

An Efficient Synthesis of Phosphinyl Oxide Pyrrolidines and Their Application to Asymmetric Direct Aldol Reactions

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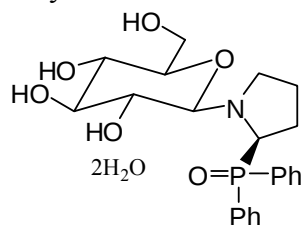
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Email: xuewei@ntu.edu.sg

Supporting Information

1. X-ray structure of compound 3a	S3
2. HPLC data	S5
3. NMR data (^1H , ^{13}C and ^{31}P)	S20

Table 1. Crystal data and structure refinement for **3a.2H₂O**



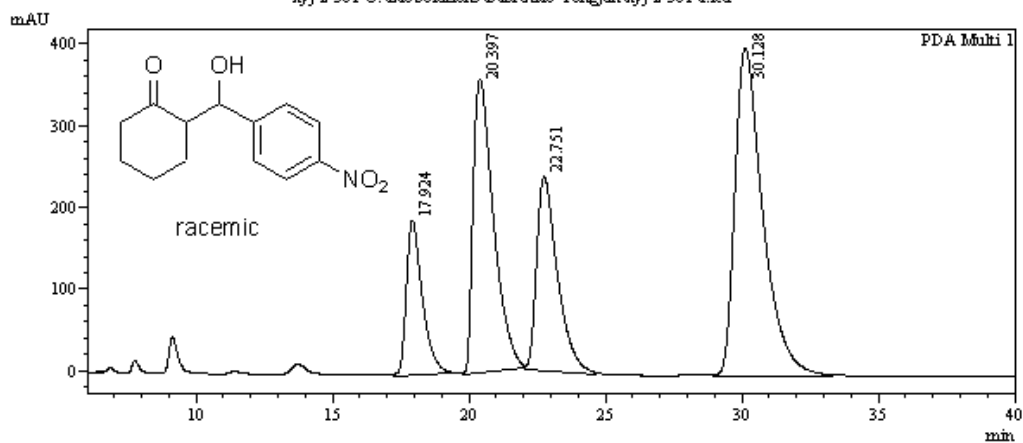
Empirical formula	C ₂₂ H ₃₂ NO ₈ P
Formula weight	469.46
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal description	Prism
Unit cell dimensions	a = 8.0227(3) Å α = 90° b = 8.8488(4) Å β = 90° c = 31.8469(13) Å γ = 90°
Volume	2260.85(16) Å ³
Space group	P2(1)2(1)2(1)
Z	4
Density (calculated)	1.739 Mg/m ³
Absorption coefficient	0.170 mm ⁻¹
F (000)	1000
Crystal size	0.30 x 0.25 x 0.25 mm ³
Theta range for data collection	2.39 to 30.42
Index ranges	-11 ≤ h ≤ 11, -12 ≤ k ≤ 45, -45 ≤ l ≤ 45
Reflections collected	6889
Absorption correction type	Multi scans
Max. and min. transmission	0.9507 and 0.9587
Flack parameter	0.00(9)
Friedel pairs used	2937

The restraint is 'DFIX 2.5 0.02 H3A C2', which restraints the torsion angle of H(3A)-O(3)-C(3)-C(2) was used in the refinement

hexane 90%
ipa 10%
AD-H 1mL/min

Chromatogram

xyj-2-361 C:\Lab Solutions\Data\Gao Yongjun\xyj-2-361-a.lcd



PeakTable

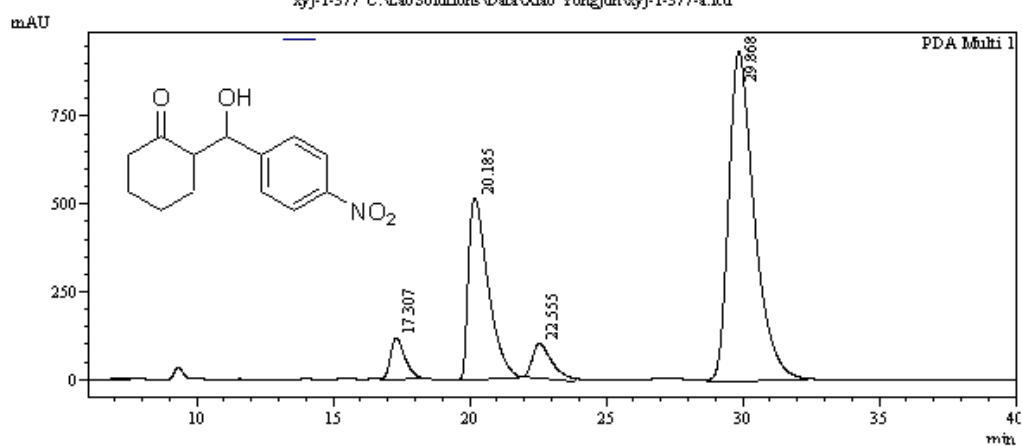
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.924	7737541	189057	11.468	15.956
2	20.397	18569390	358918	27.522	30.292
3	22.751	12638306	236775	18.732	19.983
4	30.128	28524745	400122	42.278	33.769
Total		67469983	1184871	100.000	100.000

XYJ-1-377
hexane 90%
ipa 10%
AD-H 1 mL/min

Chromatogram

xyj-1-377 C:\Lab Solutions\Data\Xiao Yongjun\xyj-1-377-a.lcd



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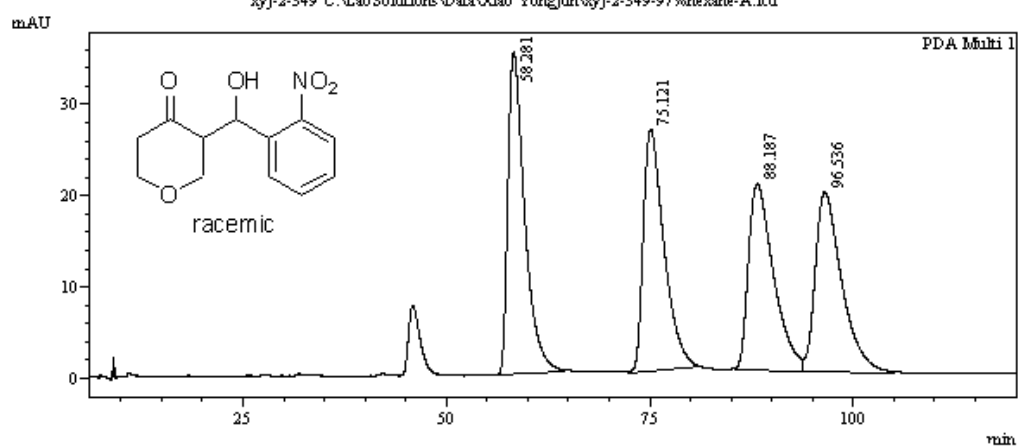
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.307	4202276	115839	4.385	6.960
2	20.185	25829624	514698	26.952	30.925
3	22.555	4866026	97862	5.077	5.880
4	29.868	60938715	935932	63.586	56.235
Total		95836641	1664332	100.000	100.000

hexane 97%
ipa 3%
AD-H 1mL/min

Chromatogram

xyj-2-349 C:\Lab Solutions\Data\Xiao Yongjun\xyj-2-349-97%hexane-A.lcd



PeakTable

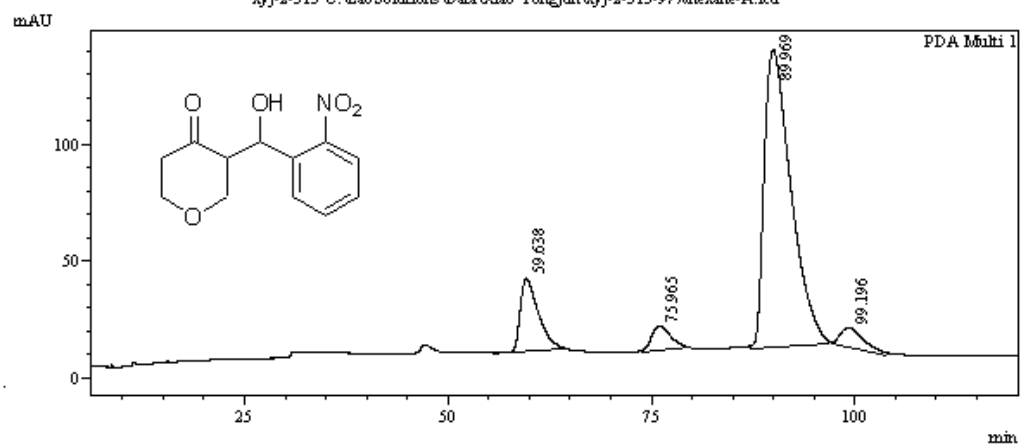
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	58.281	5066175	35268	27.227	34.658
2	75.121	4699763	26480	25.258	26.023
3	88.187	4395332	20382	23.621	20.030
4	96.536	4446103	19629	23.894	19.289
Total		18607374	101759	100.000	100.000

hexane 97%
ipa 3%
AD-H 1 mL/min

Chromatogram

xyj-2-315 C:\Lab Solutions\Data\Xiao Yongjun\xyj-2-315-97%hexane-A.lcd



PeakTable

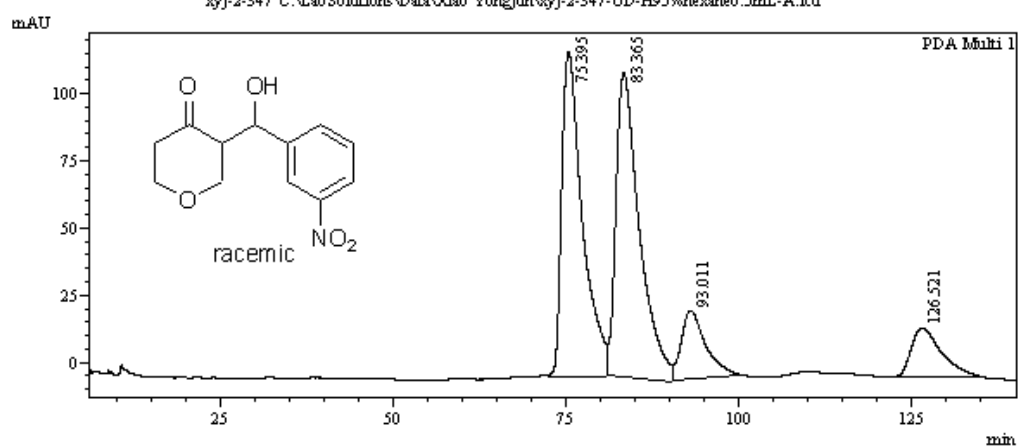
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	59.638	4832370	31102	12.895	17.543
2	75.965	1644115	10422	4.387	5.878
3	89.969	29307921	127440	78.209	71.883
4	99.196	1689404	8323	4.508	4.695
Total		37473810	177287	100.000	100.000

hexane 95%
ipa 5%
OD-H 1.mL/min

Chromatogram

xyj-2-347 C:\Lab Solutions\Data\Xiao Yongjun\xyj-2-347-OD-H95%hexane0.5mL-A.lcd



PeakTable

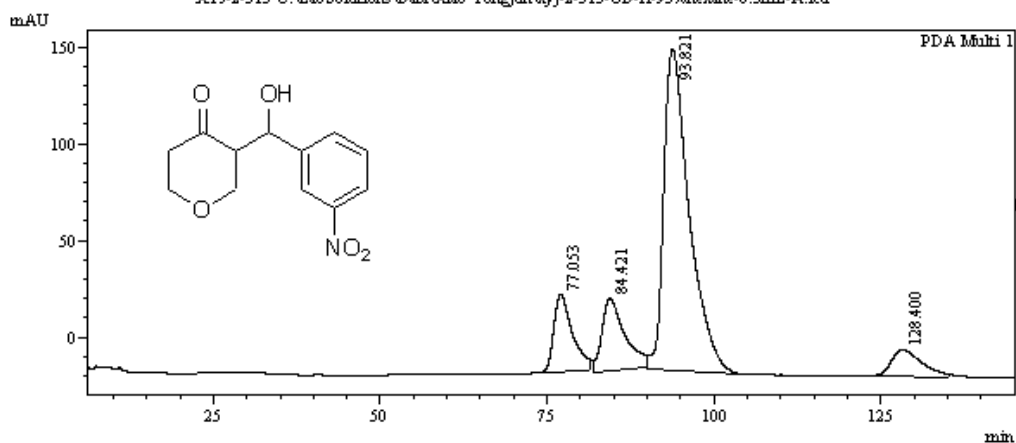
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	75.395	25229998	120906	39.558	43.560
2	83.365	26739348	113054	41.925	40.732
3	93.011	6281785	25394	9.849	9.149
4	126.521	5528579	18206	8.668	6.559
Total		63779711	277559	100.000	100.000

hexane 95%
ipa 5%
OD-H 1 mL/min

Chromatogram

XYJ-2-313 C:\Lab Solutions\Data\Gao Yongjun\xyj-2-313-OD-H-95%hexane-0.5mL-A.lcd



PeakTable

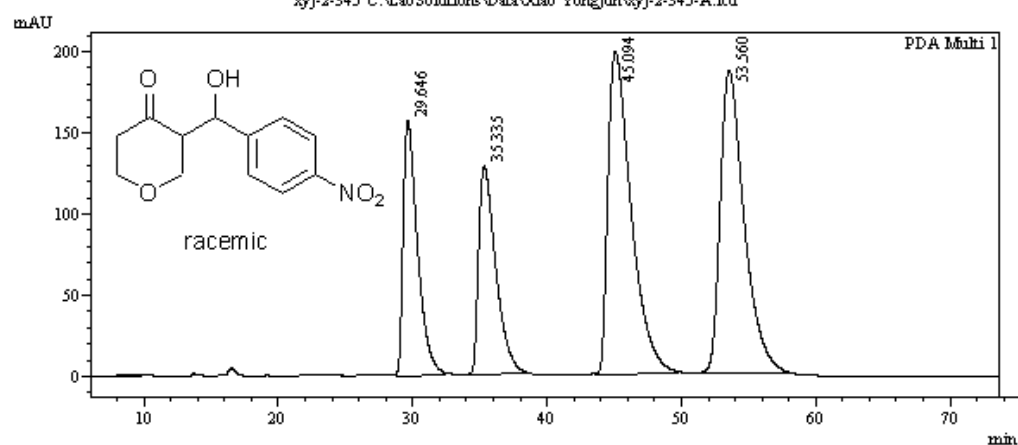
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	77.053	7954504	40031	12.136	15.567
2	84.421	9012064	37561	13.750	14.607
3	93.821	44238072	165801	67.494	64.477
4	128.400	4338789	13755	6.620	5.349
Total		65543429	257149	100.000	100.000

