Catalytic hydroamination of unactivated olefins using a Co catalyst for complex molecule synthesis

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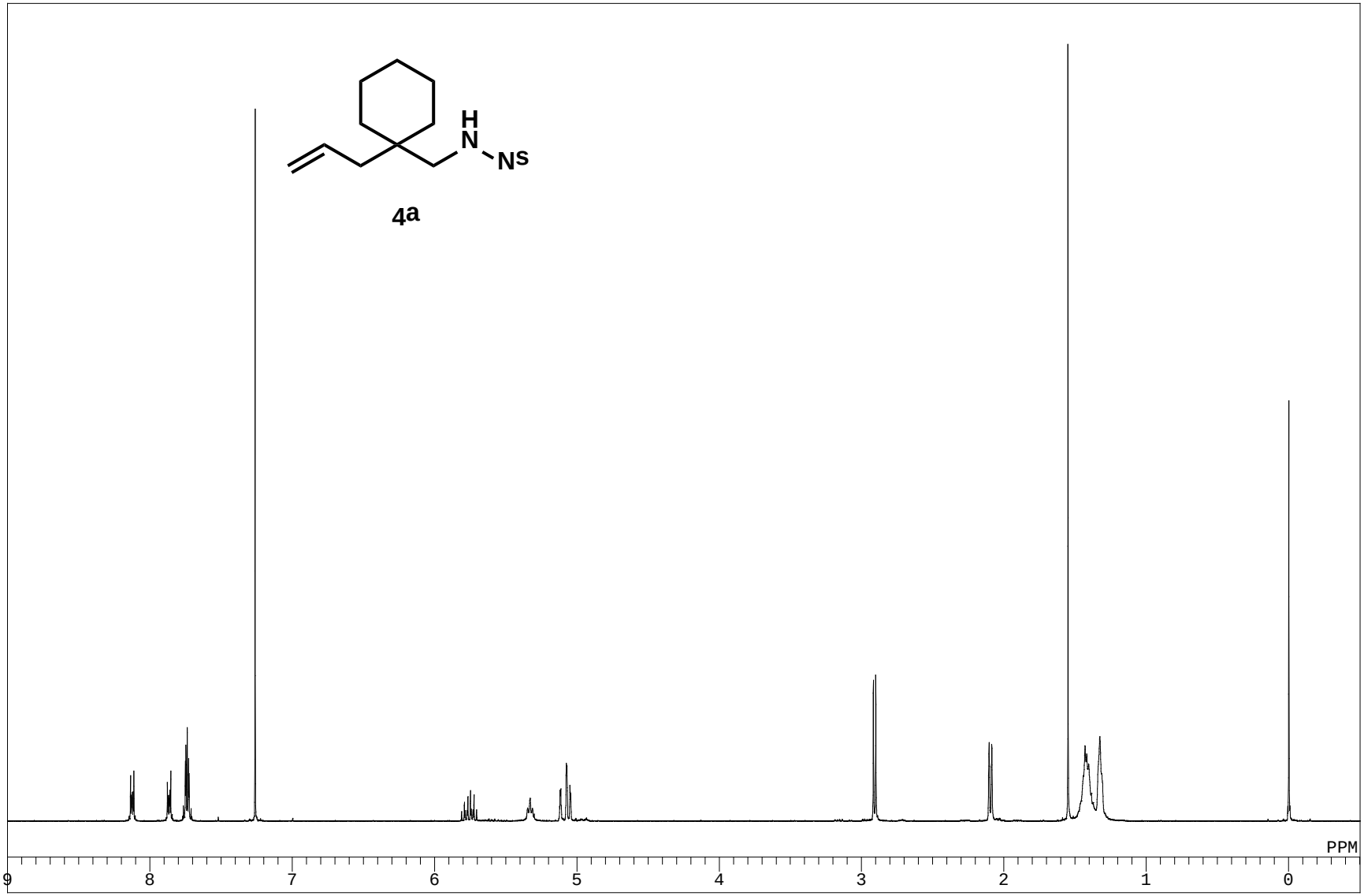
SUPPORTING INFORMATION

Part 2 ^{1}H ,^{13}C NMR, X-ray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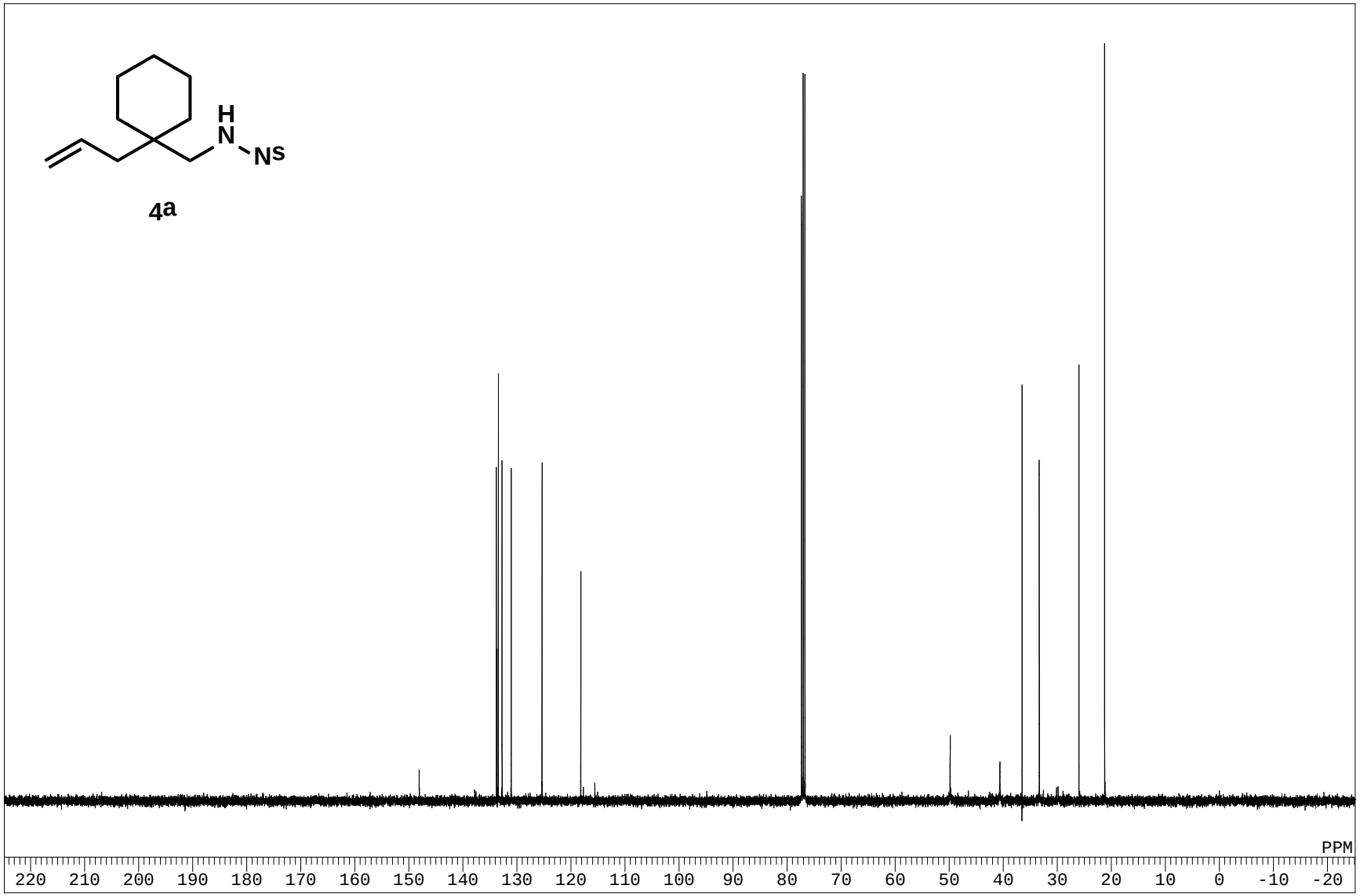
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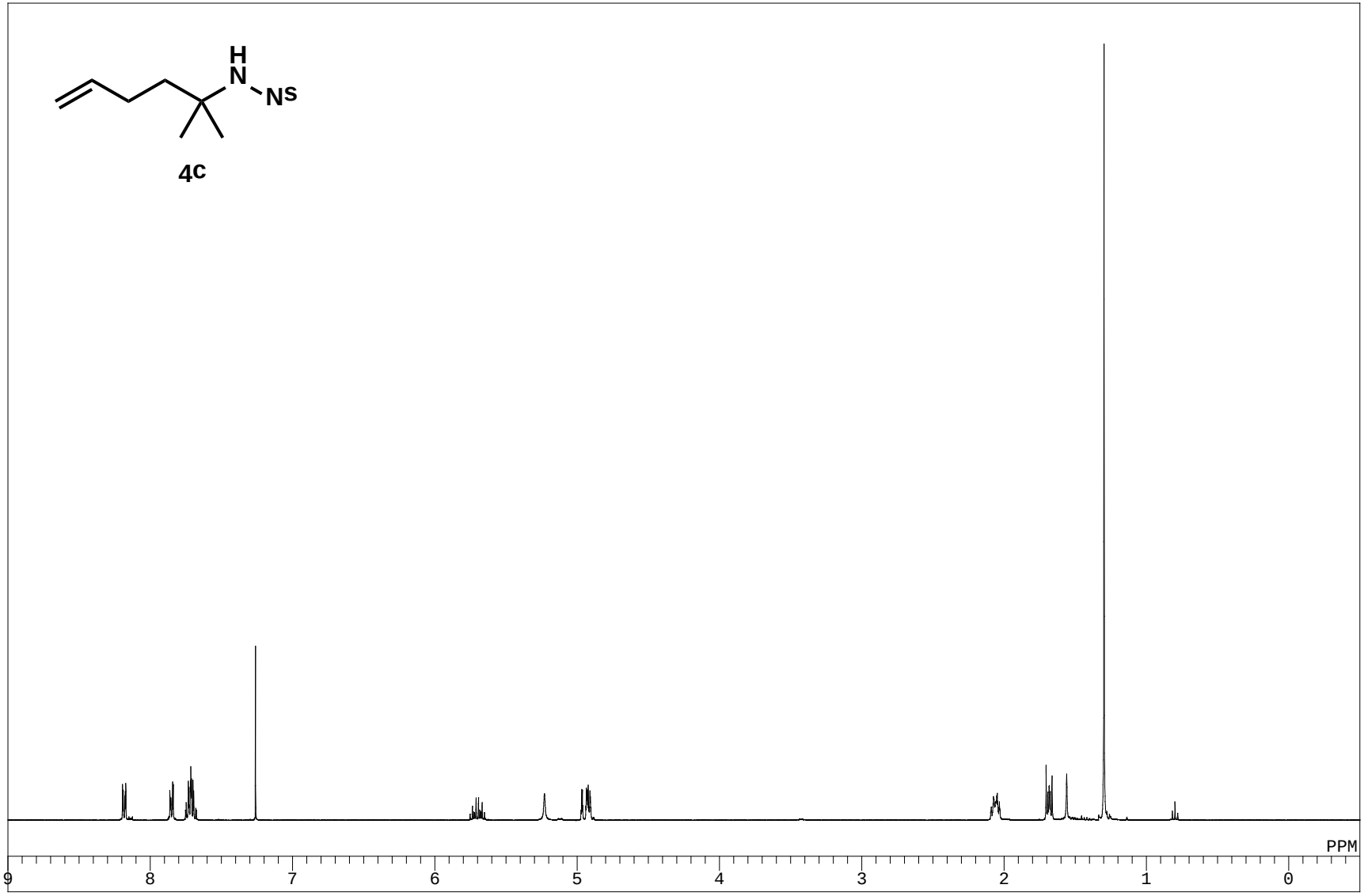
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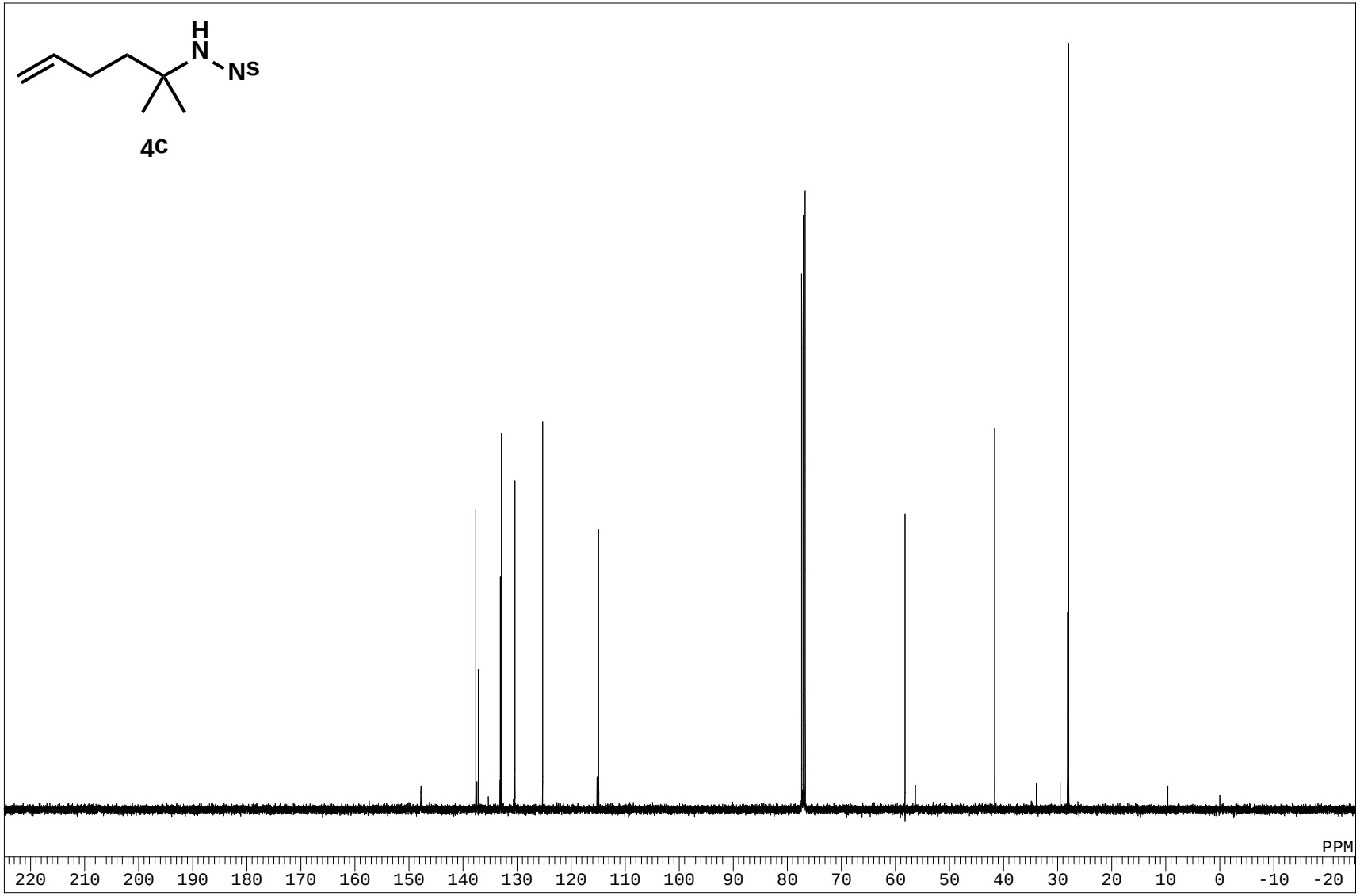
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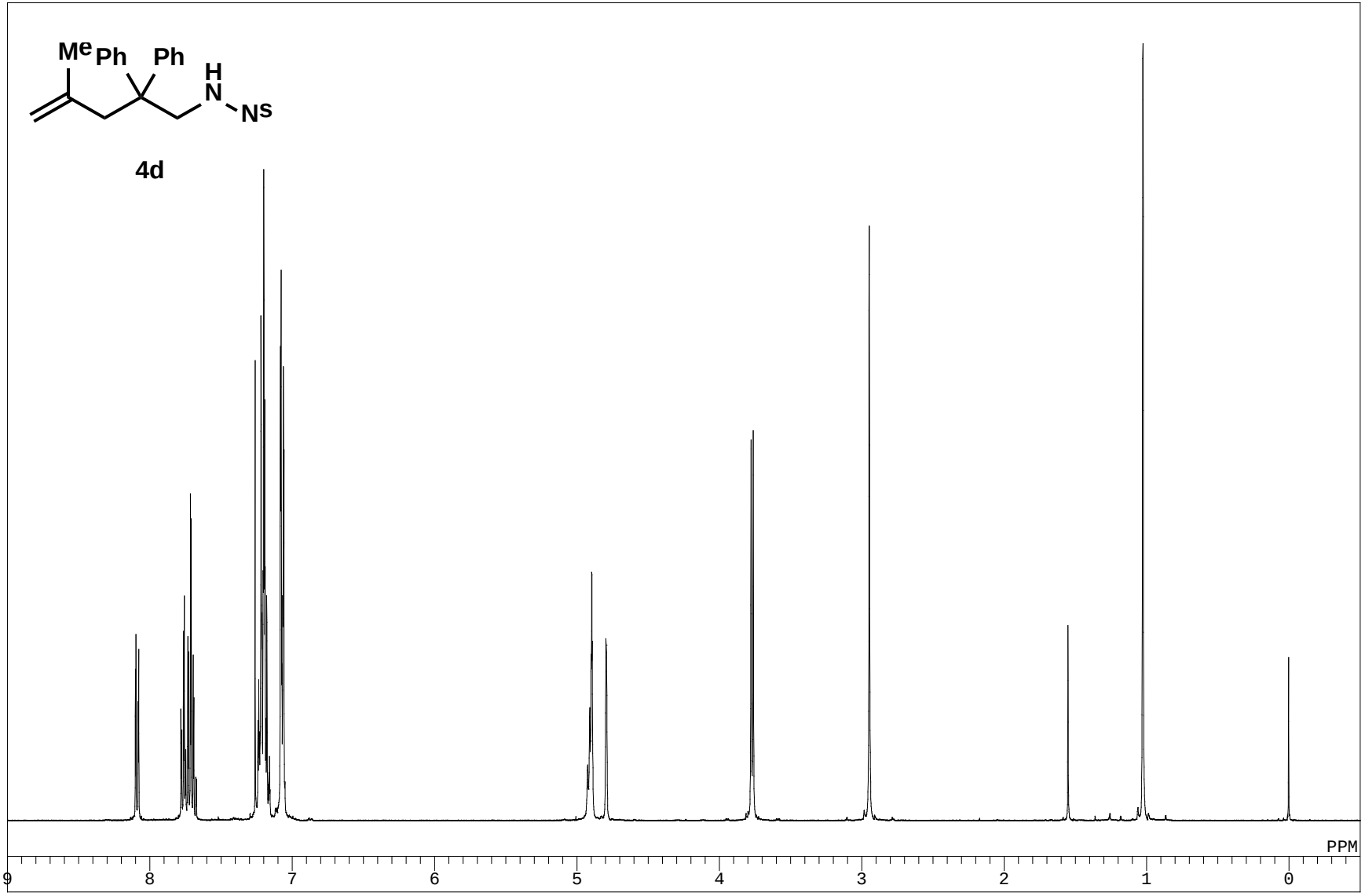
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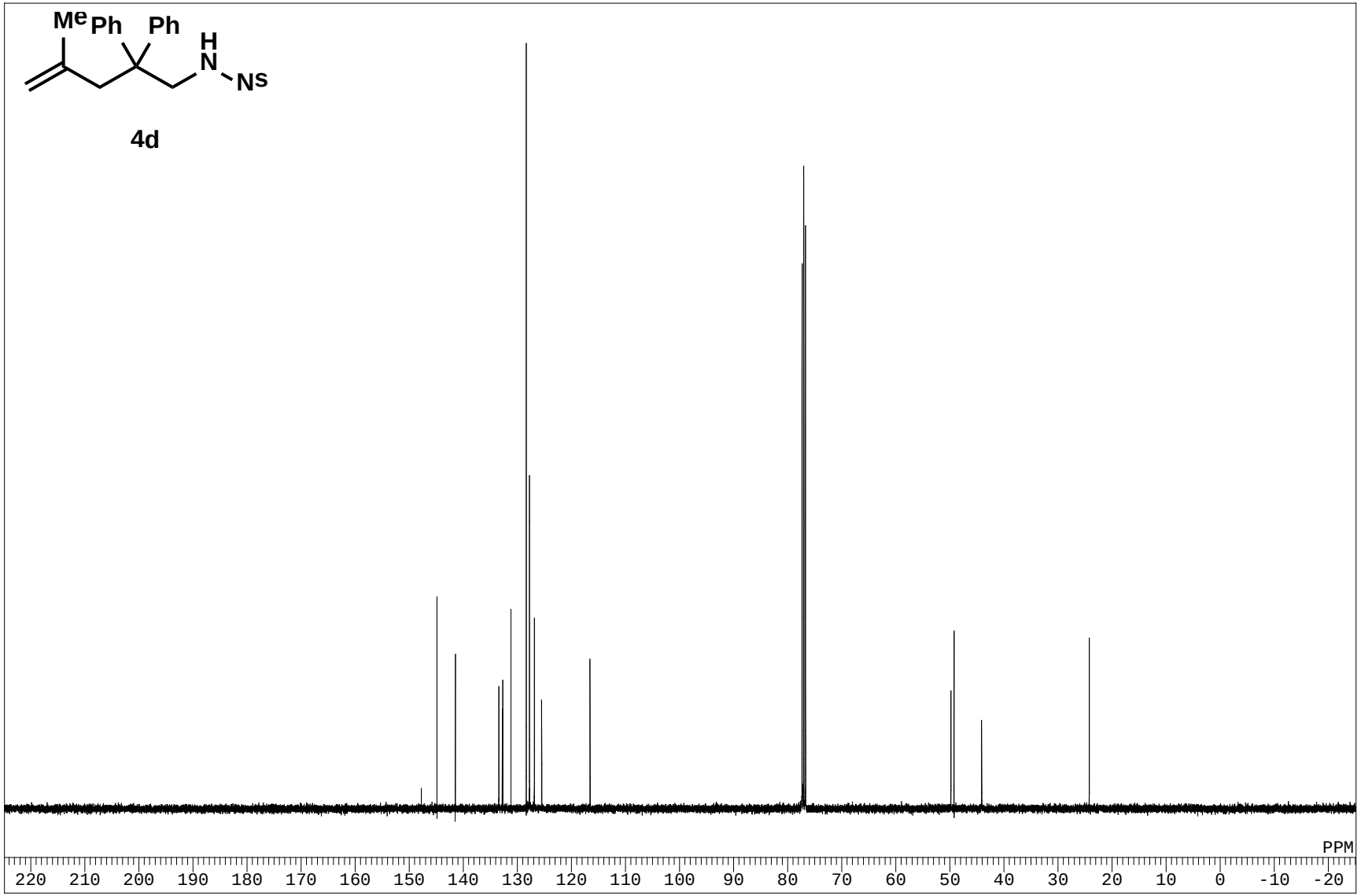
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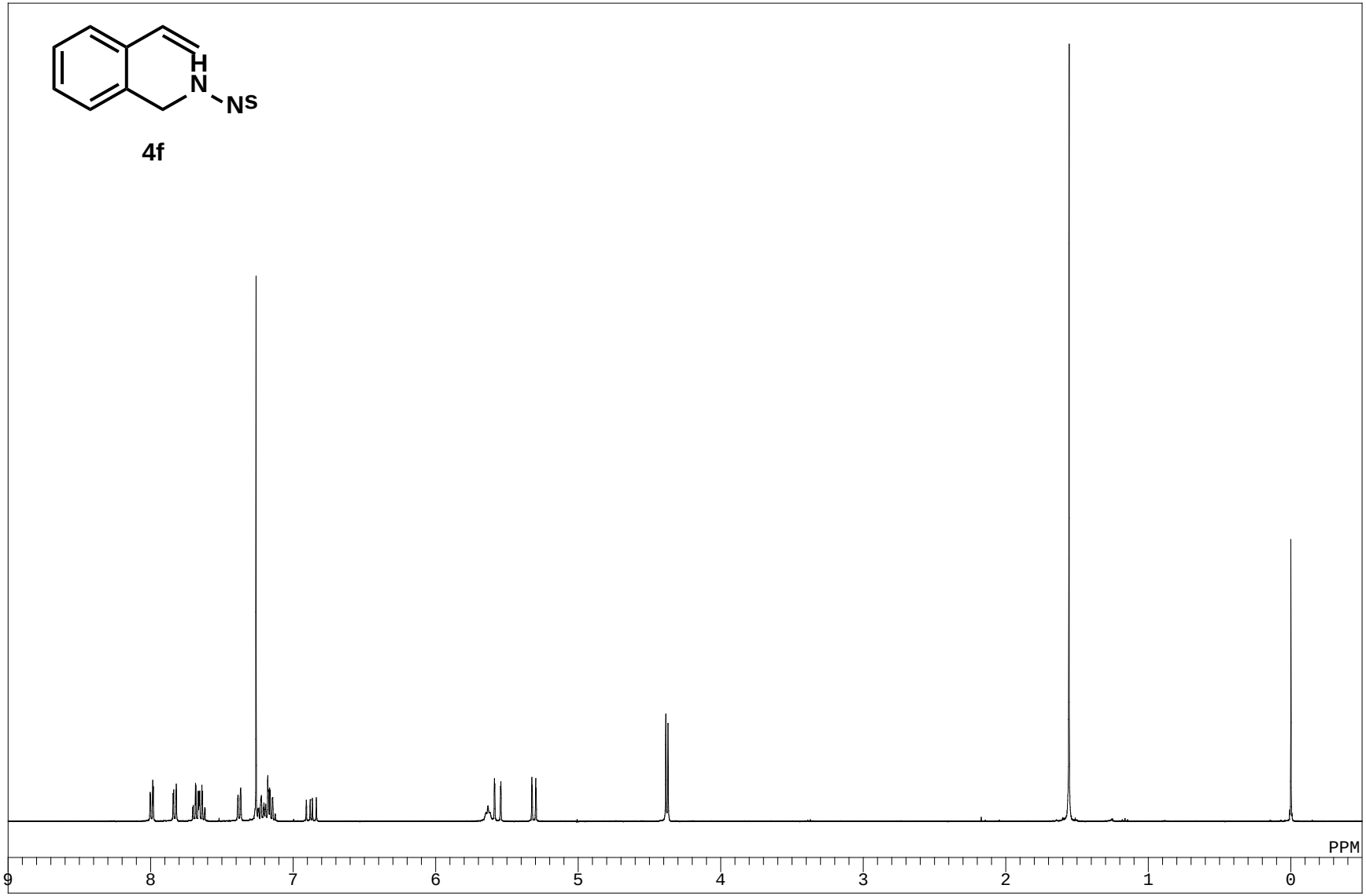
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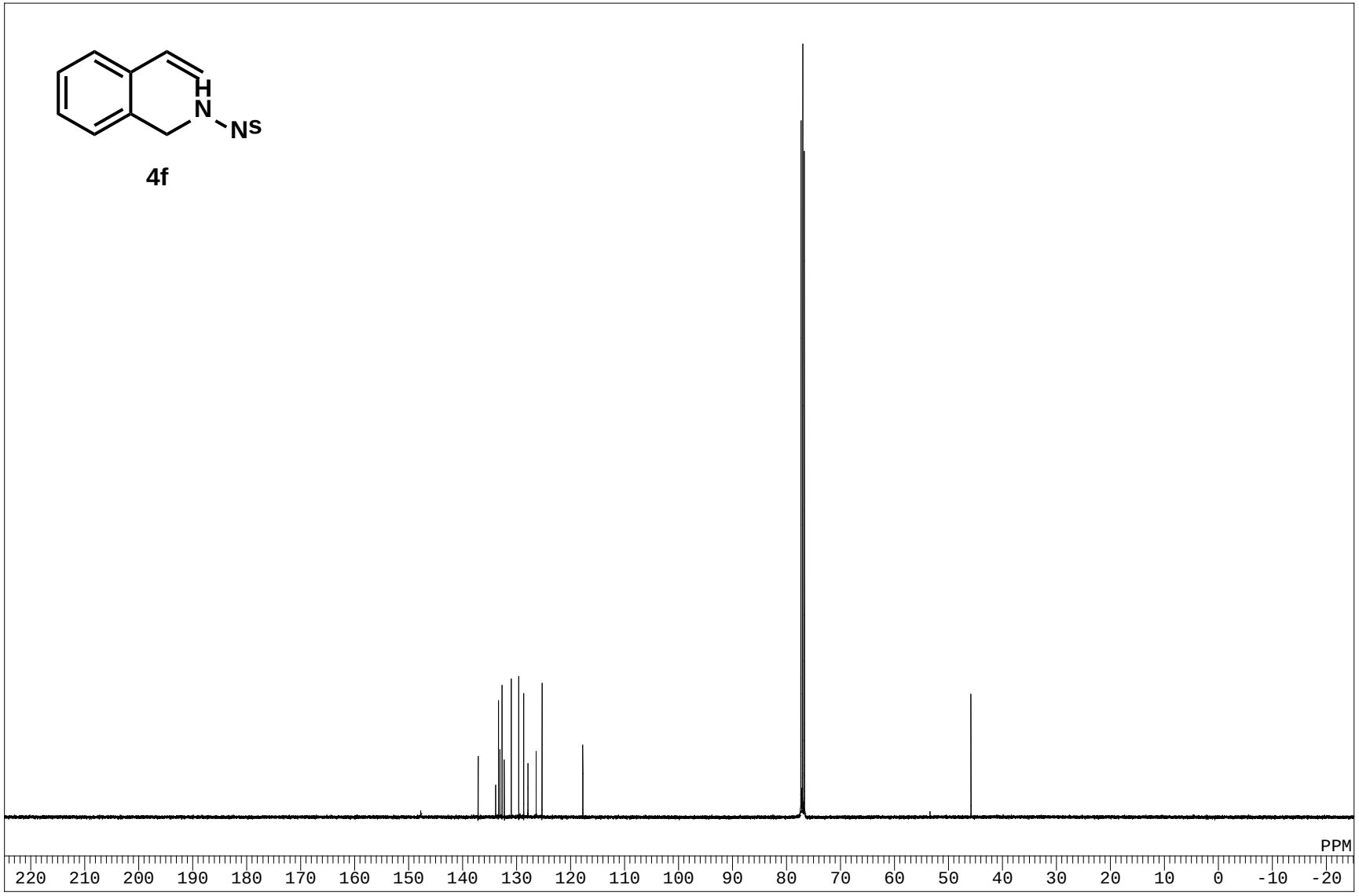
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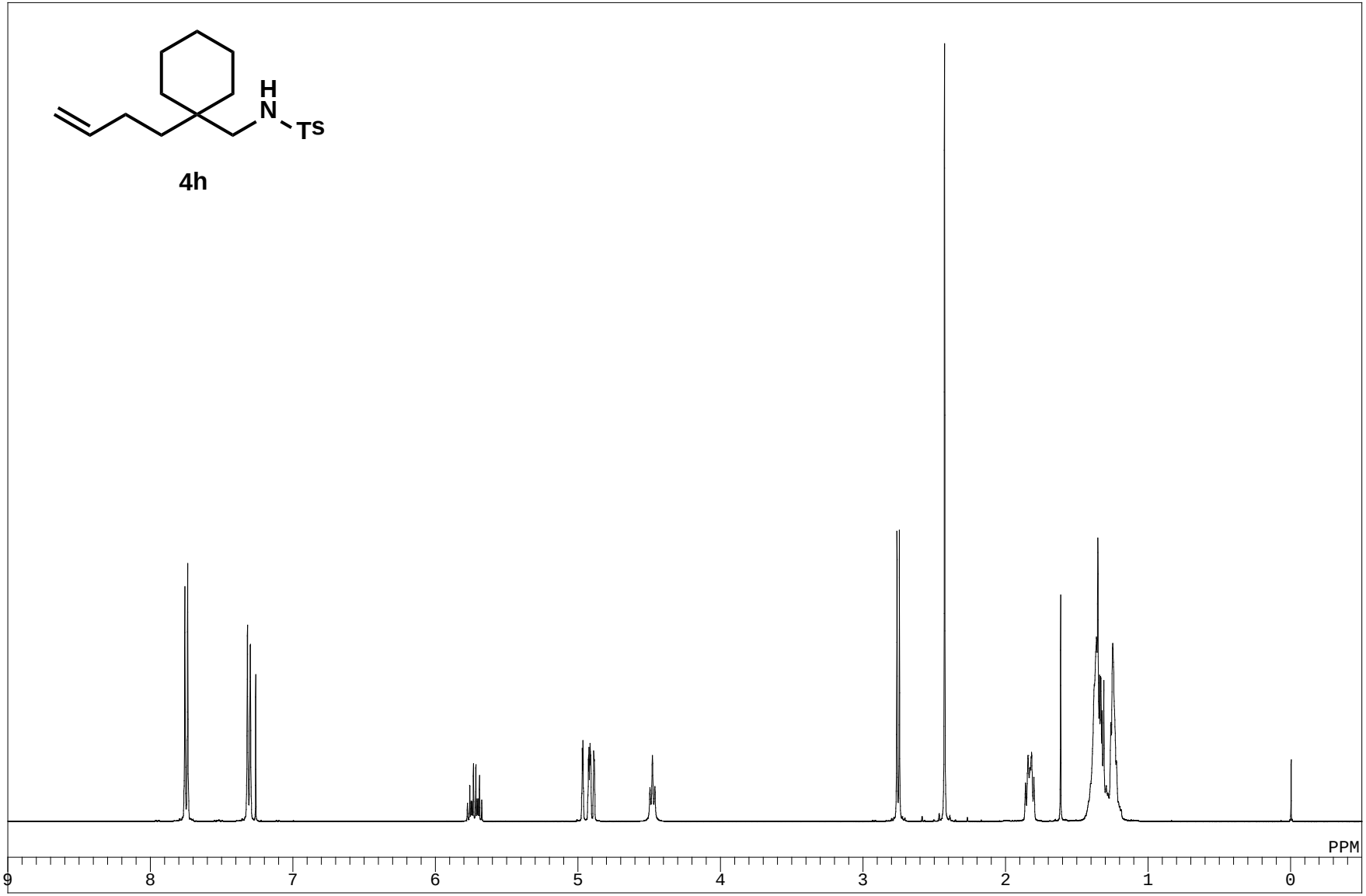
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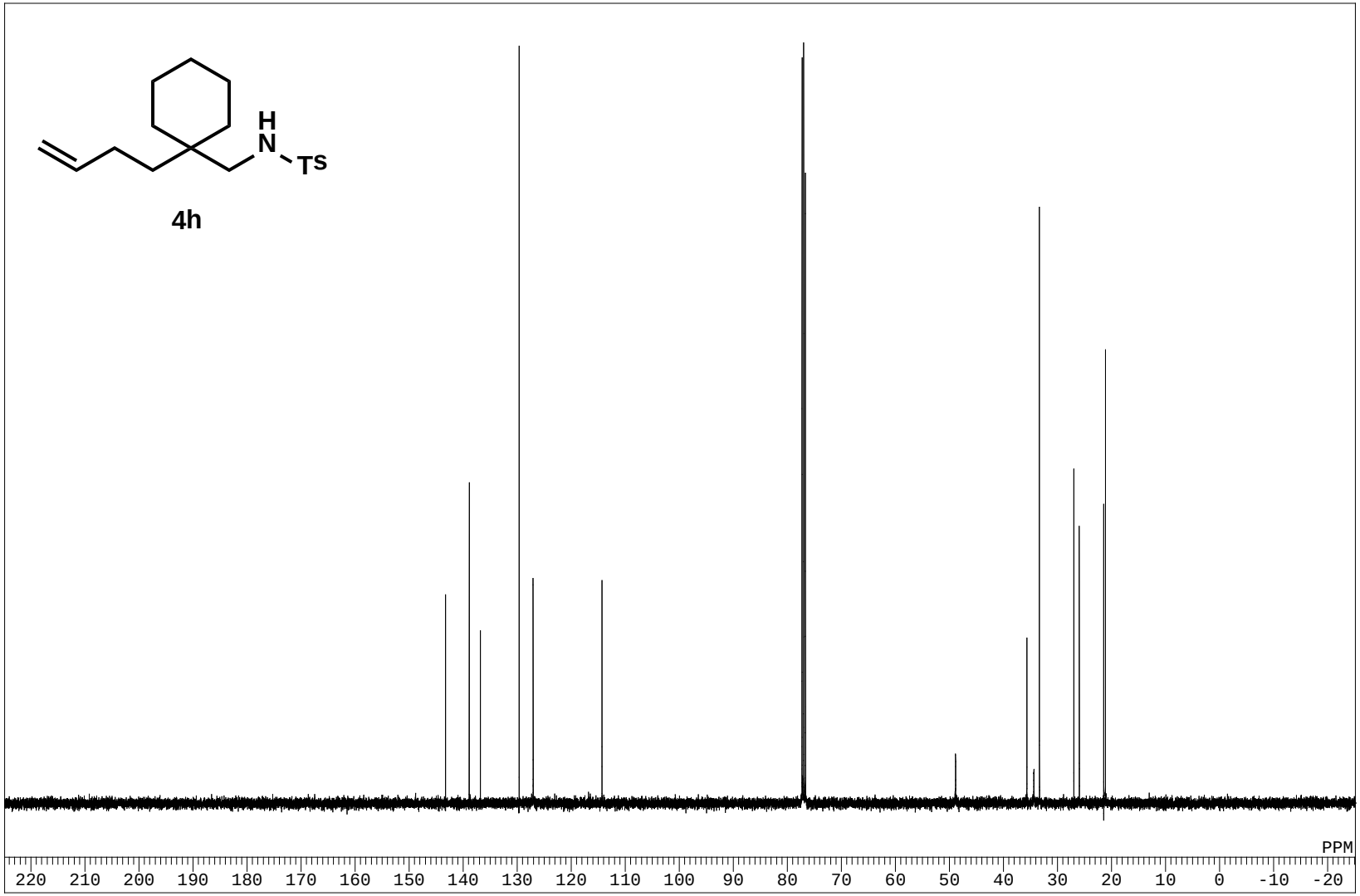
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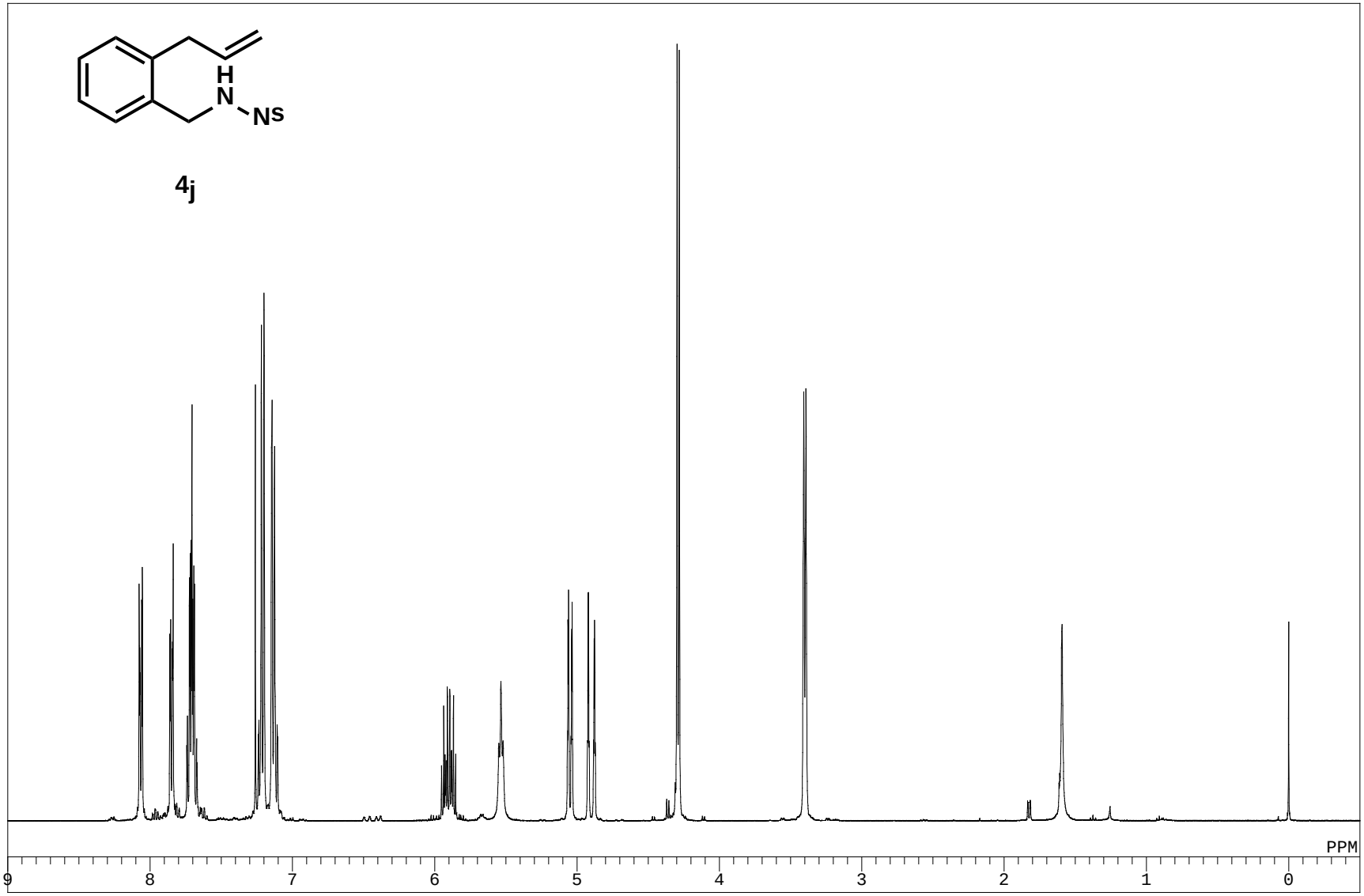
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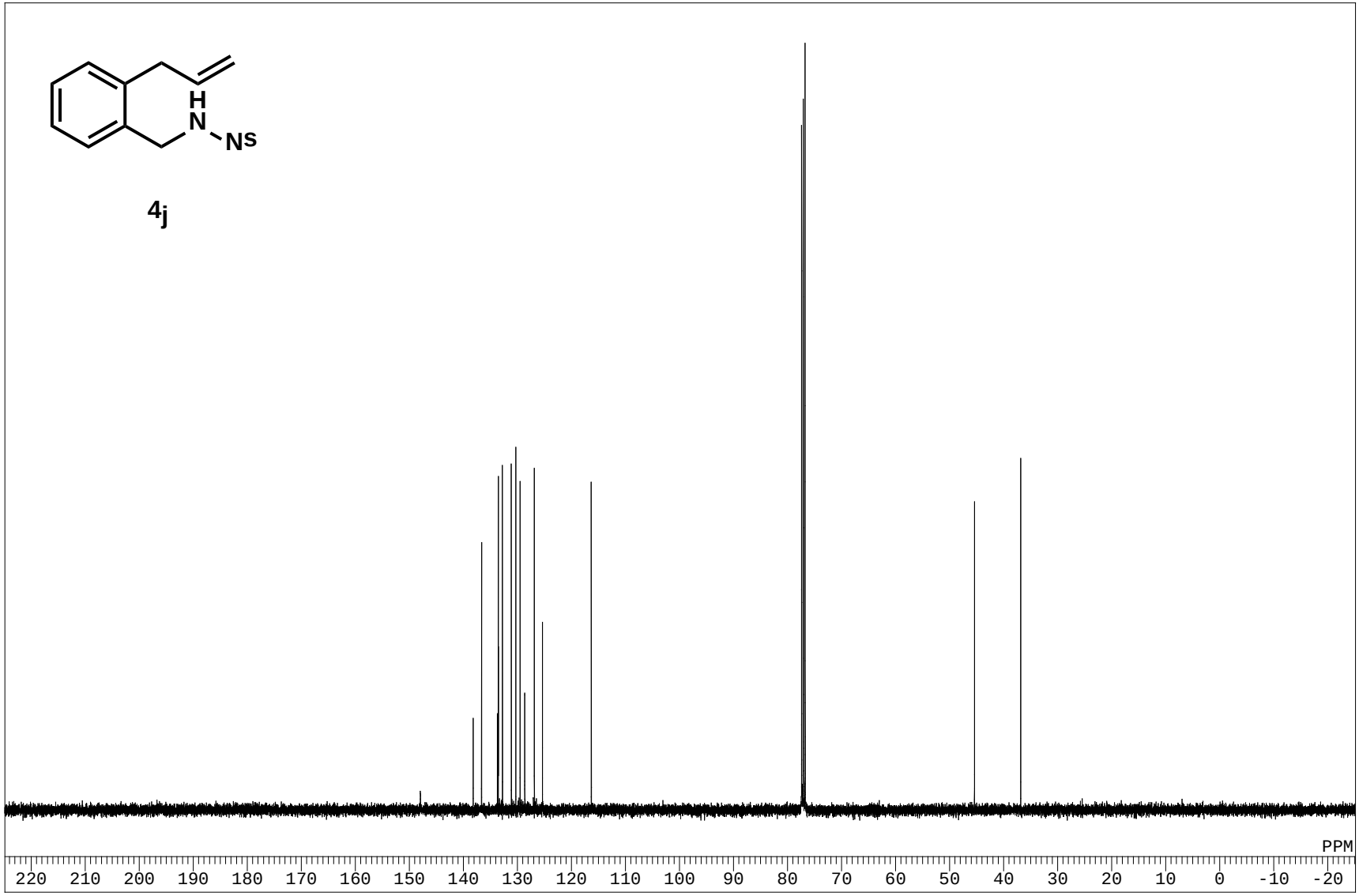
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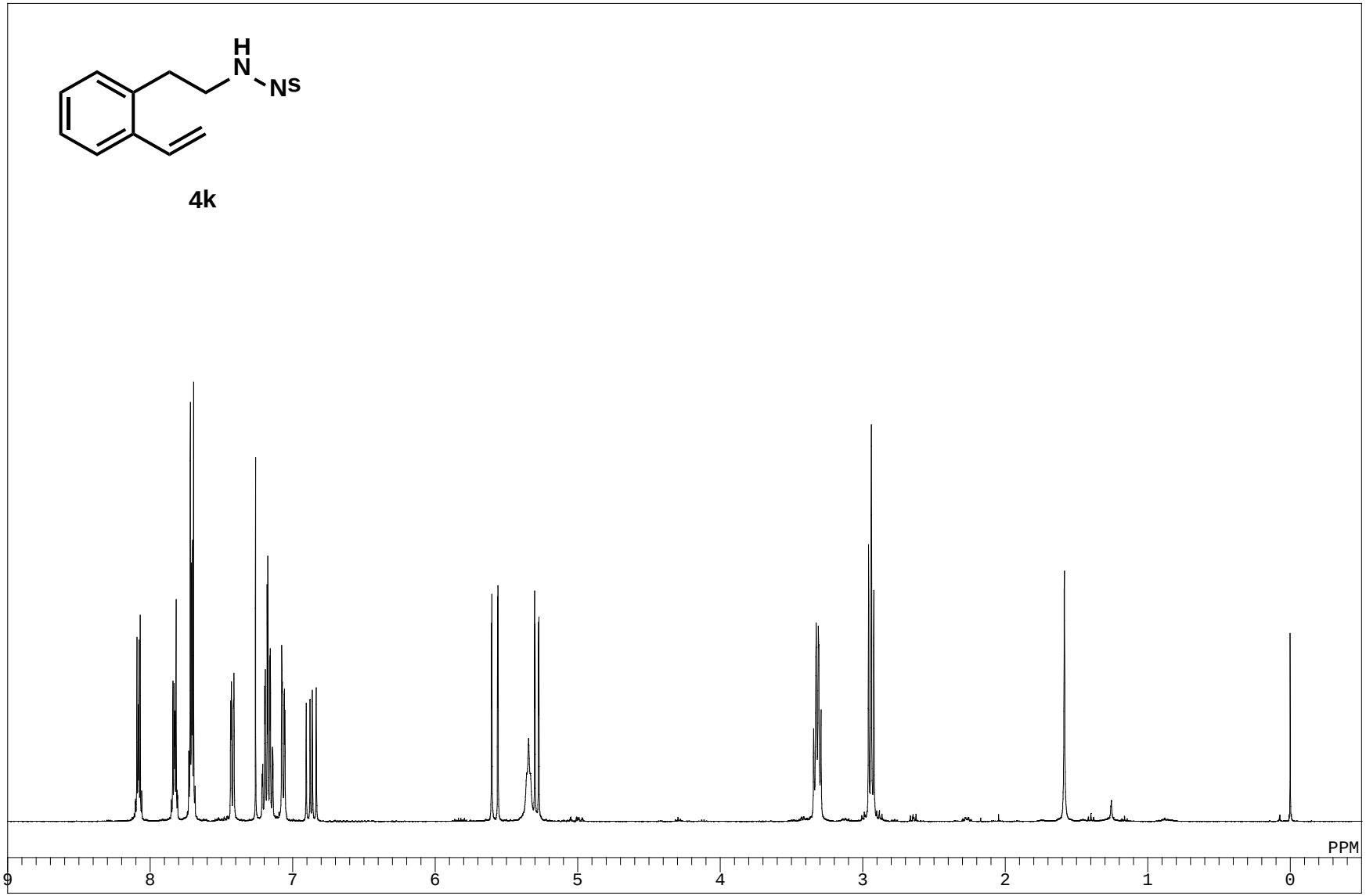
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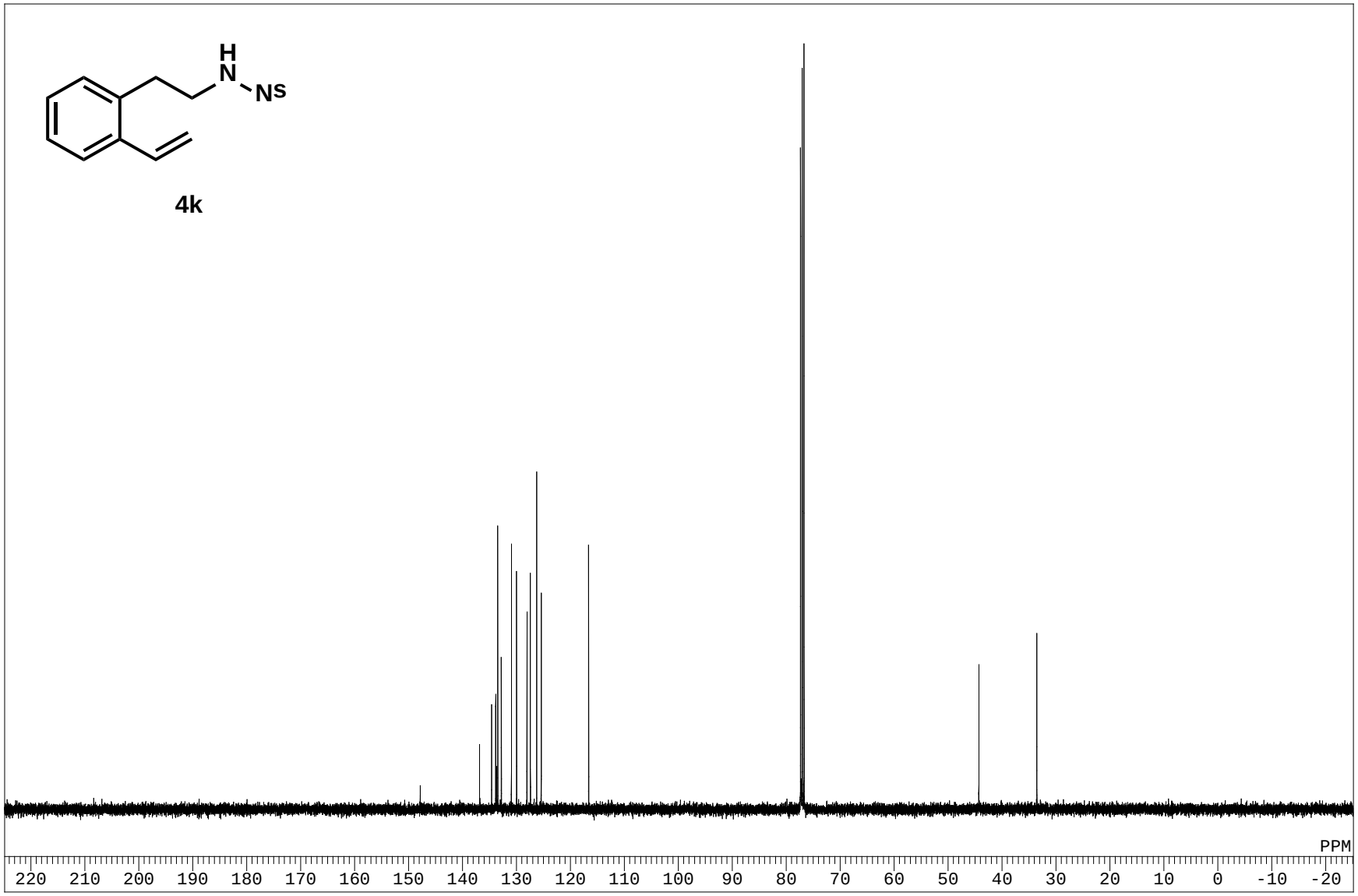
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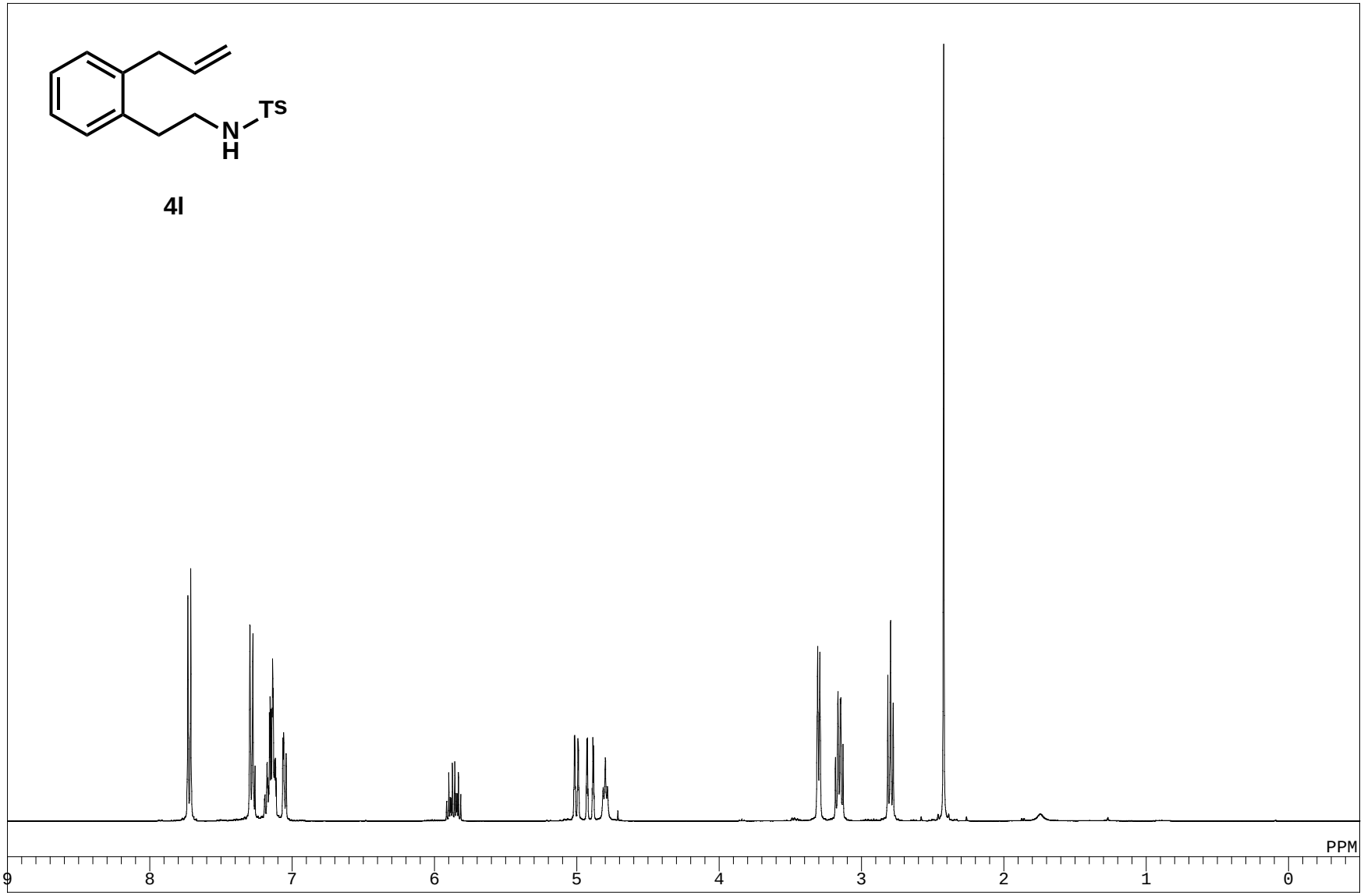
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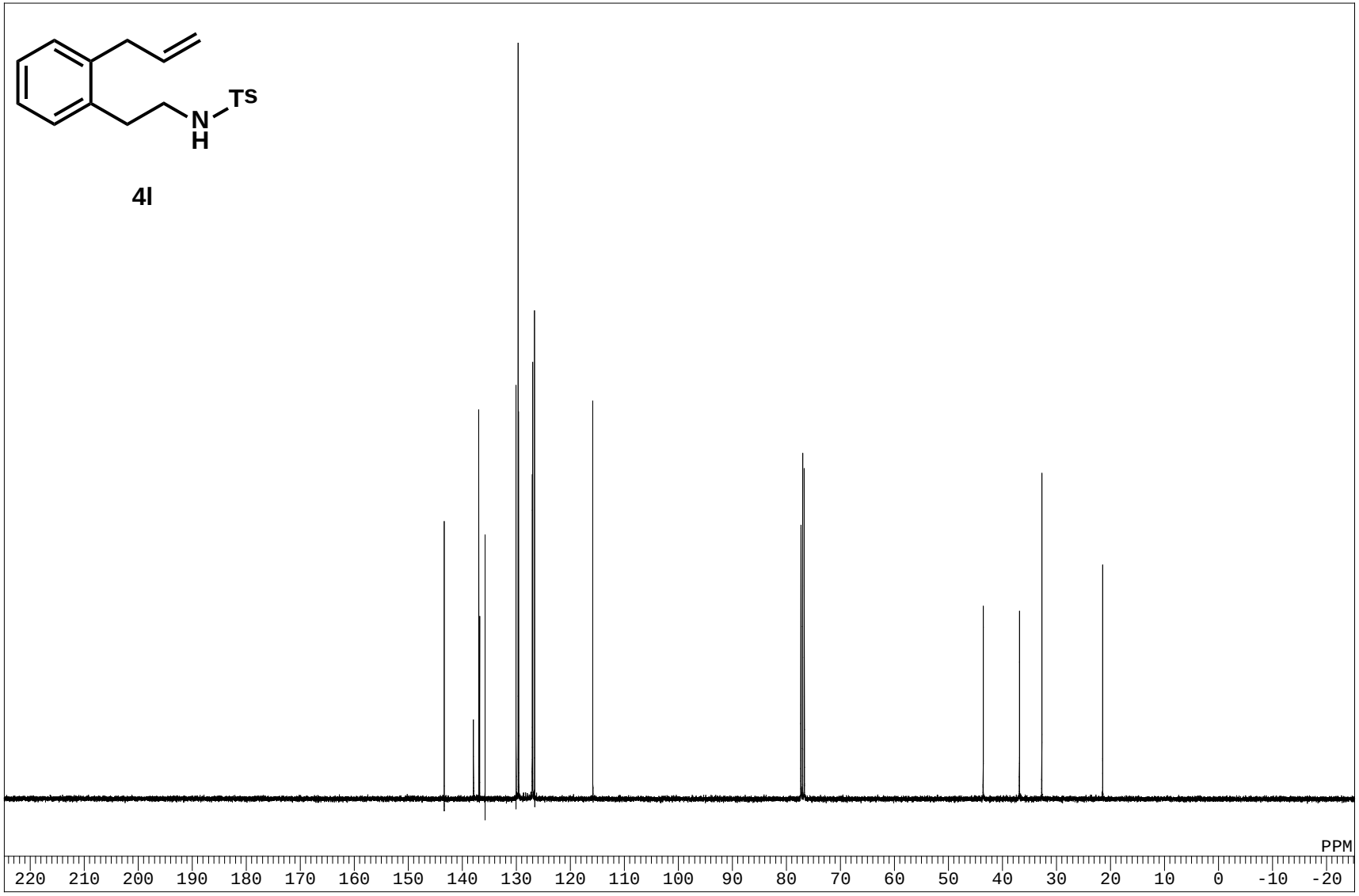
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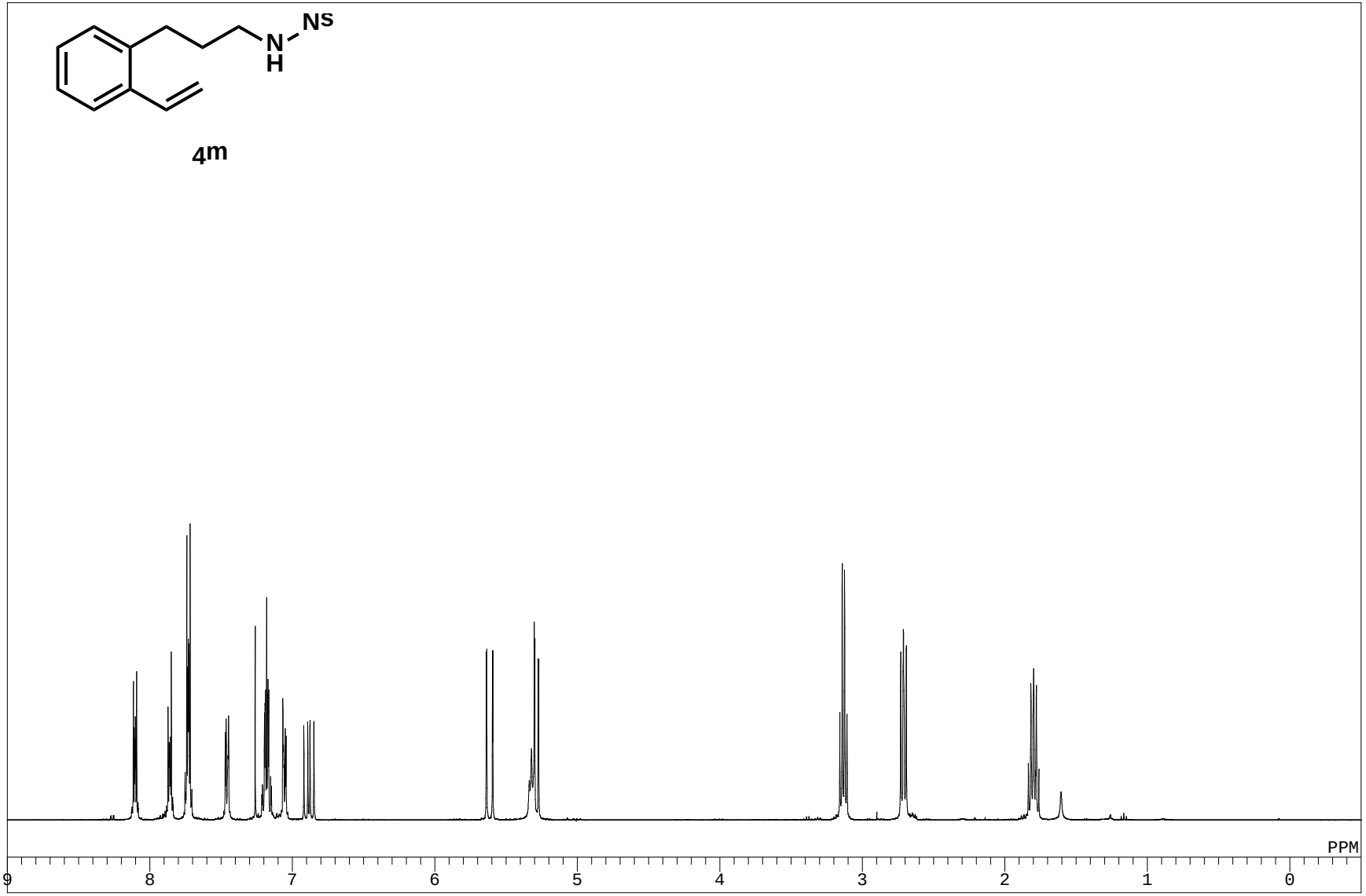
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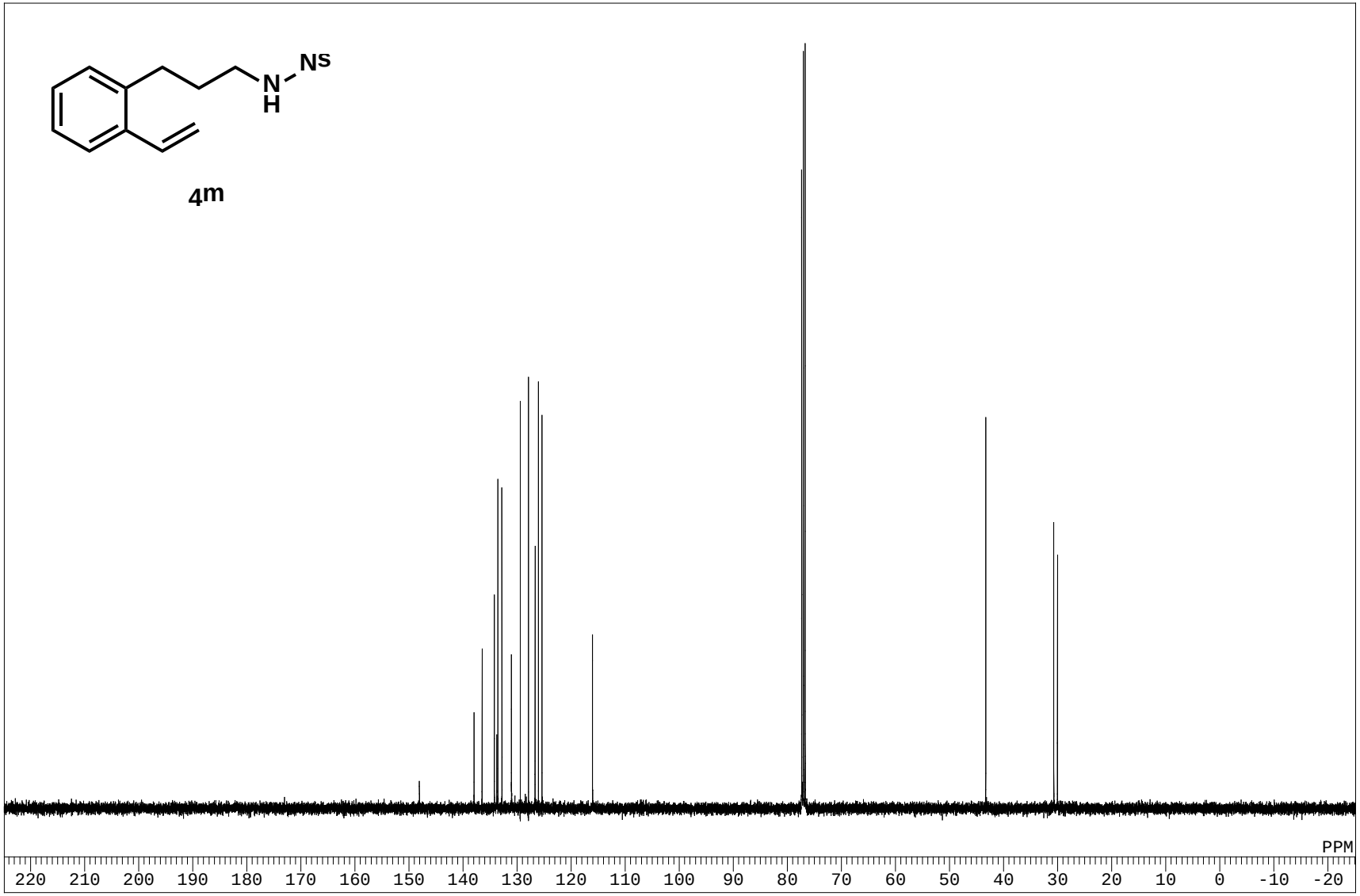
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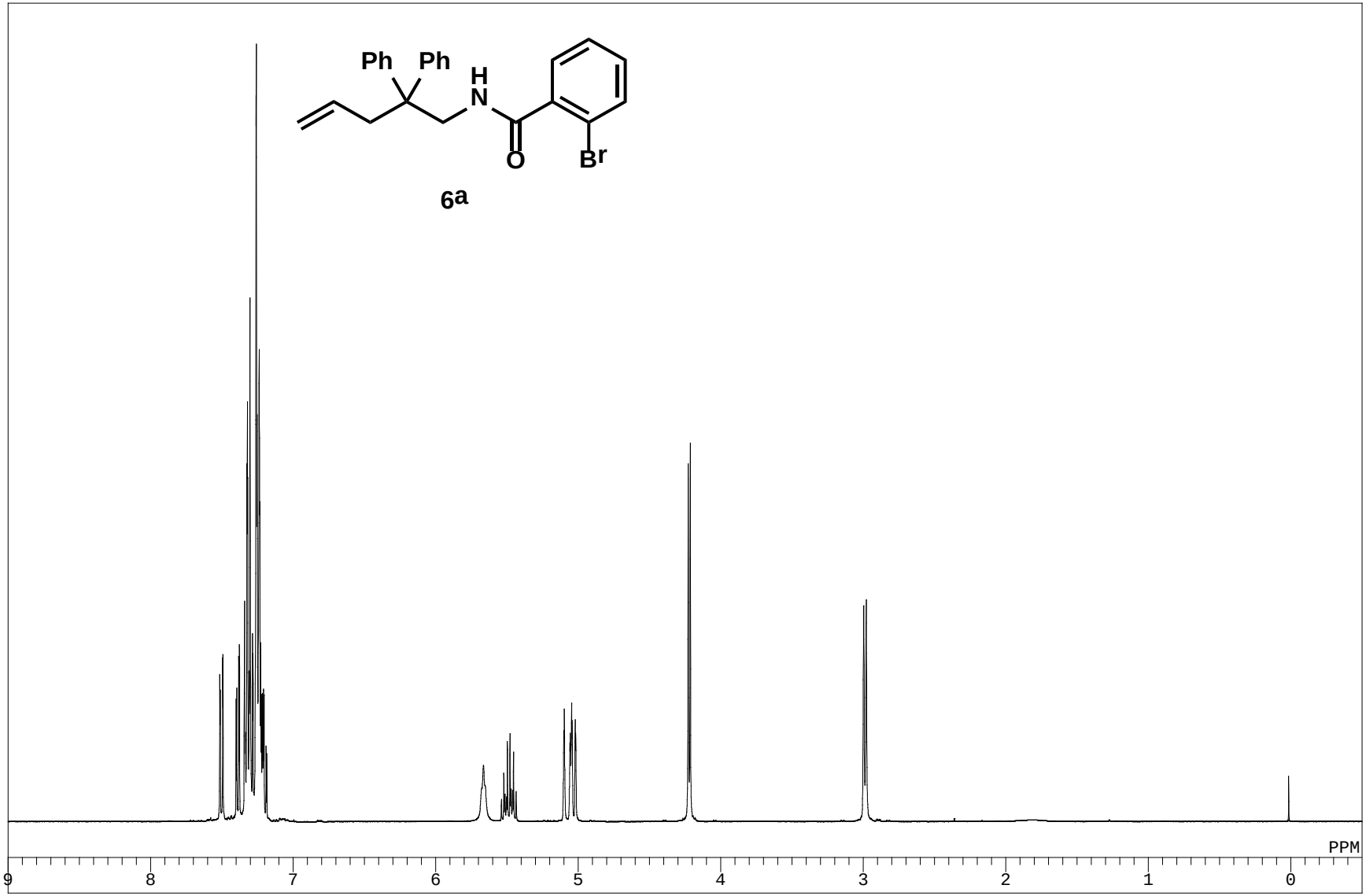
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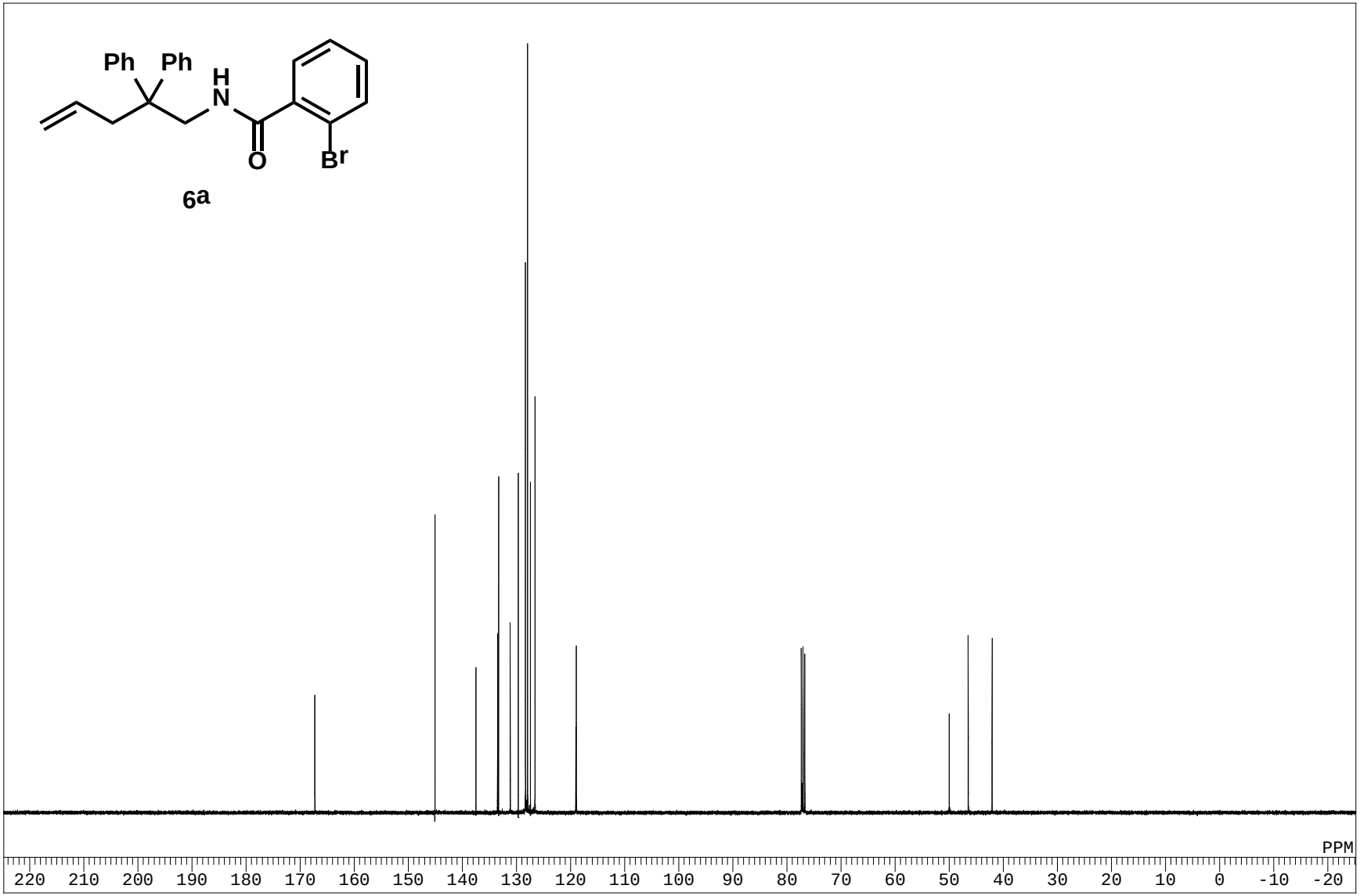
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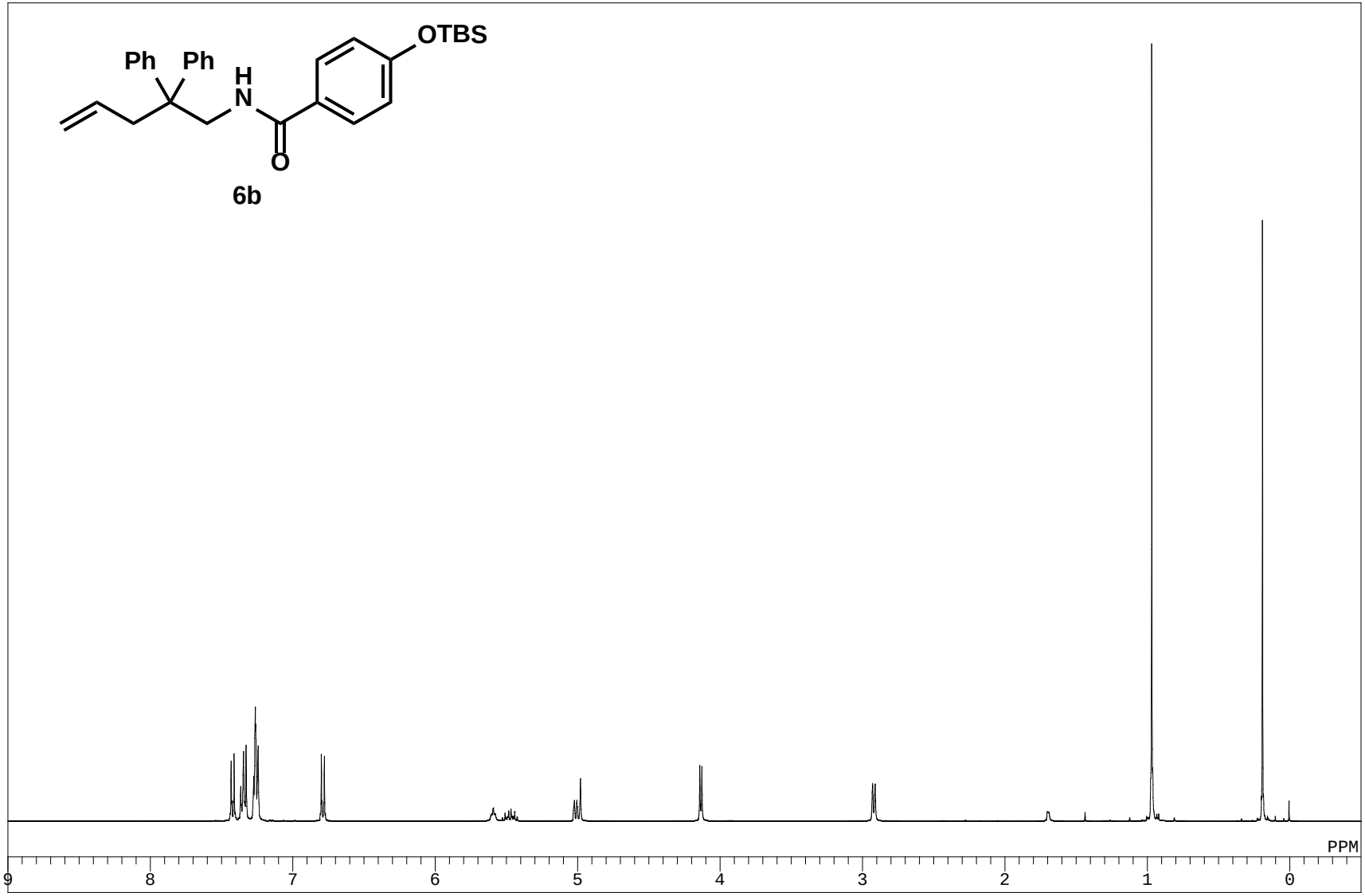
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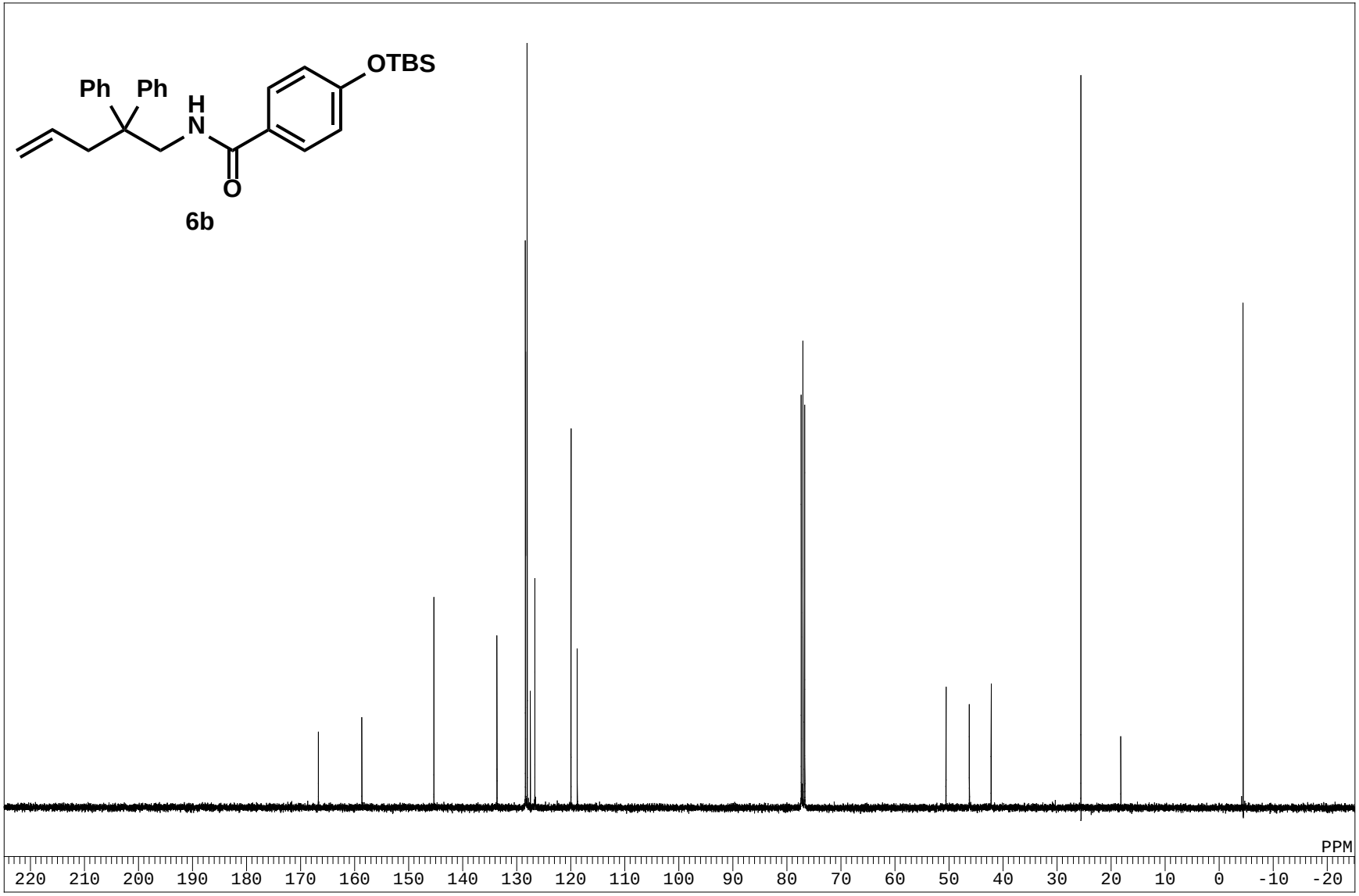
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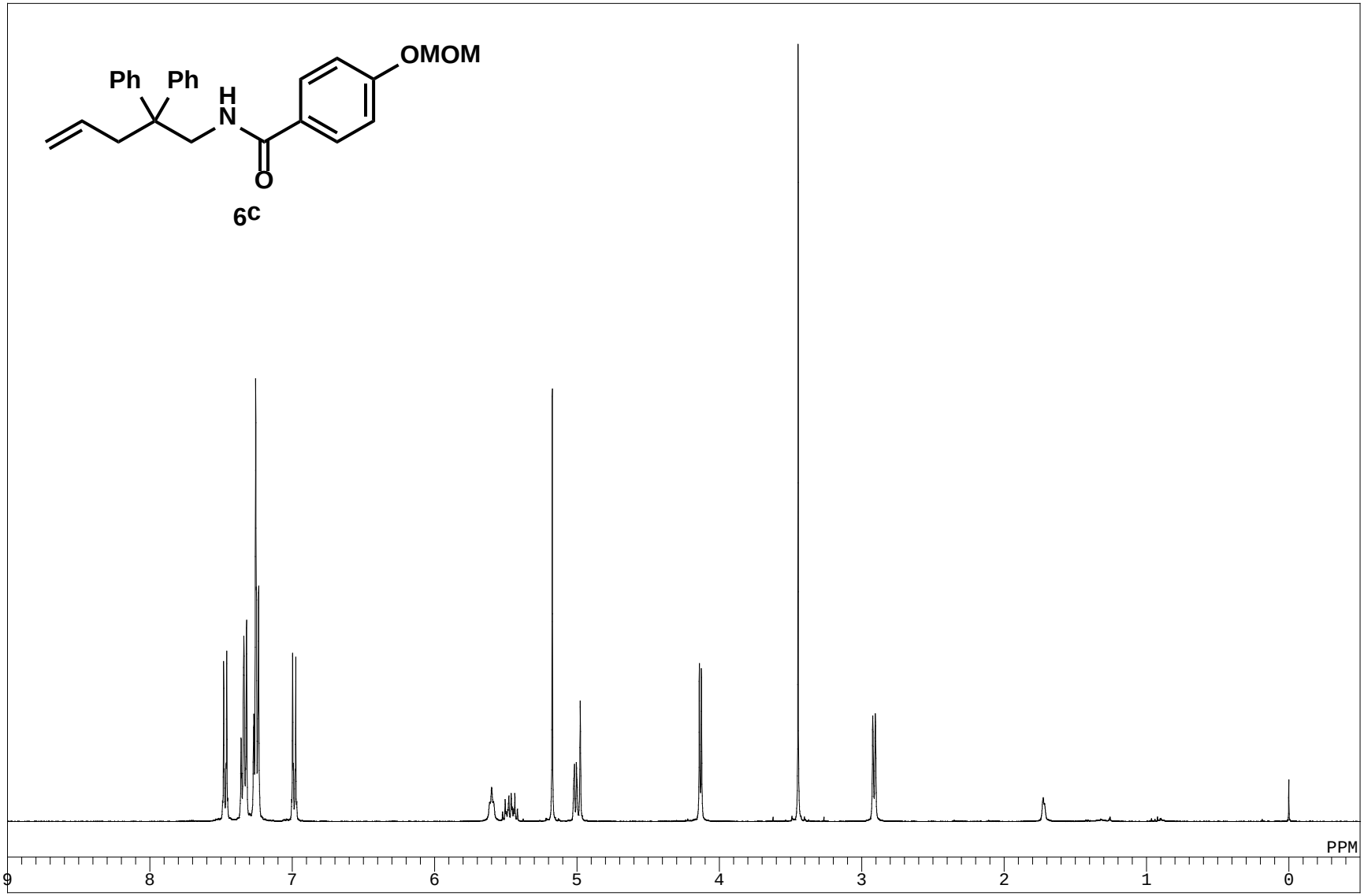
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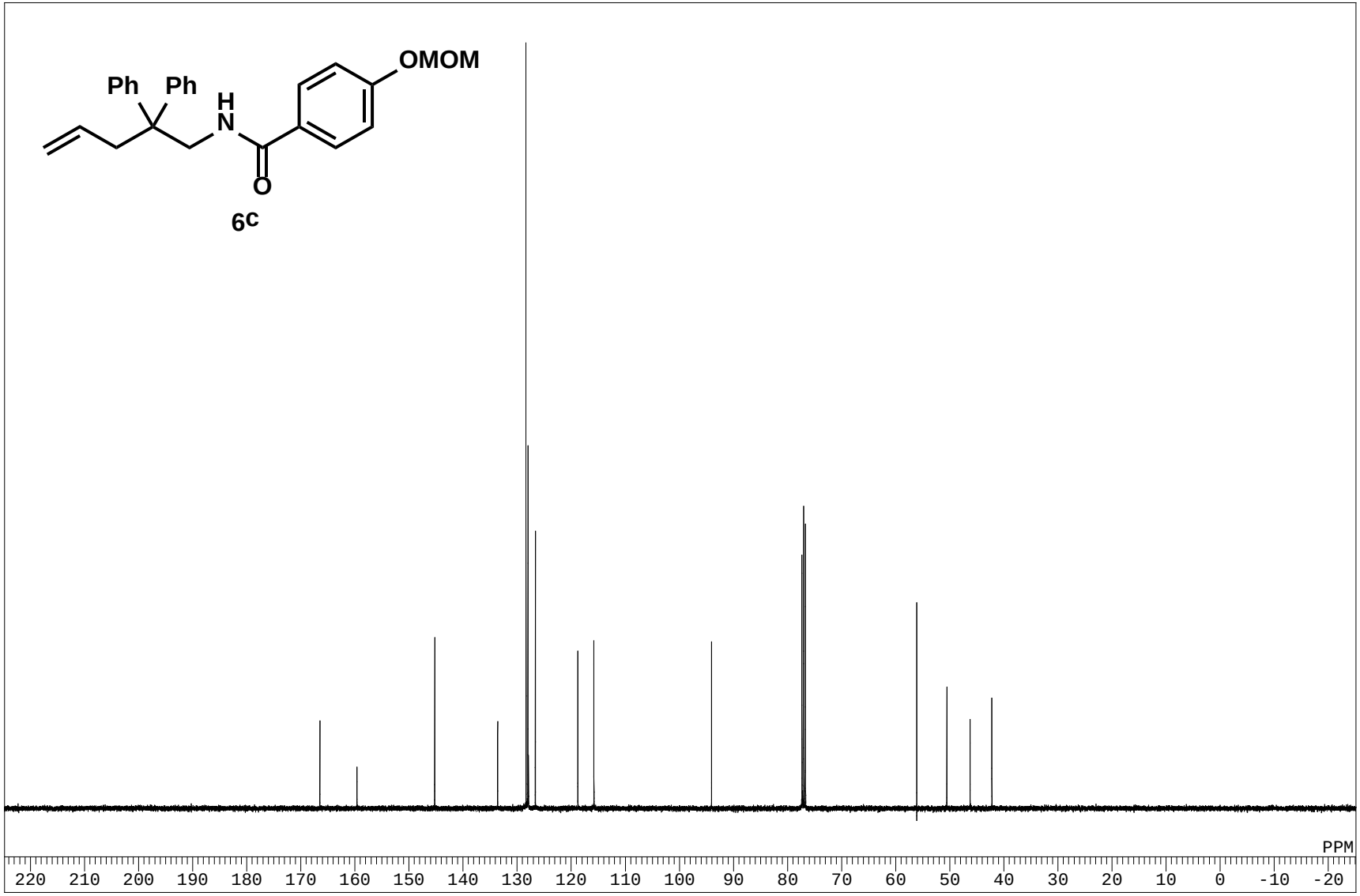
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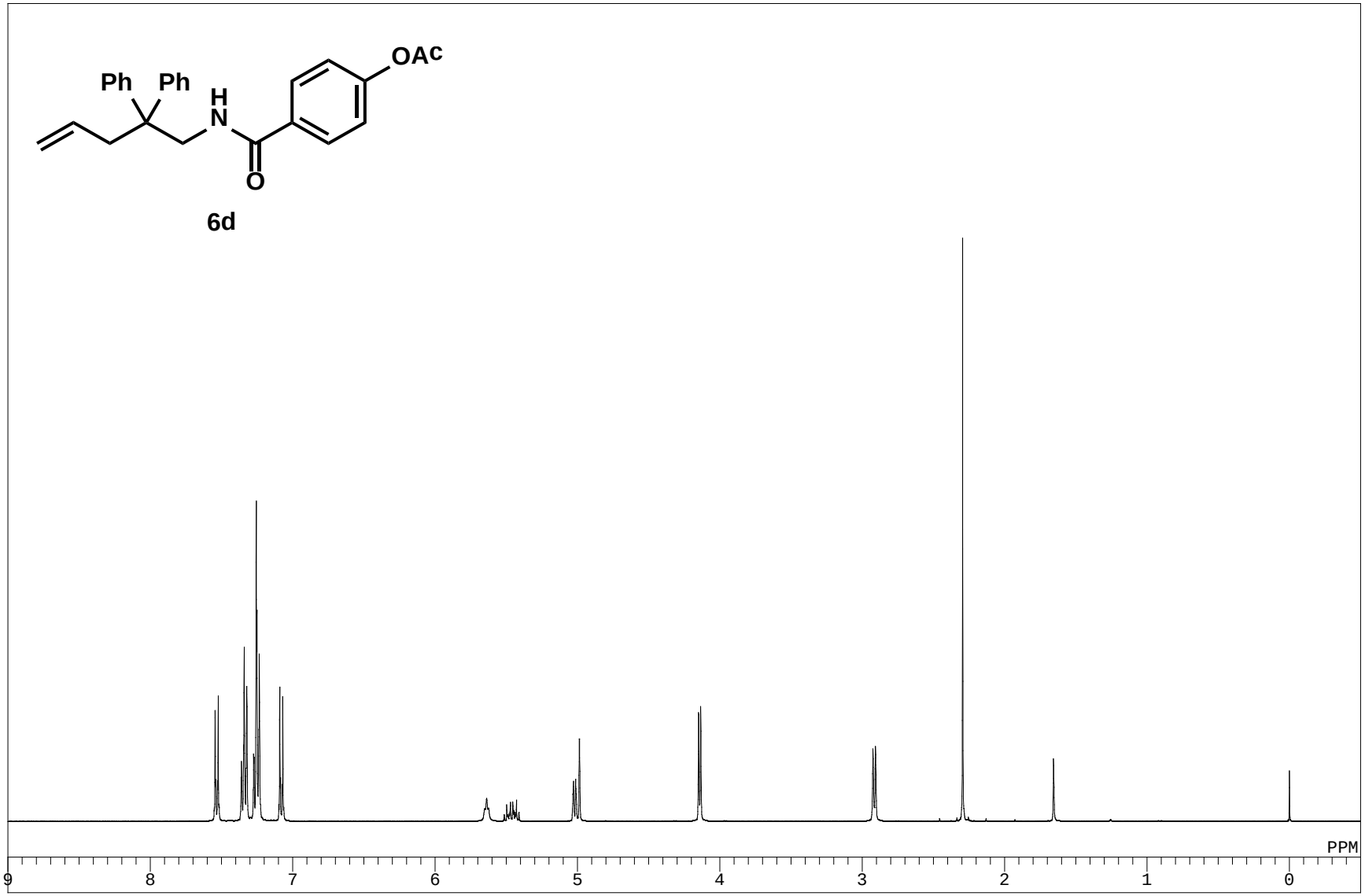
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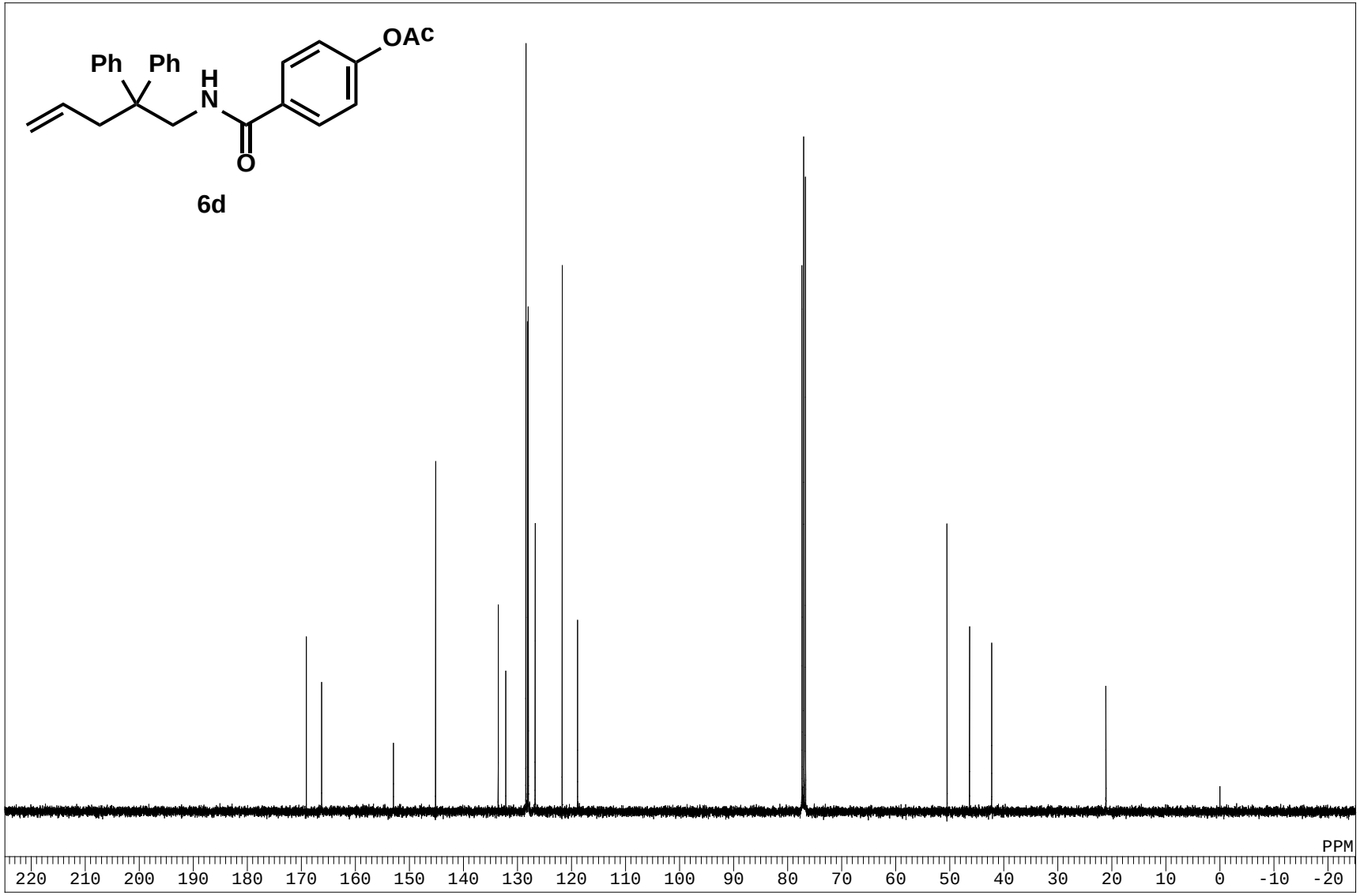
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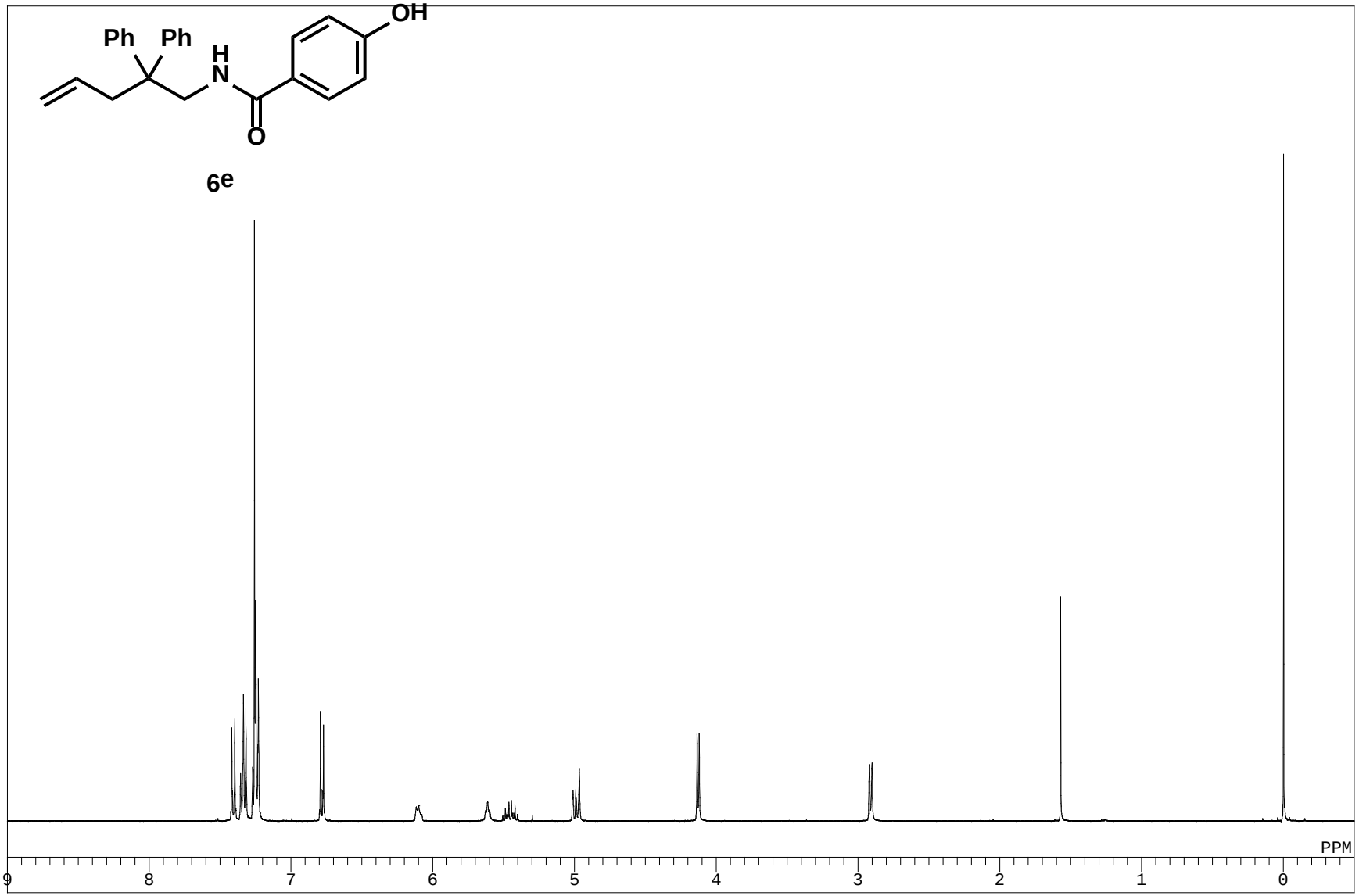
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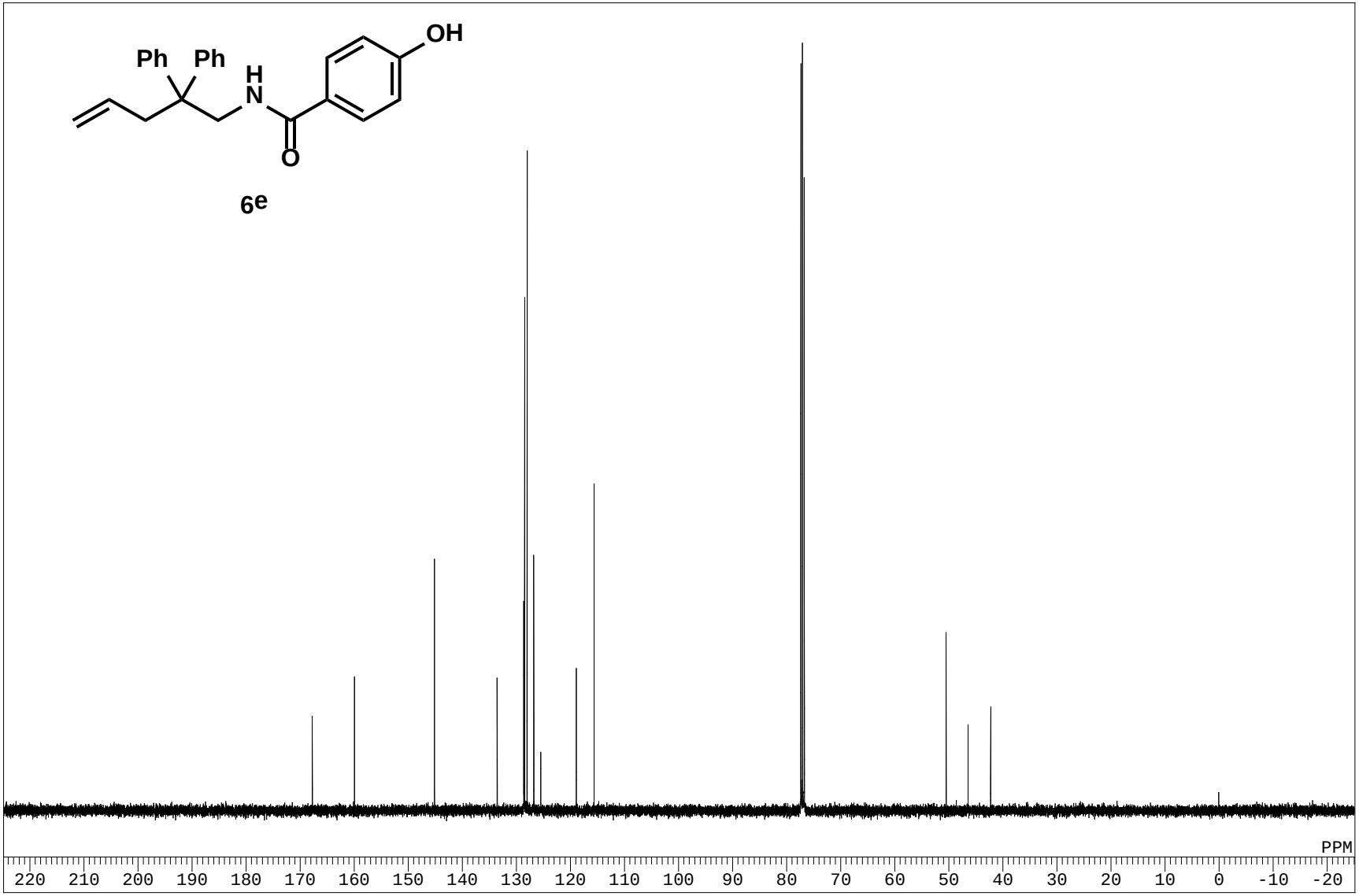
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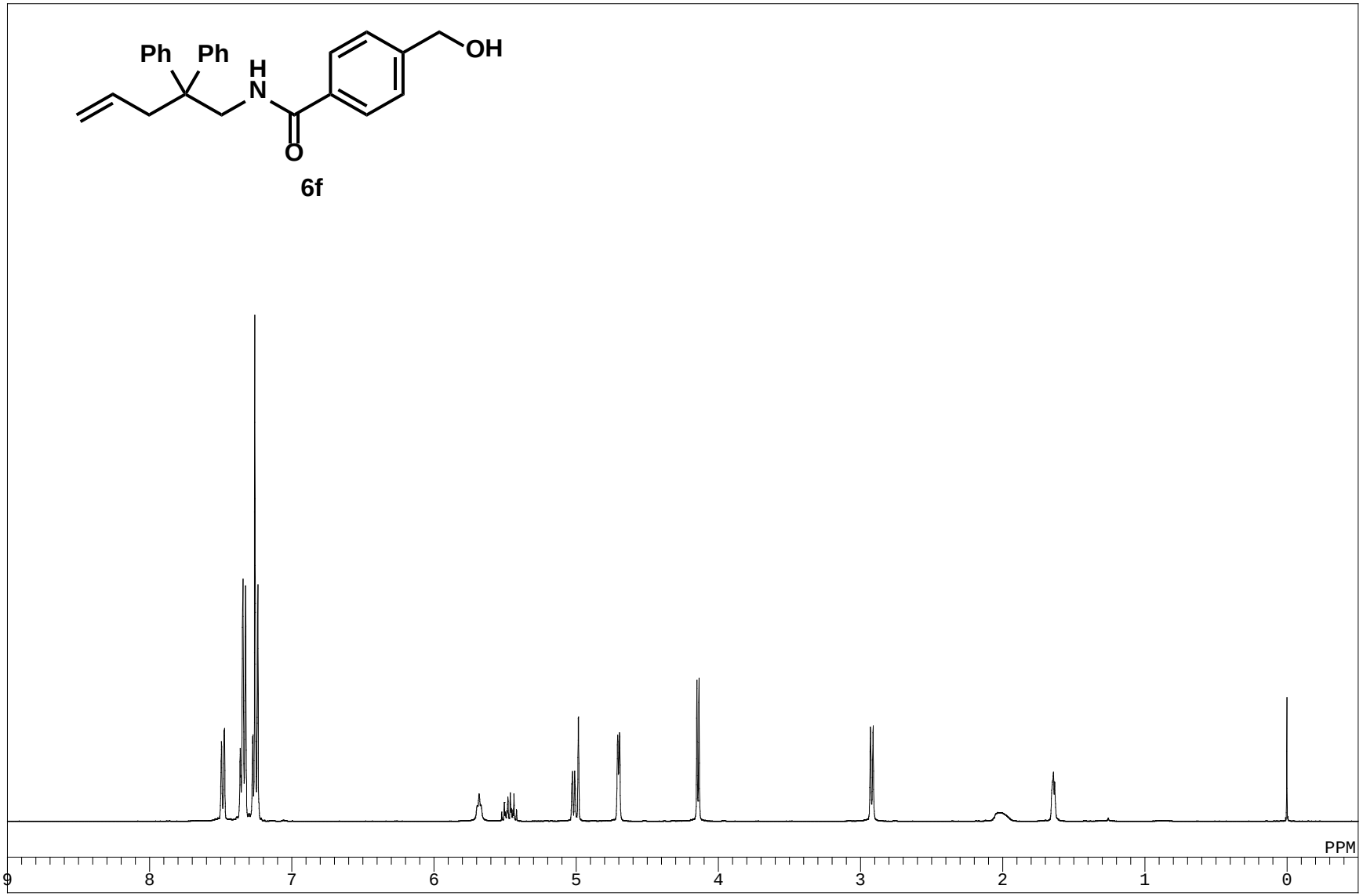
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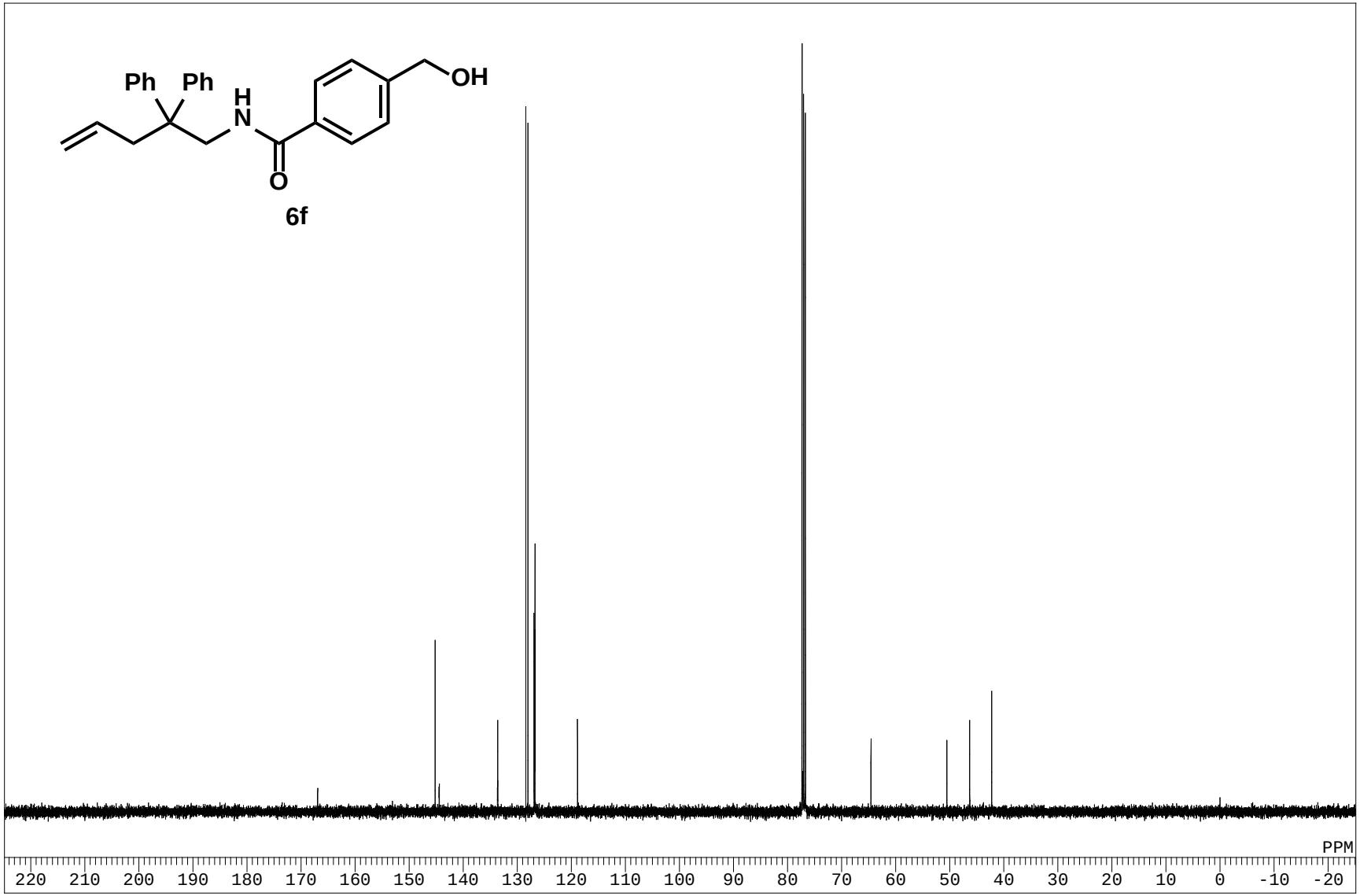
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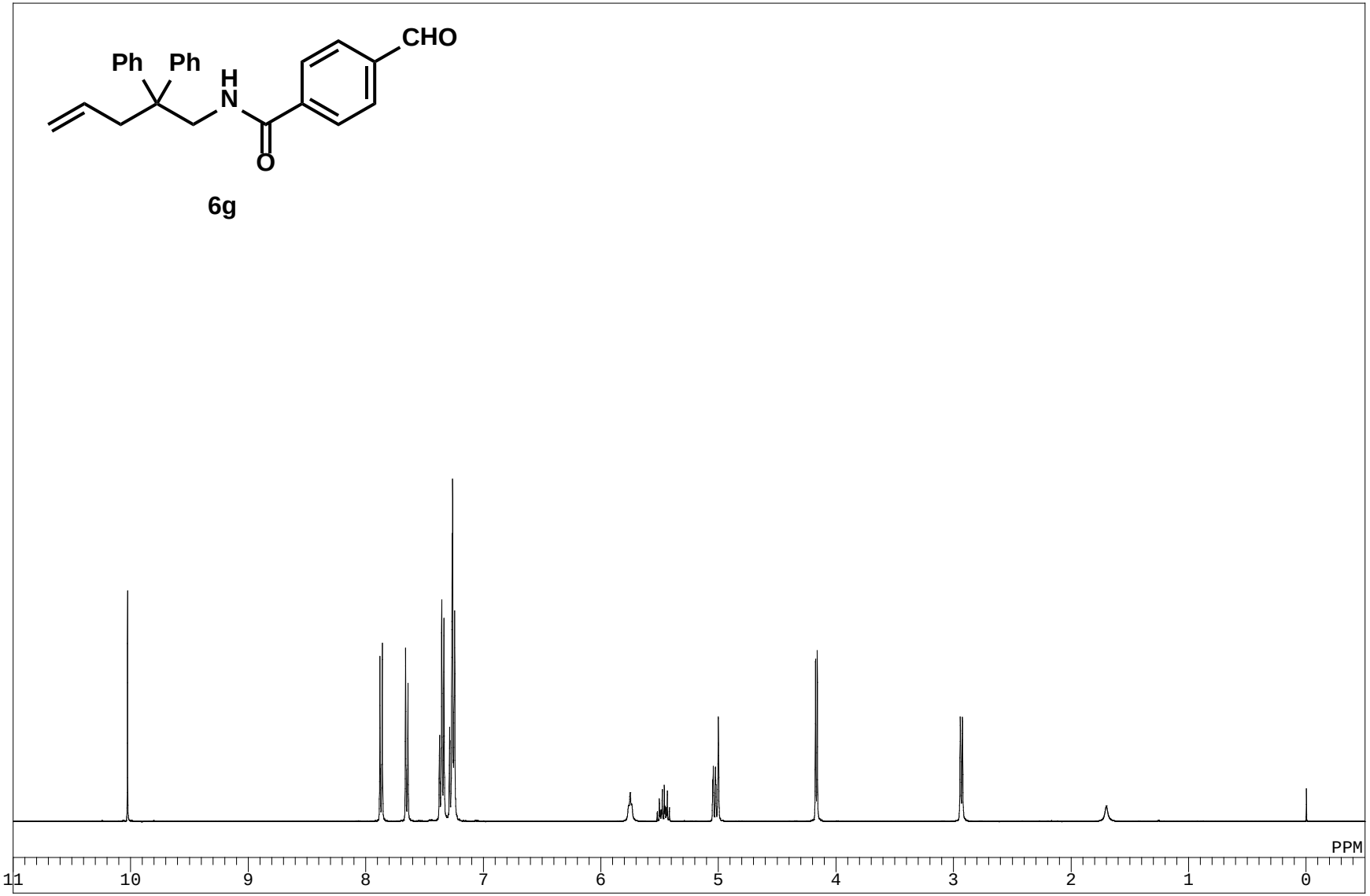

