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 ^{1}H 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 ^{1}H 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).

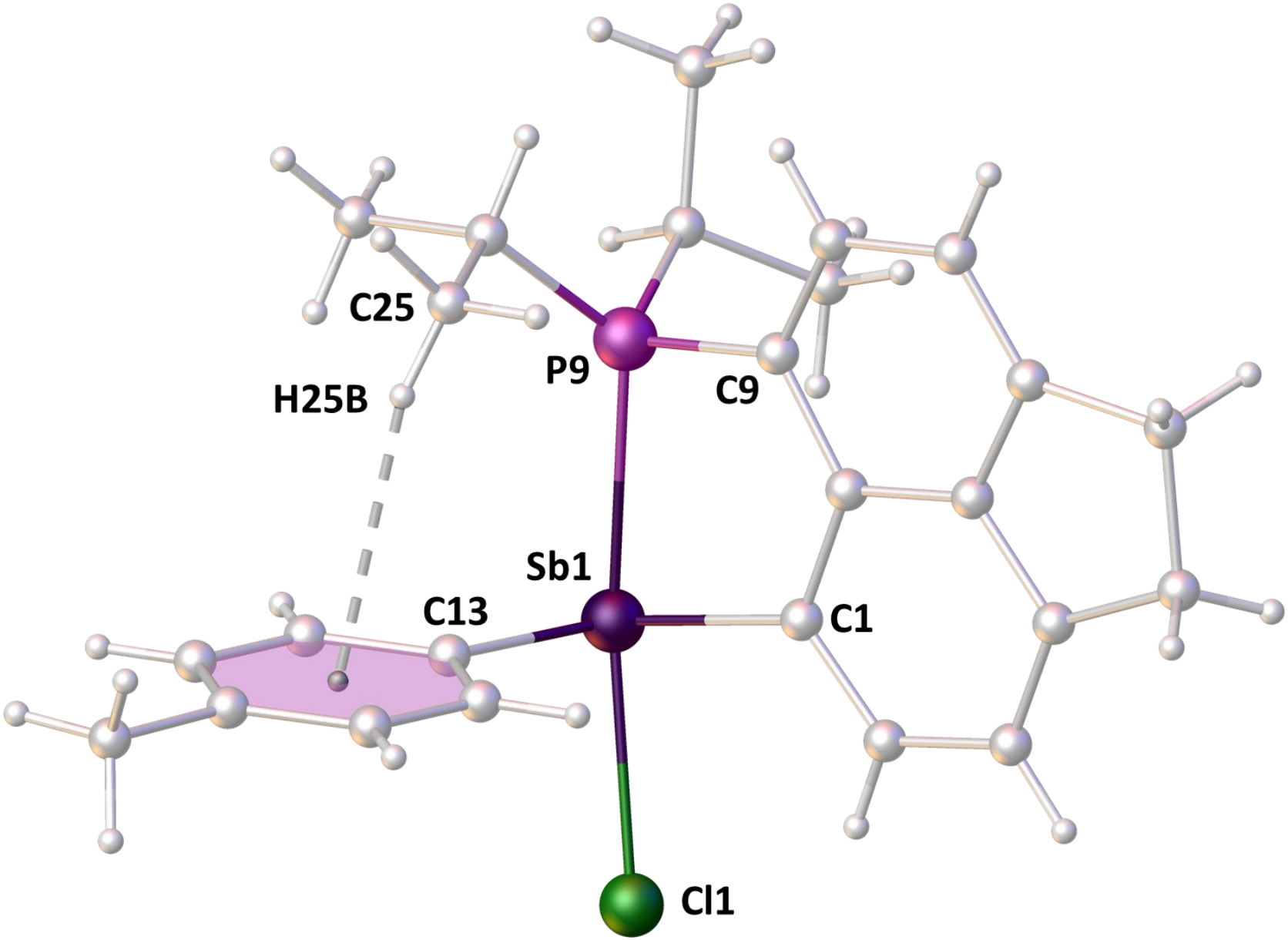

Figure S1: “ball and stick” model of the molecular structure of **1**, showing the CH···π interaction H25B to the centroid of the C13−C18 tolyl ring.

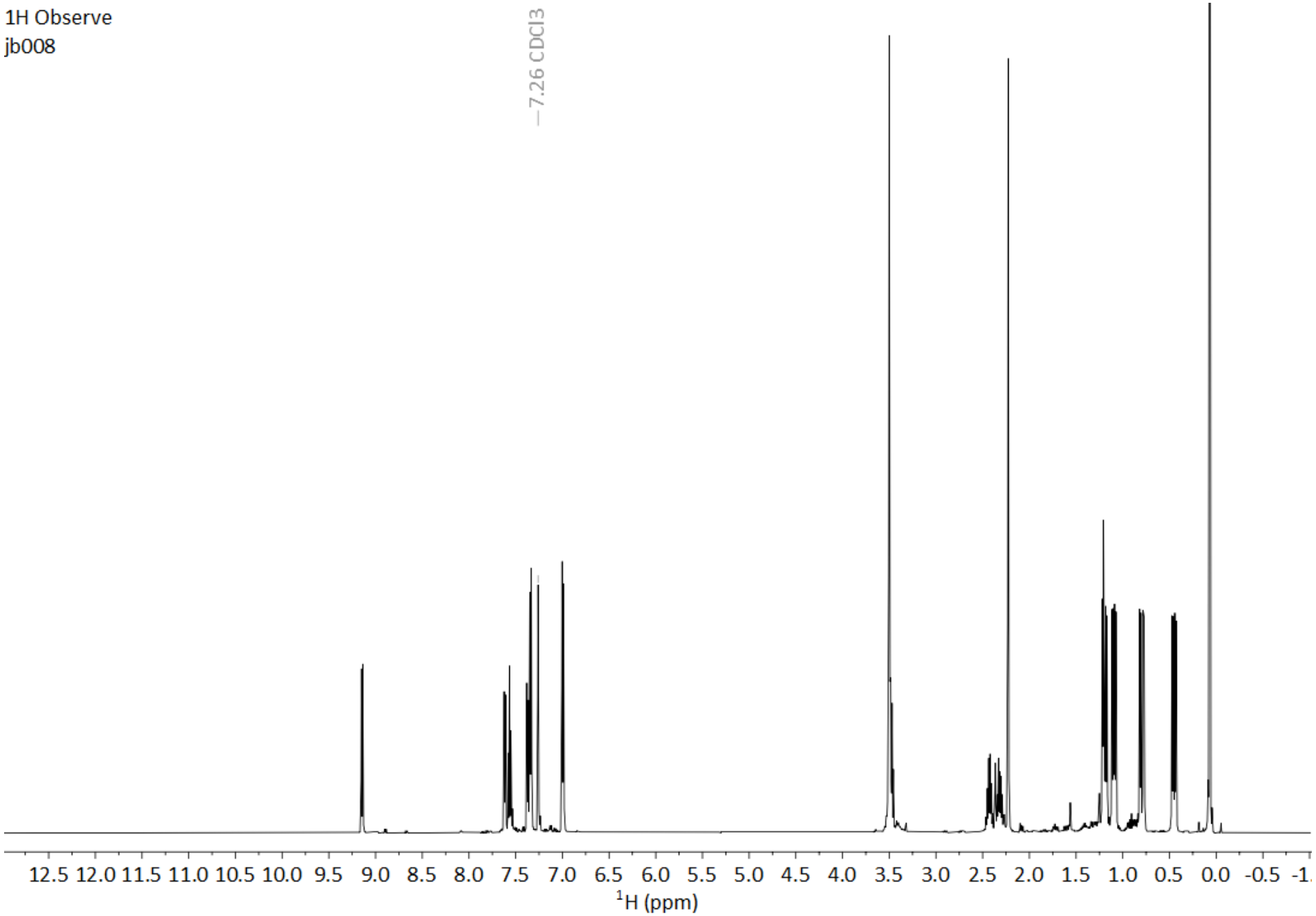

Figure S2: ^{1}H NMR spectrum of **1**, acquired in $CDCl_3$ at 500.1 MHz at ambient conditions.