hexane 90%
ipa 10%
AD-H 1 mL/min

Chromatogram

xyj-2-345 C:\Lab Solutions\Data\Xiao Yongjun\xyj-2-345-A.lcd



PeakTable

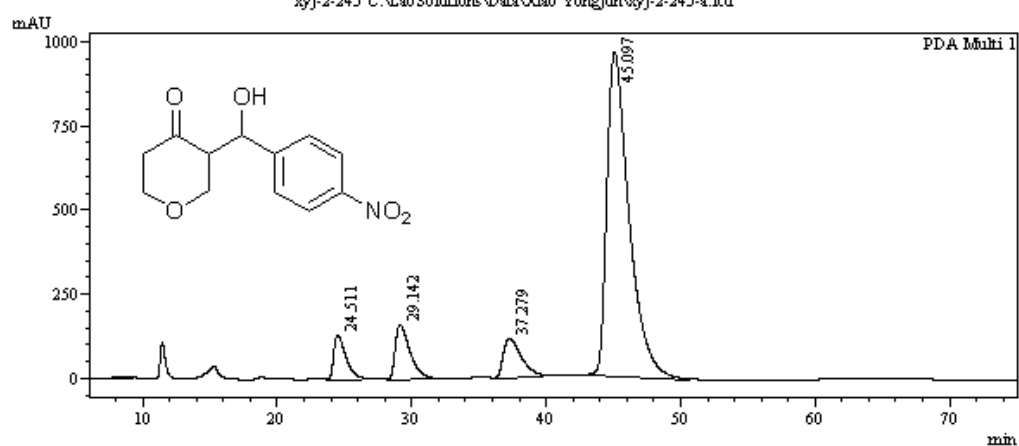
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	29.646	11785794	157190	16.738	23.402
2	35.335	11525267	128560	16.368	19.140
3	45.094	23865462	198854	33.893	29.605
4	53.560	23237511	187088	33.001	27.853
Total		70414034	671692	100.000	100.000

hexane 90%
ipa 10%
AD-H 1 mL/min

Chromatogram

xyj-2-245 C:\Lab Solutions\Data\Xiao Yongjun\xyj-2-245-a.lcd



PeakTable

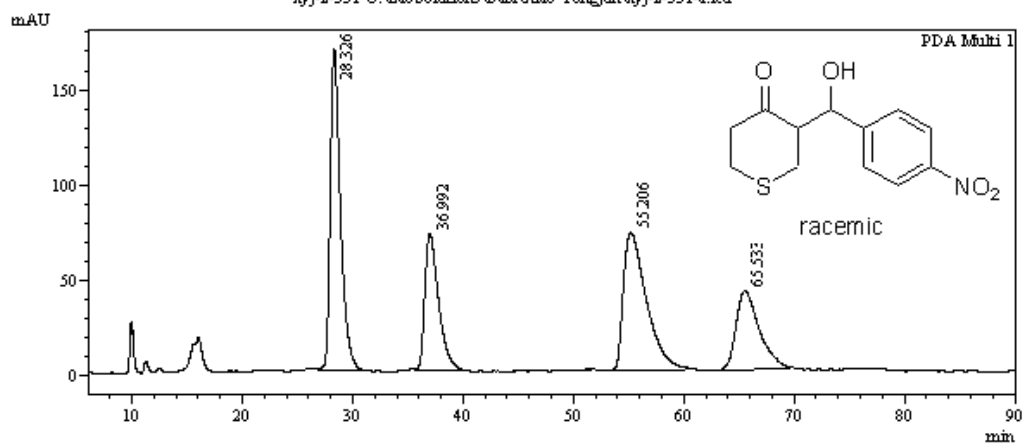
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	24.511	8418593	133872	5.902	9.712
2	29.142	12356869	162806	8.663	11.811
3	37.279	11270747	118362	7.902	8.586
4	45.097	110588972	963439	77.533	69.891
Total		142635181	1378478	100.000	100.000

hexane 90%
ipa 10%
AD-H 1 mL/min

Chromatogram

xyj-2-351 C:\Lab Solutions\Data\Gao Yongjun\xyj-2-351-a.lcd



PeakTable

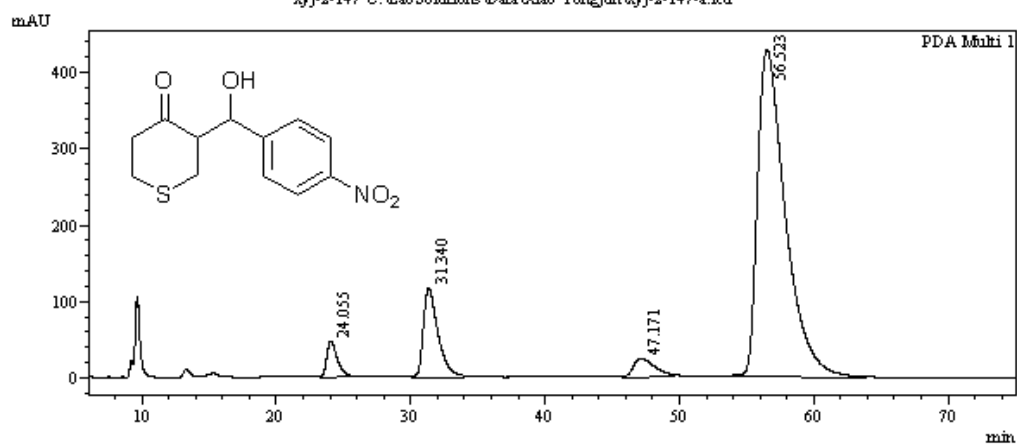
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	28.326	10860445	168770	32.017	47.508
2	36.992	6329571	72431	18.660	20.389
3	55.206	10577034	72569	31.181	20.428
4	65.533	6153966	41477	18.142	11.676
Total		33921016	355247	100.000	100.000

hexane 90%
ipa 10%
AD-H 1 mL/min

Chromatogram

xyj-2-147 C:\Lab Solutions\Data\Gao Yongjun\xyj-2-147-a.lcd



PeakTable

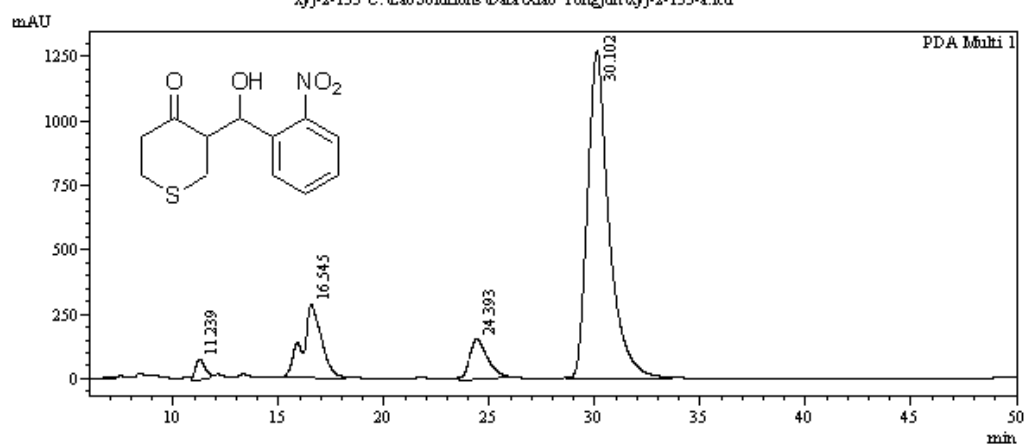
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	24.055	2562305	47085	3.380	7.655
2	31.340	8933647	116661	11.785	18.966
3	47.171	2688480	23625	3.547	3.841
4	56.523	61620051	427728	81.288	69.538
Total		75804483	615098	100.000	100.000

hexane 90%
ipa 10%
AD-H 1.mL/min

Chromatogram

xyj-2-133 C:\Lab Solutions\Data\Xiao Yongjun\xyj-2-133-a.lcd



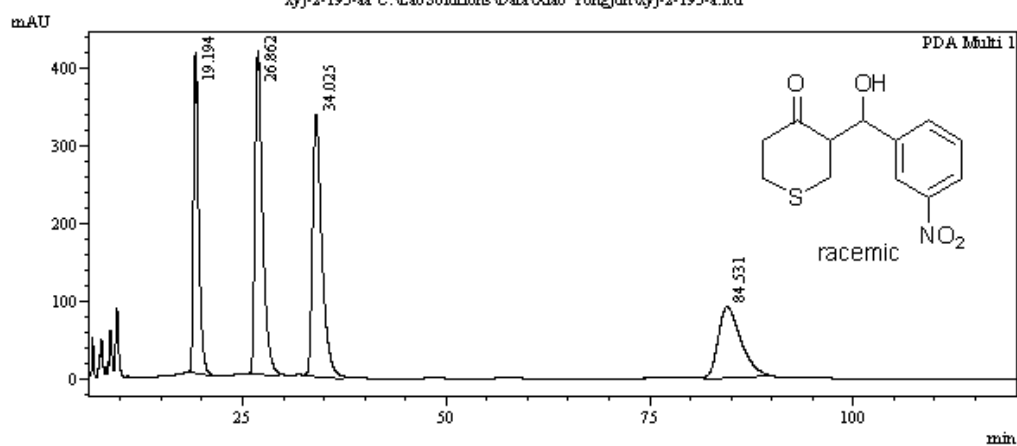
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.239	2408496	77604	2.022	4.345
2	16.545	17992477	284068	15.108	15.906
3	24.393	8926535	152824	7.495	8.557
4	30.102	89767246	1271458	75.375	71.192
Total		119094754	1785954	100.000	100.000

hexane 90%
ipa 10%
AD-H 1 mL/min

Chromatogram
xyj-2-195-aa C:\Lab Solutions\Data\Gao Yongjun\xyj-2-195-a.lcd



PeakTable

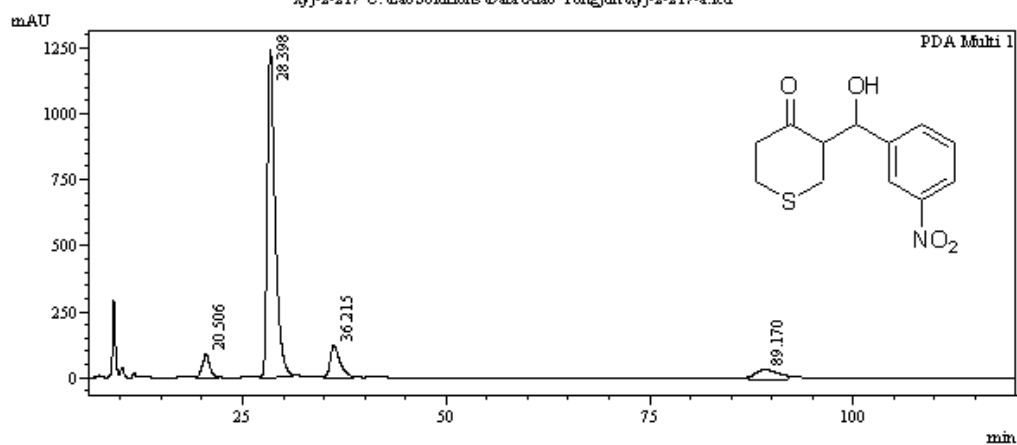
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	19.194	18542468	413013	20.545	32.832
2	26.862	27097821	416208	30.024	33.087
3	34.025	27004611	337167	29.921	26.803
4	84.531	17607626	91550	19.509	7.278
Total		90252525	1257938	100.000	100.000

hexane 90%
ipa 10%
AD-H 1 mL/min

Chromatogram

xyj-2-217 C:\Lab Solutions\Data\Gao Yongjun\xyj-2-217-a.lcd



PeakTable

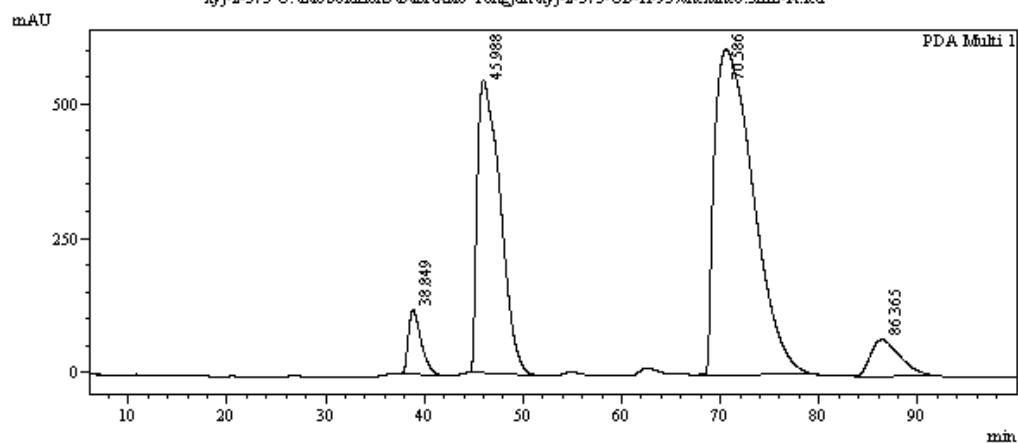
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	20.506	6035462	94821	5.543	6.310
2	28.398	83671143	1242680	76.840	82.696
3	36.215	10846465	124902	9.961	8.312
4	89.170	8336561	40309	7.656	2.682
Total		108889632	1502712	100.000	100.000

hexane 95%
ipa 5%
OD-H 0.5 mL/min

Chromatogram

xyj-2-373 C:\Lab Solutions\Data\Gao Yongjun\xyj-2-373-OD-H-95%hexane0.5mL-A.lcd



PeakTable

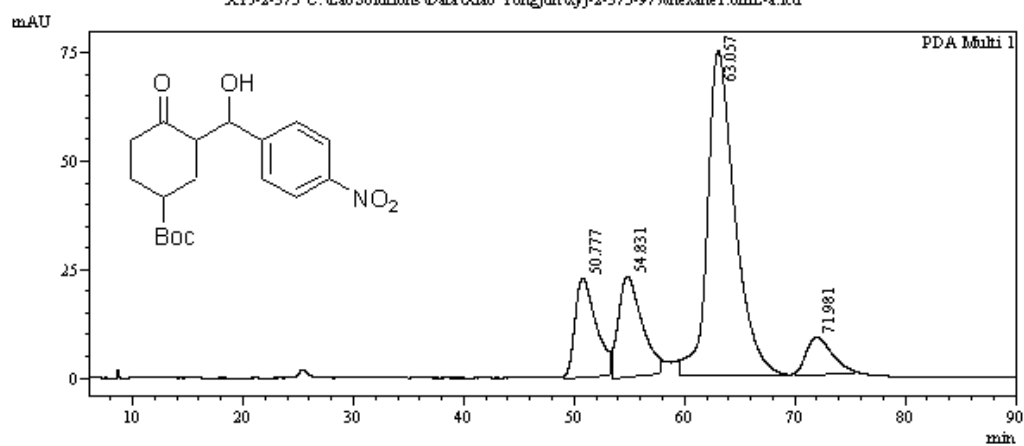
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	38.849	10817836	119943	3.884	8.940
2	45.988	88138596	546710	31.649	40.751
3	70.586	165597838	607171	59.463	45.258
4	86.365	13934627	67766	5.004	5.051
Total		278488897	1341590	100.000	100.000

