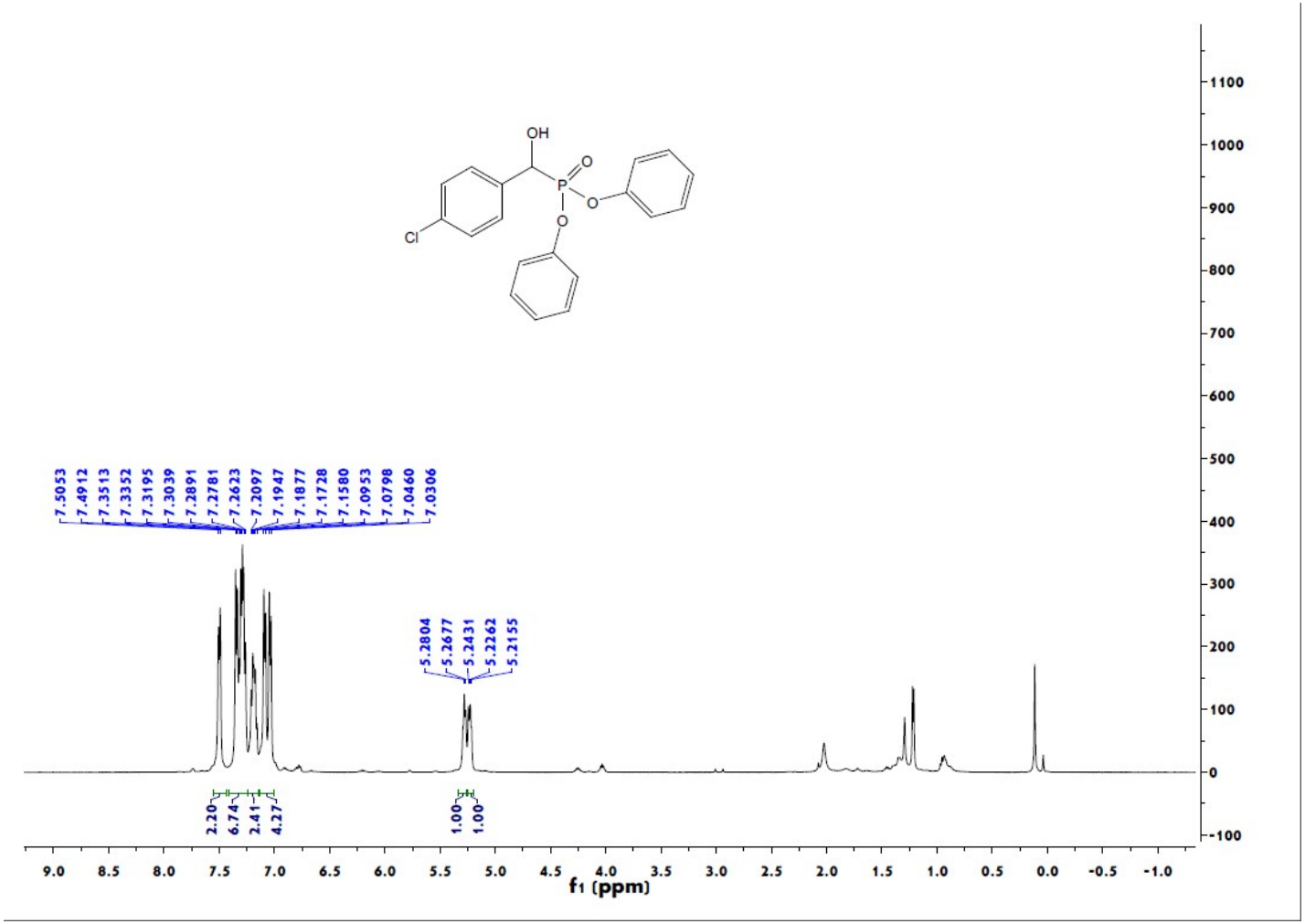

129.2037
OH
P
O
O
O
F
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
130.2
130.0
129.8
129.6
129.4
129.2
129.0
128.8
128.6
128.4
f1 (ppm)


127.4703
127.3904
127.0838
127.0329
127.0185
126.9676
123.1273
123.0250
118.4156
118.3833
118.2714
118.2391
113.1973
113.1783
113.0252
113.0062
OH
P
O
O
O
F
f1 (ppm)

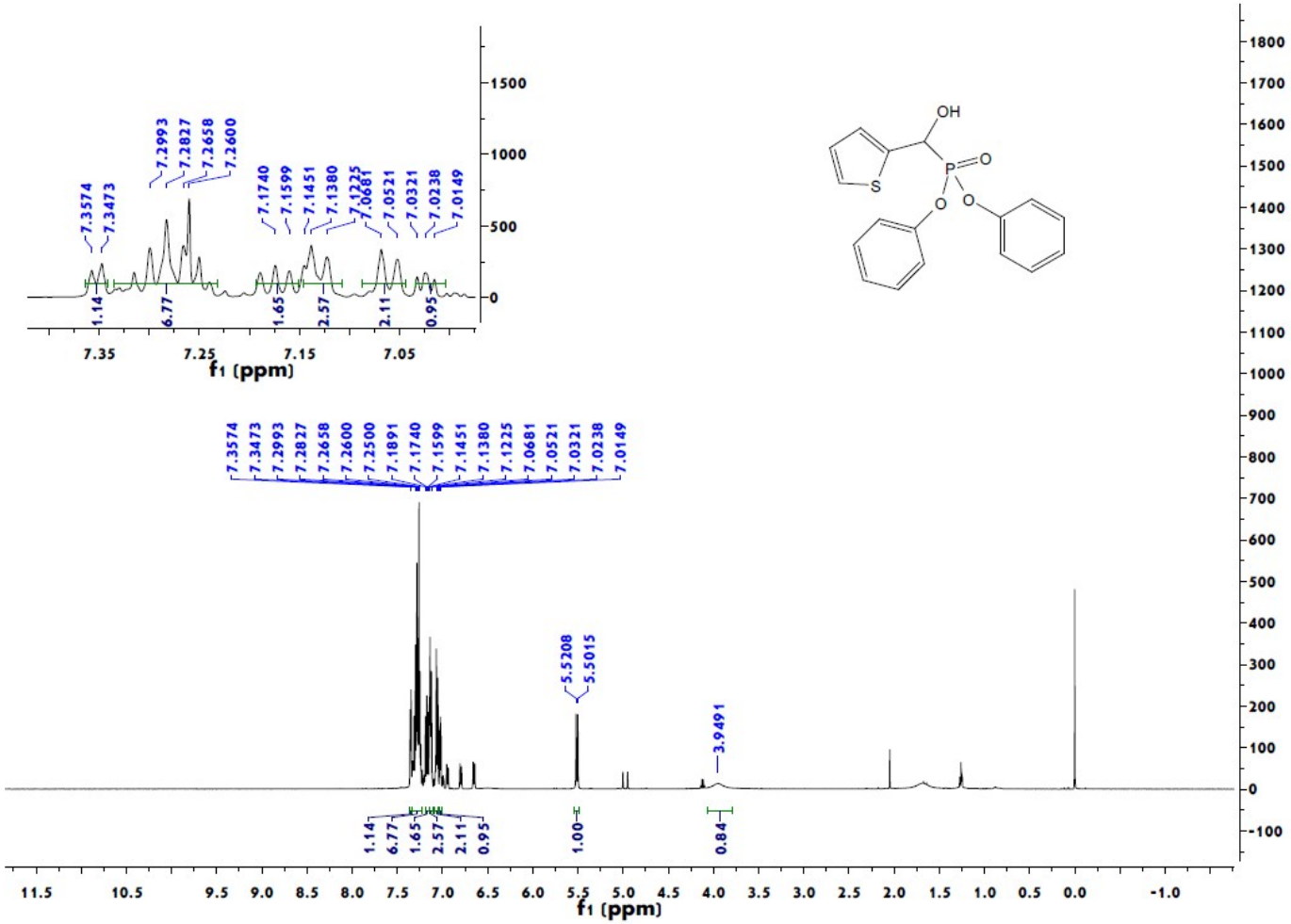
2g


OH
O
P
O
O
Br
7.4570
7.4401
7.3992
7.3951
7.3820
7.3781
7.2845
7.2688
7.2626
7.2528
7.2474
7.2308
7.1720
7.1573
7.1416
7.1270
7.1258
7.0762
7.0591
7.0068
6.9897
5.1590
5.1400
5.0823
2.00
2.02
4.15
1.99
1.96
1.99
1.00
0.62
f1 (ppm)

