

Supporting Information for
(6-(Trichloro(*p*-tolyl)- λ^5 -stibanyl)-1,2-dihydroacenaphthylen-5-yl)(diisopropyl)phosphine

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All NMR spectra were recorded using a JEOL GSX Delta (270 MHz) or Bruker Avance III (500 MHz) spectrometer at 25 °C. Assignments of ^1H and ^{13}C spectra were made in conjunction with appropriate 2D spectra. ^{13}C NMR spectra were recorded using the DEPTQ pulse sequence with broadband proton decoupling. For ^1H and ^{13}C NMR, tetramethylsilane was used as an external standard. For ^{31}P NMR, 85% H_3PO_4 in D_2O was used as the external standard. Residual solvent peaks were also used for secondary calibration (CDCl_3 δ_{H} 7.260 ppm; δ_{C} 77.160 ppm). Chemical shifts (δ) are given in parts per million (ppm). Coupling constants (J) are quoted in Hertz (Hz).

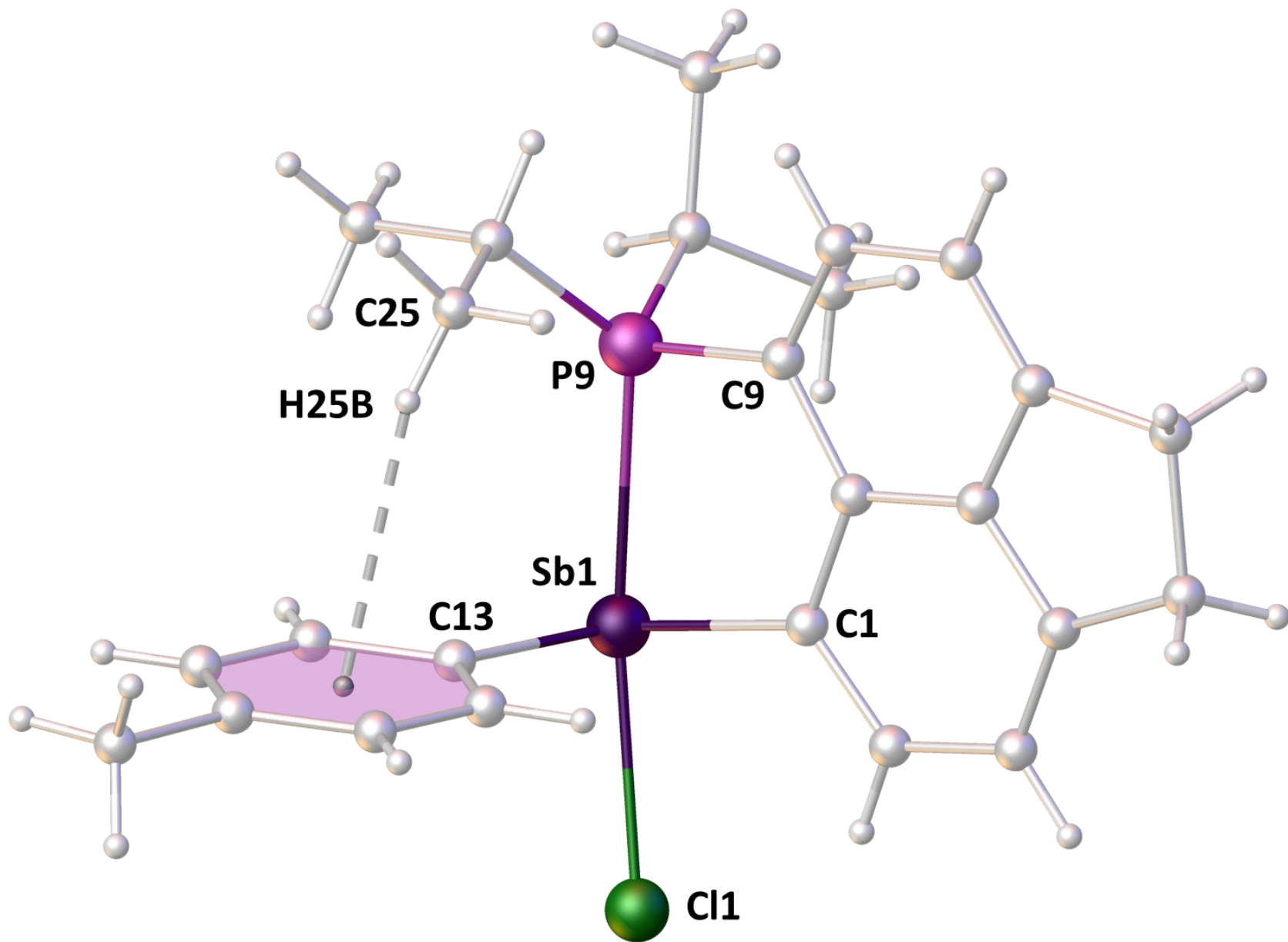


Figure S1: “ball and stick” model of the molecular structure of **1**, showing the CH... π interaction H25B to the centroid of the C13–C18 tolyl ring.

¹H Observe
jb008

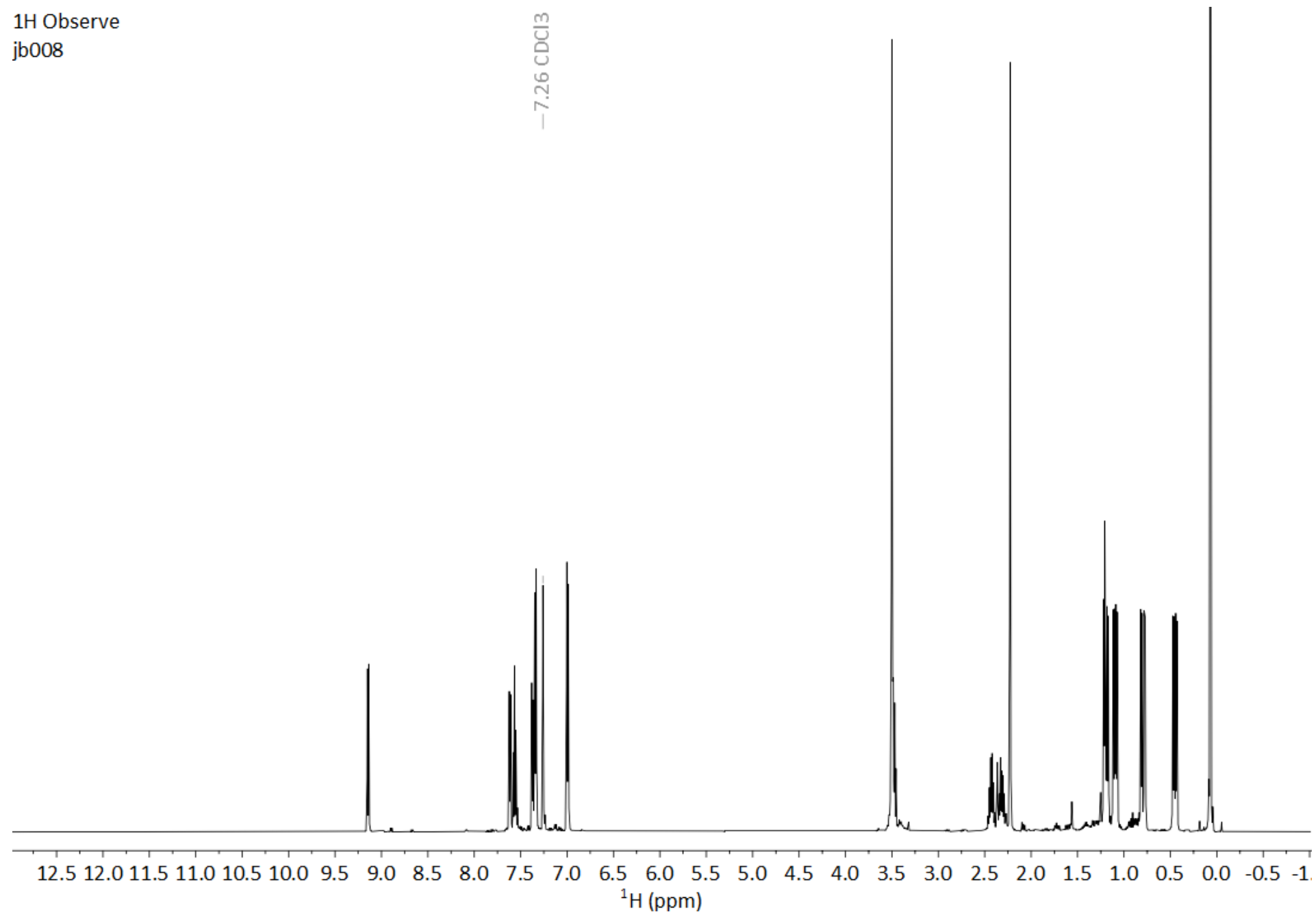


Figure S2: ¹H NMR spectrum of **1**, acquired in CDCl₃ at 500.1 MHz at ambient conditions.