hexane 97%
ipa 3%
AD-H 1 mL/min

Chromatogram

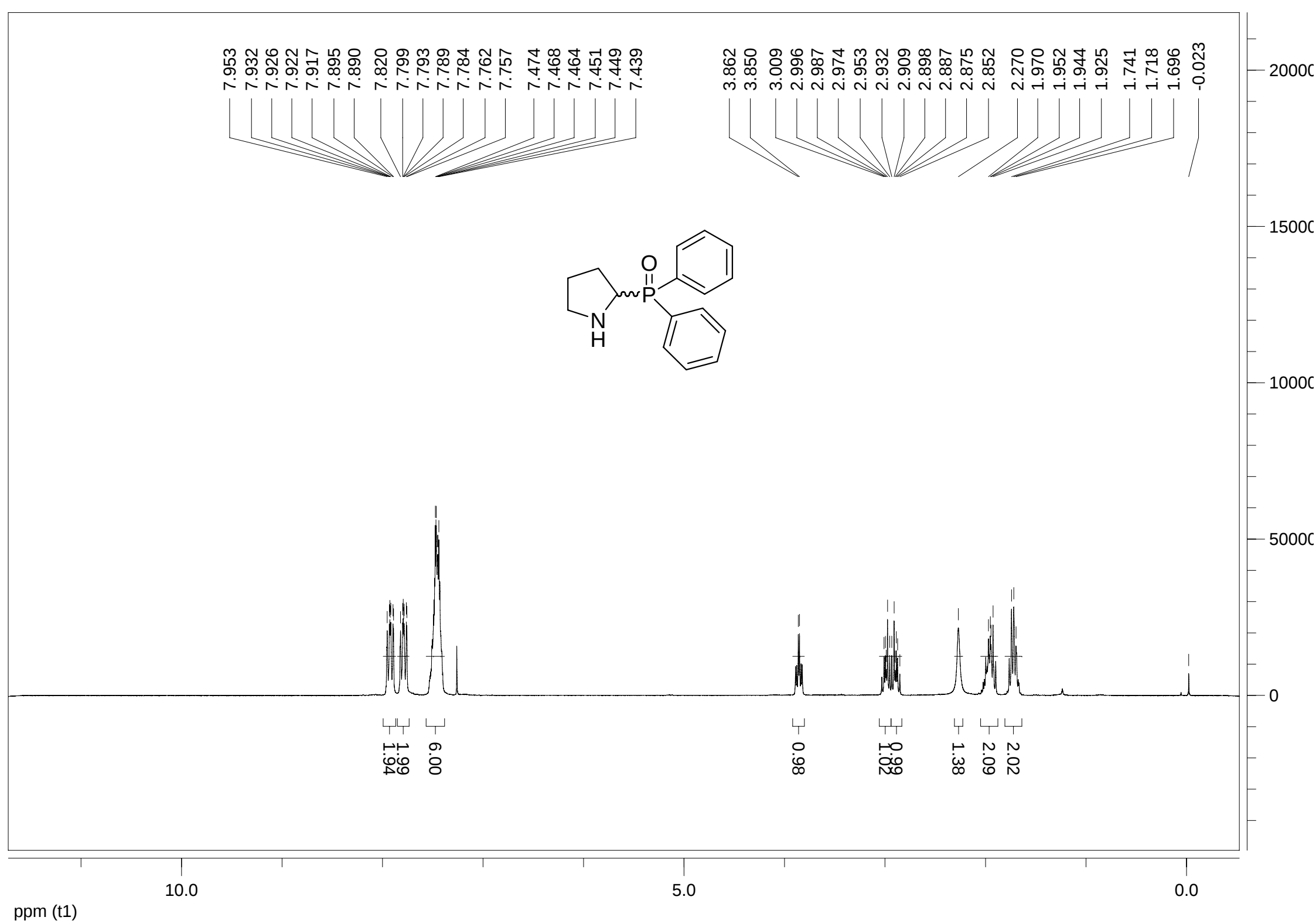
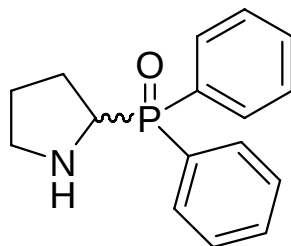
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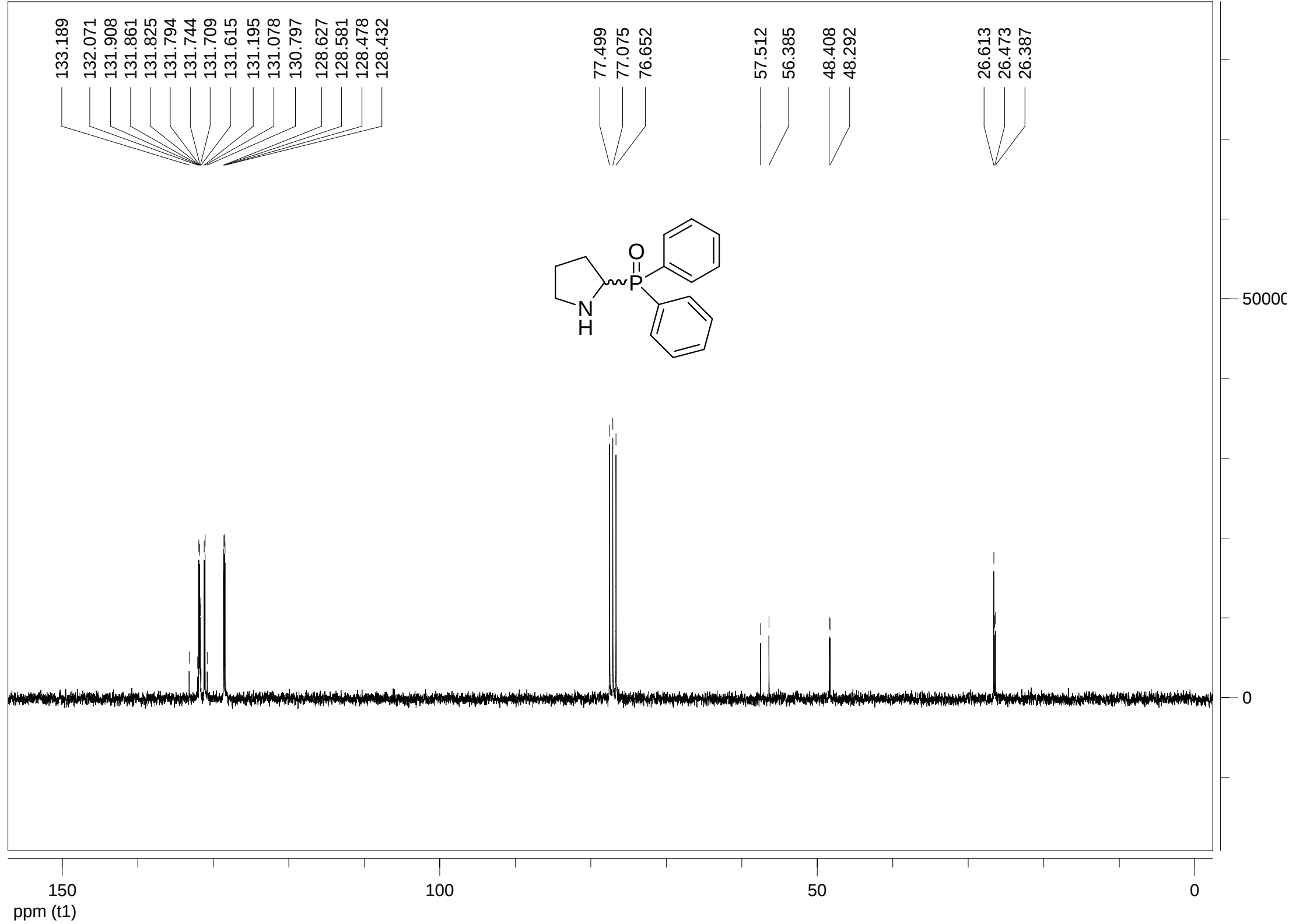


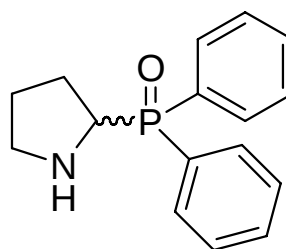
PeakTable

PDA Ch1 254nm 4nm

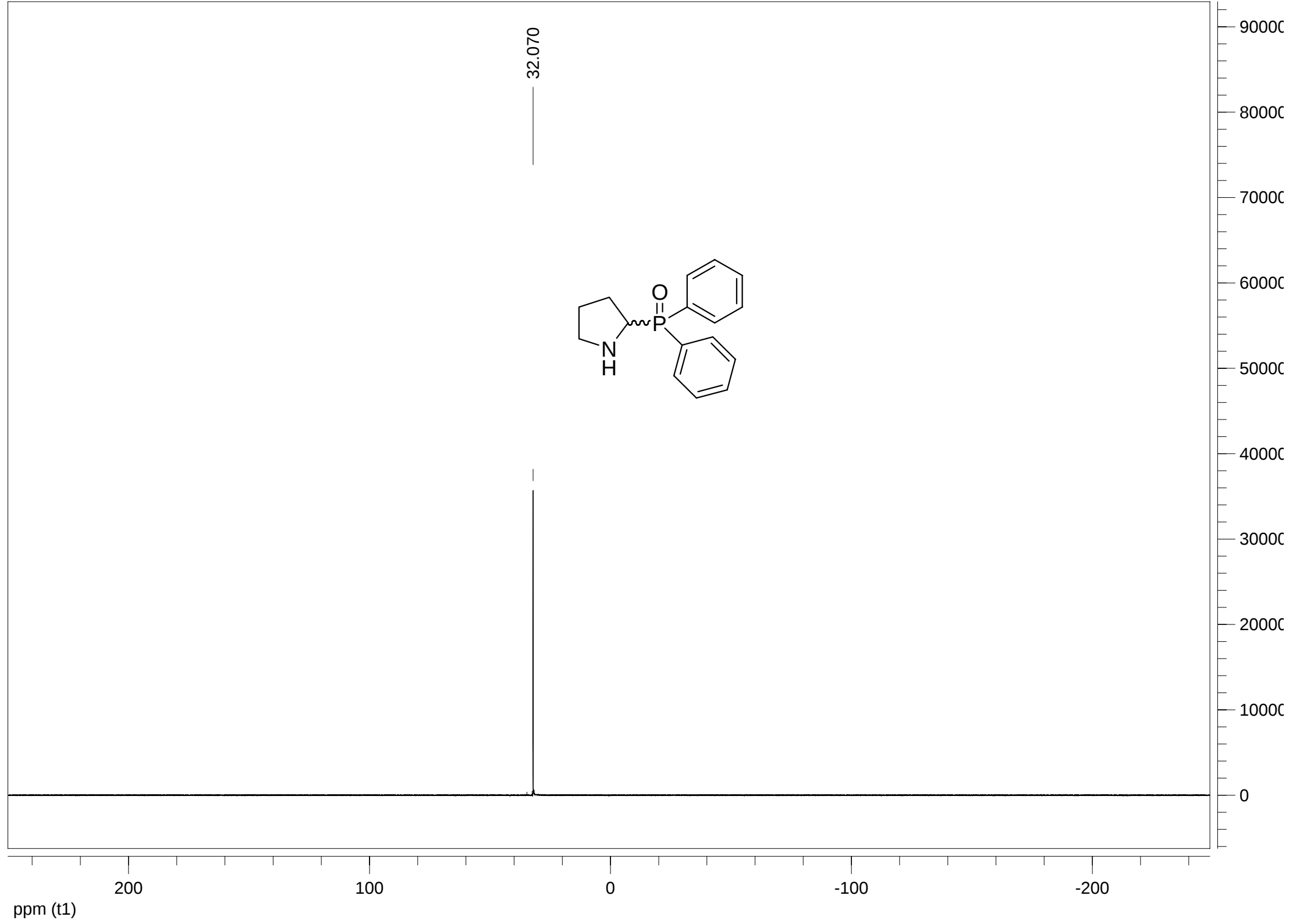
Peak#	Ret. Time	Area	Height	Area %	Height %
1	50.777	3029950	22925	14.506	17.665
2	54.831	3459063	23153	16.560	17.841
3	63.057	12974911	74955	62.118	57.758
4	71.981	1423753	8741	6.816	6.736
Total		20887678	129774	100.000	100.000