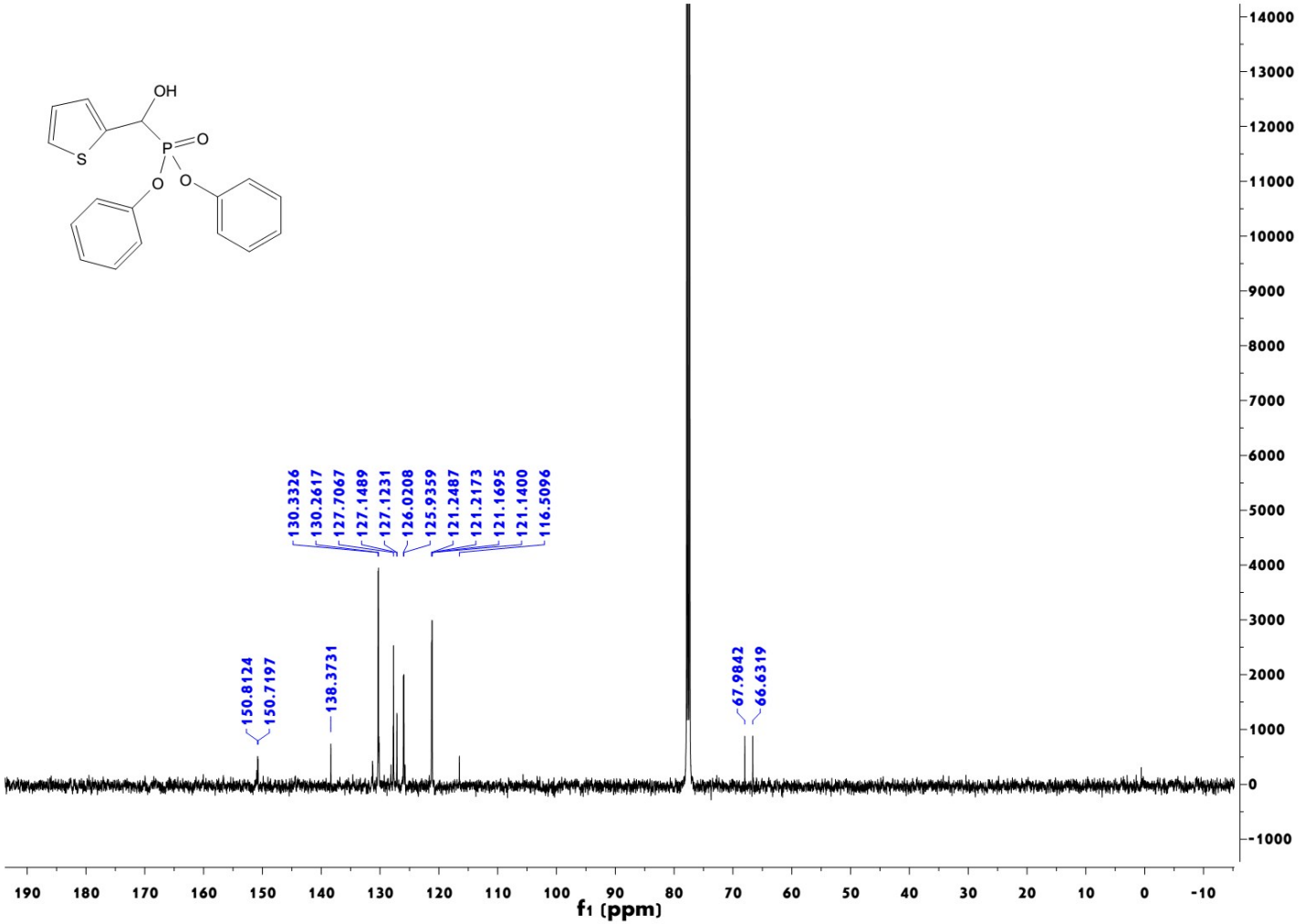
2h

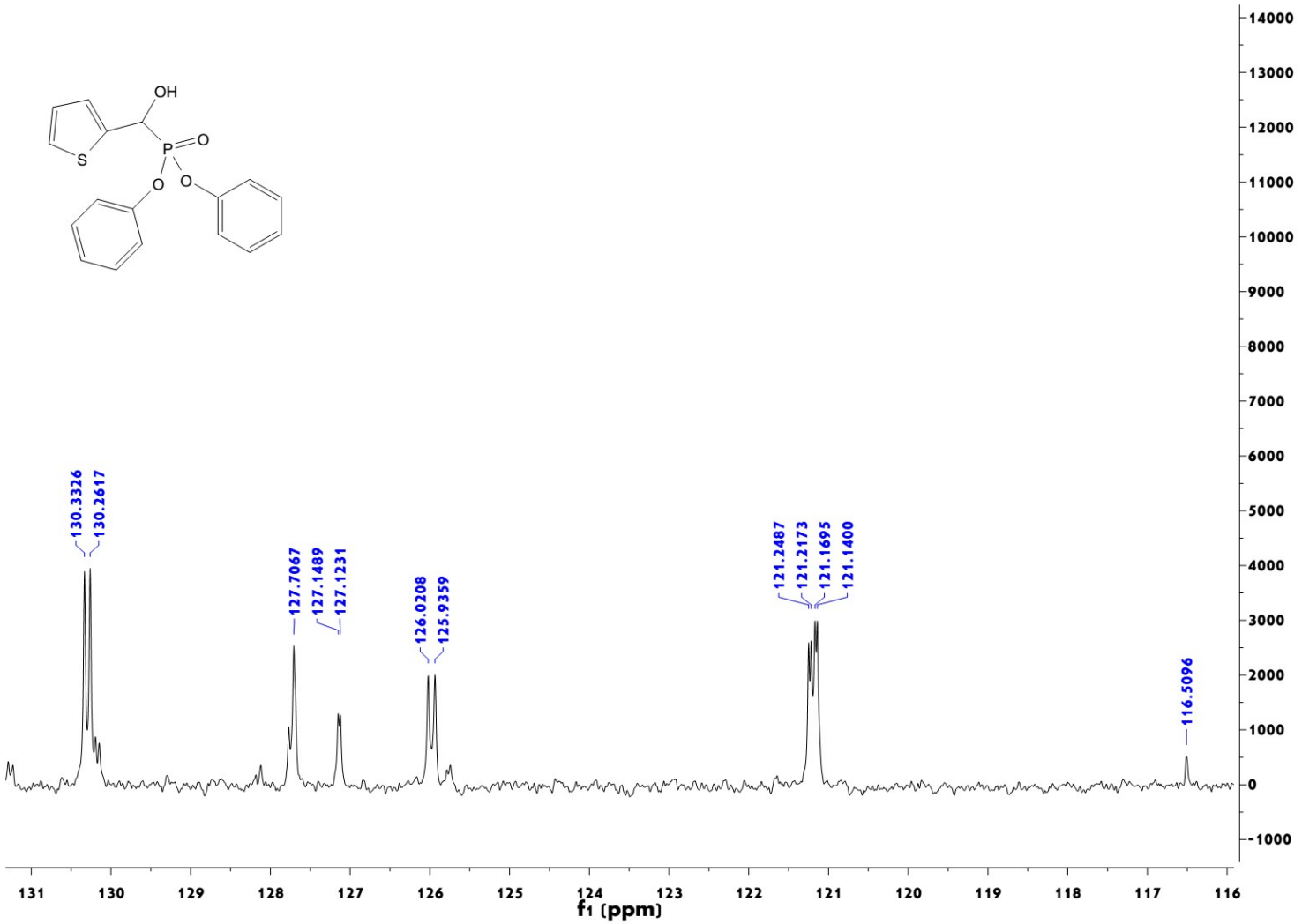

OH
O
P
O
O
Cl
7.5053
7.4912
7.3513
7.3352
7.3195
7.3039
7.2891
7.2781
7.2623
7.2097
7.1947
7.1877
7.1728
7.1580
7.0953
7.0798
7.0460
7.0306
5.2804
5.2677
5.2431
5.2262
5.2155
2.20
6.74
2.41
4.27
1.00
1.00
f1 (ppm)