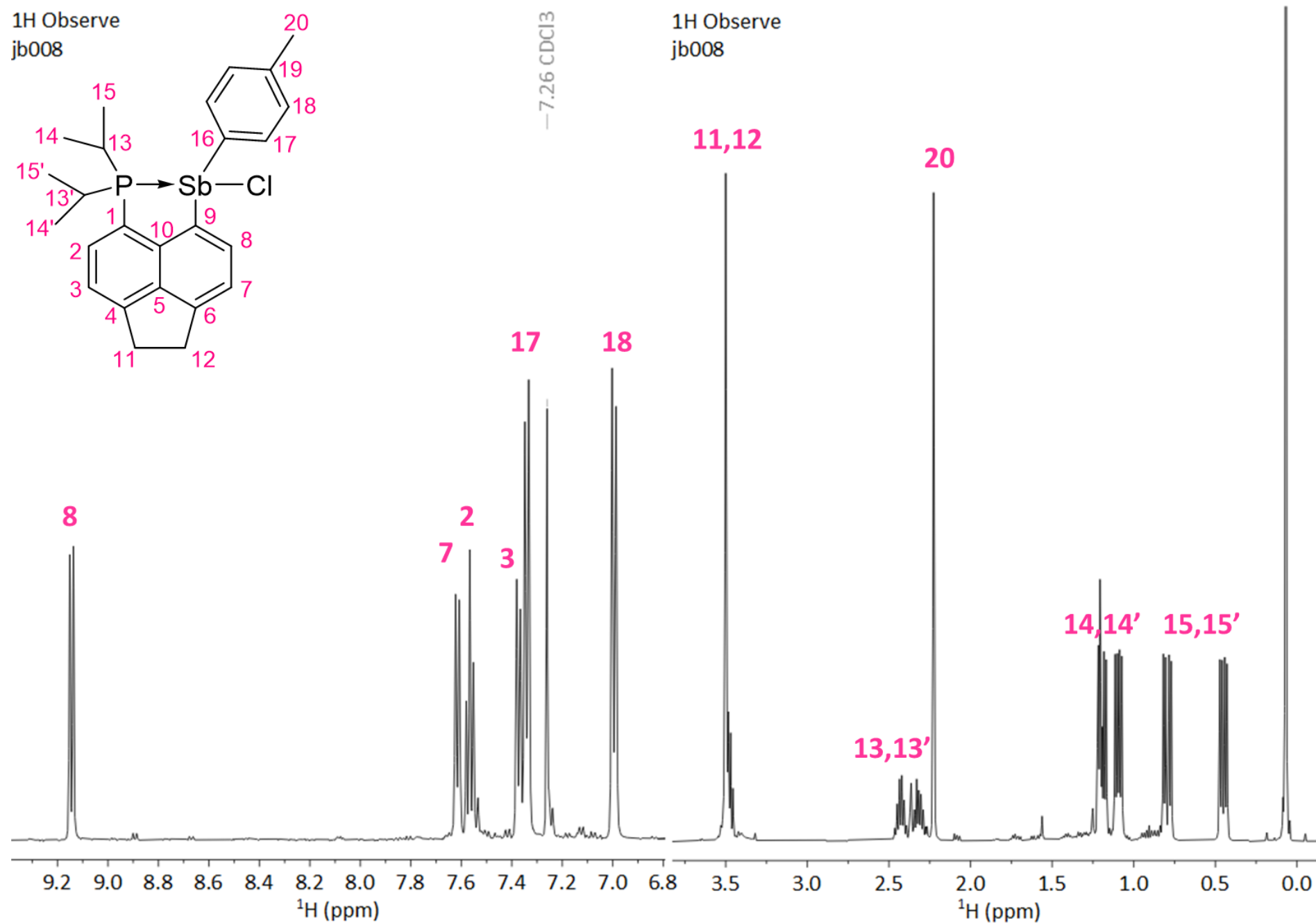


Figure S3: Expansions of the ¹H NMR spectrum of **1** with assignments.

¹³C Observe with multiplicity editing - DEPTQ
jb008

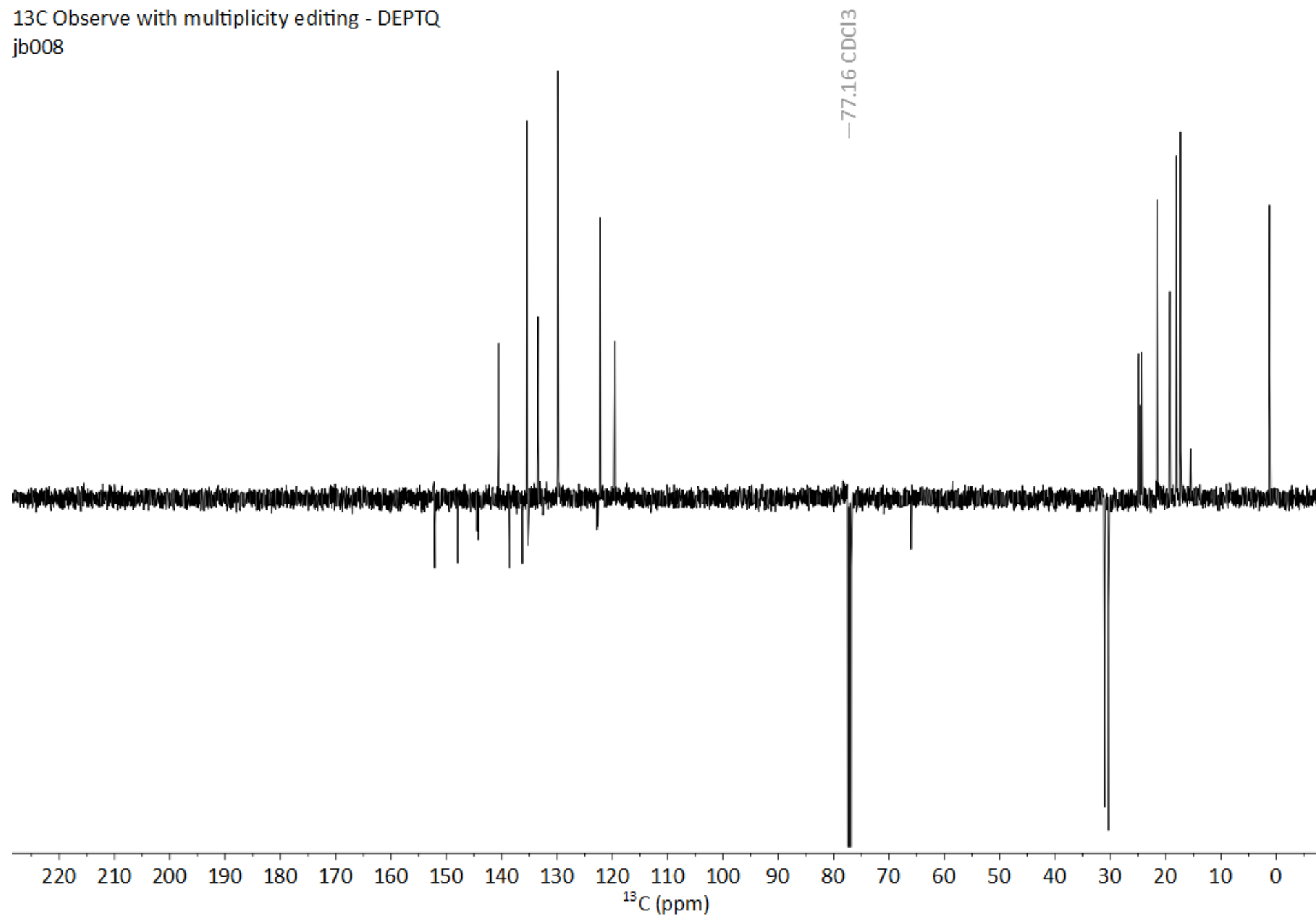
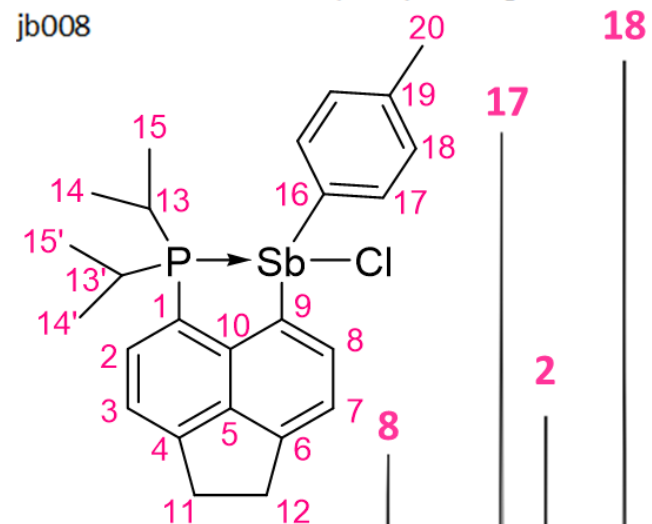