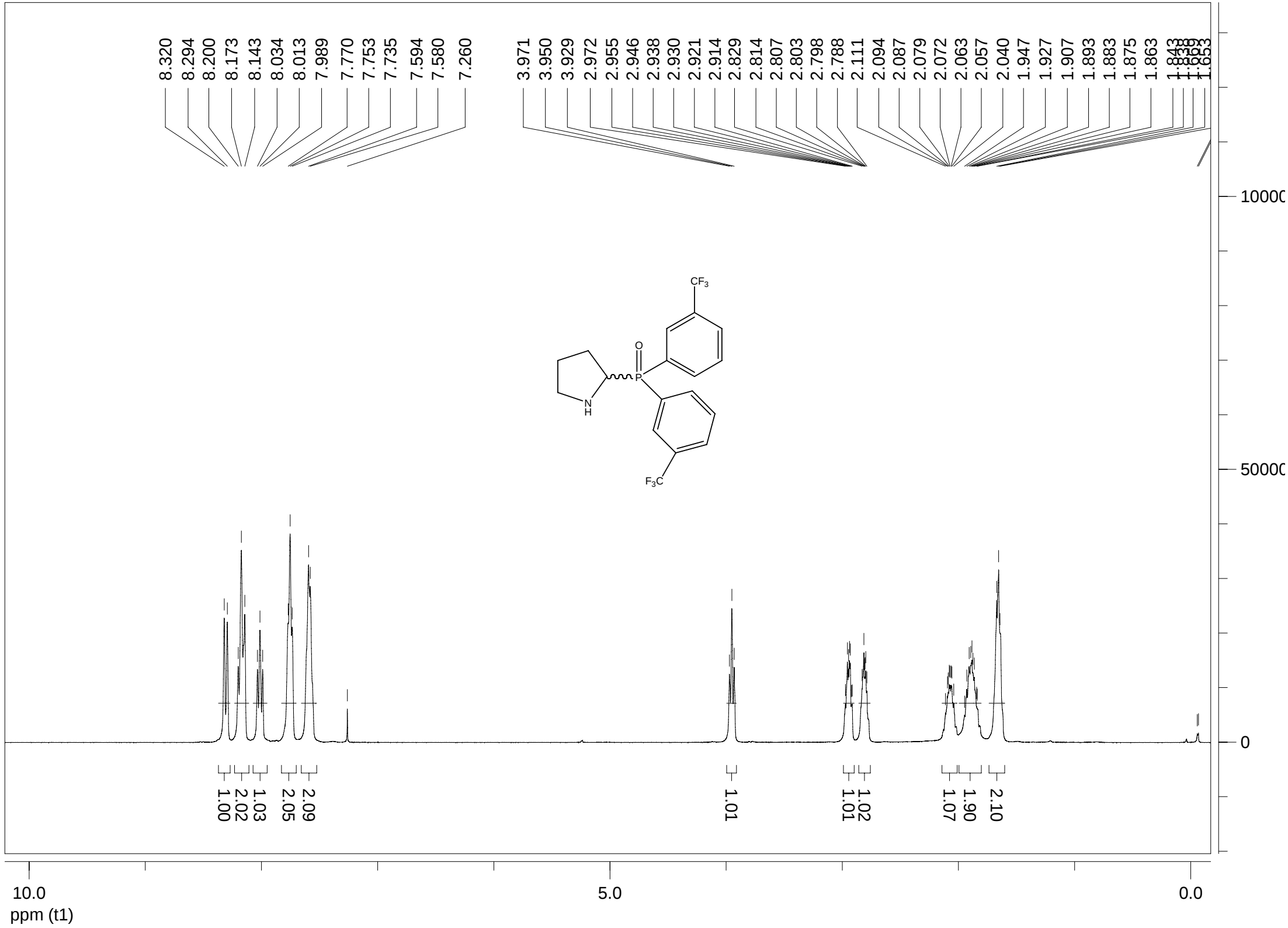


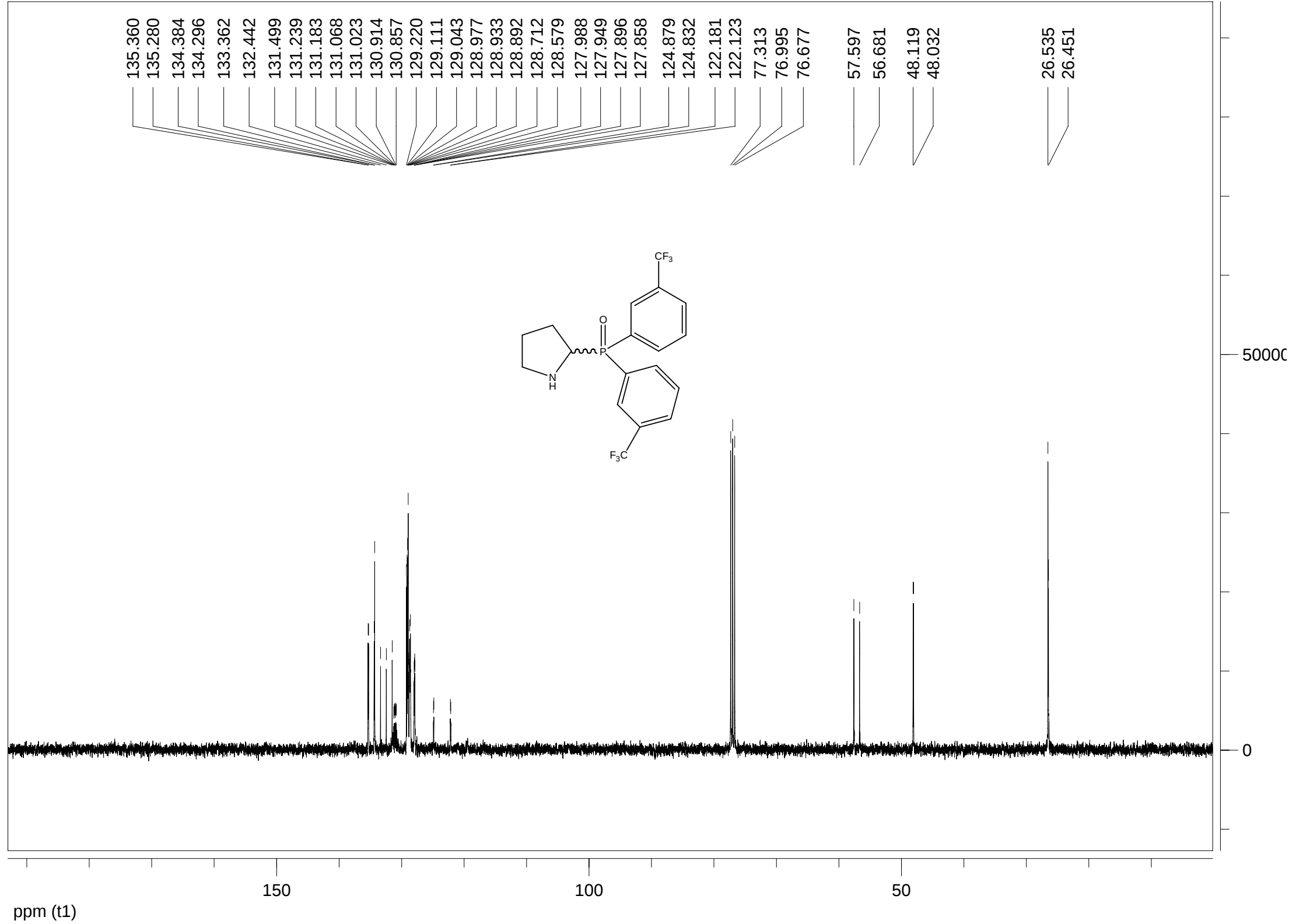


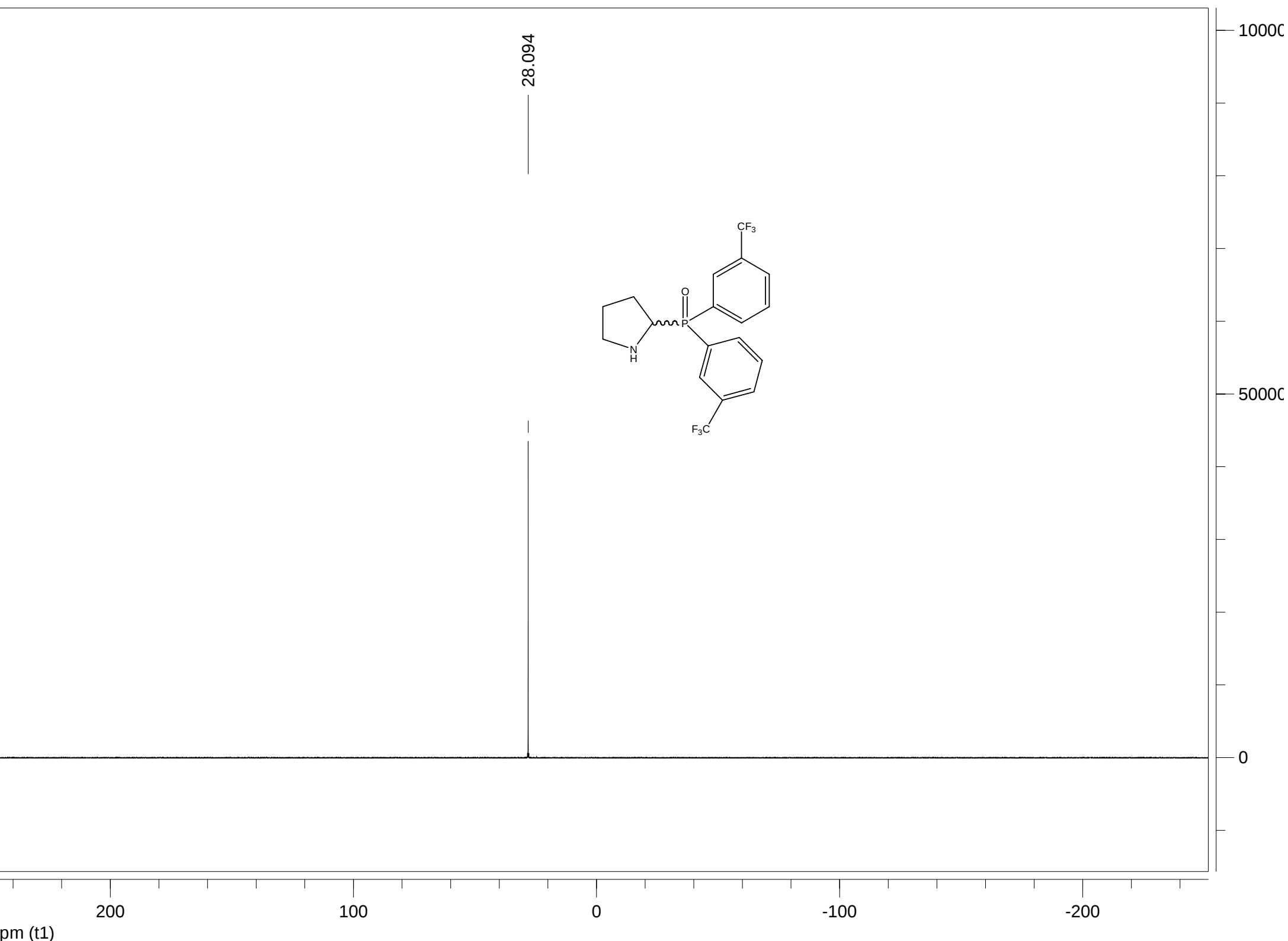


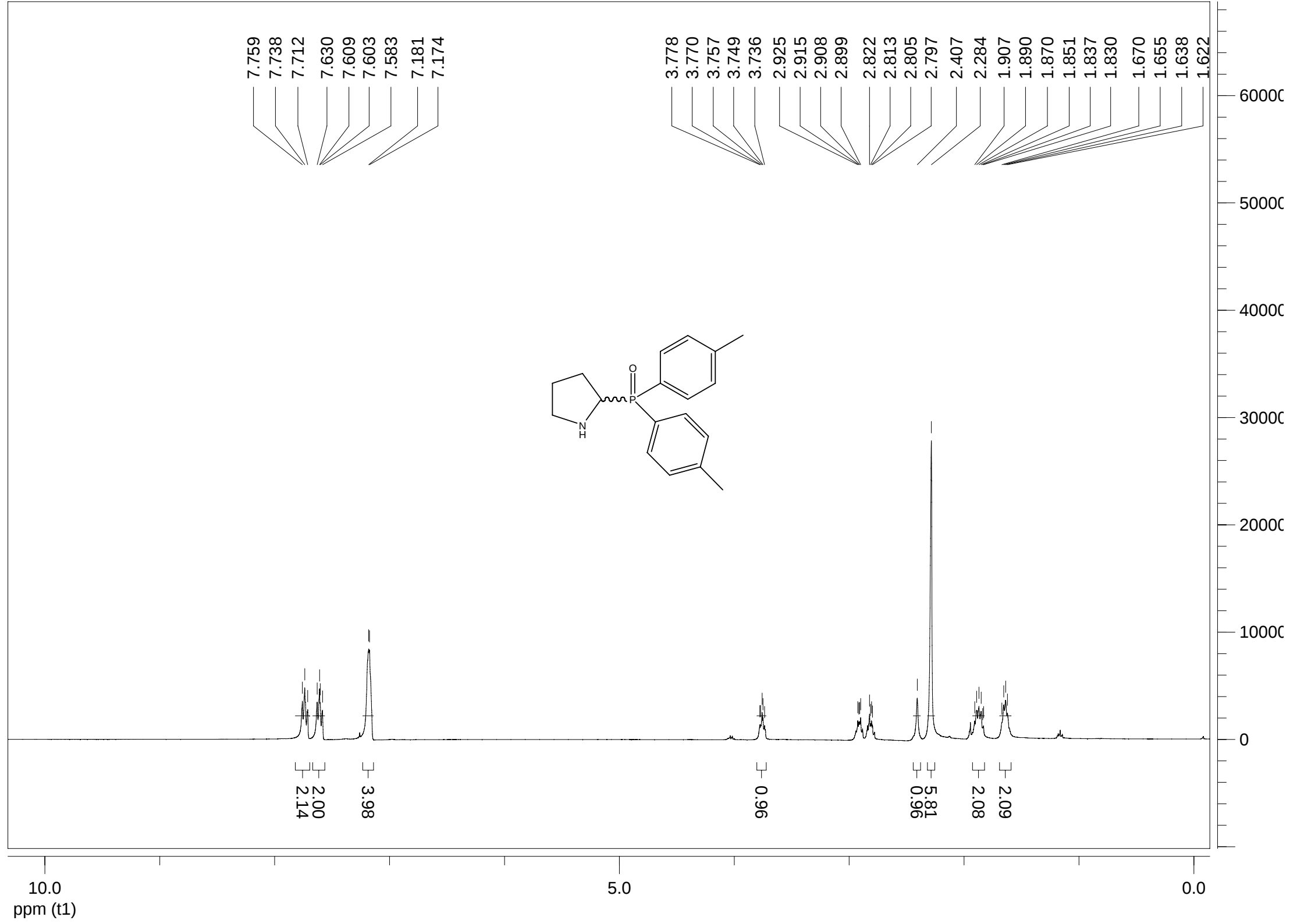
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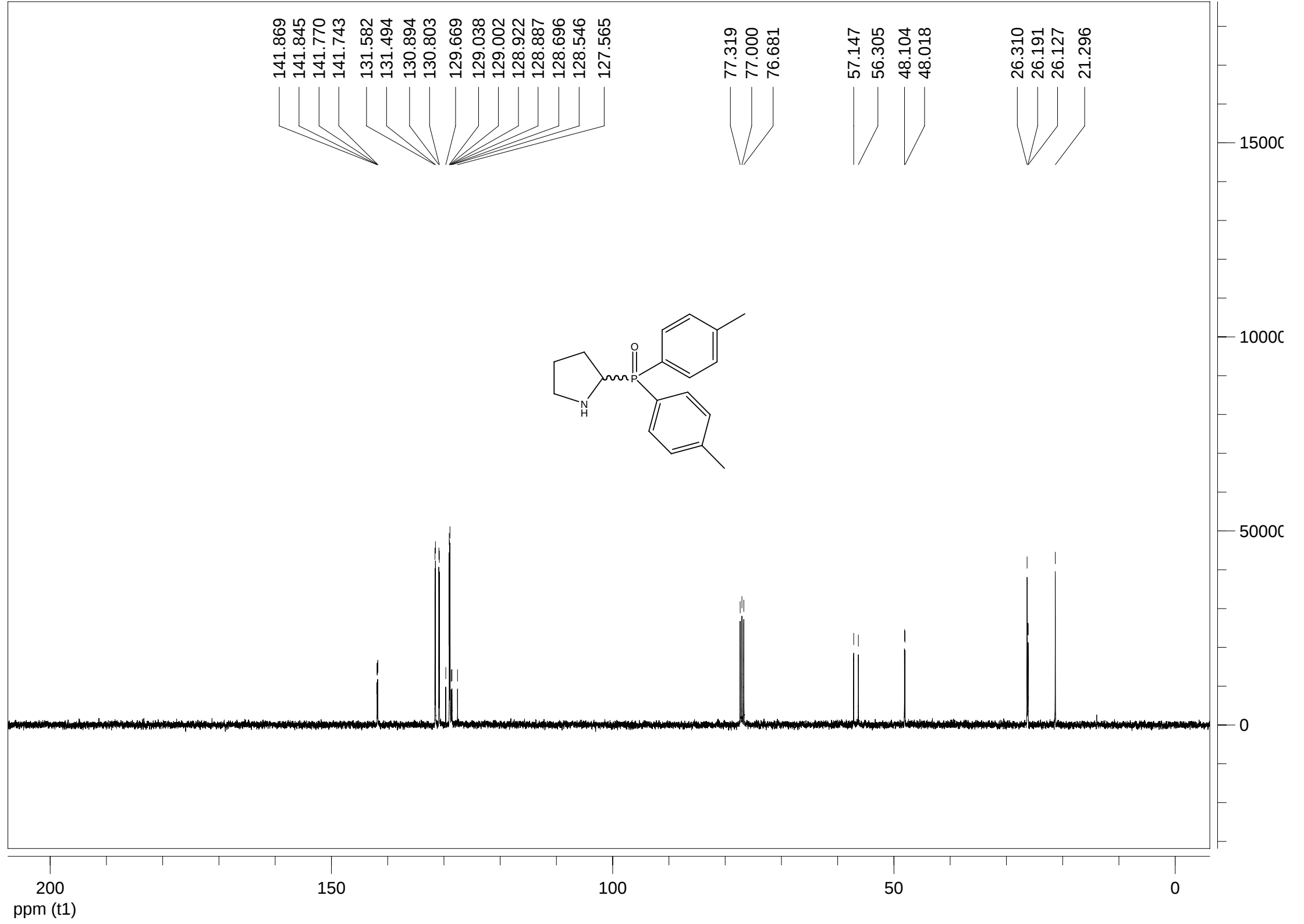