2i

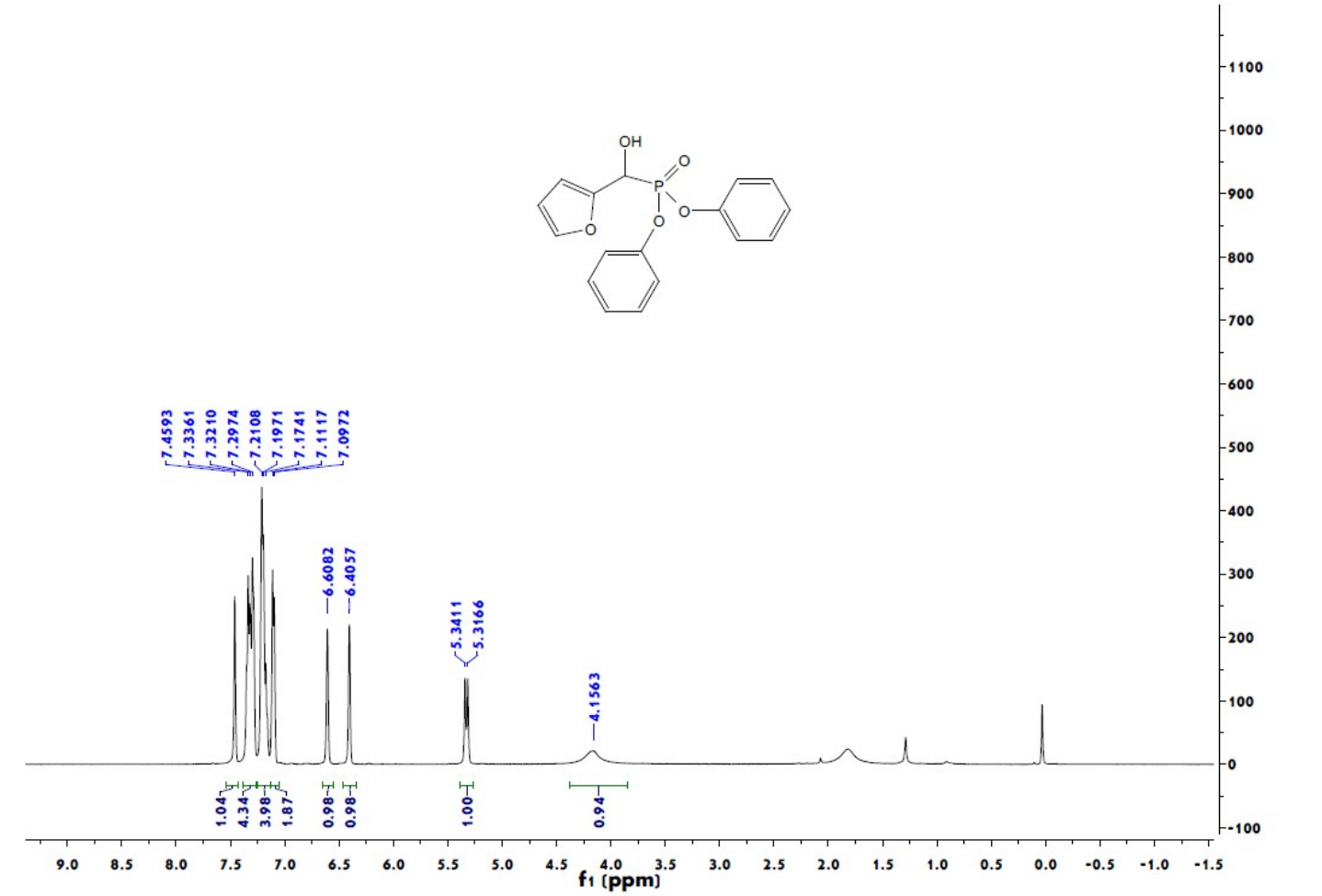

OH
S
P
O
O
O
7.3574
7.3473
7.2993
7.2827
7.2658
7.2600
7.2500
7.1891
7.1740
7.1599
7.1451
7.1380
7.1225
7.0681
7.0521
7.0321
7.0238
7.0149
5.5208
5.5015
3.9491
1.14
6.77
1.65
2.57
2.11
0.95
1.00
0.84
f1 (ppm)

2i

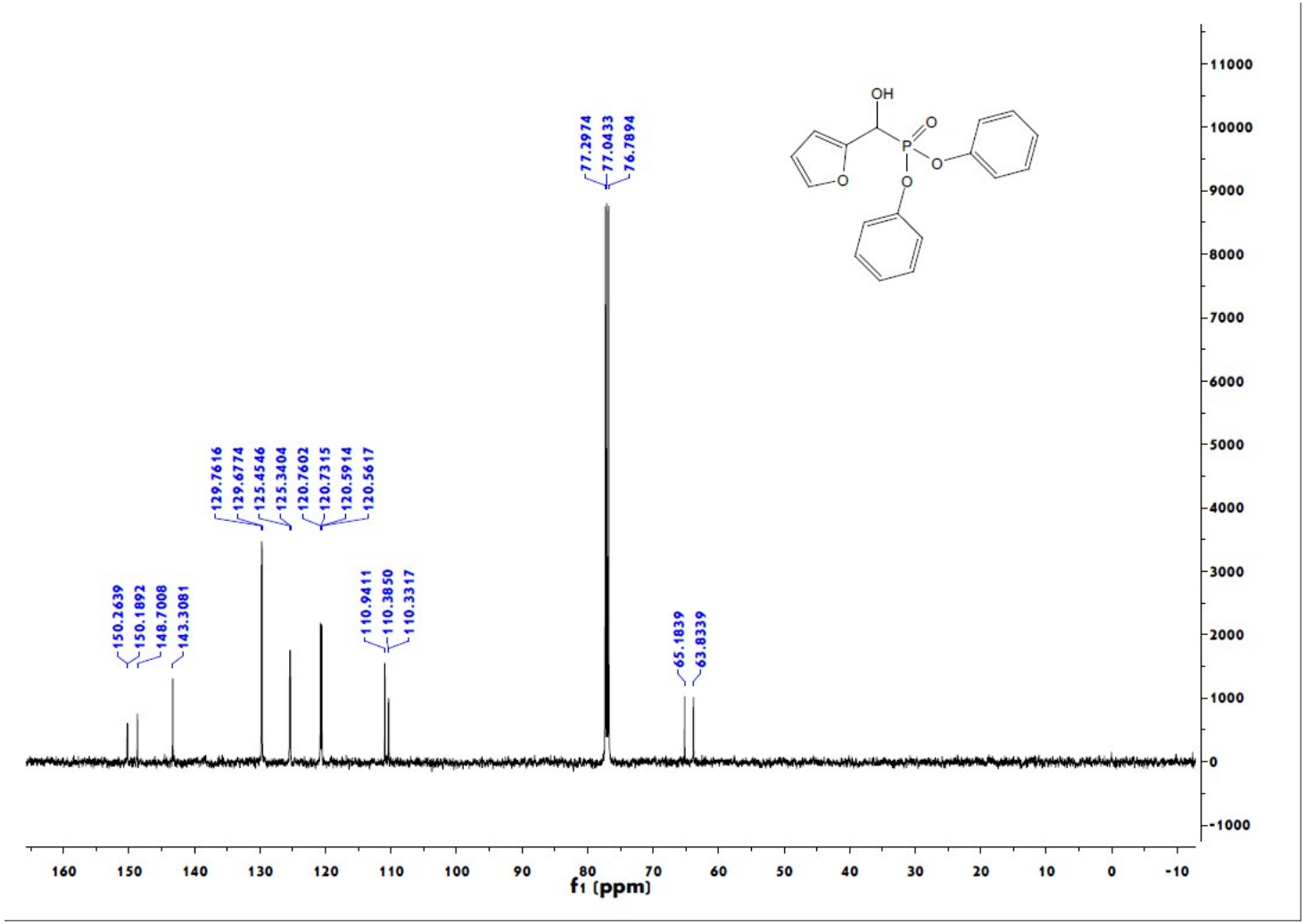

OH
S
P
O
O
O
130.3326
130.2617
127.7067
127.1489
127.1231
126.0208
125.9359
121.2487
121.2173
121.1695
121.1400
116.5096
150.8124
150.7197
138.3731
67.9842
66.6319
f1 (ppm)


OH
S
P
O
O
O
130.3326
130.2617
127.7067
127.1489
127.1231
126.0208
125.9359
121.2487
121.2173
121.1695
121.1400
116.5096
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
131
130
129
128
127
126
125
124
123
122
121
120
119
118
117
116
f1 (ppm)

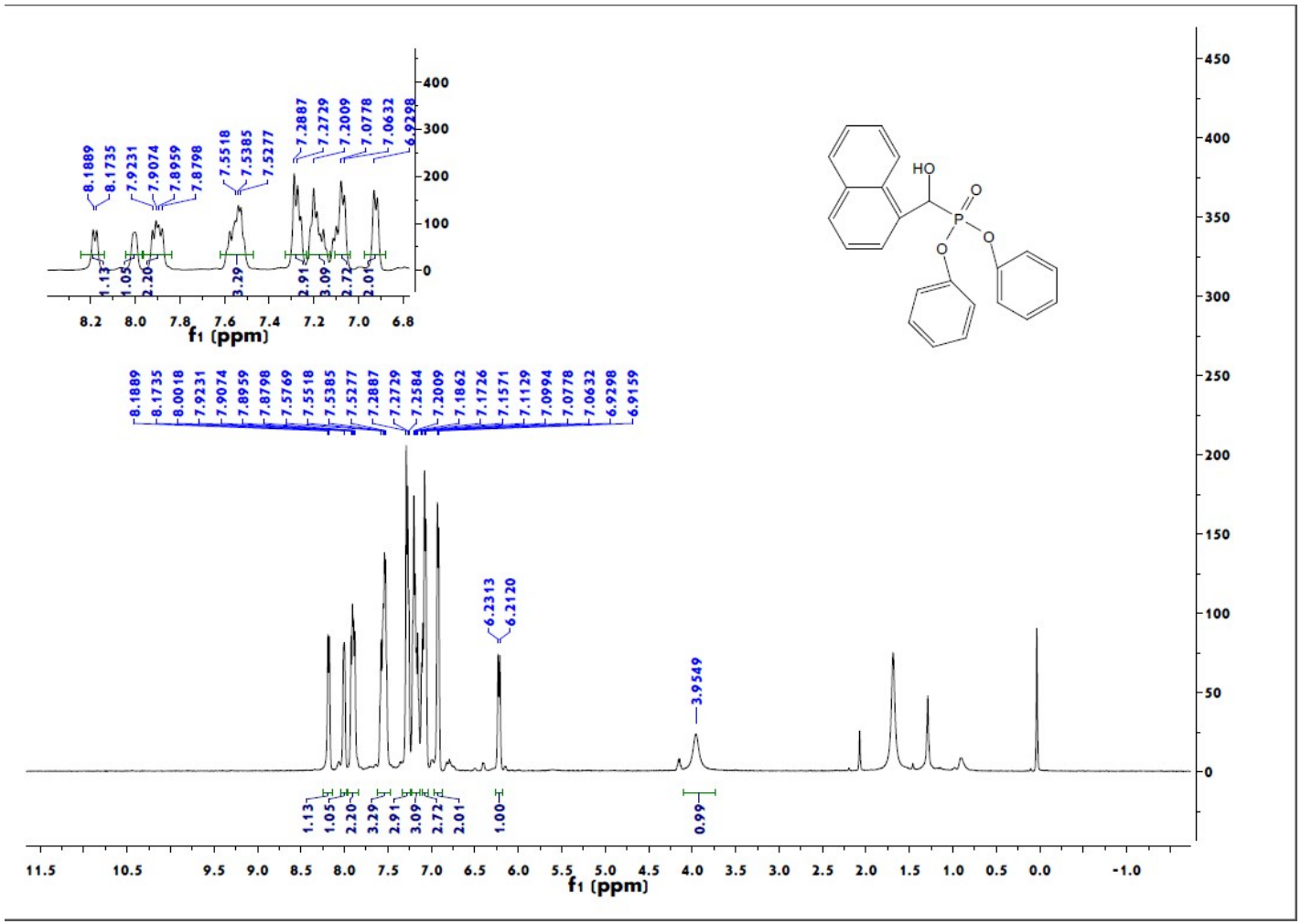
2j


OH
O
P
O
O
O
7.4593
7.3361
7.3210
7.2974
7.2108
7.1971
7.1741
7.1117
7.0972
6.6082
6.4057
5.3411
5.3166
4.1563
1.04
4.34
3.98
1.87
0.98
0.98
1.00
0.94
f1 (ppm)