Figure S4: ¹³C DEPTQ NMR Spectrum of **1** acquired in CDCl₃ at 125.8 MHz at ambient conditions.

¹³C Observe with multiplicity editing - DEPTQ
jb008



¹³C Observe with multiplicity editing - DEPTQ
jb008

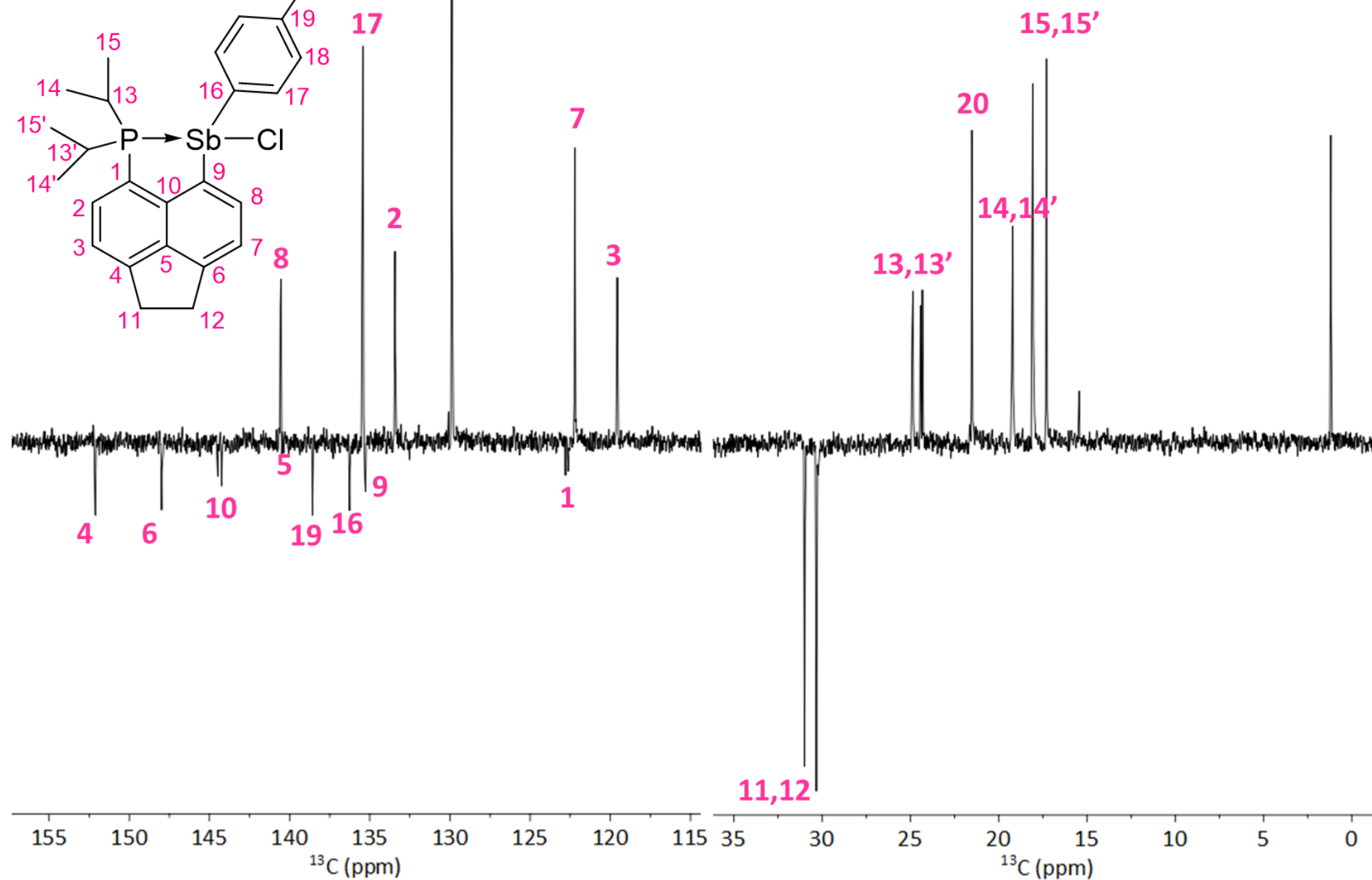


Figure S5: Expansions of the ¹³C DEPTQ NMR spectrum of **1** with assignments.