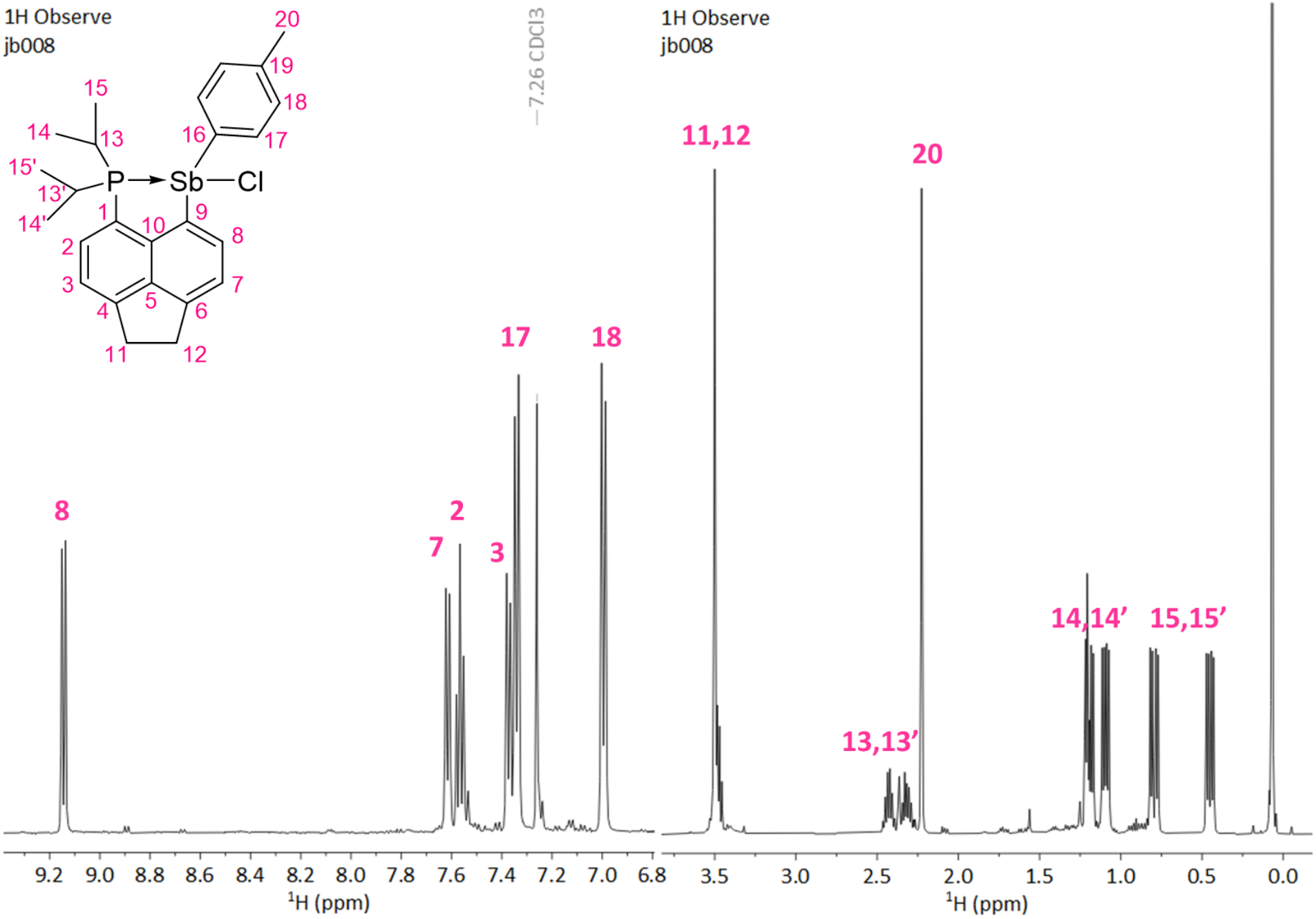

Figure S3: Expansions of the ^{1}H NMR spectrum of **1** with assignments.

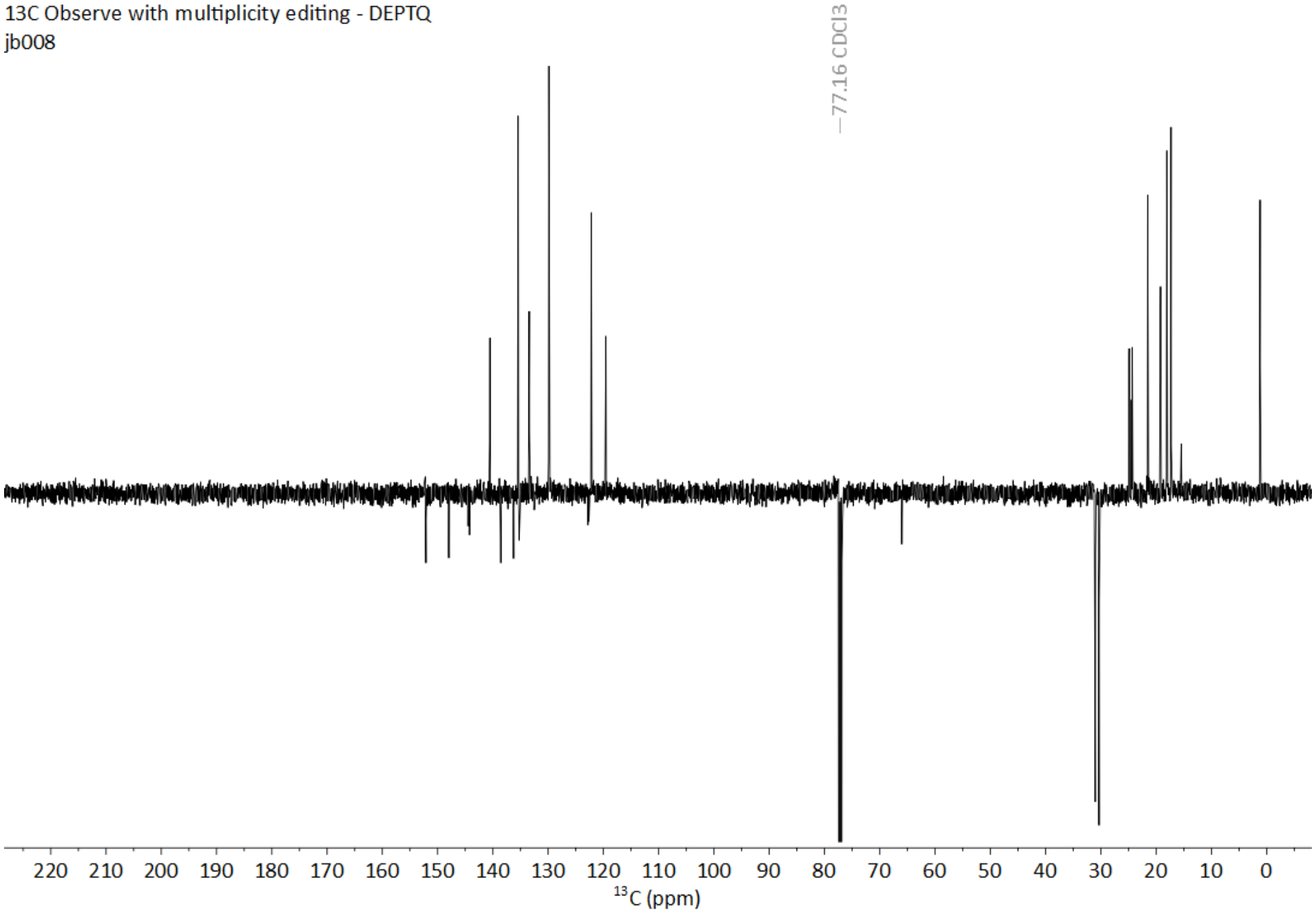

Figure S4: ^{13}C DEPTQ NMR Spectrum of **1** acquired in $CDCl_3$ at 125.8 MHz at ambient conditions.