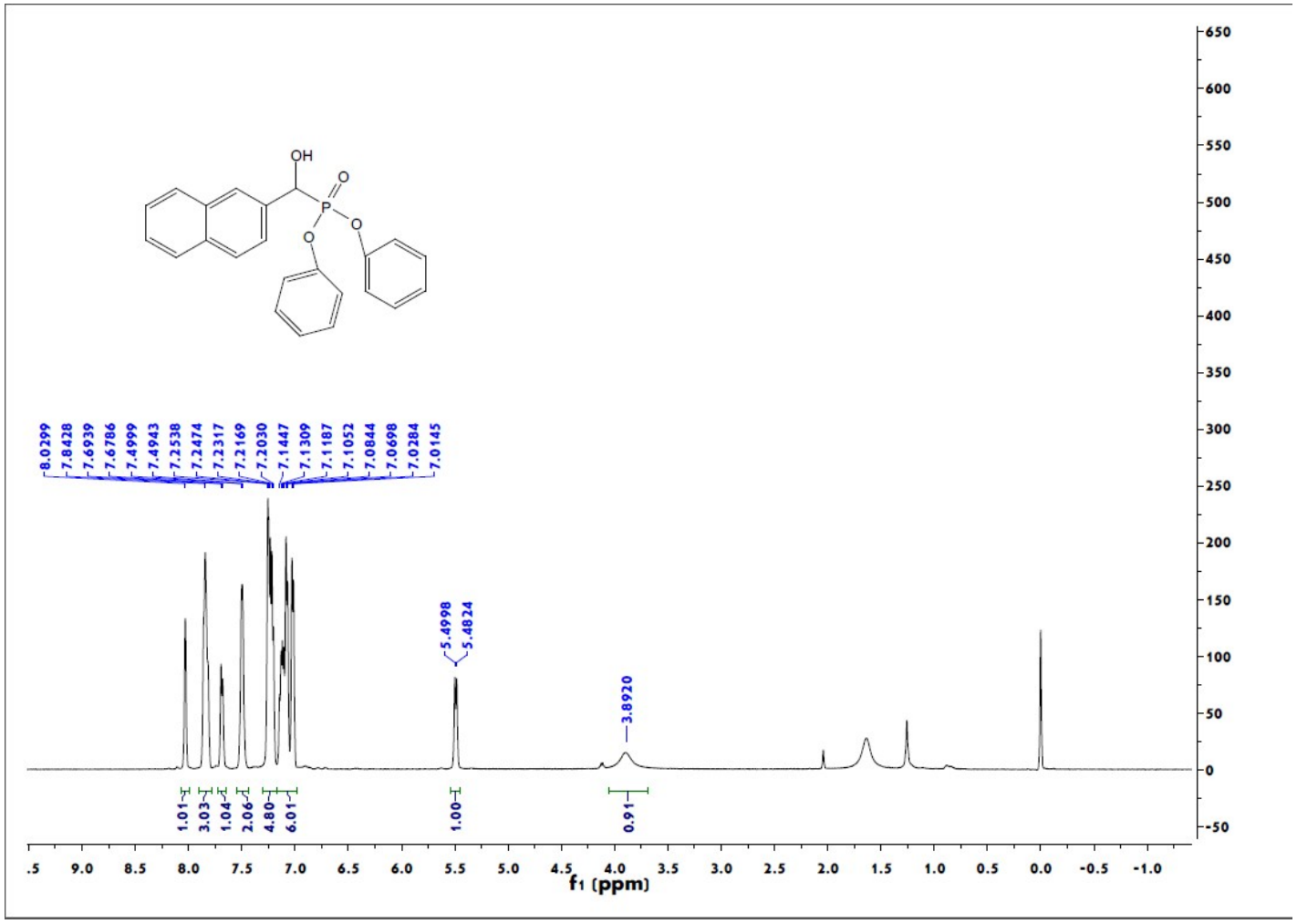
2j


77.2974
77.0433
76.7894
129.7616
129.6774
125.4546
125.3404
120.7602
120.7315
120.5914
120.5617
150.2639
150.1892
148.7008
143.3081
110.9411
110.3850
110.3317
65.1839
63.8339
OH
O
P
O
O
O
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