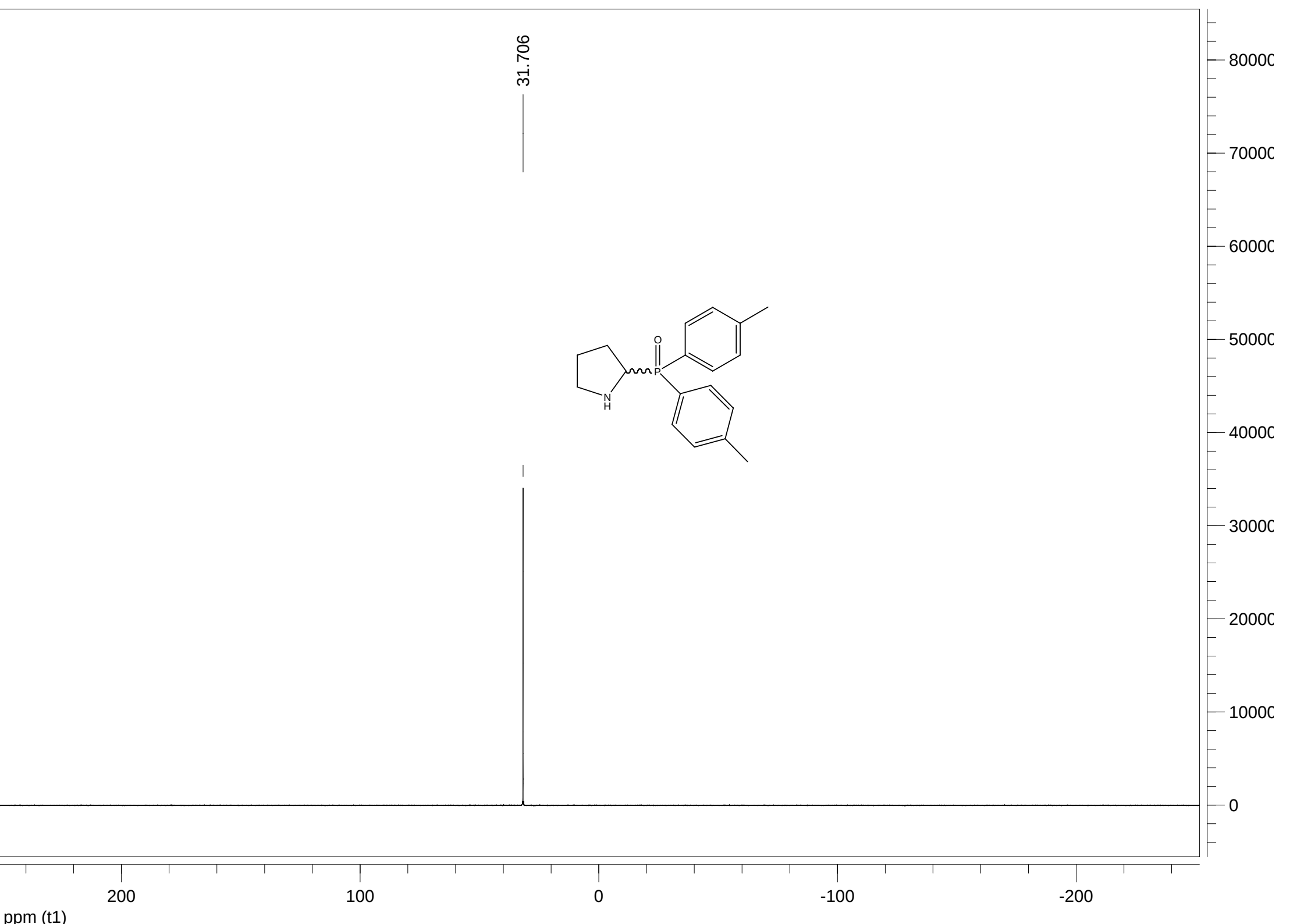


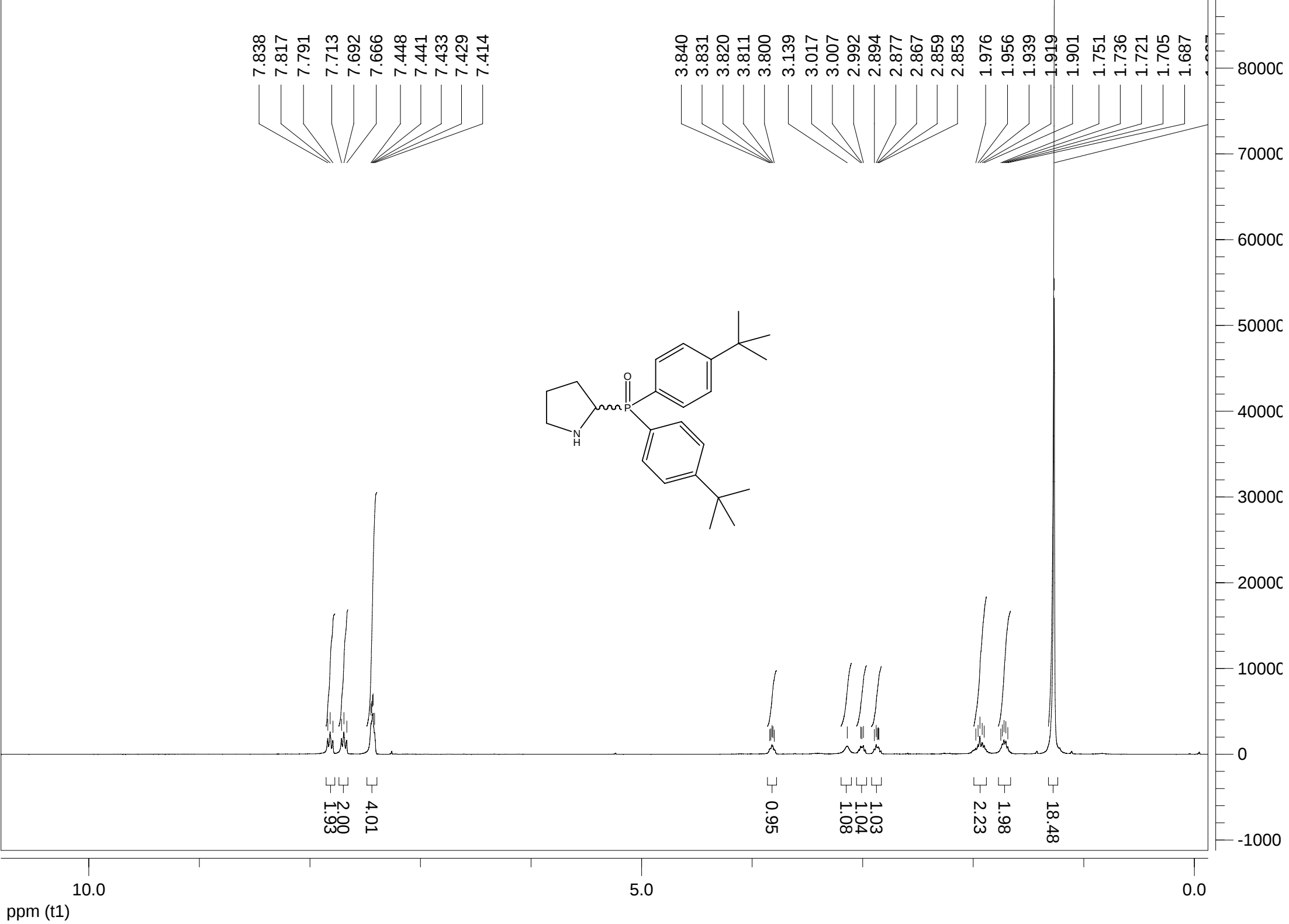


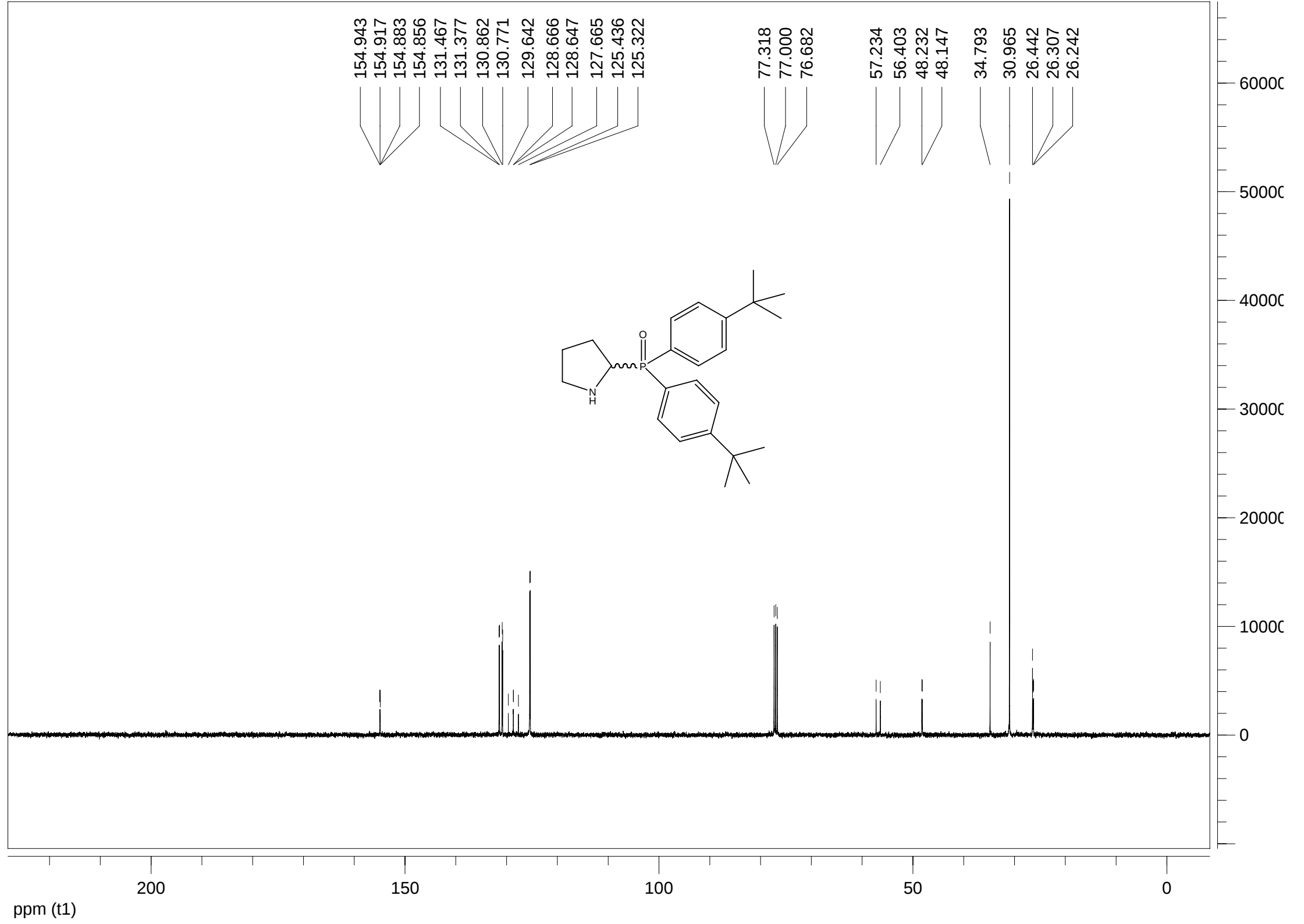


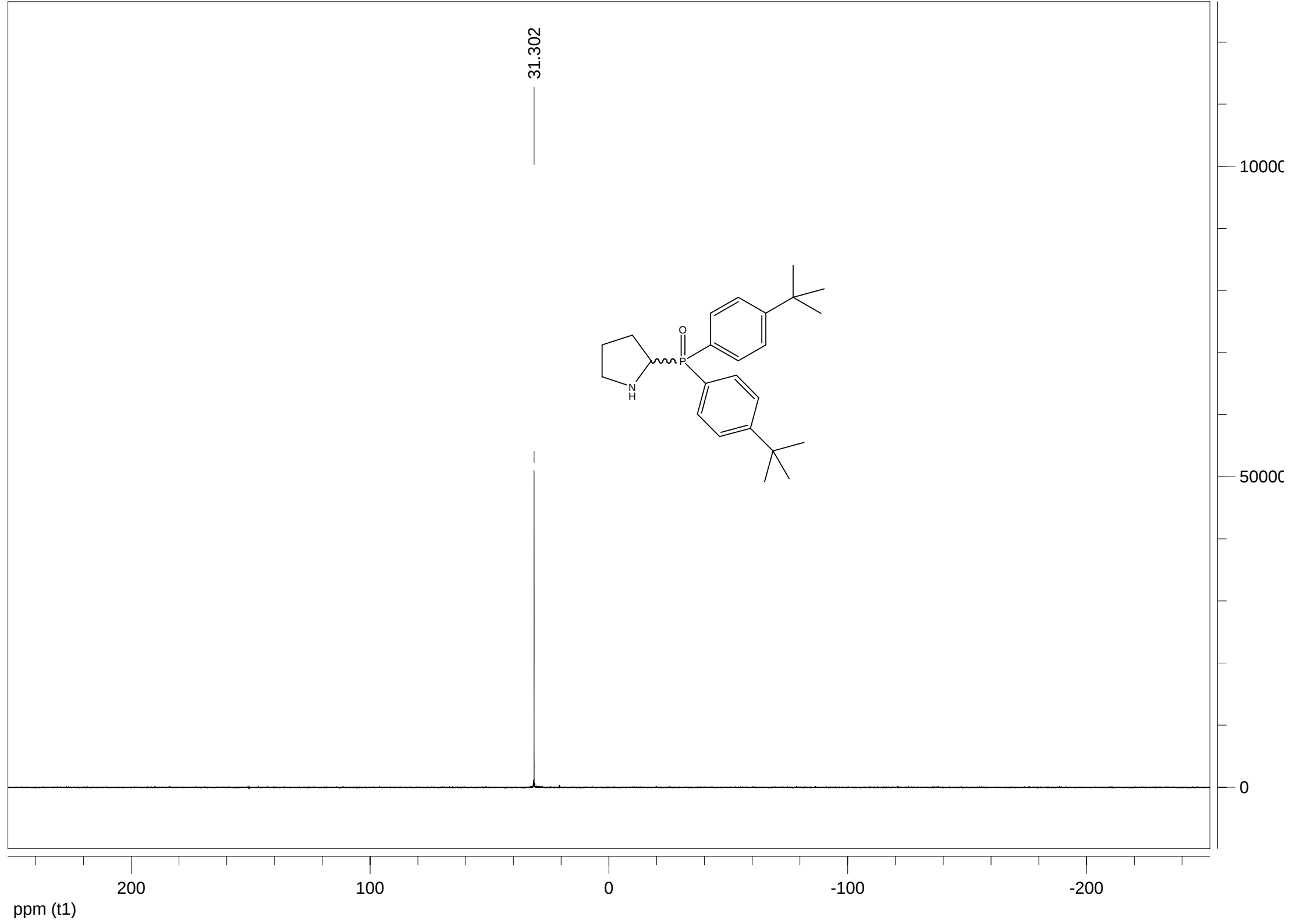
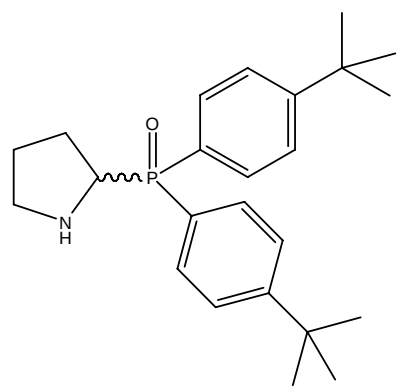