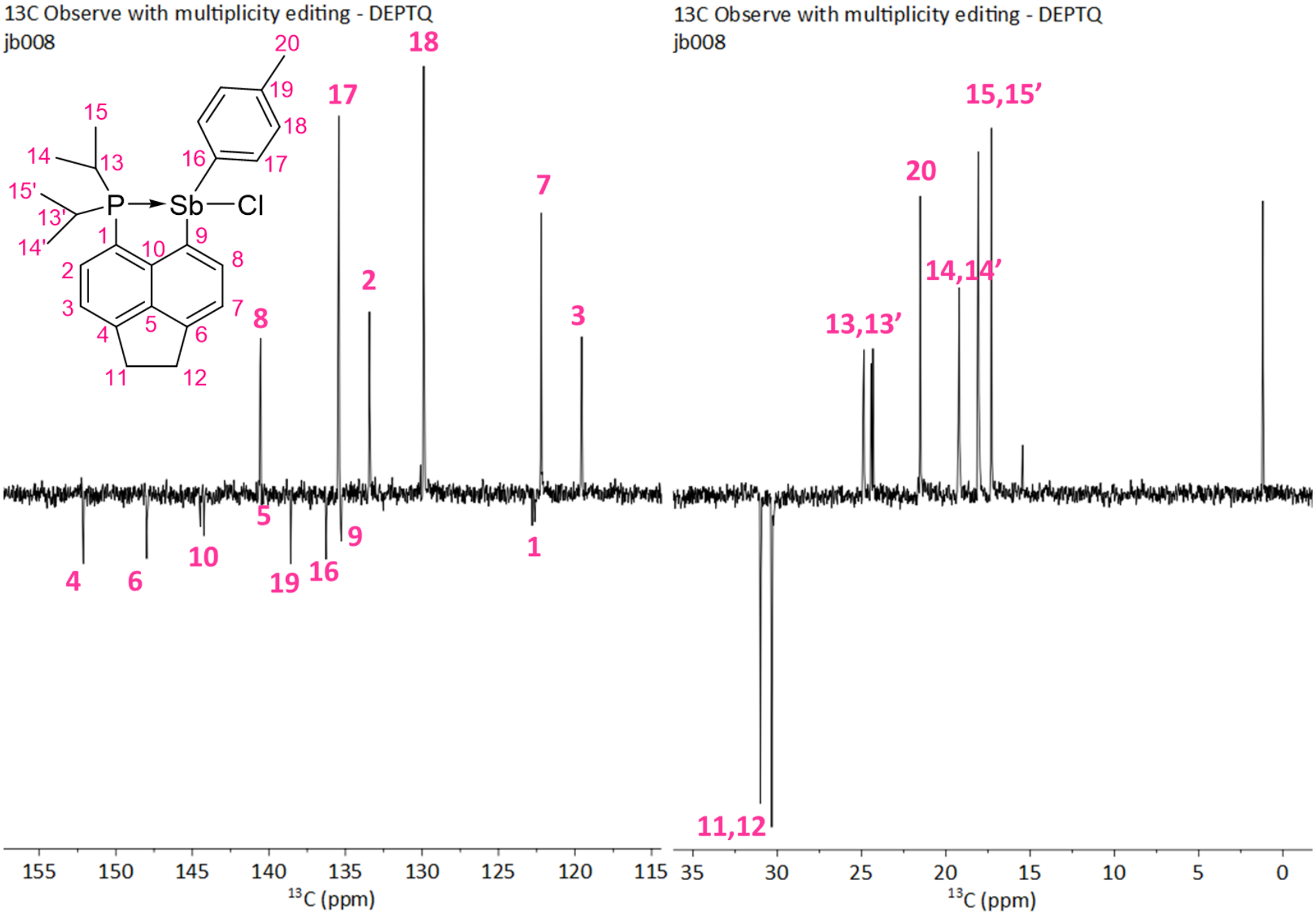

Figure S5: Expansions of the ^{13}C DEPTQ NMR spectrum of **1** with assignments.