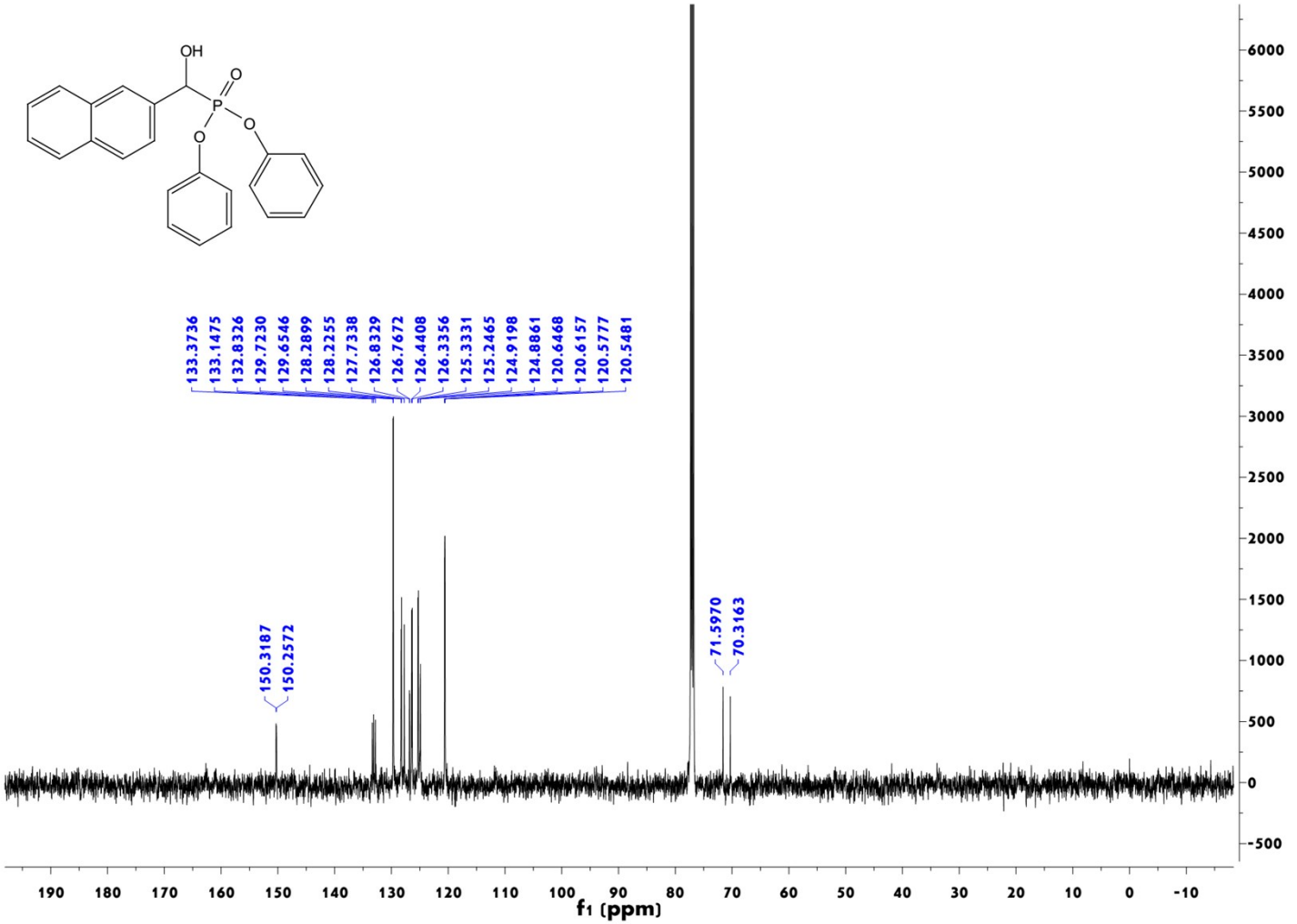
2k

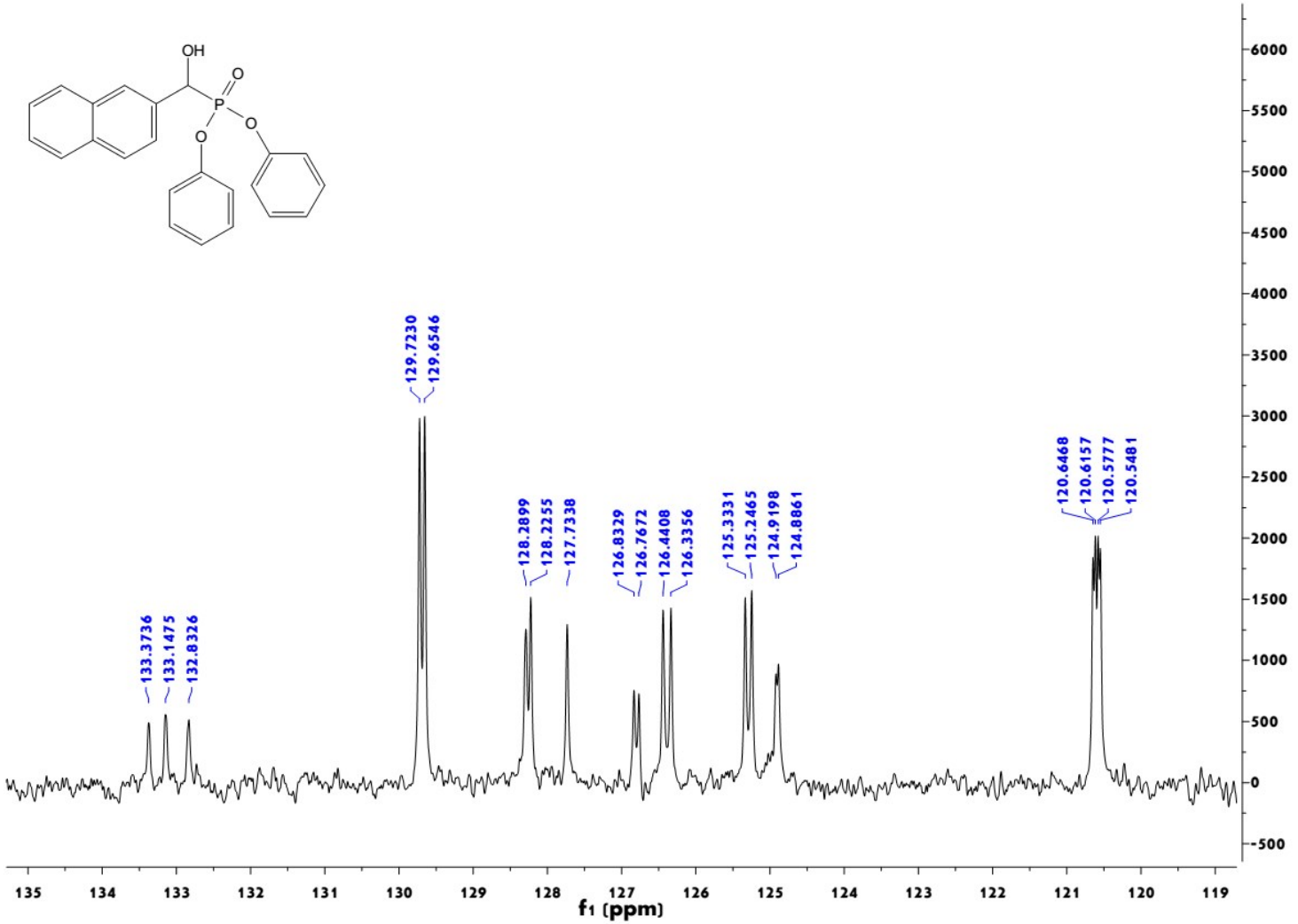


2l

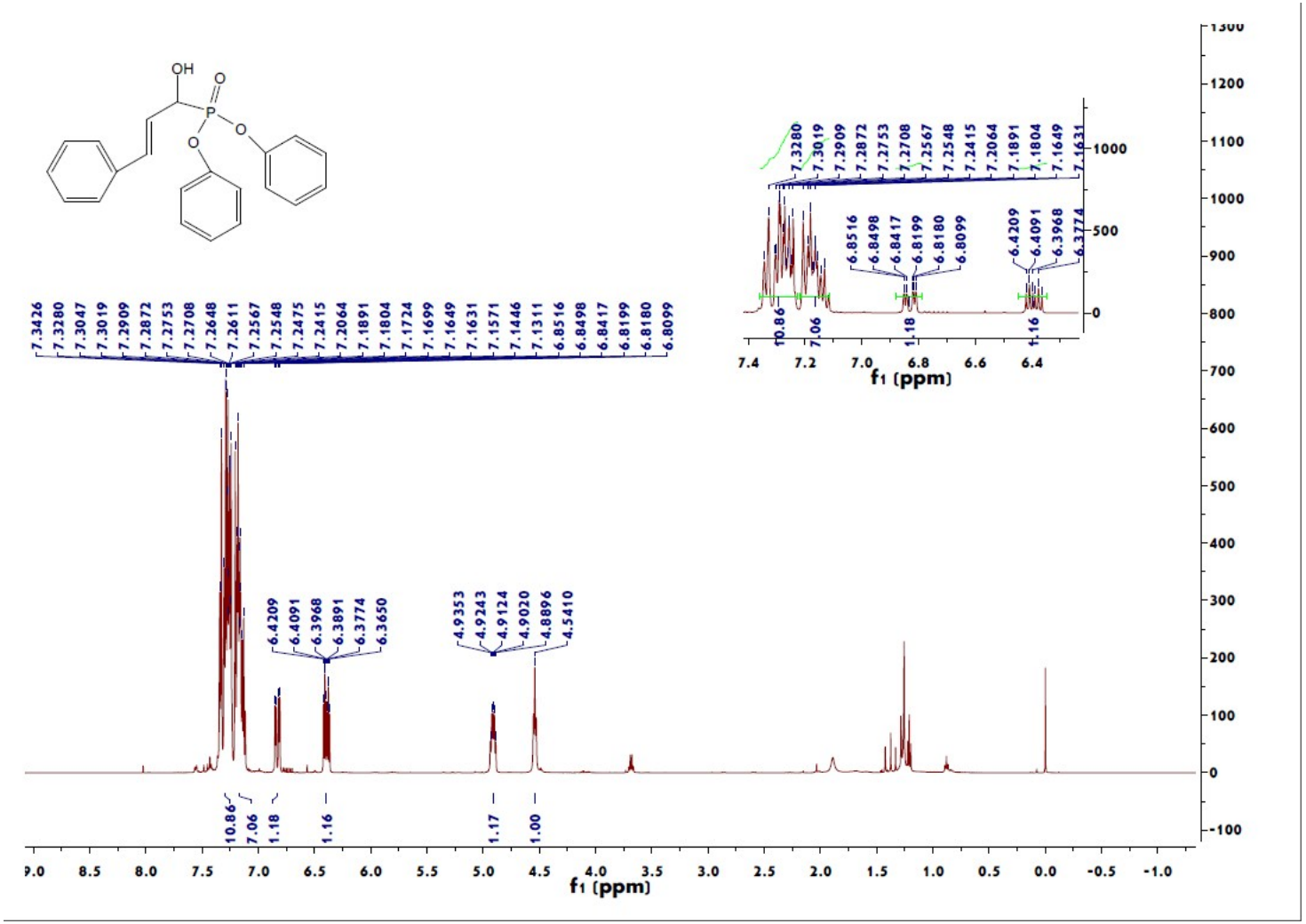

OH
O
P
O
O
8.0299
7.8428
7.6939
7.6786
7.4999
7.4943
7.2538
7.2474
7.2317
7.2169
7.2030
7.1447
7.1309
7.1187
7.1052
7.0844
7.0698
7.0284
7.0145
5.4998
5.4824
3.8920
1.01
3.03
1.04
2.06
4.80
6.01
1.00
0.91
f1 (ppm)

2l

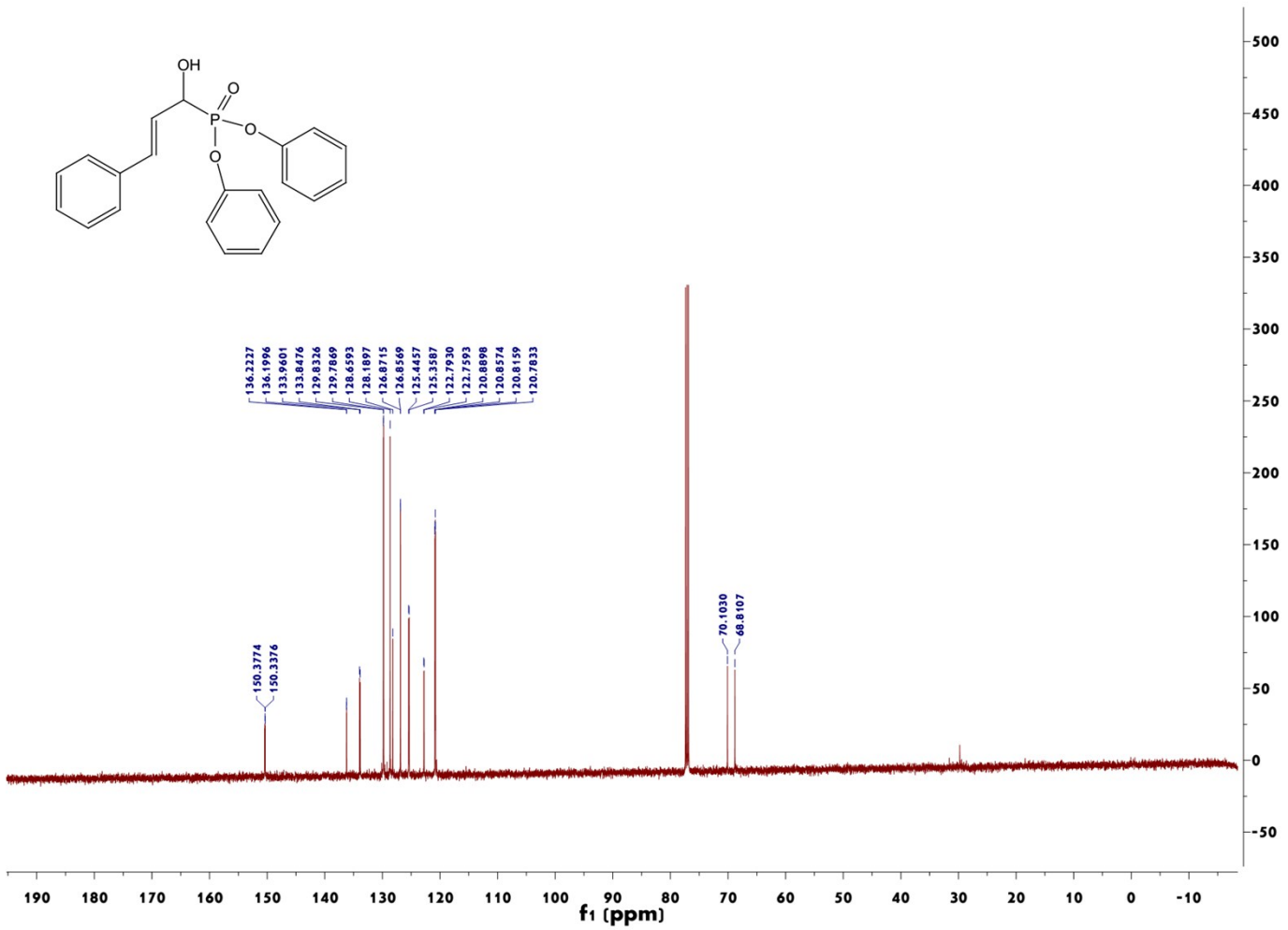

OH
O
P
O
O
O
133.3736
133.1475
132.8326
129.7230
129.6546
128.2899
128.2255
127.7338
126.8329
126.7672
126.4408
126.3356
125.3331
125.2465
124.9198
124.8861
120.6468
120.6157
120.5777
120.5481
150.3187
150.2572
71.5970
70.3163
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