^{31}P Observe with ^1H decoupling
jb008

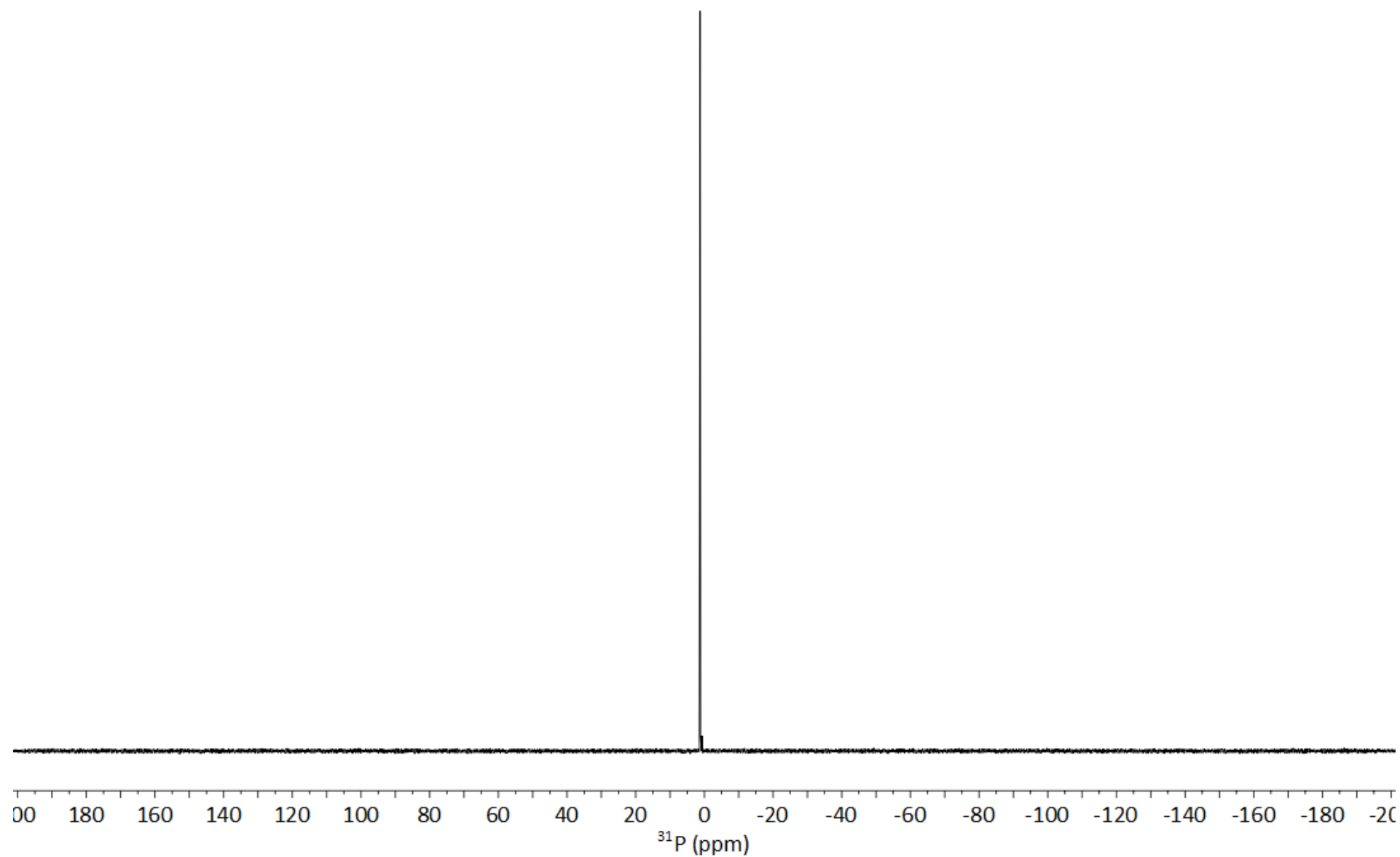


Figure S6: $^{31}\text{P}\{^1\text{H}\}$ DEPTQ NMR Spectrum of **1** acquired in CDCl_3 at 202.5 MHz at ambient conditions.

¹H Observe
JB_009

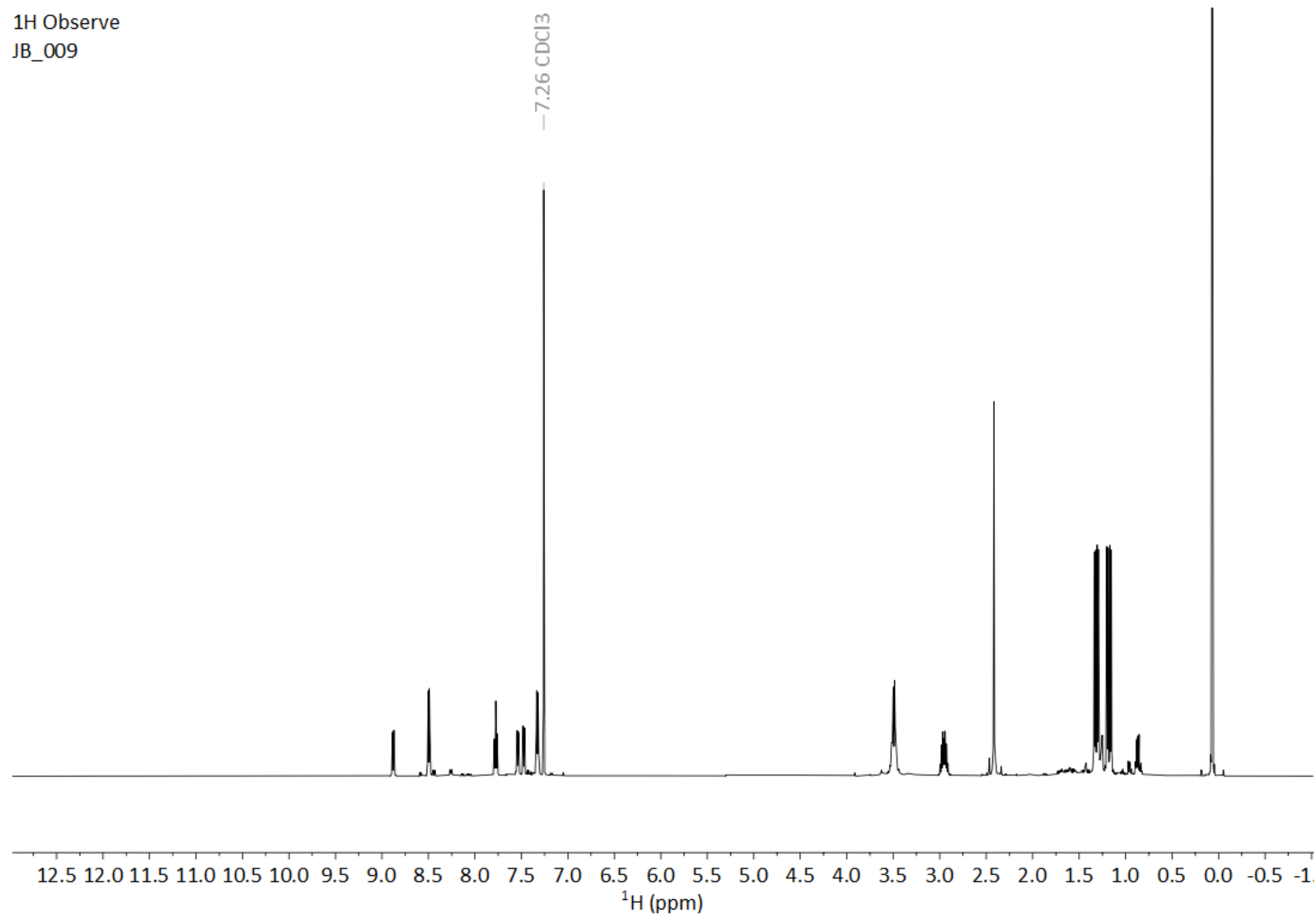


Figure S7: ¹H NMR spectrum of **2**, acquired in CDCl₃ at 500.1 MHz at ambient conditions.