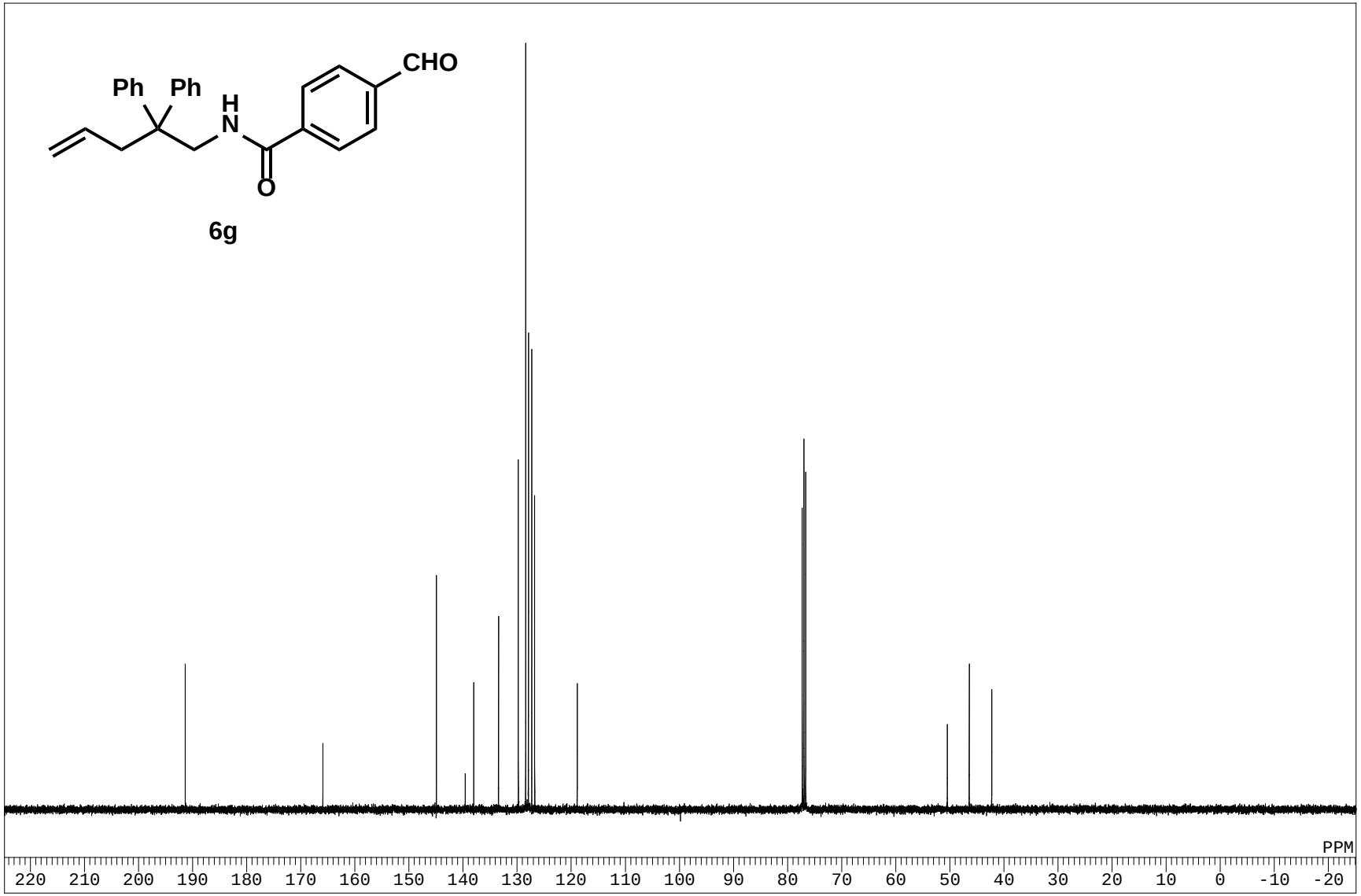
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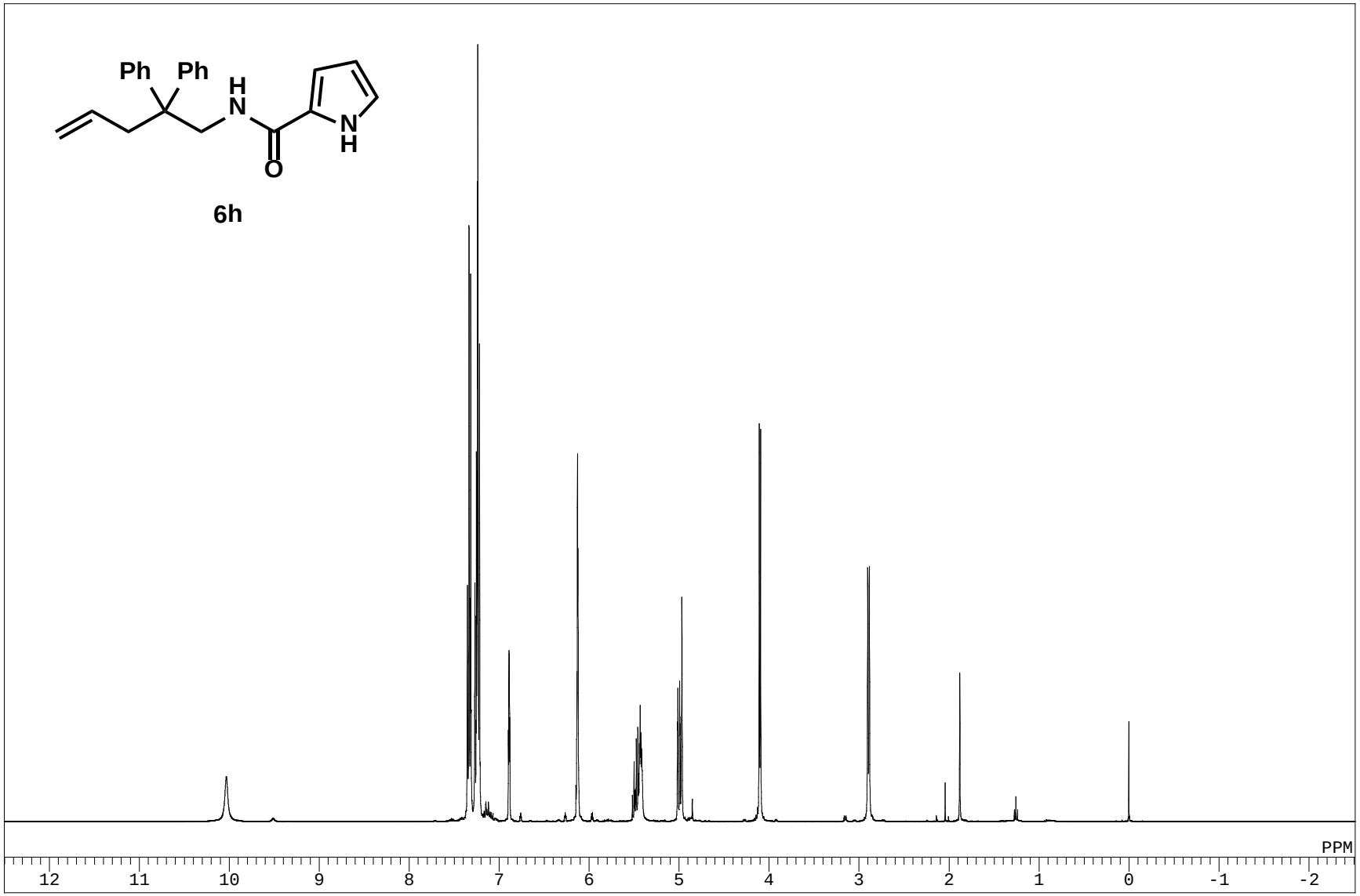
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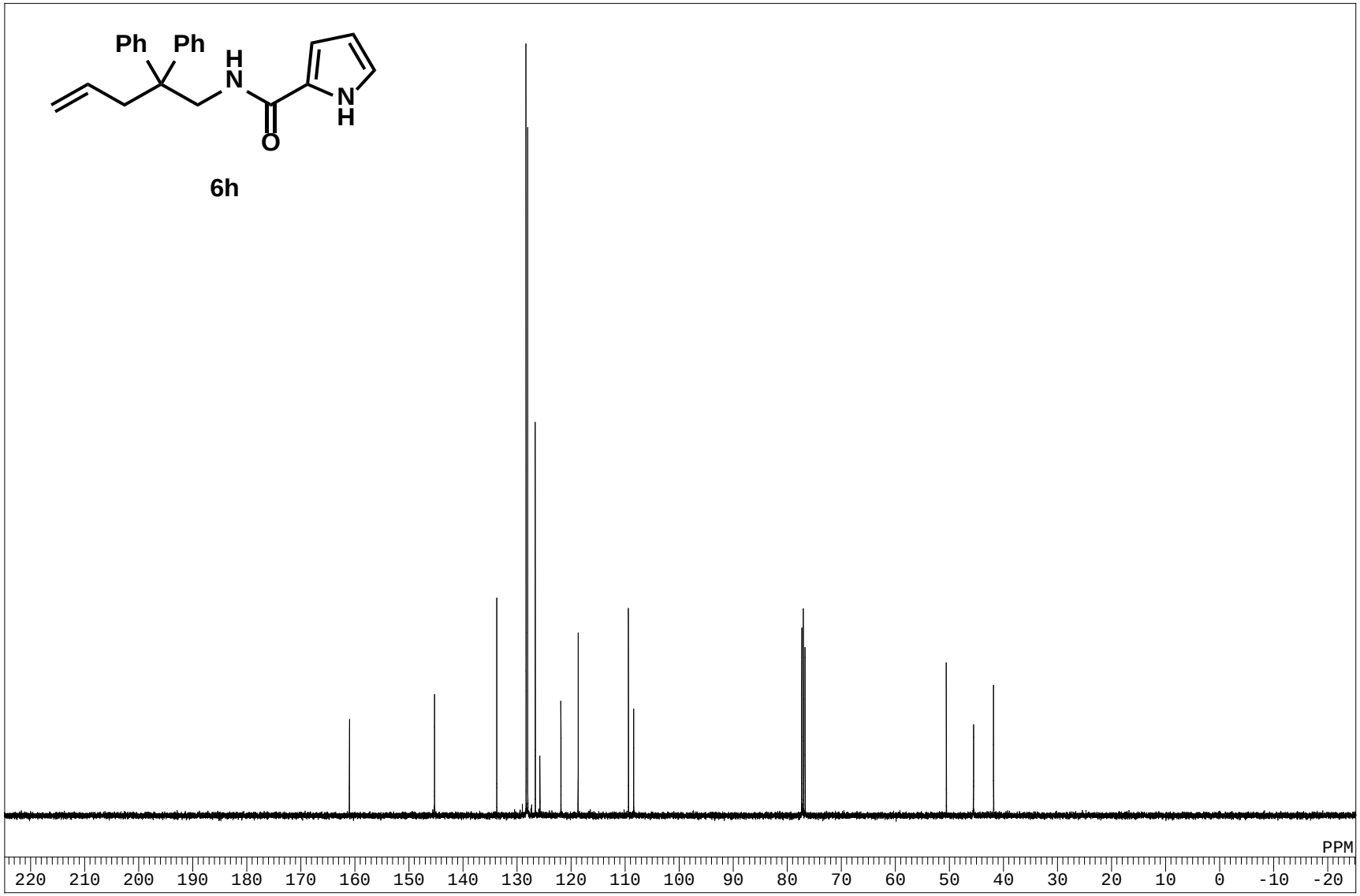
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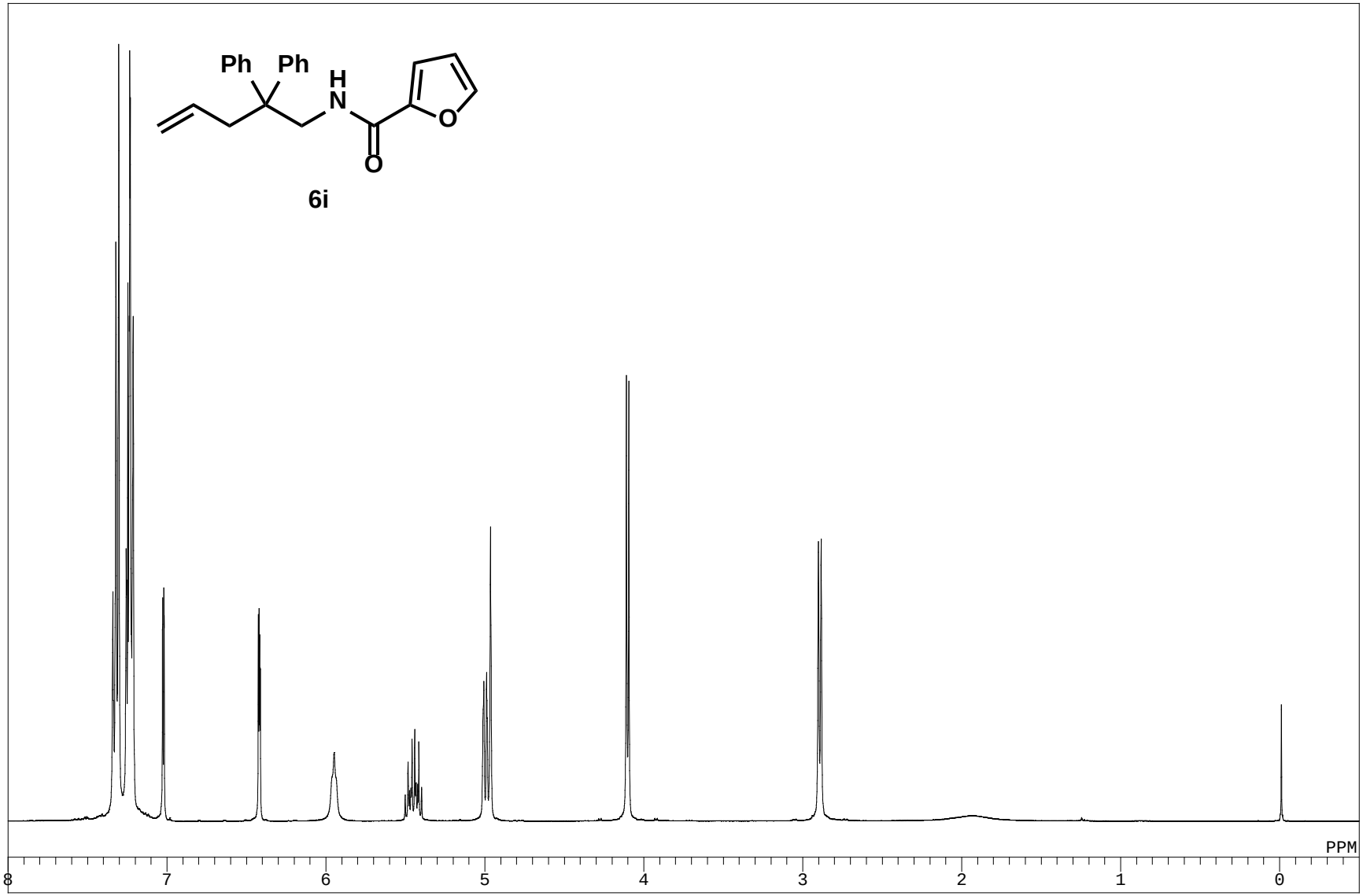
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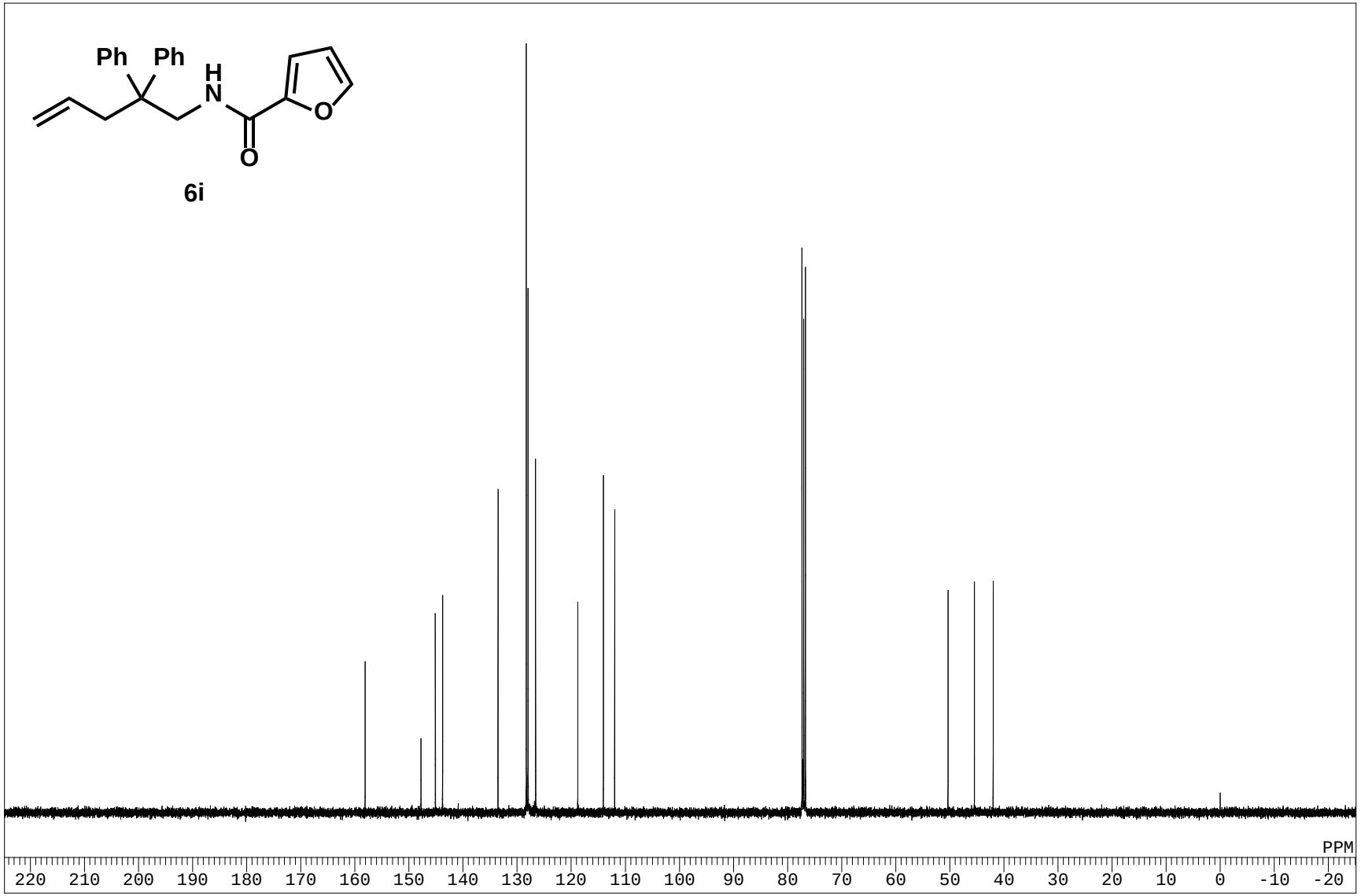
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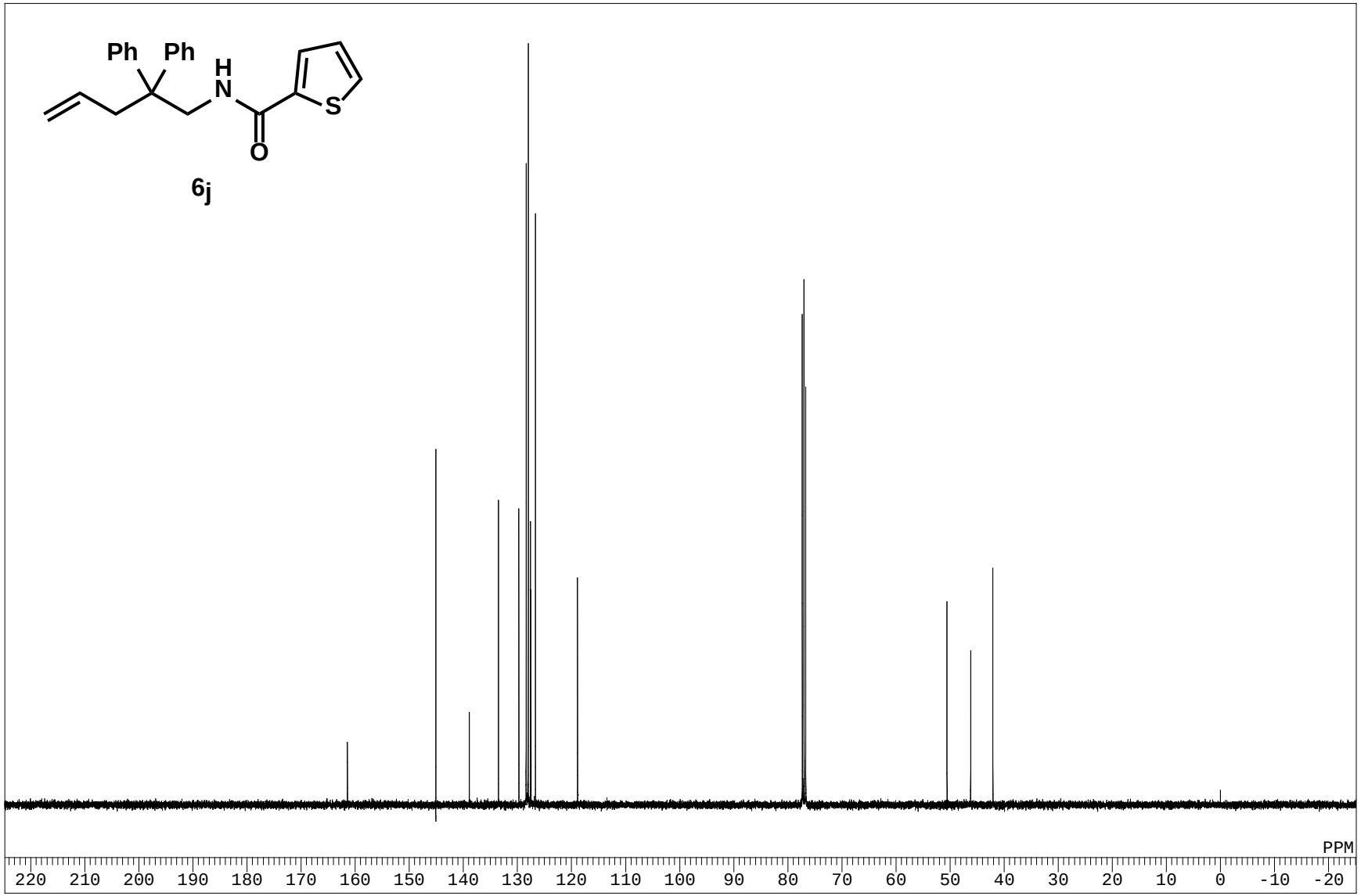
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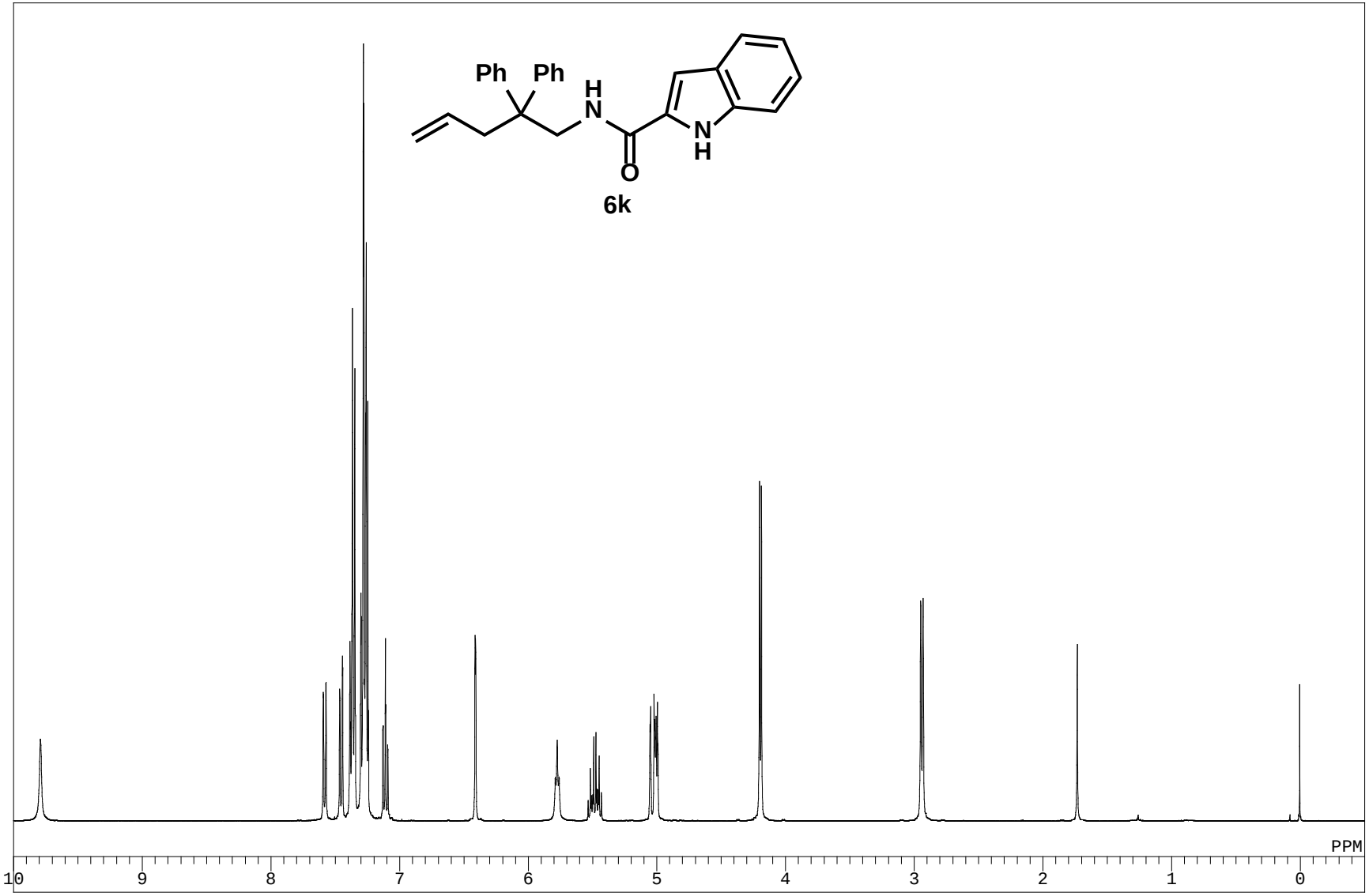
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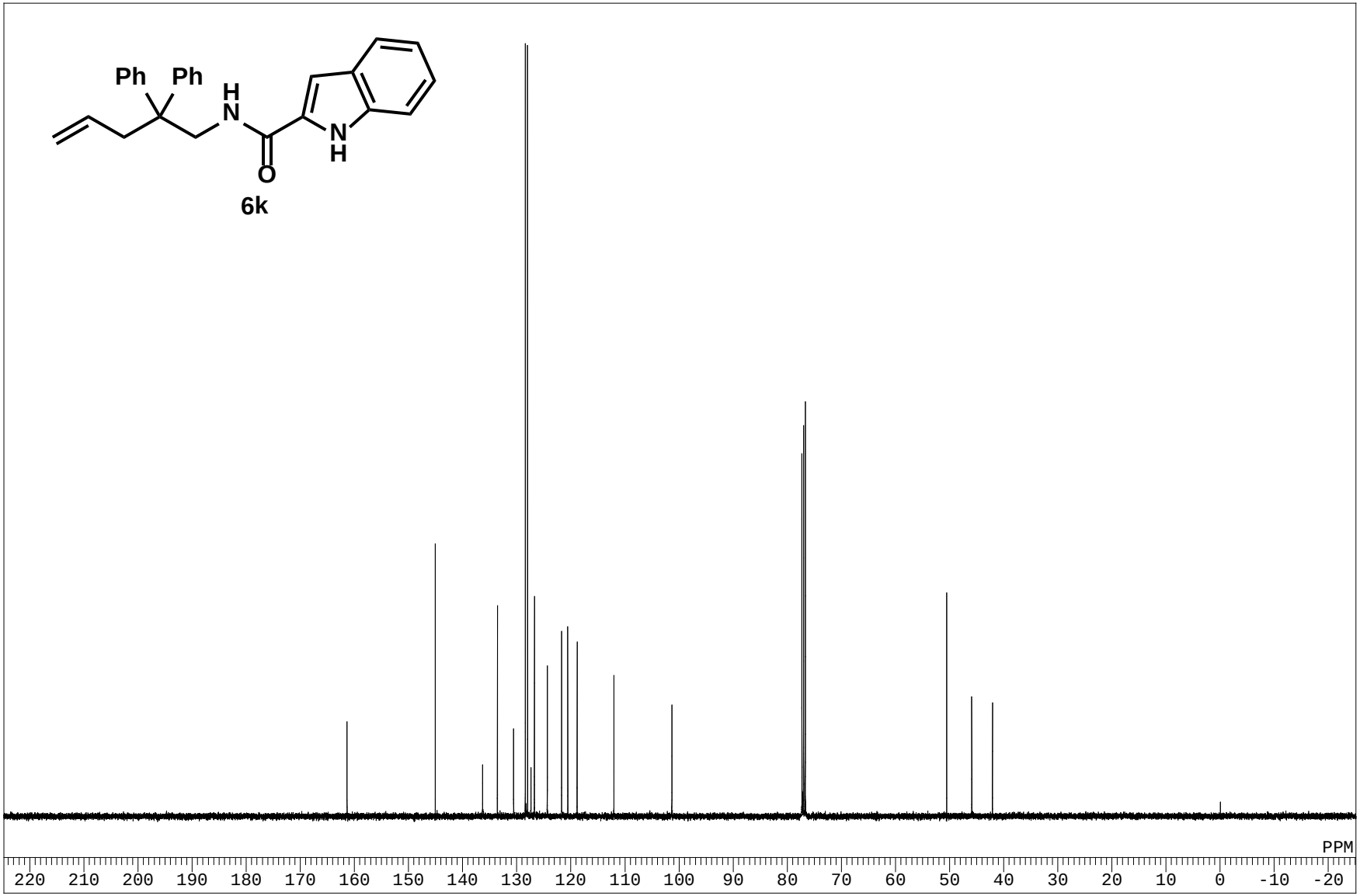
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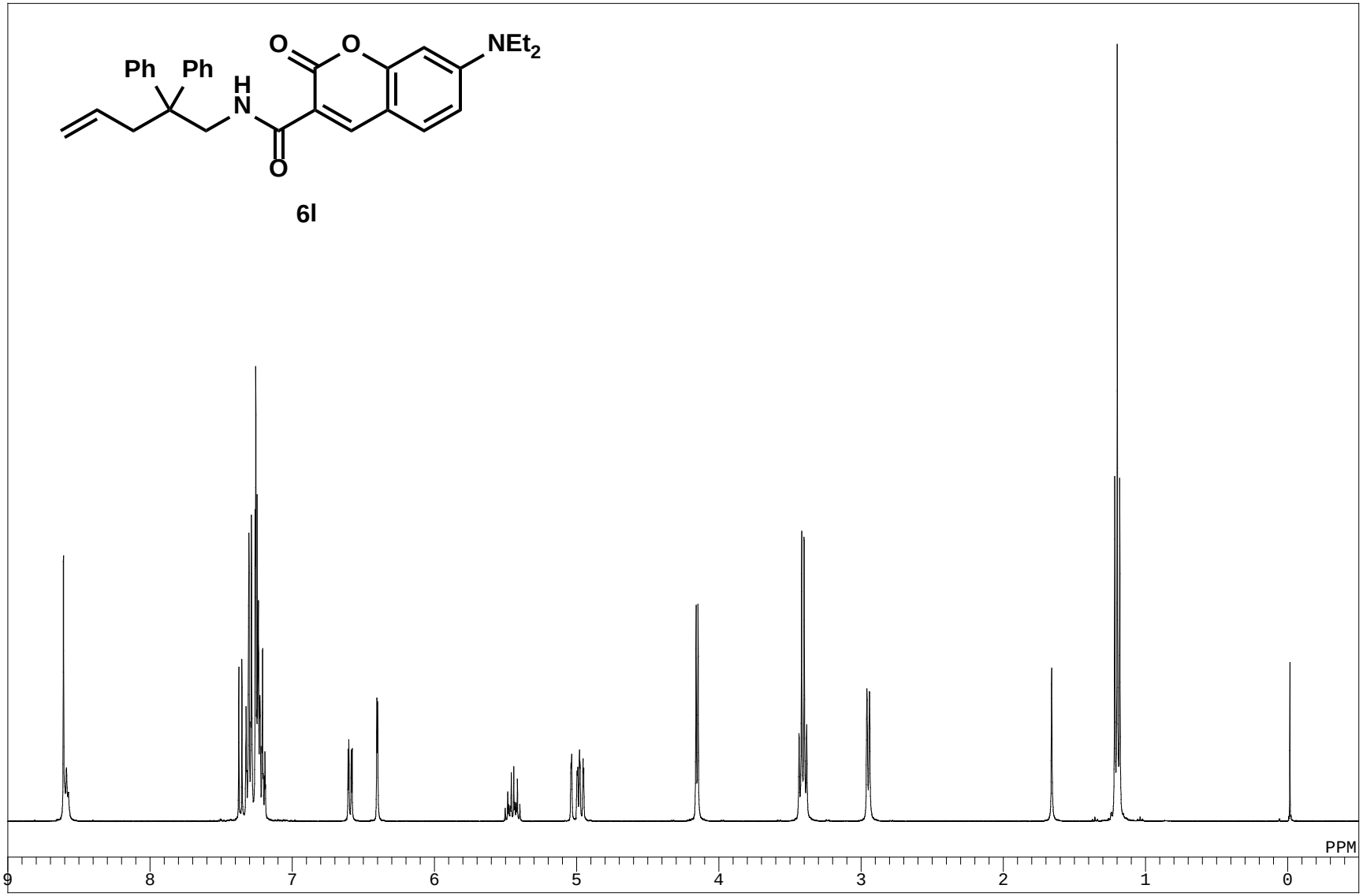
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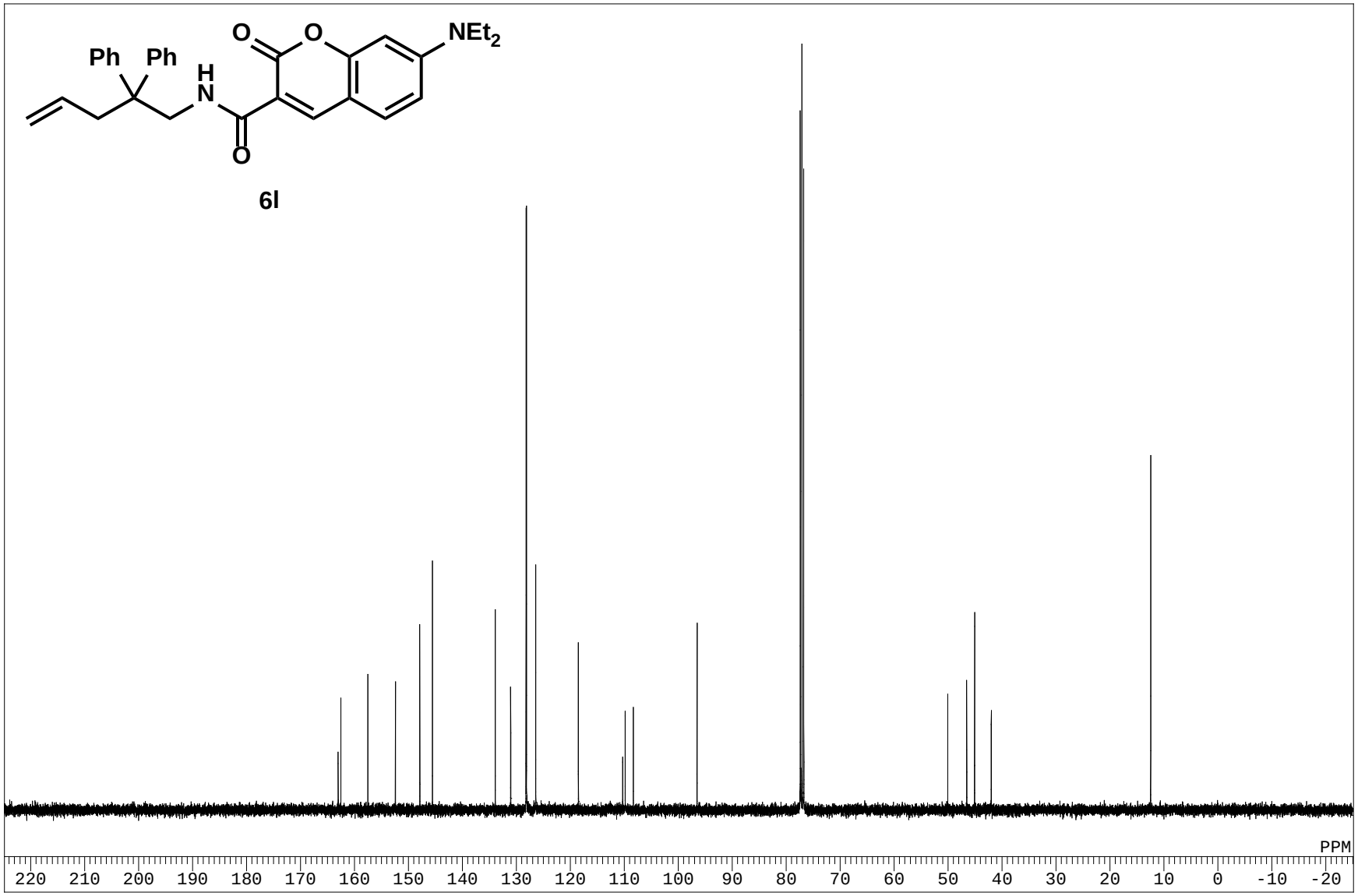
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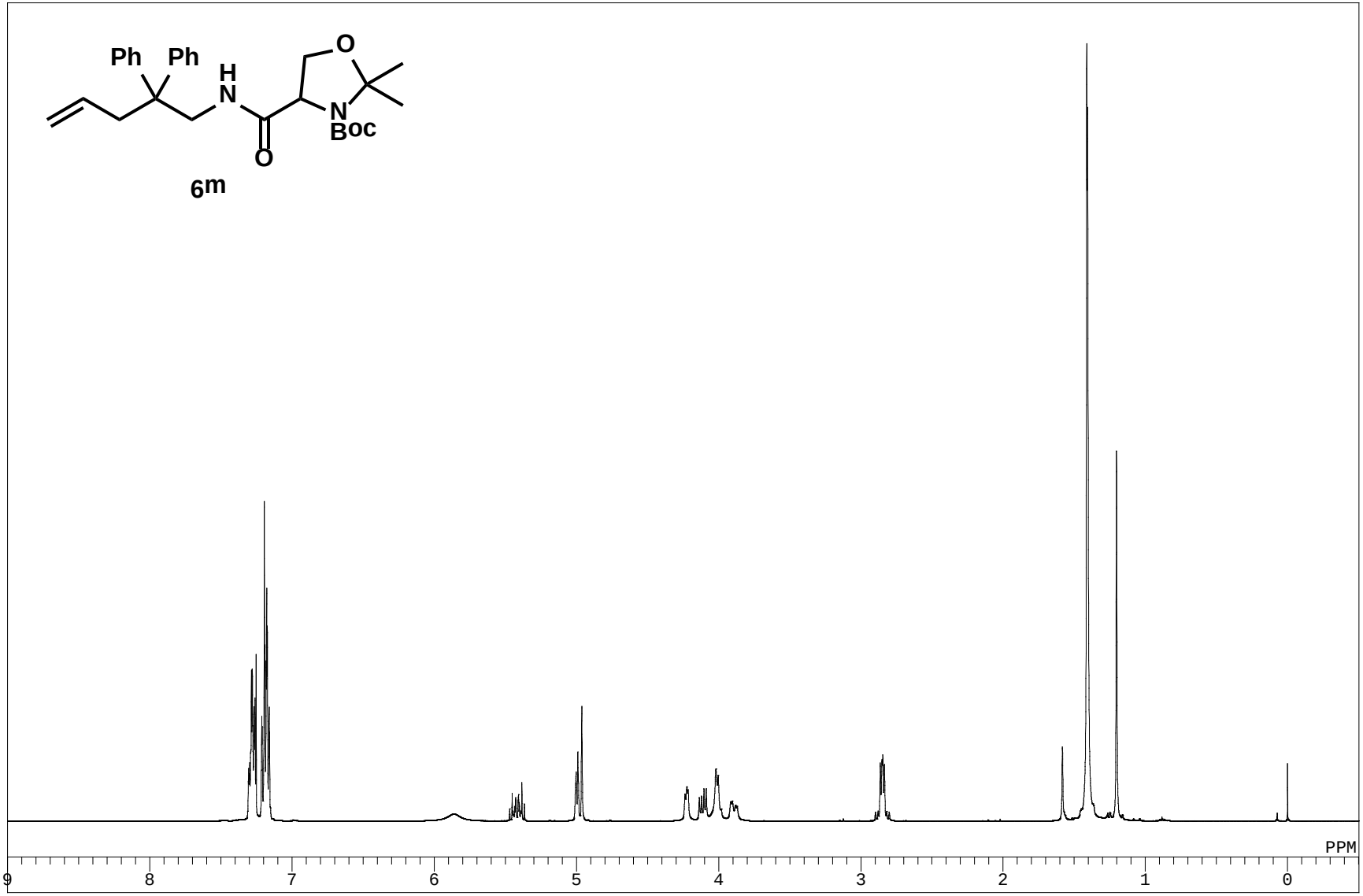
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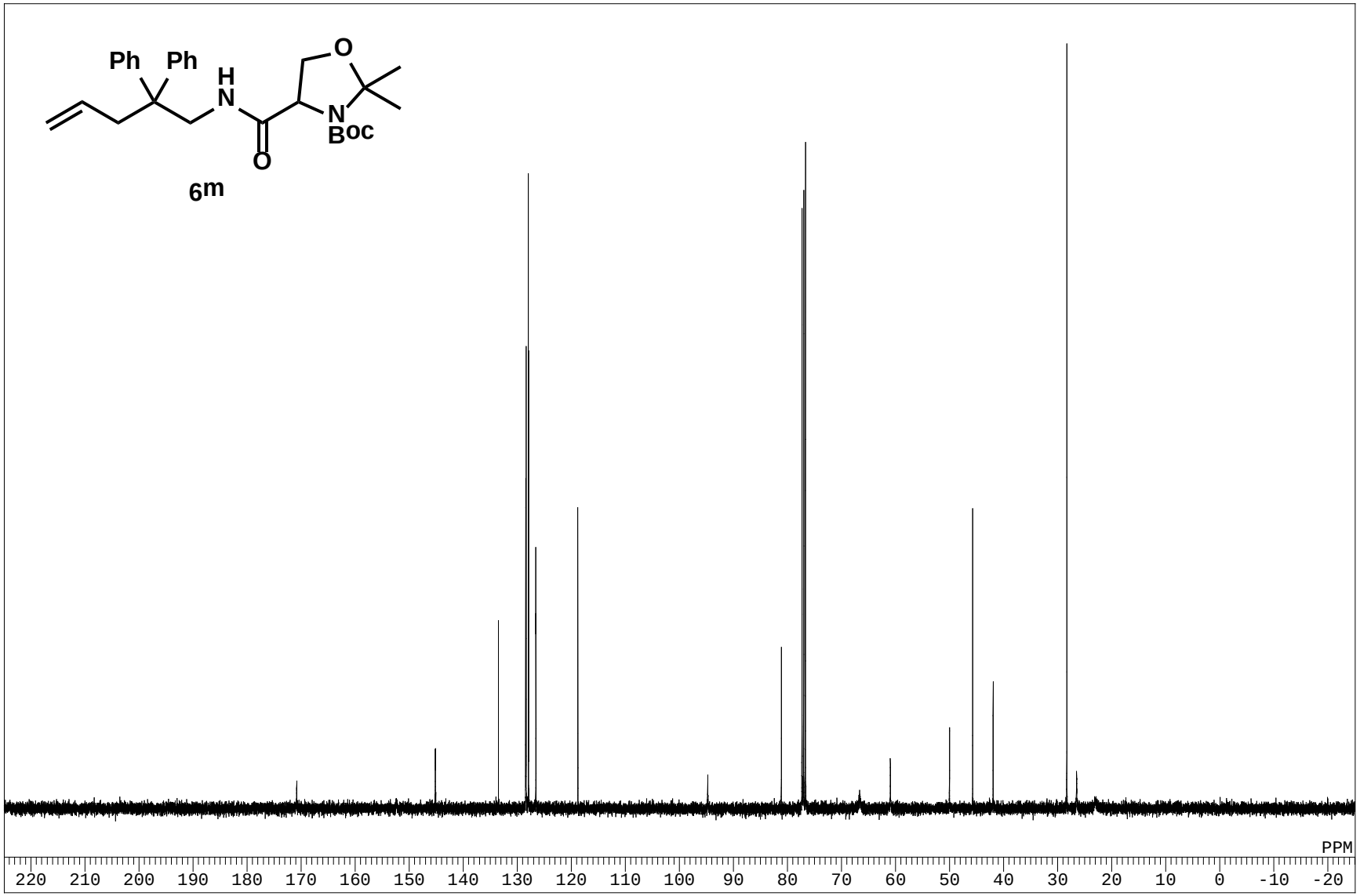
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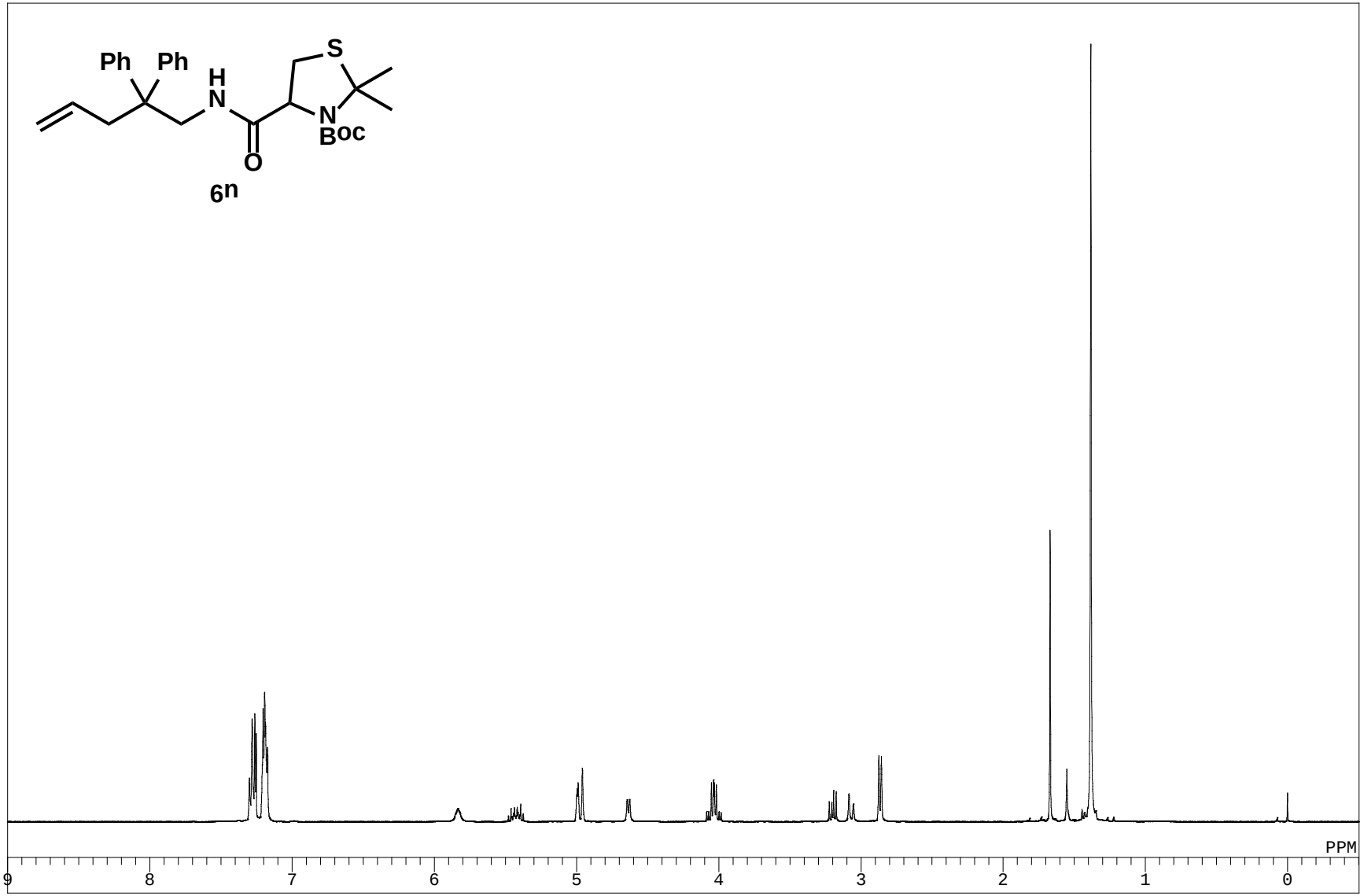
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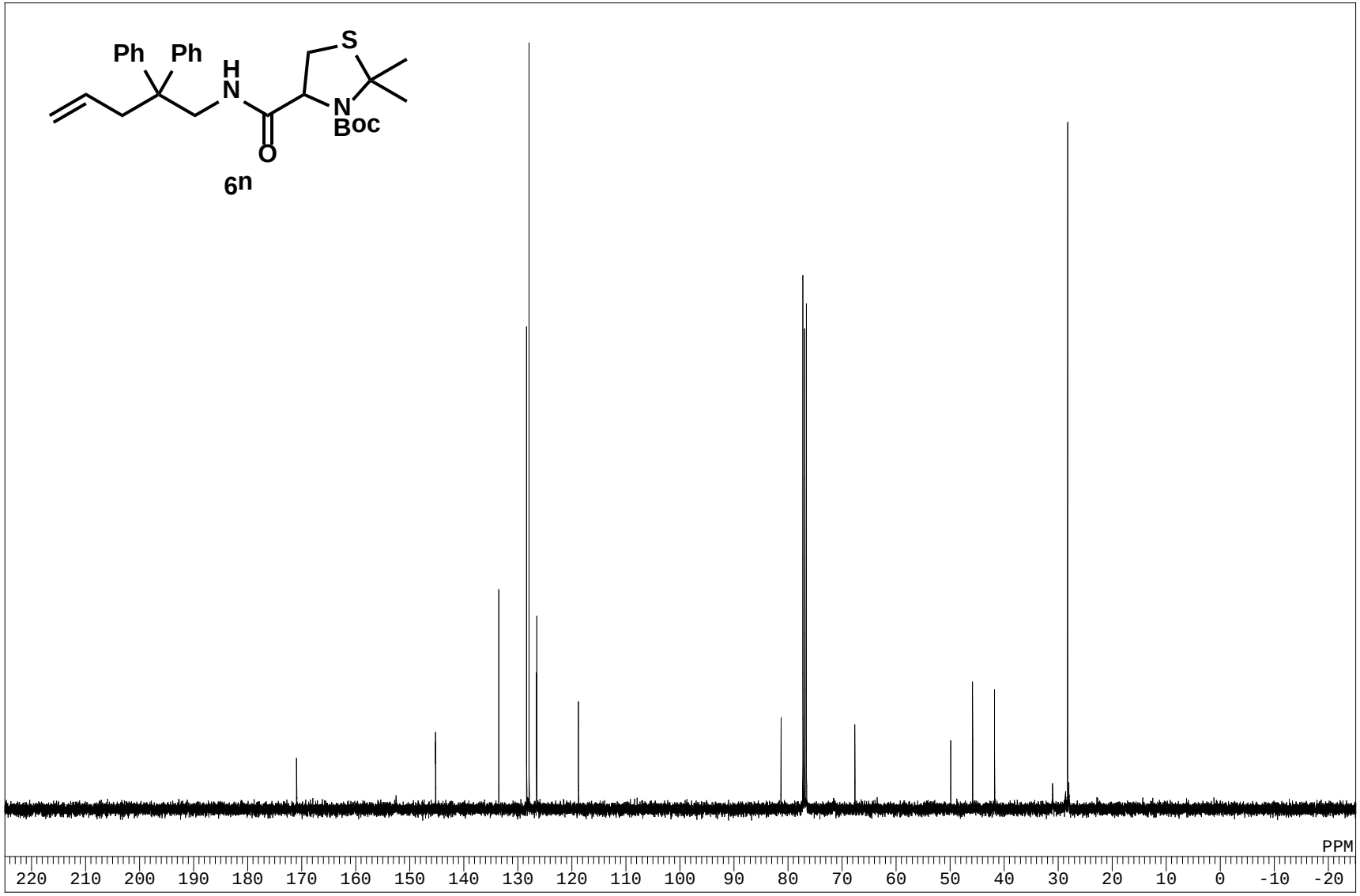
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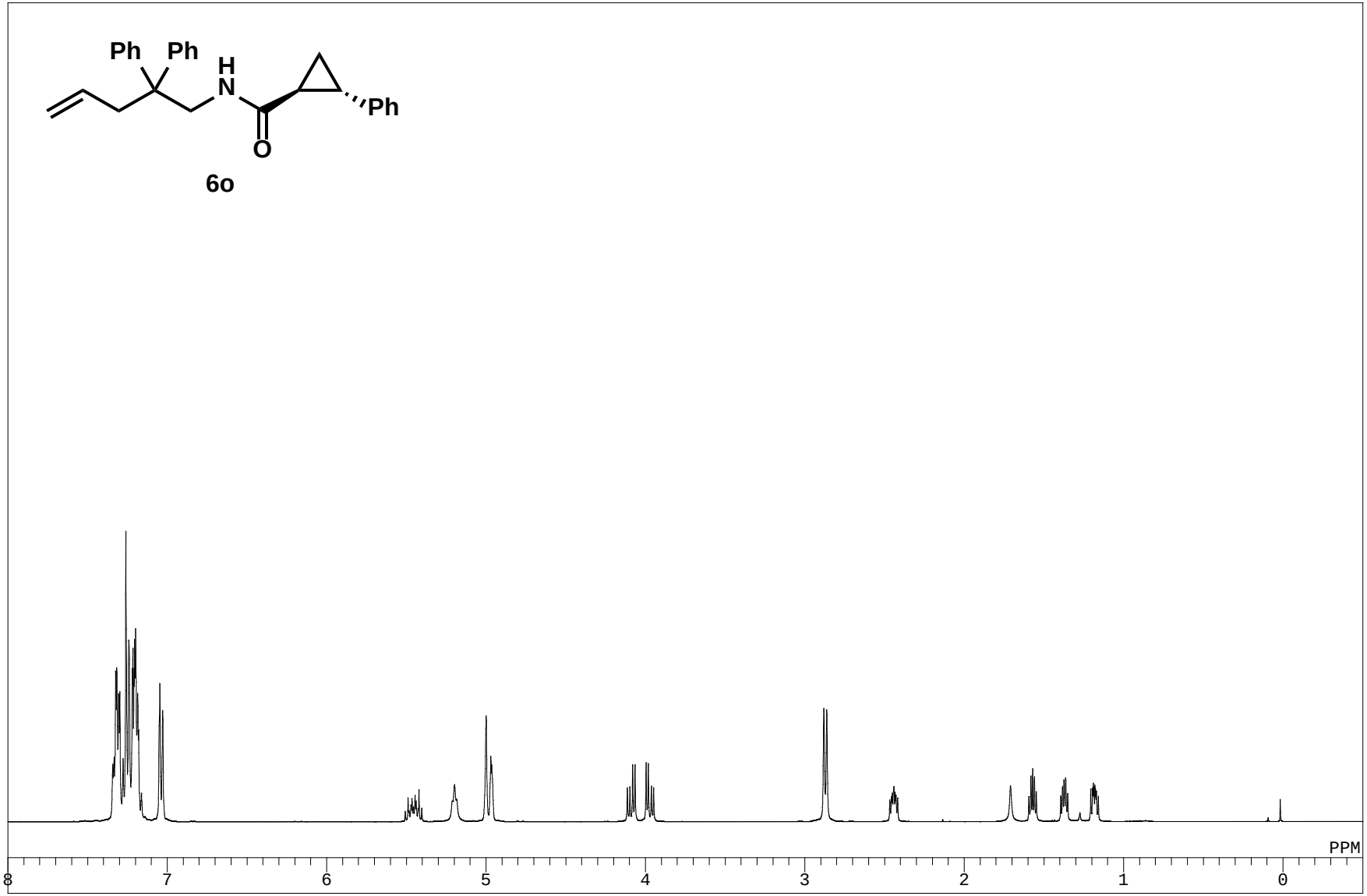
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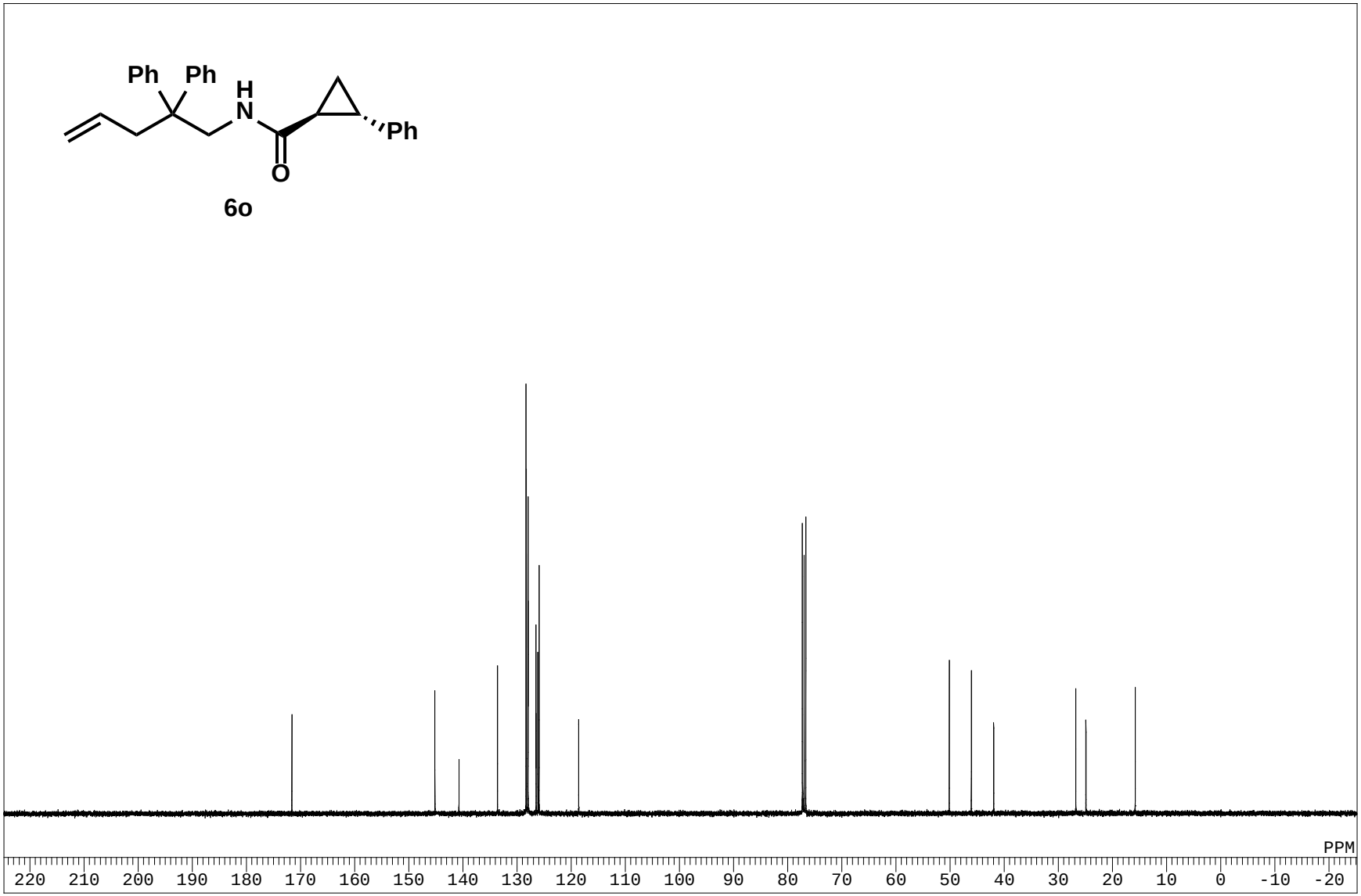
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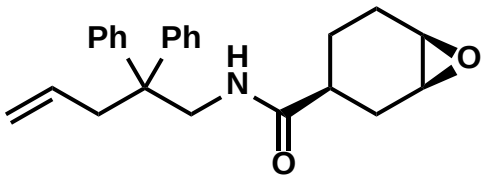