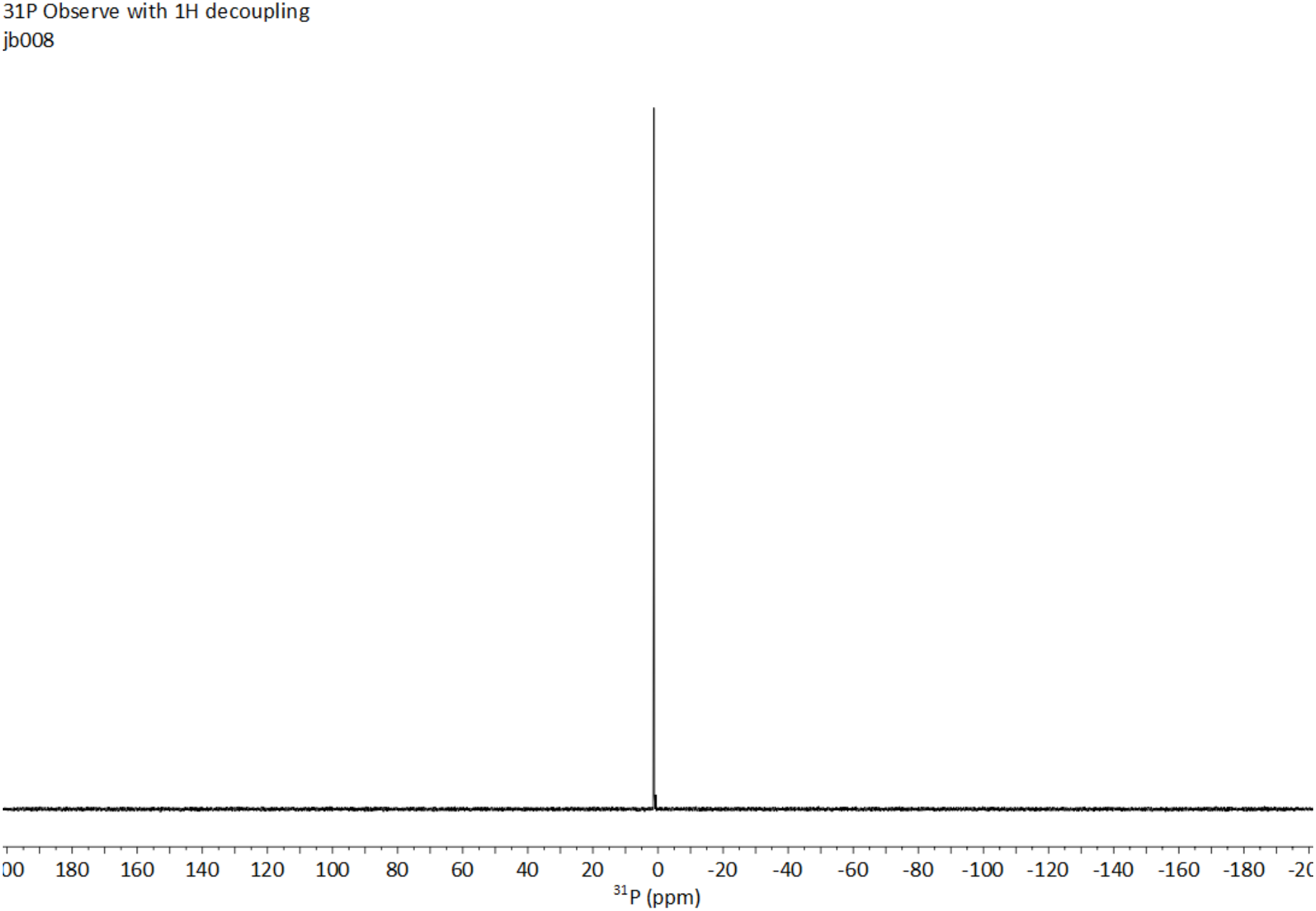

Figure S6: $^{31}P\{^{1}H\}$ DEPTQ NMR Spectrum of **1** acquired in $CDCl_3$ at 202.5 MHz at ambient conditions.

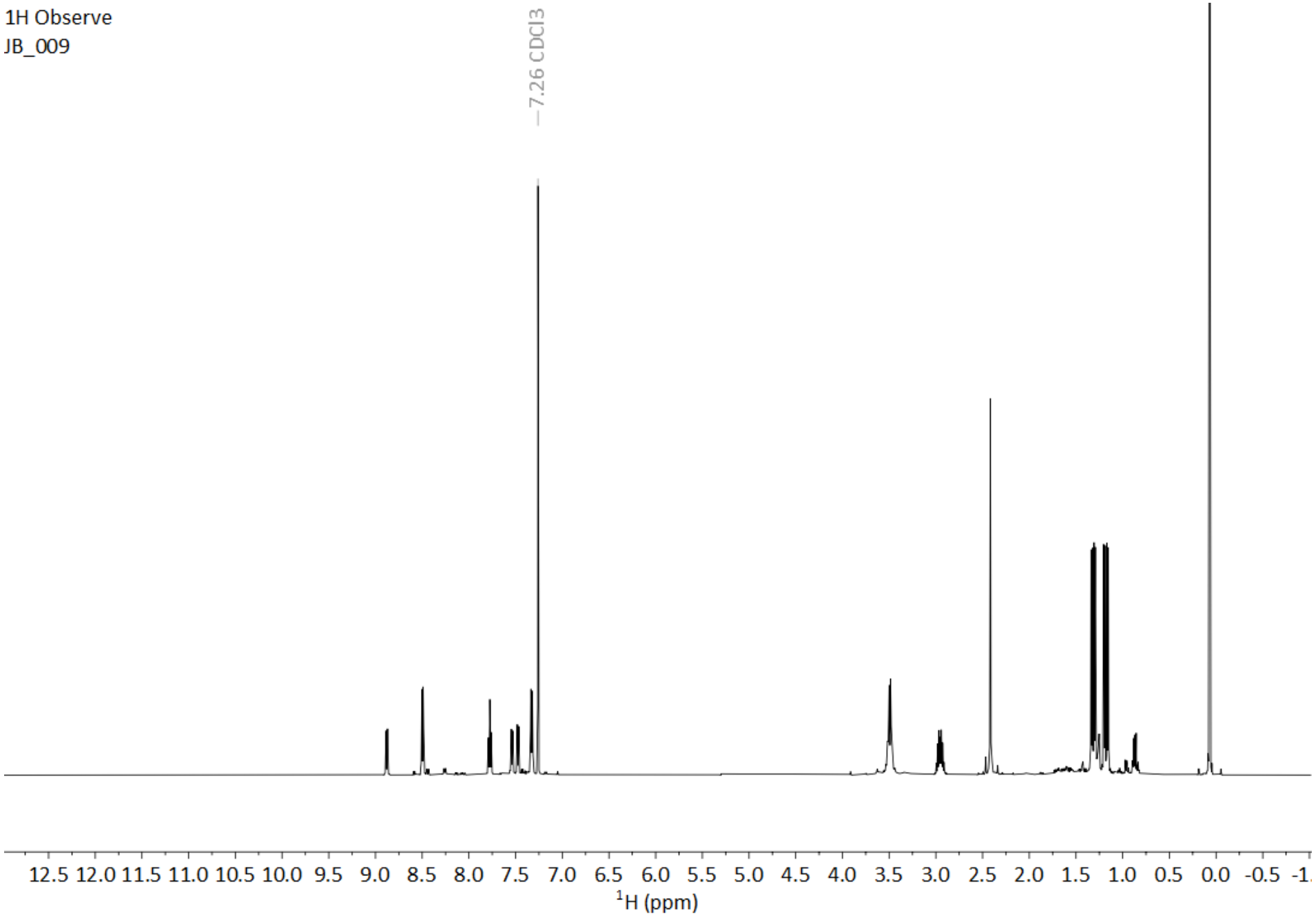

Figure S7: ^{1}H NMR spectrum of **2**, acquired in $CDCl_3$ at 500.1 MHz at ambient conditions.