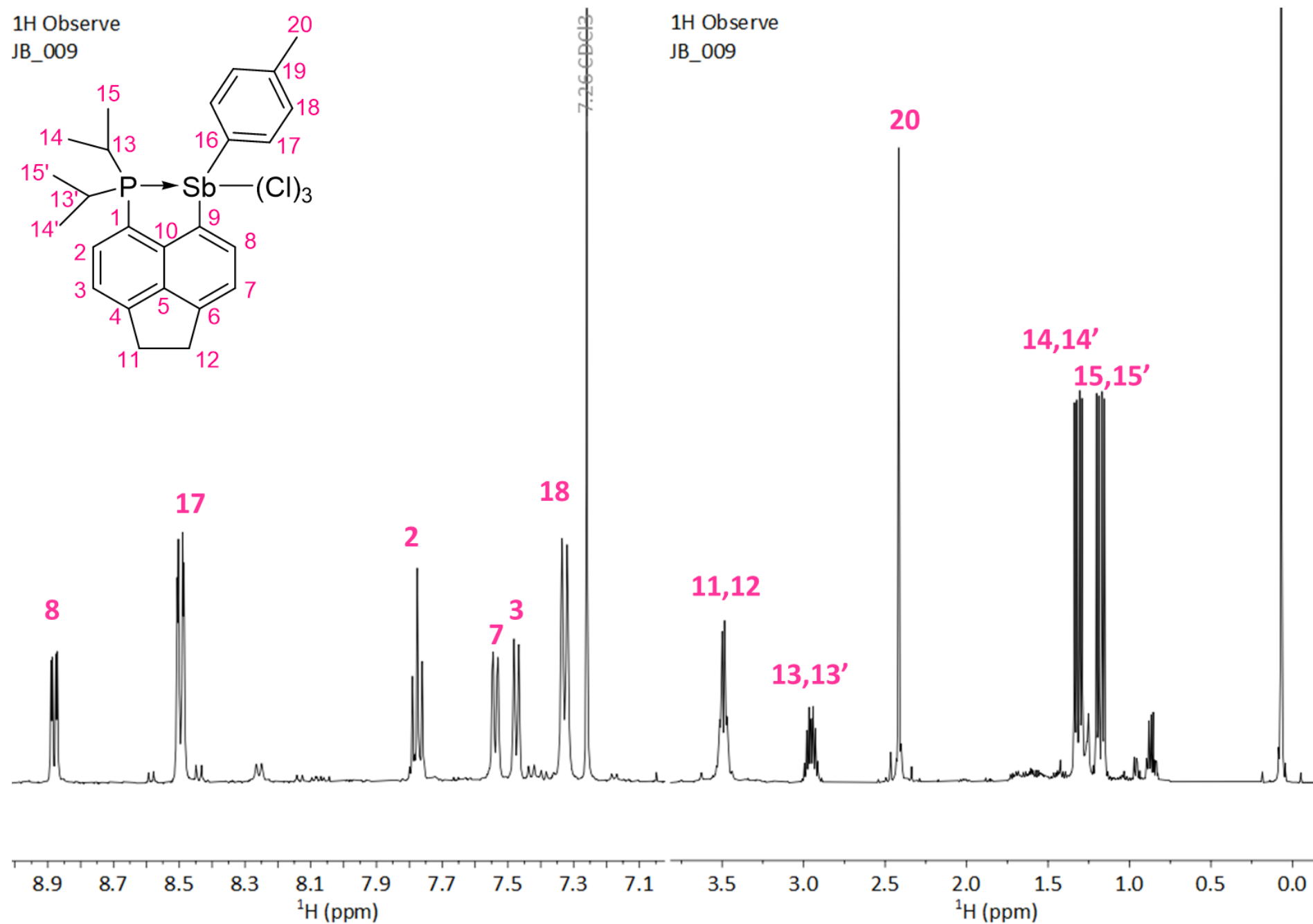


Figure S8: Expansions of the ¹H NMR spectrum of **2** with assignments.

¹³C Observe with multiplicity editing - DEPTQ
JB_009

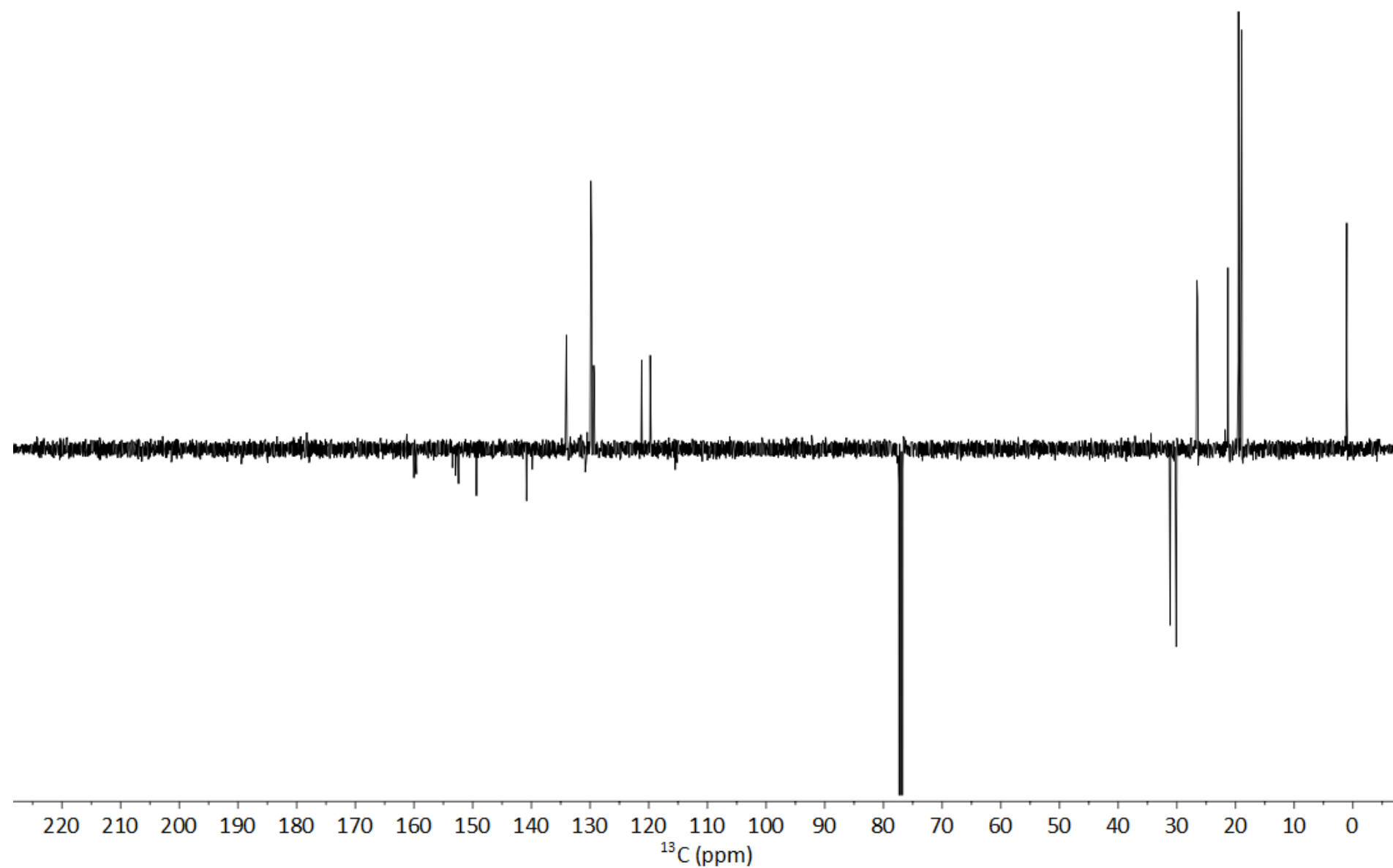


Figure S9: ¹³C DEPTQ NMR spectrum of **2**, acquired in CDCl₃ at 125.8 MHz at ambient conditions.