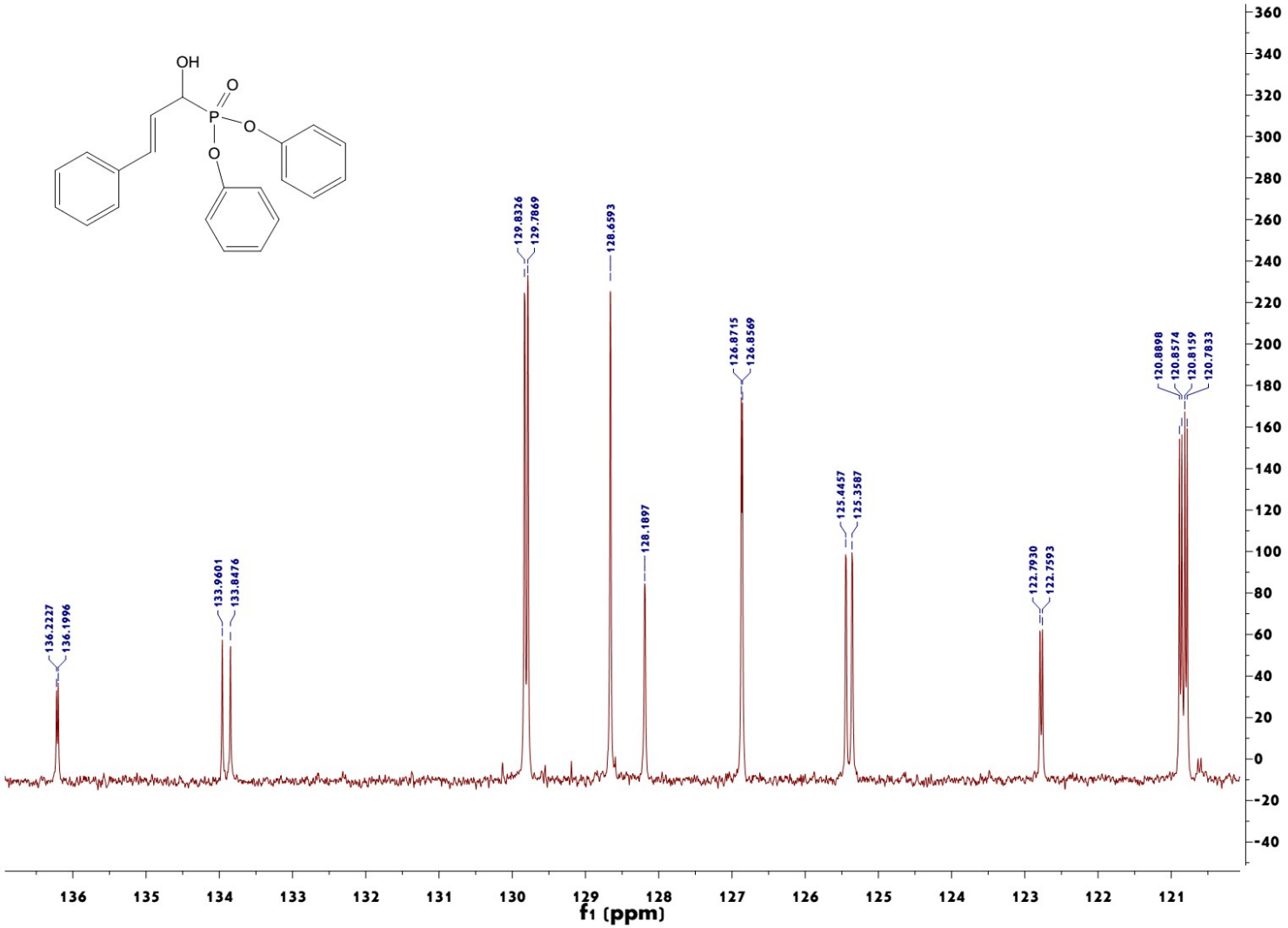

OH
O
P
O
O
133.3736
133.1475
132.8326
129.7230
129.6546
128.2899
128.2255
127.7338
126.8329
126.7672
126.4408
126.3356
125.3331
125.2465
124.9198
124.8861
120.6468
120.6157
120.5777
120.5481
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
135
134
133
132
131
130
129
128
127
126
125
124
123
122
121
120
119
f1 (ppm)

2m

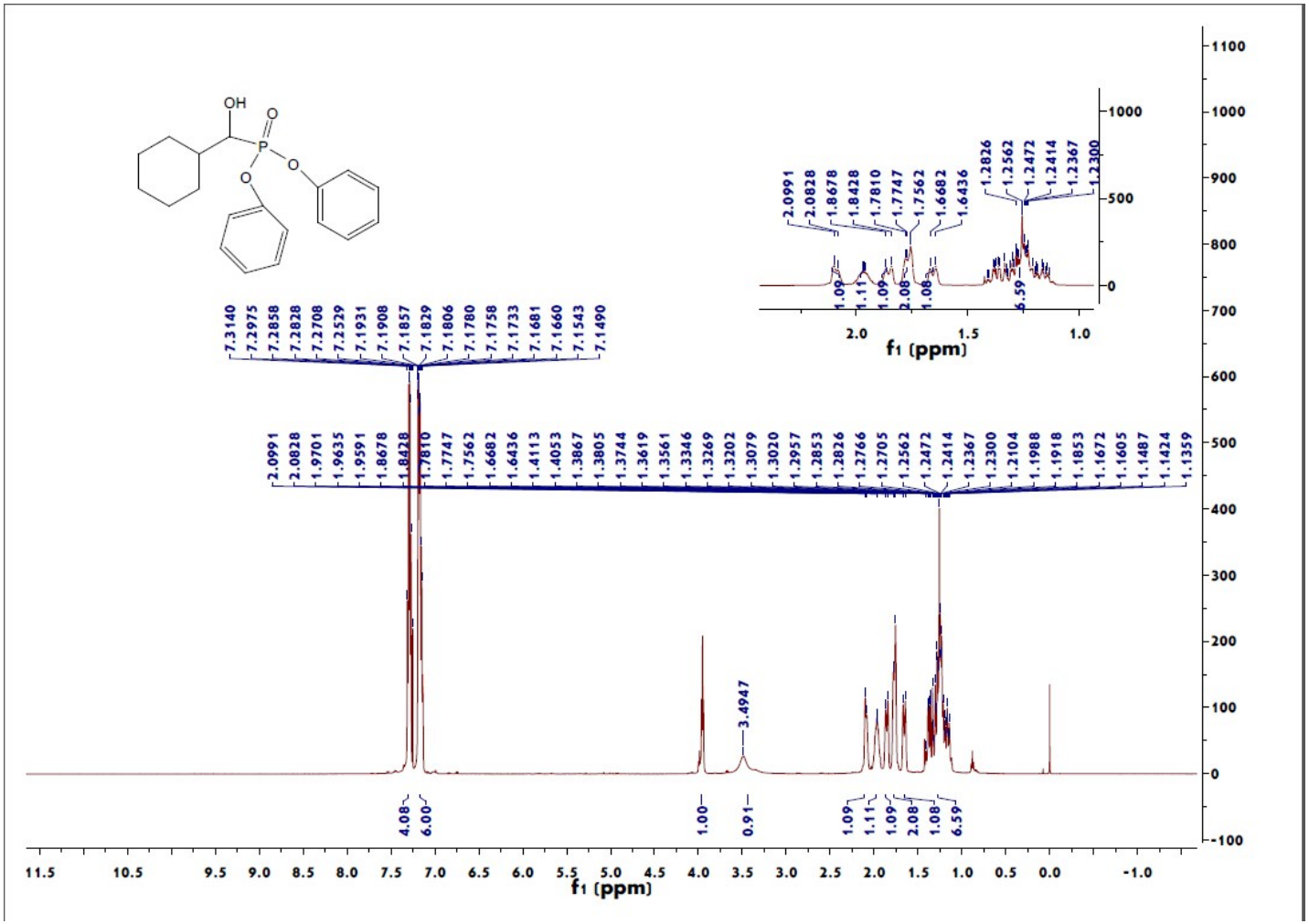

OH
O
P
O
O
7.3426
7.3280
7.3047
7.3019
7.2909
7.2872
7.2753
7.2708
7.2648
7.2611
7.2567
7.2548
7.2475
7.2415
7.2064
7.1891
7.1804
7.1724
7.1699
7.1649
7.1631
7.1571
7.1446
7.1311
6.8516
6.8498
6.8417
6.8199
6.8180
6.8099
6.4209
6.4091
6.3968
6.3891
6.3774
6.3650
4.9353
4.9243
4.9124
4.9020
4.8896
4.5410
10.86
7.06
1.18
1.16
1.17
1.00
f1 (ppm)