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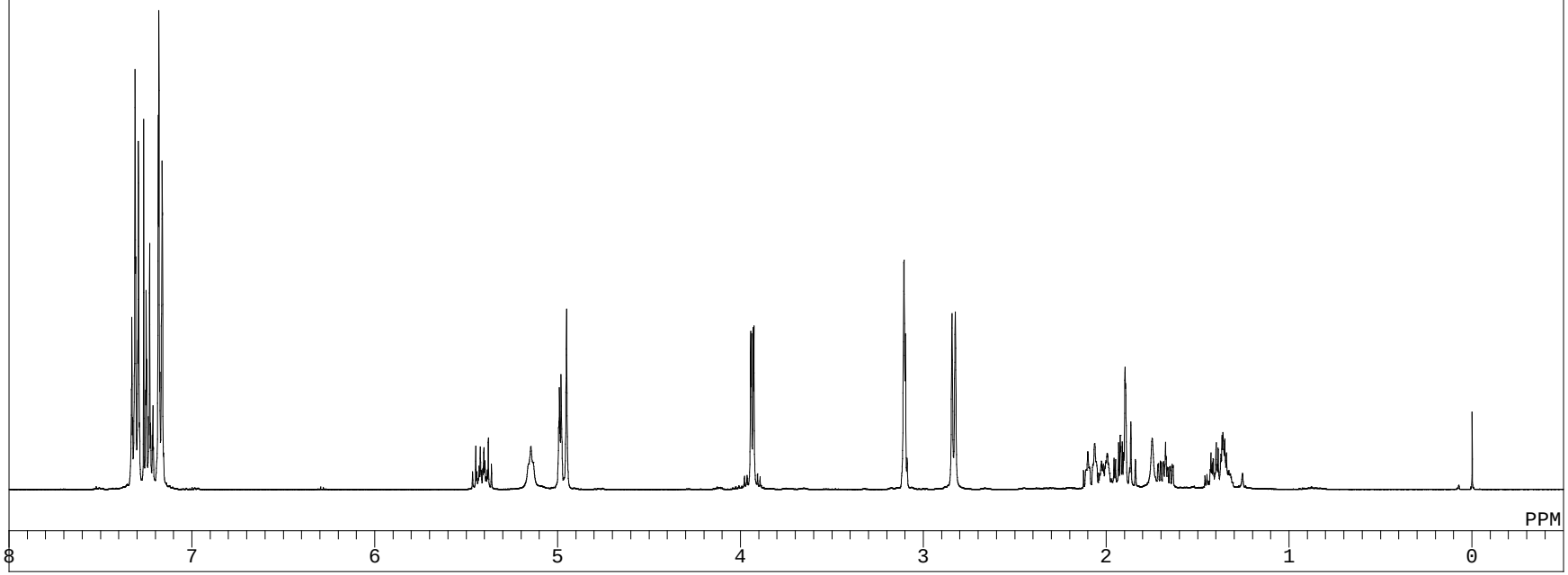