¹³C Observe with multiplicity editing - DEPTQ
JB_009

¹³C Observe with multiplicity editing - DEPTQ
JB_009

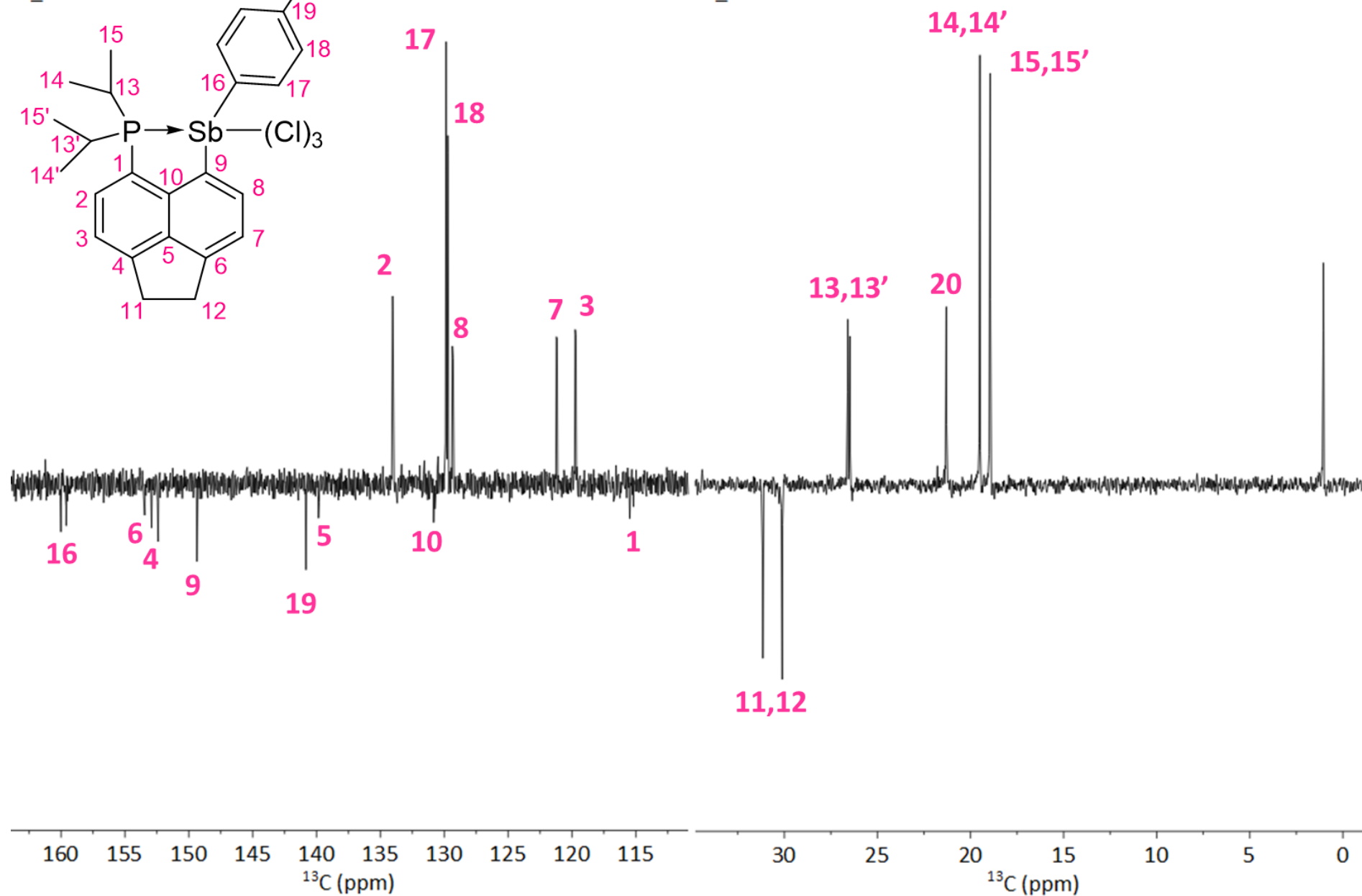


Figure S10: Expansions of the ¹³C DEPTQ NMR spectrum of **2** with assignments.

^{31}P Observe with ^1H decoupling
JB_009

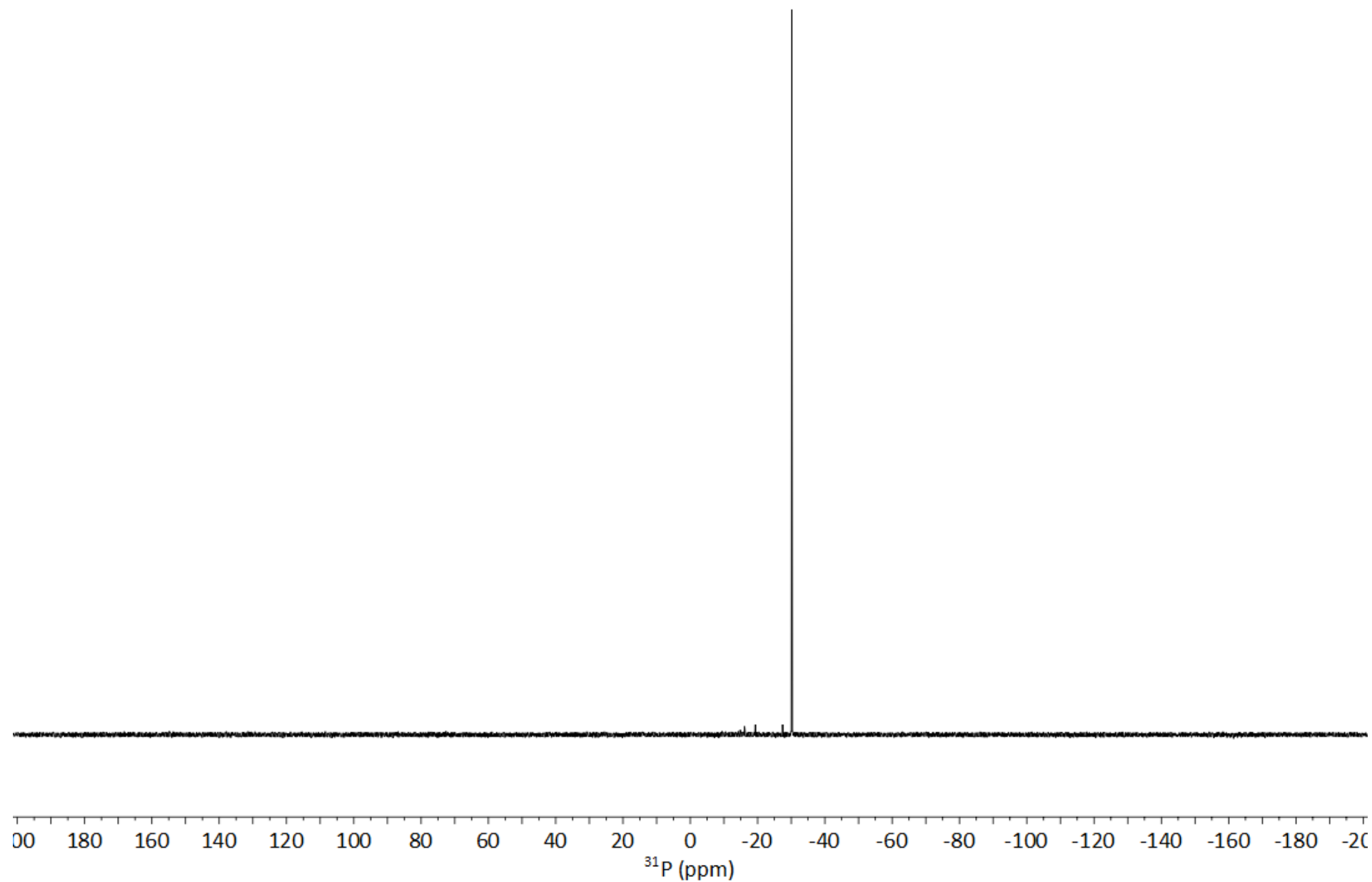


Figure S11: $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum of **2**, acquired in CDCl_3 at 202.5 MHz at ambient conditions.

ASAP (SOLID)

STAWOO244-PJ-HASP #13-39 RT: 0.34-1.05 AV: 27 NL: 1.76E8

T: FTMS + p APCI corona Full ms [100.00-800.00]