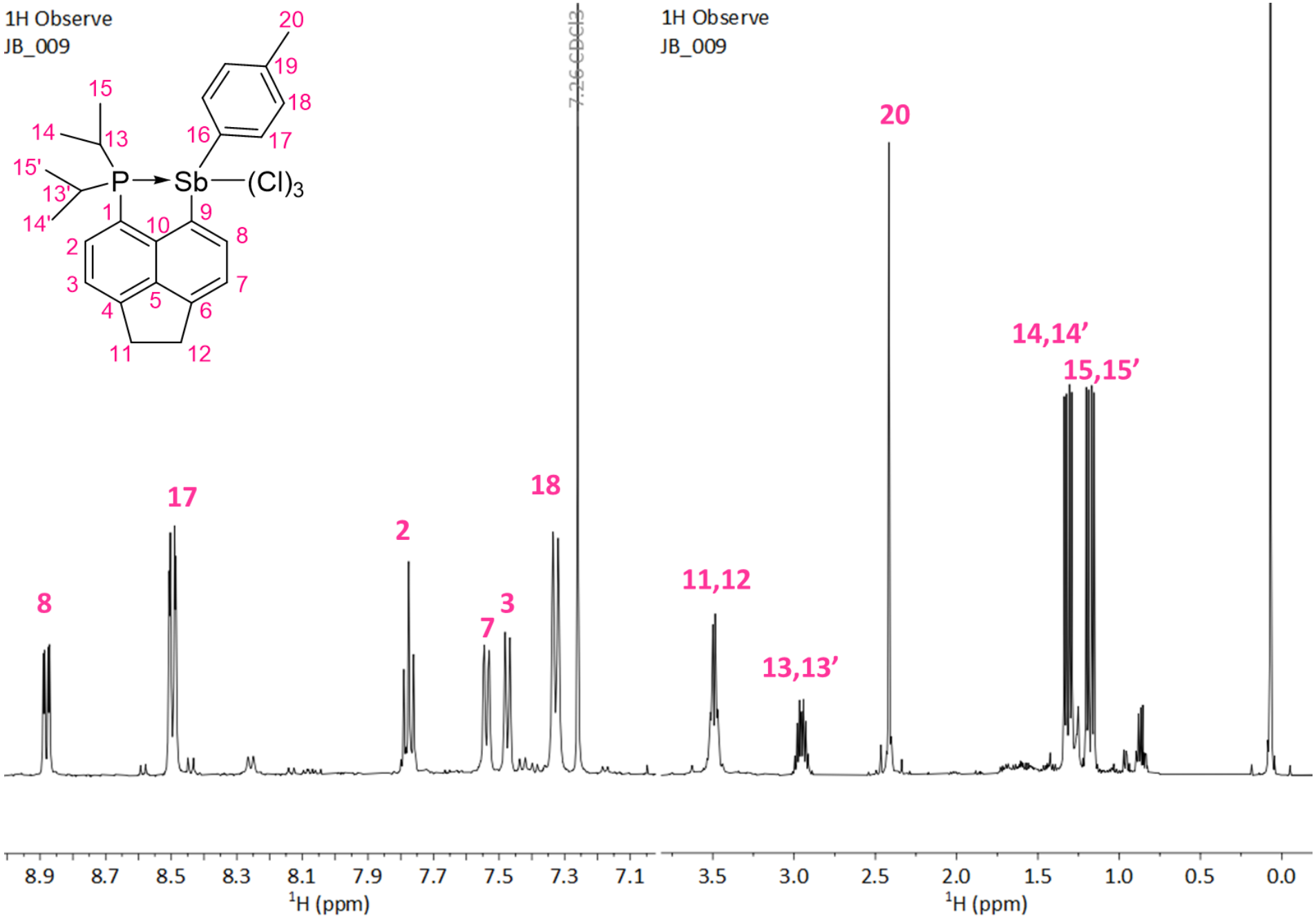

Figure S8: Expansions of the ^{1}H NMR spectrum of **2** with assignments.

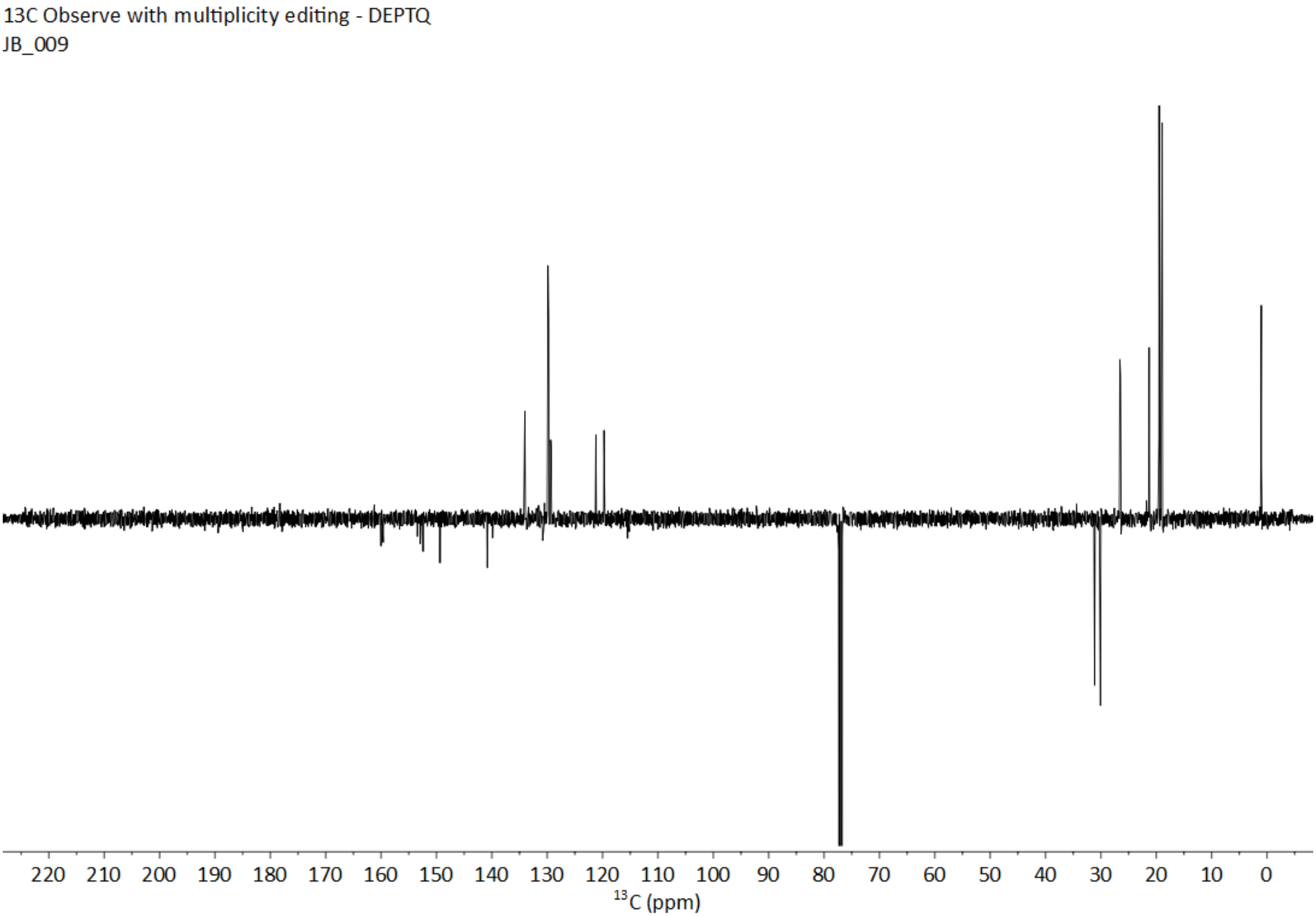

Figure S9: ^{13}C DEPTQ NMR spectrum of **2**, acquired in $CDCl_3$ at 125.8 MHz at ambient conditions.