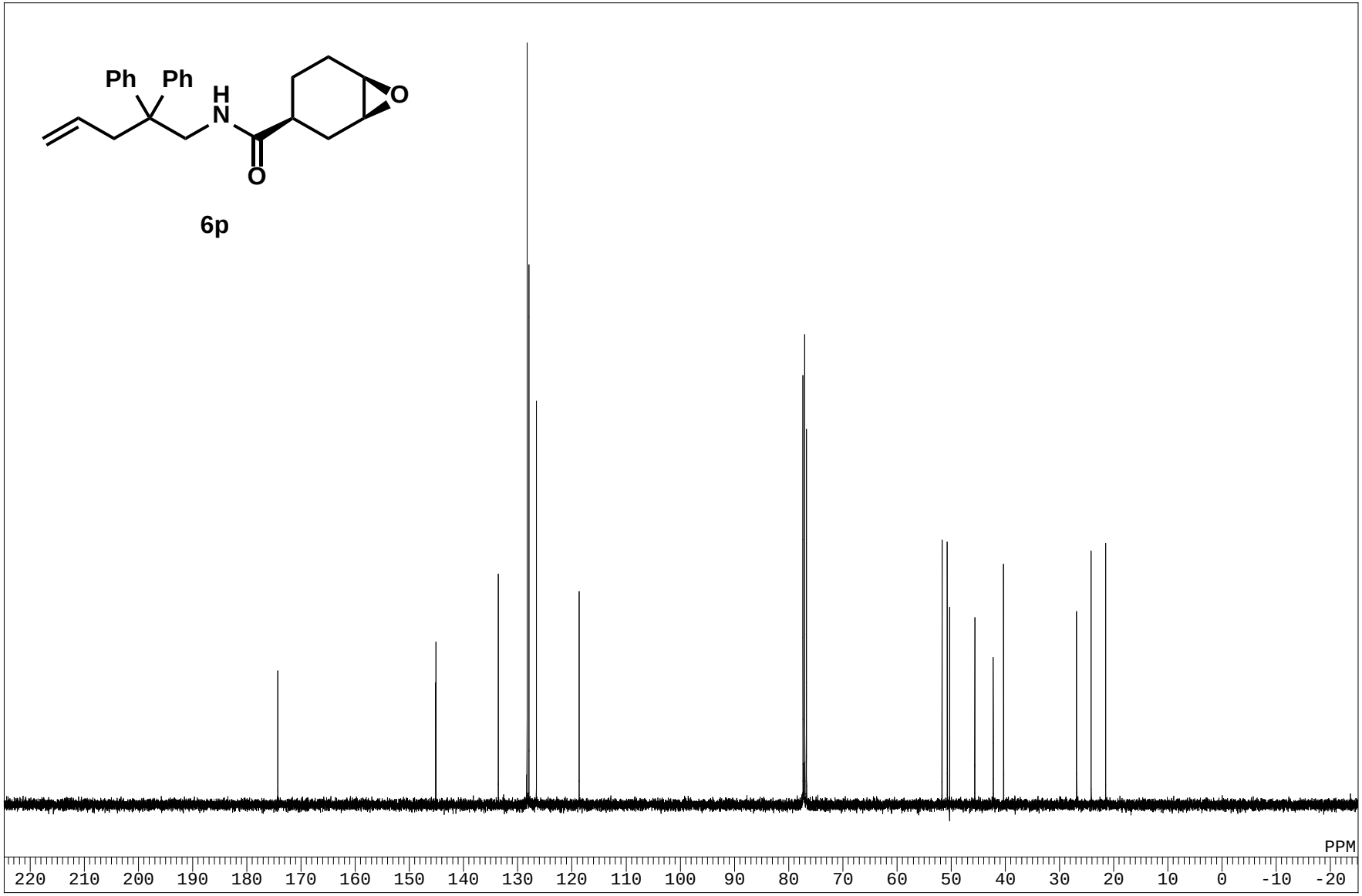
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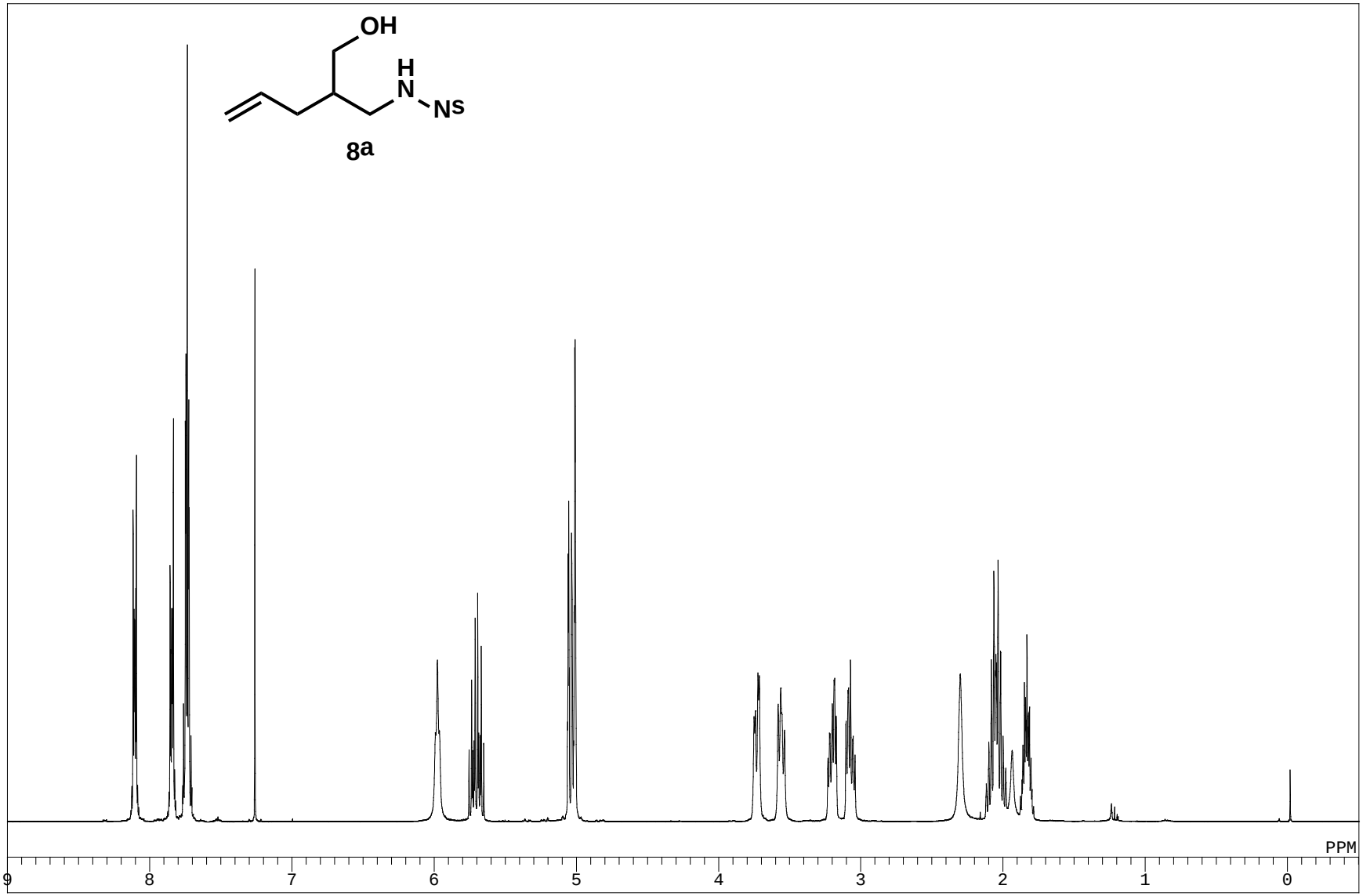
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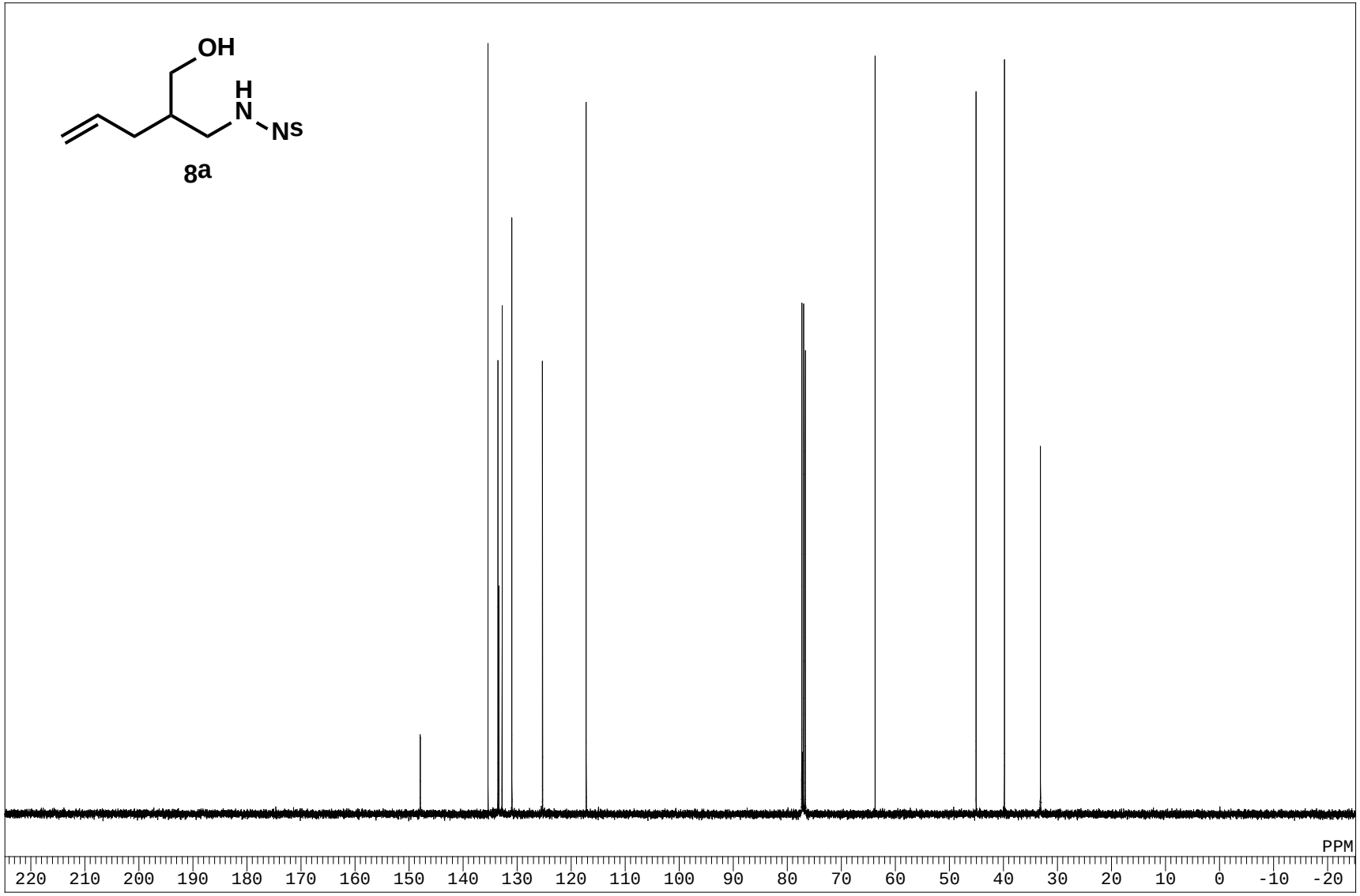
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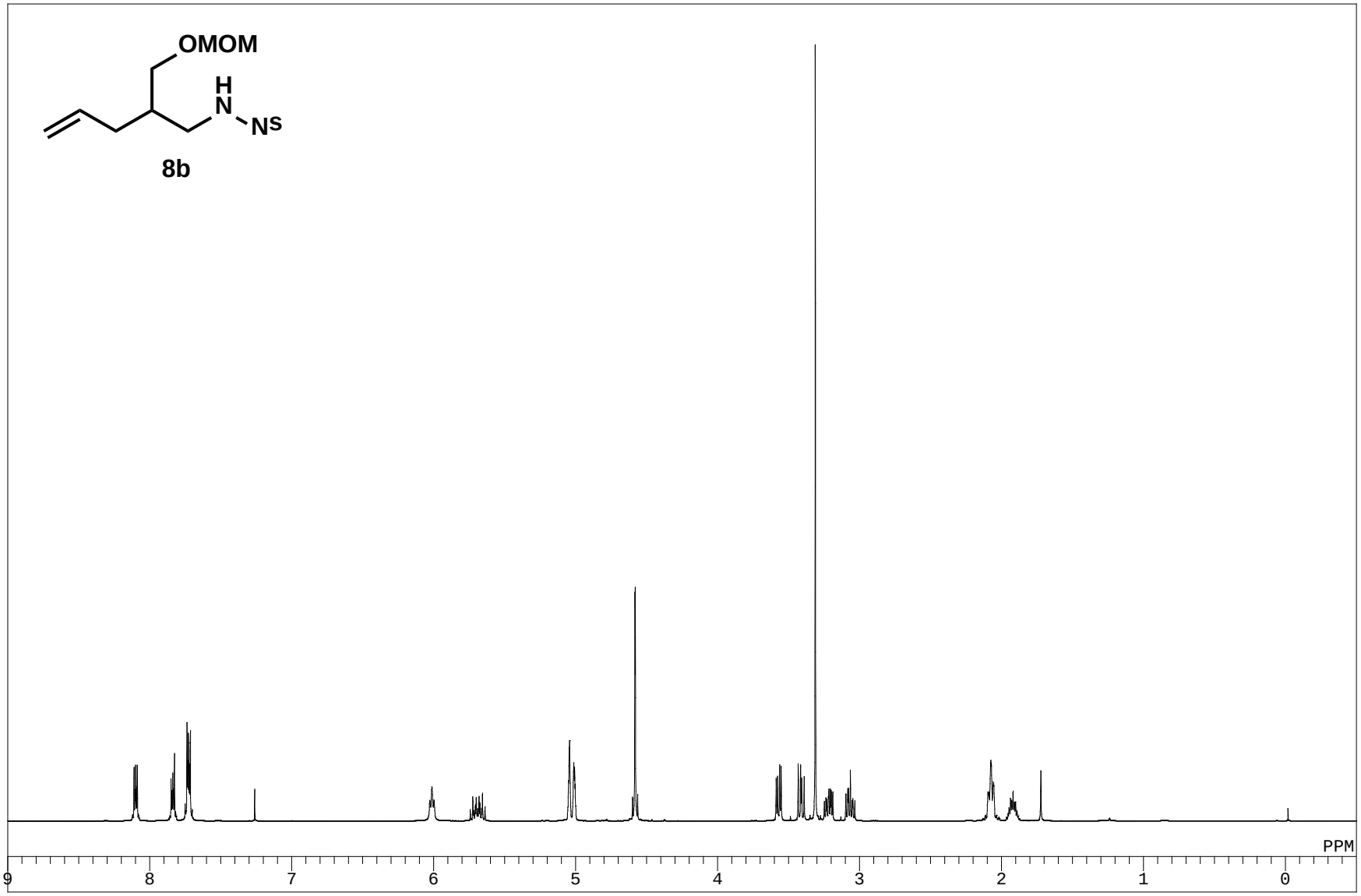
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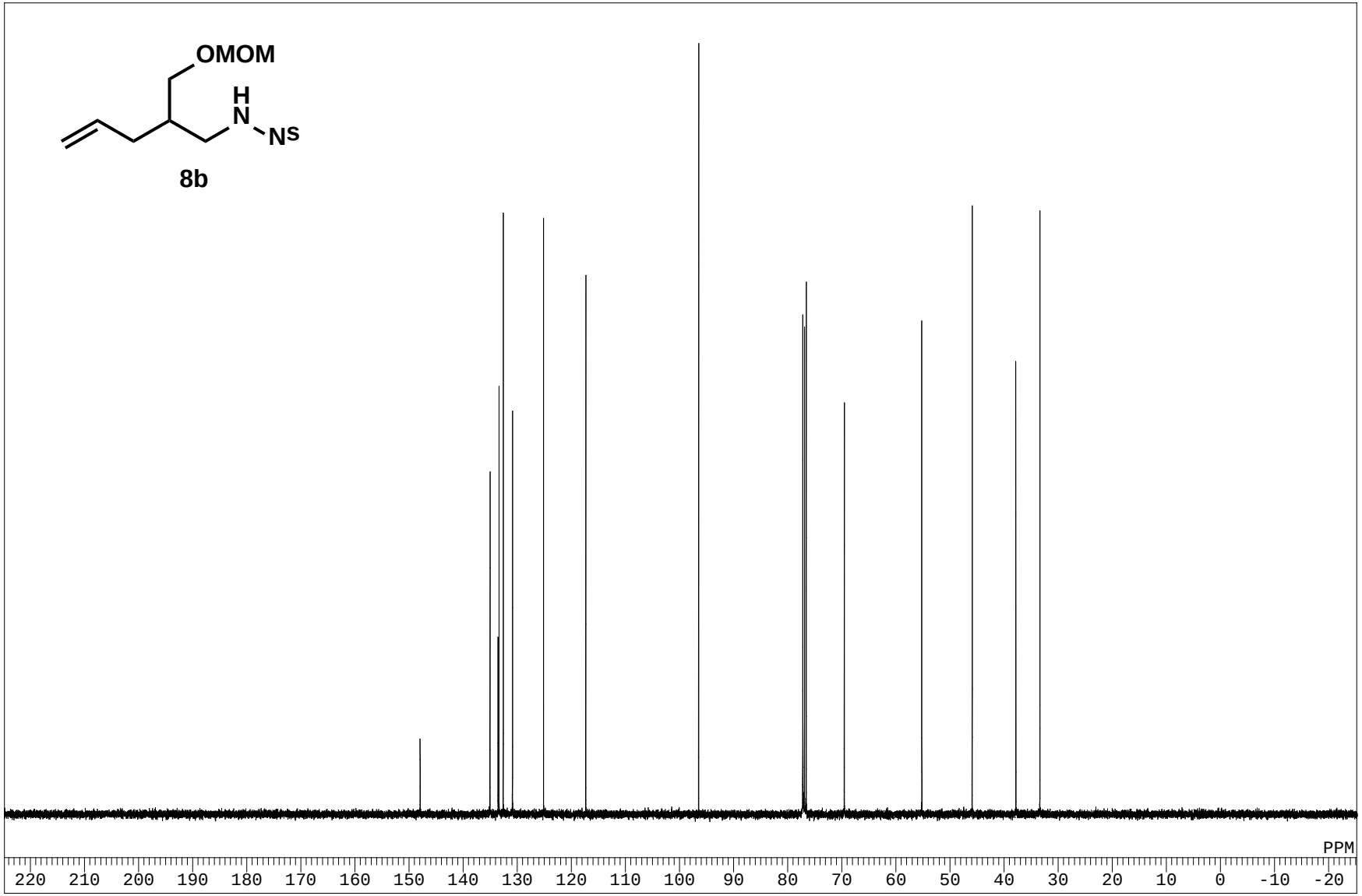
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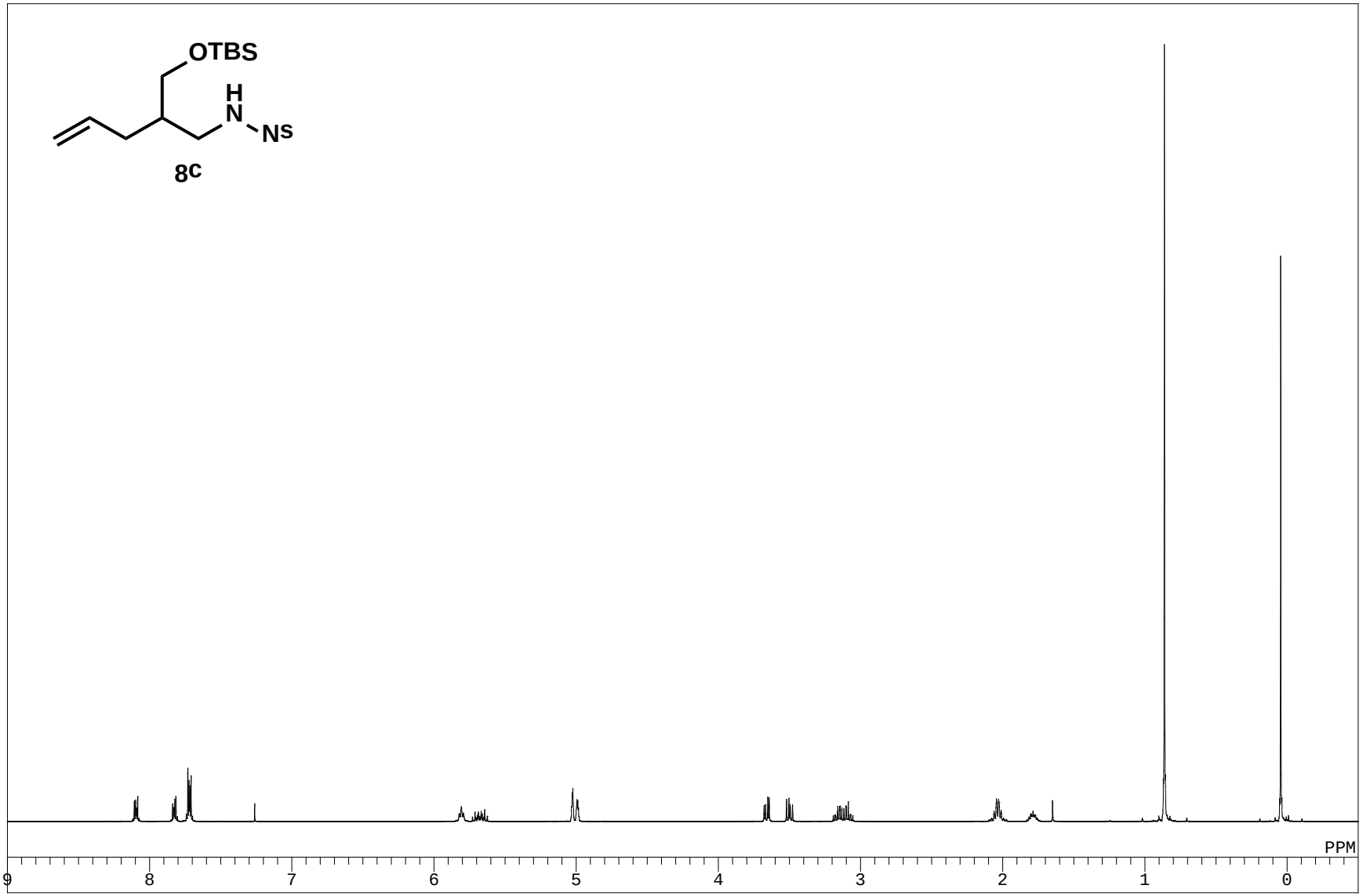
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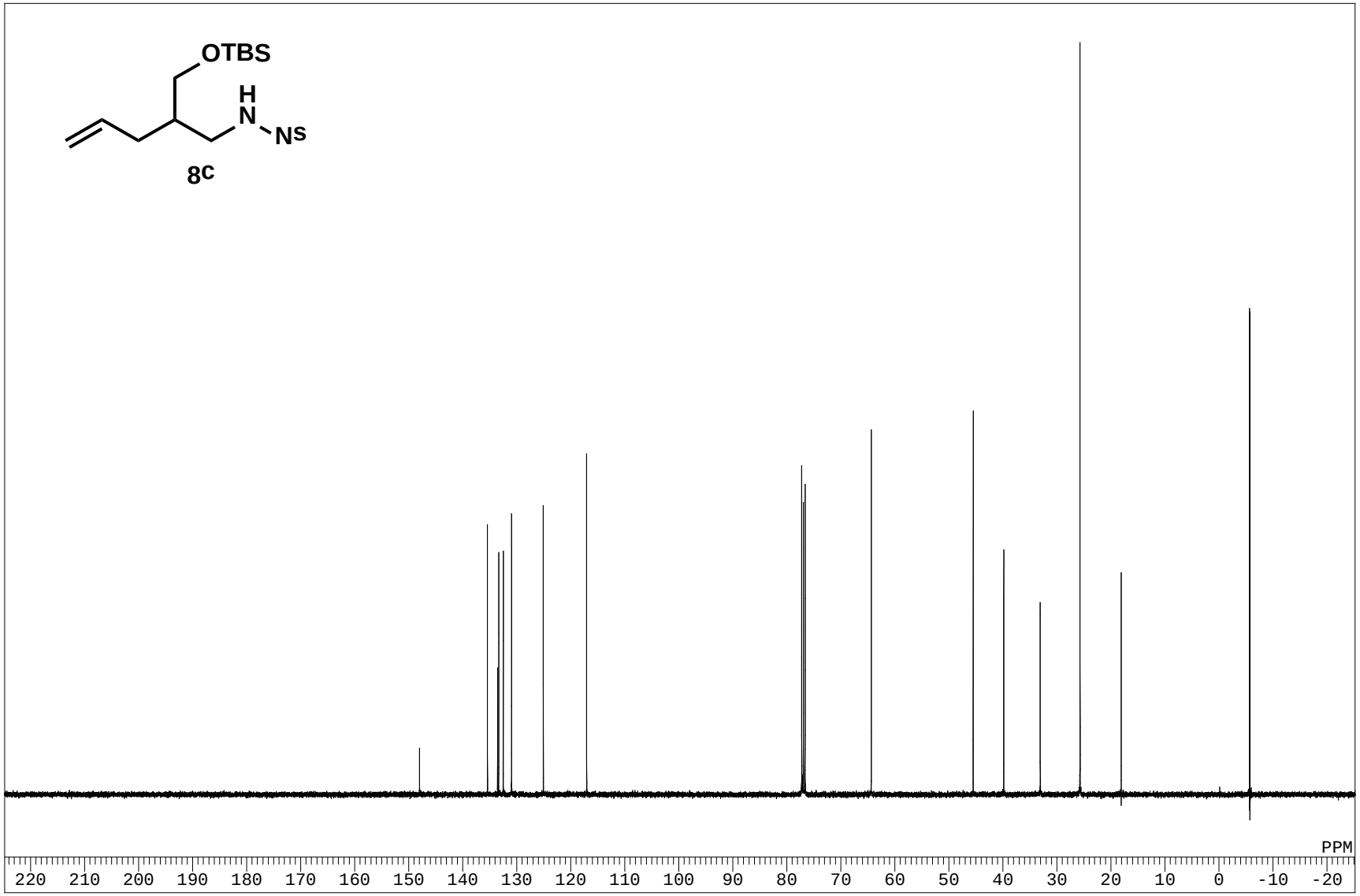
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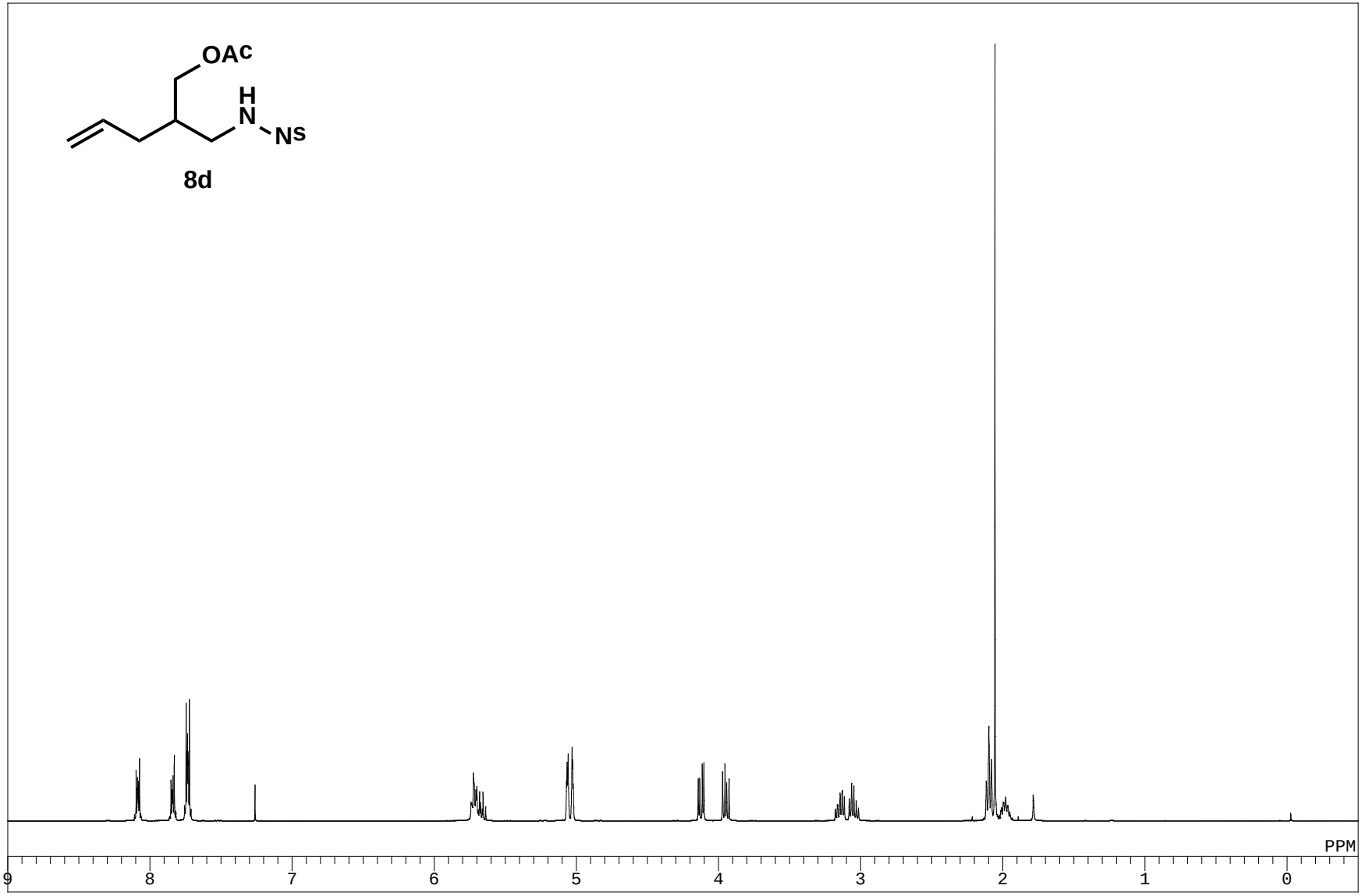
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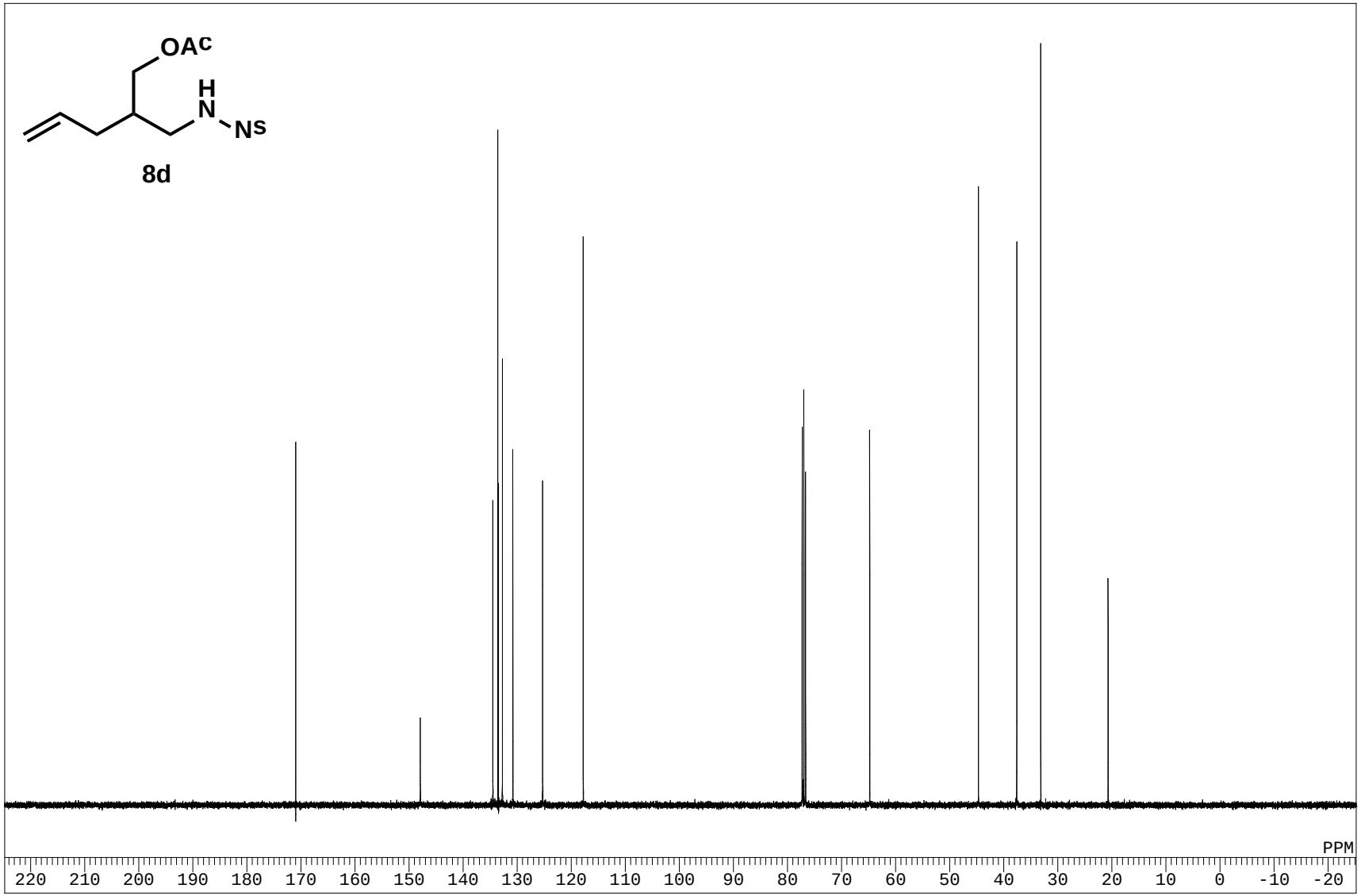
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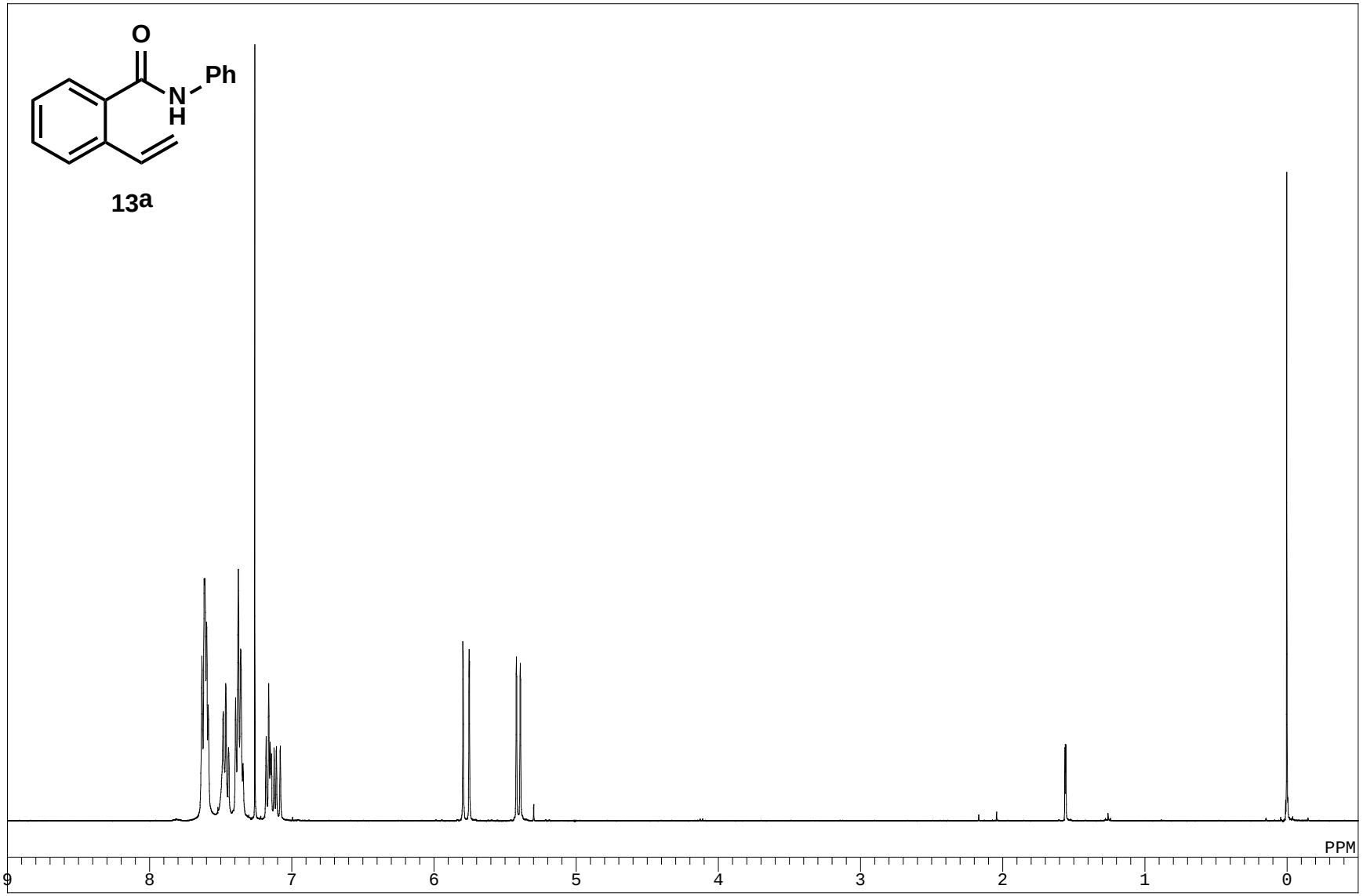
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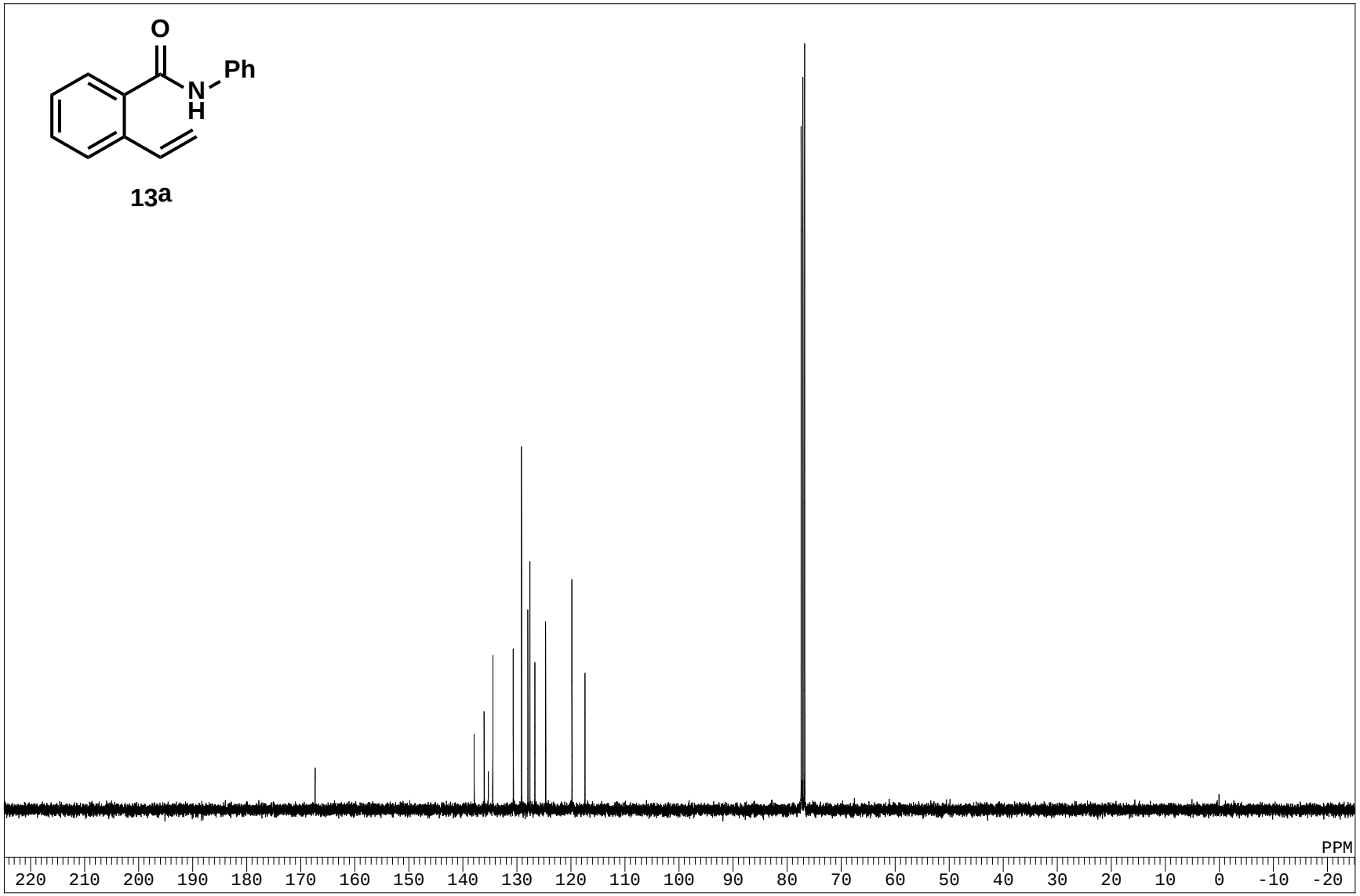
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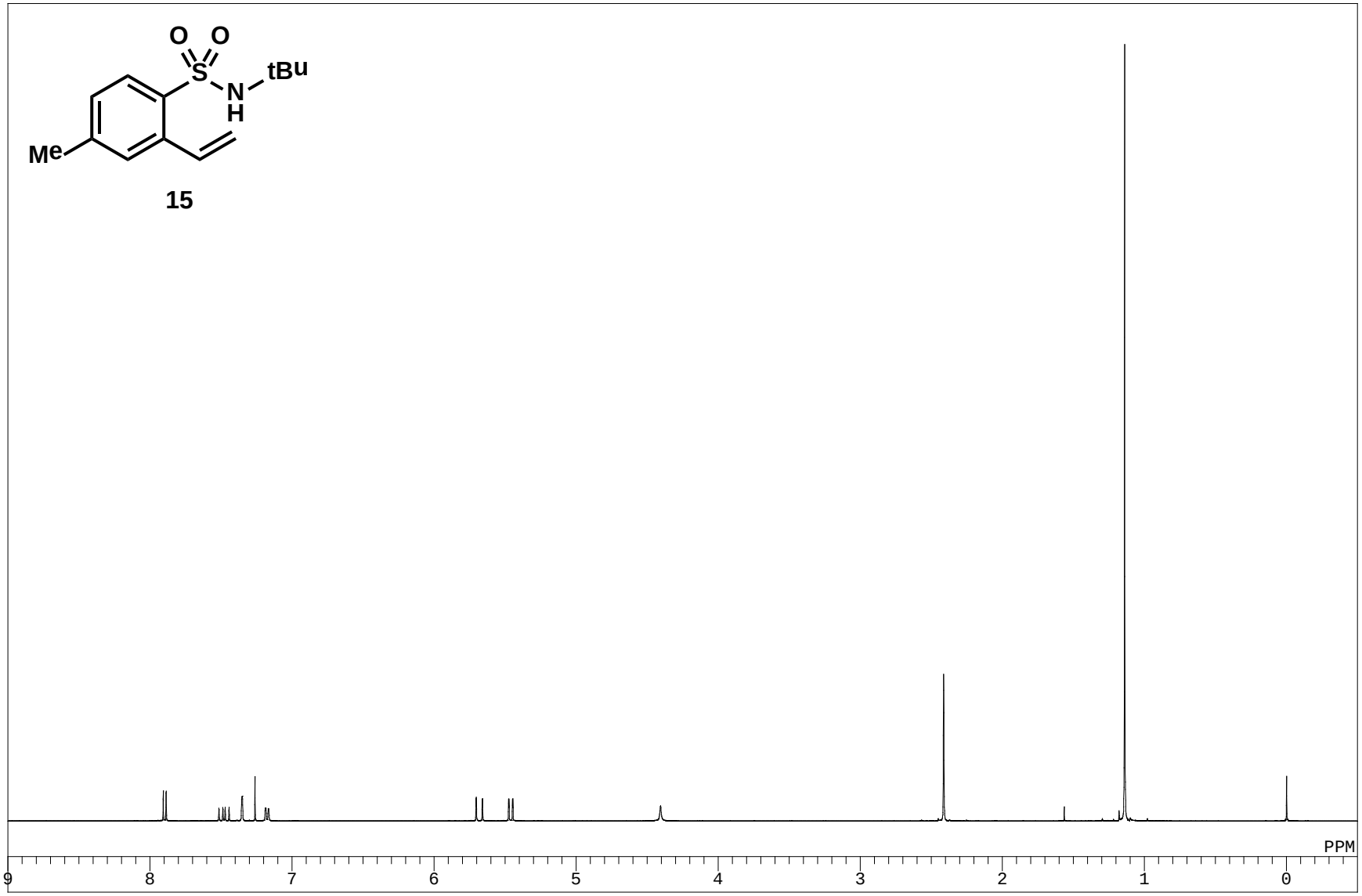
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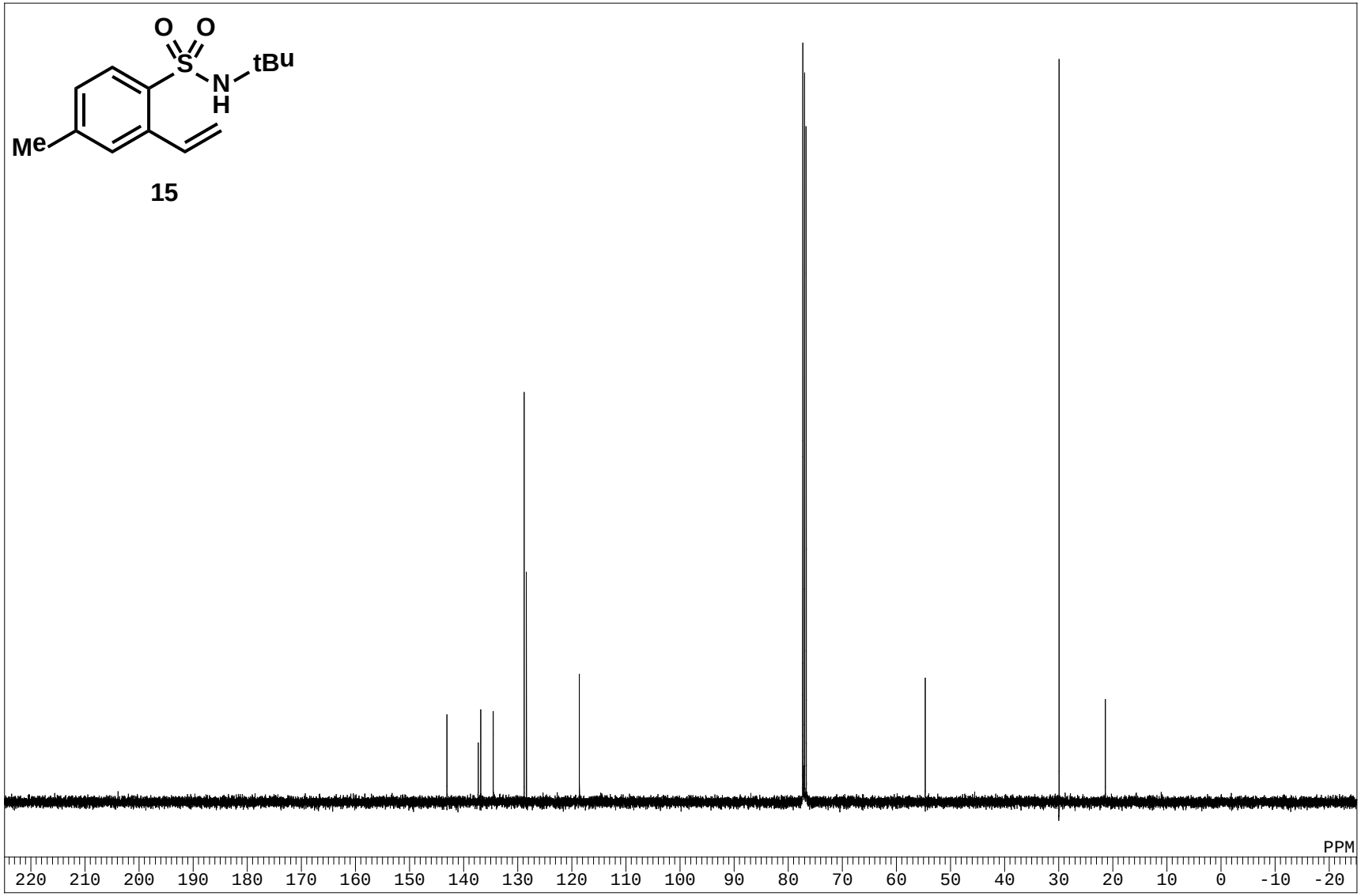
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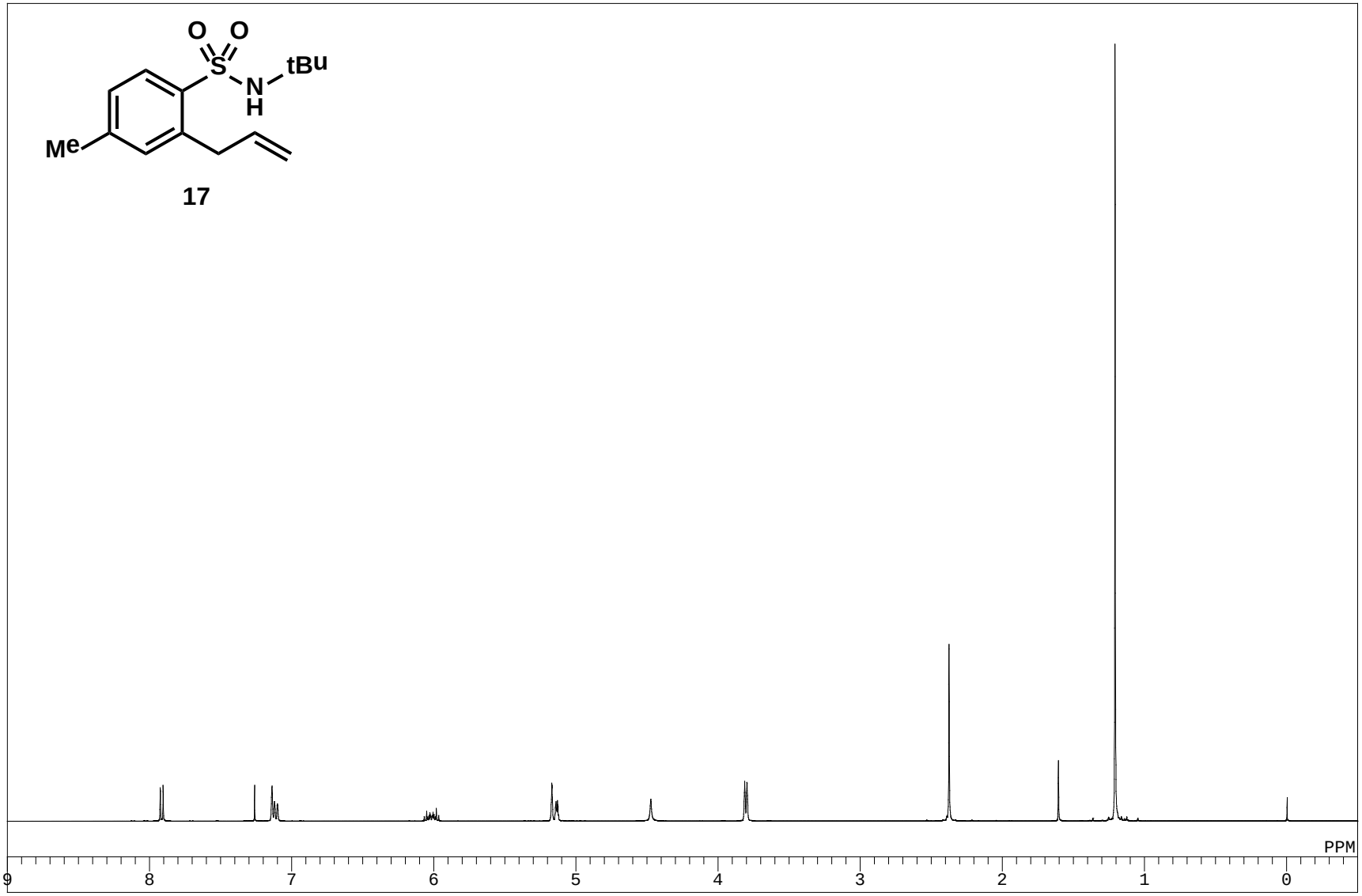
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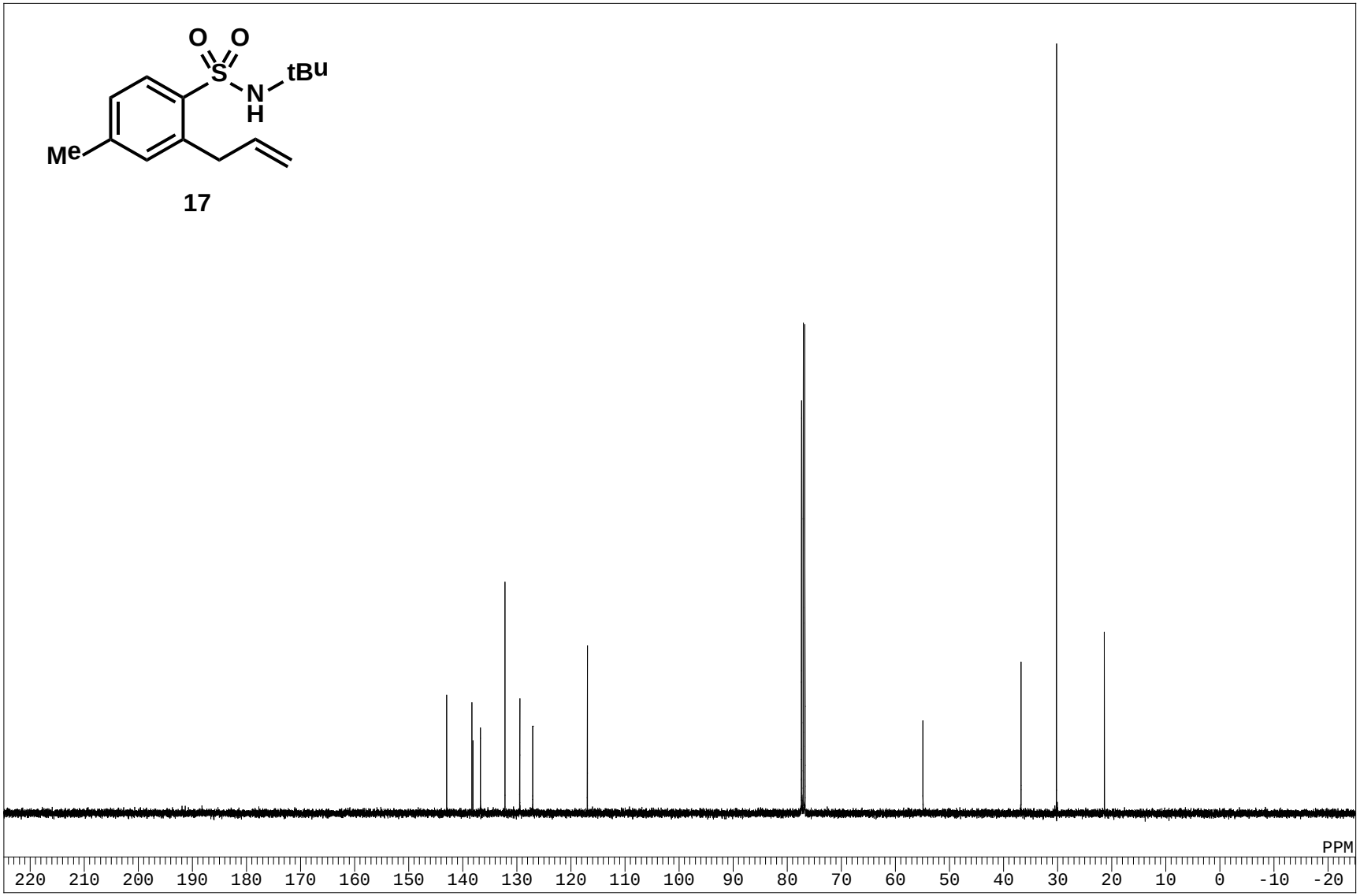
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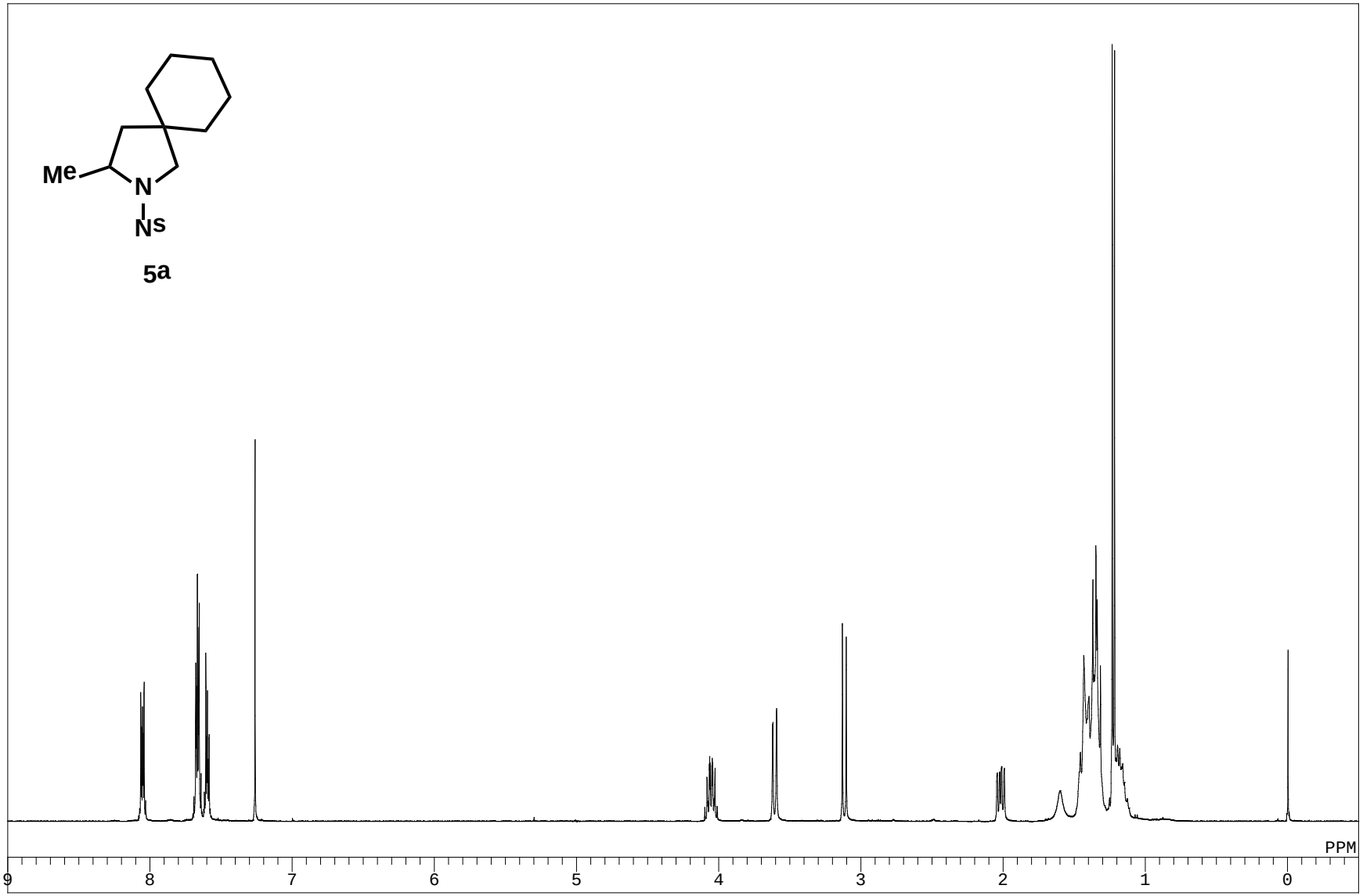
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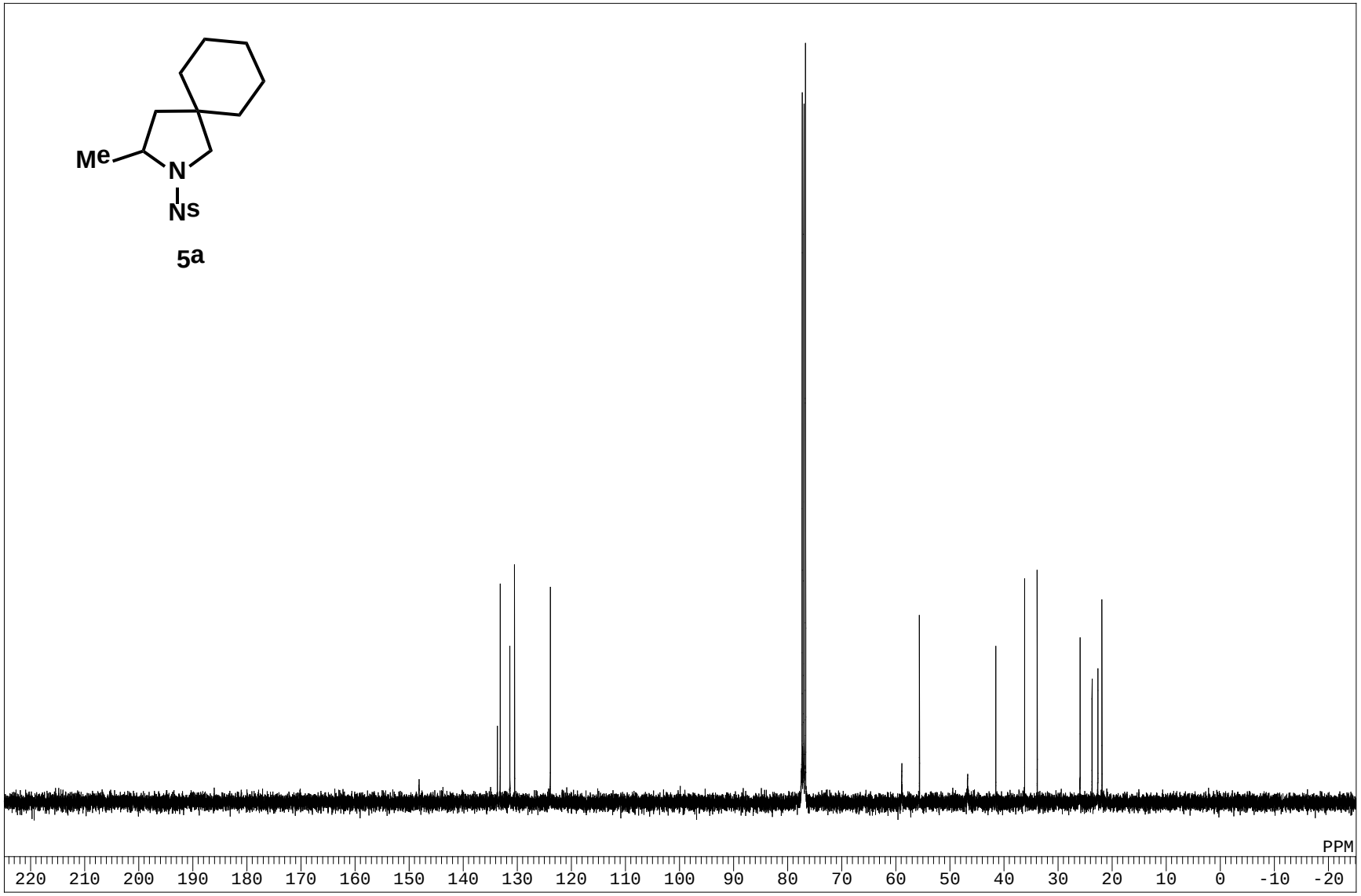
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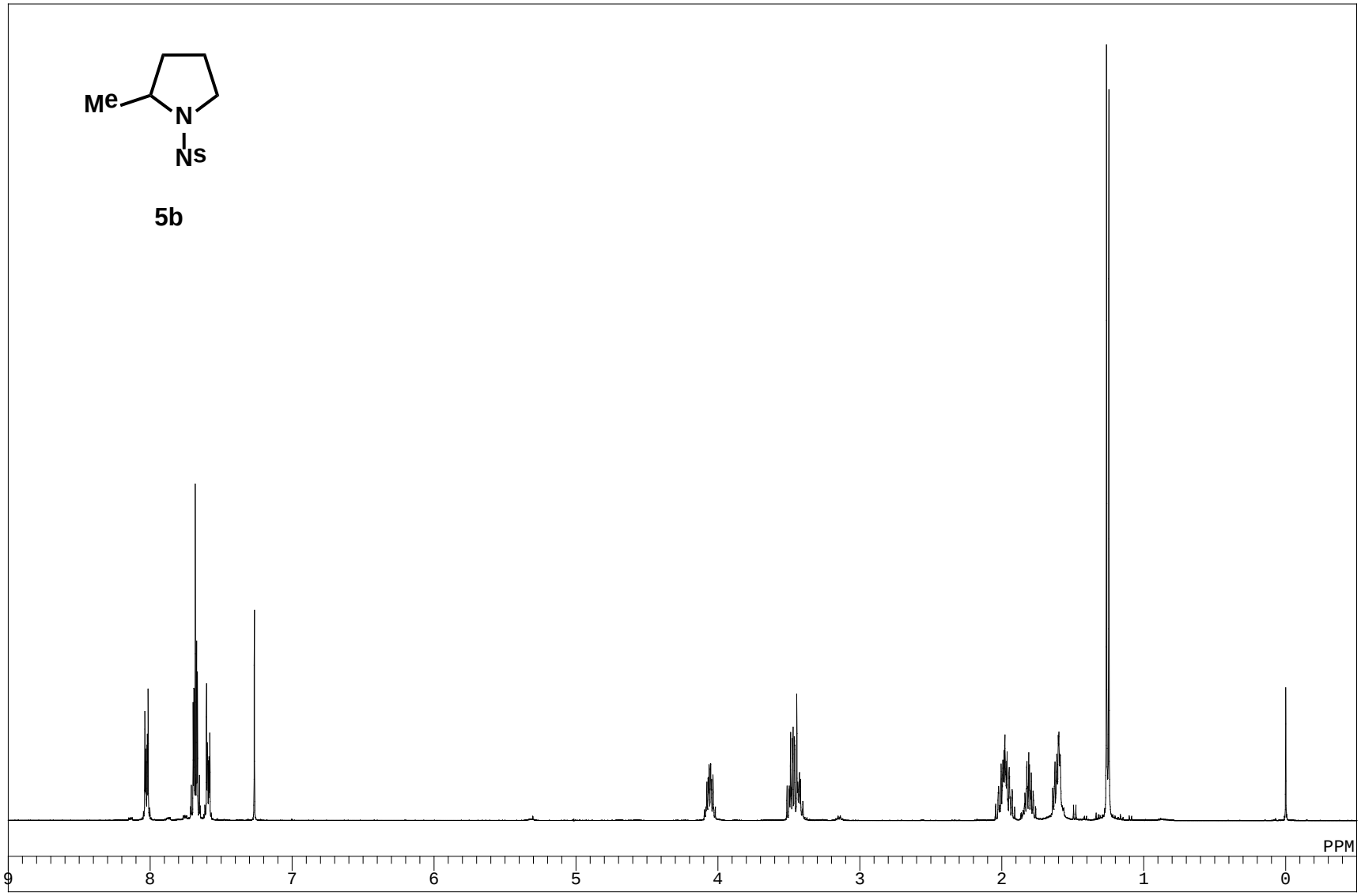
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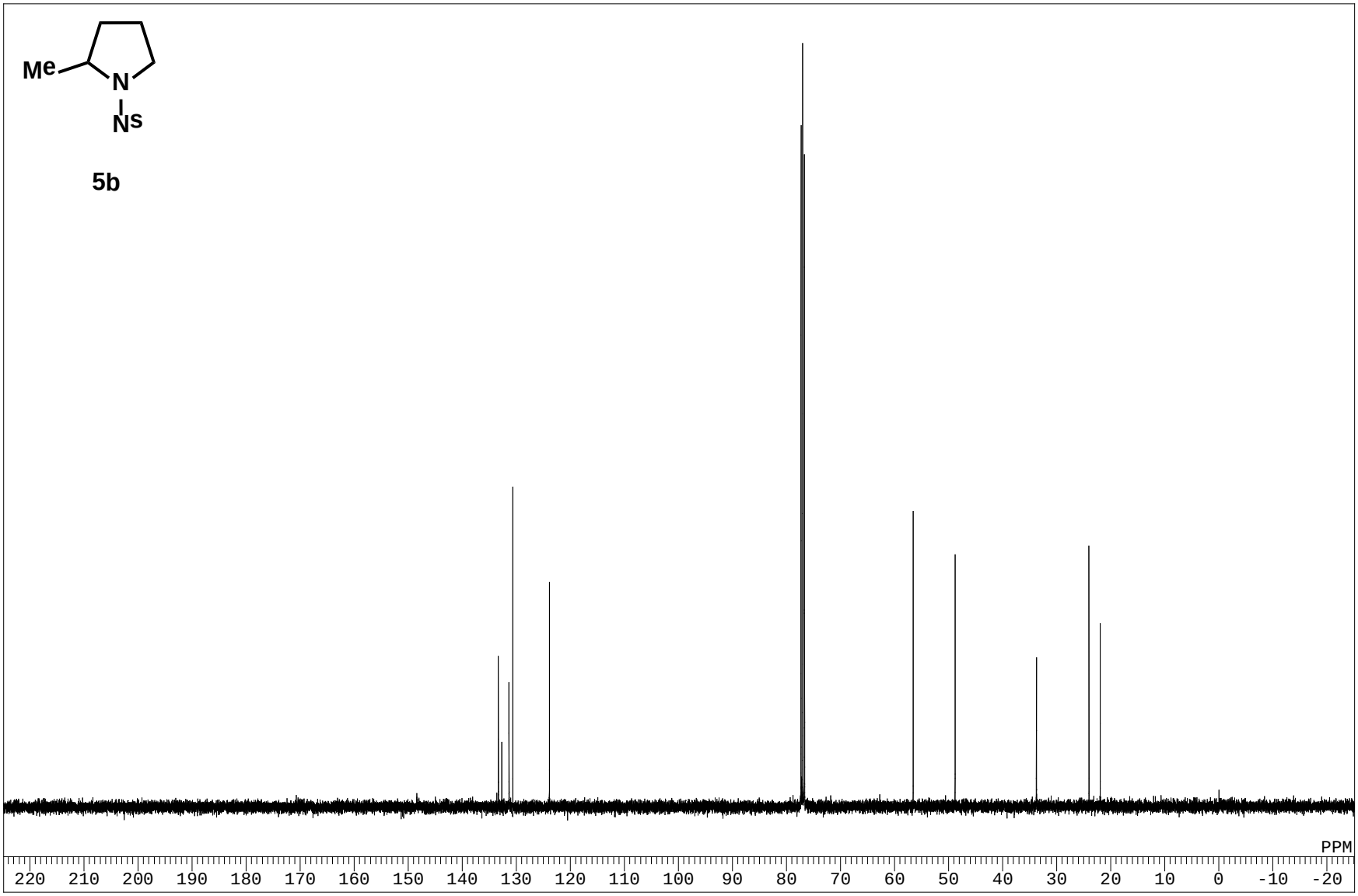
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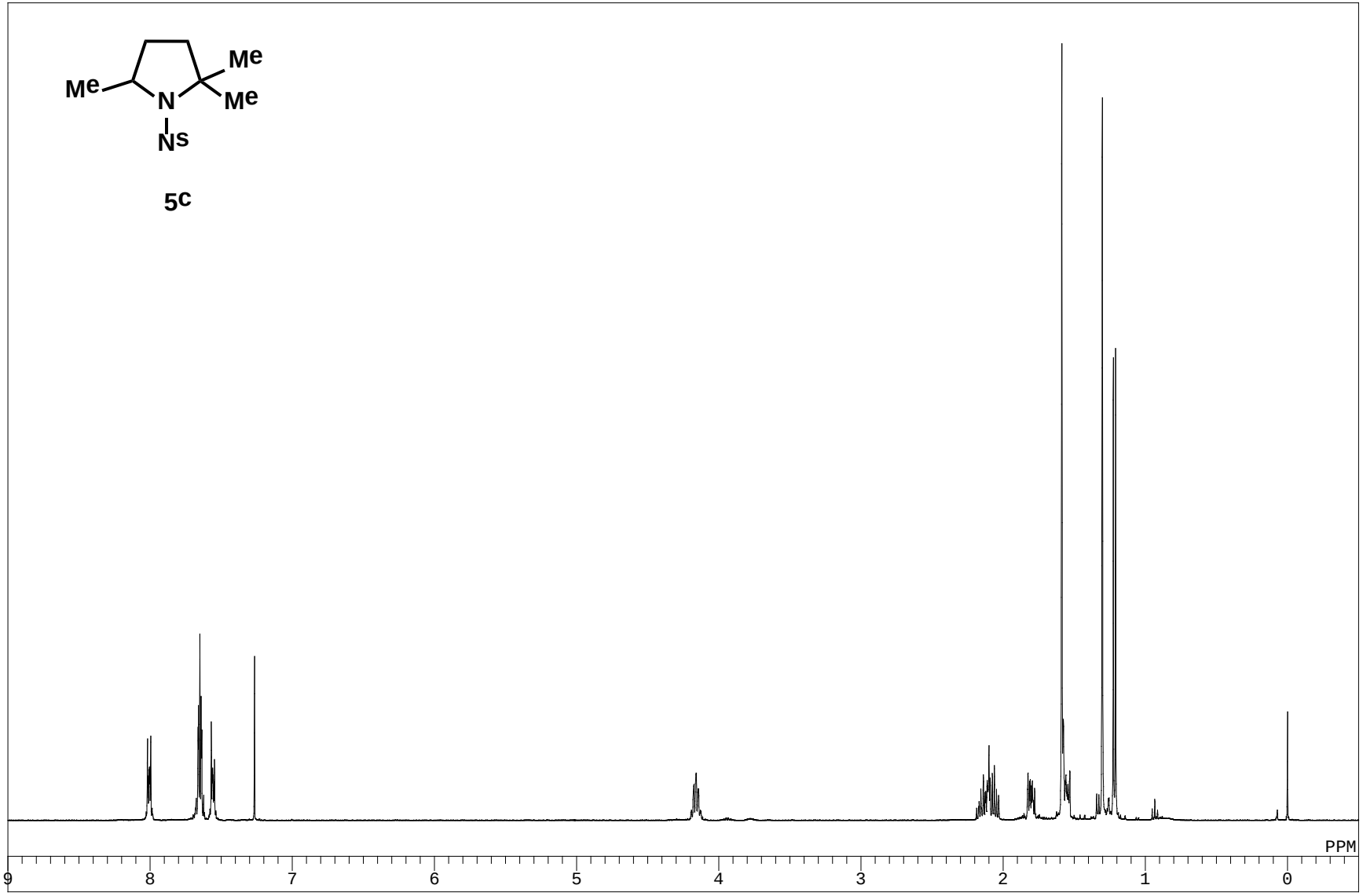
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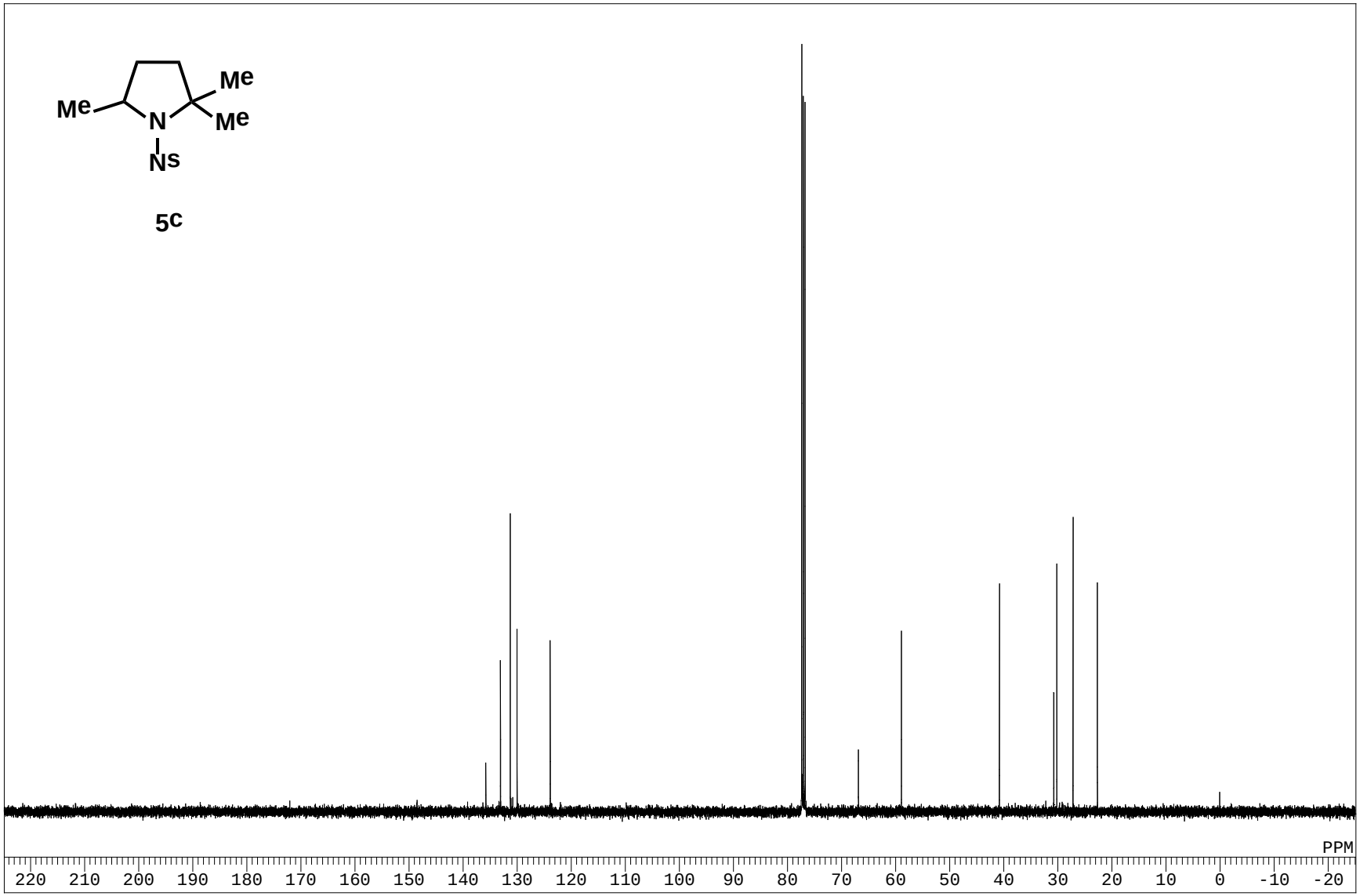
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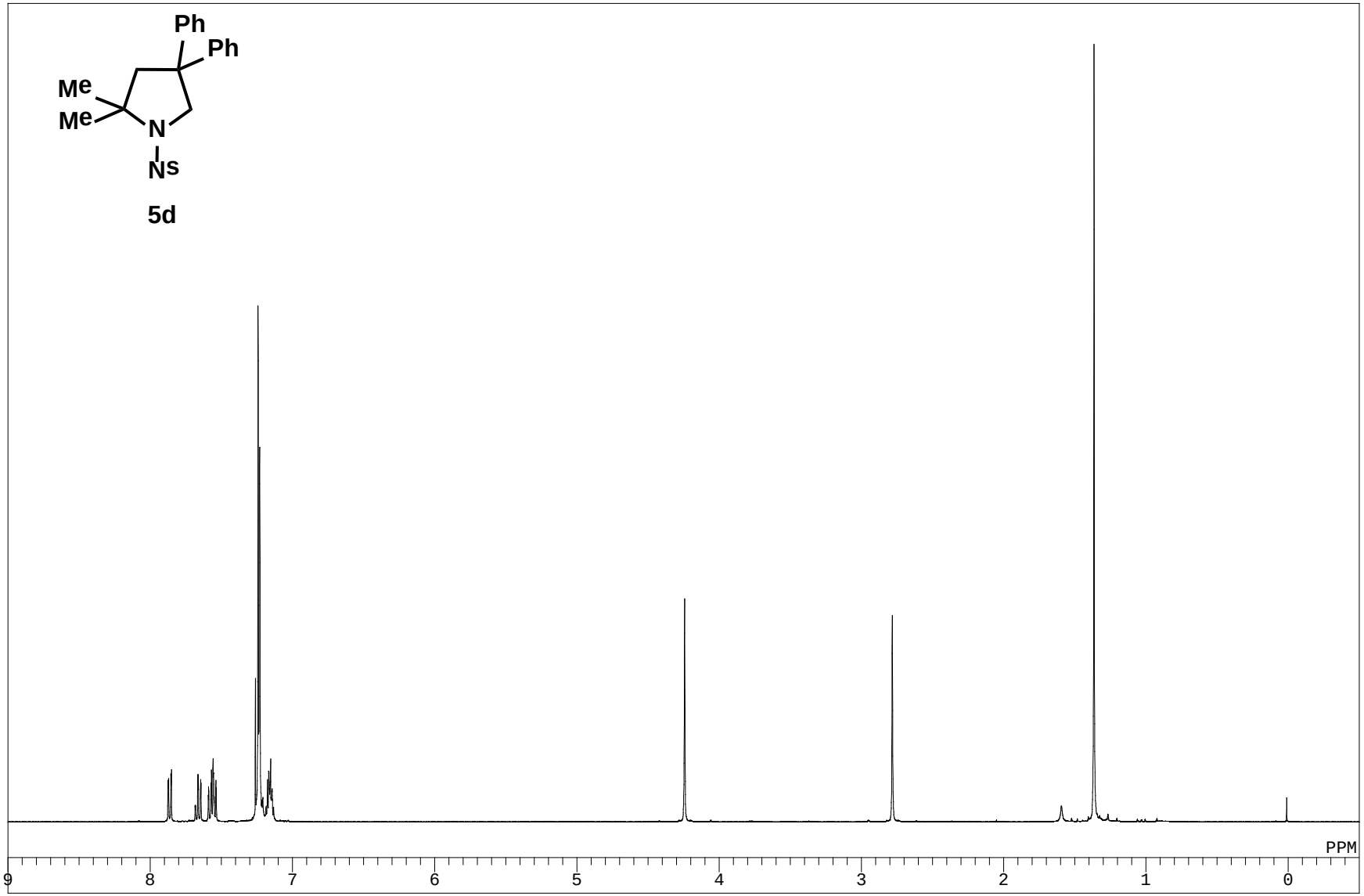
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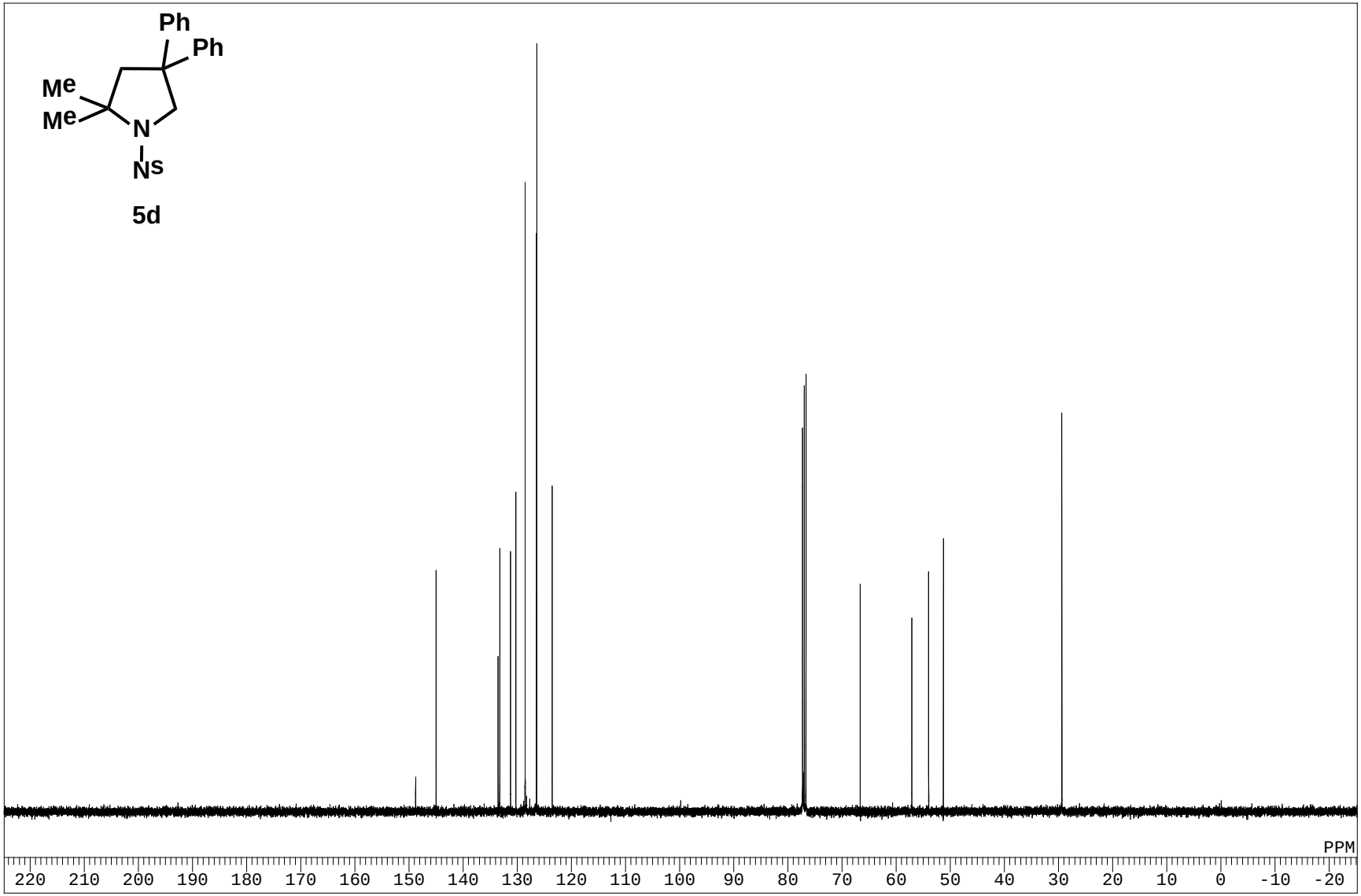
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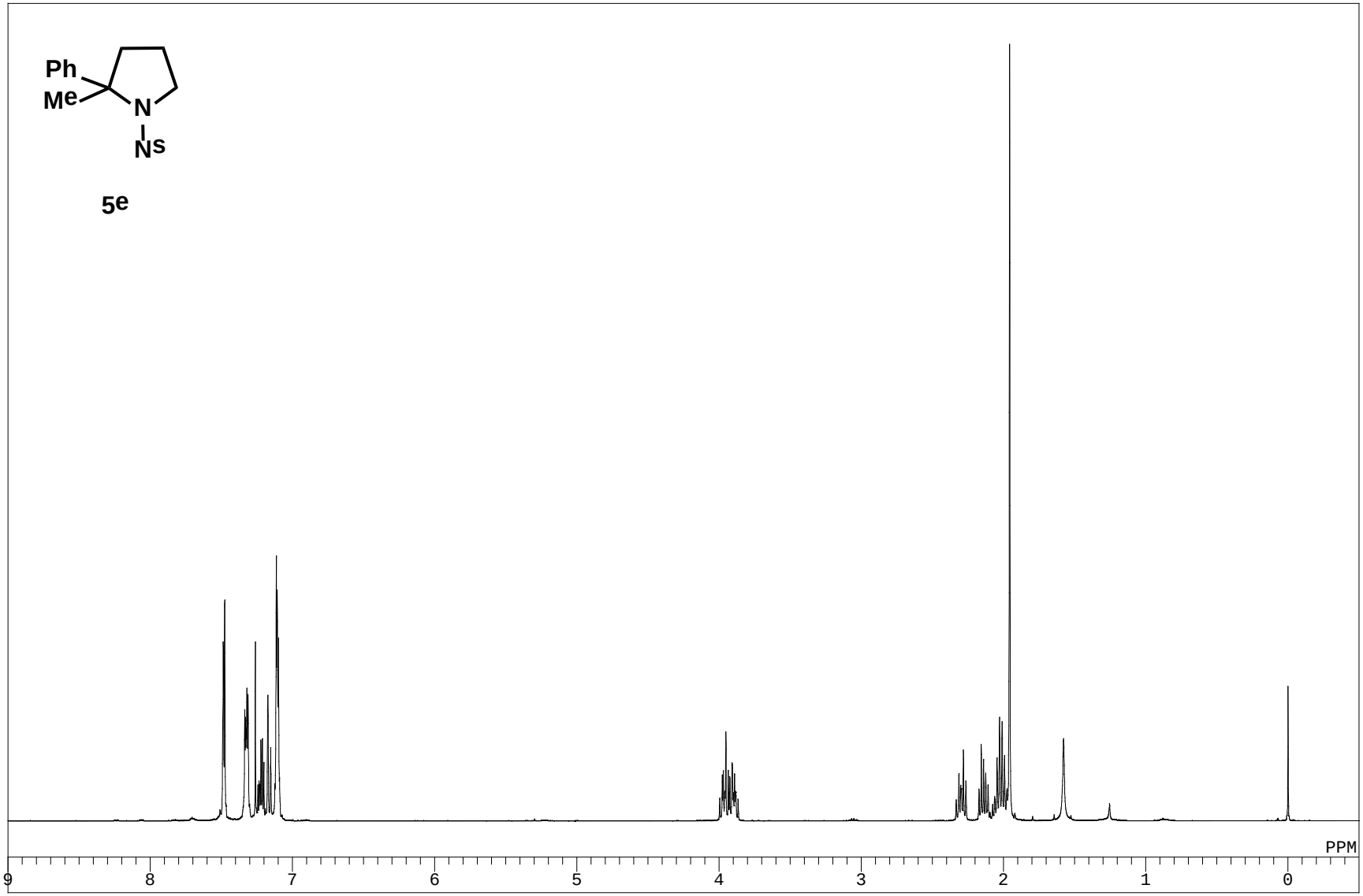
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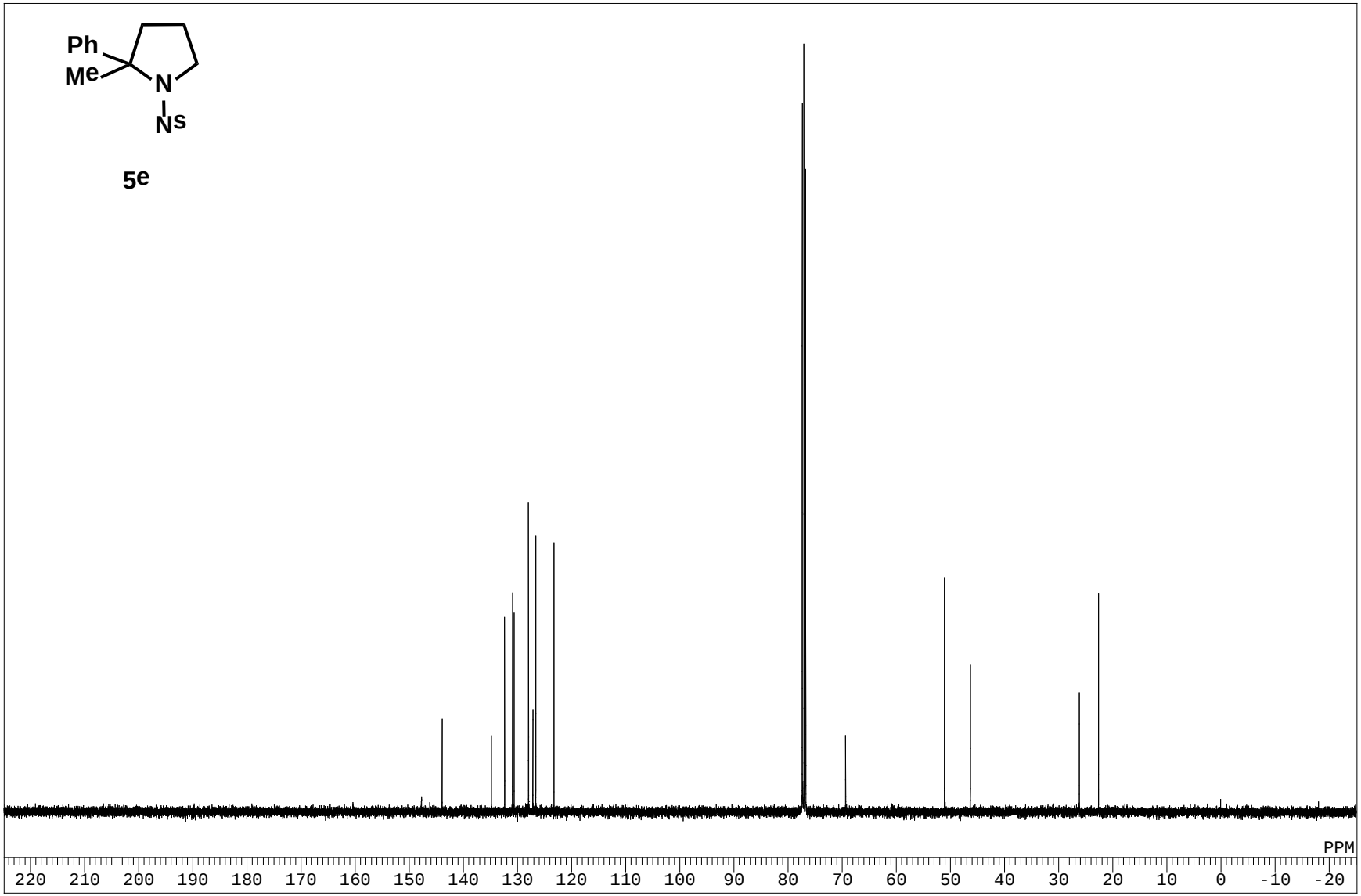
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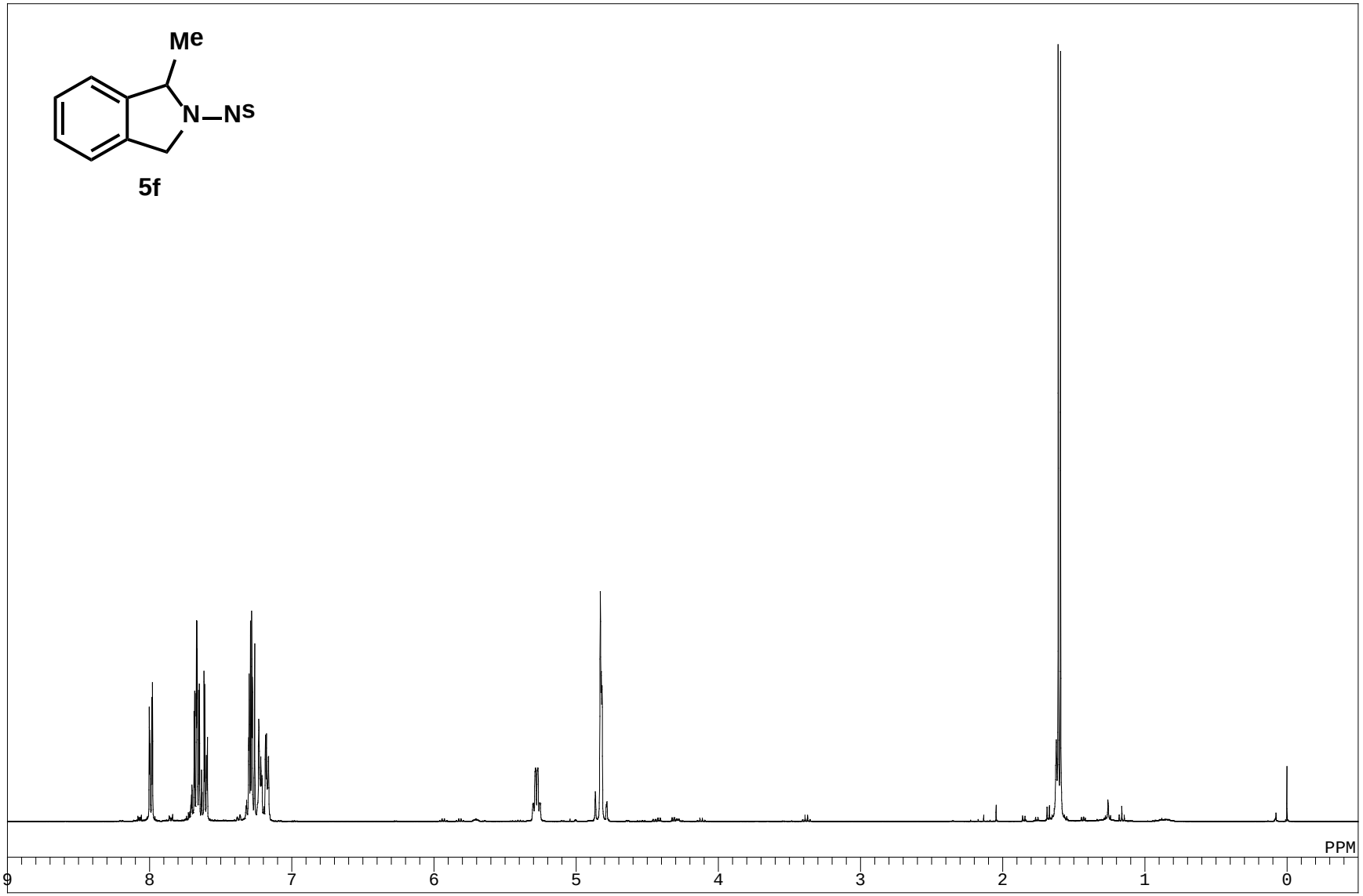
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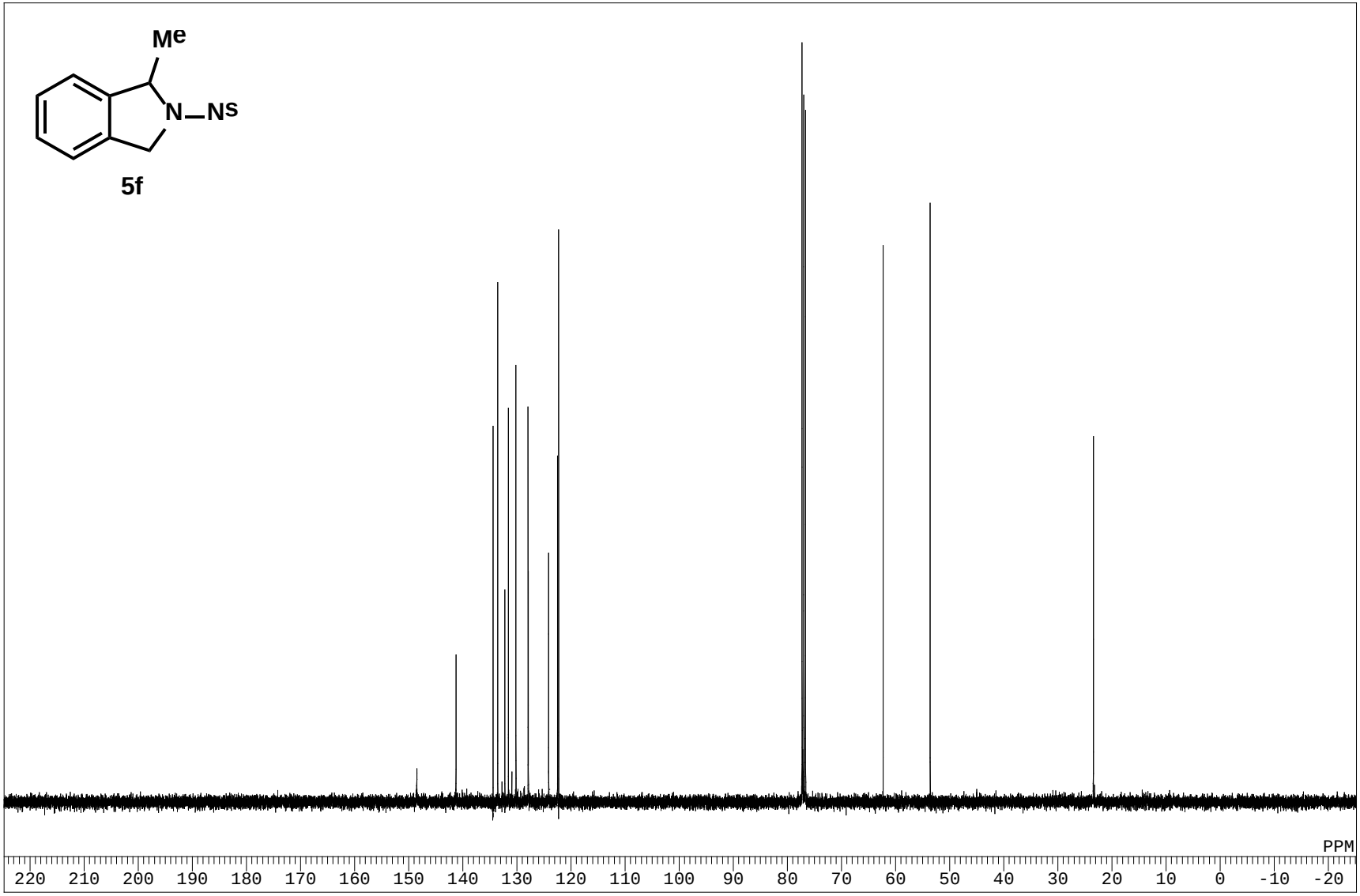
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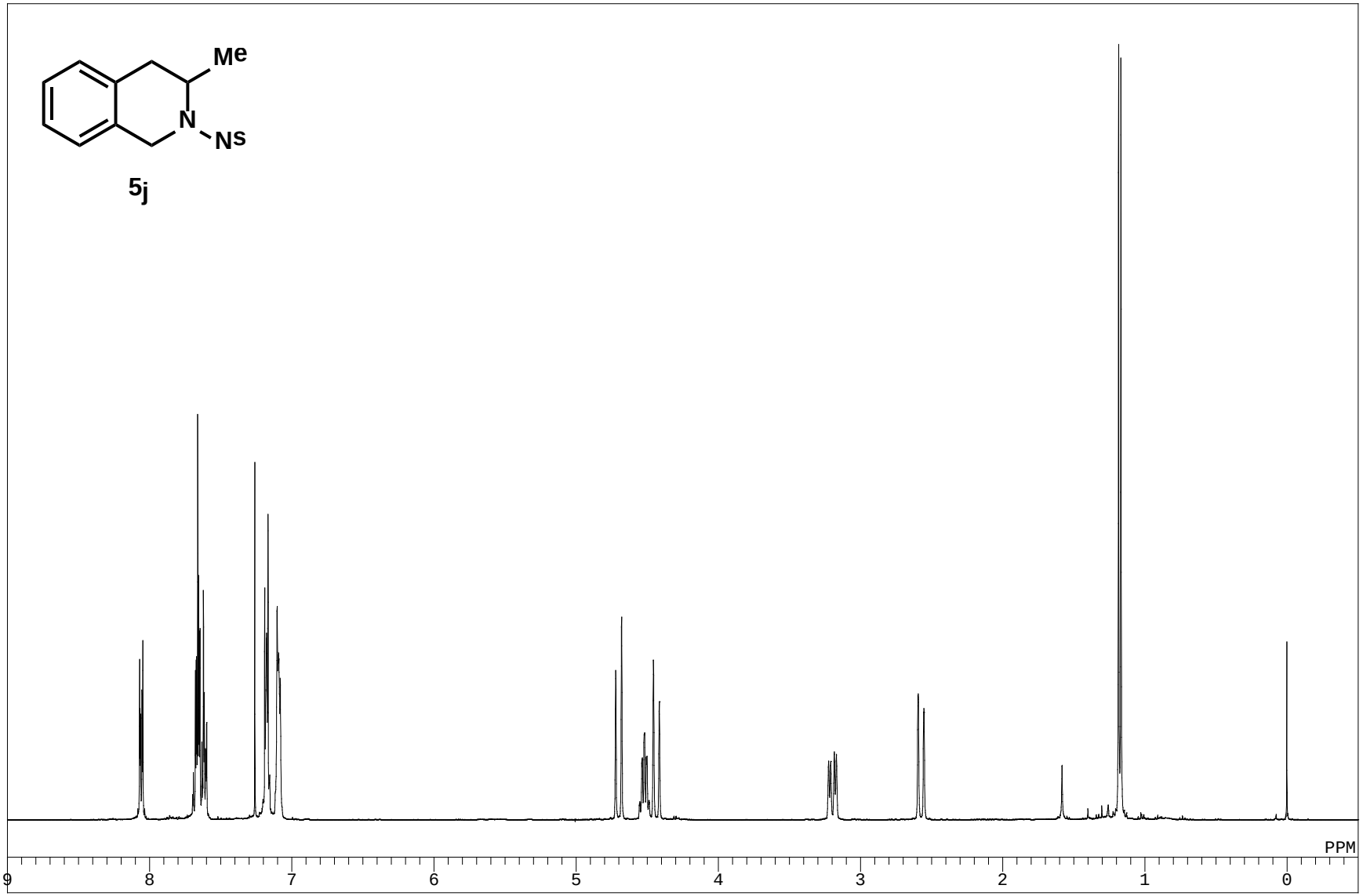
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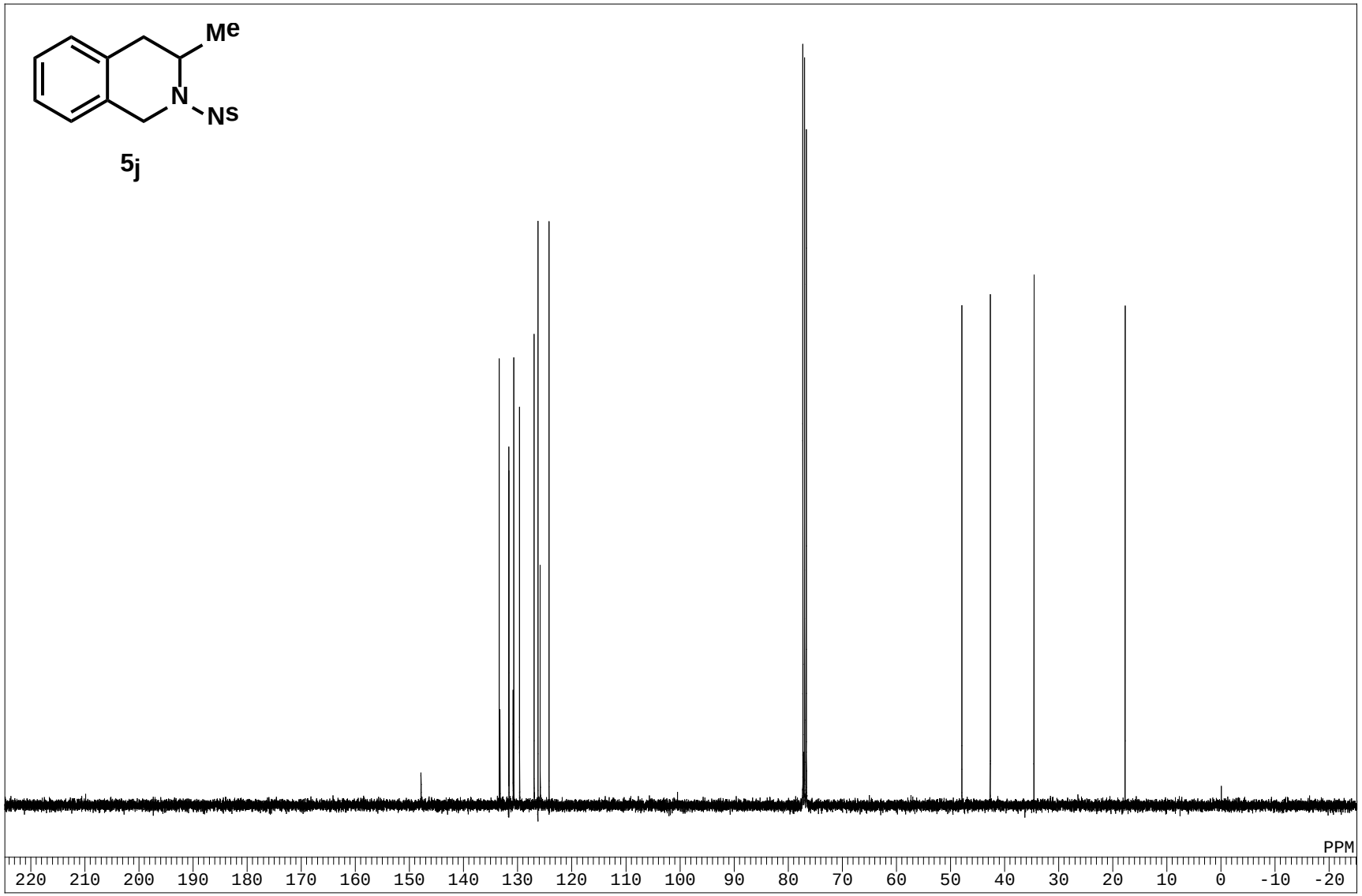
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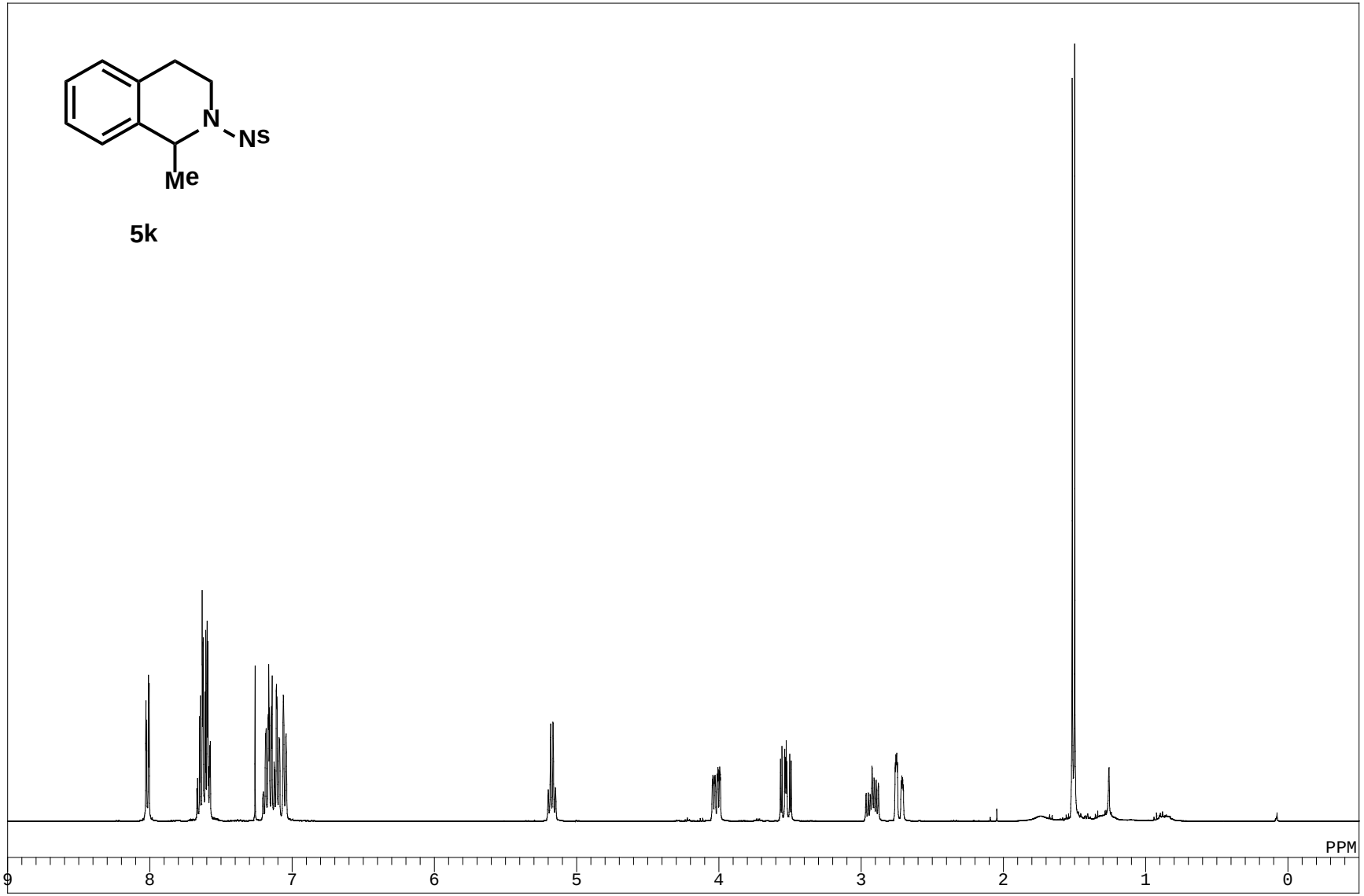
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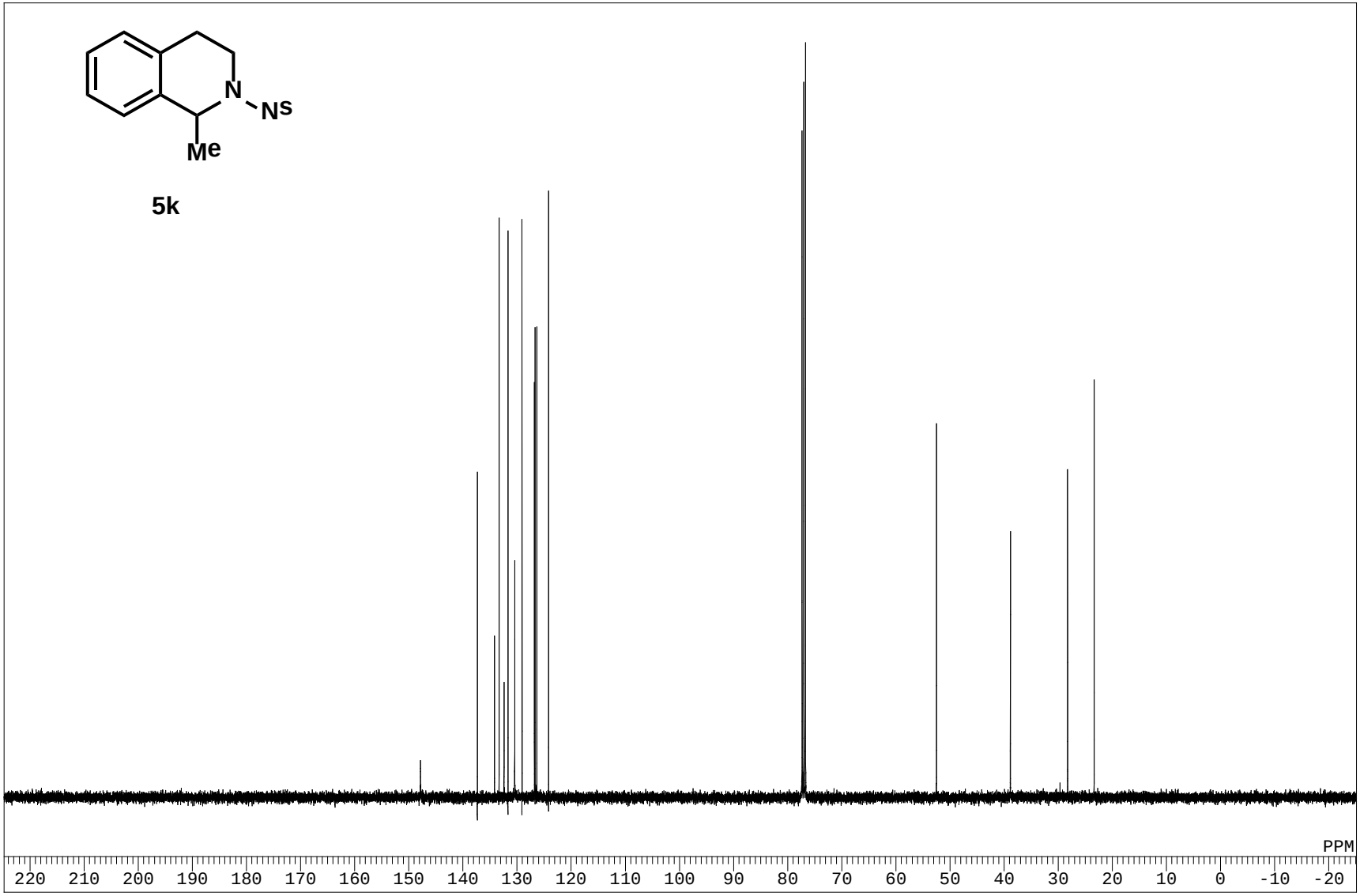
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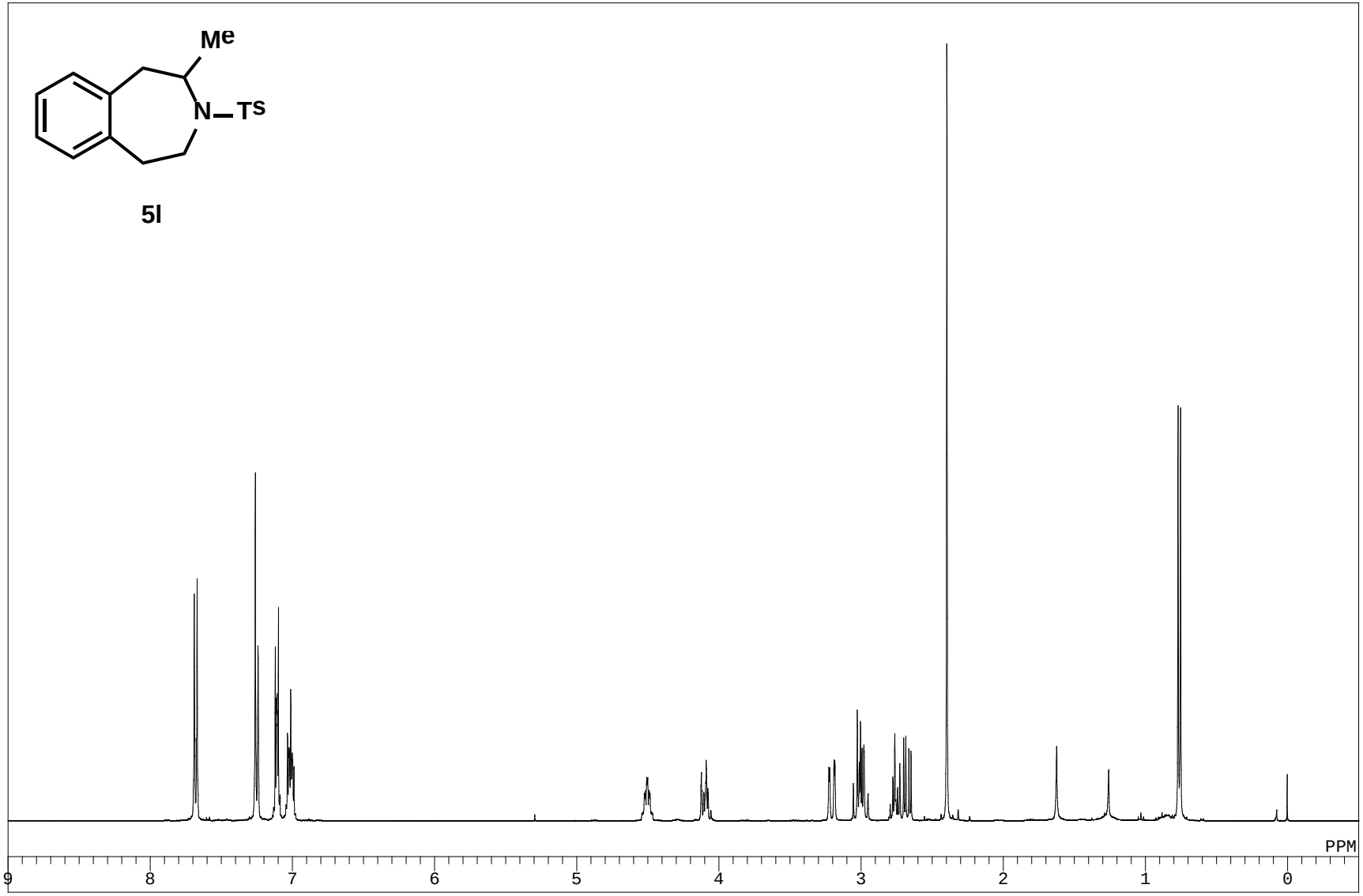
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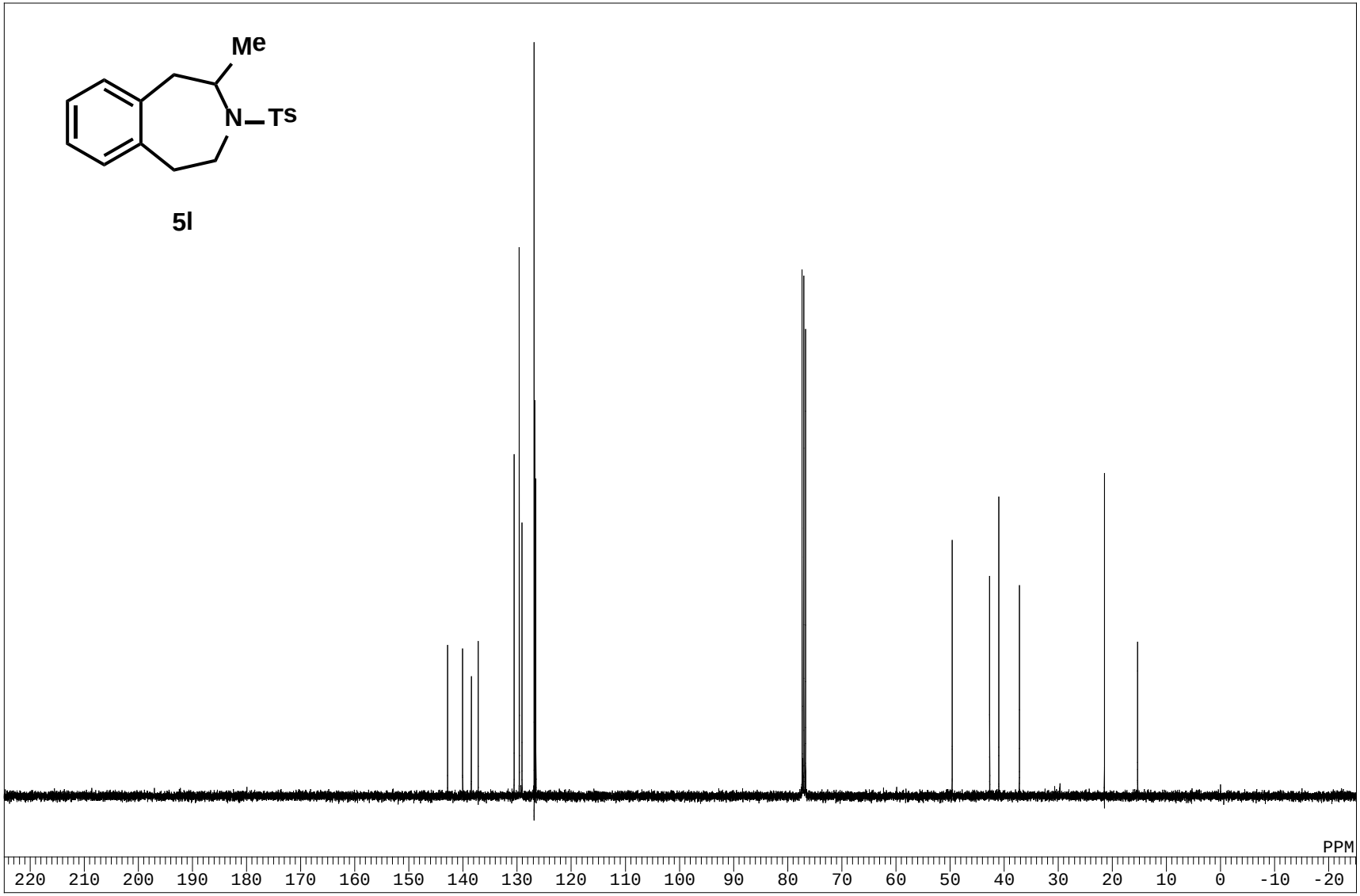