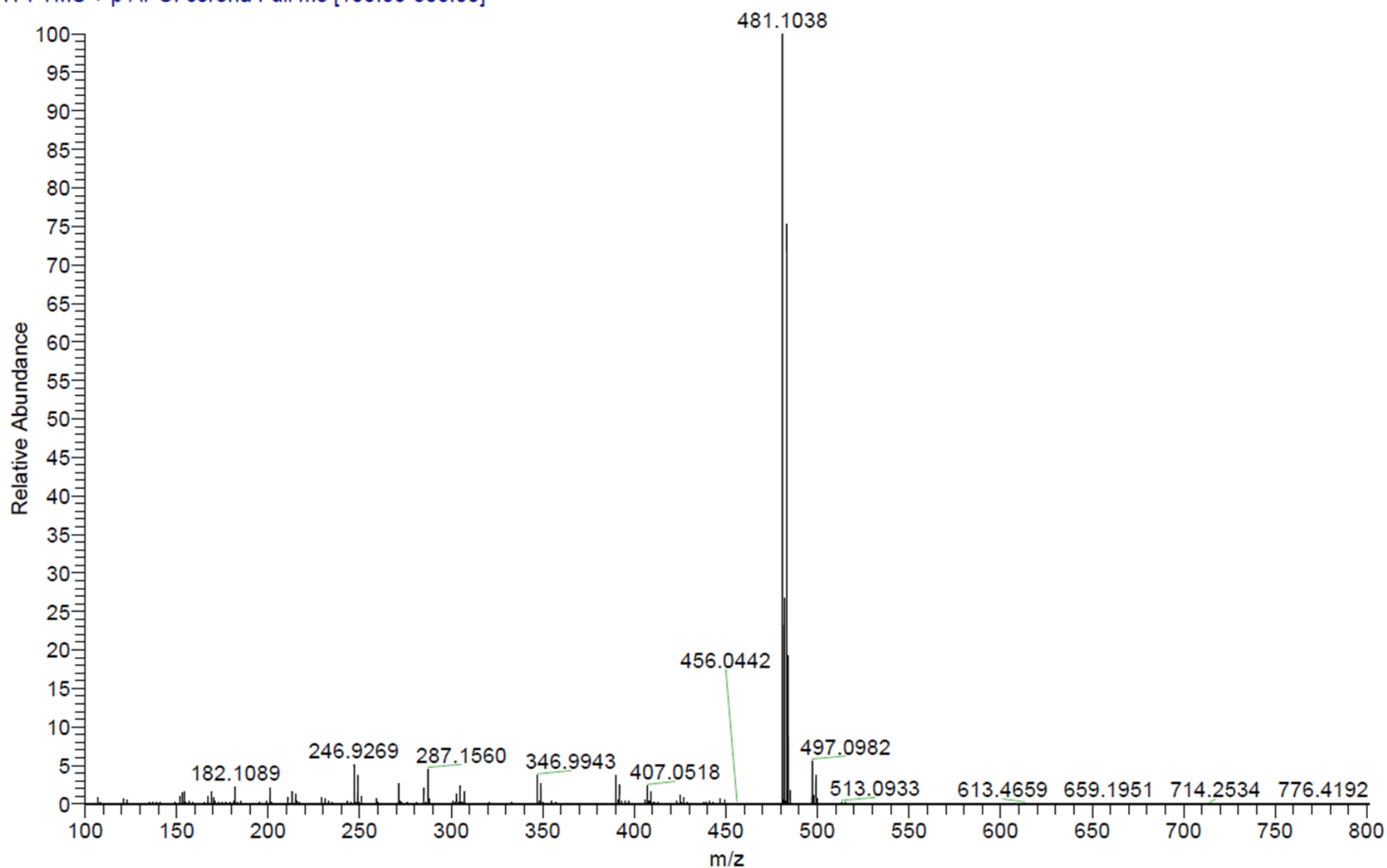
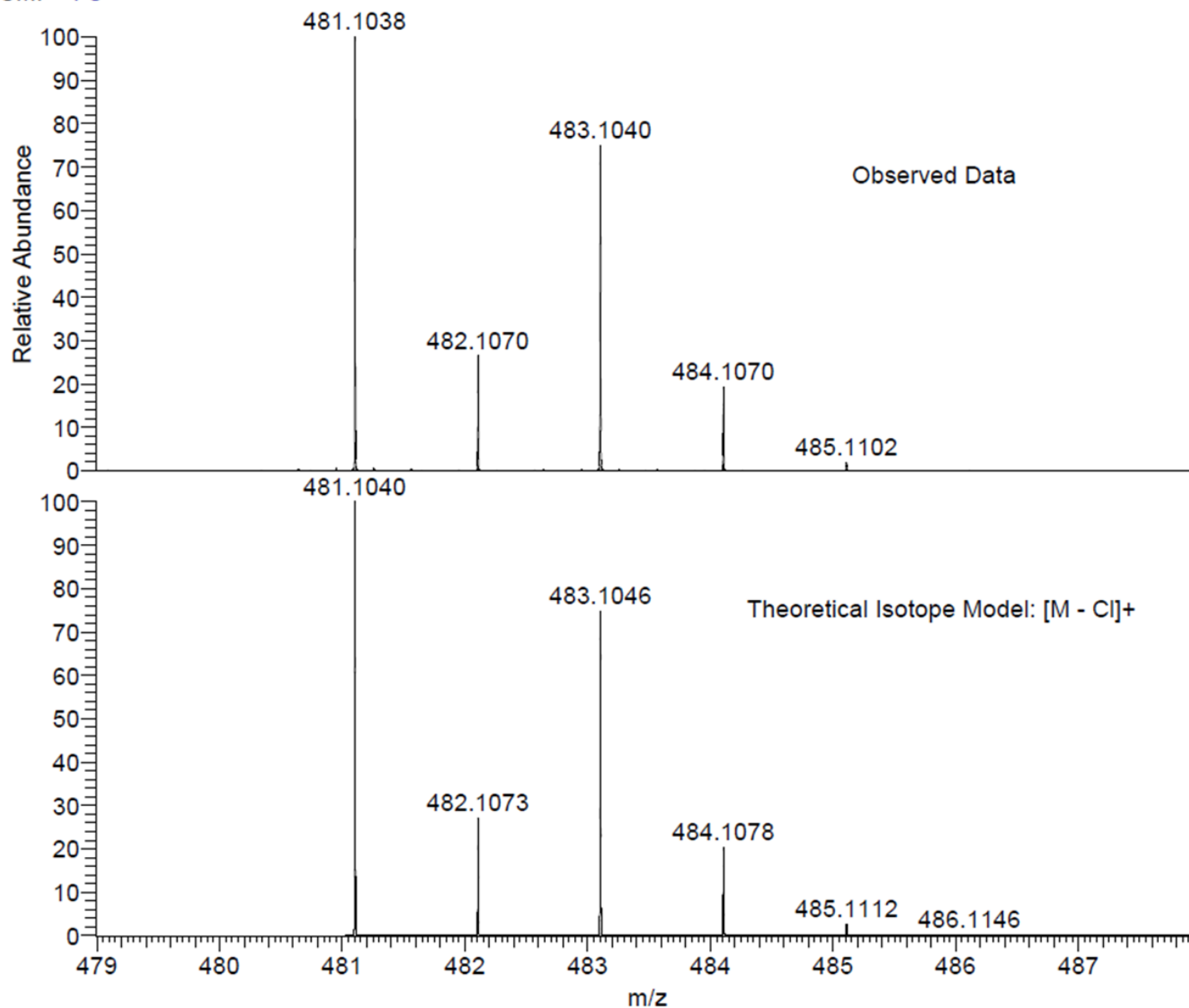


Figure S12: Full range HRMS of 1.

ASAP(SOLID)

SM: 7G

NL:
1.44E8
STAWOO244-PJ-HASP#13-39
RT: 0.34-1.05 AV: 27 T:
FTMS + p APCI corona Full ms
[100.00-800.00]



NL:
1.02E4
C₂₅ H₂₉ PSb:
C₂₅ H₂₉ P₁ Sb₁
p (gss, s /p:40) Chrg 1
R: 100000 Res .Pwr . @FWHM

Figure S13: Expansion of the molecular ion peaks in the HRMS of 1.

ASAP (SOLID)

STAWOO242-PJ-HASP #67-108 RT: 1.83-2.95 AV: 42 NL: 4.01E7

T: FTMS + p APCI corona Full ms [100.00-800.00]