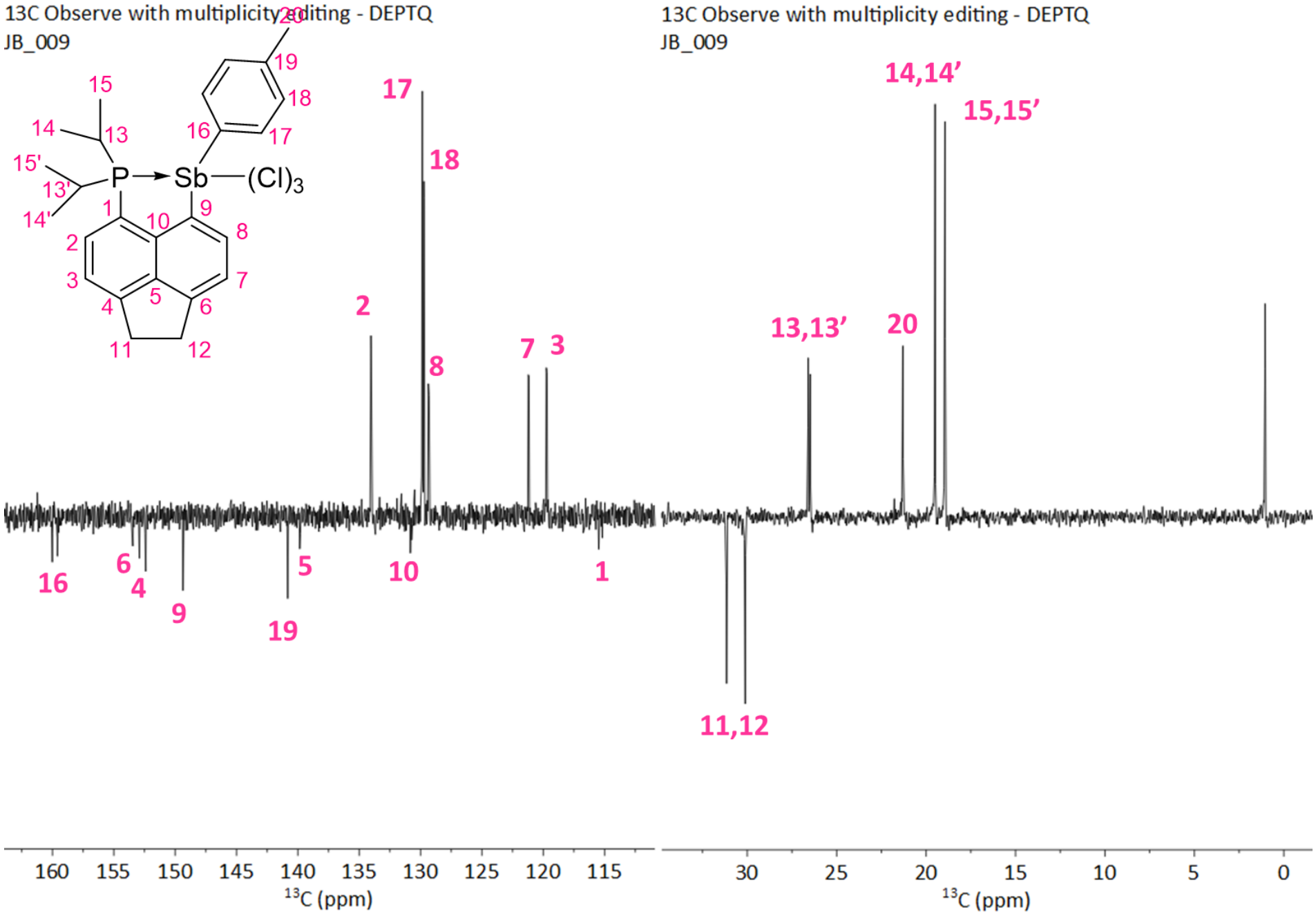

Figure S10: Expansions of the ^{13}C DEPTQ NMR spectrum of **2** with assignments.

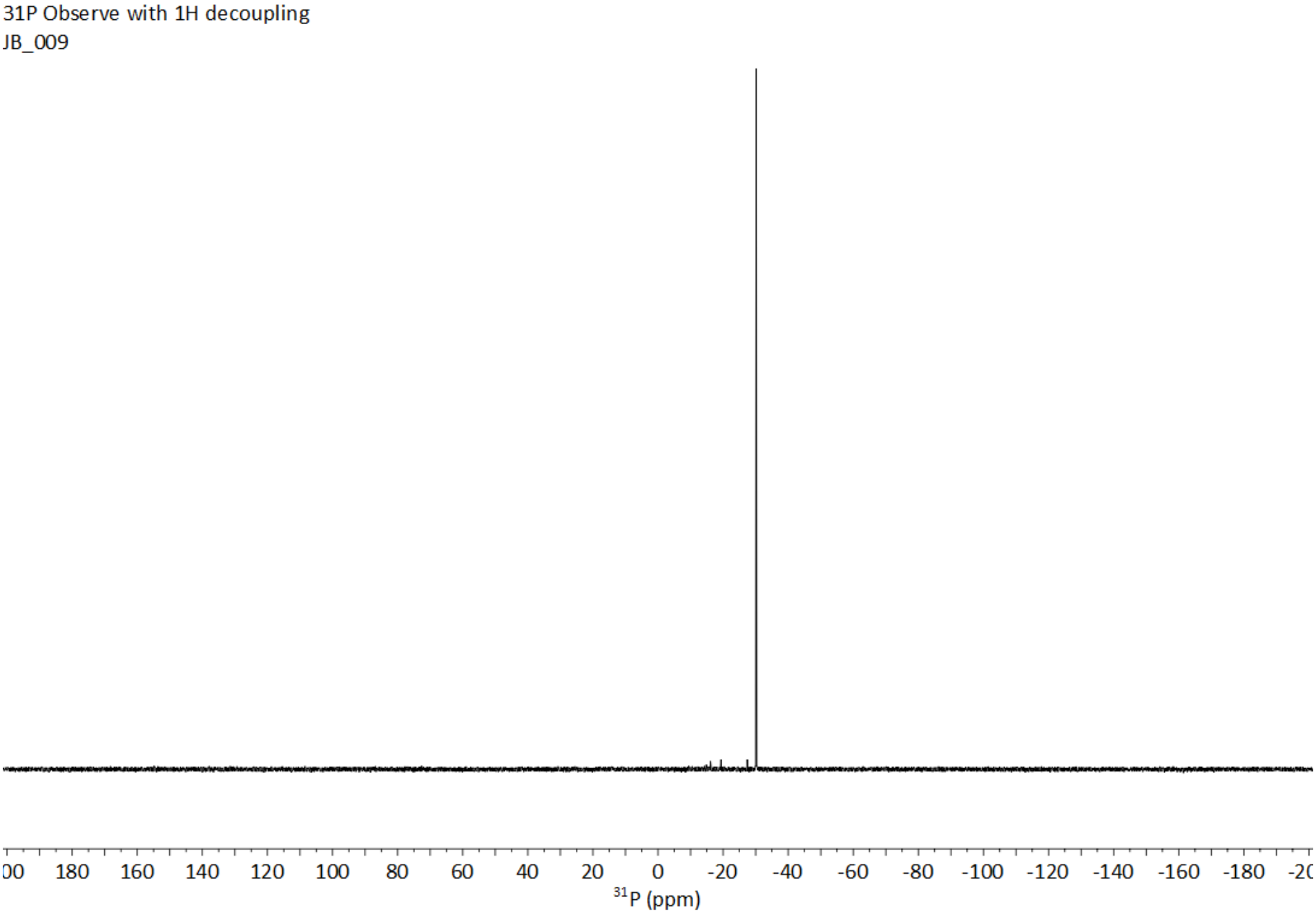

Figure S11: $^{31}P\{^{1}H\}$ NMR spectrum of **2**, acquired in $CDCl_3$ at 202.5 MHz at ambient conditions.