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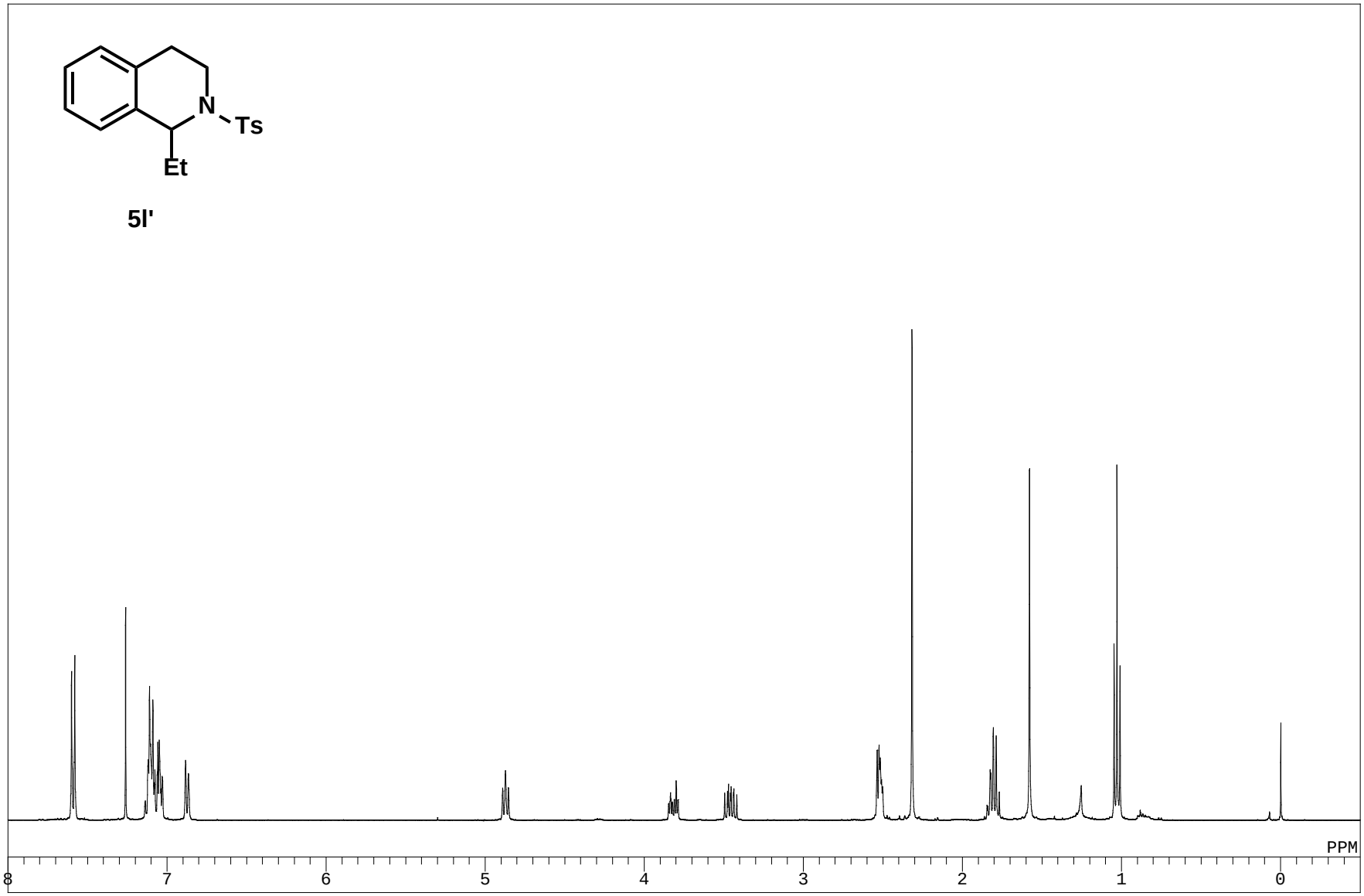
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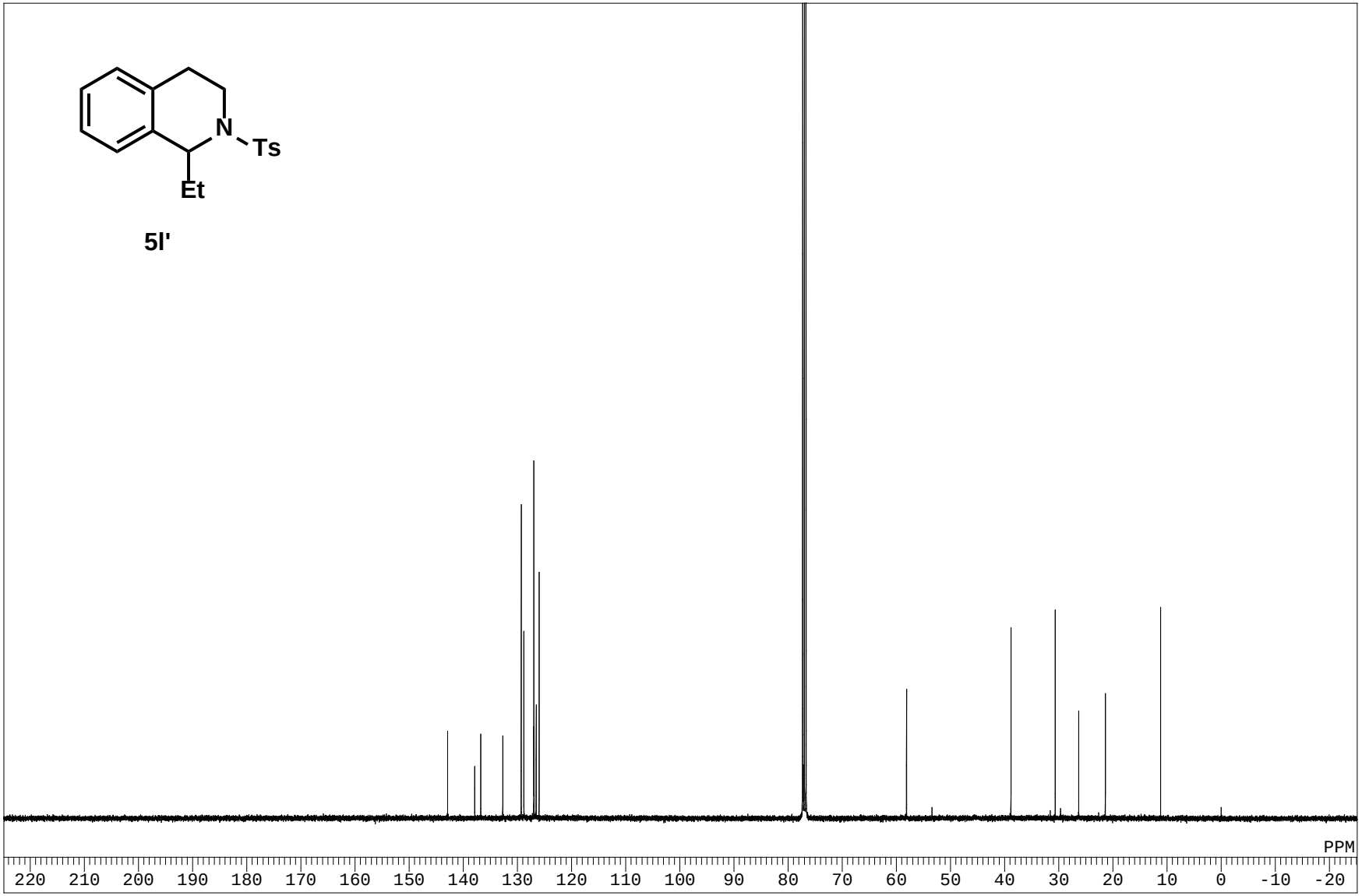
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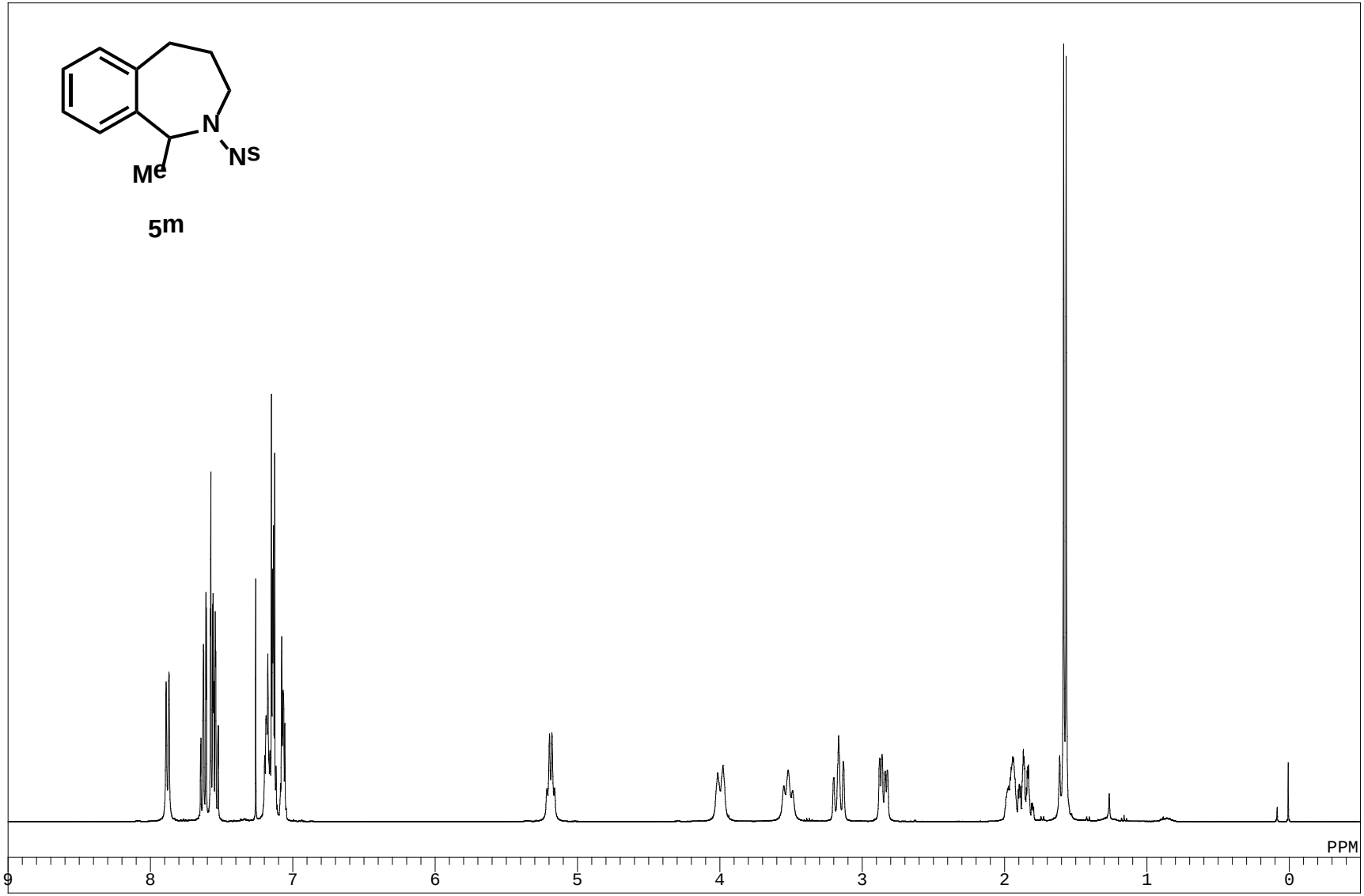
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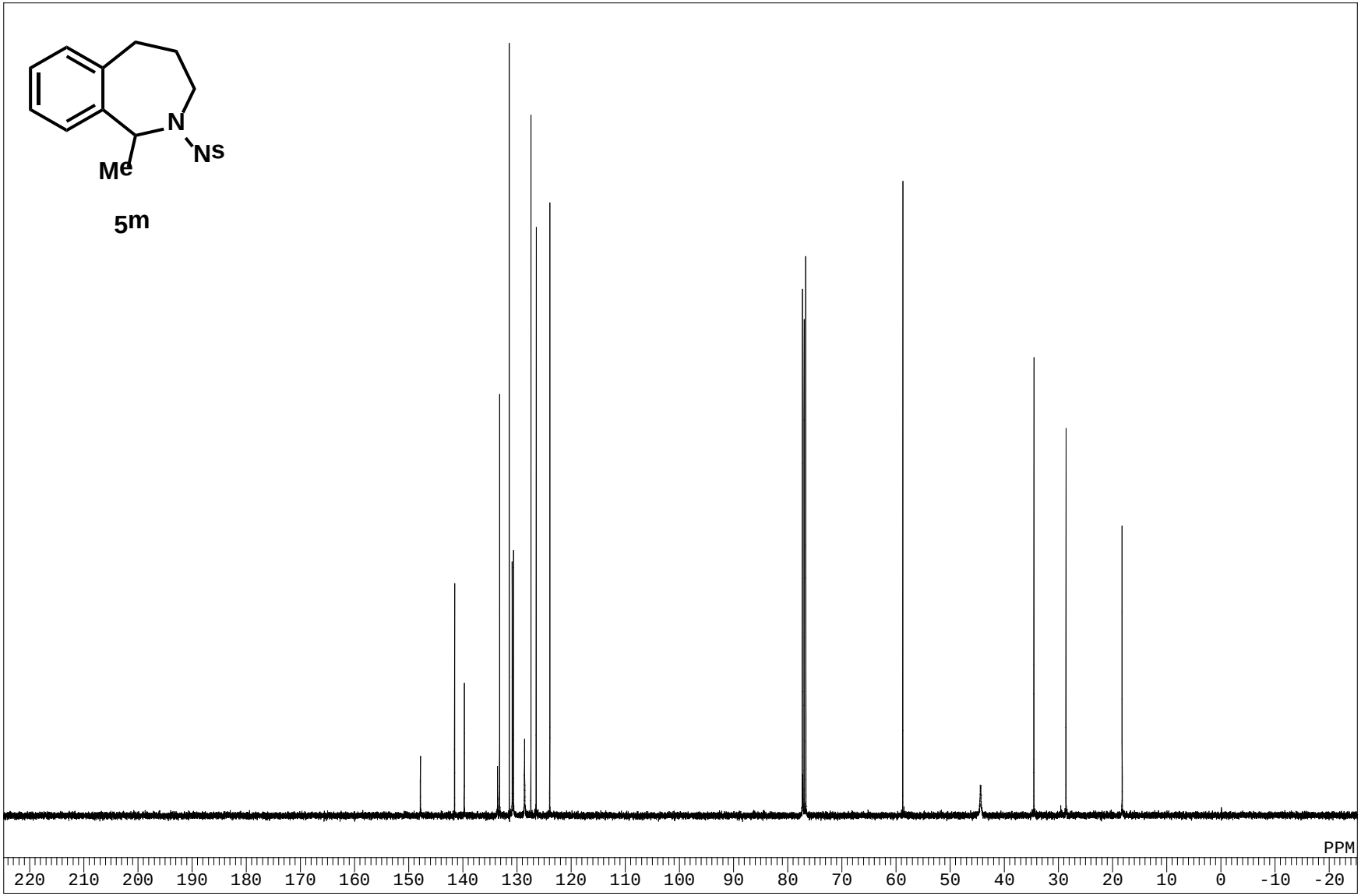
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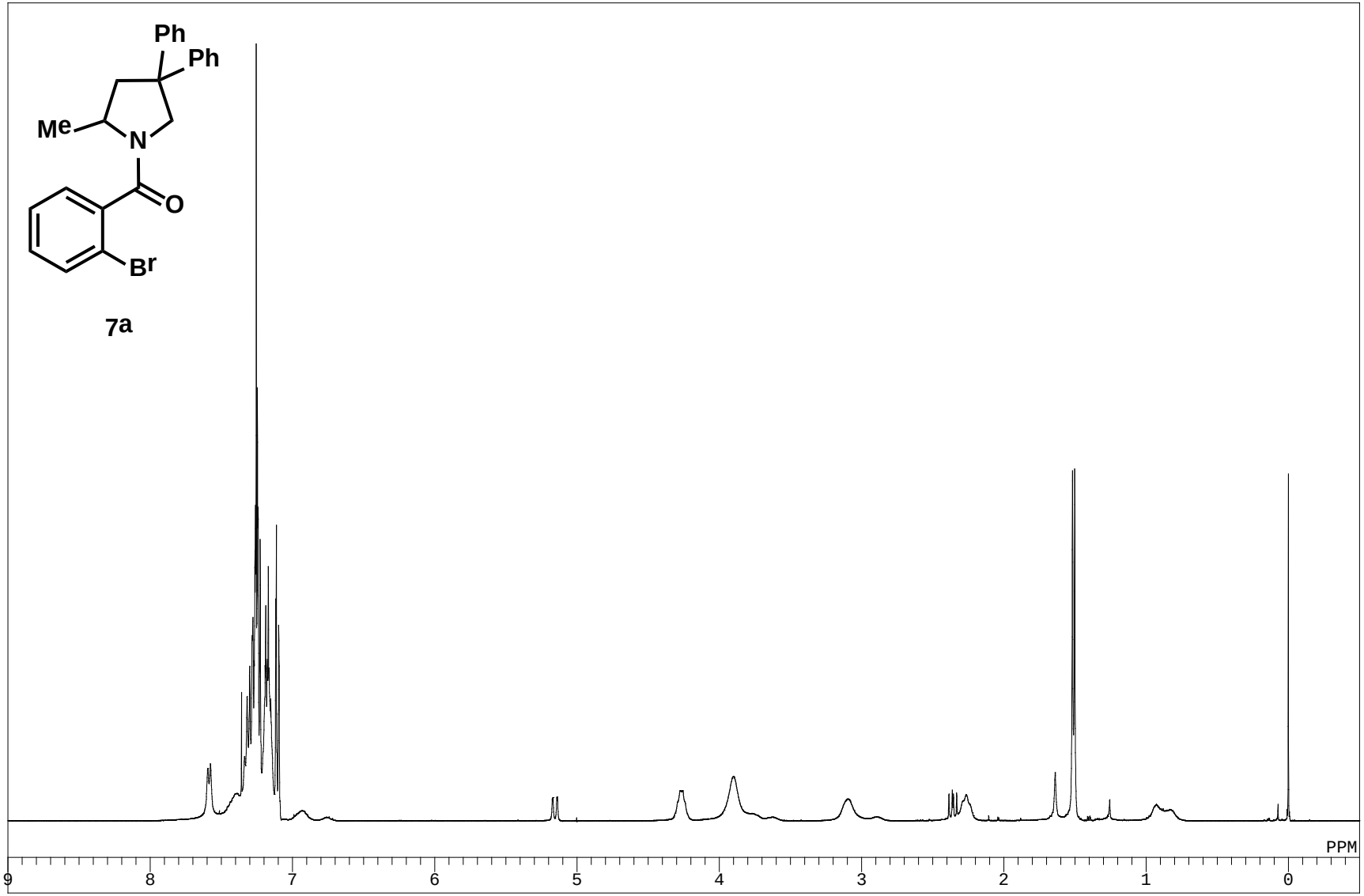
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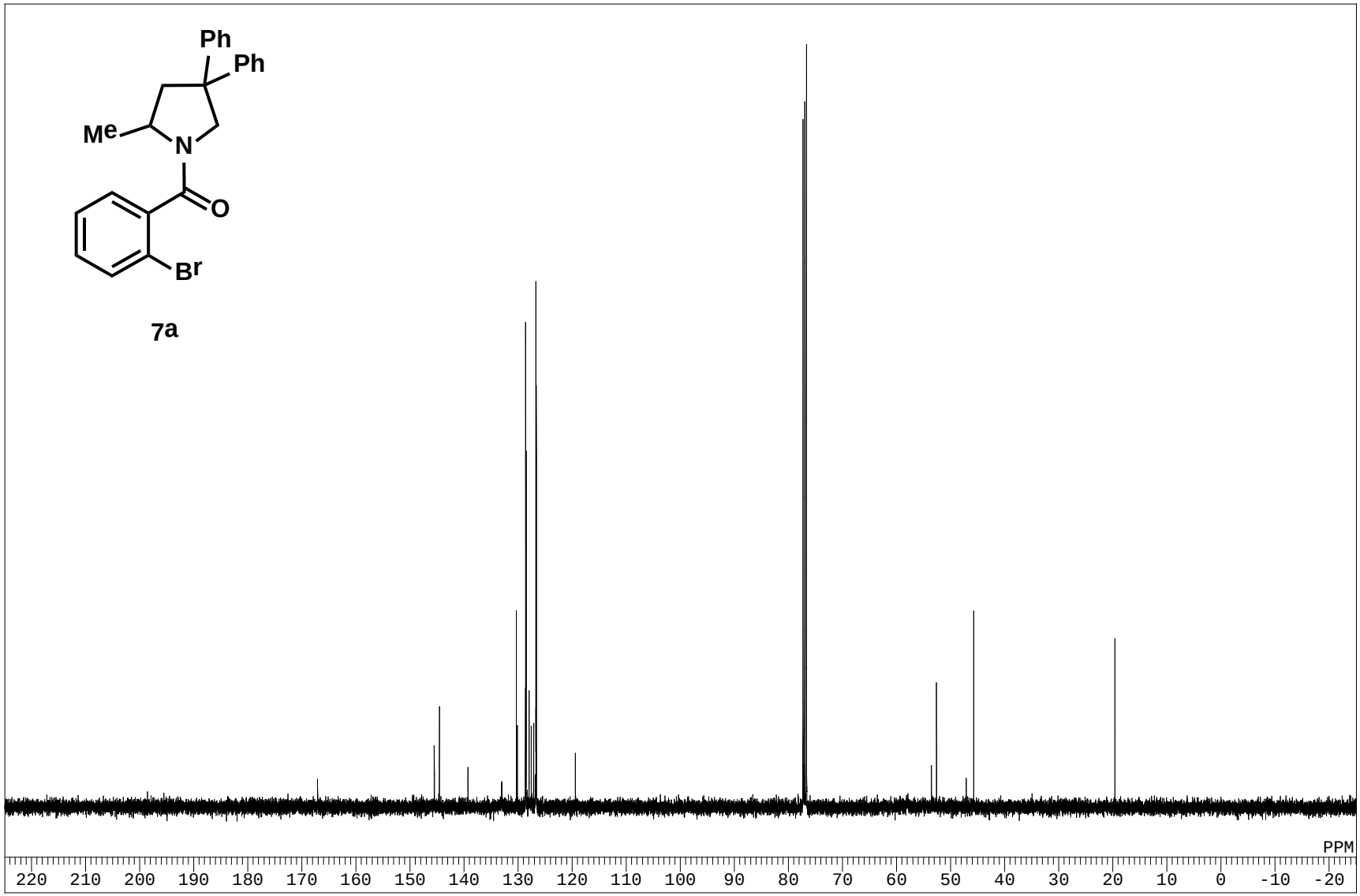
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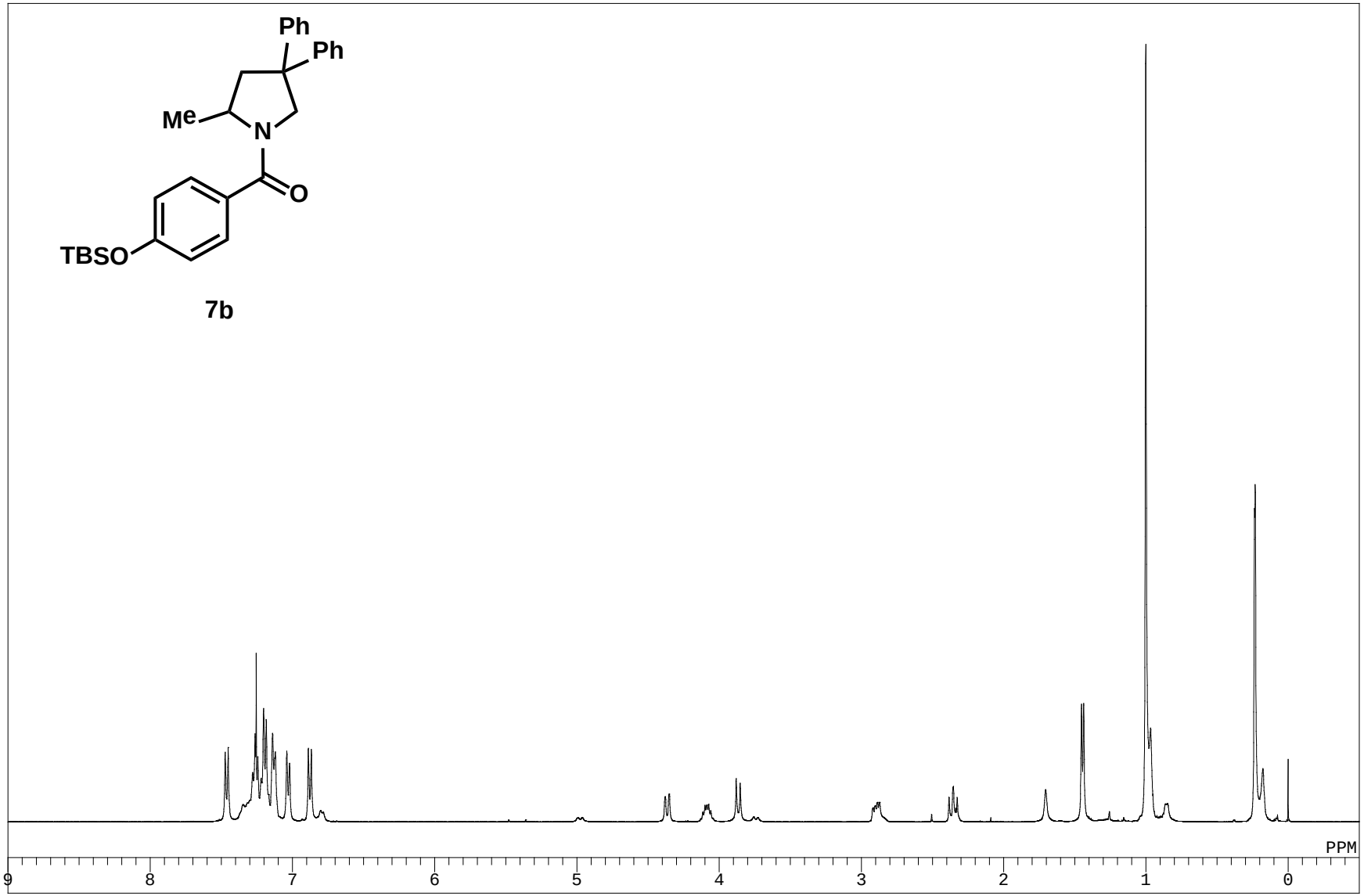
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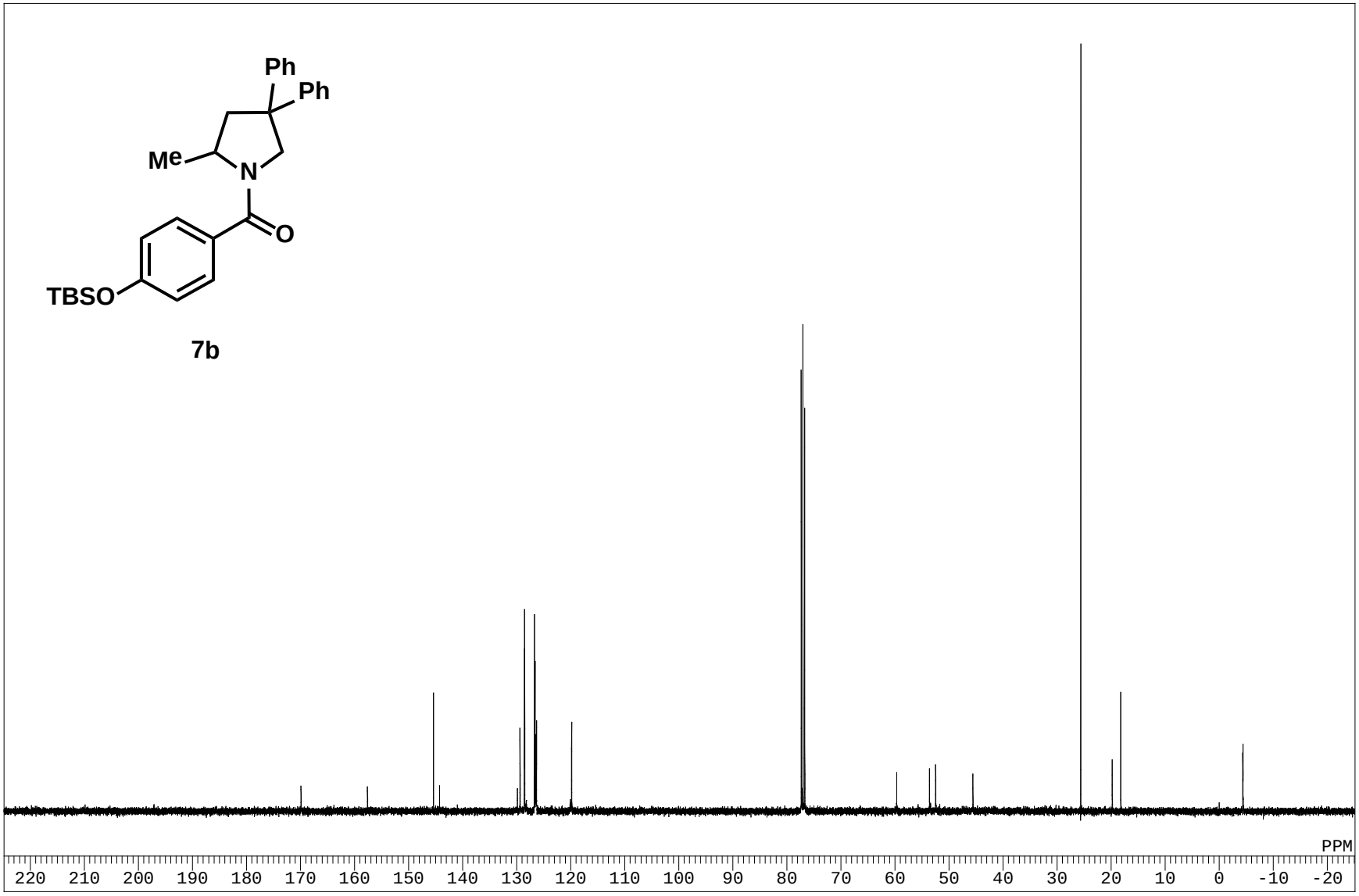
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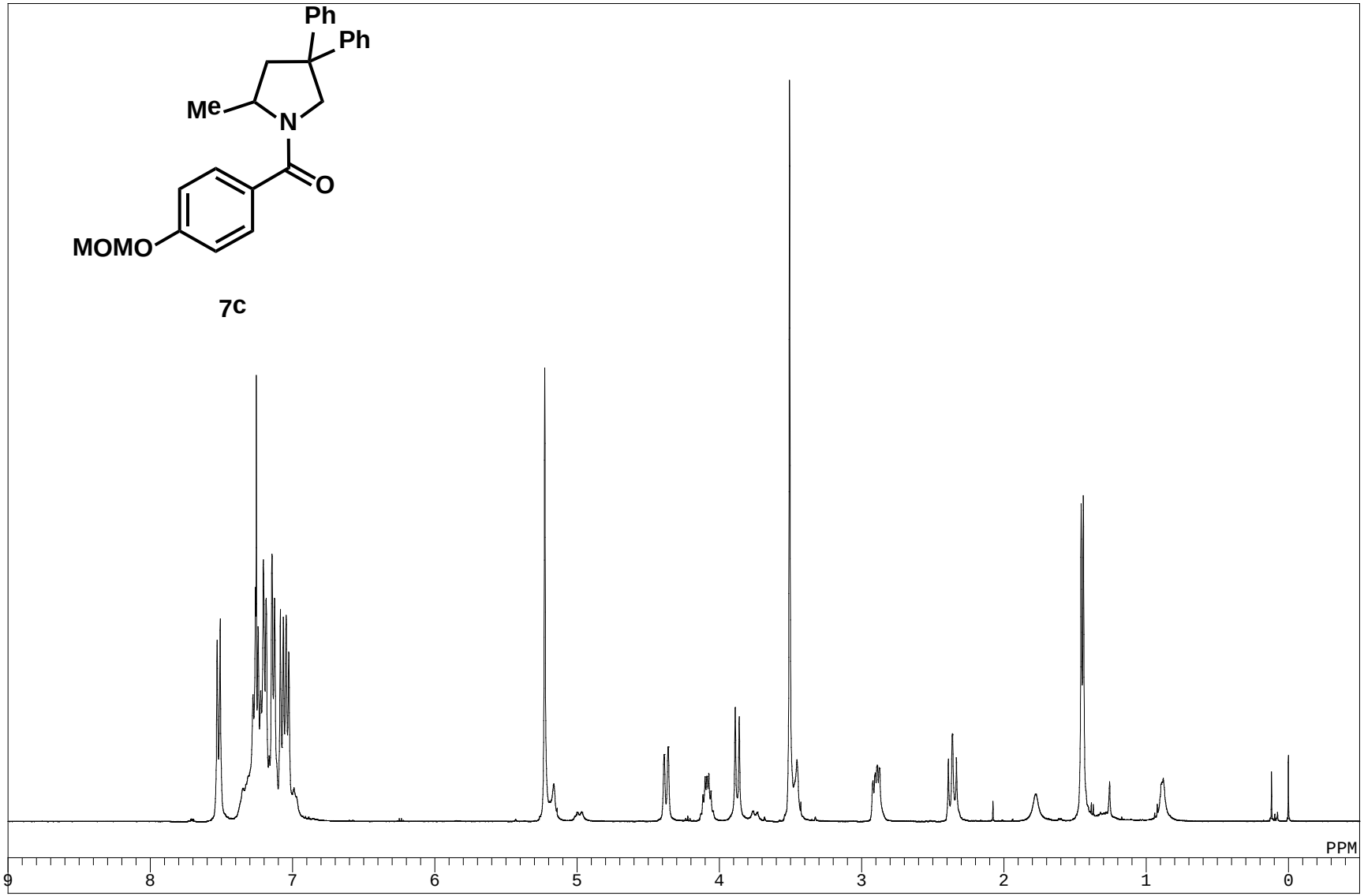
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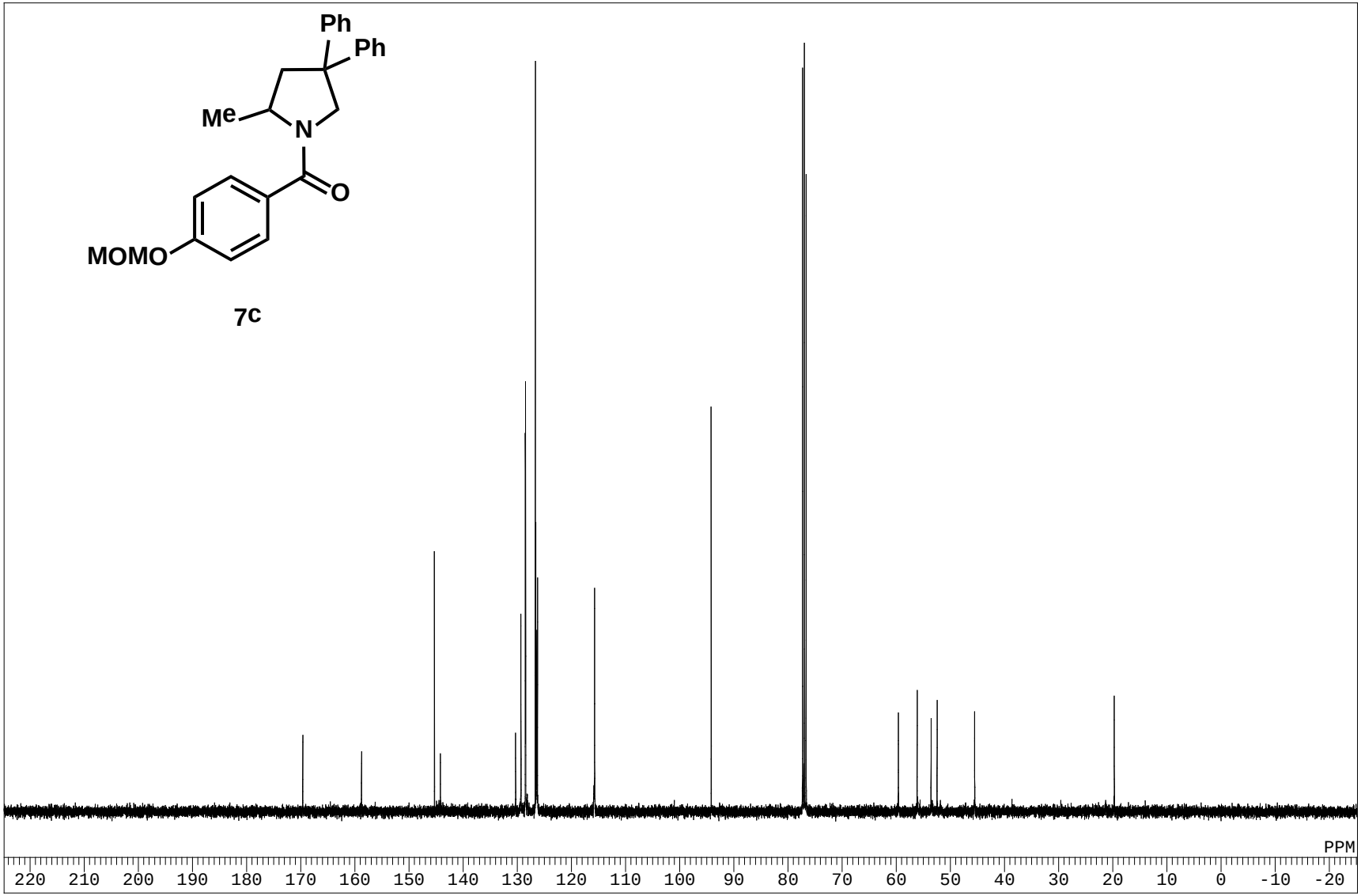
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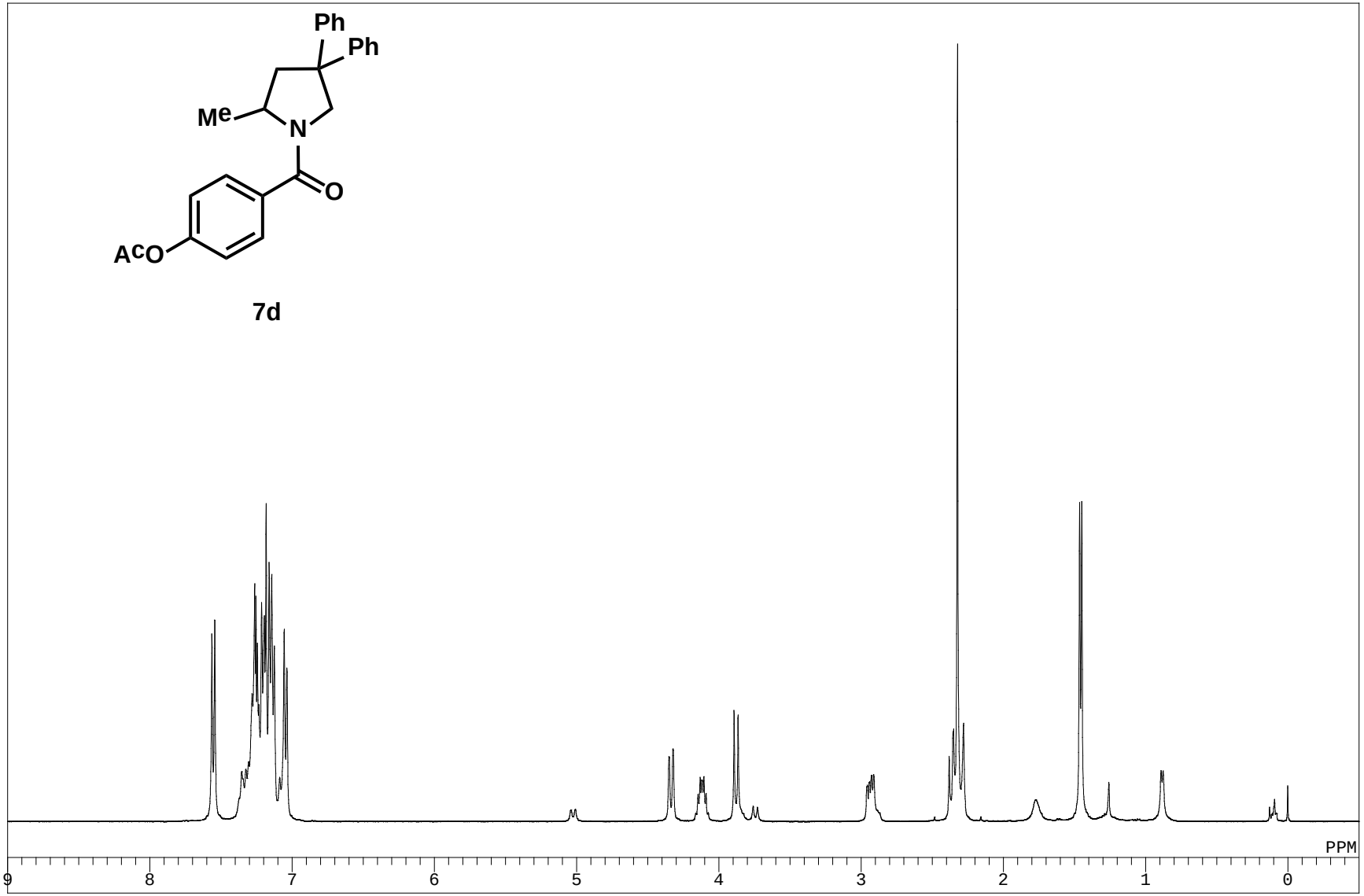
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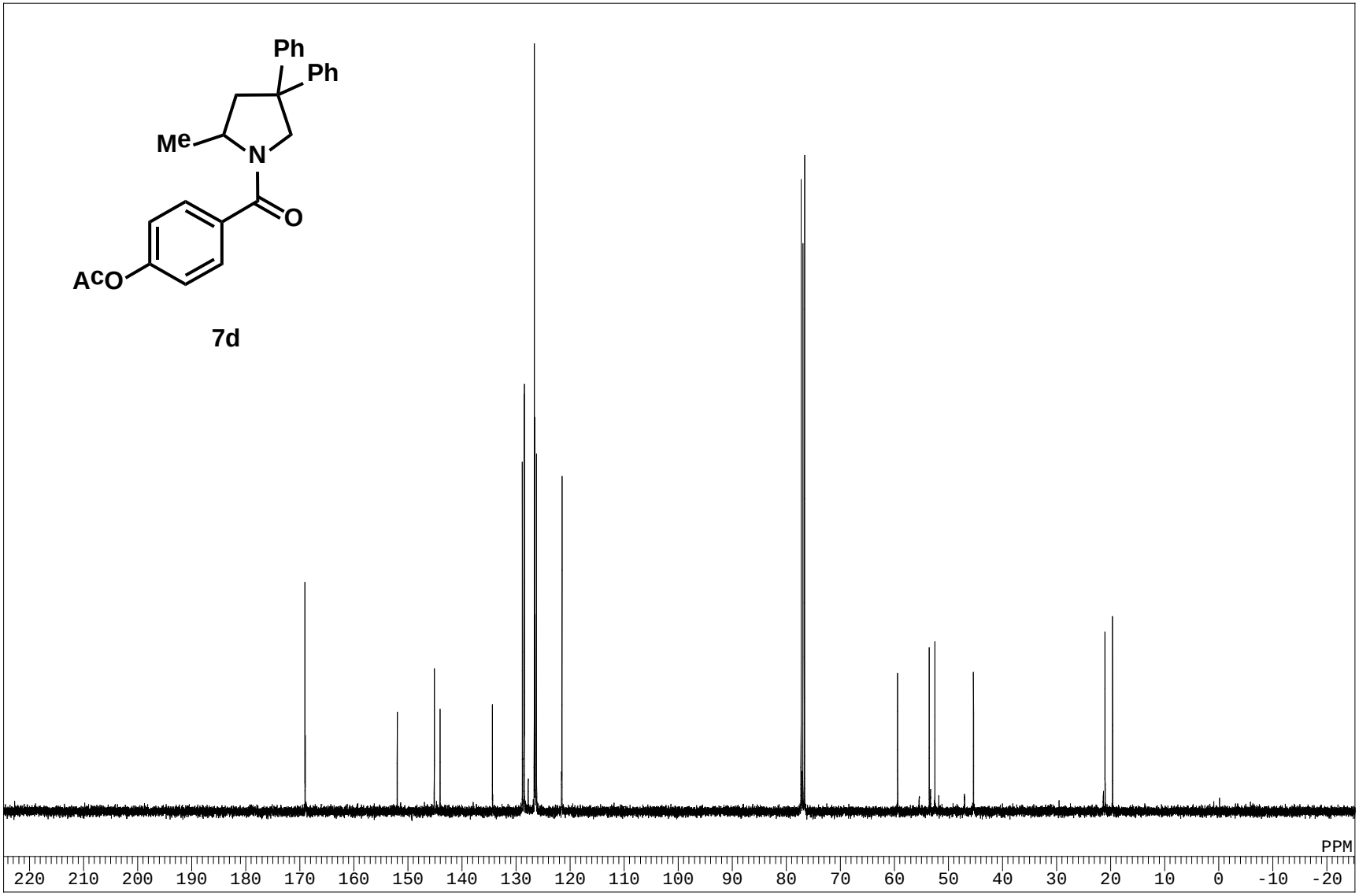
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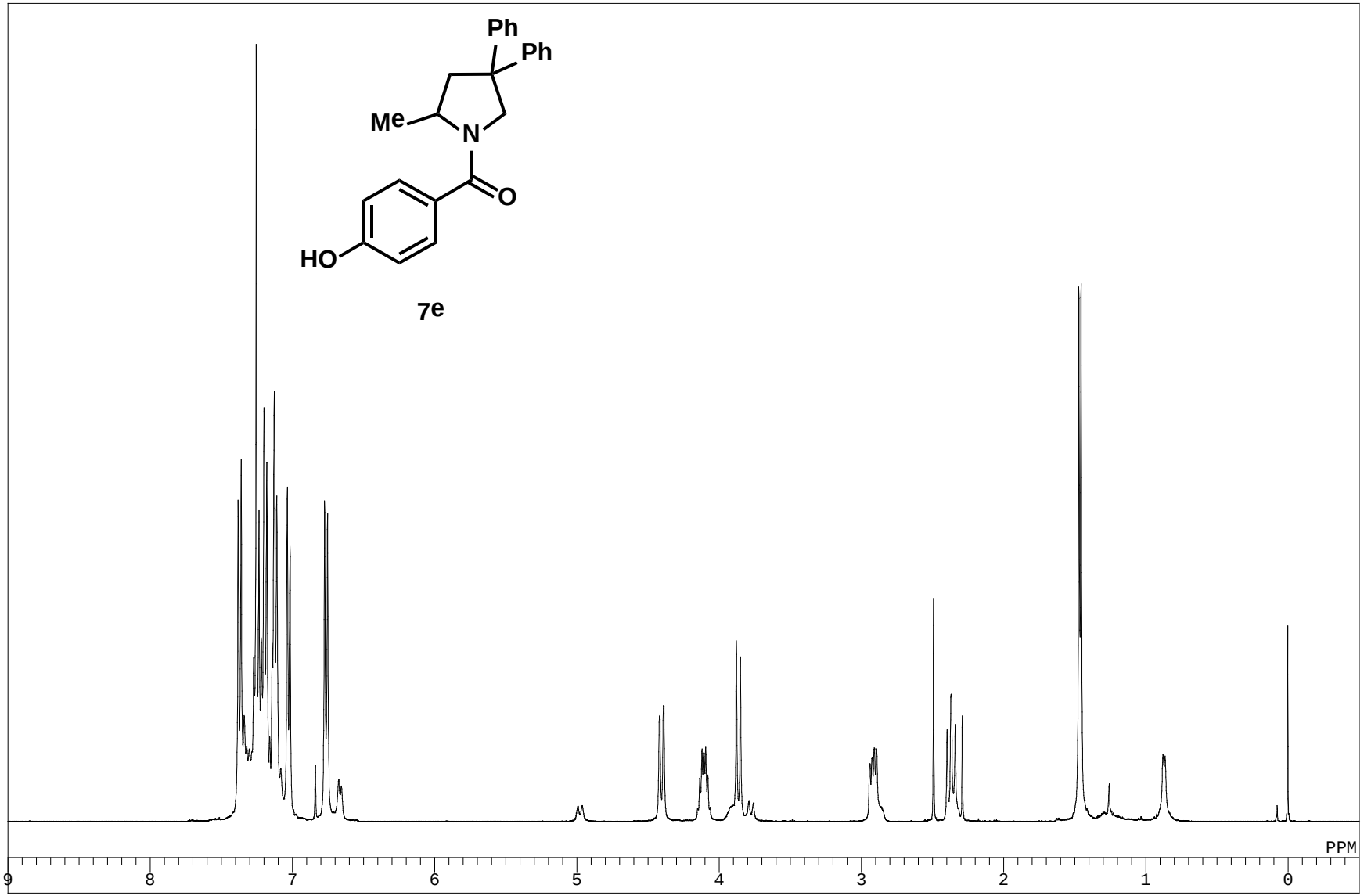
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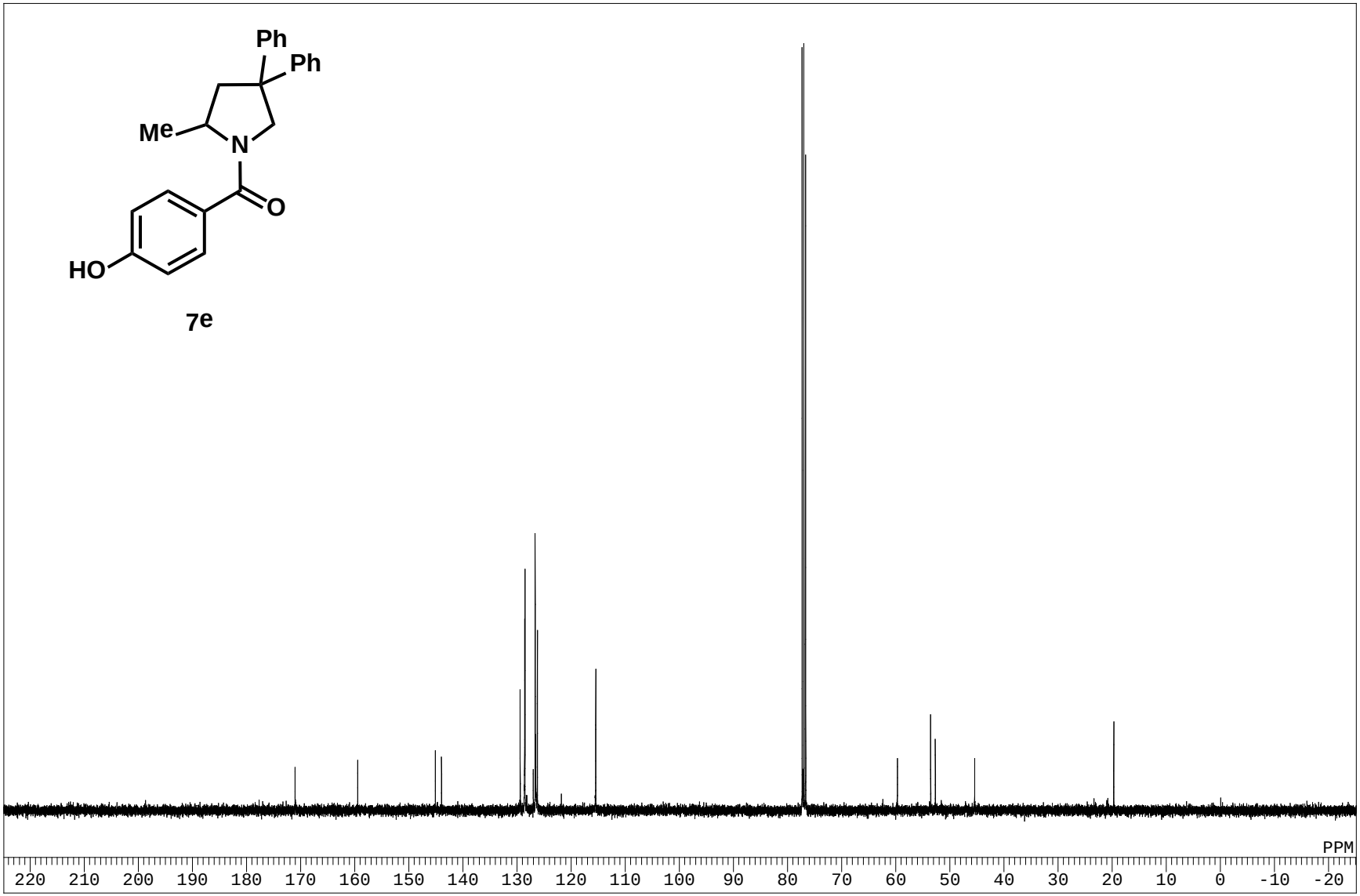
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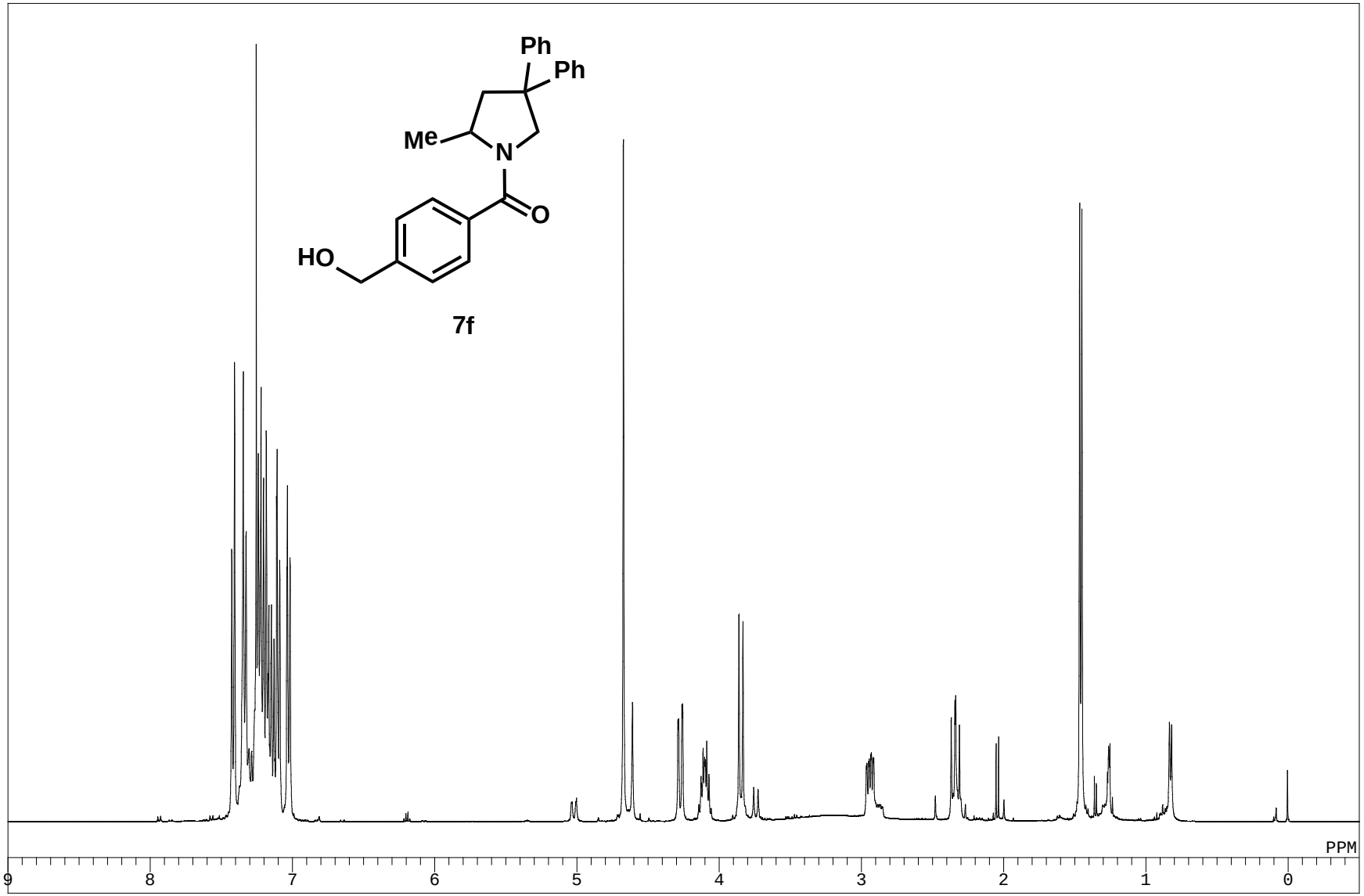
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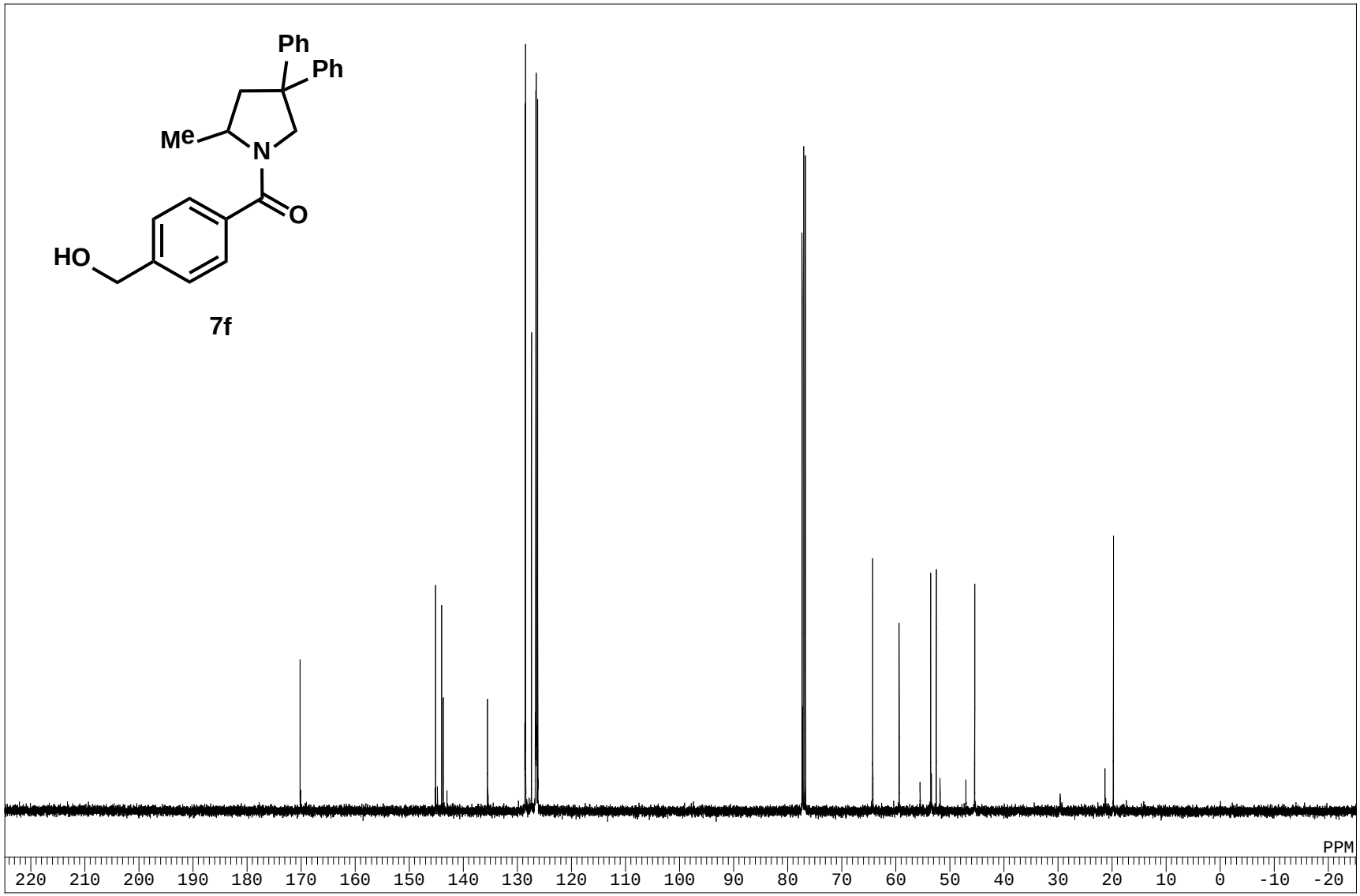
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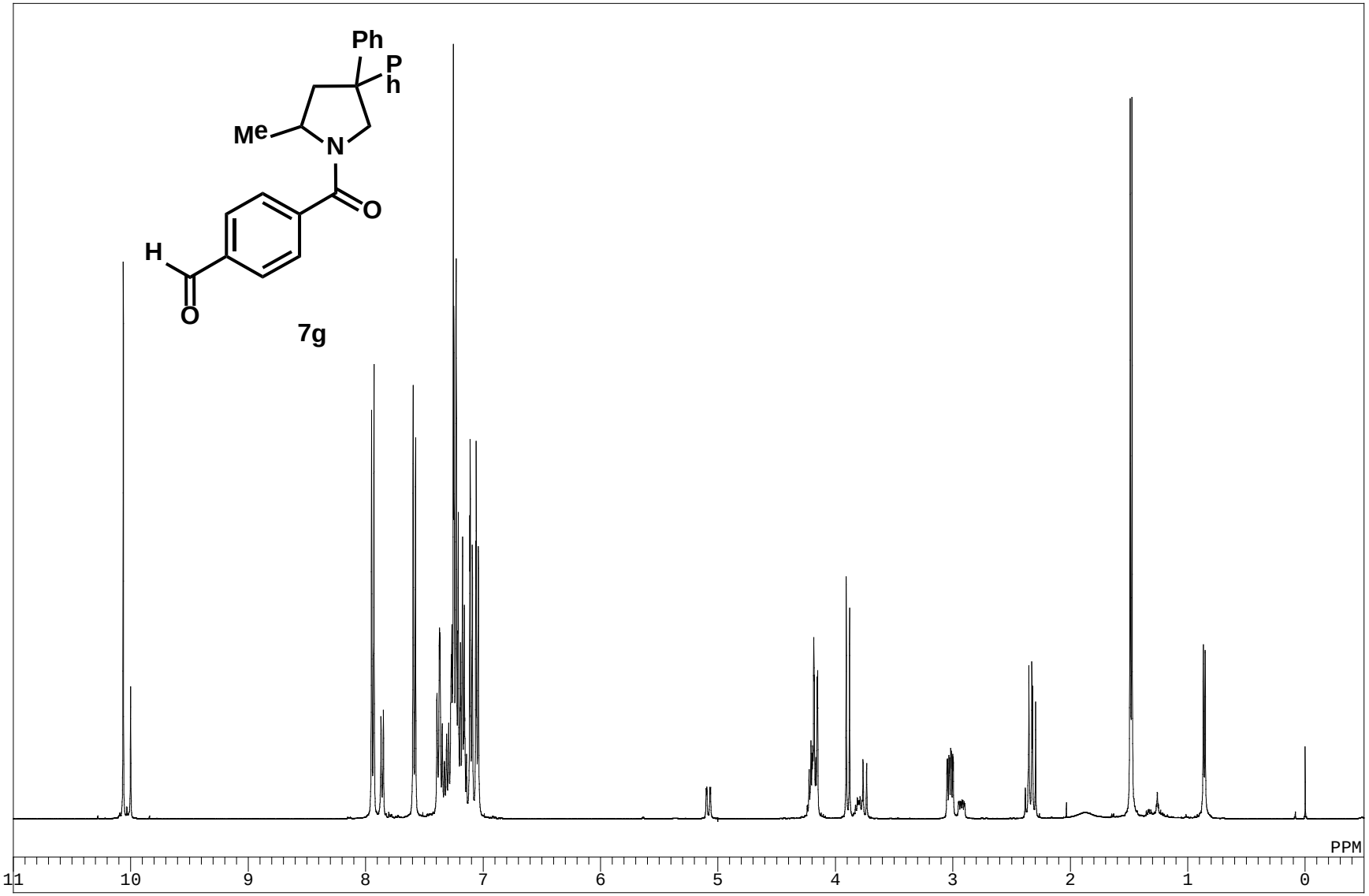
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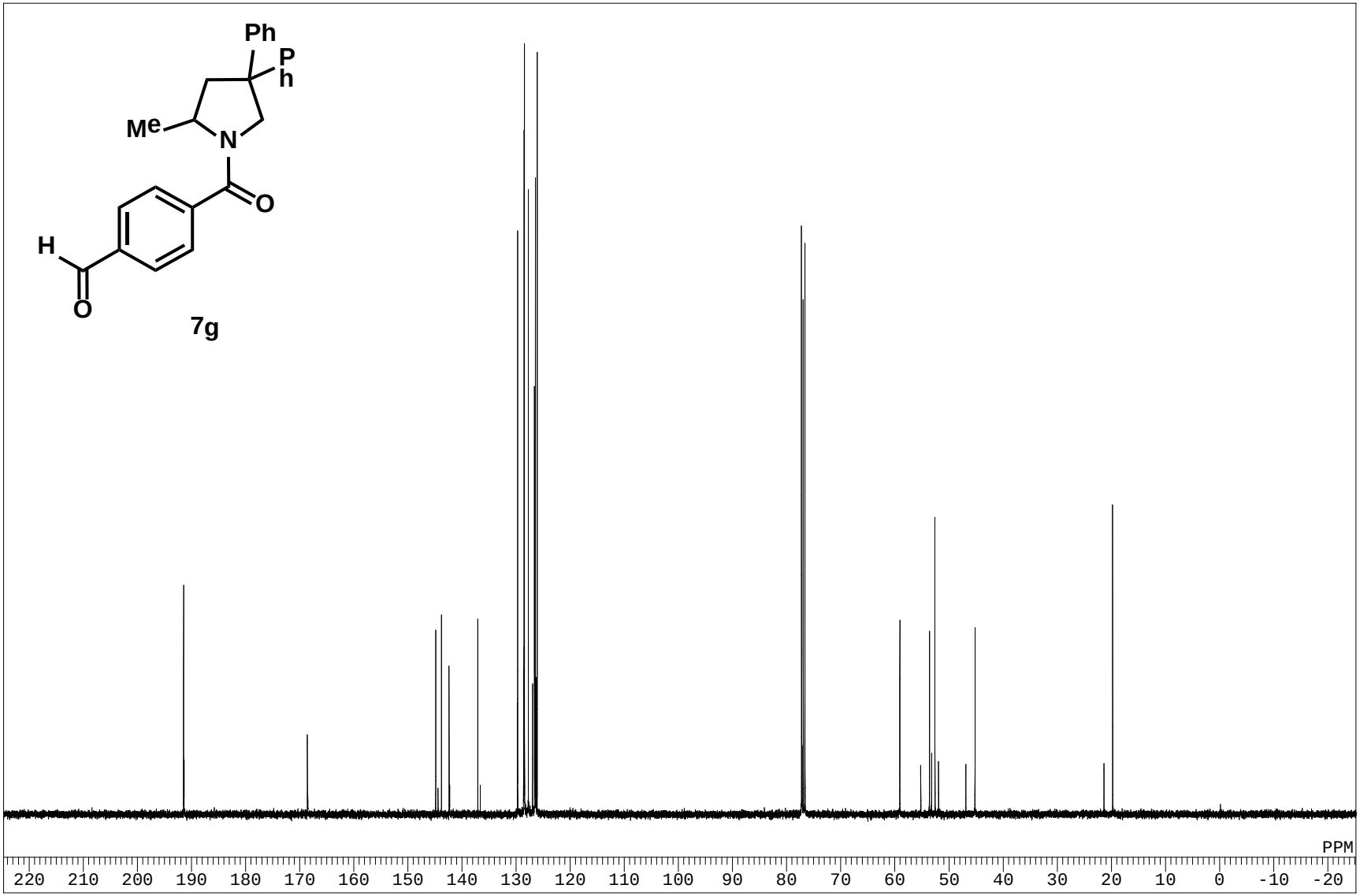
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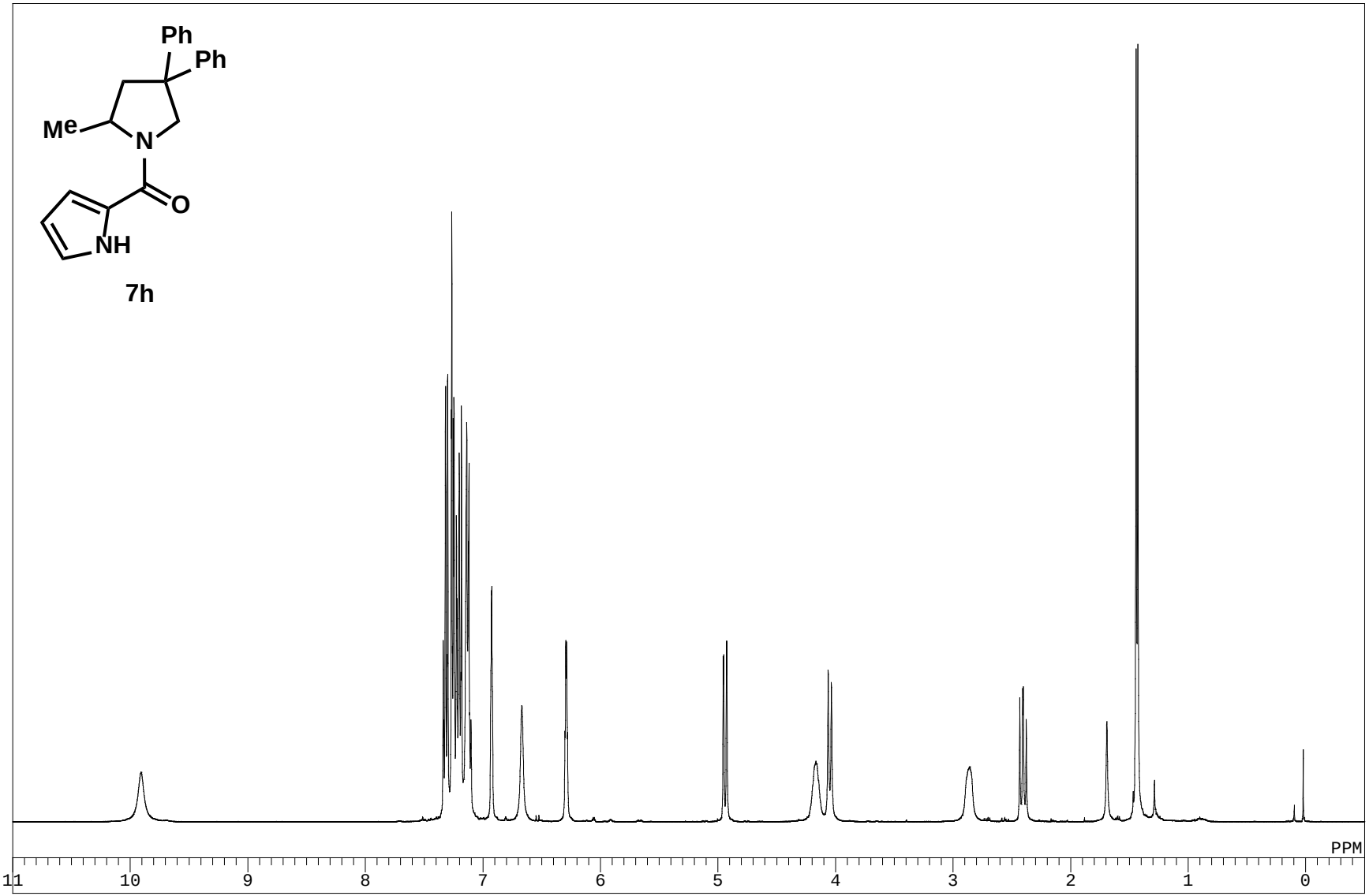
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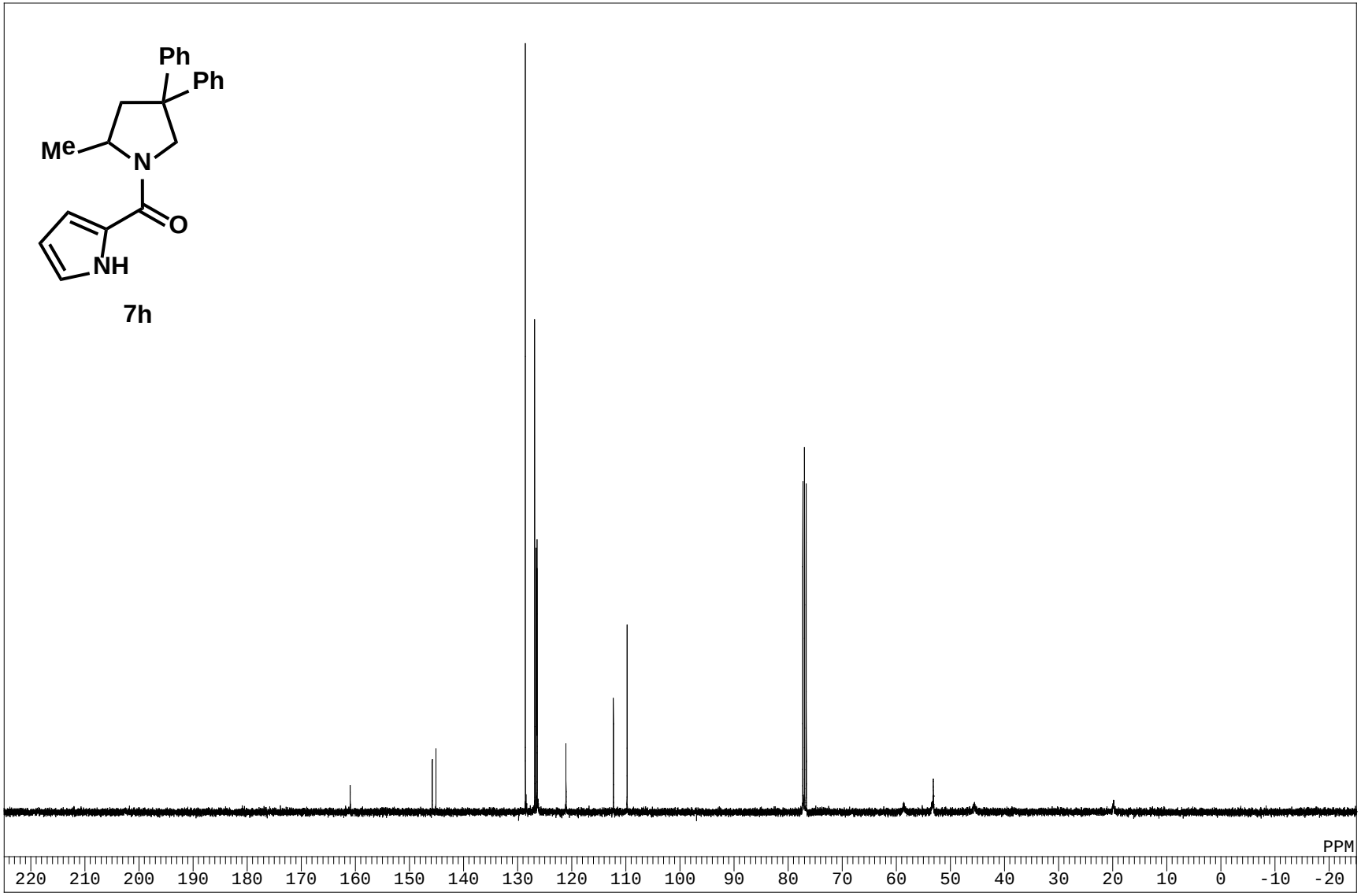
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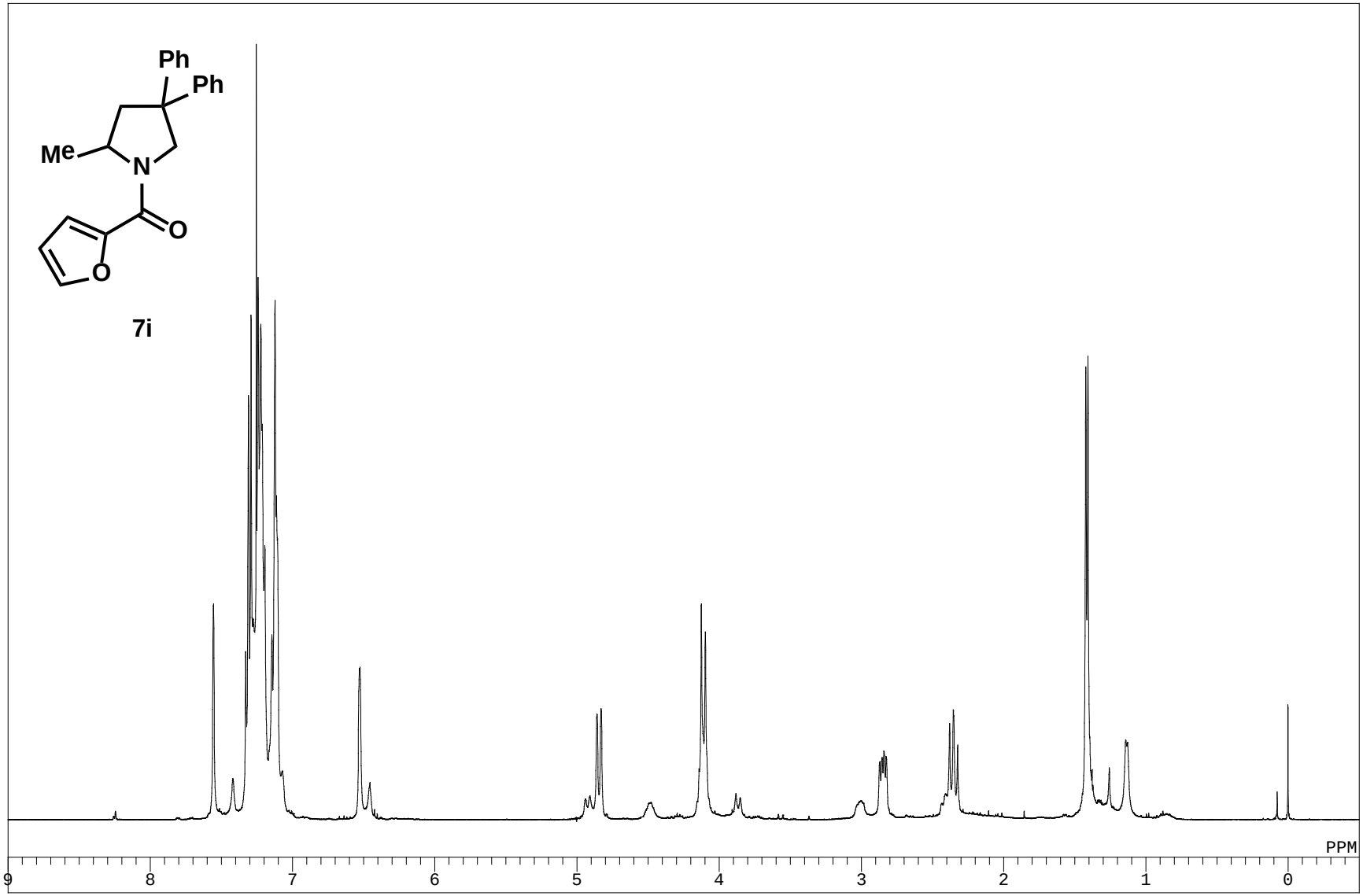
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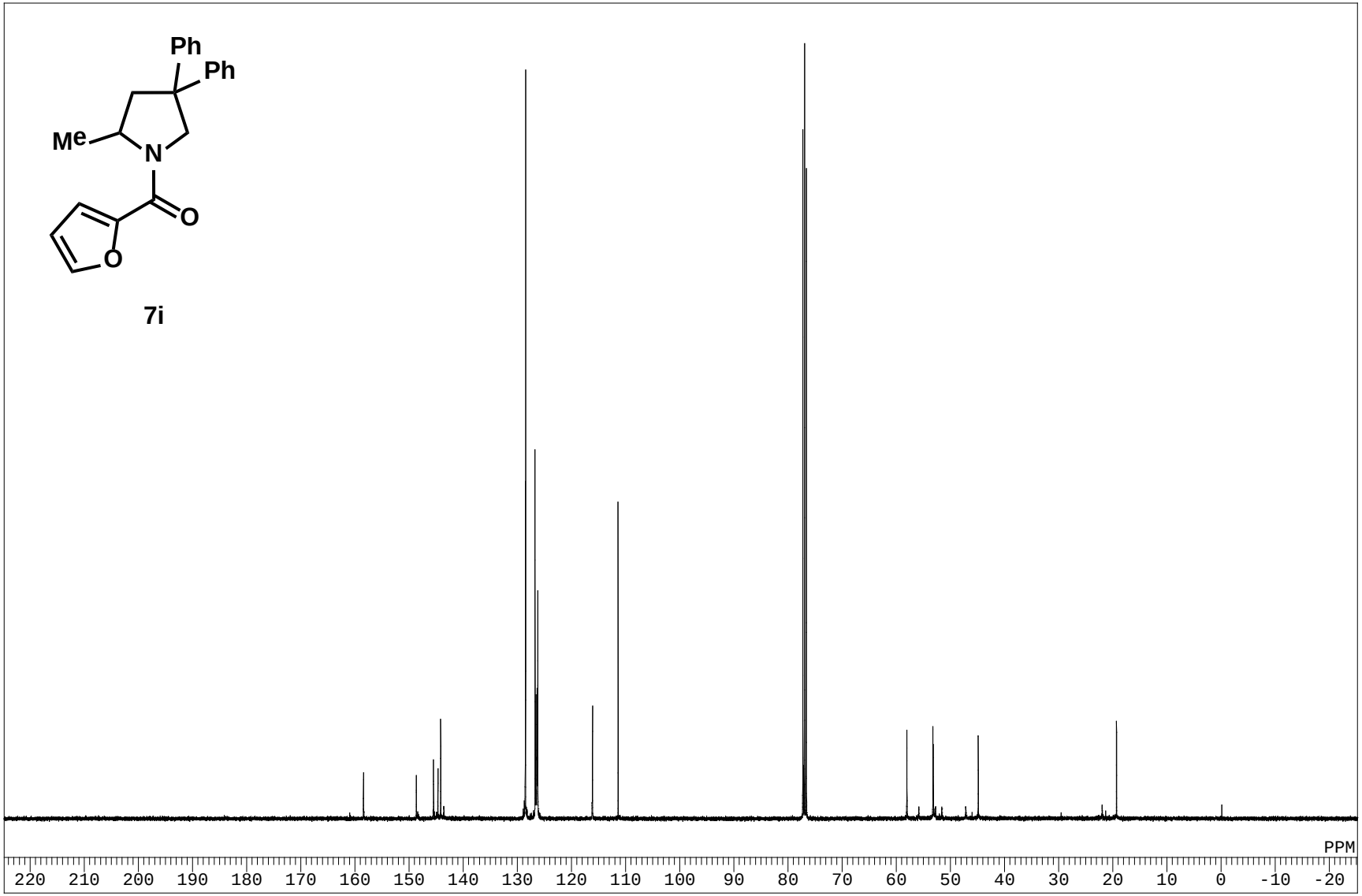
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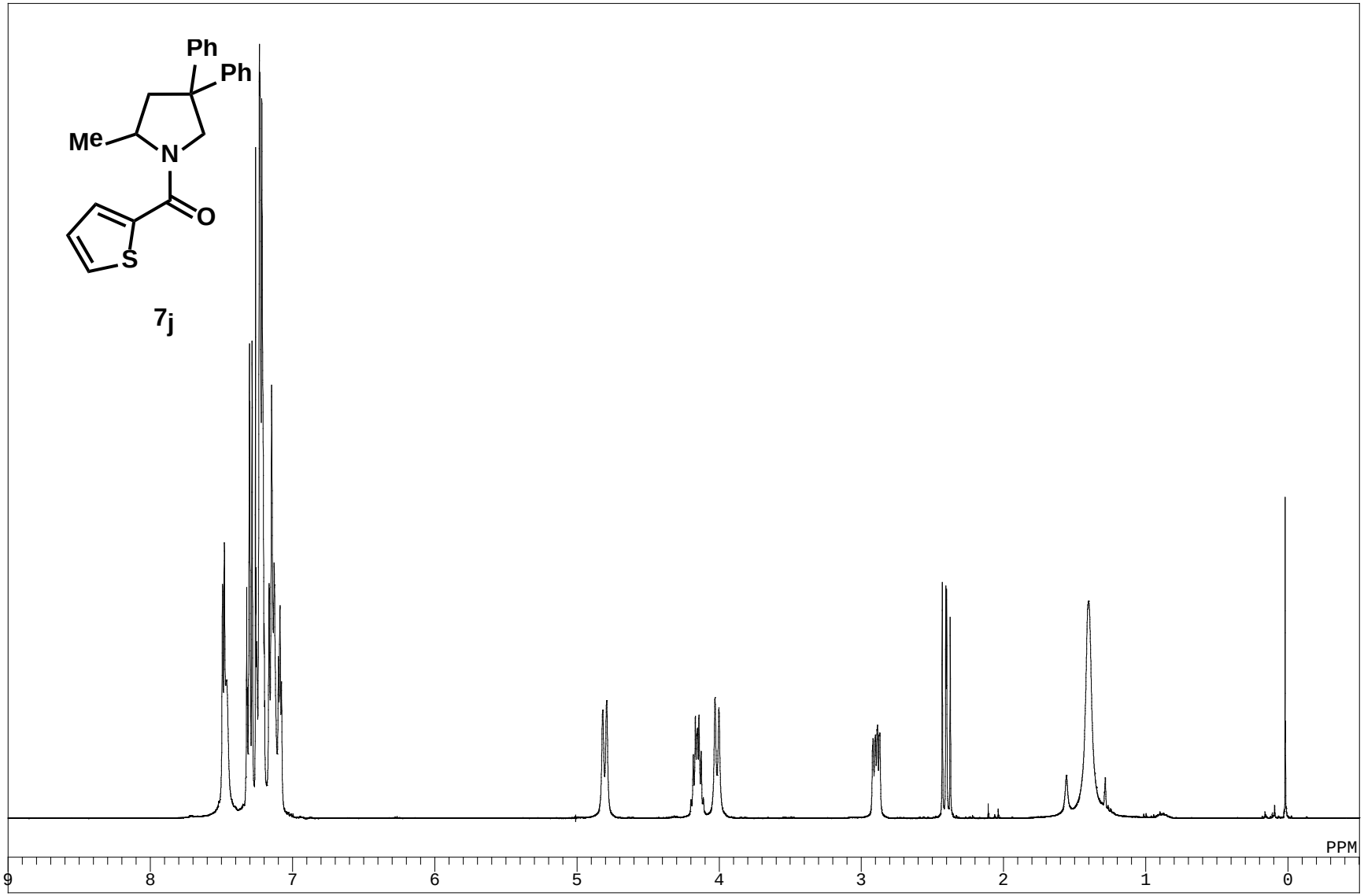
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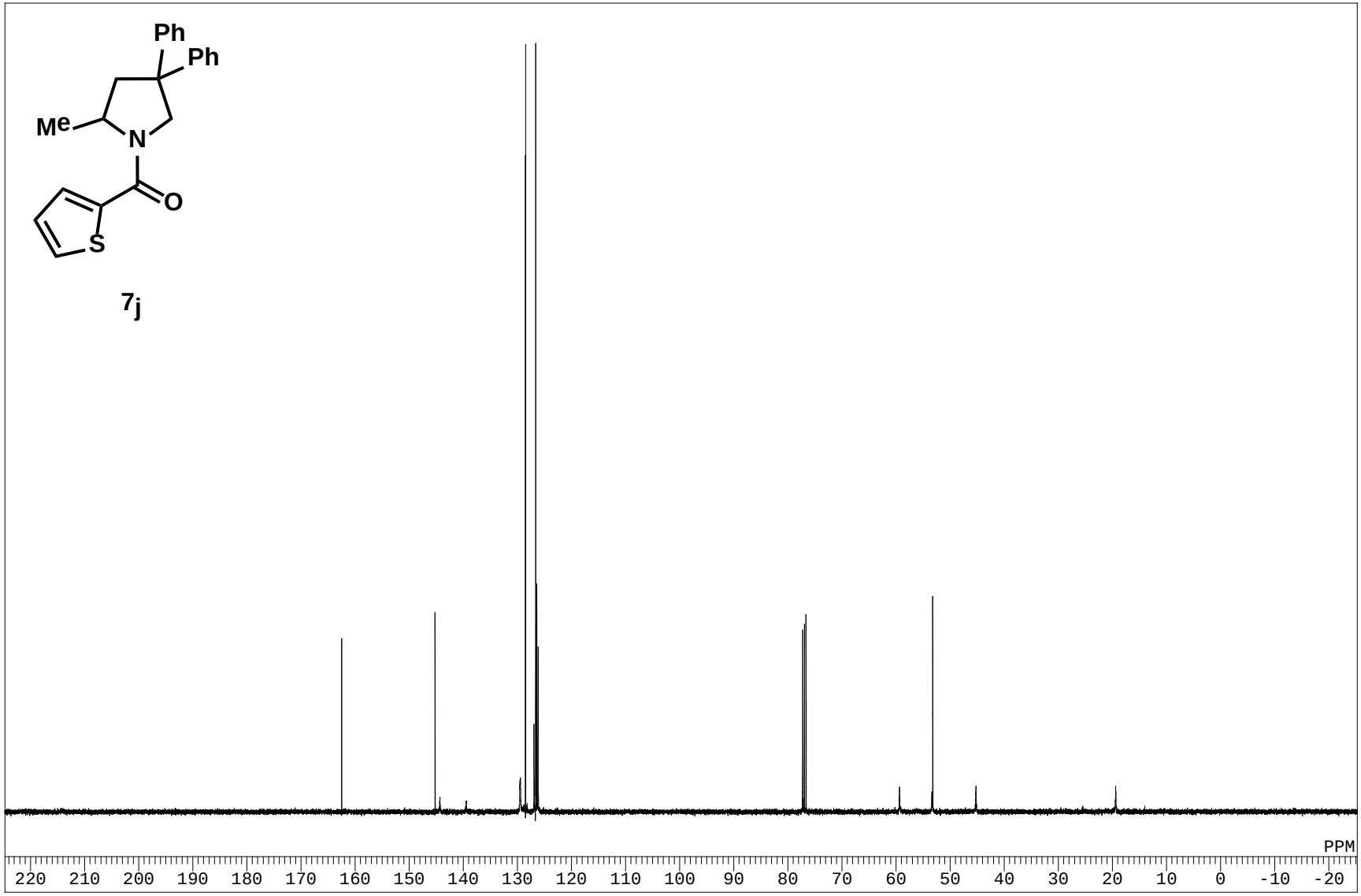
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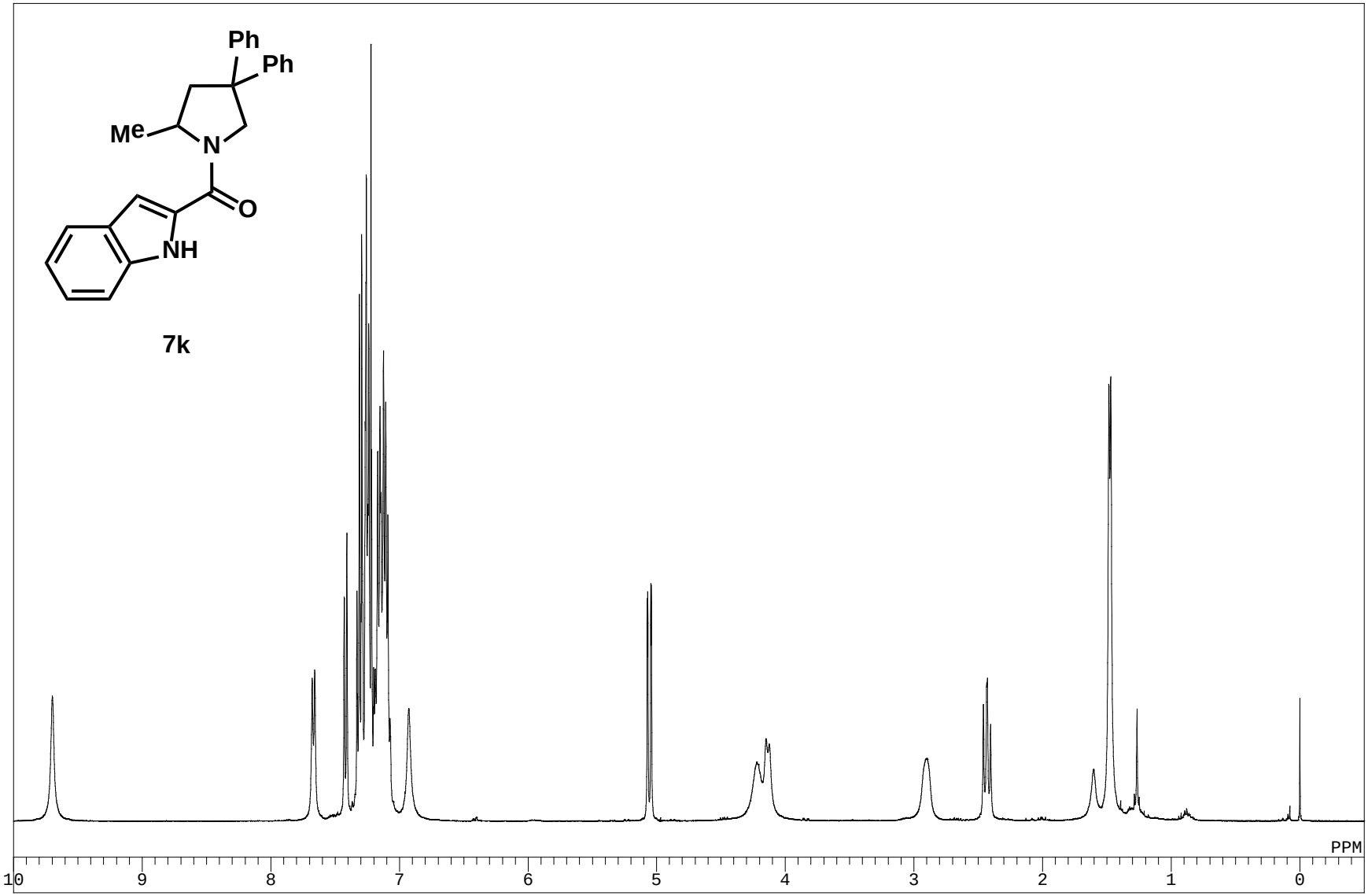
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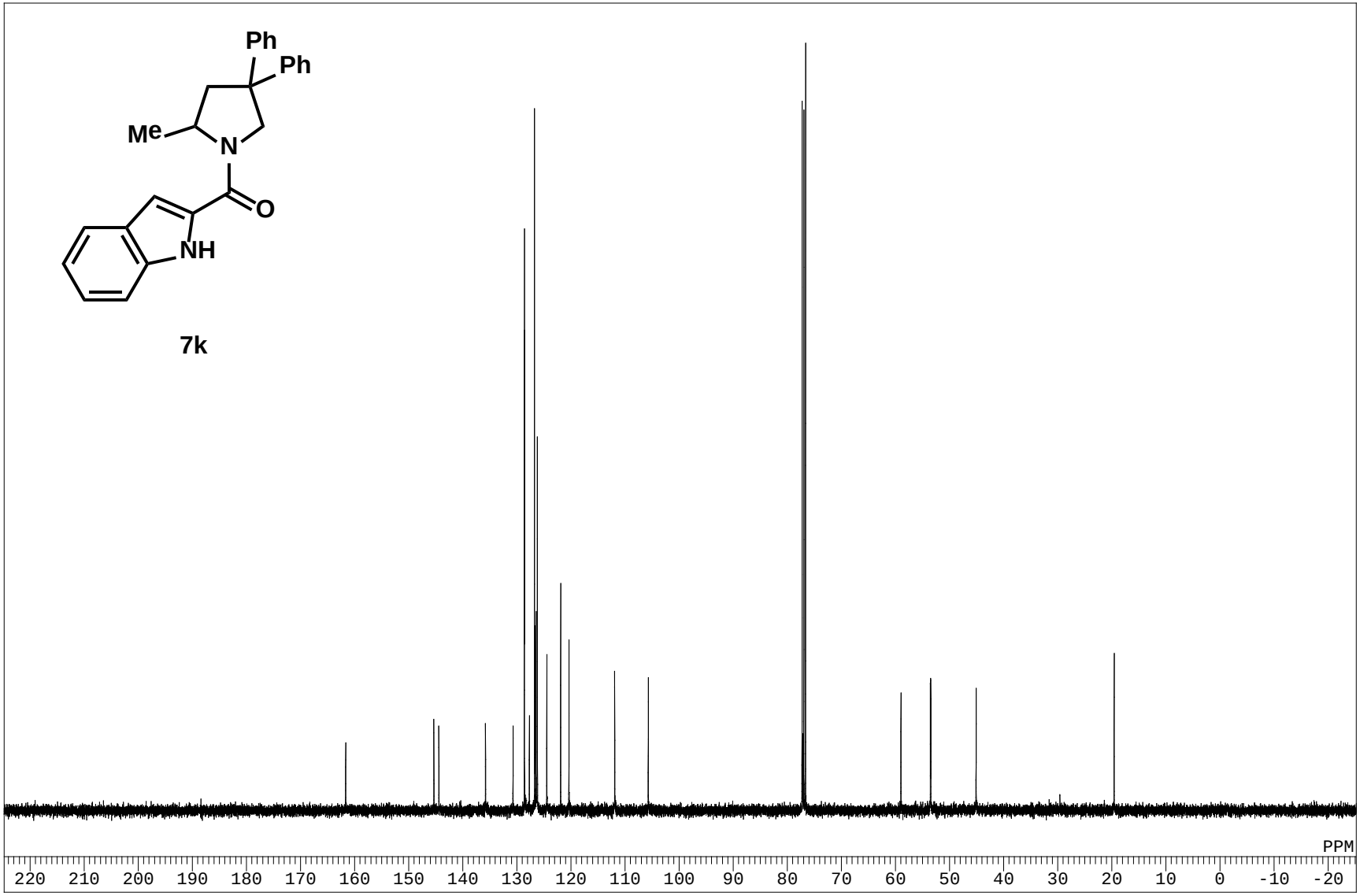
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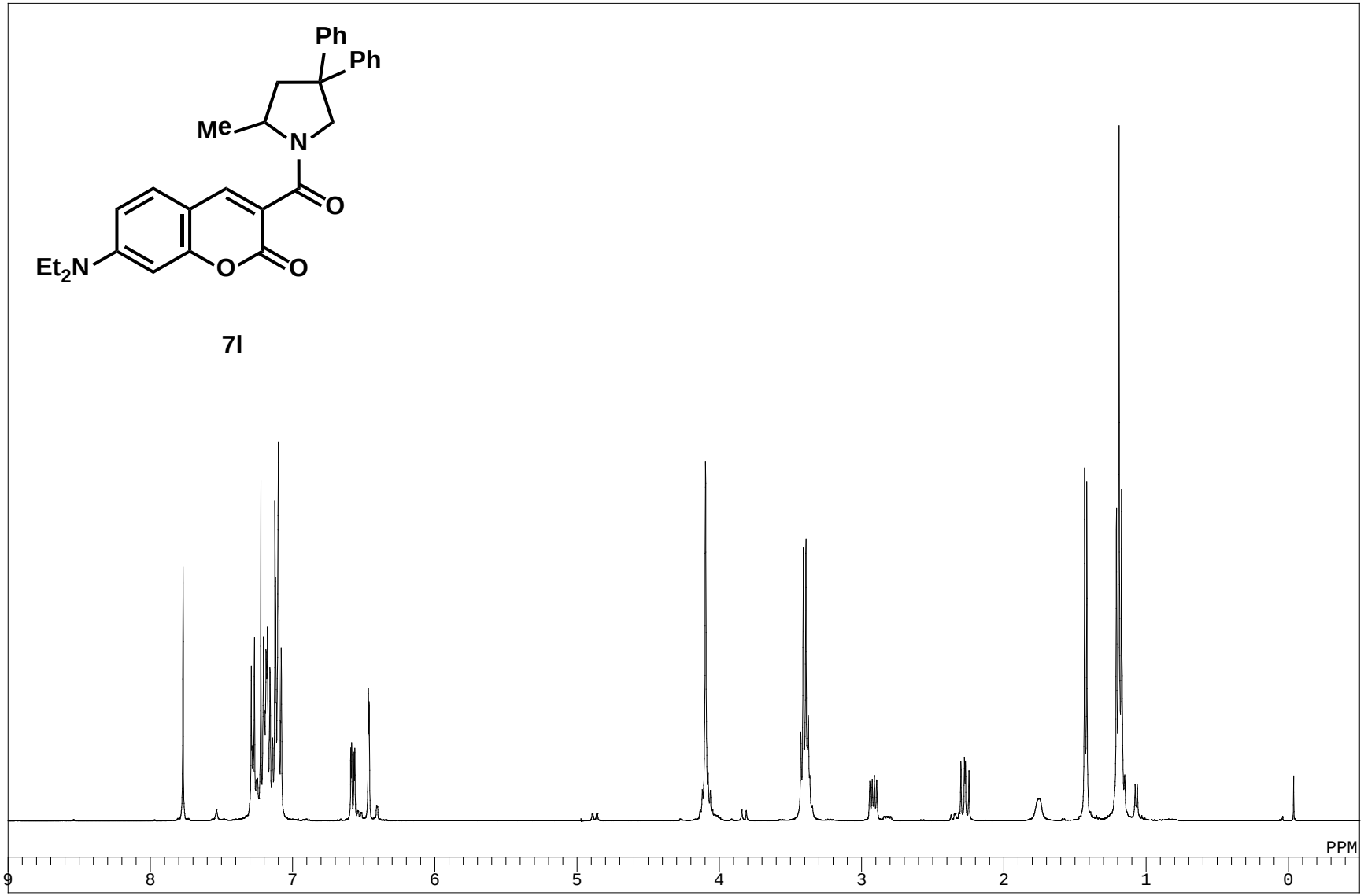
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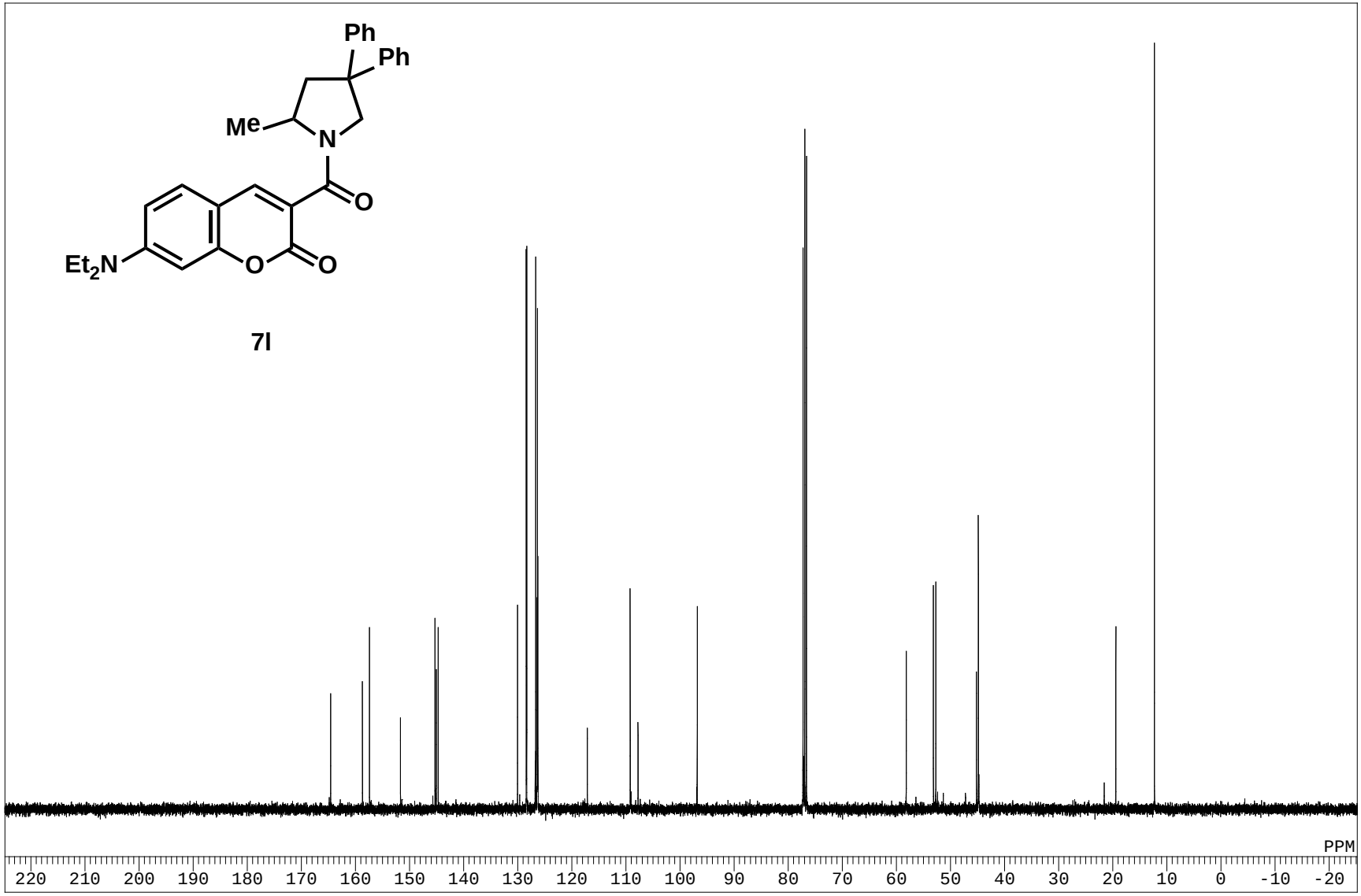
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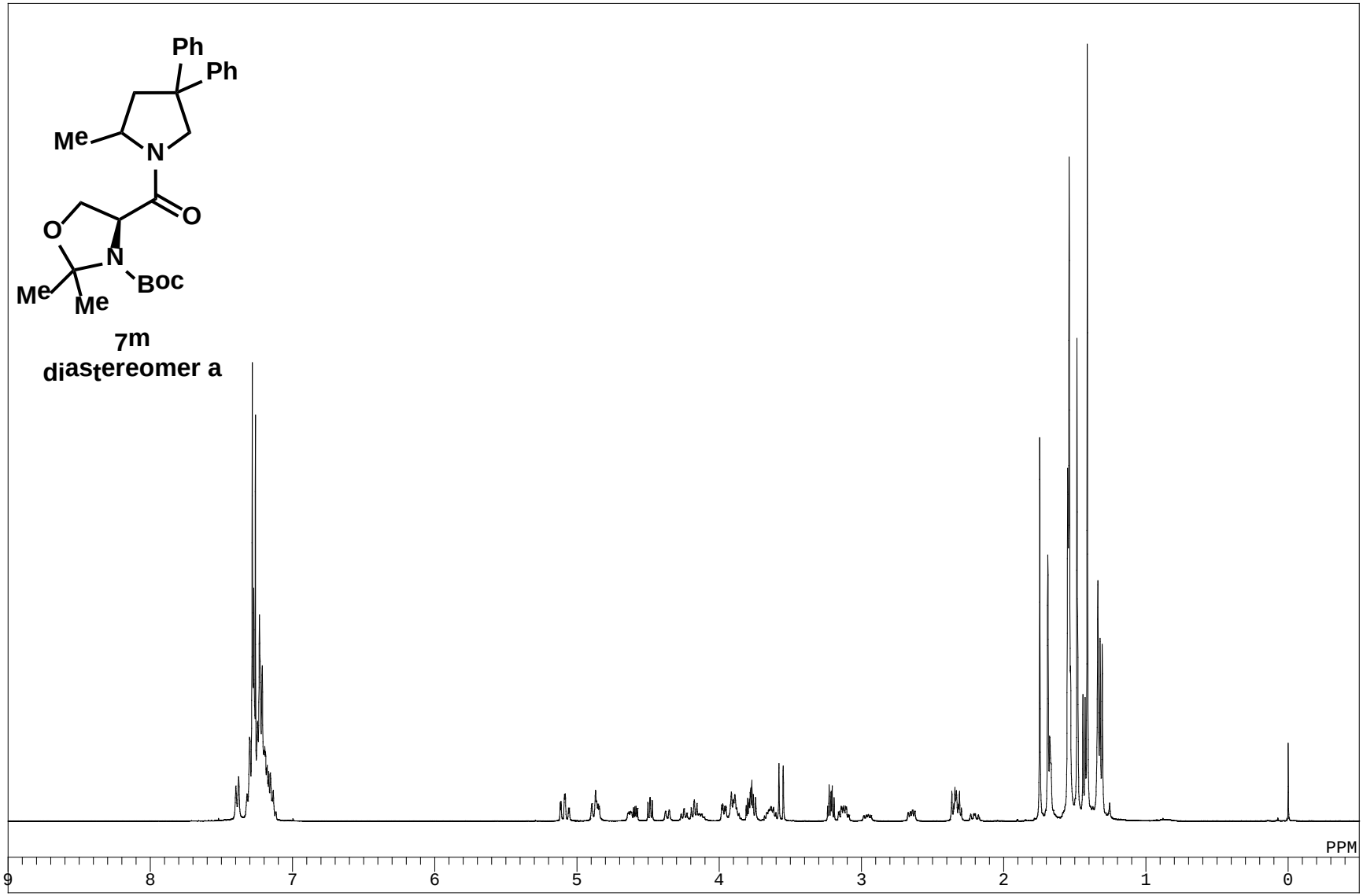
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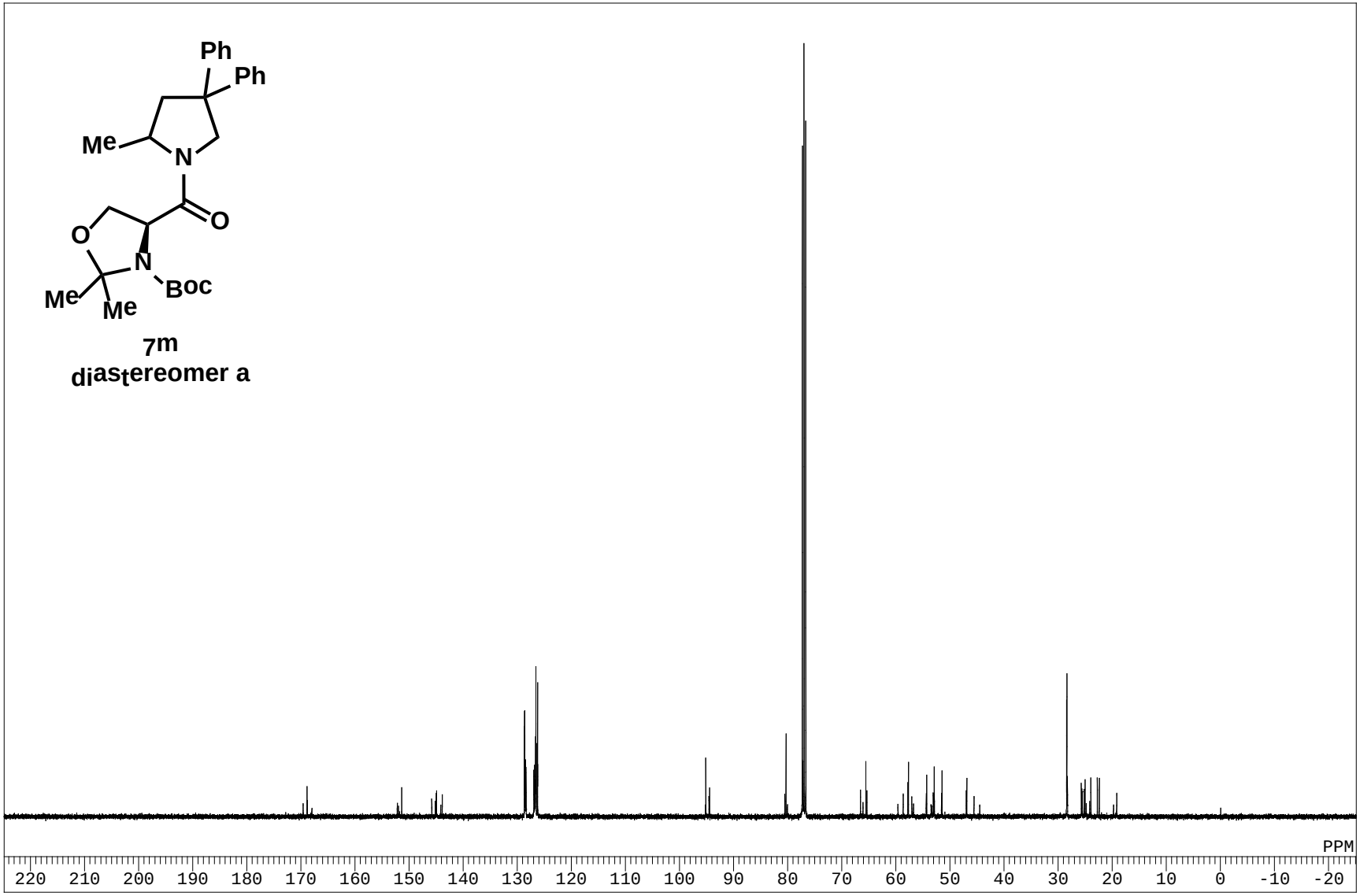
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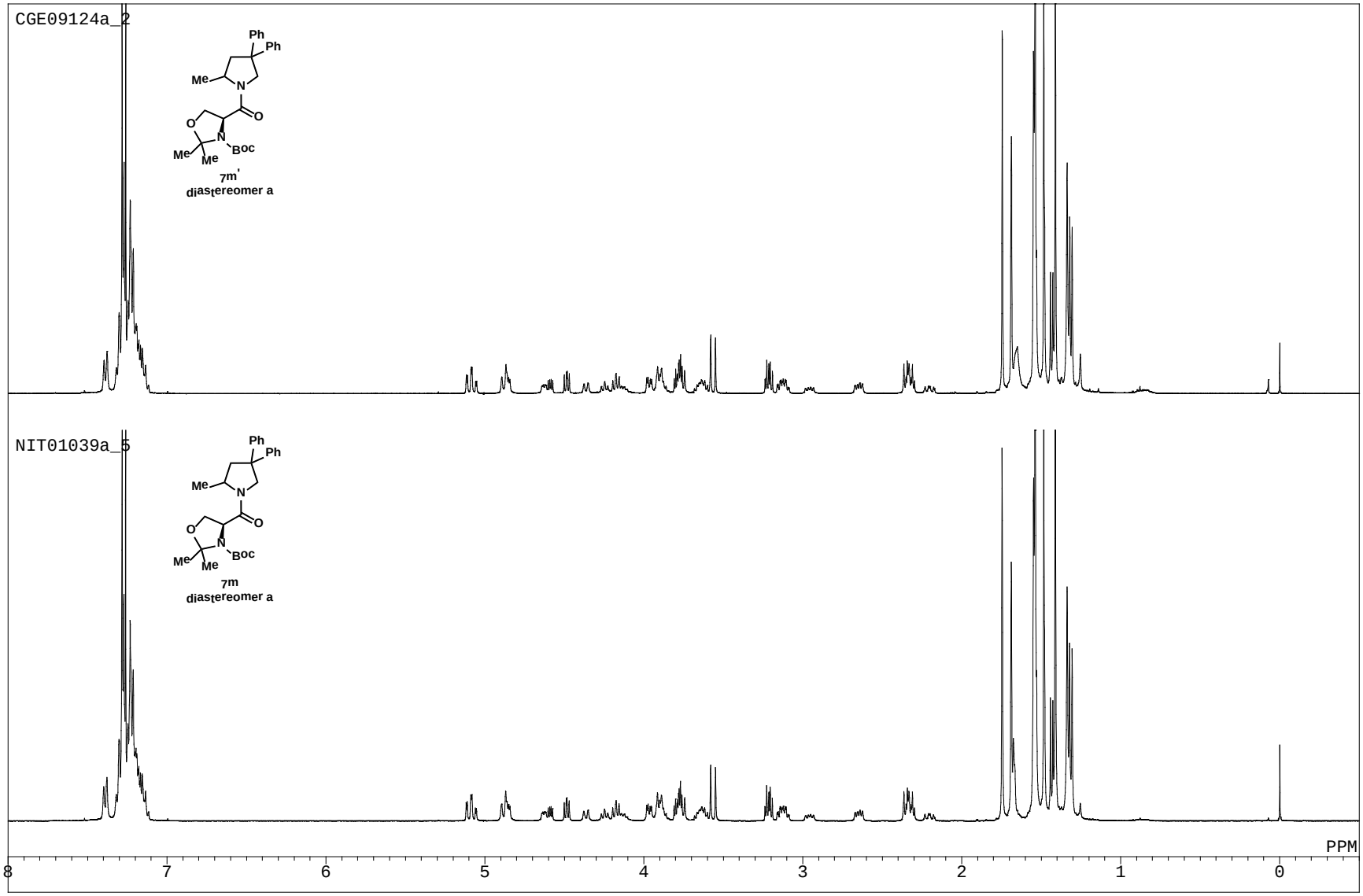
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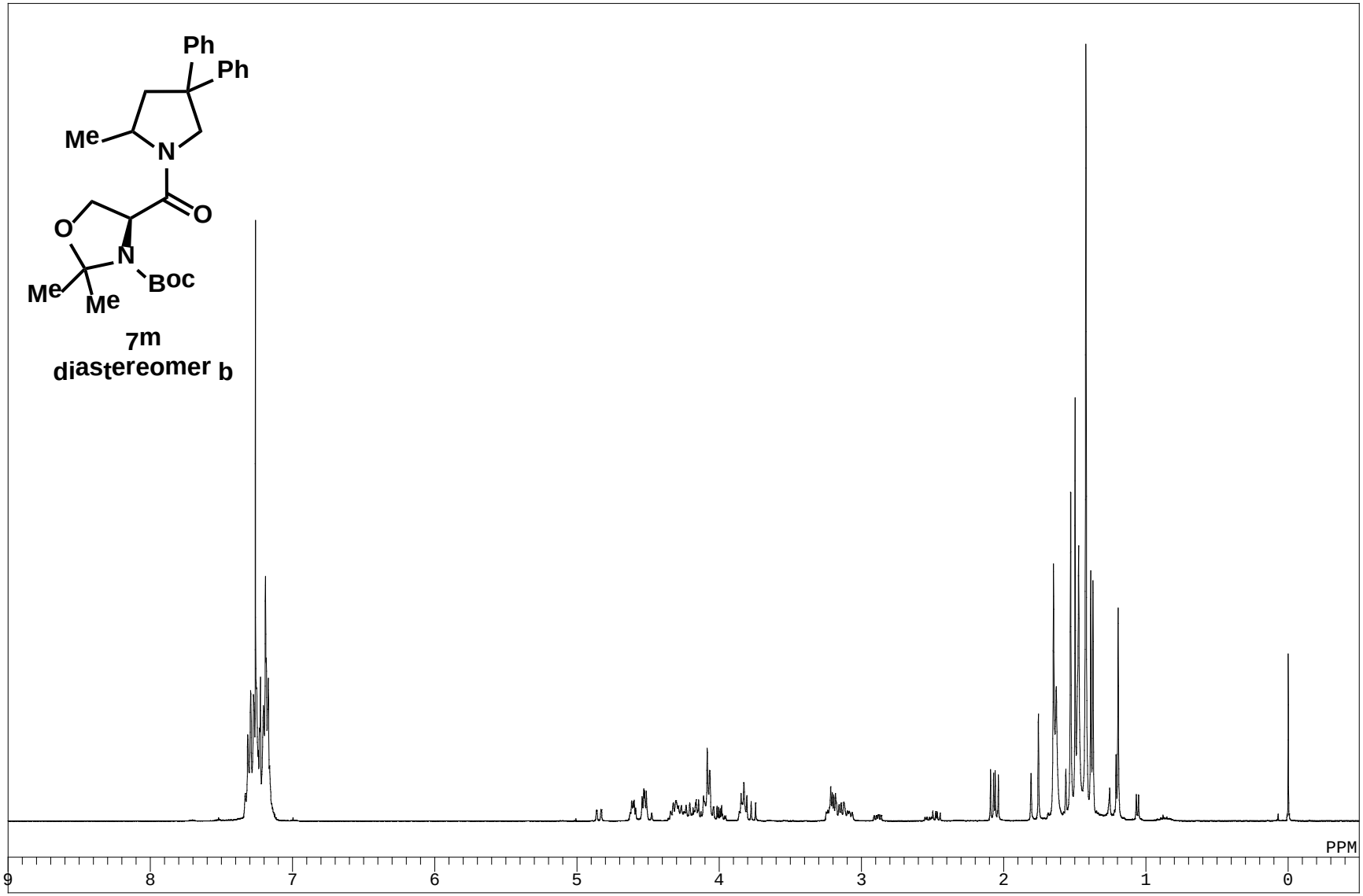