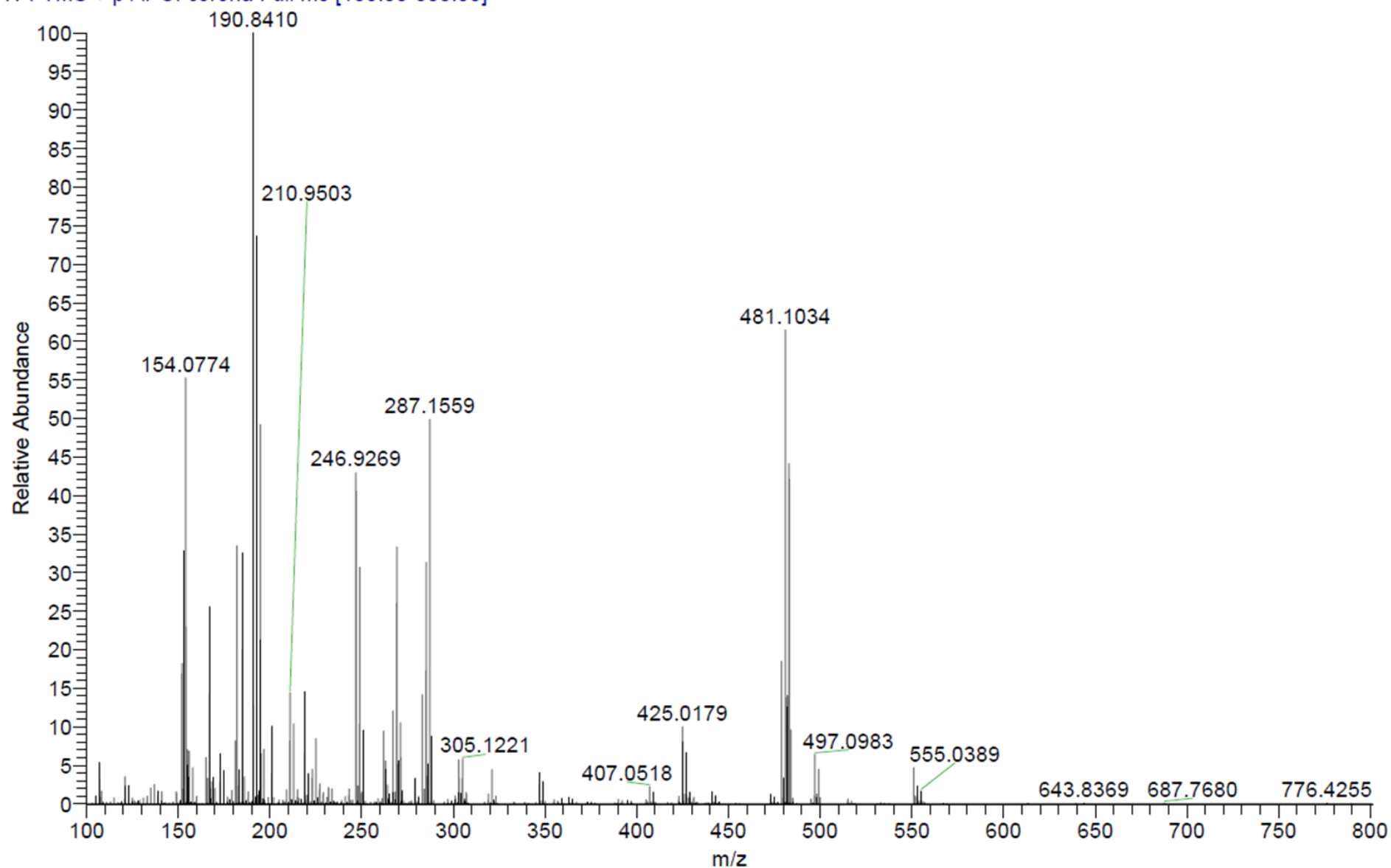
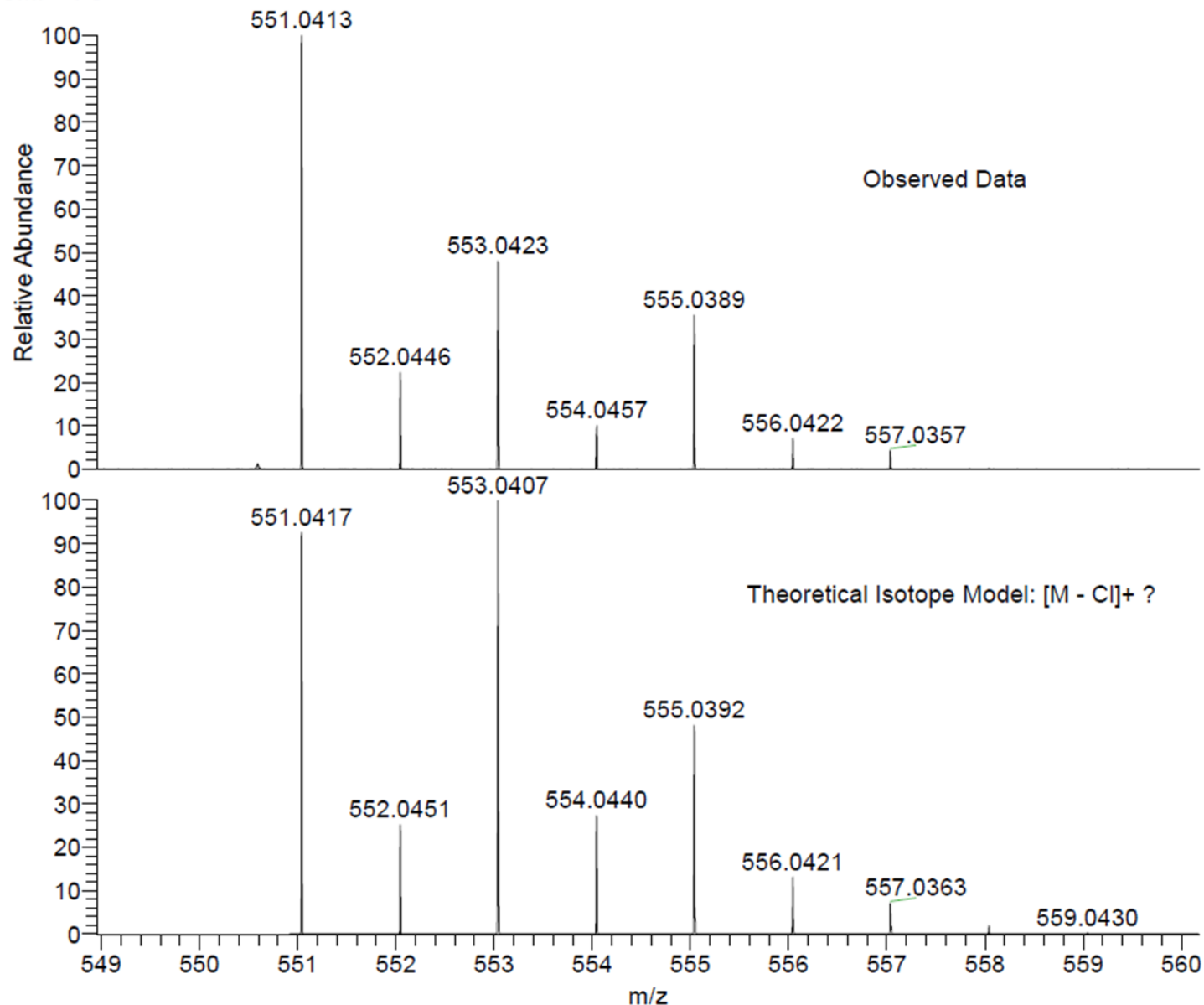


Figure S14: Full range HRMS of **2**.

ASAP(SOLID)

SM: 7G

NL:
1.55E6
STAWOO242-PJ-HASP#67-
108 RT: 1.83-2.95 AV: 42 T:
FTMS + p APCI corona Full ms
[100.00-800.00]



NL:
6.34E3
C₂₅ H₂₉ PSbCl₂:
C₂₅ H₂₉ P₁ Sb₁ Cl₂
p (gss, s /p:40) Chrg 1
R: 100000 Res .Pwr . @FWHM

Figure S15: Expansion of the molecular ion peaks in the HRMS of **2**.

Table S1. Selected crystallographic data.

	1	2
Formula	C ₂₅ H ₂₉ ClPSb	C ₂₅ H ₂₉ Cl ₃ PSb
F _w	517.68	588.59
Crystal description	Colourless prism	Colourless prism
Crystal size [mm ³]	0.100×0.100×0.100	0.100×0.030×0.030
Space group	<i>P</i> 2 ₁ / <i>n</i> (#14)	<i>P</i> 2 ₁ / <i>n</i> (#14)
<i>a</i> [Å]	7.8712(10)	12.5649(18)
<i>b</i> [Å]	18.334(2)	12.1953(17)
<i>c</i> [Å]	15.4030(17)	16.122(3)
α [°]	90	90
β [°]	92.001(4)	100.609(2)
γ [°]	90	90
vol [Å] ³	2221.5(4)	2428.1(7)
<i>Z</i>	4	4
ρ (calc) [g/cm ³]	1.548	1.610
μ [cm ⁻¹]	14.410	15.414
F(000)	1048	1184
2 θ _{max} [°]	50.7	50.6
2 θ range [°]	3.454 – 50.746	4.214 – 50.644
Index range	–9 ≤ <i>h</i> ≤ 9 –22 ≤ <i>k</i> ≤ 22 –17 ≤ <i>l</i> ≤ 18	–15 ≤ <i>h</i> ≤ 15 –14 ≤ <i>k</i> ≤ 14 –19 ≤ <i>l</i> ≤ 14
Reflections collected	23375	17206
Independent reflections (<i>R</i> _{int})	4042 (0.0292)	4421 (0.0352)
GoF on <i>F</i> ²	1.182	0.980
<i>R</i> ₁ [<i>I</i> > 2 σ (<i>I</i>)]	0.0237	0.0179
<i>wR</i> ₂ (all data)	0.0713	0.0462
Largest diff. peak/hole [e/Å ³]	0.75, –0.33	0.38, –0.31