2m

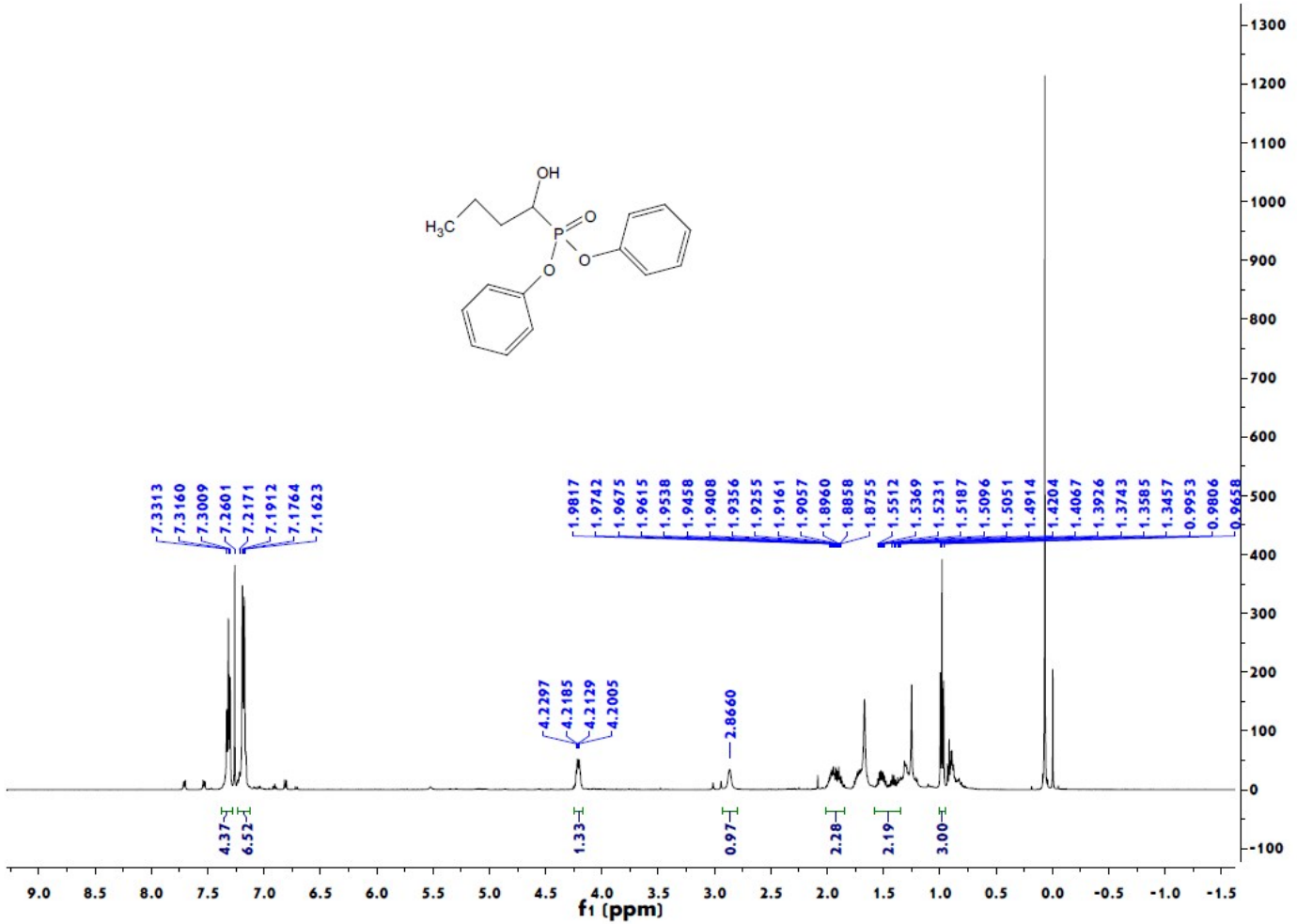

OH
O
P
O
O
136.2227
136.1996
133.9601
133.8476
129.8326
129.7869
128.6593
128.1897
126.8715
126.8569
125.4457
125.3587
122.7930
122.7593
120.8898
120.8574
120.8159
120.7833
150.3774
150.3376
70.1030
68.8107
500
450
400
350
300
250
200
150
100
50
0
-50
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


OH
O
P
O
O
136.2227
136.1996
133.9601
133.8476
129.8326
129.7869
128.6593
128.1897
126.8715
126.8569
125.4457
125.3587
122.7930
122.7593
120.8898
120.8574
120.8159
120.7833
f1 (ppm)

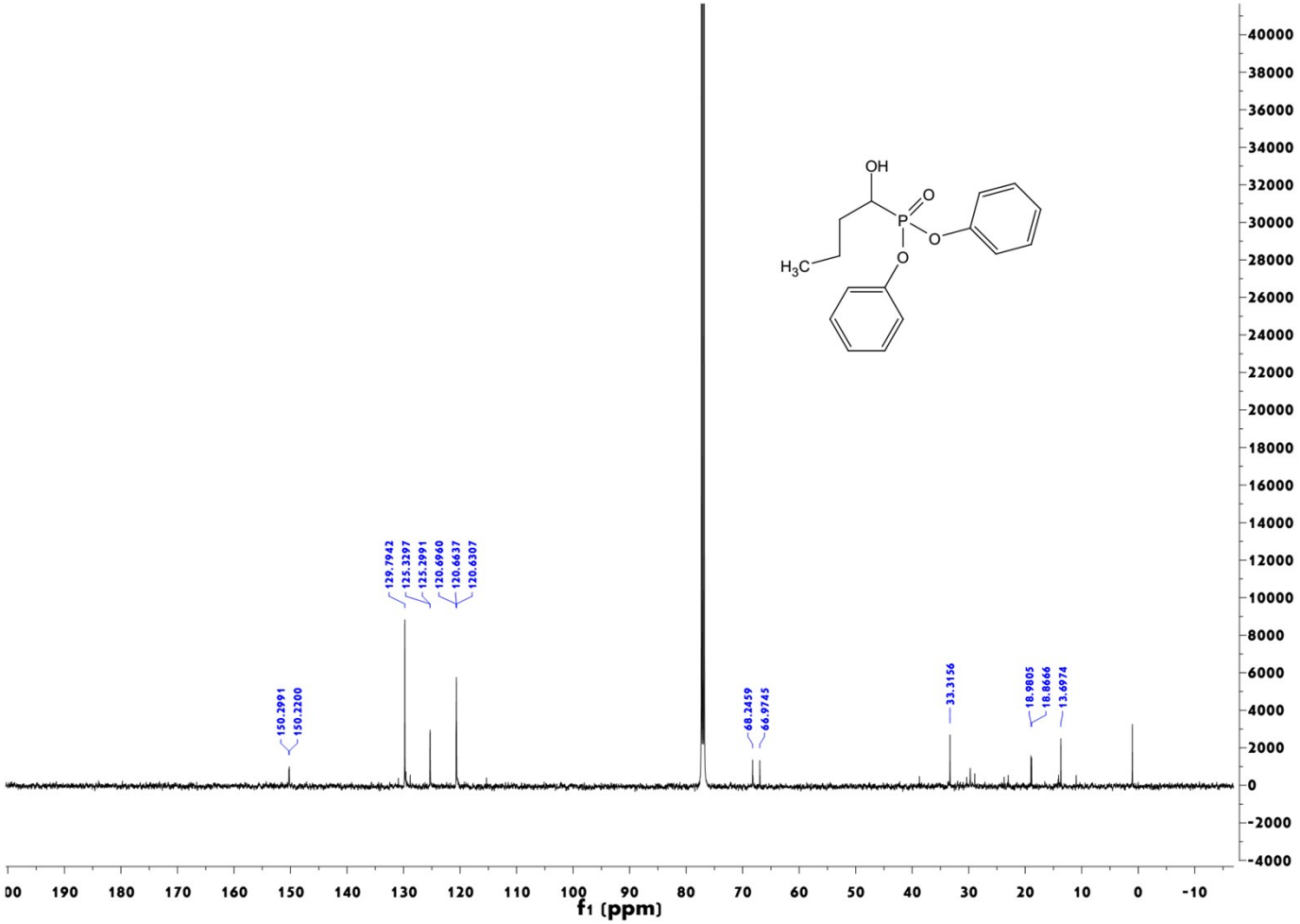
2n


OH
O
P
O
O
7.3140
7.2975
7.2858
7.2828
7.2708
7.2529
7.1931
7.1908
7.1857
7.1829
7.1806
7.1780
7.1758
7.1733
7.1681
7.1660
7.1543
7.1490
2.0991
2.0828
1.9701
1.9635
1.9591
1.8678
1.8428
1.7810
1.7747
1.7562
1.6682
1.6436
1.4113
1.4053
1.3867
1.3805
1.3744
1.3619
1.3561
1.3346
1.3269
1.3202
1.3079
1.3020
1.2957
1.2853
1.2826
1.2766
1.2705
1.2562
1.2472
1.2414
1.2367
1.2300
1.2104
1.1988
1.1918
1.1853
1.1672
1.1605
1.1487
1.1424
1.1359
3.4947
4.08
6.00
1.00
0.91
1.09
1.11
1.09
2.08
1.08
6.59
f1 (ppm)
2.0991
2.0828
1.8678
1.8428
1.7810
1.7747
1.7562
1.6682
1.6436
1.2826
1.2562
1.2472
1.2414
1.2367
1.2300
1.09
1.11
1.09
2.08
1.08
6.59
f1 (ppm)