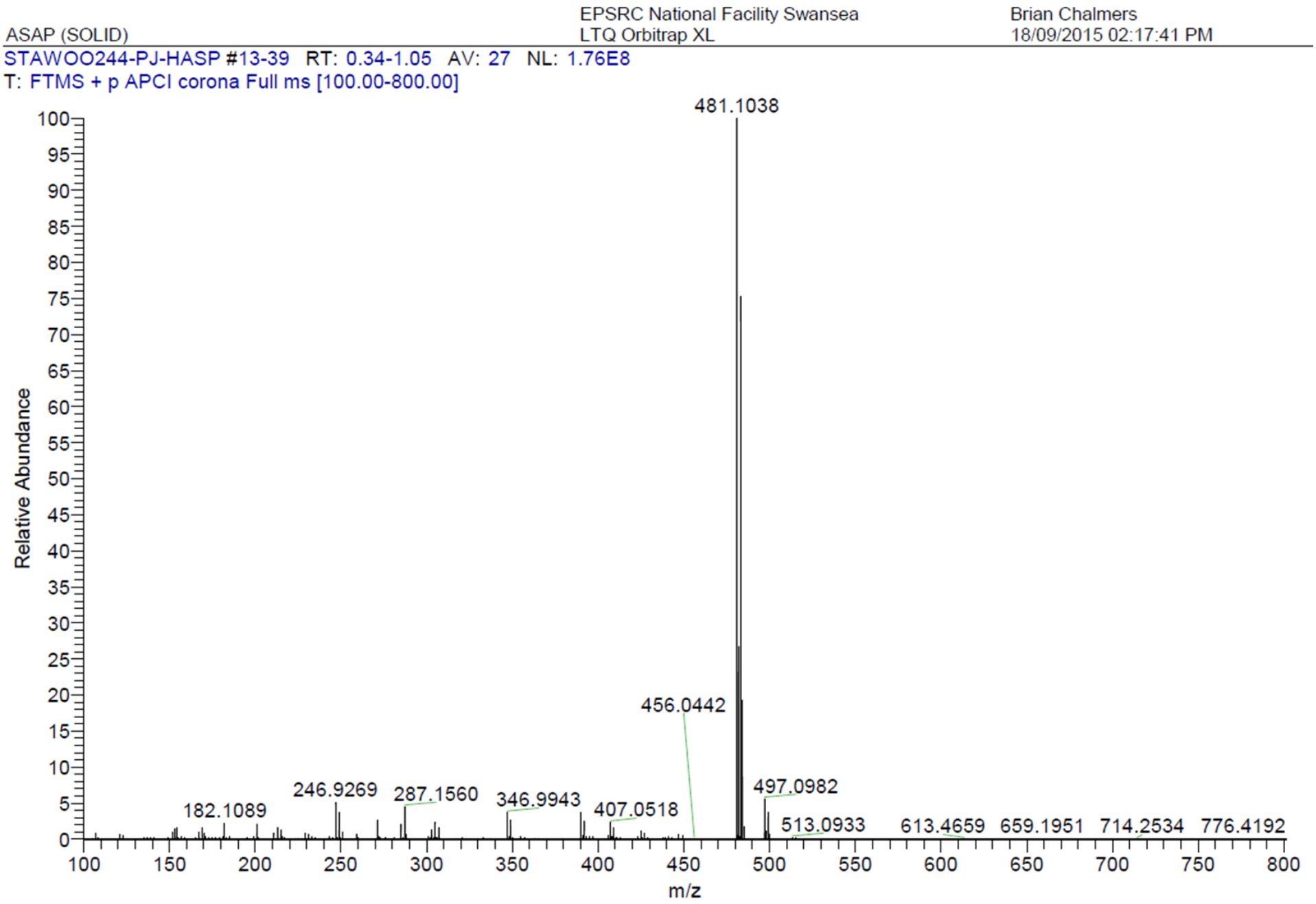

Figure S12: Full range HRMS of **1**.

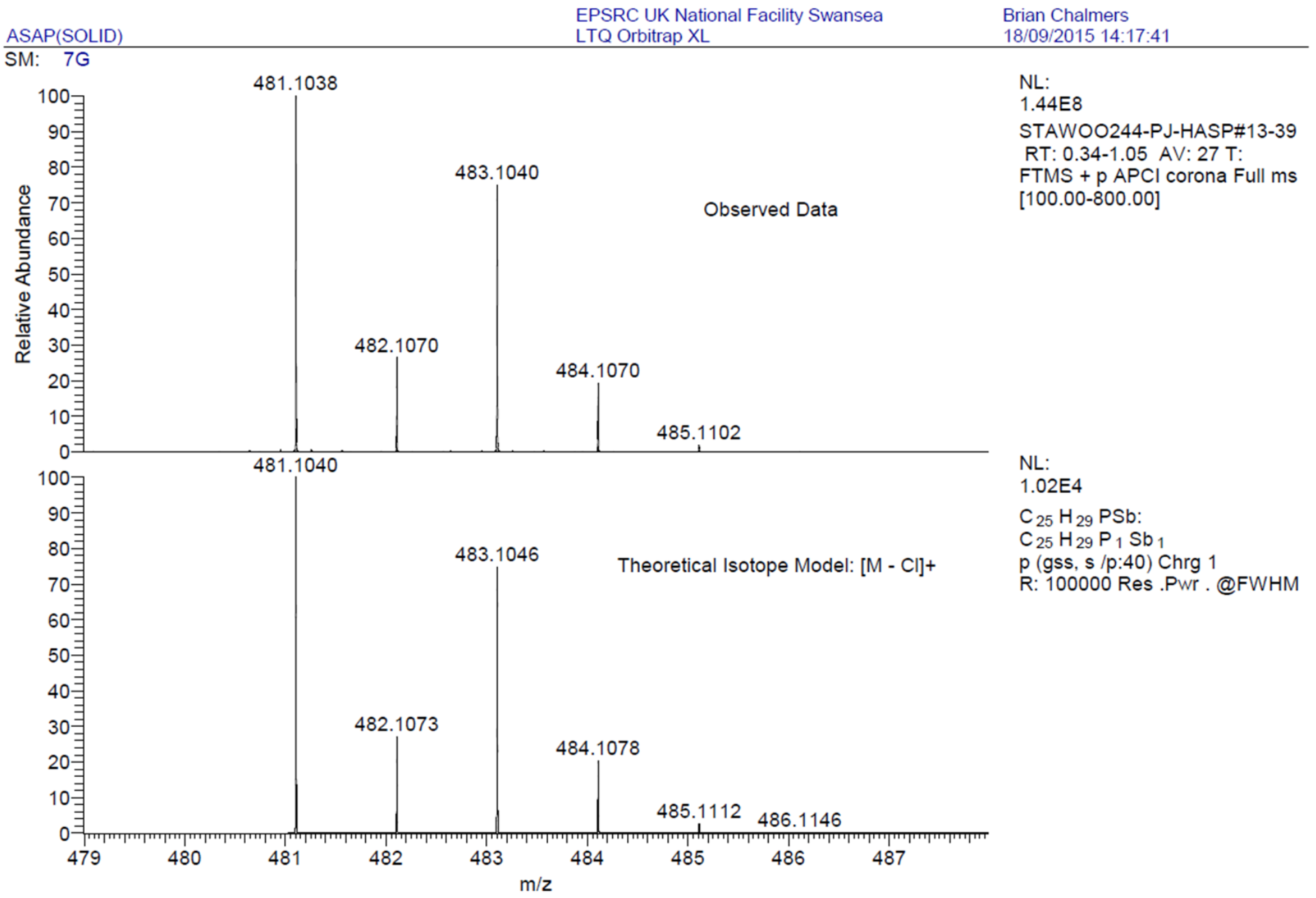

Figure S13: Expansion of the molecular ion peaks in the HRMS of **1**.