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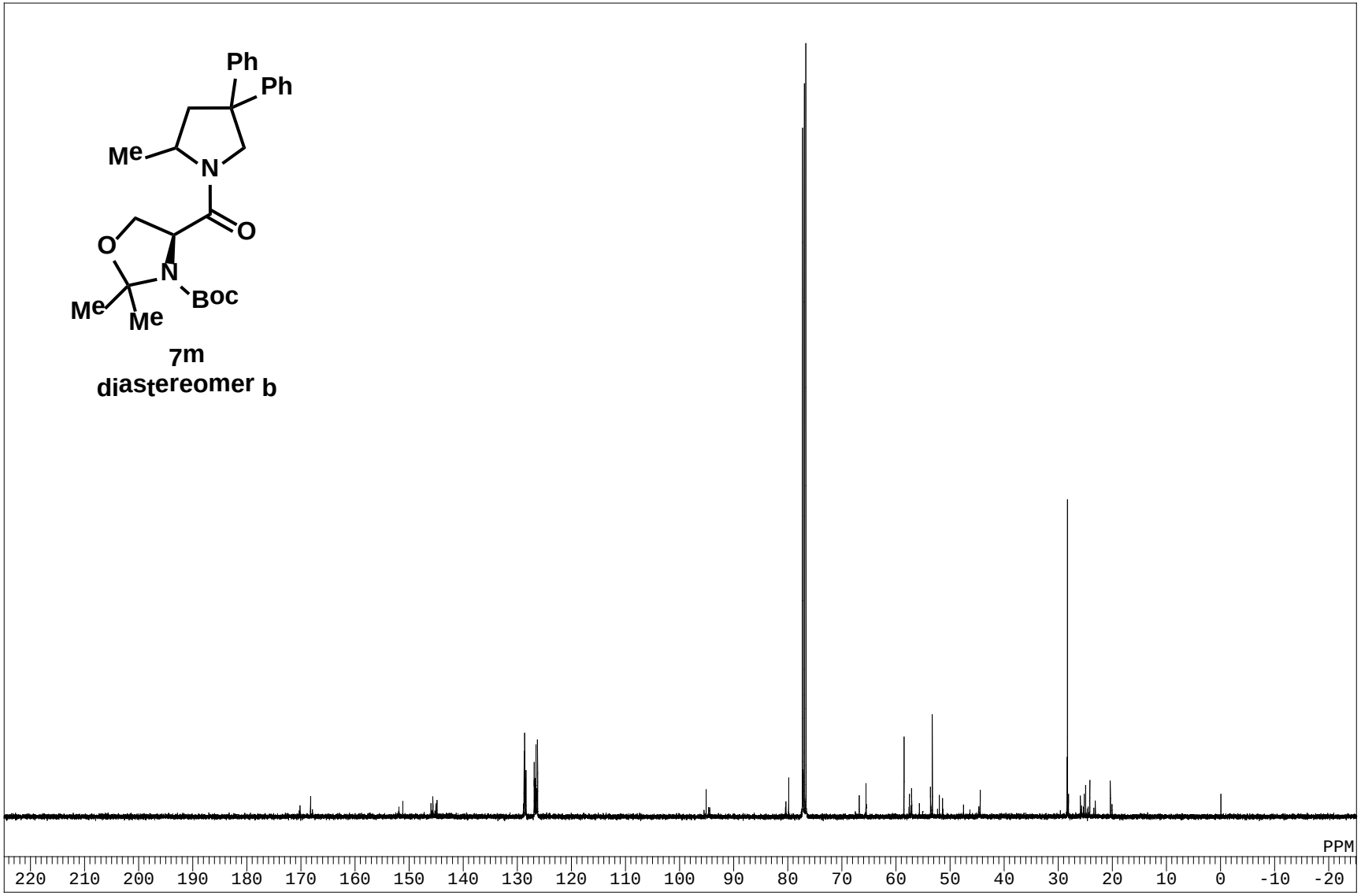
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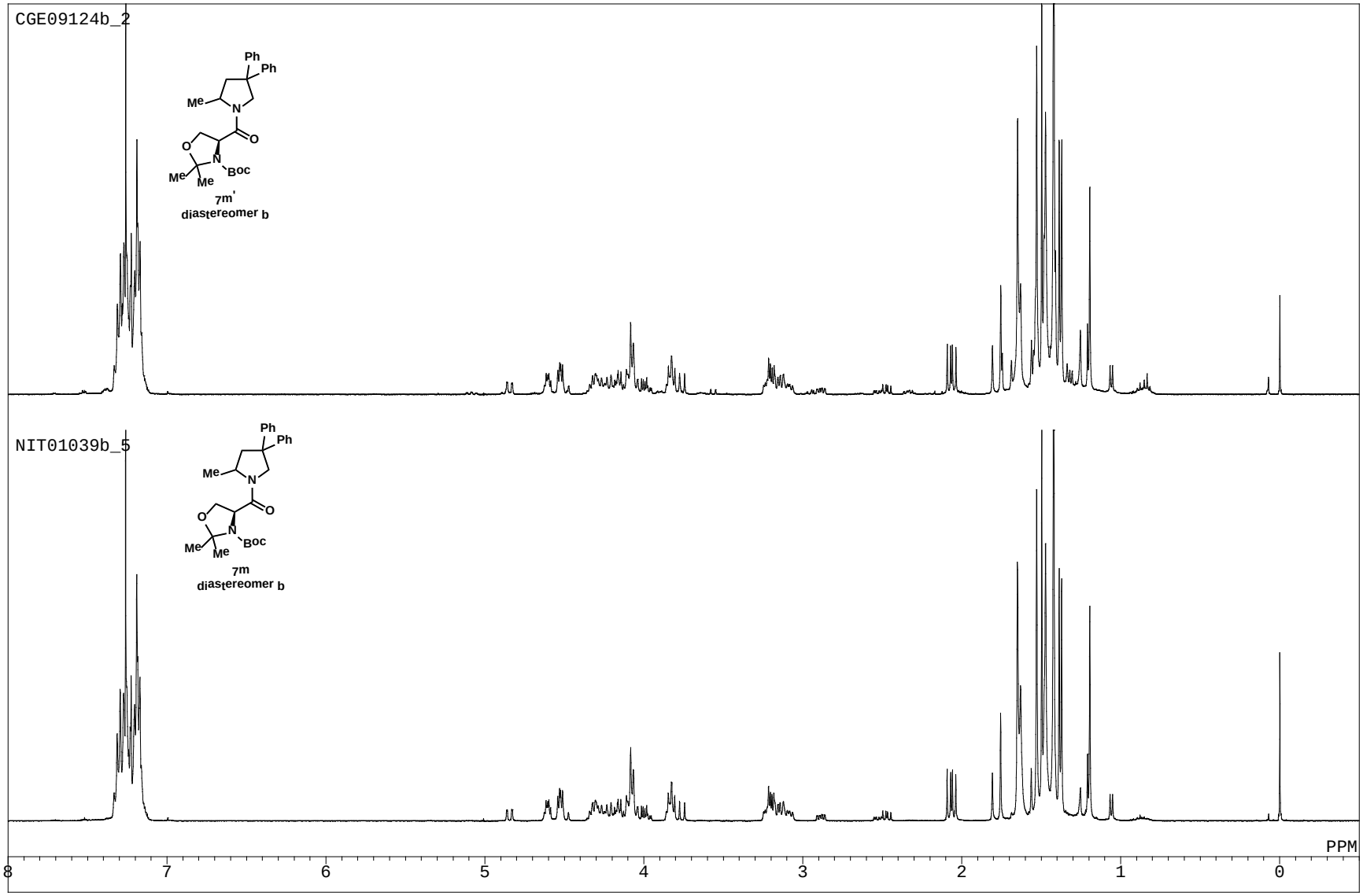
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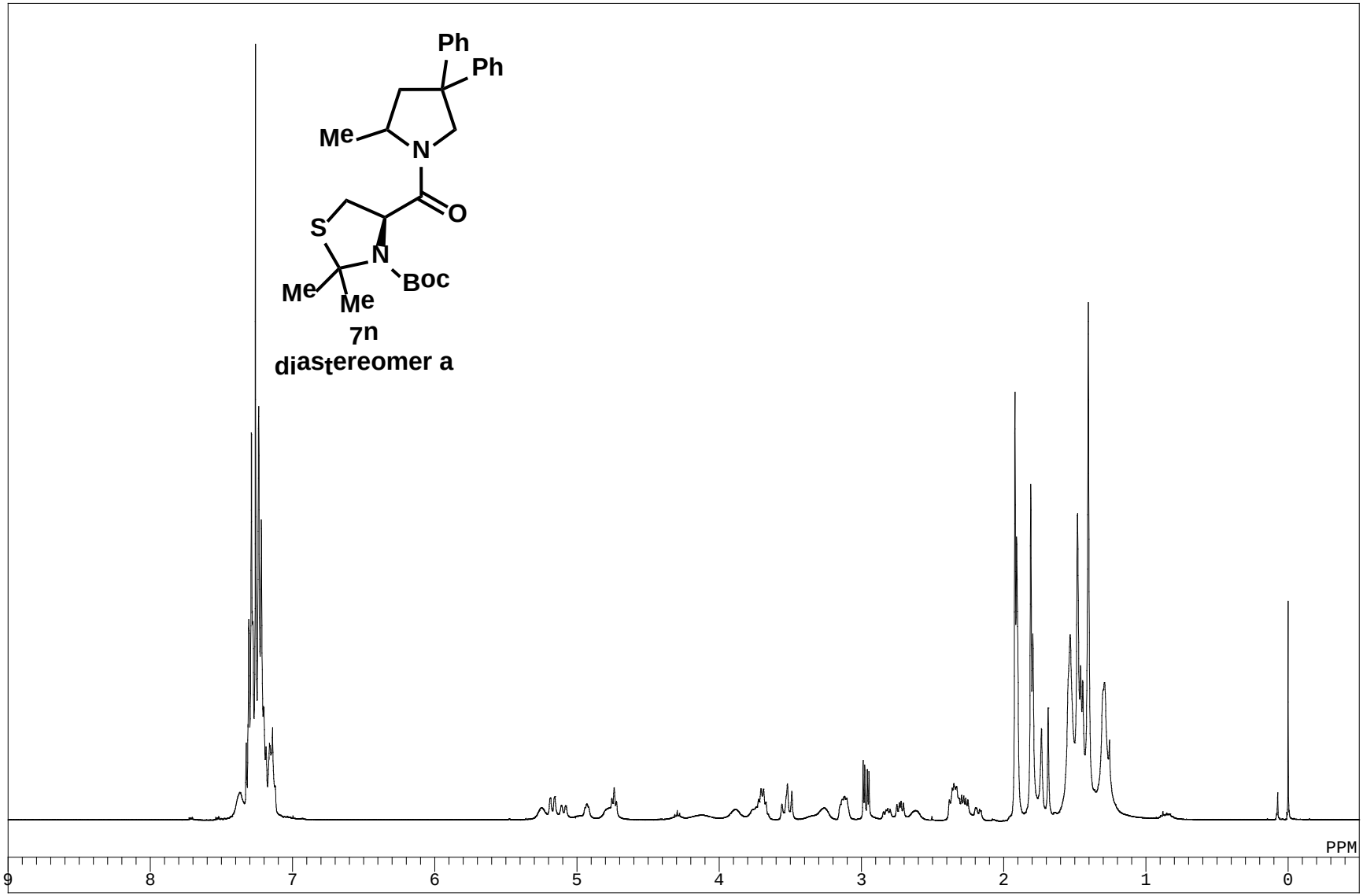
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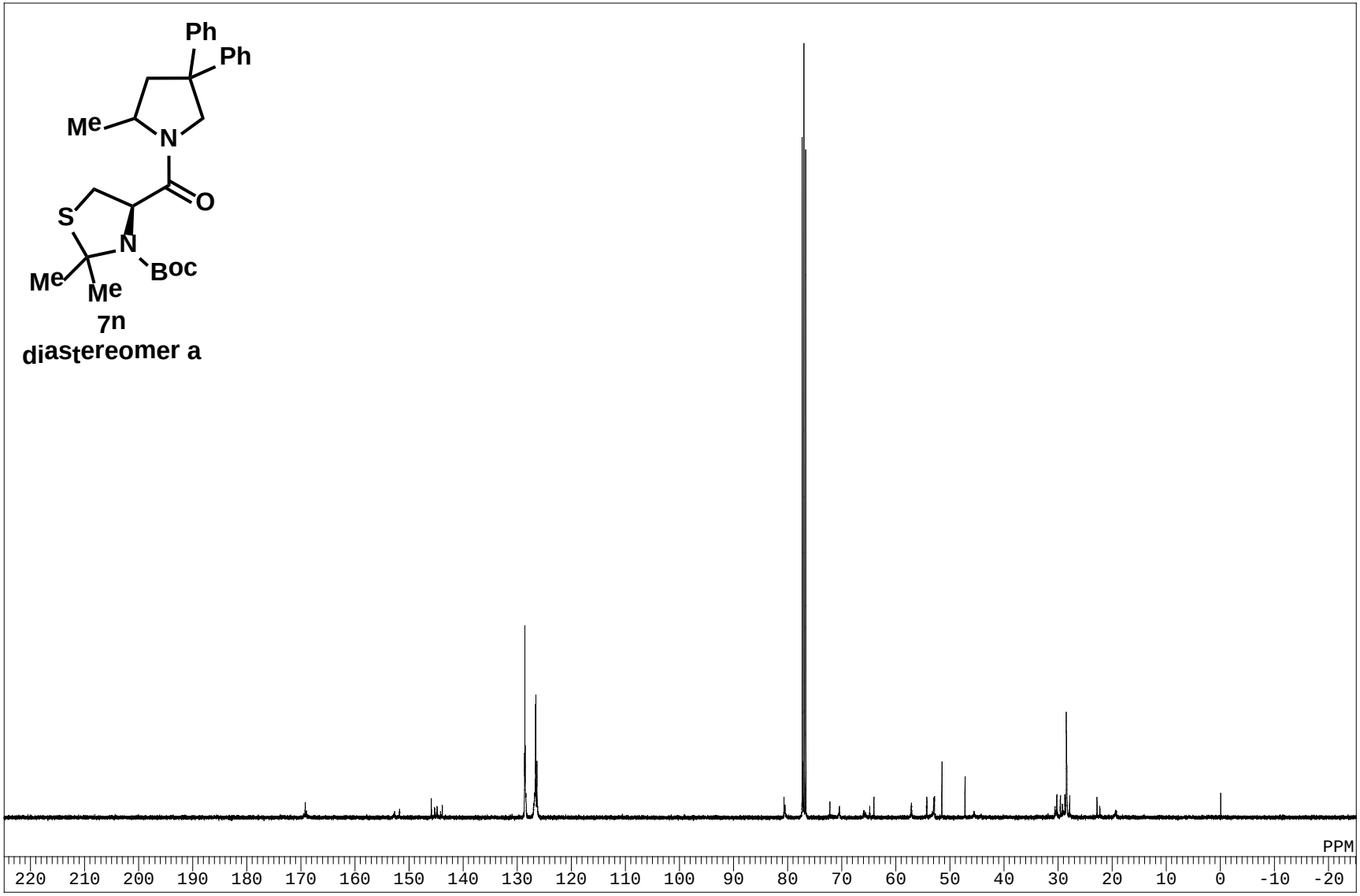
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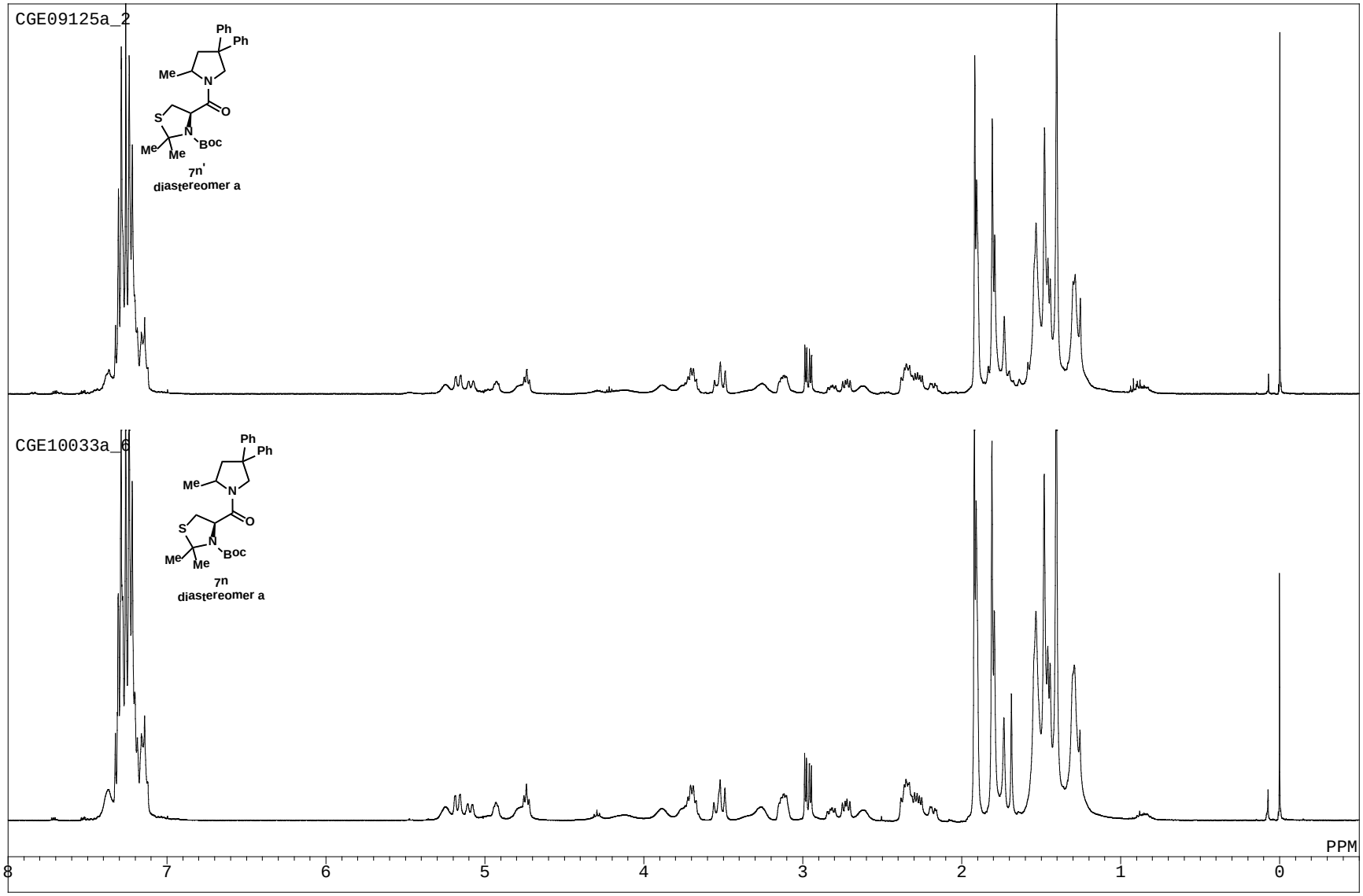
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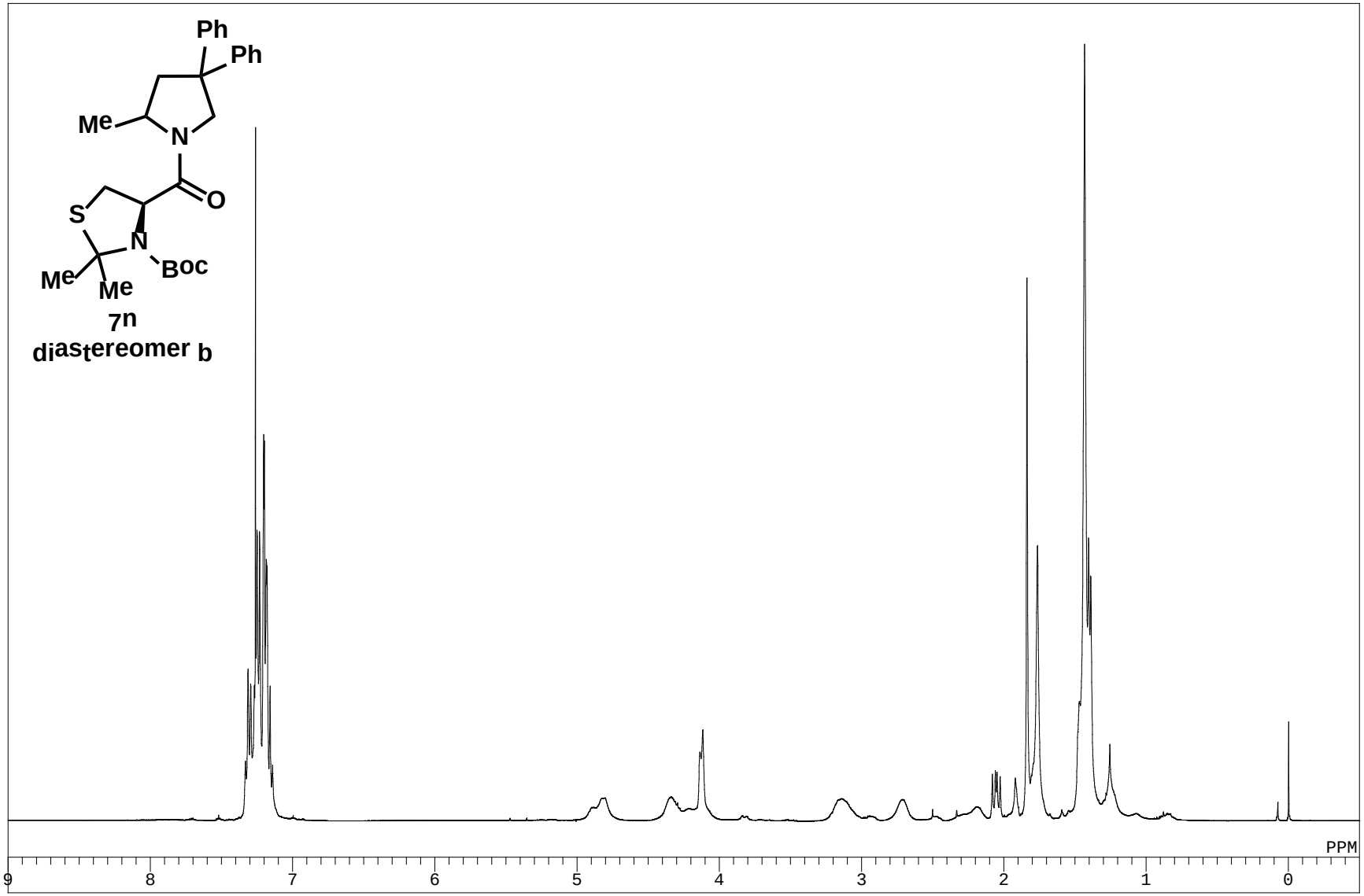
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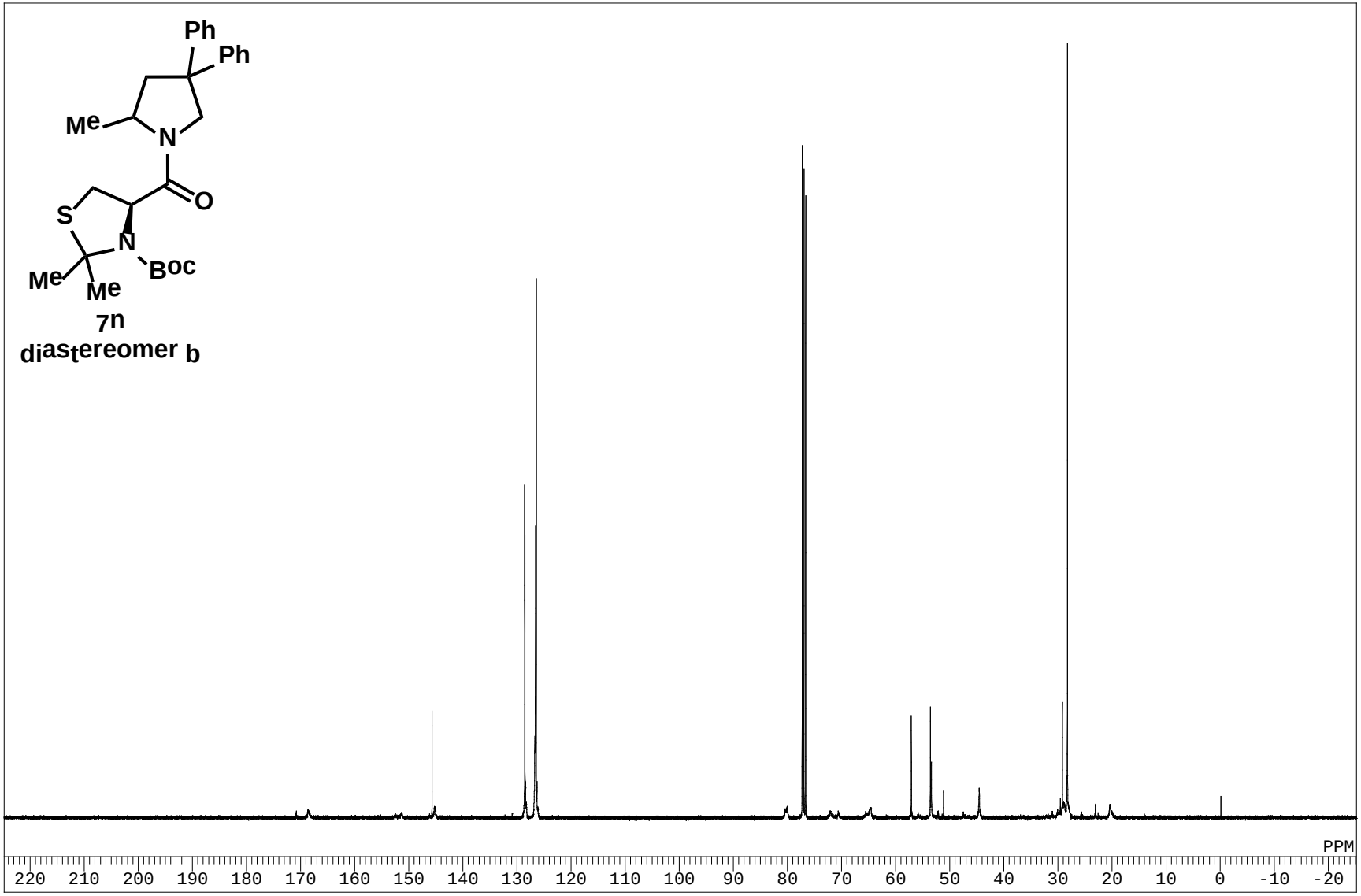
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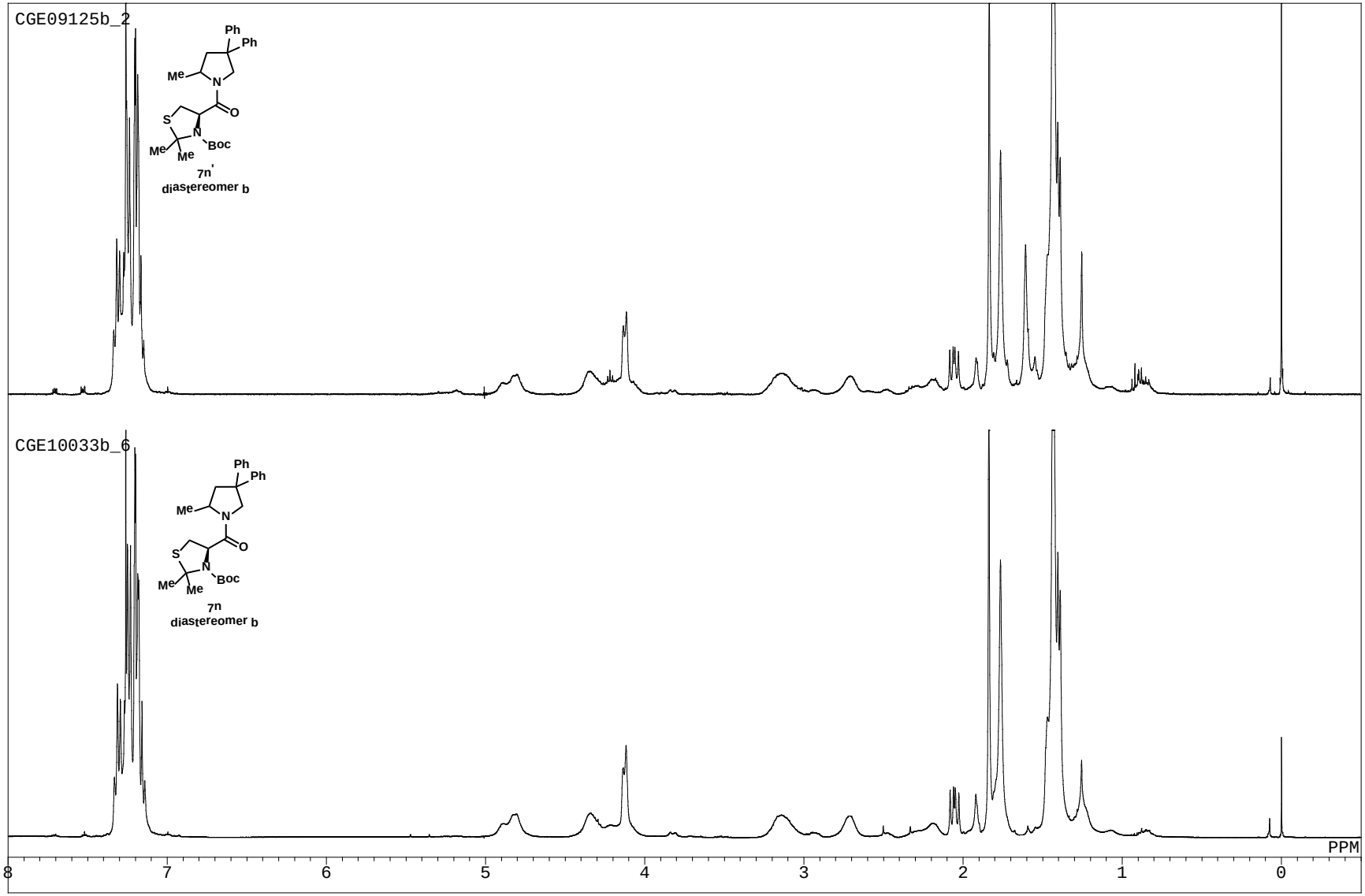
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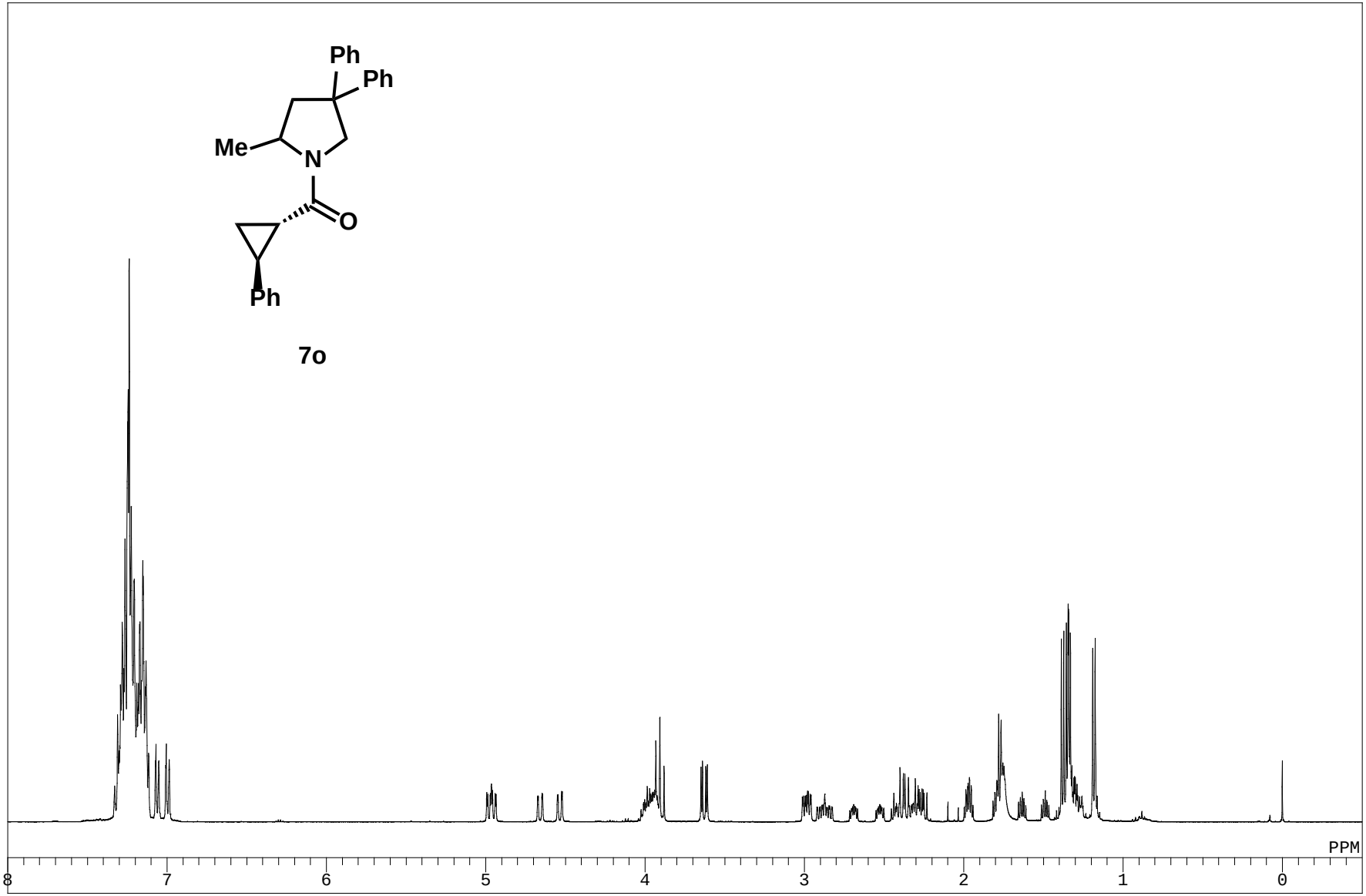
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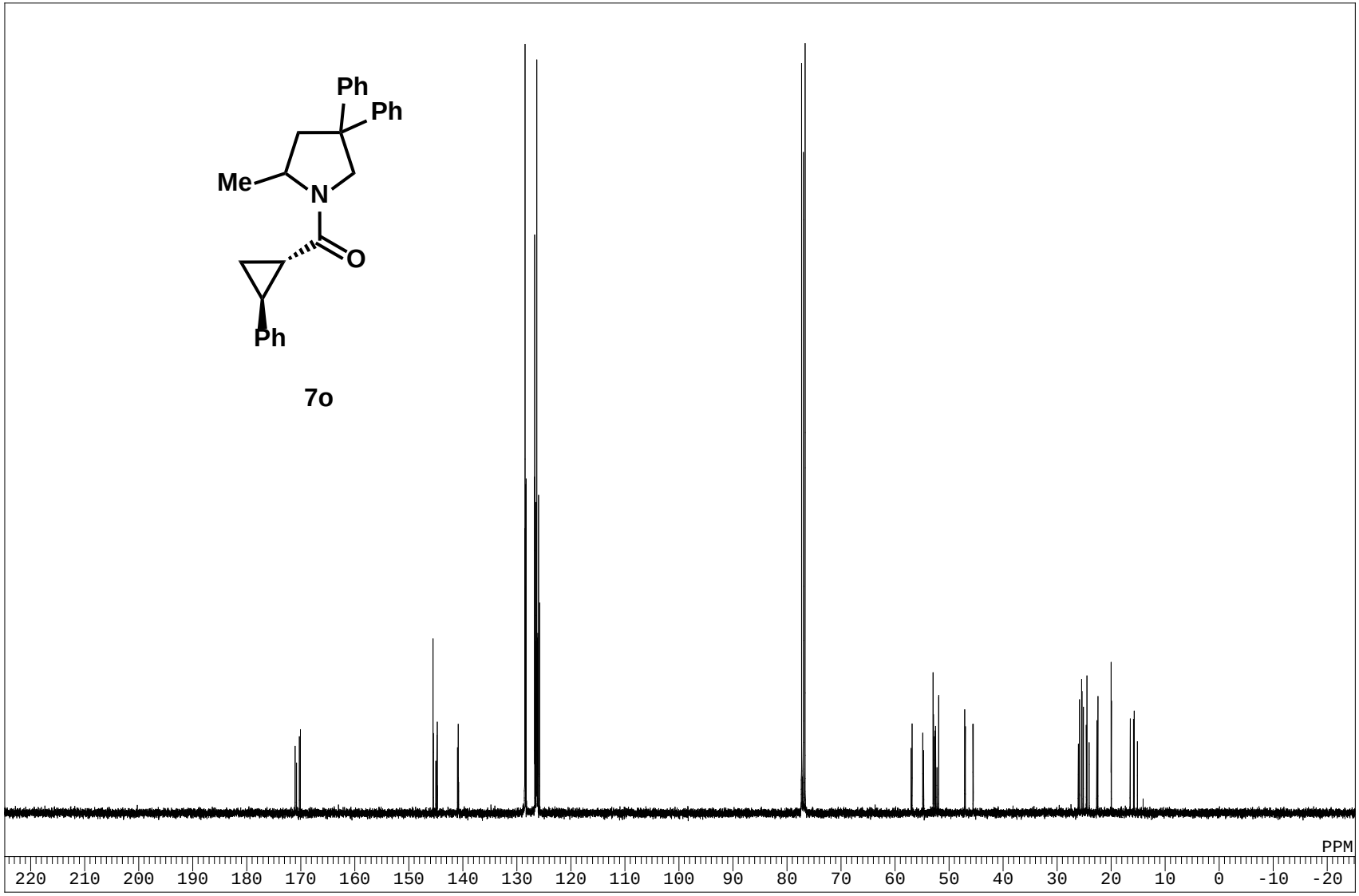
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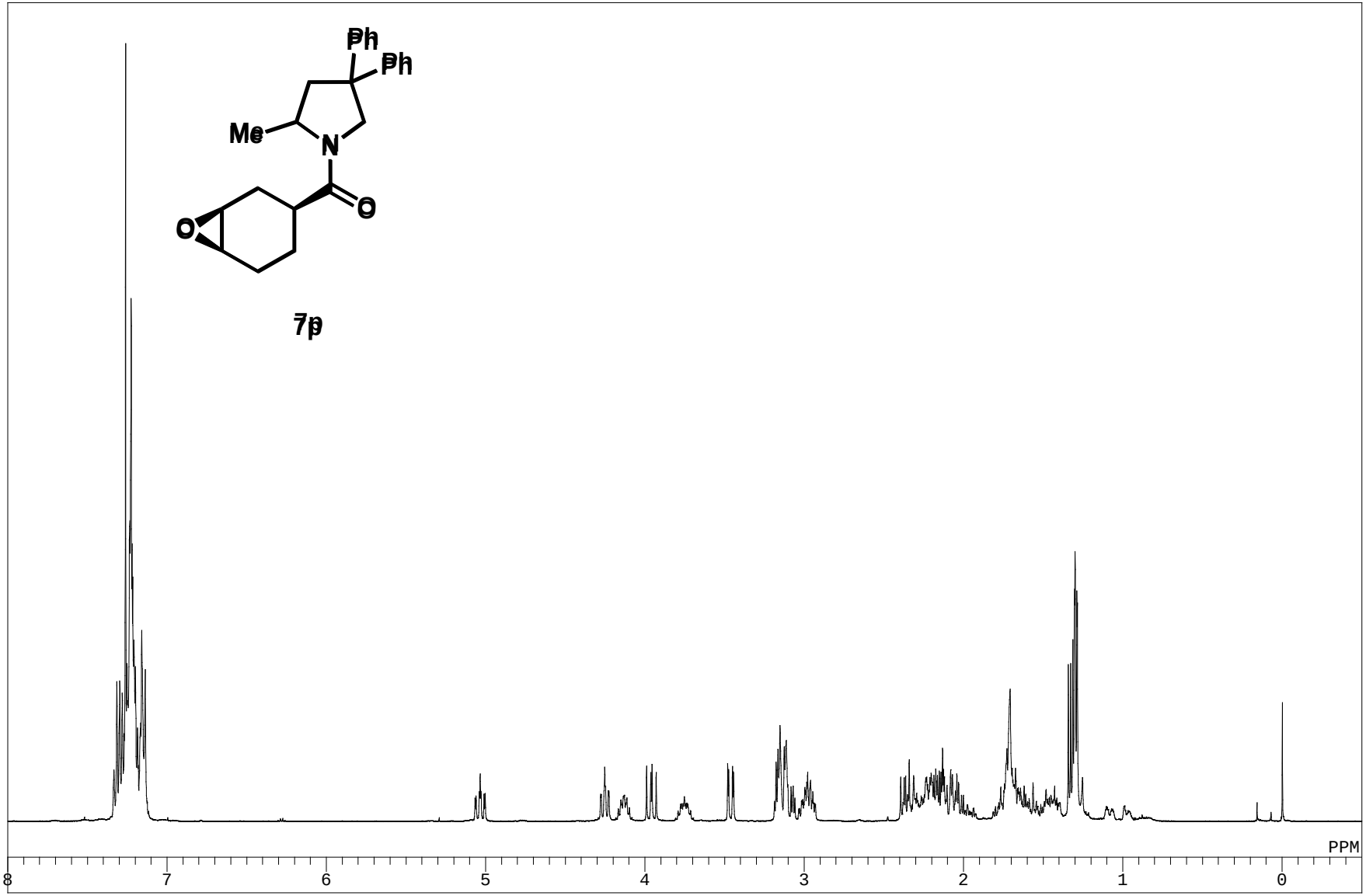
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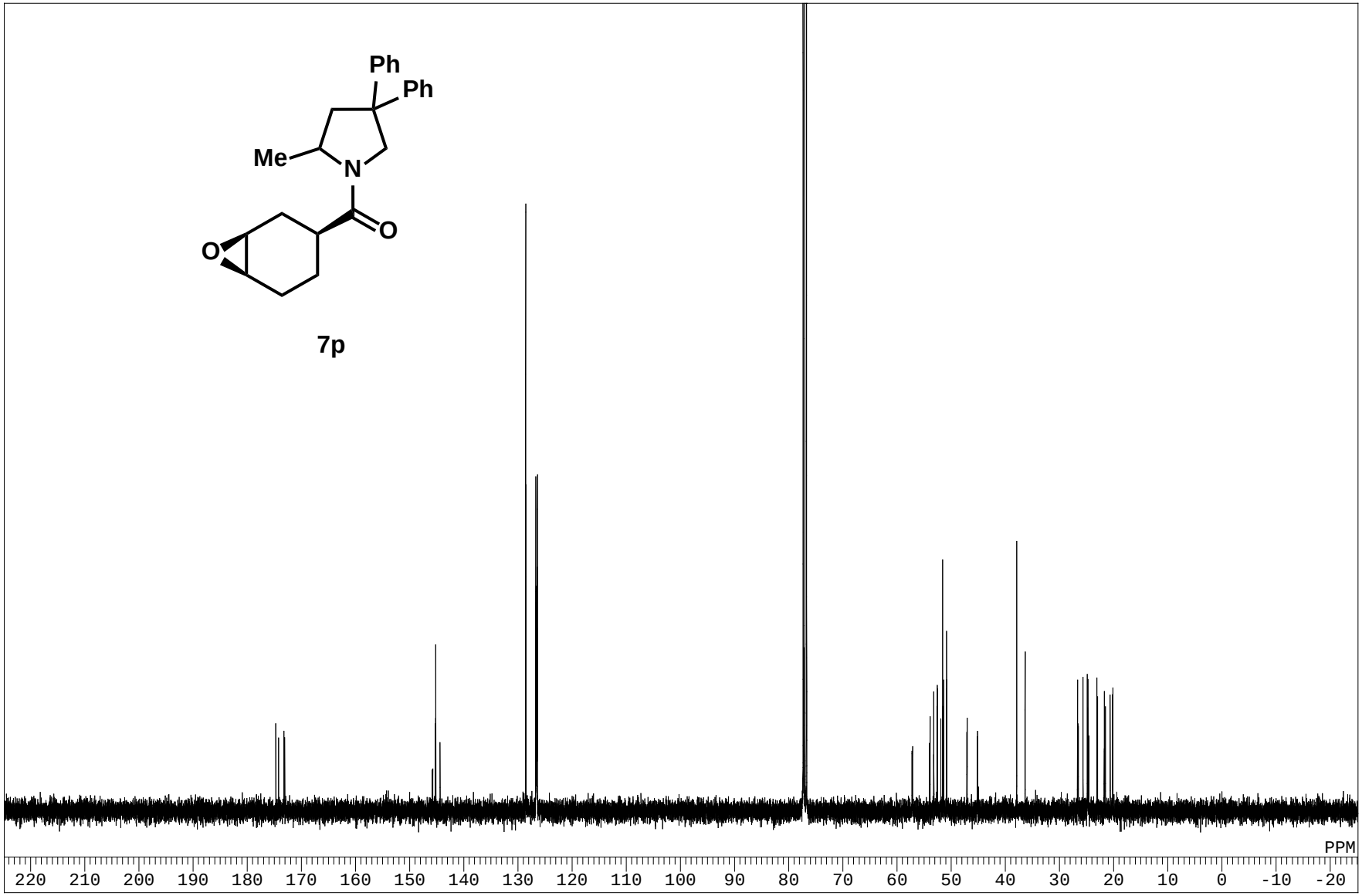
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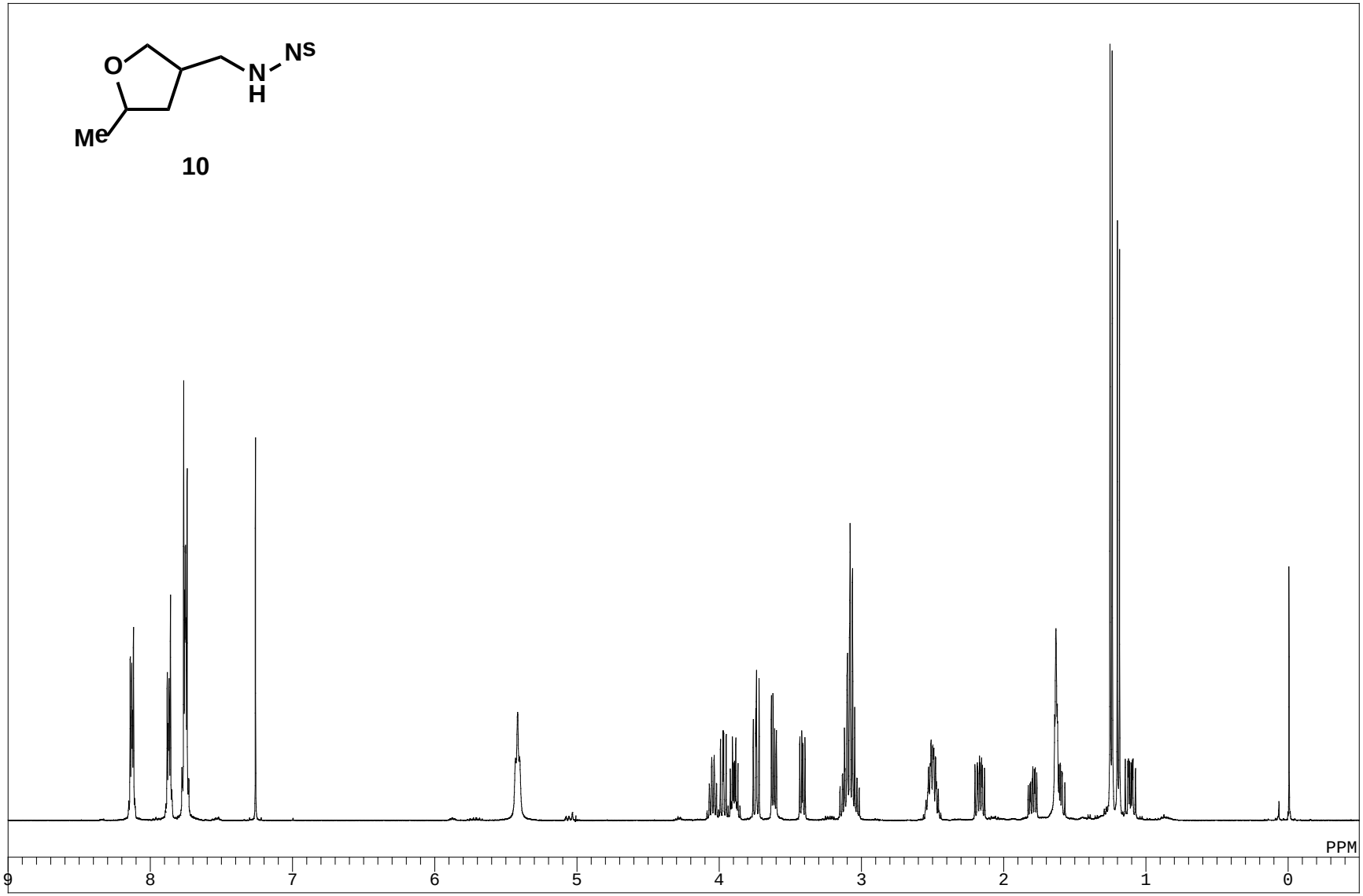
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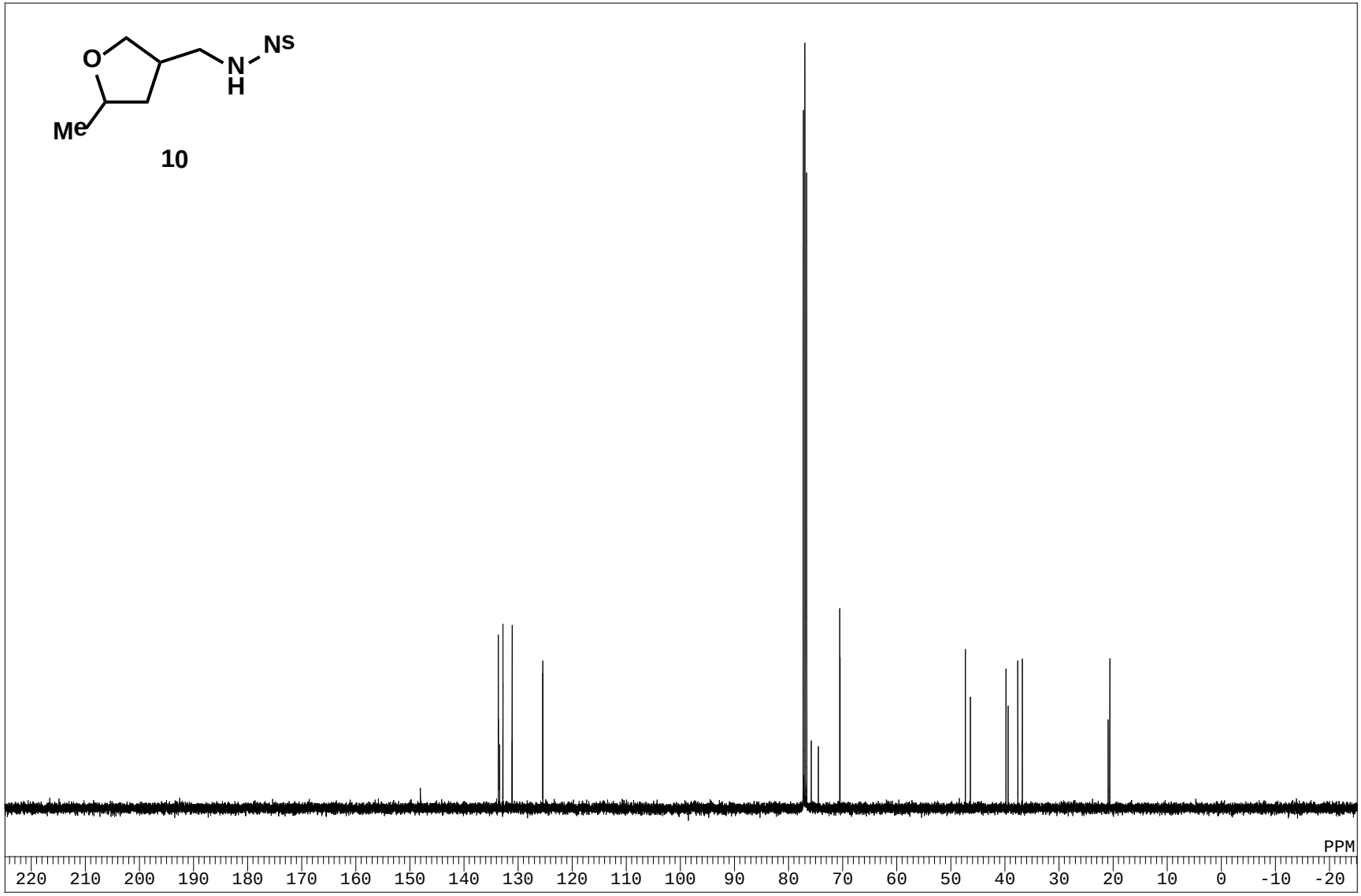
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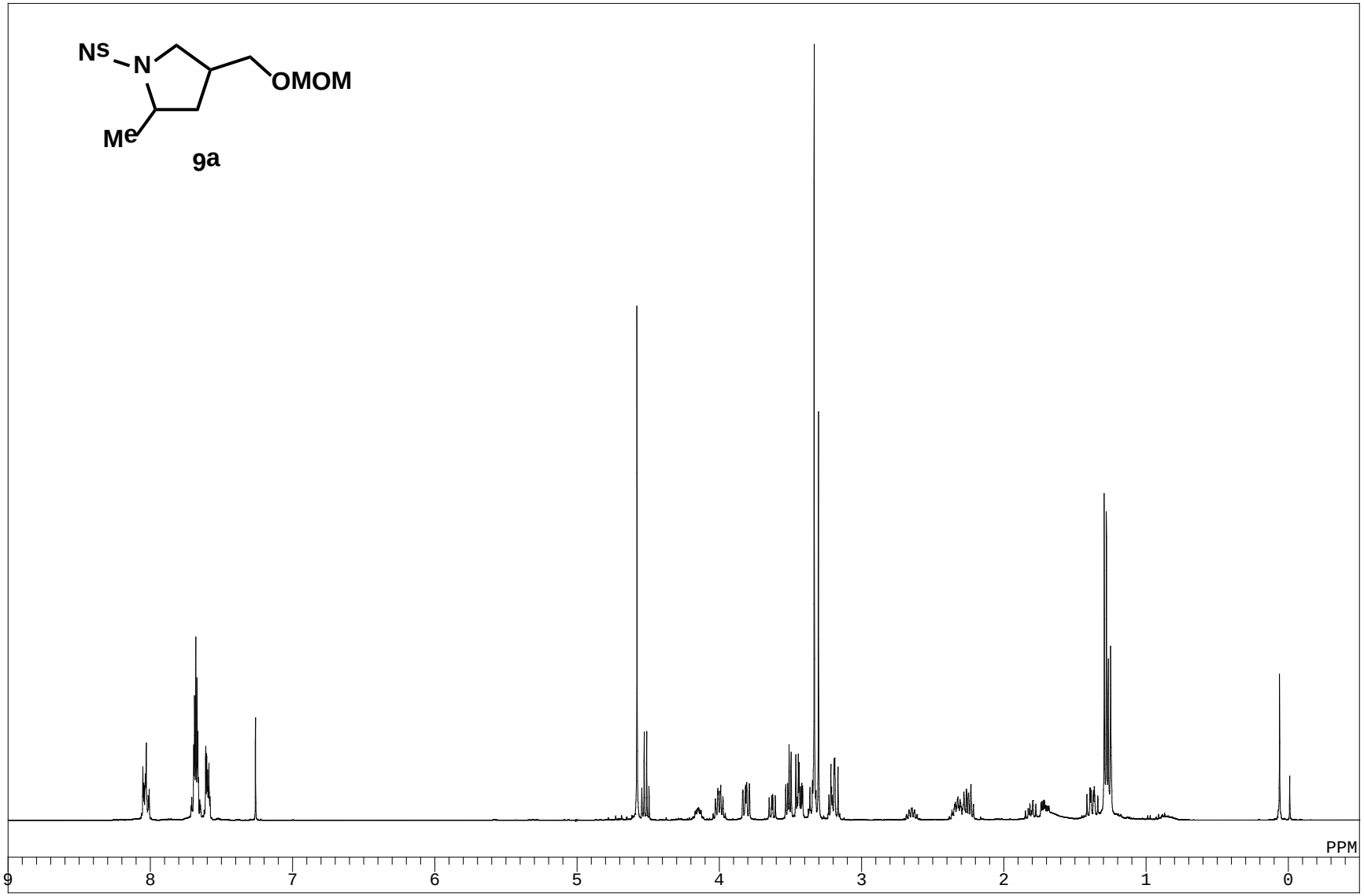
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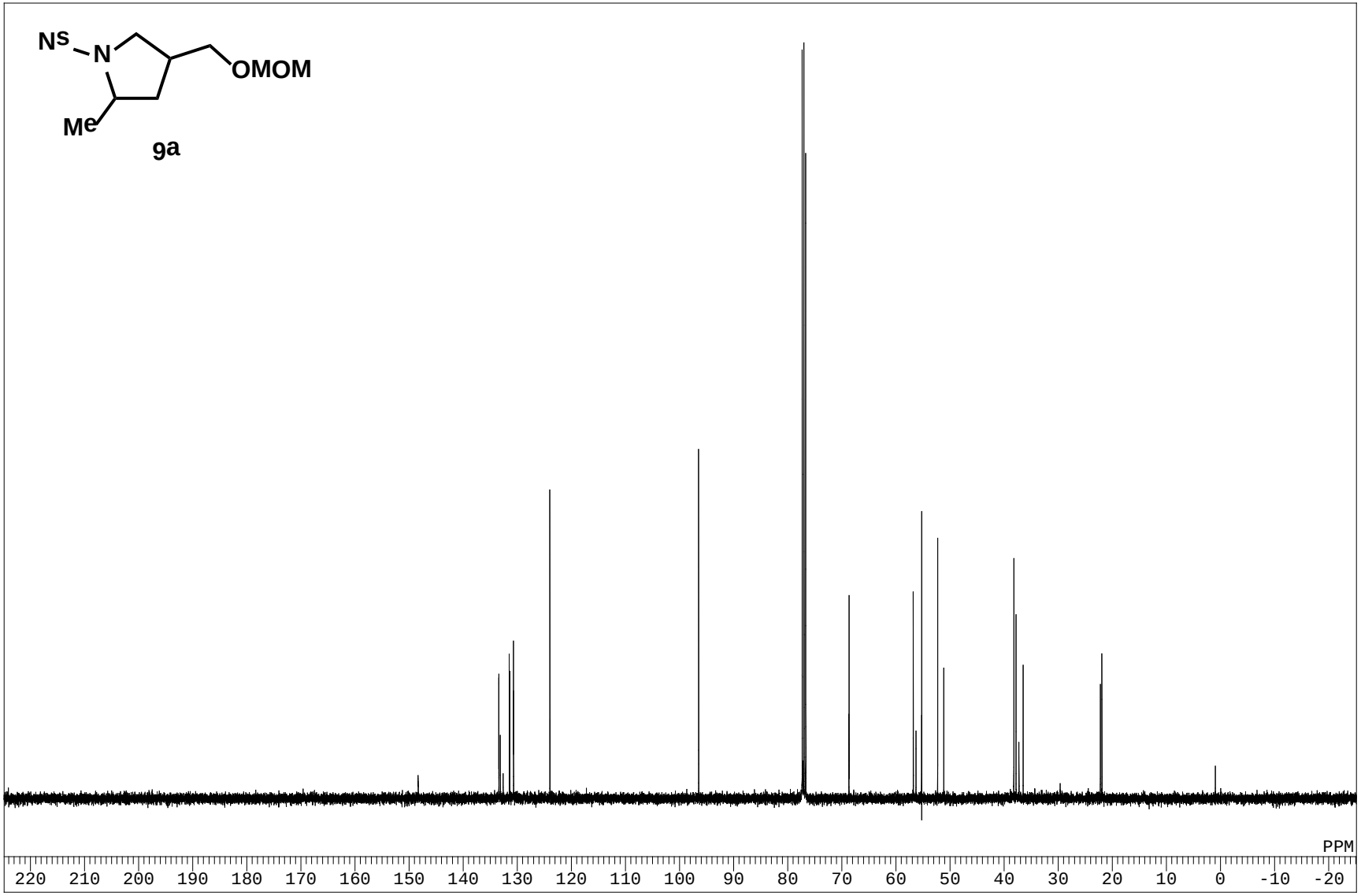
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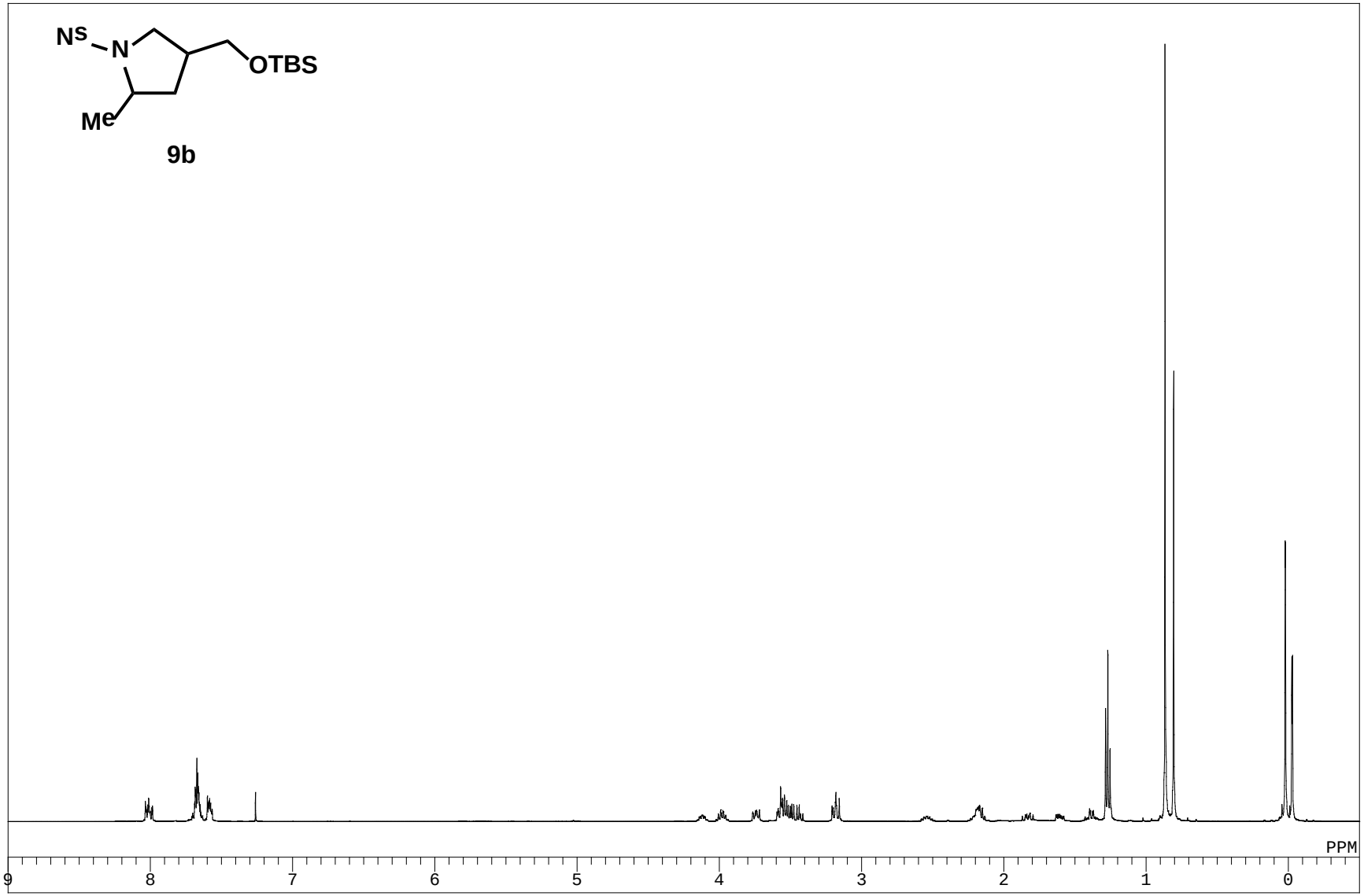
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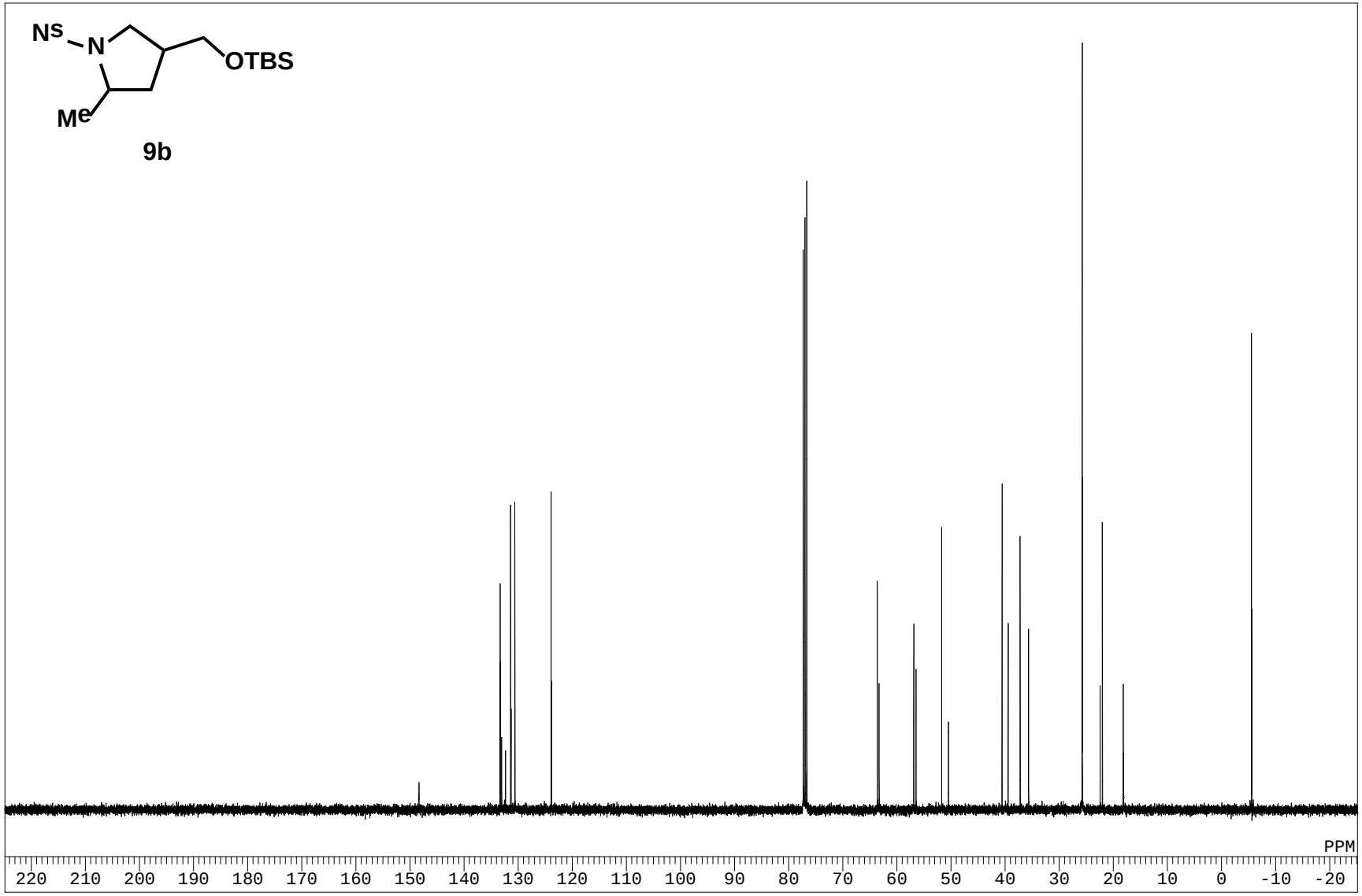
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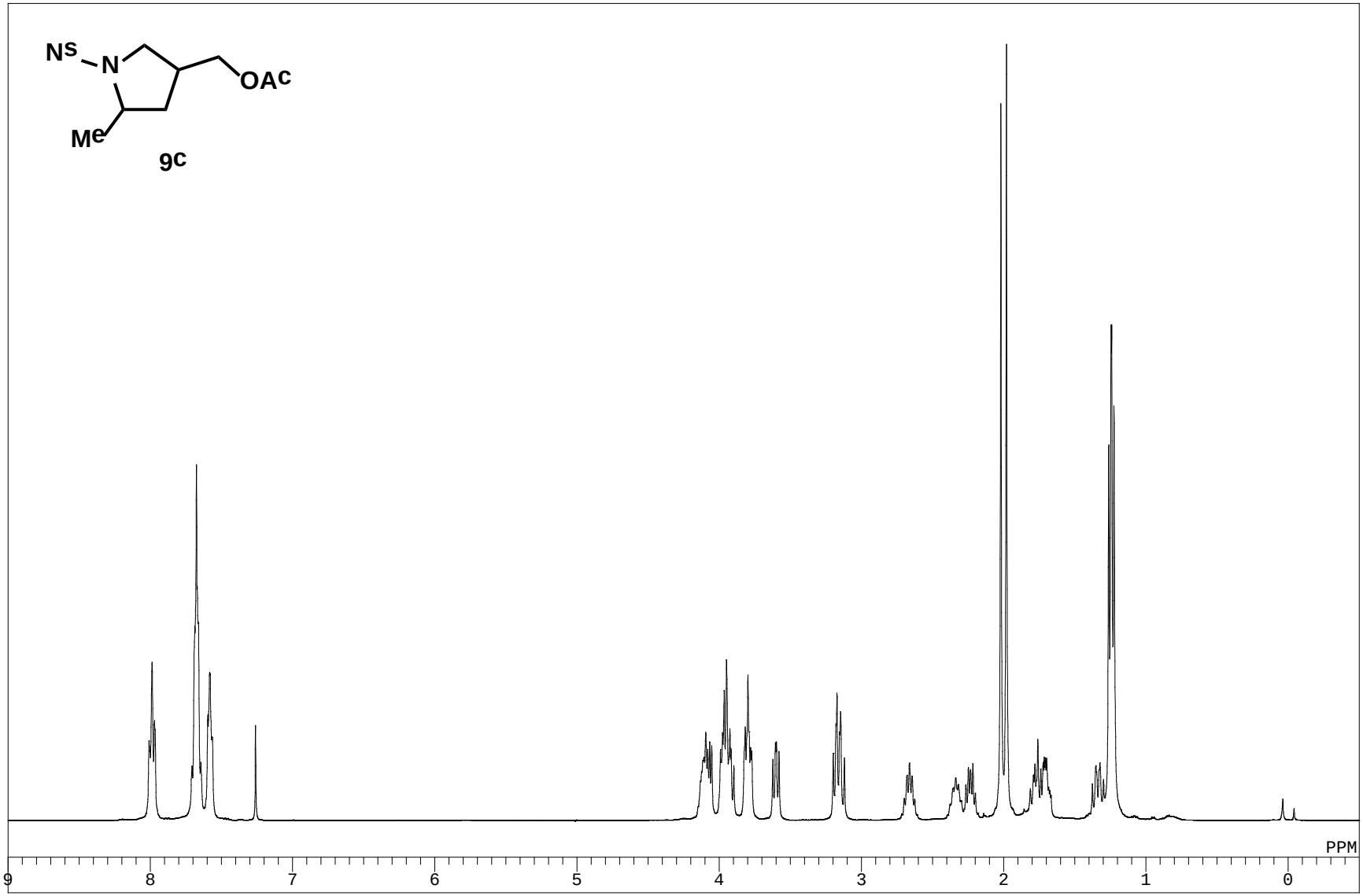
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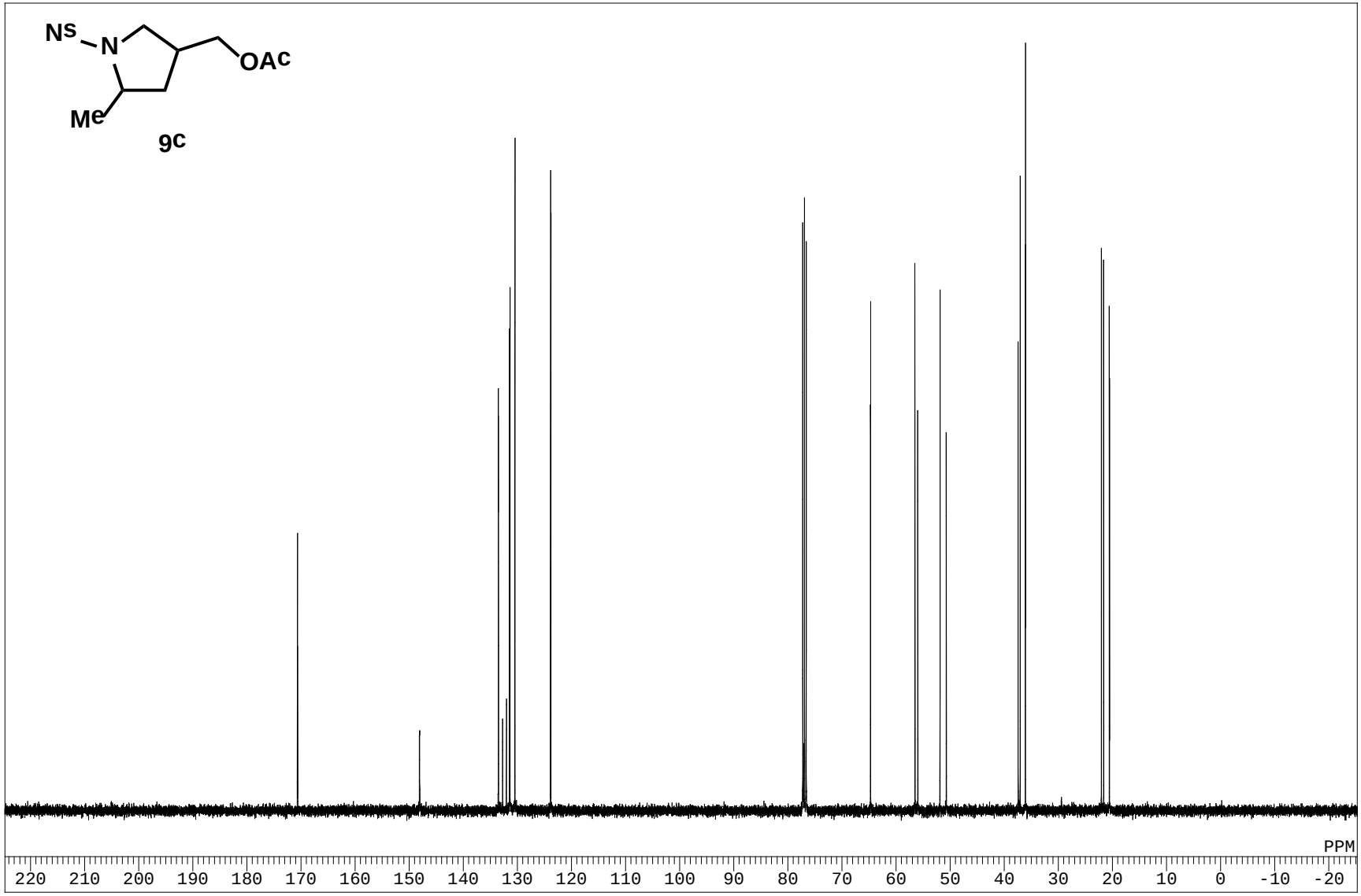
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OAc
Me
9c
PPM
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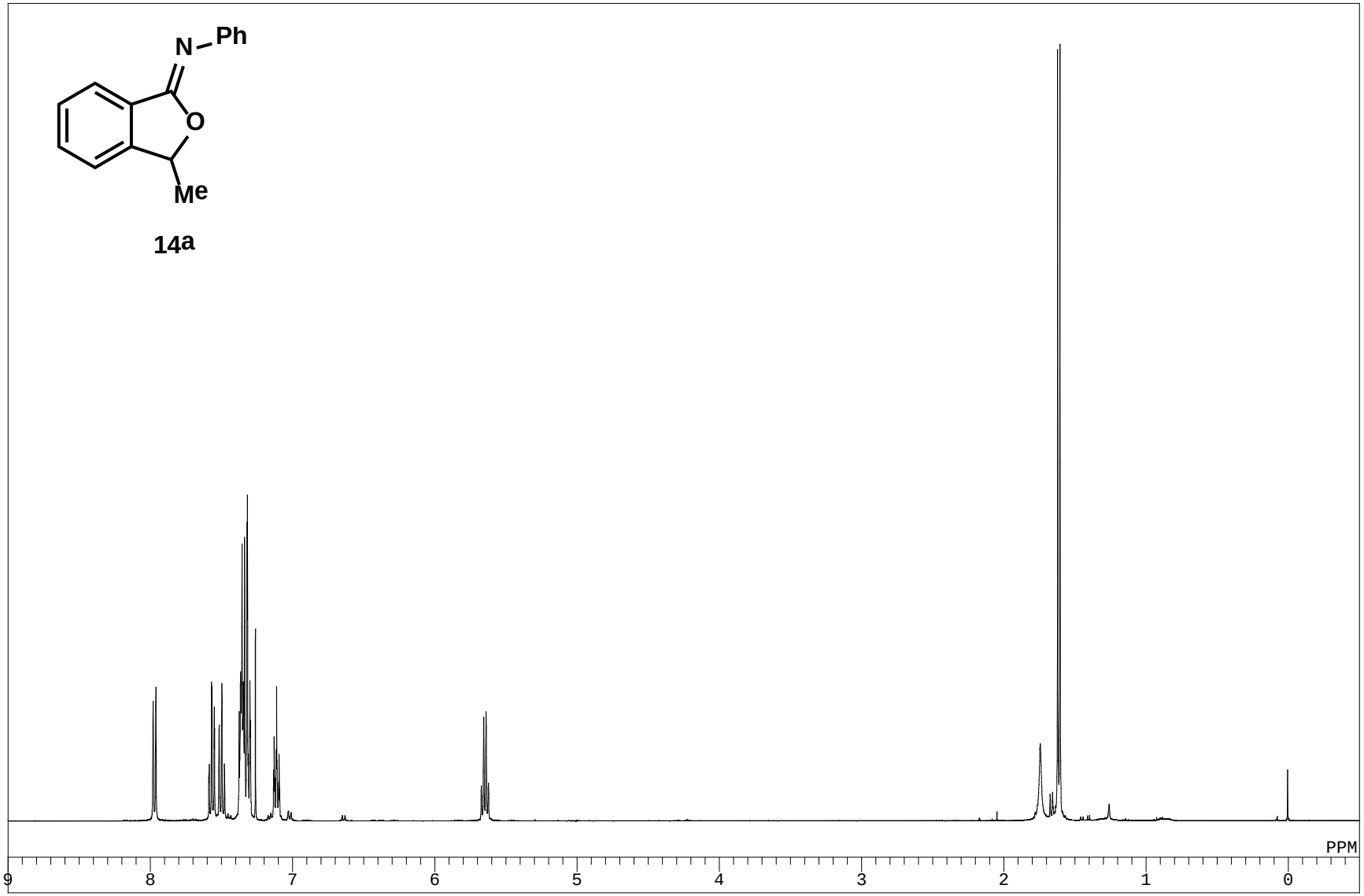
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Me
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PPM
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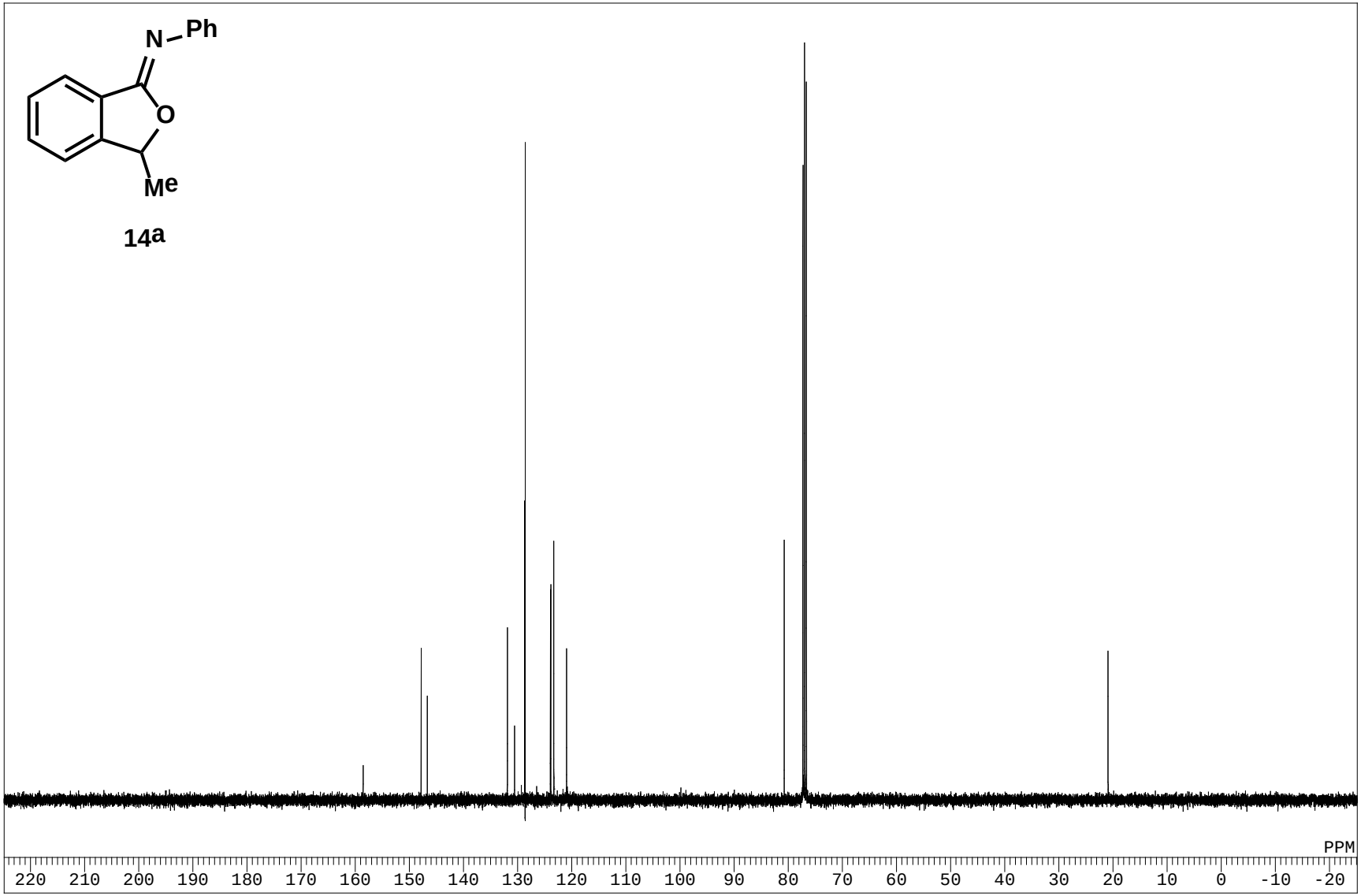
4
3
2
1
0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O
Me
14a
PPM
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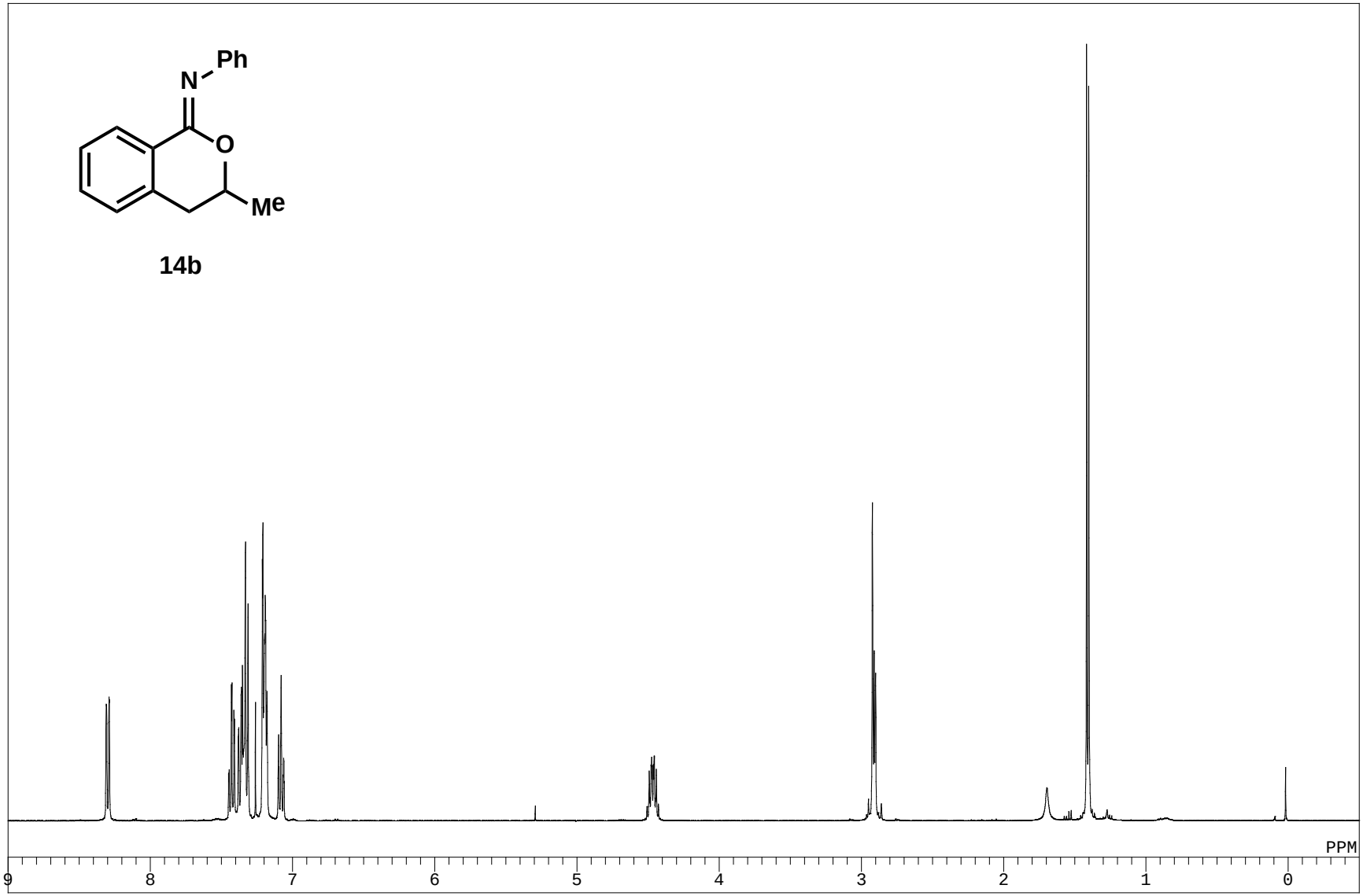
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PPM
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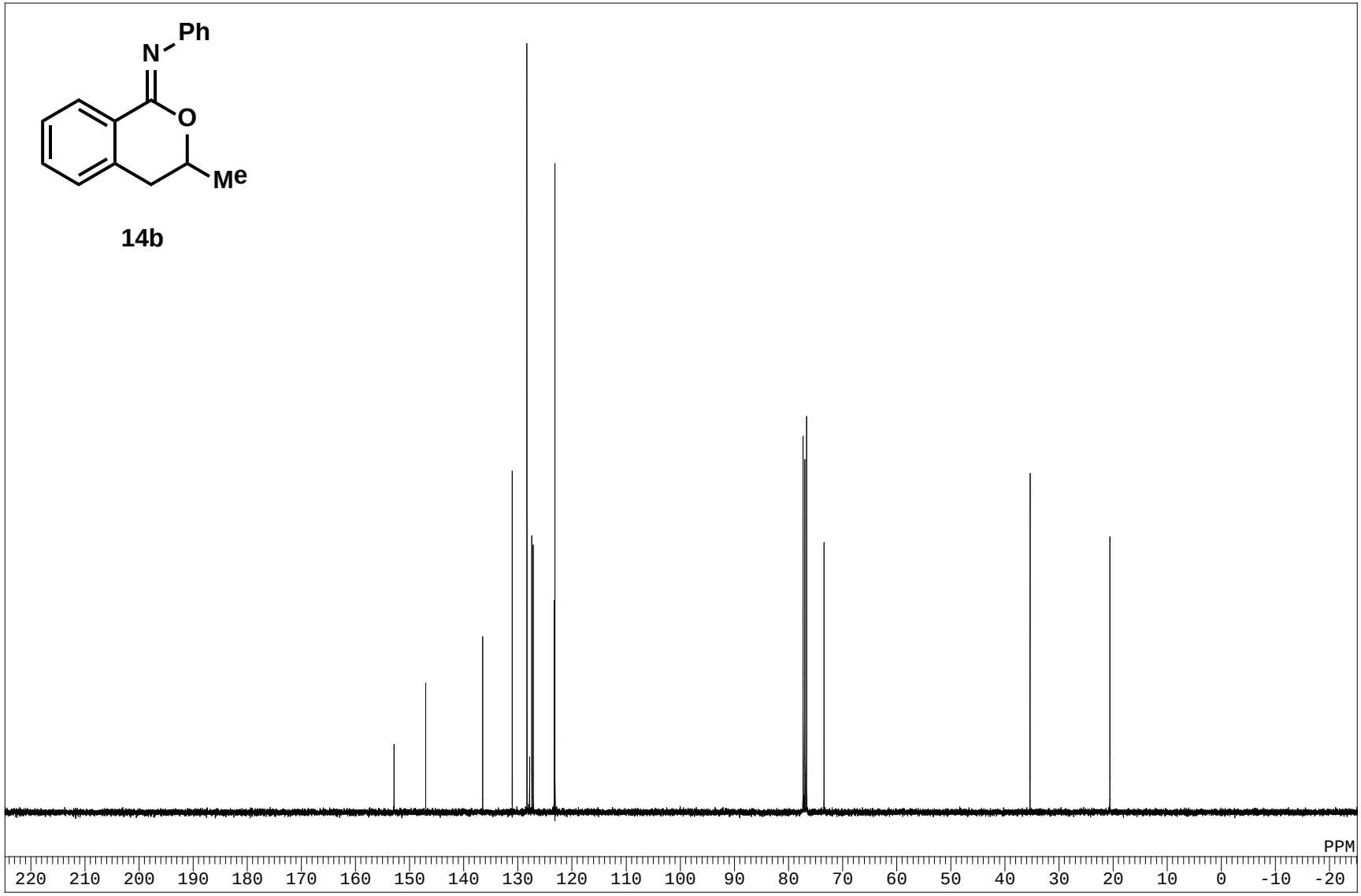
1
0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Me
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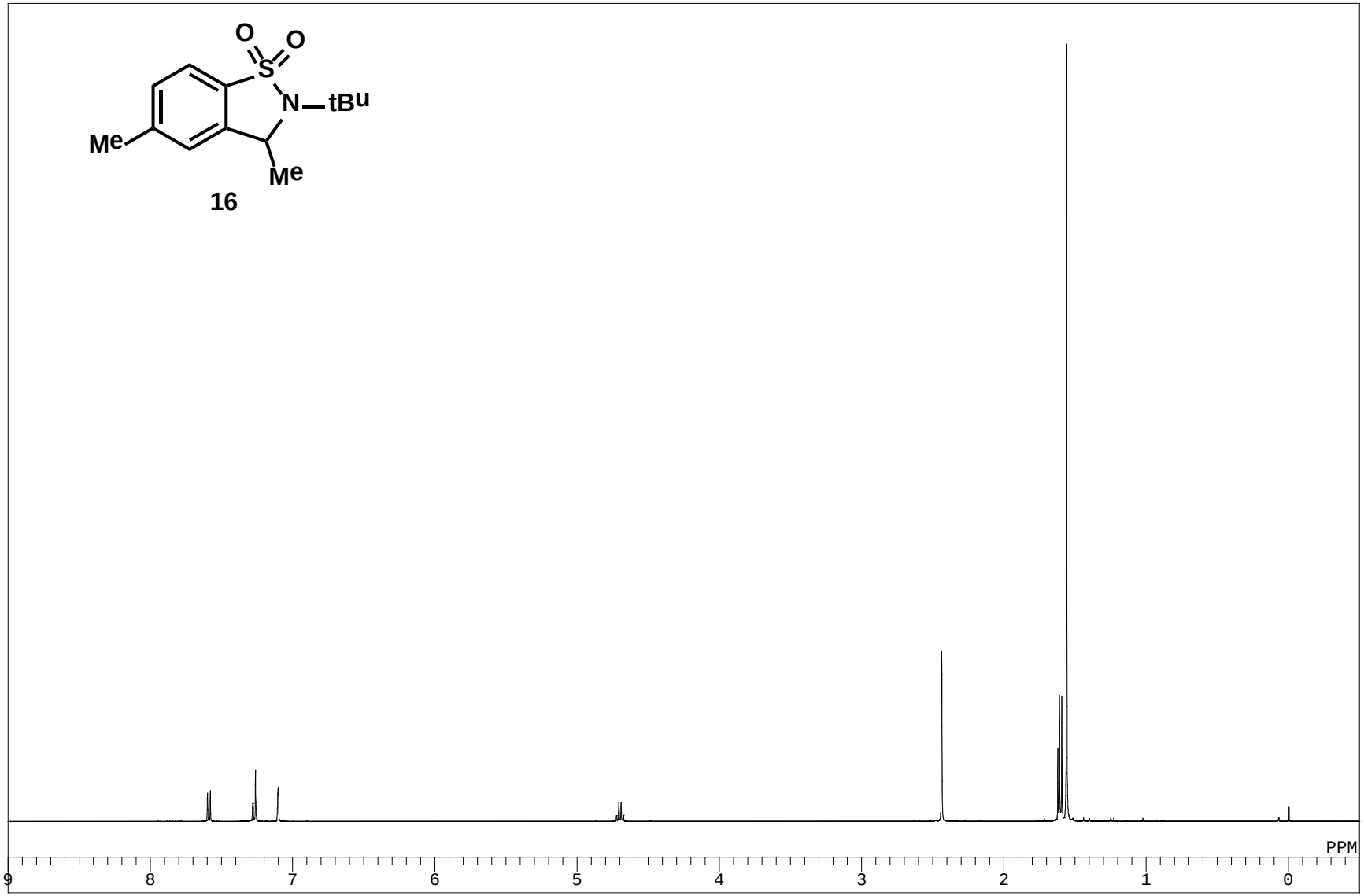
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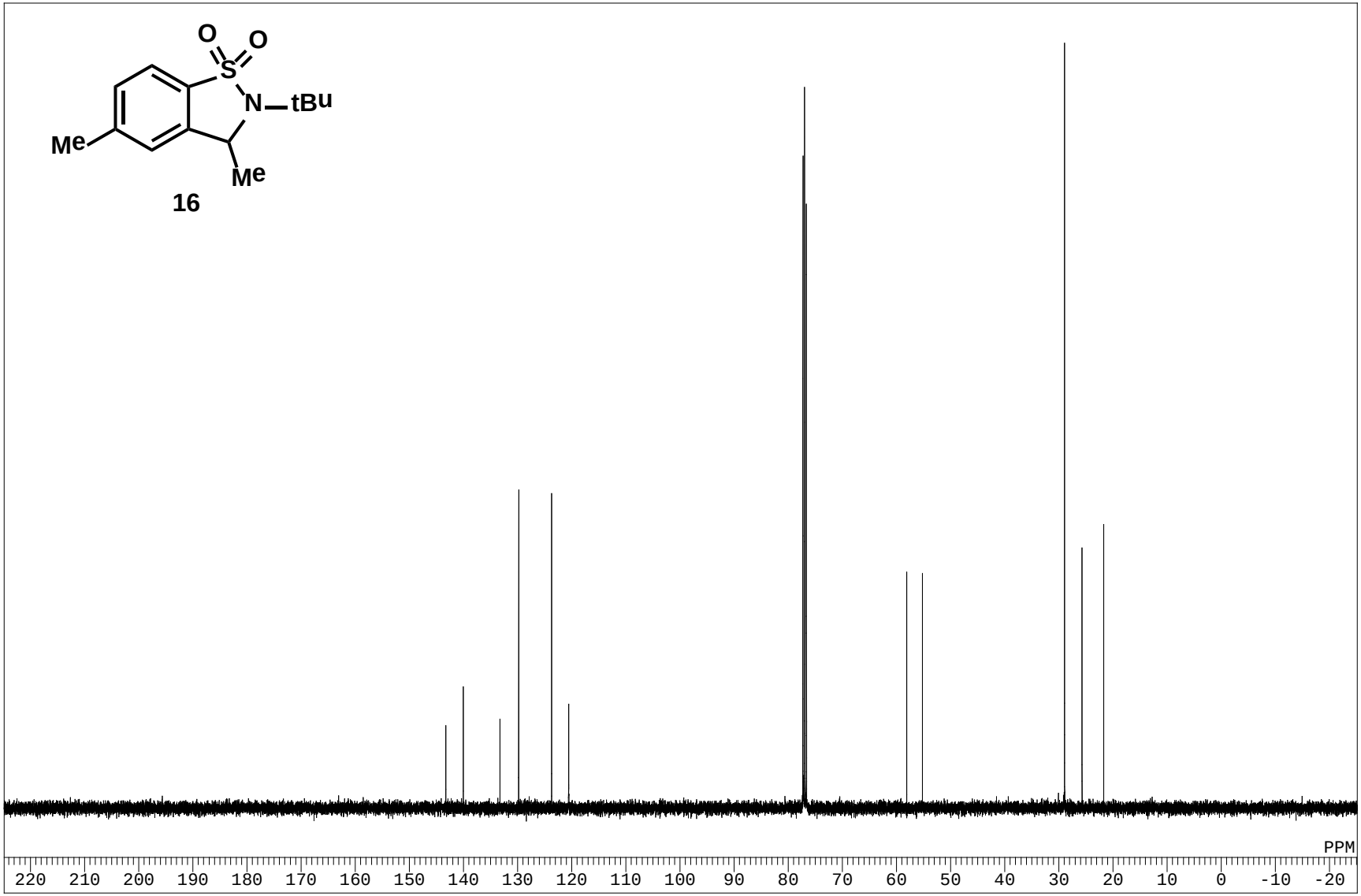
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16
PPM
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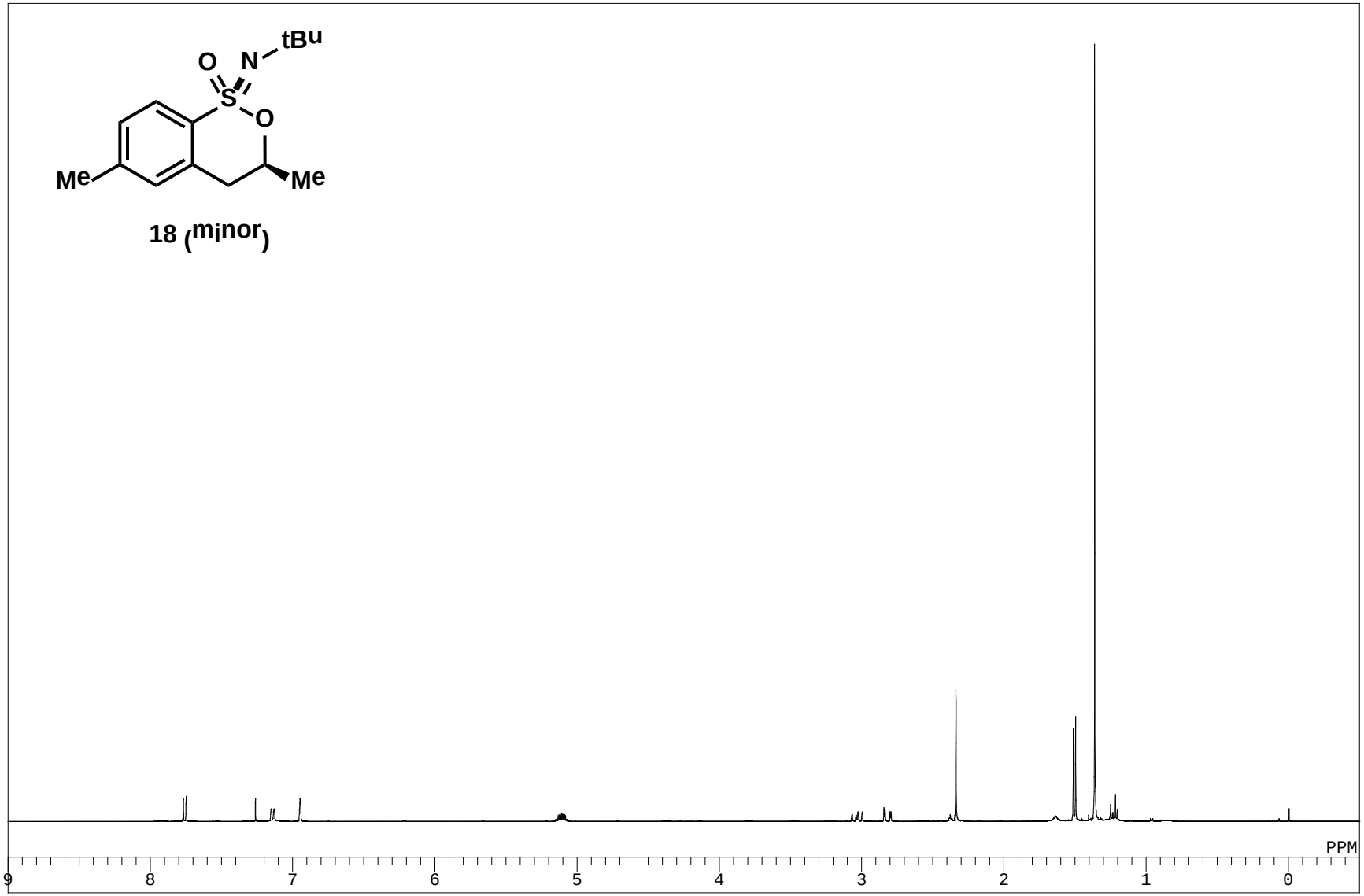
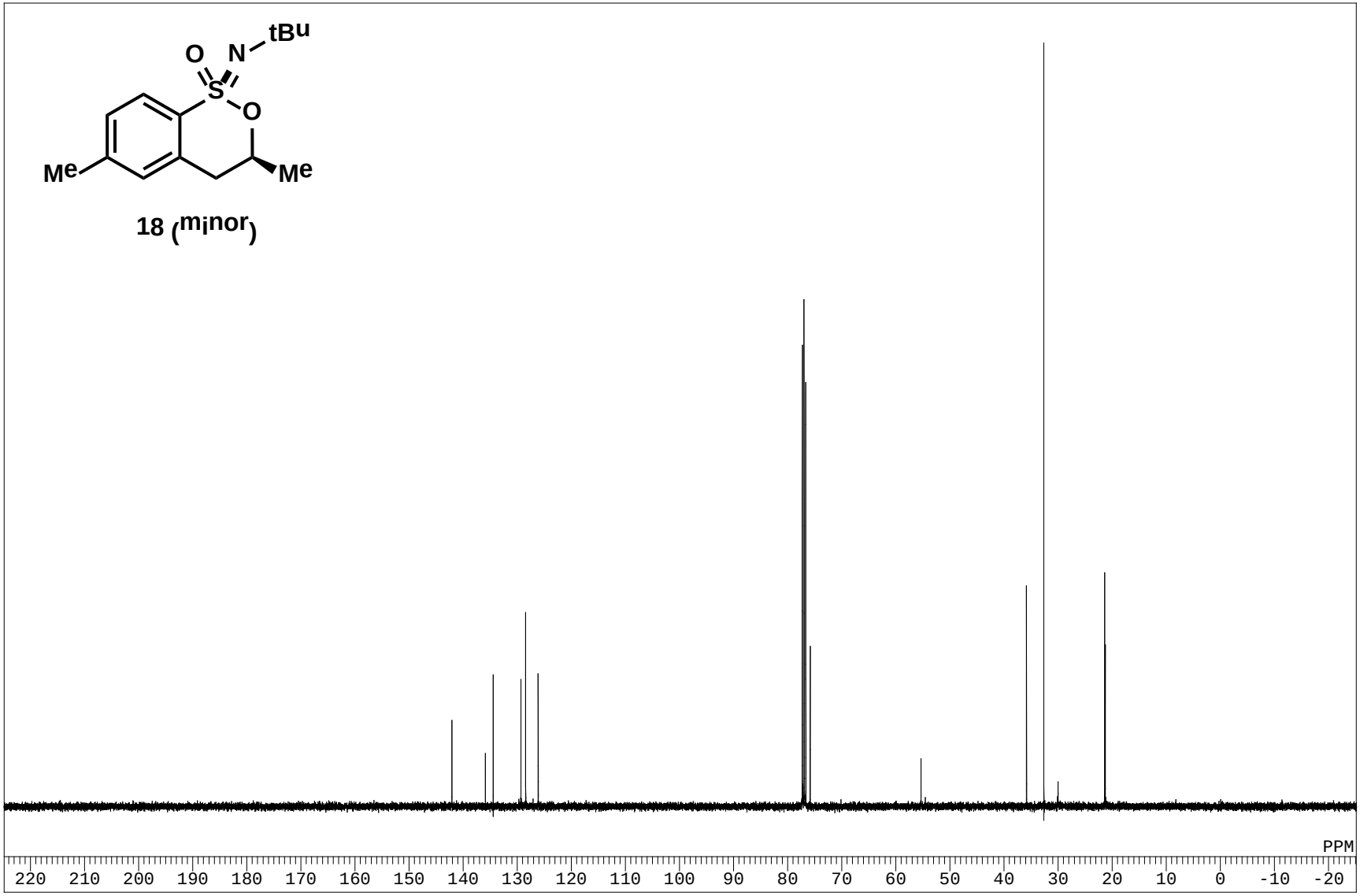
18 (minor)