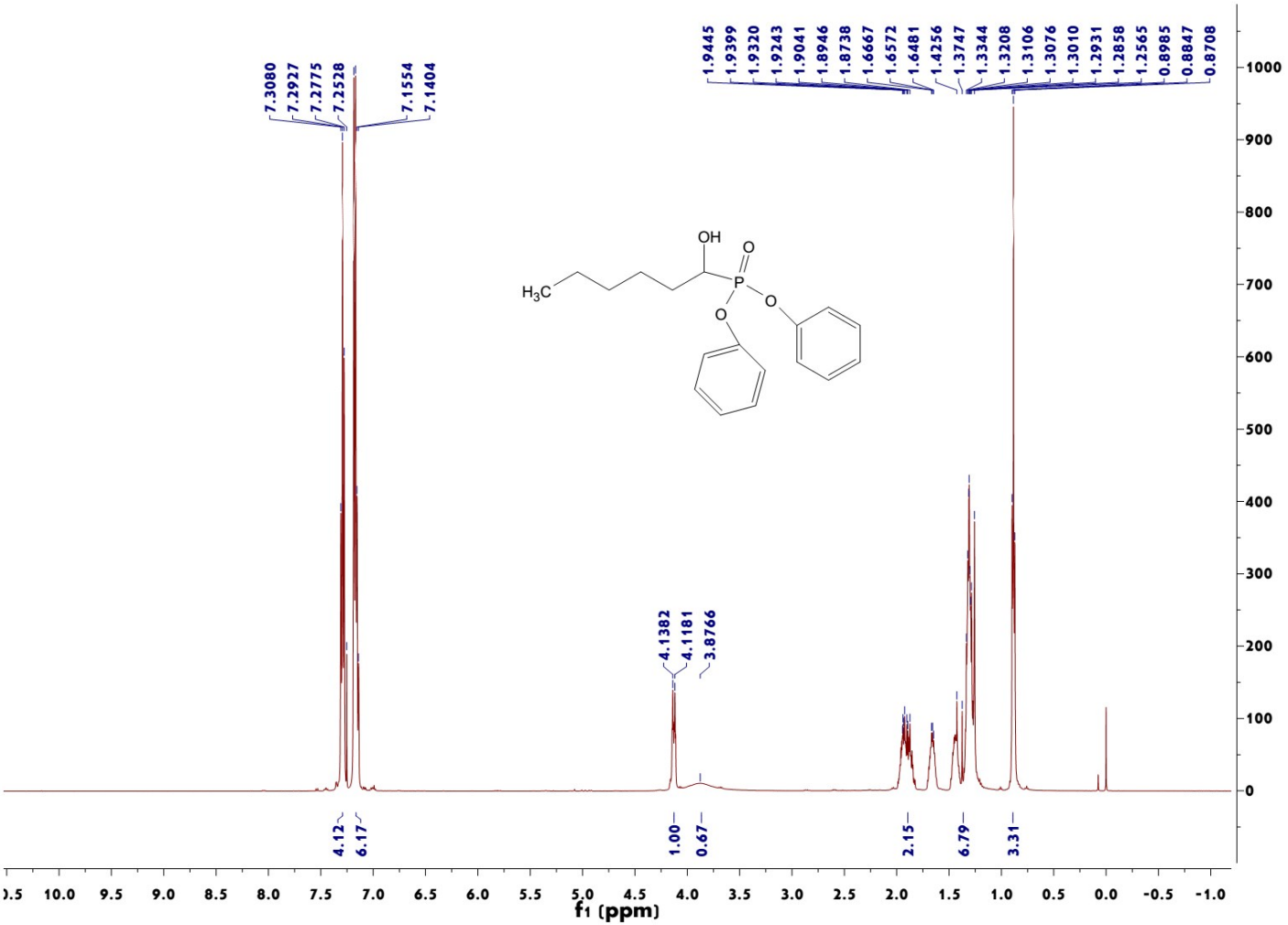
2o


OH
H3C
P
O
O
O
7.3313
7.3160
7.3009
7.2601
7.2171
7.1912
7.1764
7.1623
1.9817
1.9742
1.9675
1.9615
1.9538
1.9458
1.9408
1.9356
1.9255
1.9161
1.9057
1.8960
1.8858
1.8755
1.5512
1.5369
1.5231
1.5187
1.5096
1.5051
1.4914
1.4204
1.4067
1.3926
1.3743
1.3585
1.3457
0.9953
0.9806
0.9658
4.2297
4.2185
4.2129
4.2005
2.8660
4.37
6.52
1.33
0.97
2.28
2.19
3.00
f1 (ppm)

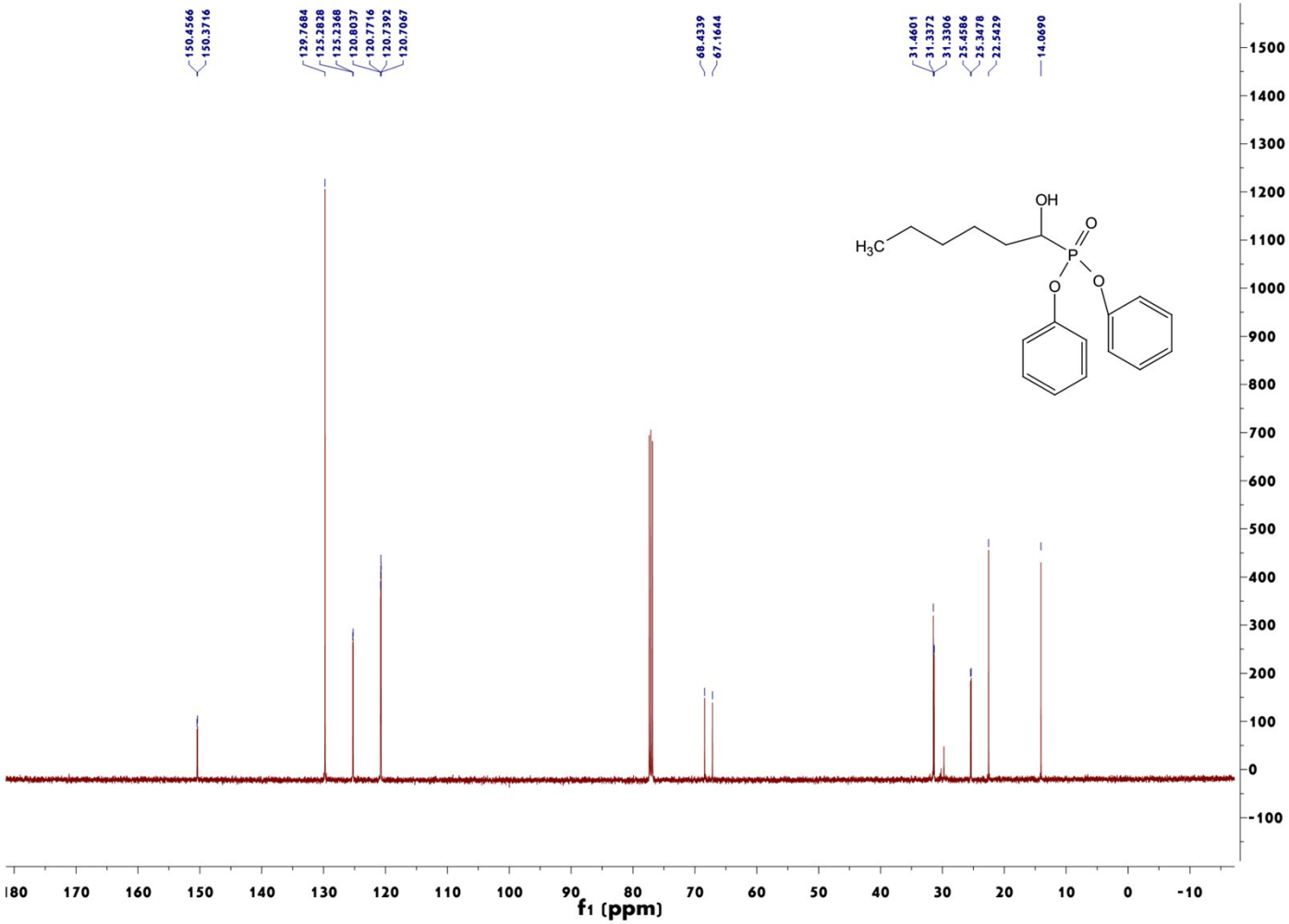
2o


OH
O
P
O
O
H3C
150.2991
150.2200
129.7942
125.3297
125.2991
120.6960
120.6637
120.6307
68.2459
66.9745
33.3156
18.9805
18.8666
13.6974
f1 (ppm)

2p


7.3080
7.2927
7.2775
7.2528
7.1554
7.1404
1.9445
1.9399
1.9320
1.9243
1.9041
1.8946
1.8738
1.6667
1.6572
1.6481
1.4256
1.3747
1.3344
1.3208
1.3106
1.3076
1.3010
1.2931
1.2858
1.2565
0.8985
0.8847
0.8708
OH
O
P
O
O
H3C
4.1382
4.1181
3.8766
4.12
6.17
1.00
0.67
2.15
6.79
3.31
f1 (ppm)

2p


150.4566
150.3716
129.7684
125.2828
125.2368
120.8037
120.7716
120.7392
120.7067
68.4339
67.1644
31.4601
31.3372
31.3306
25.4586
25.3478
22.5429
14.0690
OH
O
P
O
O
H3C
f1 (ppm)