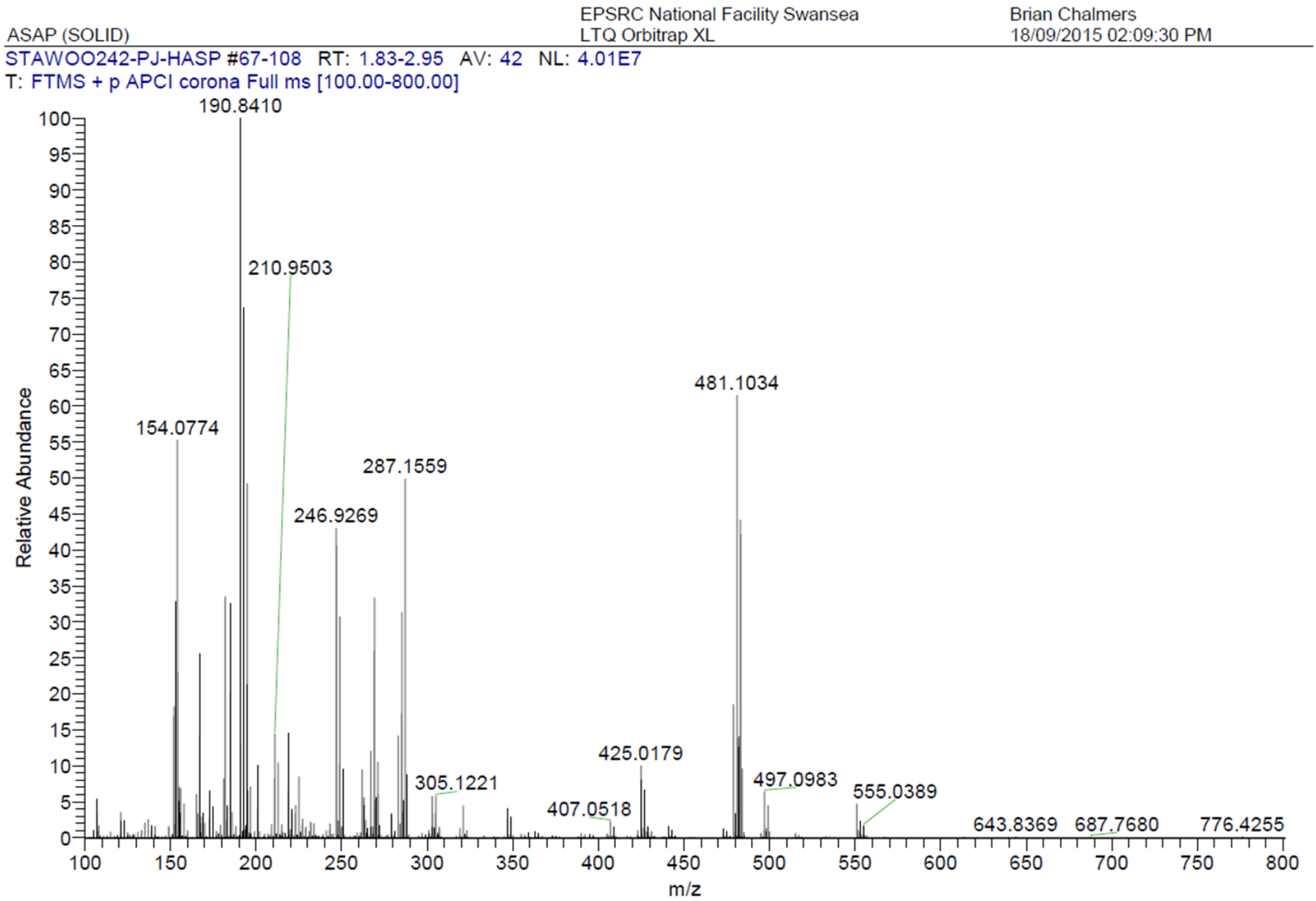

Figure S14: Full range HRMS of **2**.

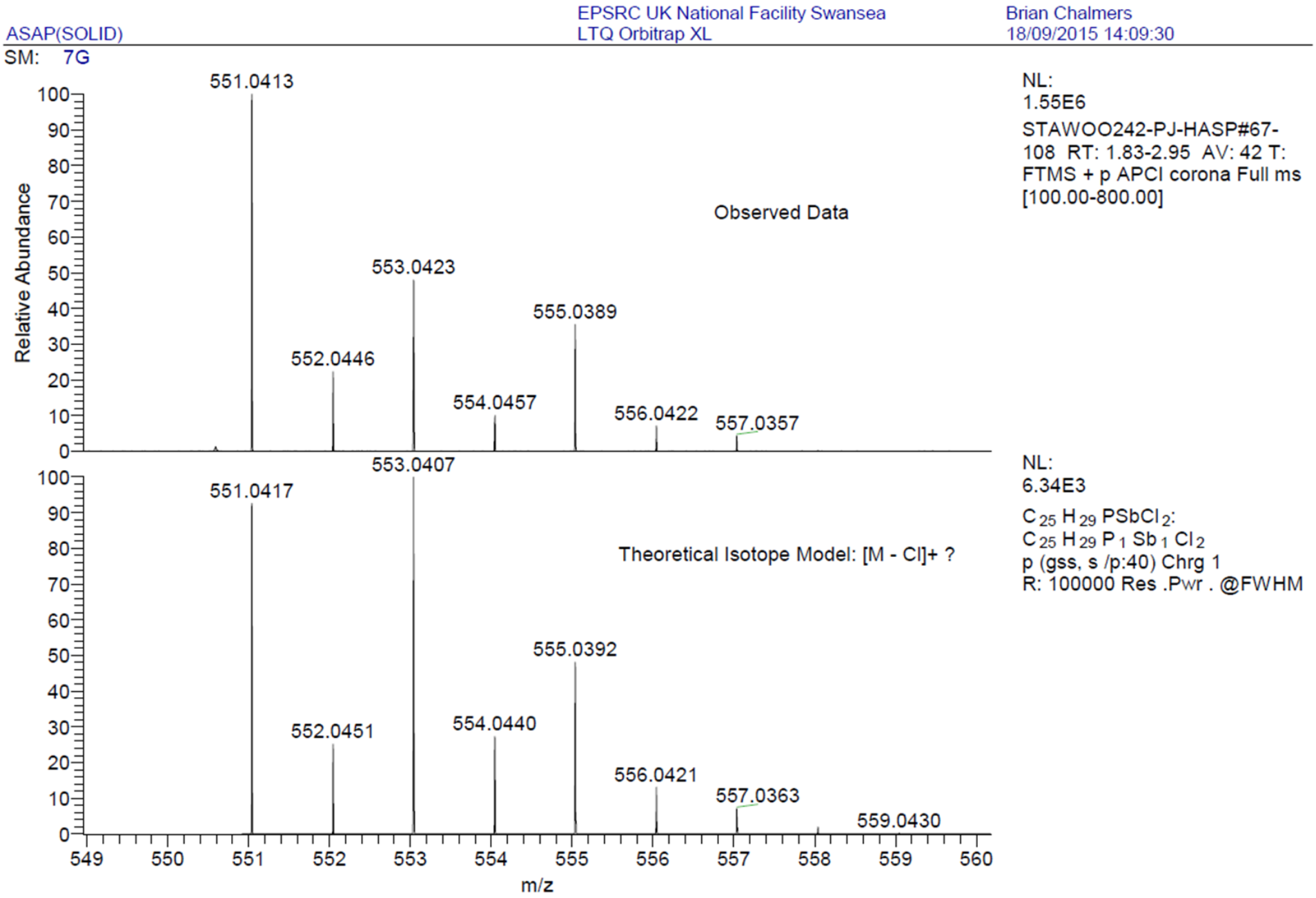

Figure S15: Expansion of the molecular ion peaks in the HRMS of **2.**

Table S1. Selected crystallographic data.

	1	**2**
Formula	$C_{25}H_{29}ClPSb$	$C_{25}H_{29}Cl_3PSb$
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	*P2₁/n* (#14)	*P2₁/n* (#14)
a [Å]	7.8712(10)	12.5649(18)
b [Å]	18.334(2)	12.1953(17)
c [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)
Z	4	4
ρ (calc) [g/cm^3]	1.548	1.610
μ [cm^{-1}]	14.410	15.414
F(000)	1048	1184
$2\theta_{max}$ [°]	50.7	50.6
2θ range [°]	3.454 – 50.746	4.214 – 50.644
Index range	−9 ≤ h ≤ 9 −22 ≤ k ≤ 22 −17 ≤ l ≤ 18	−15 ≤ h ≤ 15 −14 ≤ k ≤ 14 −19 ≤ l ≤ 14
Reflections collected	23375	17206
Independent reflections (R_{int})	4042 (0.0292)	4421 (0.0352)
GoF on F^2	1.182	0.980
R_1 [$I > 2\sigma(I)$]	0.0237	0.0179
wR_2 (all data)	0.0713	0.0462
Largest diff. peak/hole [$e/Å^3$]	0.75, −0.33	0.38, −0.31