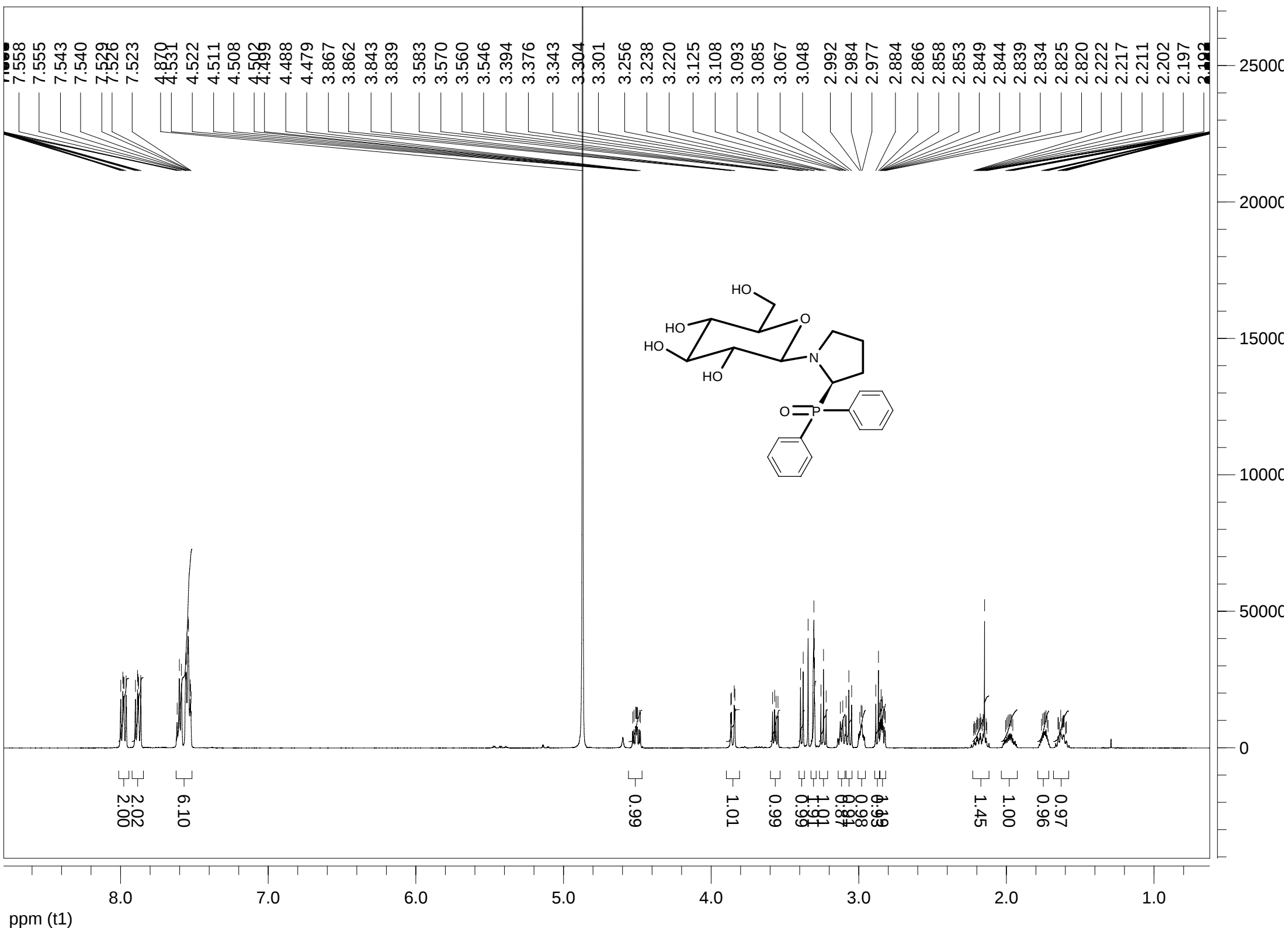


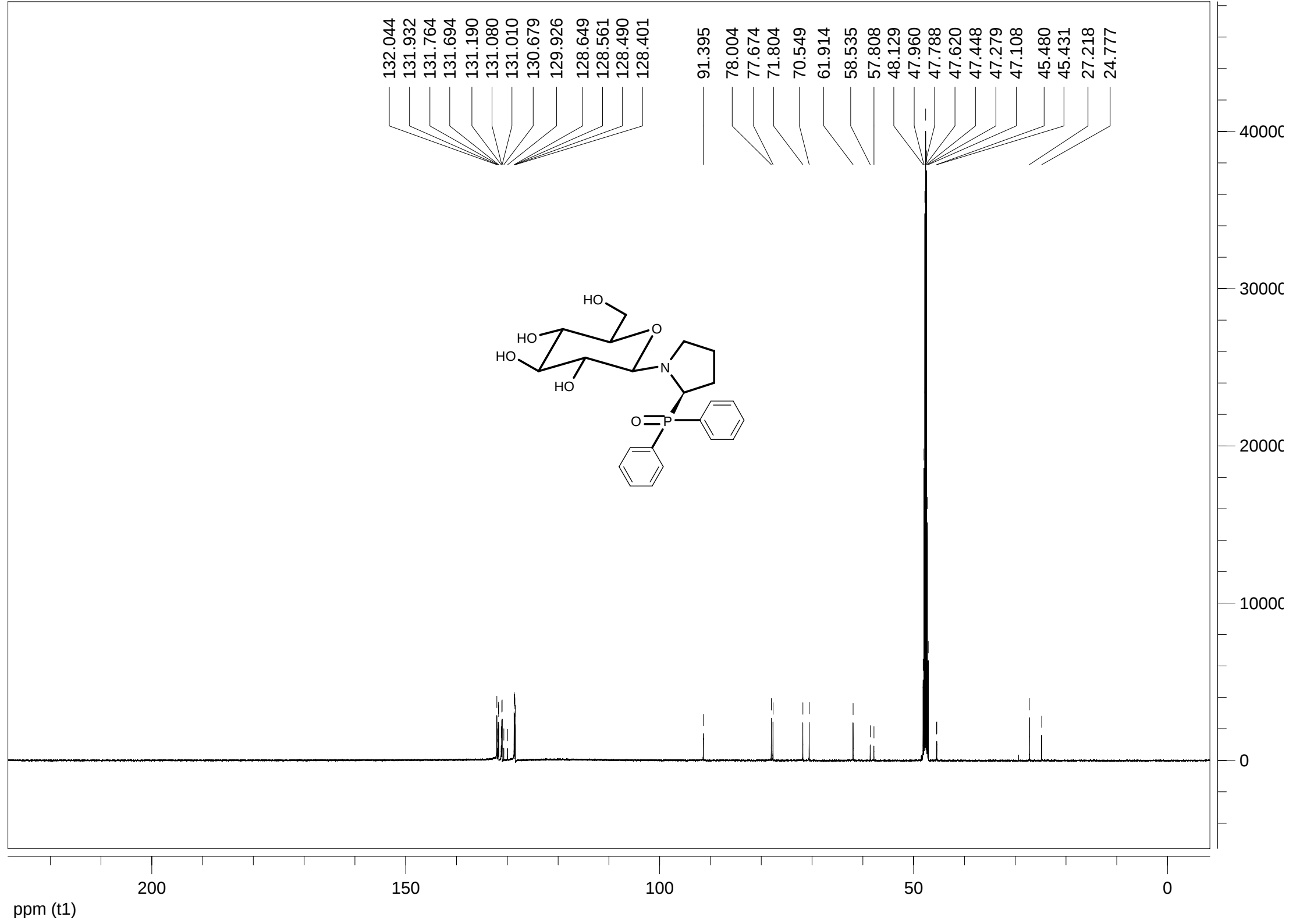


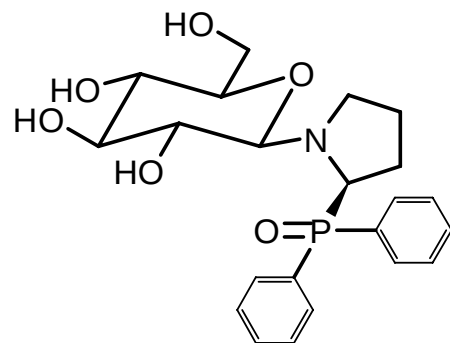










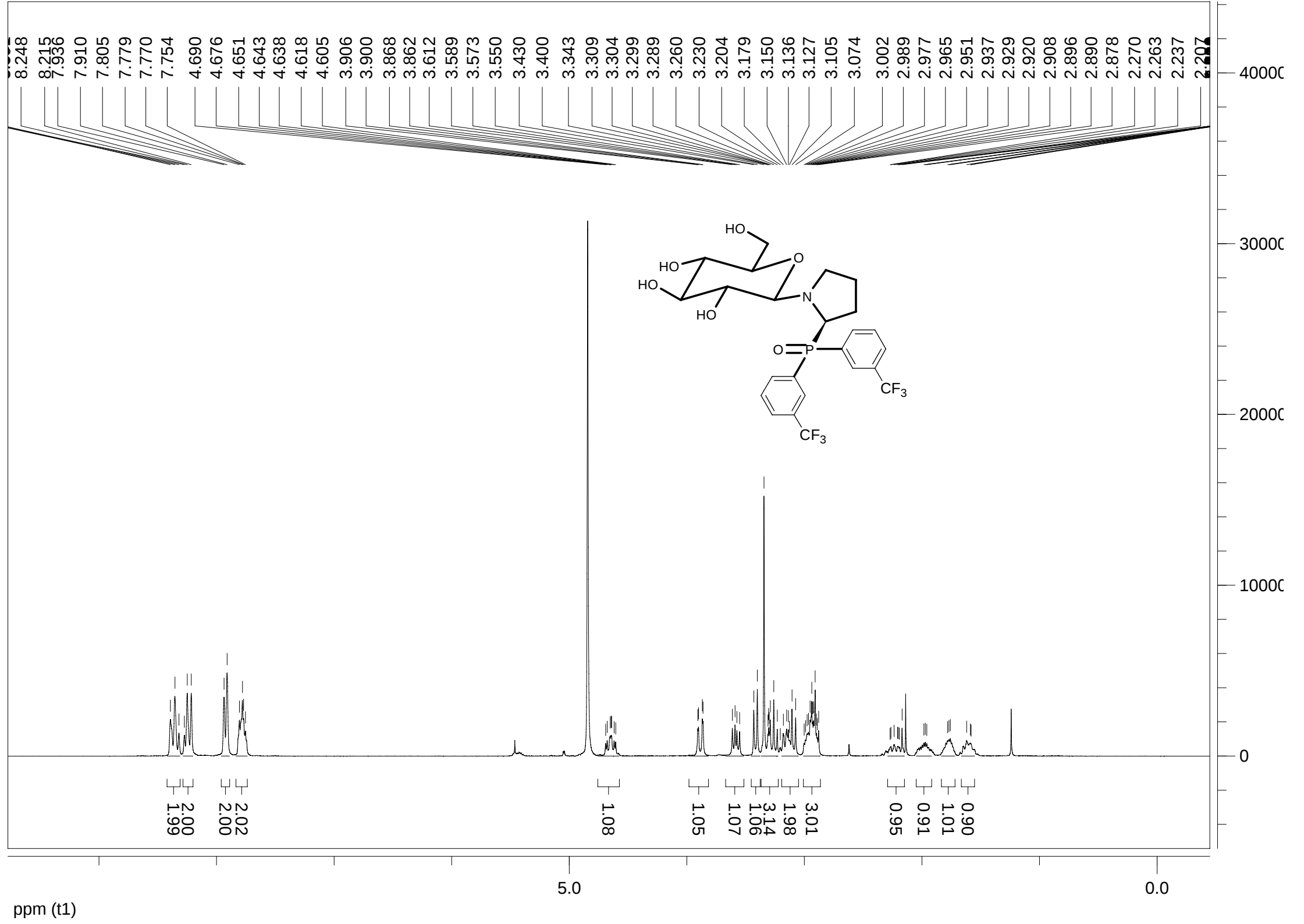


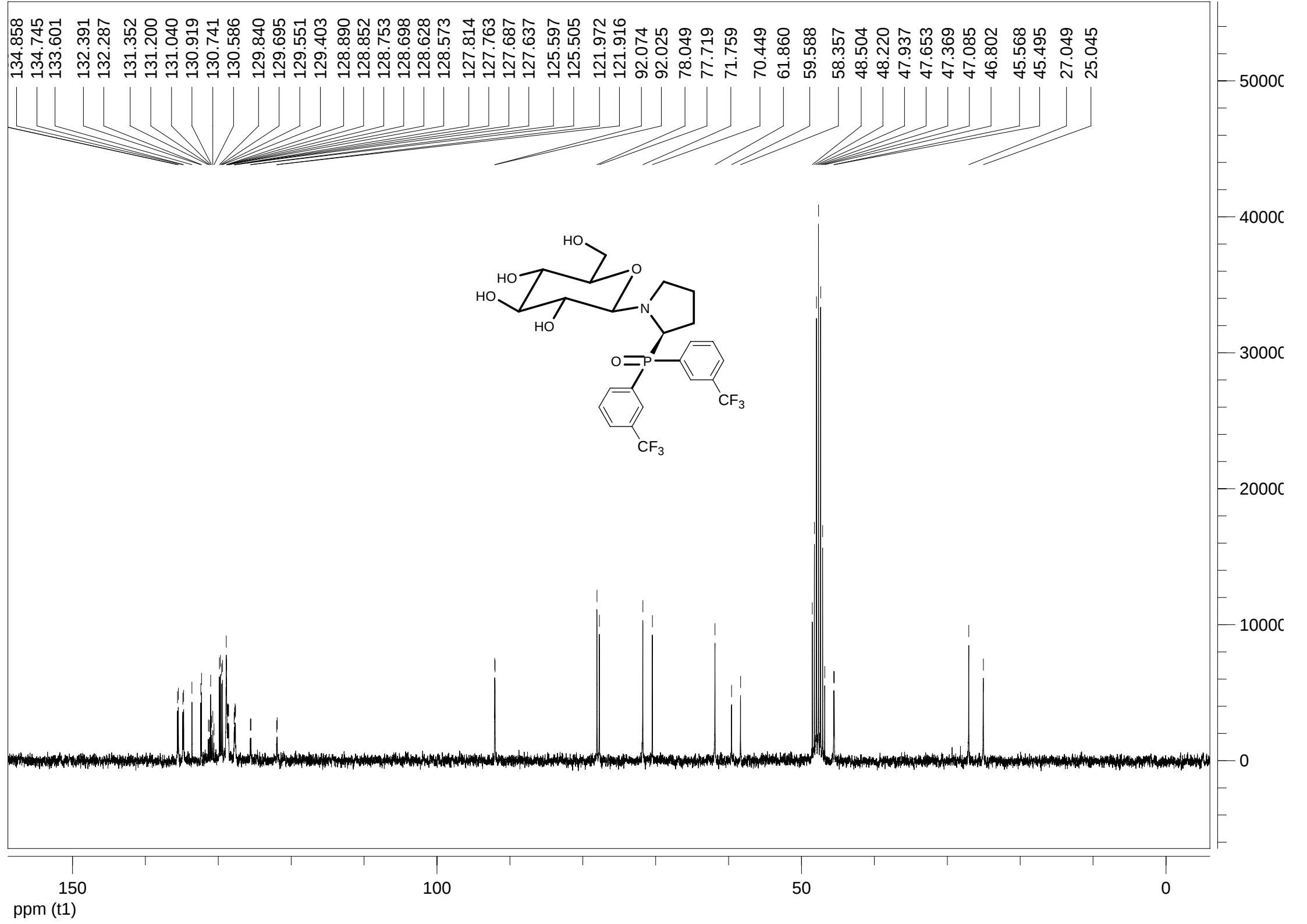
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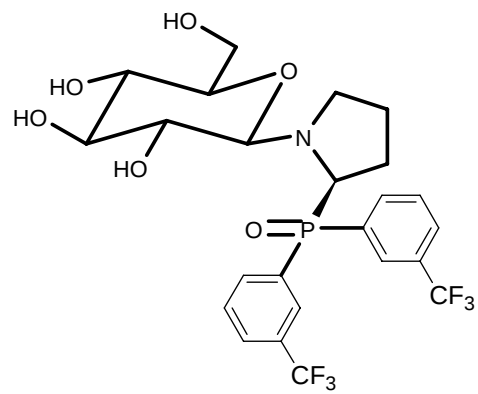
50000

0

ppm (t1)







30.900

10000

50000

0

200

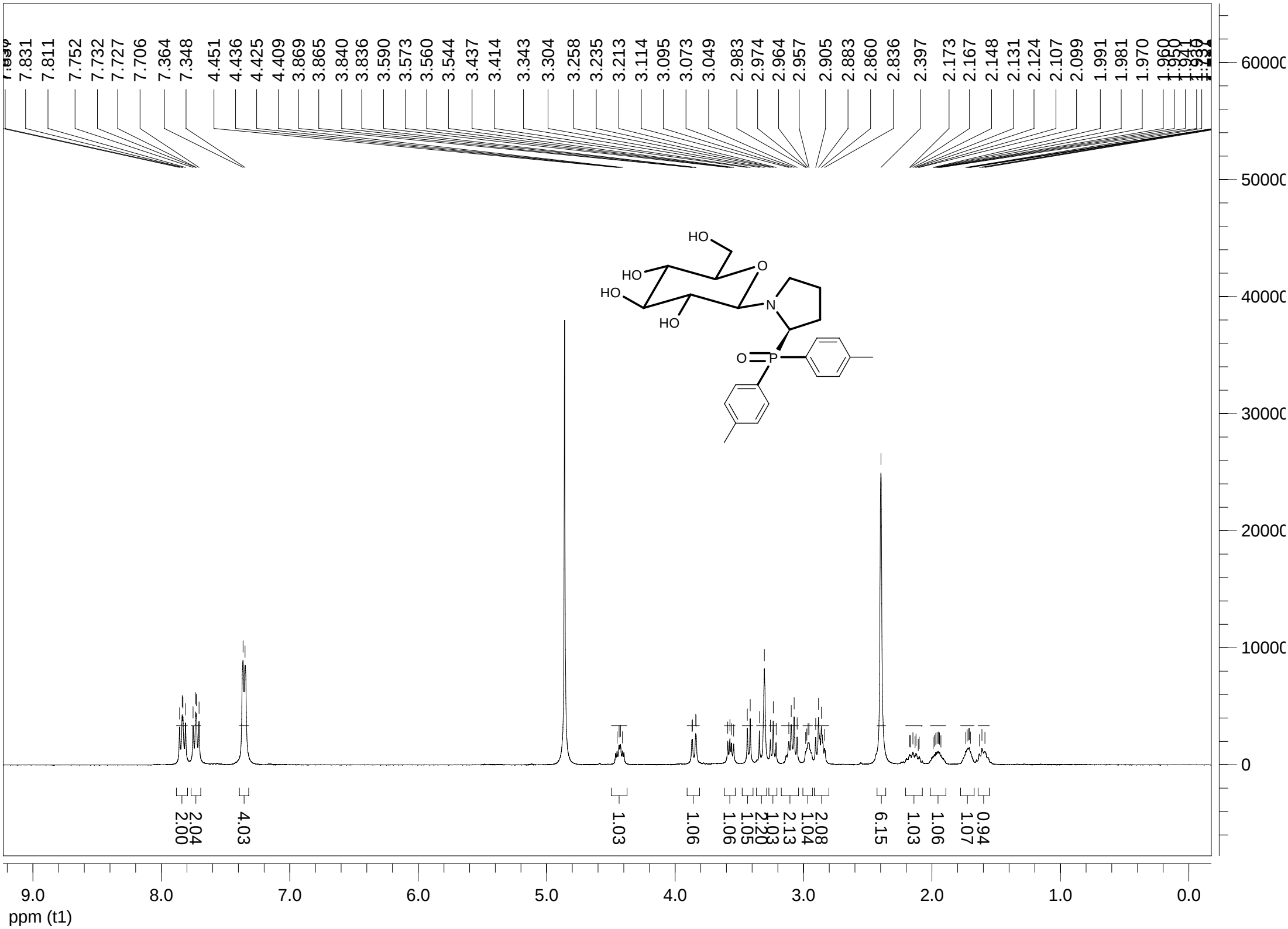
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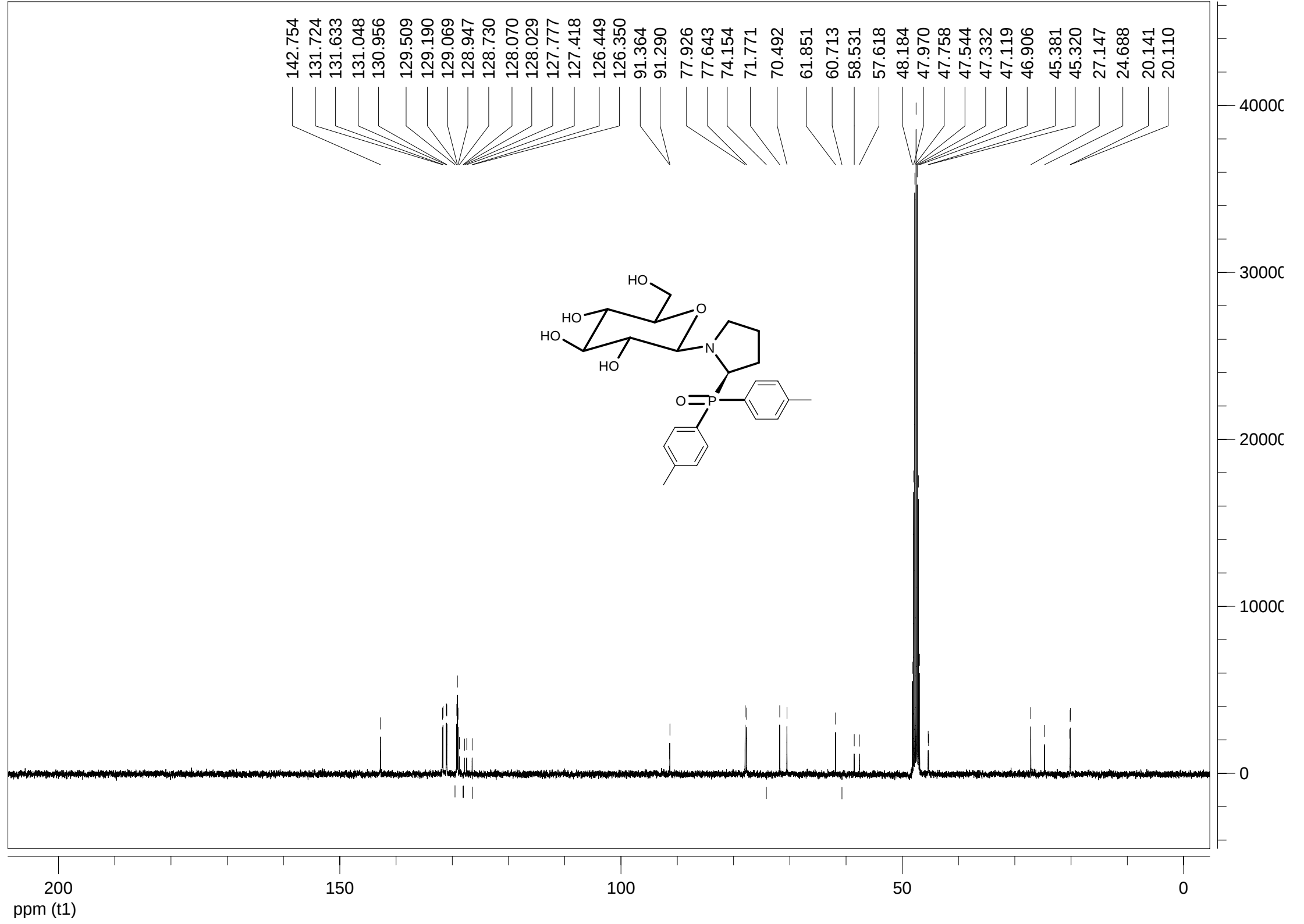
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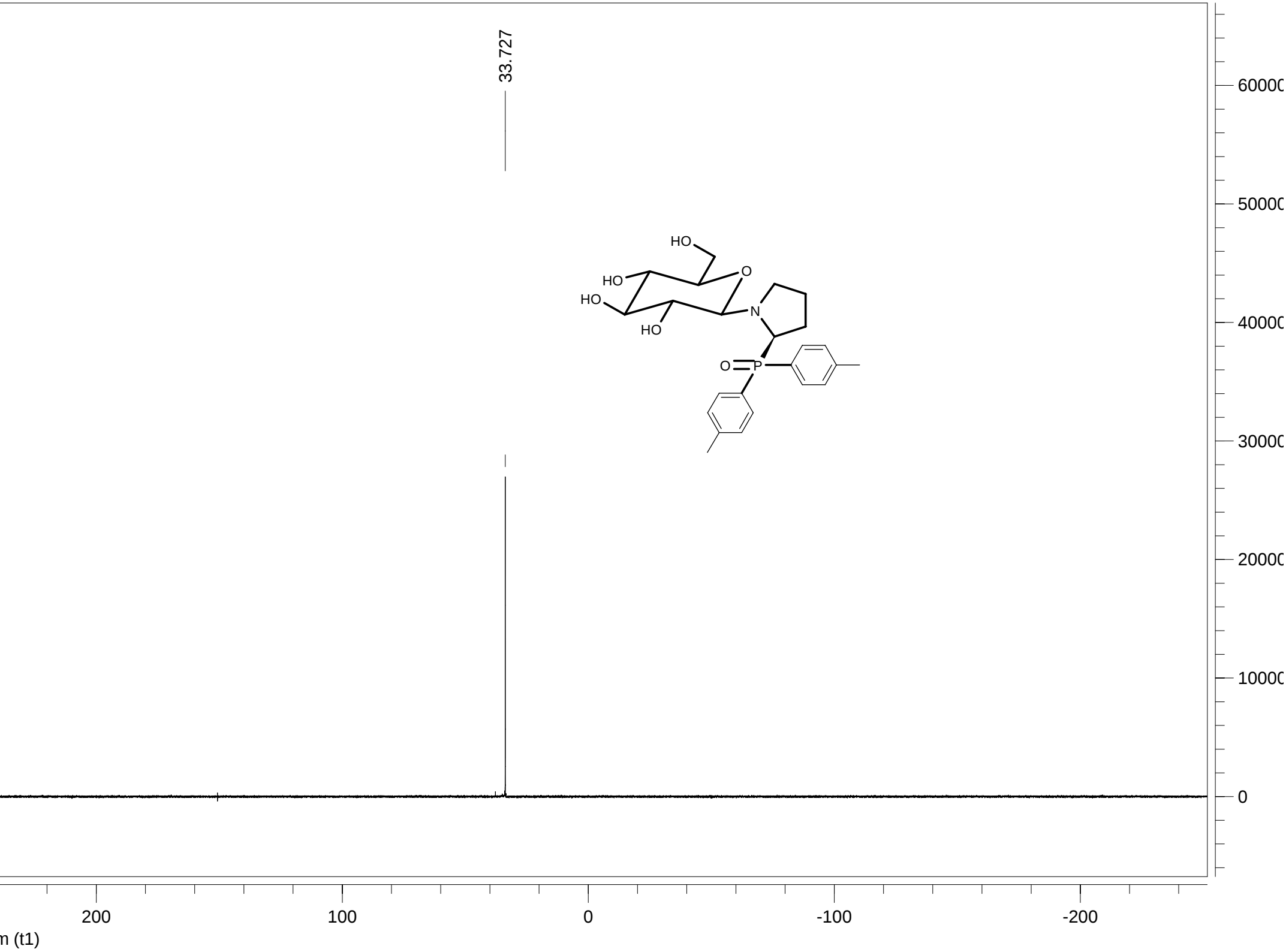
-100

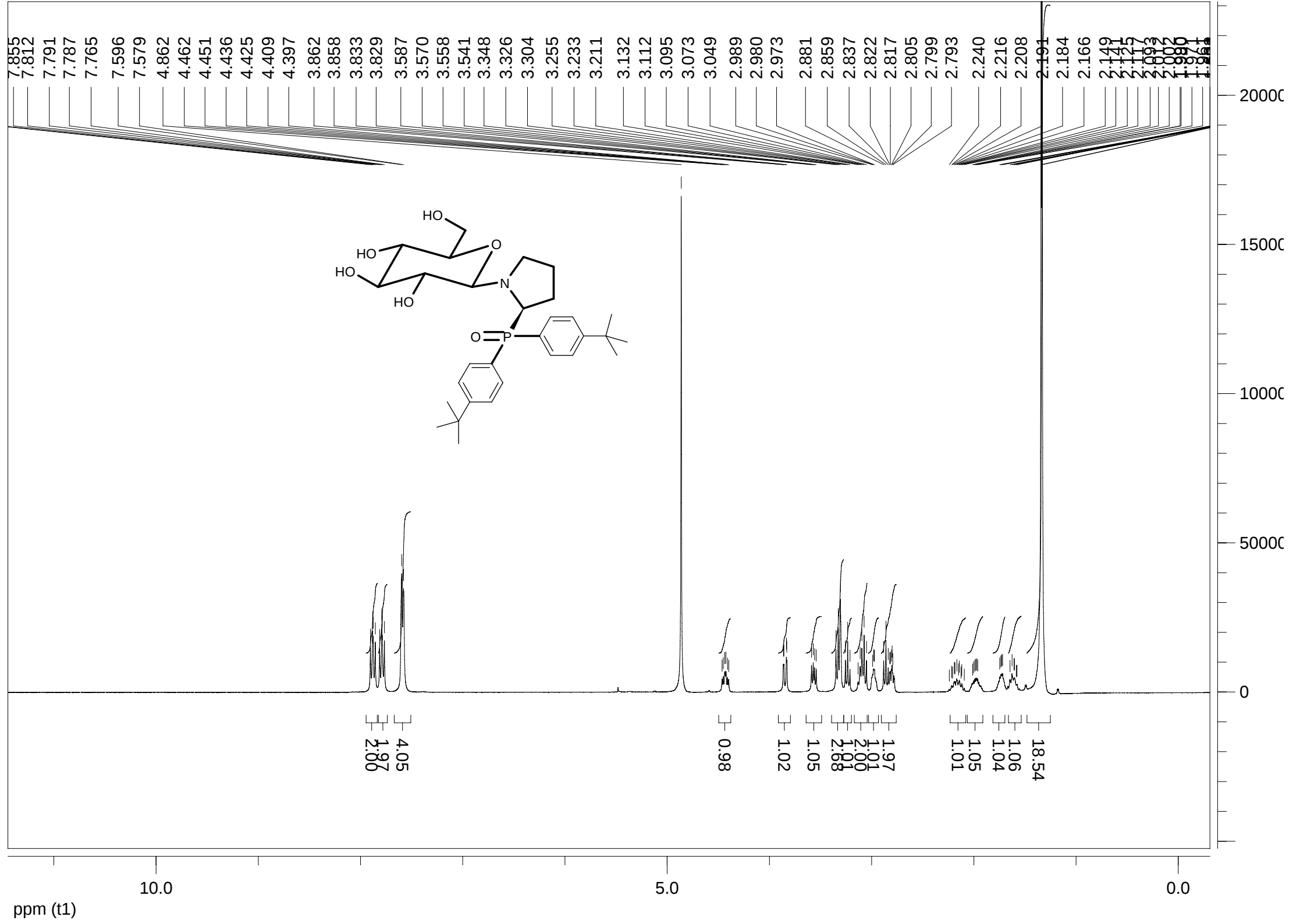
-200

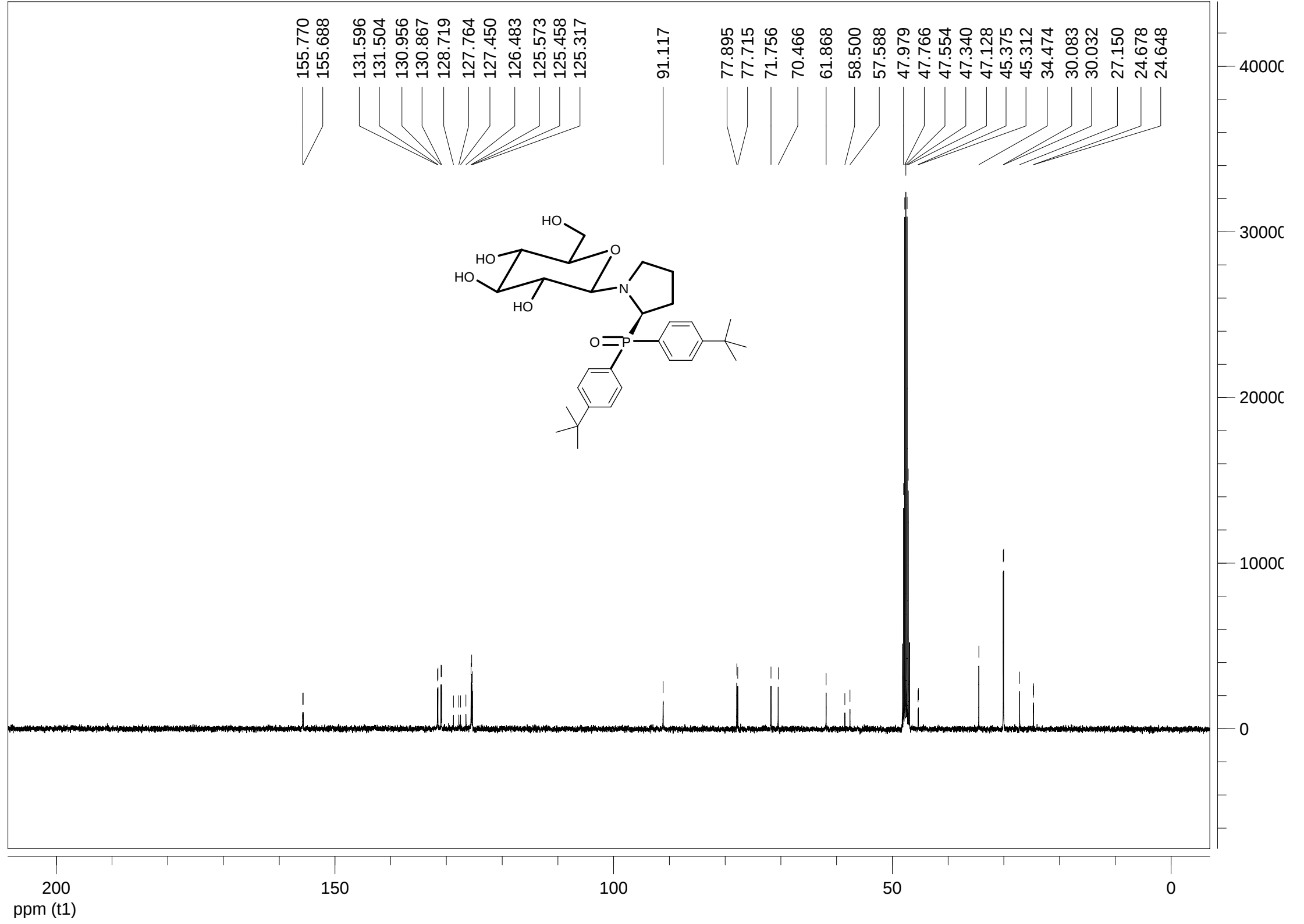
ppm (t1)

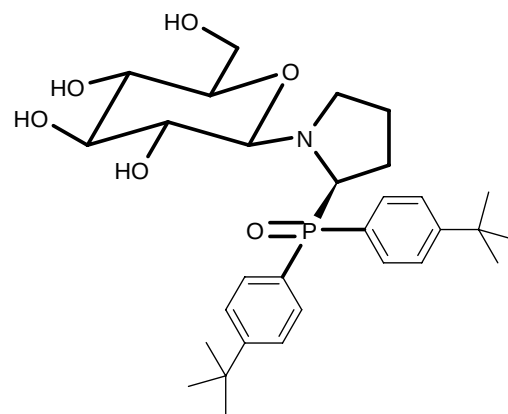




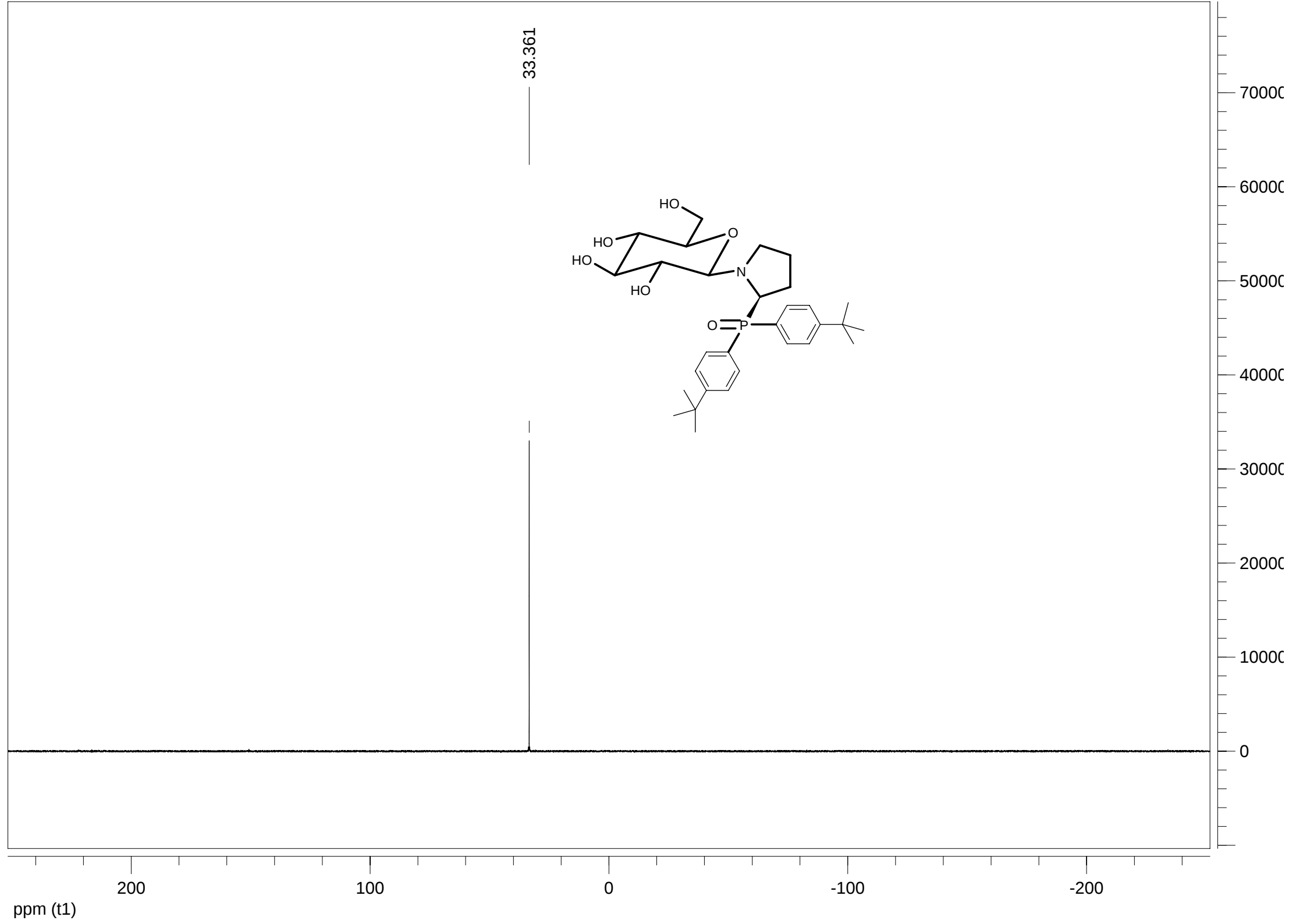




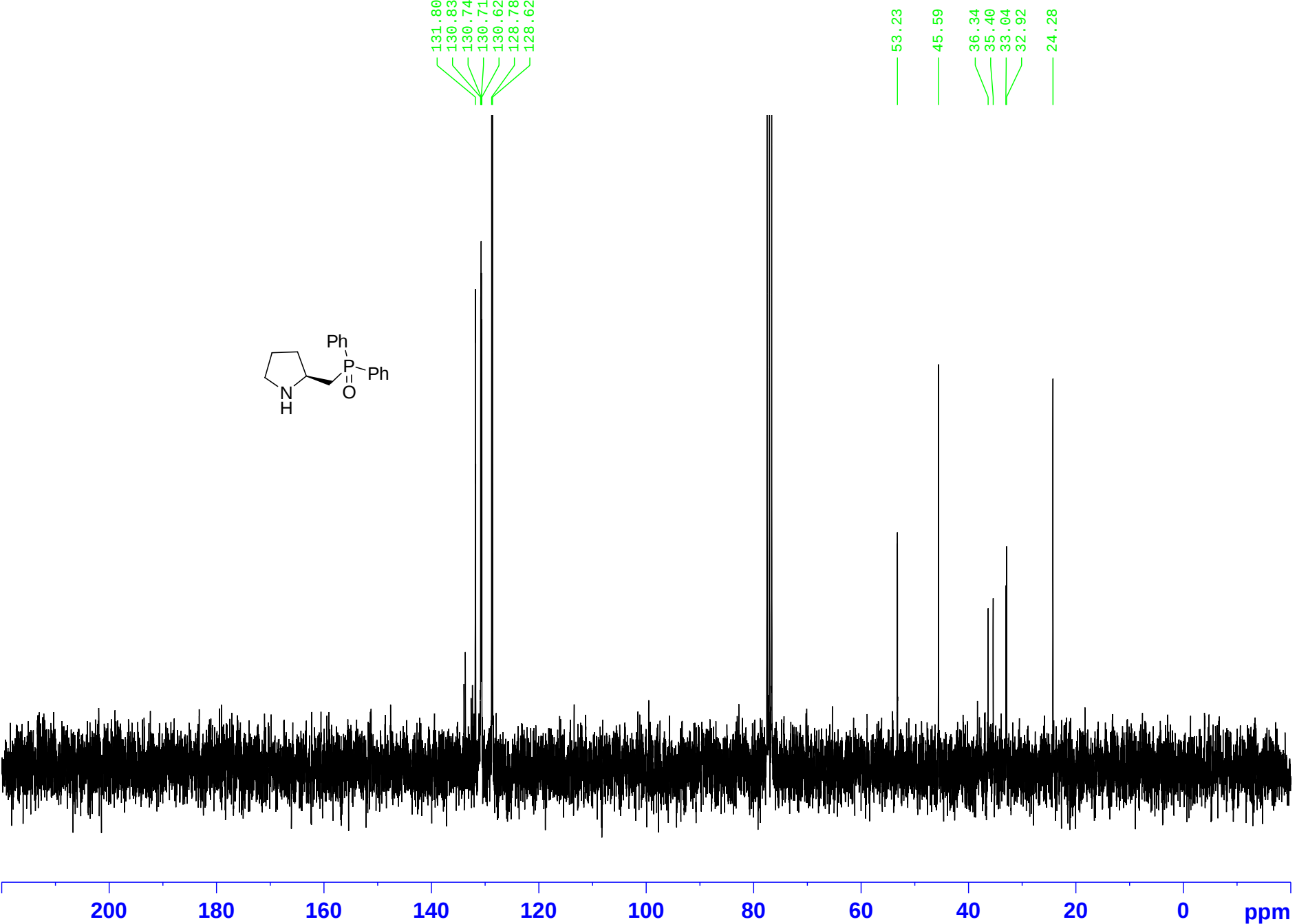
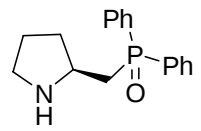




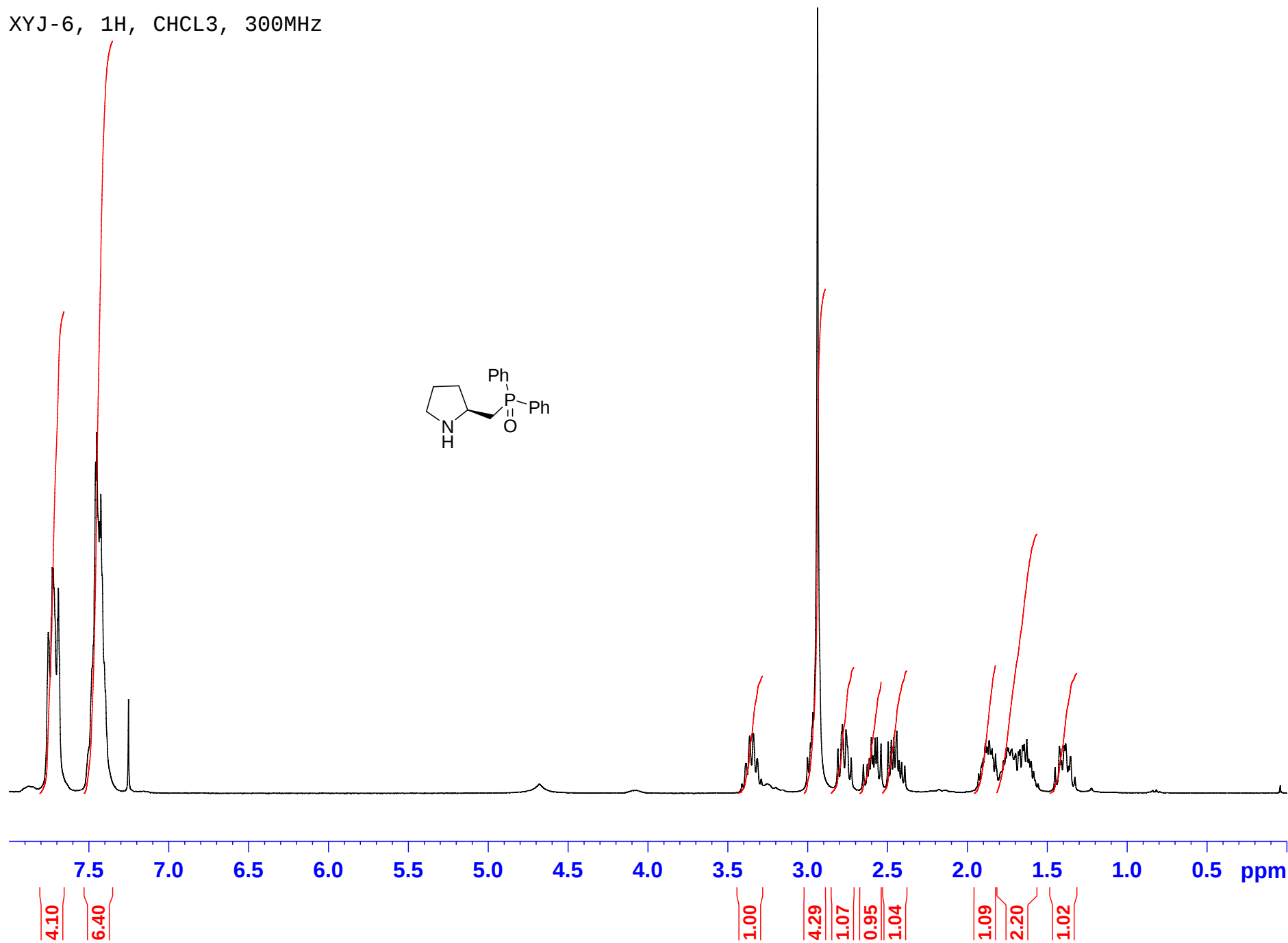
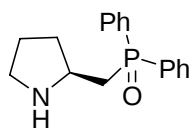
33.361



XYJ-6, ¹³C, CHCL₃, 300MHz



XYJ-6, 1H, CHCL3, 300MHz



ISO-3 31P CHCl3 400 MHz