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Me
18 (minor)
PPM
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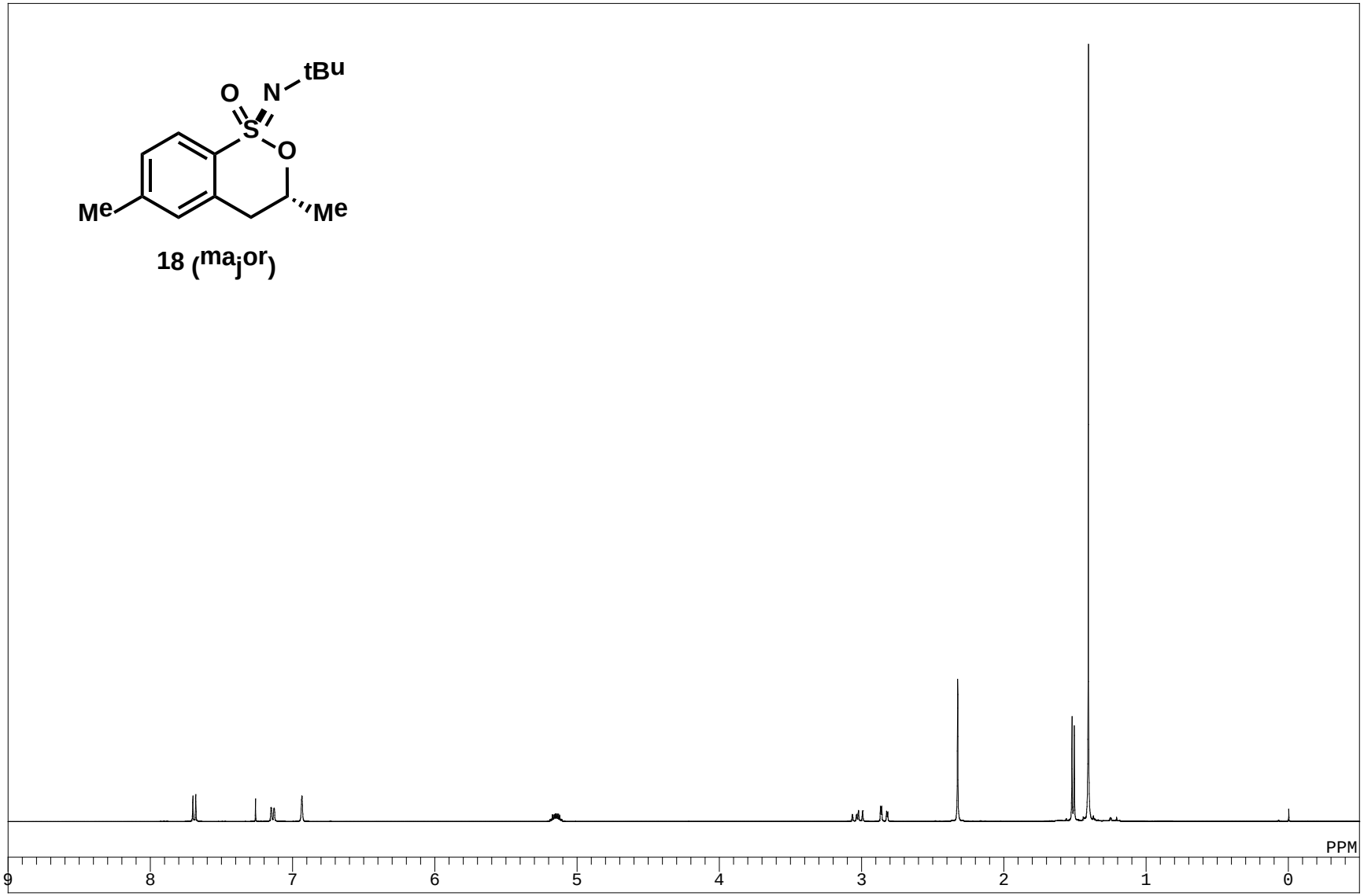
tBu
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18 (major)
PPM
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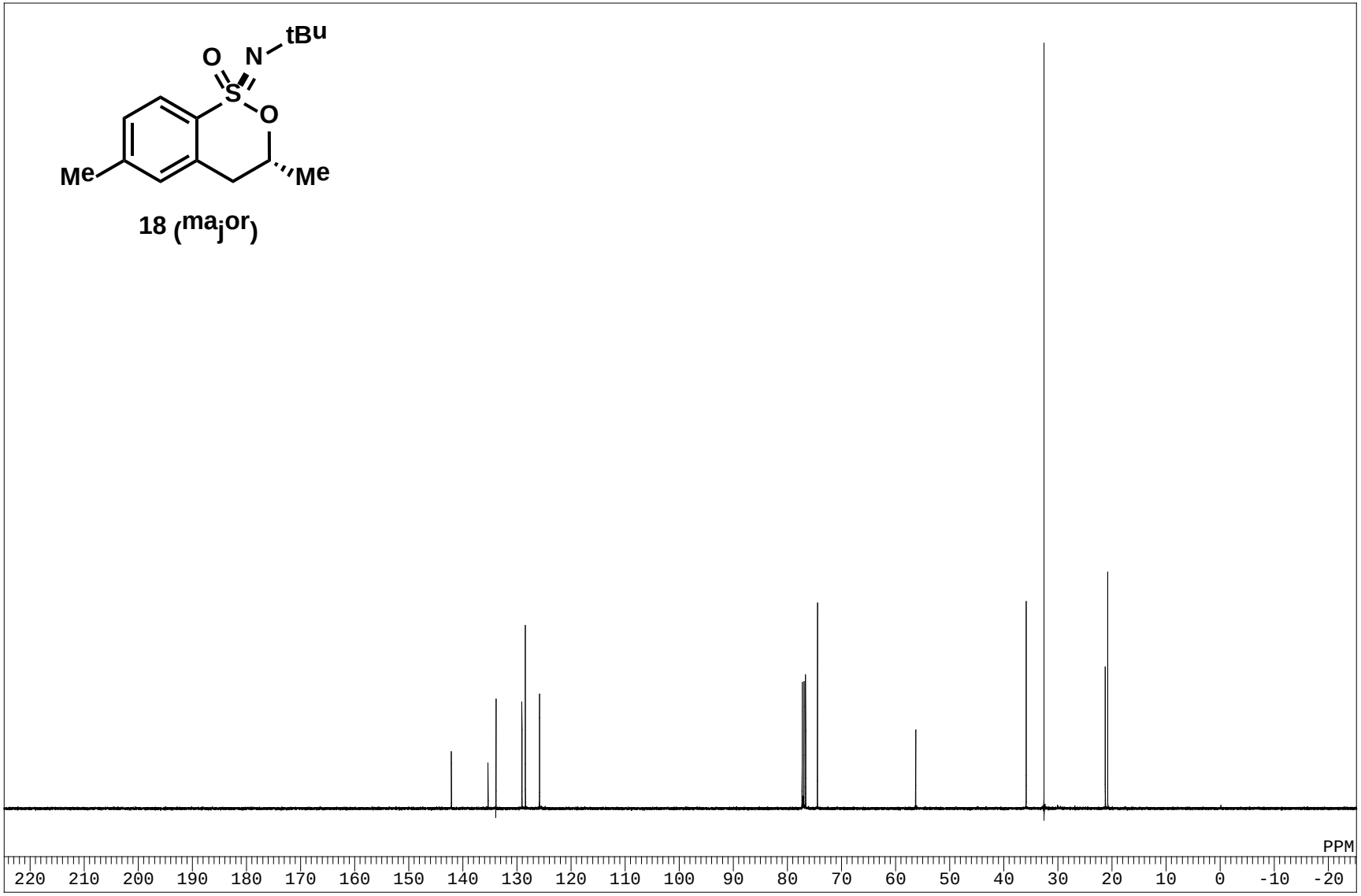
tBu
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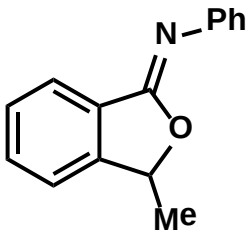
N
S
O
Me
Me
18 (major)
PPM
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20

X-ray crystallography data for 14a



14a

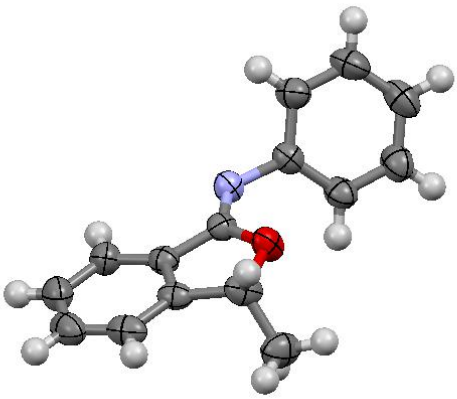


Table 1. Crystal data and structure refinement for CGE09128.

Identification code	CGE09128	
Empirical formula	C15 H13 N O	
Formula weight	223.26	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 7.7421(18) Å	α= 90°.
	b = 21.210(5) Å	β= 98.222(2)°.
	c = 7.4082(17) Å	γ = 90°.
Volume	1204.0(5) $Å^3$	
Z	4	
Density (calculated)	1.232 Mg/m^3	
Absorption coefficient	0.077 mm^{-1}	
F(000)	472	
Crystal size	0.22 x 0.20 x 0.16 mm^3	
Theta range for data collection	1.92 to 26.37°.	
Index ranges	-9<=h<=9, -26<=k<=26, -9<=l<=9	
Reflections collected	12252	
Independent reflections	2470 [R(int) = 0.0305]	
Completeness to theta = 26.37°	99.8 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9877 and 0.9832	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2470 / 0 / 155	

Goodness-of-fit on F^2	1.025
Final R indices [I>2sigma(I)]	R1 = 0.0417, wR2 = 0.1016
R indices (all data)	R1 = 0.0650, wR2 = 0.1136
Largest diff. peak and hole	0.130 and -0.150 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for CGE09128. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	9537(3)	2987(1)	8229(3)	66(1)
C(2)	10197(3)	3343(1)	6918(3)	62(1)
C(3)	9696(2)	3962(1)	6617(2)	51(1)
C(4)	8523(2)	4210(1)	7675(2)	42(1)
C(5)	7734(2)	4836(1)	7624(2)	40(1)
C(6)	7058(2)	5859(1)	6638(2)	43(1)
C(7)	7427(2)	6246(1)	8147(2)	52(1)
C(8)	6639(2)	6827(1)	8177(3)	62(1)
C(9)	5467(2)	7025(1)	6723(3)	66(1)
C(10)	5903(2)	6068(1)	5167(2)	56(1)
C(11)	5099(2)	6644(1)	5222(3)	66(1)
C(12)	6744(2)	4278(1)	9970(2)	52(1)
C(13)	7412(2)	4399(1)	11946(2)	68(1)
C(14)	7894(2)	3865(1)	9010(2)	46(1)
C(15)	8391(2)	3242(1)	9295(2)	58(1)
N(1)	7914(2)	5273(1)	6512(2)	45(1)
O(1)	6697(1)	4870(1)	8956(2)	52(1)

Table 3. Bond lengths [Å] and angles [°] for CGE09128.

C(1)-C(15)	1.380(3)
C(1)-C(2)	1.384(3)
C(1)-H(1)	0.9300
C(2)-C(3)	1.377(2)
C(2)-H(2)	0.9300
C(3)-C(4)	1.386(2)
C(3)-H(3)	0.9300
C(4)-C(14)	1.374(2)
C(4)-C(5)	1.459(2)
C(5)-N(1)	1.2613(19)
C(5)-O(1)	1.3600(18)
C(6)-C(10)	1.380(2)
C(6)-C(7)	1.383(2)
C(6)-N(1)	1.419(2)
C(7)-C(8)	1.377(2)
C(7)-H(7)	0.9300
C(8)-C(9)	1.371(3)
C(8)-H(8)	0.9300
C(9)-C(11)	1.371(3)
C(9)-H(9)	0.9300
C(10)-C(11)	1.375(2)
C(10)-H(10)	0.9300
C(11)-H(11)	0.9300
C(12)-O(1)	1.4608(19)
C(12)-C(14)	1.499(2)
C(12)-C(13)	1.503(2)
C(12)-H(12)	0.9800
C(13)-H(13A)	0.9600
C(13)-H(13B)	0.9600
C(13)-H(13C)	0.9600
C(14)-C(15)	1.385(2)
C(15)-H(15)	0.9300
C(15)-C(1)-C(2)	121.28(17)
C(15)-C(1)-H(1)	119.4
C(2)-C(1)-H(1)	119.4

C(3)-C(2)-C(1)	120.67(17)
C(3)-C(2)-H(2)	119.7
C(1)-C(2)-H(2)	119.7
C(2)-C(3)-C(4)	117.59(16)
C(2)-C(3)-H(3)	121.2
C(4)-C(3)-H(3)	121.2
C(14)-C(4)-C(3)	122.17(15)
C(14)-C(4)-C(5)	108.22(13)
C(3)-C(4)-C(5)	129.60(14)
N(1)-C(5)-O(1)	124.57(14)
N(1)-C(5)-C(4)	126.87(14)
O(1)-C(5)-C(4)	108.53(12)
C(10)-C(6)-C(7)	118.94(15)
C(10)-C(6)-N(1)	119.01(15)
C(7)-C(6)-N(1)	121.92(14)
C(8)-C(7)-C(6)	120.23(16)
C(8)-C(7)-H(7)	119.9
C(6)-C(7)-H(7)	119.9
C(9)-C(8)-C(7)	120.39(17)
C(9)-C(8)-H(8)	119.8
C(7)-C(8)-H(8)	119.8
C(11)-C(9)-C(8)	119.63(17)
C(11)-C(9)-H(9)	120.2
C(8)-C(9)-H(9)	120.2
C(11)-C(10)-C(6)	120.42(17)
C(11)-C(10)-H(10)	119.8
C(6)-C(10)-H(10)	119.8
C(9)-C(11)-C(10)	120.37(17)
C(9)-C(11)-H(11)	119.8
C(10)-C(11)-H(11)	119.8
O(1)-C(12)-C(14)	103.31(12)
O(1)-C(12)-C(13)	109.52(14)
C(14)-C(12)-C(13)	114.63(14)
O(1)-C(12)-H(12)	109.7
C(14)-C(12)-H(12)	109.7
C(13)-C(12)-H(12)	109.7
C(12)-C(13)-H(13A)	109.5
C(12)-C(13)-H(13B)	109.5

H(13A)-C(13)-H(13B)	109.5
C(12)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
C(4)-C(14)-C(15)	119.93(16)
C(4)-C(14)-C(12)	109.05(14)
C(15)-C(14)-C(12)	130.99(15)
C(1)-C(15)-C(14)	118.33(17)
C(1)-C(15)-H(15)	120.8
C(14)-C(15)-H(15)	120.8
C(5)-N(1)-C(6)	120.30(13)
C(5)-O(1)-C(12)	110.73(12)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x 10^3$) for CGE09128. 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}
C(1)	73(1)	43(1)	74(1)	-3(1)	-15(1)	4(1)
C(2)	66(1)	55(1)	62(1)	-10(1)	2(1)	11(1)
C(3)	55(1)	50(1)	49(1)	-3(1)	7(1)	4(1)
C(4)	40(1)	43(1)	43(1)	0(1)	1(1)	-2(1)
C(5)	32(1)	48(1)	40(1)	0(1)	5(1)	-1(1)
C(6)	37(1)	42(1)	50(1)	7(1)	10(1)	-2(1)
C(7)	44(1)	52(1)	59(1)	0(1)	1(1)	1(1)
C(8)	59(1)	50(1)	76(1)	-10(1)	3(1)	0(1)
C(9)	59(1)	46(1)	92(2)	7(1)	8(1)	8(1)
C(10)	57(1)	55(1)	53(1)	5(1)	0(1)	0(1)
C(11)	58(1)	61(1)	72(1)	15(1)	-6(1)	8(1)
C(12)	38(1)	62(1)	56(1)	14(1)	10(1)	-6(1)
C(13)	61(1)	91(2)	54(1)	7(1)	15(1)	2(1)
C(14)	38(1)	48(1)	48(1)	6(1)	-4(1)	-7(1)
C(15)	58(1)	51(1)	62(1)	11(1)	-8(1)	-11(1)
N(1)	44(1)	45(1)	48(1)	4(1)	12(1)	2(1)
O(1)	46(1)	57(1)	57(1)	11(1)	20(1)	7(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^{3}) for CGE09128.

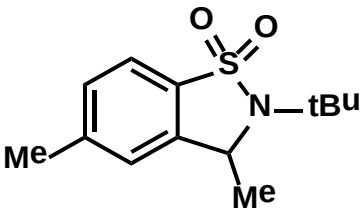
	x	y	z	U(eq)
H(1)	9872	2567	8394	79
H(2)	10986	3163	6234	74
H(3)	10130	4204	5736	62
H(7)	8210	6113	9145	63
H(8)	6903	7087	9190	75
H(9)	4925	7415	6754	79
H(10)	5668	5817	4131	67
H(11)	4300	6776	4236	79
H(12)	5568	4097	9855	62
H(13A)	8572	4568	12056	102
H(13B)	7430	4010	12615	102
H(13C)	6661	4695	12431	102
H(15)	7964	3001	10183	70

Table 6. Torsion angles [°] for CGE09128.

C(15)-C(1)-C(2)-C(3)	-1.3(3)
C(1)-C(2)-C(3)-C(4)	0.2(3)
C(2)-C(3)-C(4)-C(14)	1.4(2)
C(2)-C(3)-C(4)-C(5)	-178.03(15)
C(14)-C(4)-C(5)-N(1)	-175.76(15)
C(3)-C(4)-C(5)-N(1)	3.7(3)
C(14)-C(4)-C(5)-O(1)	2.13(16)
C(3)-C(4)-C(5)-O(1)	-178.38(15)
C(10)-C(6)-C(7)-C(8)	0.5(2)
N(1)-C(6)-C(7)-C(8)	176.40(14)
C(6)-C(7)-C(8)-C(9)	0.8(3)
C(7)-C(8)-C(9)-C(11)	-0.9(3)
C(7)-C(6)-C(10)-C(11)	-1.7(2)
N(1)-C(6)-C(10)-C(11)	-177.72(15)
C(8)-C(9)-C(11)-C(10)	-0.3(3)
C(6)-C(10)-C(11)-C(9)	1.6(3)
C(3)-C(4)-C(14)-C(15)	-2.0(2)
C(5)-C(4)-C(14)-C(15)	177.57(14)
C(3)-C(4)-C(14)-C(12)	176.70(14)
C(5)-C(4)-C(14)-C(12)	-3.76(16)
O(1)-C(12)-C(14)-C(4)	3.89(16)
C(13)-C(12)-C(14)-C(4)	-115.21(15)
O(1)-C(12)-C(14)-C(15)	-177.64(15)
C(13)-C(12)-C(14)-C(15)	63.3(2)
C(2)-C(1)-C(15)-C(14)	0.8(3)
C(4)-C(14)-C(15)-C(1)	0.8(2)
C(12)-C(14)-C(15)-C(1)	-177.49(16)
O(1)-C(5)-N(1)-C(6)	2.5(2)
C(4)-C(5)-N(1)-C(6)	-179.96(13)
C(10)-C(6)-N(1)-C(5)	-120.85(17)
C(7)-C(6)-N(1)-C(5)	63.3(2)
N(1)-C(5)-O(1)-C(12)	178.39(14)
C(4)-C(5)-O(1)-C(12)	0.44(16)
C(14)-C(12)-O(1)-C(5)	-2.58(16)
C(13)-C(12)-O(1)-C(5)	119.99(14)

Symmetry transformations used to generate equivalent atoms:

X-ray crystallography data for 16



16

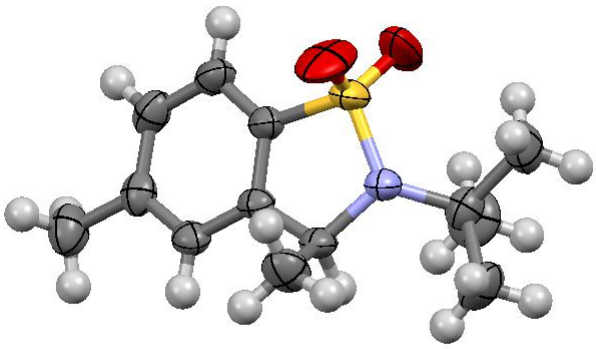


Table 1. Crystal data and structure refinement for CGE09105.

Identification code	CGE09105	
Empirical formula	C13 H19 N O2 S	
Formula weight	253.35	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 6.5204(11) Å	α= 90°.
	b = 26.602(4) Å	β= 101.144(2)°.
	c = 7.9704(13) Å	γ = 90°.
Volume	1356.4(4) $Å^3$	
Z	4	
Density (calculated)	1.241 Mg/m^3	
Absorption coefficient	0.229 mm^{-1}	
F(000)	544	
Crystal size	0.18 x 0.10 x 0.06 mm^3	
Theta range for data collection	1.53 to 26.36°.	
Index ranges	-8<=h<=8, -33<=k<=33, -9<=l<=9	
Reflections collected	14091	
Independent reflections	2760 [R(int) = 0.0324]	
Completeness to theta = 26.36°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9864 and 0.9599	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2760 / 0 / 159	
Goodness-of-fit on F^2	1.004	

Final R indices [I>2sigma(I)]	R1 = 0.0535, wR2 = 0.1223
R indices (all data)	R1 = 0.0632, wR2 = 0.1285
Largest diff. peak and hole	0.271 and -0.432 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for CGE09105. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	3523(5)	4248(1)	1105(4)	74(1)
C(2)	5702(4)	4123(1)	2131(3)	52(1)
C(3)	7625(3)	4063(1)	5257(3)	42(1)
C(4)	7542(3)	3605(1)	6347(3)	38(1)
C(5)	9161(3)	3392(1)	7514(3)	44(1)
C(6)	8832(4)	2967(1)	8419(3)	48(1)
C(7)	10577(5)	2727(1)	9671(4)	69(1)
C(8)	7139(5)	4567(1)	1955(4)	76(1)
C(9)	5580(3)	3392(1)	6144(3)	42(1)
C(10)	7954(4)	4538(1)	6333(4)	65(1)
C(11)	6822(4)	2757(1)	8156(3)	54(1)
C(12)	5189(4)	2968(1)	7036(3)	53(1)
C(13)	6544(6)	3646(1)	1465(4)	85(1)
N(1)	5618(3)	4079(1)	3985(2)	44(1)
O(1)	2659(4)	3436(1)	3375(3)	109(1)
O(2)	2647(3)	4063(1)	5586(3)	100(1)
S(1)	3833(1)	3744(1)	4669(1)	60(1)

Table 3. Bond lengths [Å] and angles [°] for CGE09105.

C(1)-C(2)	1.532(4)
C(1)-H(1A)	0.9600
C(1)-H(1B)	0.9600
C(1)-H(1C)	0.9600
C(2)-N(1)	1.494(3)
C(2)-C(13)	1.519(4)
C(2)-C(8)	1.529(4)
C(3)-N(1)	1.493(3)
C(3)-C(4)	1.504(3)
C(3)-C(10)	1.520(3)
C(3)-H(3)	0.9800
C(4)-C(9)	1.381(3)
C(4)-C(5)	1.385(3)
C(5)-C(6)	1.382(3)
C(5)-H(5)	0.9300
C(6)-C(11)	1.401(3)
C(6)-C(7)	1.503(3)
C(7)-H(7A)	0.9600
C(7)-H(7B)	0.9600
C(7)-H(7C)	0.9600
C(8)-H(8A)	0.9600
C(8)-H(8B)	0.9600
C(8)-H(8C)	0.9600
C(9)-C(12)	1.382(3)
C(9)-S(1)	1.743(2)
C(10)-H(10A)	0.9600
C(10)-H(10B)	0.9600
C(10)-H(10C)	0.9600
C(11)-C(12)	1.370(3)
C(11)-H(11)	0.9300
C(12)-H(12)	0.9300
C(13)-H(13A)	0.9600
C(13)-H(13B)	0.9600
C(13)-H(13C)	0.9600
N(1)-S(1)	1.6419(17)
O(1)-S(1)	1.419(2)

O(2)-S(1)	1.439(2)

C(2)-C(1)-H(1A)	109.5
C(2)-C(1)-H(1B)	109.5
H(1A)-C(1)-H(1B)	109.5
C(2)-C(1)-H(1C)	109.5
H(1A)-C(1)-H(1C)	109.5
H(1B)-C(1)-H(1C)	109.5
N(1)-C(2)-C(13)	111.4(2)
N(1)-C(2)-C(8)	106.9(2)
C(13)-C(2)-C(8)	110.1(2)
N(1)-C(2)-C(1)	109.7(2)
C(13)-C(2)-C(1)	110.8(2)
C(8)-C(2)-C(1)	107.8(2)
N(1)-C(3)-C(4)	106.91(16)
N(1)-C(3)-C(10)	111.61(18)
C(4)-C(3)-C(10)	111.44(19)
N(1)-C(3)-H(3)	108.9
C(4)-C(3)-H(3)	108.9
C(10)-C(3)-H(3)	108.9
C(9)-C(4)-C(5)	118.66(19)
C(9)-C(4)-C(3)	113.68(18)
C(5)-C(4)-C(3)	127.64(18)
C(6)-C(5)-C(4)	120.7(2)
C(6)-C(5)-H(5)	119.7
C(4)-C(5)-H(5)	119.7
C(5)-C(6)-C(11)	118.9(2)
C(5)-C(6)-C(7)	121.4(2)
C(11)-C(6)-C(7)	119.8(2)
C(6)-C(7)-H(7A)	109.5
C(6)-C(7)-H(7B)	109.5
H(7A)-C(7)-H(7B)	109.5
C(6)-C(7)-H(7C)	109.5
H(7A)-C(7)-H(7C)	109.5
H(7B)-C(7)-H(7C)	109.5
C(2)-C(8)-H(8A)	109.5
C(2)-C(8)-H(8B)	109.5
H(8A)-C(8)-H(8B)	109.5

C(2)-C(8)-H(8C)	109.5
H(8A)-C(8)-H(8C)	109.5
H(8B)-C(8)-H(8C)	109.5
C(4)-C(9)-C(12)	122.3(2)
C(4)-C(9)-S(1)	109.16(16)
C(12)-C(9)-S(1)	128.58(17)
C(3)-C(10)-H(10A)	109.5
C(3)-C(10)-H(10B)	109.5
H(10A)-C(10)-H(10B)	109.5
C(3)-C(10)-H(10C)	109.5
H(10A)-C(10)-H(10C)	109.5
H(10B)-C(10)-H(10C)	109.5
C(12)-C(11)-C(6)	121.4(2)
C(12)-C(11)-H(11)	119.3
C(6)-C(11)-H(11)	119.3
C(11)-C(12)-C(9)	118.1(2)
C(11)-C(12)-H(12)	121.0
C(9)-C(12)-H(12)	121.0
C(2)-C(13)-H(13A)	109.5
C(2)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
C(2)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
C(3)-N(1)-C(2)	118.61(17)
C(3)-N(1)-S(1)	110.10(13)
C(2)-N(1)-S(1)	122.34(15)
O(1)-S(1)-O(2)	116.22(16)
O(1)-S(1)-N(1)	112.94(12)
O(2)-S(1)-N(1)	109.38(12)
O(1)-S(1)-C(9)	111.79(13)
O(2)-S(1)-C(9)	108.67(12)
N(1)-S(1)-C(9)	95.93(9)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x 10^3$) for CGE09105. 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}
C(1)	78(2)	76(2)	60(2)	14(1)	-5(1)	4(2)
C(2)	61(1)	49(1)	49(1)	6(1)	15(1)	4(1)
C(3)	29(1)	51(1)	49(1)	6(1)	9(1)	-3(1)
C(4)	33(1)	42(1)	42(1)	-3(1)	13(1)	-3(1)
C(5)	35(1)	52(1)	47(1)	-1(1)	11(1)	2(1)
C(6)	57(1)	49(1)	39(1)	-1(1)	13(1)	10(1)
C(7)	73(2)	74(2)	59(2)	15(1)	11(1)	20(1)
C(8)	86(2)	75(2)	73(2)	24(2)	27(2)	-9(2)
C(9)	38(1)	48(1)	40(1)	-1(1)	9(1)	-6(1)
C(10)	66(2)	50(1)	76(2)	-1(1)	3(1)	-13(1)
C(11)	74(2)	44(1)	46(1)	2(1)	18(1)	-8(1)
C(12)	55(1)	55(1)	49(1)	-2(1)	14(1)	-21(1)
C(13)	103(2)	77(2)	80(2)	-16(2)	30(2)	17(2)
N(1)	32(1)	54(1)	47(1)	7(1)	9(1)	-3(1)
O(1)	90(2)	138(2)	78(2)	36(1)	-32(1)	-70(2)
O(2)	49(1)	129(2)	135(2)	47(2)	54(1)	33(1)
S(1)	30(1)	83(1)	64(1)	21(1)	4(1)	-11(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^{-3}) for CGE09105.

	x	y	z	U(eq)
H(1A)	2623	3962	1100	110
H(1B)	3623	4331	-49	110
H(1C)	2957	4529	1620	110
H(3)	8785	4027	4646	51
H(5)	10482	3538	7690	53
H(7A)	11886	2872	9545	104
H(7B)	10599	2372	9453	104
H(7C)	10362	2782	10814	104
H(8A)	6647	4861	2449	114
H(8B)	7137	4626	767	114
H(8C)	8534	4491	2539	114
H(10A)	7995	4824	5603	98
H(10B)	9250	4515	7139	98
H(10C)	6824	4577	6936	98
H(11)	6593	2469	8755	65
H(12)	3856	2829	6880	63
H(13A)	7913	3577	2116	127
H(13B)	6623	3690	283	127
H(13C)	5629	3371	1574	127

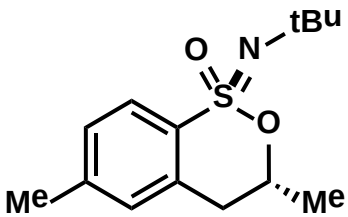
Table 6. Torsion angles [°] for CGE09105.

N(1)-C(3)-C(4)-C(9)	-12.5(2)
C(10)-C(3)-C(4)-C(9)	109.7(2)
N(1)-C(3)-C(4)-C(5)	169.20(19)
C(10)-C(3)-C(4)-C(5)	-68.6(3)
C(9)-C(4)-C(5)-C(6)	2.0(3)
C(3)-C(4)-C(5)-C(6)	-179.8(2)
C(4)-C(5)-C(6)-C(11)	-1.0(3)
C(4)-C(5)-C(6)-C(7)	179.0(2)
C(5)-C(4)-C(9)-C(12)	-1.5(3)
C(3)-C(4)-C(9)-C(12)	180.0(2)
C(5)-C(4)-C(9)-S(1)	177.82(16)
C(3)-C(4)-C(9)-S(1)	-0.6(2)
C(5)-C(6)-C(11)-C(12)	-0.5(3)
C(7)-C(6)-C(11)-C(12)	179.5(2)
C(6)-C(11)-C(12)-C(9)	0.9(3)
C(4)-C(9)-C(12)-C(11)	0.1(3)
S(1)-C(9)-C(12)-C(11)	-179.12(18)
C(4)-C(3)-N(1)-C(2)	-127.21(19)
C(10)-C(3)-N(1)-C(2)	110.7(2)
C(4)-C(3)-N(1)-S(1)	20.8(2)
C(10)-C(3)-N(1)-S(1)	-101.26(19)
C(13)-C(2)-N(1)-C(3)	69.2(3)
C(8)-C(2)-N(1)-C(3)	-51.1(3)
C(1)-C(2)-N(1)-C(3)	-167.8(2)
C(13)-C(2)-N(1)-S(1)	-74.8(3)
C(8)-C(2)-N(1)-S(1)	164.92(18)
C(1)-C(2)-N(1)-S(1)	48.3(3)
C(3)-N(1)-S(1)-O(1)	-135.97(18)
C(2)-N(1)-S(1)-O(1)	10.7(2)
C(3)-N(1)-S(1)-O(2)	92.92(17)
C(2)-N(1)-S(1)-O(2)	-120.45(19)
C(3)-N(1)-S(1)-C(9)	-19.27(16)
C(2)-N(1)-S(1)-C(9)	127.35(17)
C(4)-C(9)-S(1)-O(1)	129.48(18)
C(12)-C(9)-S(1)-O(1)	-51.2(3)
C(4)-C(9)-S(1)-O(2)	-100.93(18)

C(12)-C(9)-S(1)-O(2)	78.4(2)
C(4)-C(9)-S(1)-N(1)	11.86(17)
C(12)-C(9)-S(1)-N(1)	-168.8(2)

Symmetry transformations used to generate equivalent atoms:

X-ray crystallography data for 18



18 (major)

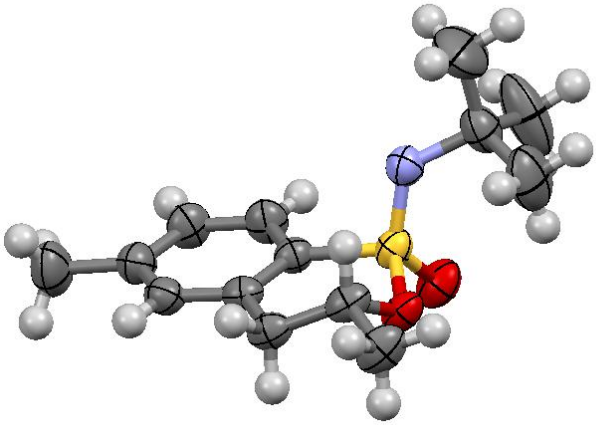


Table 1. Crystal data and structure refinement for Shigehisa_01.

Identification code	Shigehisa_01	
Empirical formula	C14 H21 N O2 S	
Formula weight	267.38	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pbca	
Unit cell dimensions	a = 17.888(3) Å	α= 90°.
	b = 9.4477(14) Å	β= 90°.
	c = 18.022(3) Å	γ = 90°.
Volume	3045.7(8) $Å^3$	
Z	8	
Density (calculated)	1.166 Mg/m^3	
Absorption coefficient	0.208 mm^{-1}	
F(000)	1152	
Crystal size	0.25 x 0.18 x 0.10 mm^3	
Theta range for data collection	2.26 to 26.37°.	
Index ranges	-22<=h<=22, -11<=k<=11, -22<=l<=22	
Reflections collected	30548	
Independent reflections	3118 [R(int) = 0.0270]	
Completeness to theta = 26.37°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9795 and 0.9499	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3118 / 0 / 168	
Goodness-of-fit on F^2	0.939	

Final R indices [I>2sigma(I)]	R1 = 0.0407, wR2 = 0.1119
R indices (all data)	R1 = 0.0491, wR2 = 0.1205
Largest diff. peak and hole	0.320 and -0.316 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for Shigehisa_01. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	1328(1)	3898(2)	1904(1)	44(1)
C(2)	1958(1)	4398(2)	2276(1)	44(1)
C(3)	2746(1)	4002(2)	2052(1)	50(1)
C(4)	2801(1)	3434(2)	1274(1)	50(1)
C(5)	3558(1)	2815(3)	1087(1)	68(1)
C(6)	1836(1)	5287(2)	2879(1)	52(1)
C(7)	1128(1)	5671(2)	3115(1)	56(1)
C(8)	514(1)	5157(2)	2732(1)	60(1)
C(9)	610(1)	4277(2)	2131(1)	56(1)
C(10)	1028(2)	6623(3)	3777(1)	83(1)
C(11)	1180(1)	3182(3)	-295(1)	64(1)
C(12)	1188(2)	4530(4)	-756(2)	122(1)
C(13)	1847(2)	2284(4)	-522(2)	100(1)
C(14)	471(2)	2368(5)	-408(2)	127(2)
N(1)	1241(1)	3672(2)	486(1)	52(1)
O(1)	1015(1)	1444(1)	1278(1)	63(1)
O(2)	2259(1)	2265(1)	1213(1)	51(1)
S(1)	1403(1)	2754(1)	1141(1)	46(1)

Table 3. Bond lengths [Å] and angles [°] for Shigehisa_01.

C(1)-C(2)	1.394(2)
C(1)-C(9)	1.393(2)
C(1)-S(1)	1.7549(18)
C(2)-C(6)	1.391(3)
C(2)-C(3)	1.513(2)
C(3)-C(4)	1.504(3)
C(3)-H(3A)	0.9700
C(3)-H(3B)	0.9700
C(4)-O(2)	1.473(2)
C(4)-C(5)	1.513(2)
C(4)-H(4)	0.9800
C(5)-H(5A)	0.9600
C(5)-H(5B)	0.9600
C(5)-H(5C)	0.9600
C(6)-C(7)	1.385(3)
C(6)-H(6)	0.9300
C(7)-C(8)	1.384(3)
C(7)-C(10)	1.506(3)
C(8)-C(9)	1.376(3)
C(8)-H(8)	0.9300
C(9)-H(9)	0.9300
C(10)-H(10A)	0.9600
C(10)-H(10B)	0.9600
C(10)-H(10C)	0.9600
C(11)-N(1)	1.486(2)
C(11)-C(14)	1.497(3)
C(11)-C(13)	1.520(3)
C(11)-C(12)	1.520(4)
C(12)-H(12A)	0.9600
C(12)-H(12B)	0.9600
C(12)-H(12C)	0.9600
C(13)-H(13A)	0.9600
C(13)-H(13B)	0.9600
C(13)-H(13C)	0.9600
C(14)-H(14A)	0.9600
C(14)-H(14B)	0.9600

C(14)-H(14C)	0.9600
N(1)-S(1)	1.4919(17)
O(1)-S(1)	1.4403(14)
O(2)-S(1)	1.6056(13)

C(2)-C(1)-C(9)	121.20(17)
C(2)-C(1)-S(1)	121.54(13)
C(9)-C(1)-S(1)	117.25(14)
C(1)-C(2)-C(6)	116.94(16)
C(1)-C(2)-C(3)	122.81(16)
C(6)-C(2)-C(3)	120.25(16)
C(4)-C(3)-C(2)	113.47(15)
C(4)-C(3)-H(3A)	108.9
C(2)-C(3)-H(3A)	108.9
C(4)-C(3)-H(3B)	108.9
C(2)-C(3)-H(3B)	108.9
H(3A)-C(3)-H(3B)	107.7
O(2)-C(4)-C(5)	106.41(15)
O(2)-C(4)-C(3)	107.11(14)
C(5)-C(4)-C(3)	113.89(17)
O(2)-C(4)-H(4)	109.8
C(5)-C(4)-H(4)	109.8
C(3)-C(4)-H(4)	109.8
C(4)-C(5)-H(5A)	109.5
C(4)-C(5)-H(5B)	109.5
H(5A)-C(5)-H(5B)	109.5
C(4)-C(5)-H(5C)	109.5
H(5A)-C(5)-H(5C)	109.5
H(5B)-C(5)-H(5C)	109.5
C(7)-C(6)-C(2)	122.74(18)
C(7)-C(6)-H(6)	118.6
C(2)-C(6)-H(6)	118.6
C(6)-C(7)-C(8)	118.75(18)
C(6)-C(7)-C(10)	120.5(2)
C(8)-C(7)-C(10)	120.7(2)
C(9)-C(8)-C(7)	120.37(18)
C(9)-C(8)-H(8)	119.8
C(7)-C(8)-H(8)	119.8

C(8)-C(9)-C(1)	120.00(18)
C(8)-C(9)-H(9)	120.0
C(1)-C(9)-H(9)	120.0
C(7)-C(10)-H(10A)	109.5
C(7)-C(10)-H(10B)	109.5
H(10A)-C(10)-H(10B)	109.5
C(7)-C(10)-H(10C)	109.5
H(10A)-C(10)-H(10C)	109.5
H(10B)-C(10)-H(10C)	109.5
N(1)-C(11)-C(14)	110.56(19)
N(1)-C(11)-C(13)	111.74(19)
C(14)-C(11)-C(13)	110.0(2)
N(1)-C(11)-C(12)	104.8(2)
C(14)-C(11)-C(12)	111.3(3)
C(13)-C(11)-C(12)	108.3(2)
C(11)-C(12)-H(12A)	109.5
C(11)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
C(11)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
C(11)-C(13)-H(13A)	109.5
C(11)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
C(11)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
C(11)-C(14)-H(14A)	109.5
C(11)-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14B)	109.5
C(11)-C(14)-H(14C)	109.5
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
C(11)-N(1)-S(1)	125.62(14)
C(4)-O(2)-S(1)	114.75(11)
O(1)-S(1)-N(1)	122.80(9)
O(1)-S(1)-O(2)	101.44(8)
N(1)-S(1)-O(2)	114.54(8)

O(1)-S(1)-C(1)	111.00(9)
N(1)-S(1)-C(1)	104.32(9)
O(2)-S(1)-C(1)	100.78(7)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x 10^3$) for Shigehisa_01. 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}
C(1)	41(1)	44(1)	47(1)	3(1)	2(1)	2(1)
C(2)	41(1)	42(1)	48(1)	9(1)	-1(1)	2(1)
C(3)	37(1)	49(1)	64(1)	7(1)	-1(1)	1(1)
C(4)	39(1)	44(1)	68(1)	8(1)	8(1)	1(1)
C(5)	44(1)	66(1)	93(2)	0(1)	17(1)	7(1)
C(6)	51(1)	52(1)	52(1)	2(1)	-7(1)	0(1)
C(7)	60(1)	58(1)	51(1)	-4(1)	1(1)	8(1)
C(8)	45(1)	72(1)	63(1)	-8(1)	8(1)	9(1)
C(9)	39(1)	67(1)	61(1)	-8(1)	0(1)	2(1)
C(10)	83(2)	95(2)	72(2)	-30(1)	-1(1)	16(2)
C(11)	61(1)	80(1)	50(1)	-11(1)	1(1)	2(1)
C(12)	185(4)	123(3)	58(2)	8(2)	-6(2)	20(3)
C(13)	86(2)	134(3)	80(2)	-42(2)	14(1)	14(2)
C(14)	76(2)	222(4)	83(2)	-59(2)	1(2)	-46(2)
N(1)	53(1)	51(1)	51(1)	-6(1)	-1(1)	5(1)
O(1)	54(1)	46(1)	88(1)	-2(1)	7(1)	-10(1)
O(2)	43(1)	39(1)	71(1)	1(1)	6(1)	3(1)
S(1)	41(1)	40(1)	56(1)	-2(1)	4(1)	0(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for Shigehisa_01.

	x	y	z	U(eq)
H(3A)	3063	4831	2095	60
H(3B)	2934	3293	2394	60
H(4)	2675	4180	918	60
H(5A)	3684	2099	1444	101
H(5B)	3928	3550	1098	101
H(5C)	3542	2403	600	101
H(6)	2248	5638	3135	62
H(8)	35	5408	2881	72
H(9)	196	3935	1875	67
H(10A)	1277	6217	4198	125
H(10B)	505	6723	3884	125
H(10C)	1238	7536	3672	125
H(12A)	775	5120	-613	183
H(12B)	1145	4293	-1272	183
H(12C)	1648	5028	-672	183
H(13A)	2300	2799	-430	150
H(13B)	1812	2060	-1040	150
H(13C)	1850	1424	-238	150
H(14A)	491	1504	-128	190
H(14B)	412	2152	-925	190
H(14C)	54	2926	-242	190

Table 6. Torsion angles [°] for Shigehisa_01.

C(9)-C(1)-C(2)-C(6)	0.0(3)
S(1)-C(1)-C(2)-C(6)	-179.32(13)
C(9)-C(1)-C(2)-C(3)	179.46(17)
S(1)-C(1)-C(2)-C(3)	0.1(2)
C(1)-C(2)-C(3)-C(4)	18.6(2)
C(6)-C(2)-C(3)-C(4)	-161.96(16)
C(2)-C(3)-C(4)-O(2)	-53.24(19)
C(2)-C(3)-C(4)-C(5)	-170.60(16)
C(1)-C(2)-C(6)-C(7)	0.3(3)
C(3)-C(2)-C(6)-C(7)	-179.18(18)
C(2)-C(6)-C(7)-C(8)	-0.4(3)
C(2)-C(6)-C(7)-C(10)	179.3(2)
C(6)-C(7)-C(8)-C(9)	0.3(3)
C(10)-C(7)-C(8)-C(9)	-179.4(2)
C(7)-C(8)-C(9)-C(1)	0.0(3)
C(2)-C(1)-C(9)-C(8)	-0.2(3)
S(1)-C(1)-C(9)-C(8)	179.19(16)
C(14)-C(11)-N(1)-S(1)	72.3(3)
C(13)-C(11)-N(1)-S(1)	-50.6(3)
C(12)-C(11)-N(1)-S(1)	-167.6(2)
C(5)-C(4)-O(2)-S(1)	-162.09(13)
C(3)-C(4)-O(2)-S(1)	75.75(16)
C(11)-N(1)-S(1)-O(1)	-48.1(2)
C(11)-N(1)-S(1)-O(2)	75.56(18)
C(11)-N(1)-S(1)-C(1)	-175.25(16)
C(4)-O(2)-S(1)-O(1)	-166.32(13)
C(4)-O(2)-S(1)-N(1)	59.26(15)
C(4)-O(2)-S(1)-C(1)	-52.07(14)
C(2)-C(1)-S(1)-O(1)	121.10(15)
C(9)-C(1)-S(1)-O(1)	-58.25(17)
C(2)-C(1)-S(1)-N(1)	-104.72(16)
C(9)-C(1)-S(1)-N(1)	75.93(17)
C(2)-C(1)-S(1)-O(2)	14.29(16)
C(9)-C(1)-S(1)-O(2)	-165.07(15)

Symmetry transformations used to generate equivalent atoms: