Electronic Supplementary Information

Rhodium-catalyzed sequential B(3)−, B(4)−, and B(5)-trifunctionalization of *o*-carboranes with three different substituents

Biao Cheng, † Yu Chen, † Peng Zhou, and Zuowei Xie*

Department of Chemistry and State Key Laboratory of Synthetic Chemistry, The Chinese University of Hong Kong, Shatin, New Territories, Hong Kong, China

E-mail: zxie@cuhk.edu.hk

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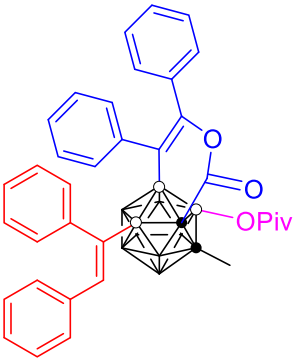
General Information.

All reactions were carried out in flame-dried glassware under an atmosphere of dry argon with the exclusion of air and moisture using standard Schlenk techniques or in a drybox. All organic solvents were dried by standard procedures prior to use. ^{1}H, ^{13}C, and ^{11}B NMR spectra were recorded on Bruker DPX 400/500 spectrometer at 400/500, 100/125, and 128/160 MHz, respectively. ^{1}H NMR chemical shifts were referenced to tetramethylsilane signal (0.00 ppm) or the residual solvent resonances of the deuterated solvents. ^{13}C NMR chemical shifts were referenced to the residual solvent resonances of the deuterated solvents. ^{11}B NMR chemical shifts were referenced to external $BF_3{\cdot}OEt_2$ (0.00 ppm). High Resolution Mass Spectra (HRMS) were obtained on a Thermo Q Exactive™ Focus Hybrid Quadrupole-Orbitrap™ Mass Spectrometer. GC-MS analyses were performed on Agilent GC-MS 6890N. Melting points were measured using a Nikon Polarizing Microscope ECLIPSE 50i POL equipped with an INTEC HCS302 heating stage without calibration. Carboranyl carboxylic acid (**1**), 1-COOH-2-Me-4-[(Ph)C=CH(Ph)]-*o*-$C_2B_{10}H_9$, and 1,4-[COOC(Ph)=C(Ph)]-2-Me-*o*-$C_2B_{10}H_9$ were prepared according to the literature method.[1,2] All other chemicals were purchased from either Aldrich or Acros Chemical Co. and used as received unless otherwise specified.

Experimental Section.

Preparation of B(3,4,5)-trifunctionalized carboranes (4). A representative procedure. A $PhCF_3$ (10 mL) suspension of 1-COOH-2-CH_3-*o*-$C_2B_{10}H_{10}$ (**1**; 20.2 mg, 0.10 mmol), diphenylacetylene (**2a**; 53.4 mg, 0.30 mmol), $Cu(OPiv)_2$ (**3a**; 79.8 mg, 0.30 mmol), $[Cp^*RhCl_2]_2$ (3.1 mg, 0.005 mmol), $AgSbF_6$ (6.9 mg, 0.02 mmol) and Li_2CO_3 (44.4 mg, 0.6 mmol) in a closed Schlenk flask was heated at 160 °C (bath temperature) for 2 h. After removal of the solvent, the residue was then subjected to column chromatography on silica gel (230-400 mesh) using *n*-hexane and ethyl acetate (100/1 in V/V) as an eluent to give **4a**.



4a: 46.0 mg, 70% yield. Colorless crystals. Mp: 171–173 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32 (m, 3H), 7.19 (m, 4H), 7.13 (m, 6H), 7.03 (m, 3H), 6.95 (m, 2H) (aryl C*H*), 6.85 (s, 1H) (alkenyl C*H*), 6.80 (m, 2H) (aryl C*H*), 2.18 (s, 3H) (cage CH_3), 1.14 (s, 9H) (^{t}Bu). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 175.8, 153.4 (*C*=O), 151.9, 141.2, 140.8, 138.2, 137.0, 132.7, 129.8, 129.6, 129.3, 129.2, 129.1, 128.8, 128.5, 128.0, 127.9, 127.4, 127.1, 127.0 (aryl & alkenyl *C*), 76.1, 67.4 (cage *C*), 39.9, 27.2 (^{t}Bu), 21.0 (cage *C*H_3). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -3.81 (1B), -6.00 (2B), -7.09 (2B), -14.17 (5B). HRMS (ESI) *m/z*: $[M+Na]^+$ Calcd for $C_{37}H_{40}B_{10}O_4Na$ 679.3840; Found 679.3821.

Single crystals of **4a** were grown from slow evaporation of *n*-hexane solution at room temperature over 3 days. The data were collected on a Bruker AXS Kappa ApexII Duo Diffractometer.

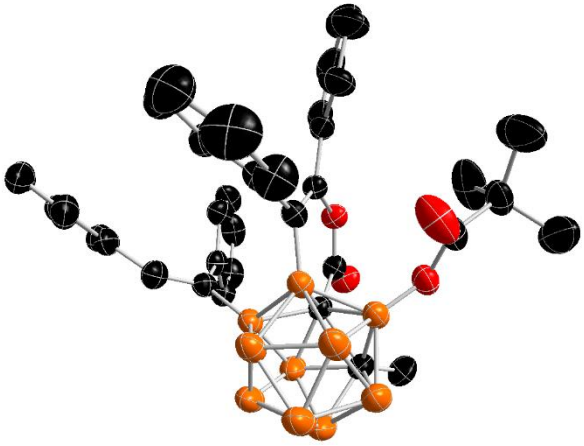
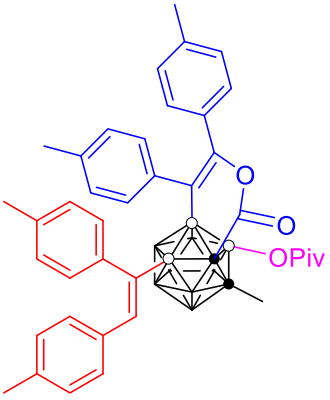


Figure S1. Molecular Structure of **4a**. The thermal ellipsoids are drawn at the 40% probability level.

Table S1. Crystal data and structure refinement for **4a**.

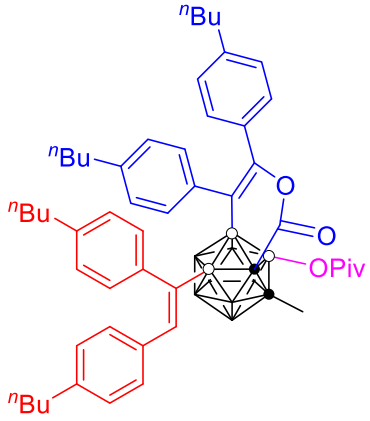
Empirical formula	C37 H40 B10 O4
Formula weight	656.79
Temperature	300(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic

Space group	P-1	
Unit cell dimensions	a = 10.417(8) Å	α= 82.25(2)°.
	b = 13.107(9) Å	β= 69.77(2)°.
	c = 14.452(10) Å	γ = 86.78(2)°.
Volume	1834(2) $Å^3$	
Z	2	
Density (calculated)	1.189 Mg/m^3	
Absorption coefficient	0.070 mm^{-1}	
F(000)	688	
Crystal size	0.400 x 0.300 x 0.200 mm^3	
Theta range for data collection	2.310 to 25.249°.	
Index ranges	-12<=h<=12, -15<=k<=15, -17<=l<=17	
Reflections collected	49399	
Independent reflections	6610 [R(int) = 0.0579]	
Completeness to theta = 25.242°	99.2 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7456 and 0.6839	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6610 / 0 / 461	
Goodness-of-fit on F^2	1.053	
Final R indices [I>2sigma(I)]	R1 = 0.0701, wR2 = 0.1757	
R indices (all data)	R1 = 0.1093, wR2 = 0.2055	
Extinction coefficient	0.091(6)	
Largest diff. peak and hole	0.576 and -0.621 e.$Å^{-3}$	

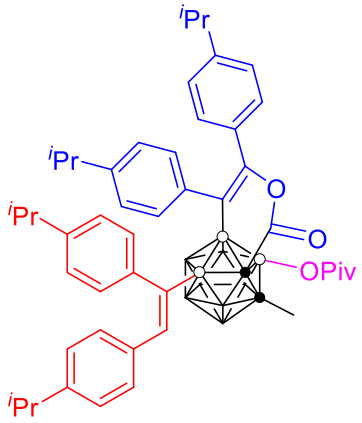


4b: 51.3 mg, 72% yield. Pale yellow solid. Mp: 123–125 °C. The crude product was purified by column chromatography on silica gel (230-400 mesh) using *n*-hexane and ethyl acetate (100/1 in V/V) as eluent. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.12 (d, *J* = 8.0 Hz, 2H), 7.03 (d, *J* = 8.0 Hz, 2H), 6.98 (m, 4H), 6.91 (d, *J* = 8.0 Hz, 2H), 6.84 (m, 4H) (aryl C*H*), 6.78 (s, 1H) (alkenyl C*H*), 6.69 (d, *J* = 8.0 Hz, 2H) (aryl C*H*), 2.39 (s, 3H), 2.30 (s, 3H), 2.24 (s, 3H), 2.19 (s, 3H), 2.16 (s, 3H) (cage CH_3 & aryl CH_3), 1.13 (s, 9H) (^{t}Bu). $^{13}C\{^1H\}$ NMR (125

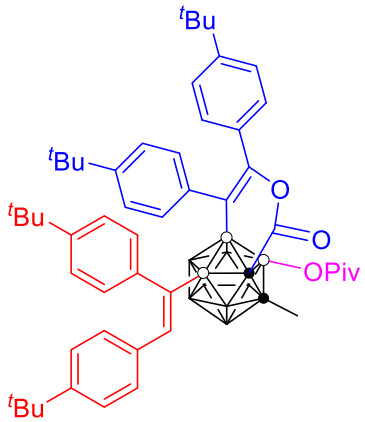
MHz, $CDCl_3$): δ 175.8, 153.6 (*C*=O), 151.8, 140.9, 138.9, 138.0, 137.2, 136.5, 136.4, 135.4, 134.4, 130.0, 129.7, 129.6, 129.4, 129.3, 129.2, 129.1, 128.6, 128.5 (aryl & alkenyl *C*), 75.9, 67.4 (cage *C*), 39.9, 27.2 (^{t}Bu), 21.5, 21.4, 21.3, 21.2, 21.0 (CH_3). $^{11}B\{^1H\}$ NMR (160 MHz, $CDCl_3$): δ -3.55 (1B), -6.65 (4B), -13.85. HRMS (ESI) *m/z*: $[M+Na]^+$ Calcd for $C_{41}H_{48}B_{10}O_4Na$ 736.4442; Found 736.4421.



4c: 65.2 mg, 74% yield. Colorless oil. The crude product was purified by column chromatography on silica gel (230-400 mesh) using *n*-hexane and ethyl acetate (100/1 in V/V) as eluent. ^1H NMR (400 MHz, $CDCl_3$): δ 7.12 (d, *J* = 8.0 Hz, 2H), 7.03 (dd, *J* = 8.0, 8.0 Hz, 4H), 6.96 (d, *J* = 8.0 Hz, 2H), 6.91 (d, *J* = 8.0 Hz, 2H), 6.84 (d, *J* = 8.0 Hz, 2H), 6.79 (m, 3H), 6.69 (d, *J* = 8.0 Hz, 2H) (aryl & alkenyl C*H*), 2.60 (m, 4H), 2.47 (m, 4H) (^{n}Bu), 2.18 (s, 3H) (cage CH_3), 1.62 (m, 4H), 1.50 (m, 4H), 1.38 (m, 4H), 1.30 (m, 4H) (^{n}Bu), 1.15 (s, 9H) (^{t}Bu), 0.98 (t, *J* = 7.4 Hz, 3H), 0.93 (t, *J* = 7.4 Hz, 3H), 0.89 (t, *J* = 7.4 Hz, 3H), 0.86 (t, *J* = 7.4 Hz, 3H). $^{13}C\{^1H\}$ NMR (125 MHz, CD_2Cl_2): δ 175.9, 153.8 (*C*=O), 152.3, 144.5, 142.7, 142.0, 141.9, 141.0, 138.6, 135.8, 134.8, 130.5, 129.9, 129.8, 129.7, 129.3, 128.9, 128.7, 128.2, 128.1 (aryl & alkenyl *C*), 76.6, 68.0 (cage *C*), 40.1 (^{t}Bu), 35.8, 35.7, 35.6, 35.5, 34.3, 33.8, 33.7, 33.6 (^{n}Bu), 27.2 (^{t}Bu), 22.9, 22.8, 22.7, 22.6 (^{n}Bu), 21.2 (cage CH_3), 14.3, 14.2, 14.1, 14.0 (^{n}Bu). $^{11}B\{^1H\}$ NMR (128 MHz, $CDCl_3$): δ -4.00 (1B), -6.40 (4B), -14.34 (5B). HRMS (ESI) *m/z*: $[M+Na]^+$ Calcd for $C_{53}H_{72}B_{10}O_4Na$ 904.6327; Found 904.6320.

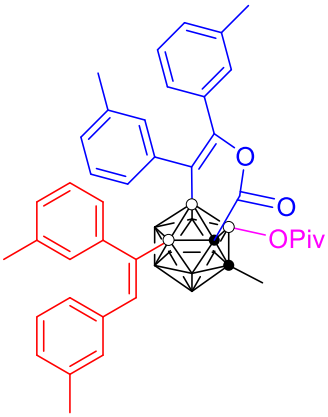


4d: 61.9 mg, 75% yield. Pale yellow solid. Mp: 160–162 °C. The crude product was purified by column chromatography on silica gel (230-400 mesh) using *n*-hexane and ethyl acetate (100/1 in V/V) as eluent. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.17 (d, J = 8.0 Hz, 2H), 7.06 (dd, J = 8.0, 8.0 Hz, 4H), 6.99 (dd, J = 8.0, 8.0 Hz, 4H), 6.90 (d, J = 8.0 Hz, 2H) (aryl C*H*), 6.82 (s, 1H) (alkenyl C*H*), 6.74 (dd, J = 8.0, 8.0 Hz, 4H) (aryl C*H*), 2.83 (m, 4H) (^{i}Pr), 2.21 (s, 3H) (cage CH_3), 1.32 (d, J = 7.4 Hz, 6H), 1.22 (d, J = 7.4 Hz, 6H) (^{i}Pr), 1.19 (s, 9H) (^{t}Bu), 1.18 (d, J = 7.4 Hz, 6H), 1.15 (d, J = 7.4 Hz, 6H) (^{i}Pr). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 175.8, 153.7 (*C*=O), 151.7, 149.6, 148.1, 147.6, 147.3, 140.4, 138.6, 135.5, 134.8, 130.3, 129.9, 129.6, 129.5, 129.1, 126.7, 126.4, 126.0, 125.8 (aryl & alkenyl *C*), 76.0, 67.7 (cage *C*), 39.9 (^{t}Bu), 34.0, 33.9, 33.8, 33.7 (^{i}Pr), 27.3 (^{t}Bu), 24.3, 24.0, 23.9, 23.8 (^{i}Pr), 21.0 (cage *C*H$_3$). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -3.76 (1B), -6.42 (4B), -14.05 (5B). HRMS (ESI) *m/z*: $[M+Na]^+$ Calcd for $C_{49}H_{64}B_{10}O_4Na$ 848.5698; Found 848.5676.

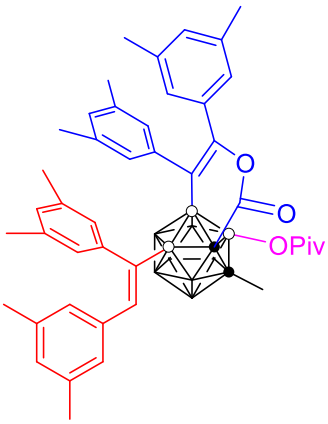


4e: 65.2 mg, 74% yield. Pale yellow solid. Mp: 138–140 °C. The crude product was purified by column chromatography on silica gel (230-400 mesh) using *n*-hexane and ethyl acetate (100/1 in V/V) as eluent. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37 (d, J = 8.0 Hz, 1H), 7.29 (d, J = 8.0 Hz, 2H), 7.10 (m, 3H), 7.05 (m, 5H) (aryl C*H*), 6.81 (s, 1H) (alkenyl C*H*), 6.75 (m, 3H), 6.68 (m, 2H) (aryl C*H*), 2.20 (s, 3H) (cage CH_3), 1.35 (s, 9H), 1.27 (s, 9H), 1.22 (s, 9H), 1.19 (s, 9H), 1.18 (s, 9H) (^{t}Bu). ^{13}C{^{1}H} NMR (125 MHz, CD_2Cl_2): δ 177.4, 155.4 (*C*=O), 154.0, 153.6, 152.4, 151.8, 151.6, 141.9,

140.0, 136.9, 136.1, 131.8, 131.2, 131.1, 130.9, 130.6, 127.3, 127.0, 126.7, 126.6 (aryl & alkenyl *C*), 78.2, 69.7 (cage *C*), 41.6, 36.4, 36.3, 36.2, 36.1, 33.2, 32.9, 32.8, 32.7, 28.8 (^{t}Bu), 22.8 (cage *C*H$_3$). ^{11}B{^{1}H} NMR (128 MHz, CDCl$_3$): δ -2.99 (1B), -5.36 (4B), -13.26 (5B). HRMS (ESI) *m/z*: [M+Na]$^+$ Calcd for $C_{53}H_{72}B_{10}O_4Na$ 904.6327; Found 904.6315.



4f: 47.0 mg, 66% yield. Pale yellow solid. Mp: 127–129 °C. The crude product was purified by column chromatography on silica gel (230-400 mesh) using *n*-hexane and ethyl acetate (100/1 in V/V) as eluent. ^{1}H NMR (400 MHz, CD_2Cl_2): δ 7.16 (m, 2H), 7.00 (m, 4H), 6.86 (m, 7H), 6.69 (m, 2H), 6.53 (m, 2H) (aryl & alkenyl C*H*), 2.27 (s, 3H), 2.20 (s, 3H), 2.17 (s, 3H), 2.16 (s, 3H), 2.10 (s, 3H) (cage C*H*$_3$ & aryl C*H*$_3$), 1.18 (s, 9H) (^{t}Bu). ^{13}C{^{1}H} NMR (125 MHz, CD_2Cl_2): δ 175.9, 153.8 (*C*=O), 152.3, 141.2, 140.9, 138.5, 138.4, 138.2, 137.9, 137.8, 137.2, 133.0, 131.0, 130.8, 130.6, 130.2, 130.0, 128.7, 128.4, 128.0, 127.9, 127.8, 127.7, 127.4, 127.1, 126.9, 126.8, 125.7 (aryl & alkenyl *C*), 76.6, 67.9 (cage *C*), 40.1, 27.2 (^{t}Bu), 21.6, 21.5, 21.4, 21.3, 21.2 (*C*H$_3$). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -3.38 (1B), -6.04 (4B), -13.64 (5B). HRMS (ESI) *m/z*: [M+Na]$^+$ Calcd for $C_{41}H_{48}B_{10}O_4Na$ 736.4442; Found 736.4422.



4g: 41.5 mg, 54% yield. Colorless crystals. Mp: 160–162 °C. The crude product was purified by column chromatography on silica gel (230-400 mesh) using *n*-hexane and ethyl acetate (100/1 in V/V) as eluent. ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.94 (s, 1H), 6.78 (s, 2H), 6.75 (m, 4H), 6.66 (m, 2H), 6.44 (s, 2H), 6.32 (s, 2H) (aryl & alkenyl C*H*), 2.28 (s, 3H), 2.23 (s, 3H), 2.20 (s, 3H), 2.12 (s, 6H), 2.08 (s, 6H), 2.05 (s, 6H) (cage & aryl C*H*$_3$), 1.18 (s, 9H) (^{t}Bu).

$^{13}C\{^{1}H\}$ NMR (125 MHz, CD_2Cl_2): δ 177.3, 155.5 (*C*=O), 153.6, 142.7, 141.8, 139.8, 139.7, 139.3, 138.9, 138.6, 134.3, 132.3, 130.8, 130.1, 129.6, 129.4, 129.2, 128.9, 128.8, 128.6 (aryl & alkenyl *C*), 78.0, 69.5 (cage *C*), 41.5, 31.6, 28.7, 22.9, 22.8, 22.7, 22.6 (^{t}Bu & aryl & cage *C*H_3). $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$): δ -3.37 (1B), -5.62 (4B), -13.38 (5B). HRMS (ESI) *m/z*: $[M+Na]^+$ Calcd for $C_{45}H_{56}B_{10}O_4Na$ 792.5070; Found 792.5053.

Single crystals of **4g** were grown from slow evaporation of *n*-hexane solution at room temperature over 3 days. The data were collected on a Bruker AXS Kappa ApexII Duo Diffractometer.

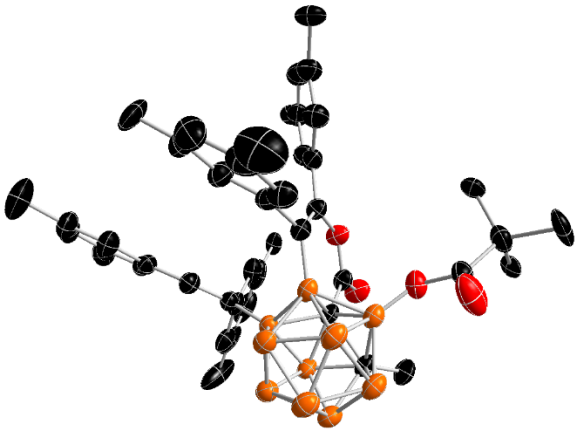
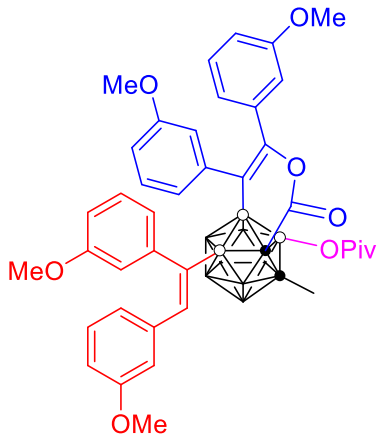


Figure S2. Molecular Structure of **4g**. The thermal ellipsoids are drawn at the 50% probability level.

Table S2. Crystal data and structure refinement for **4g**.

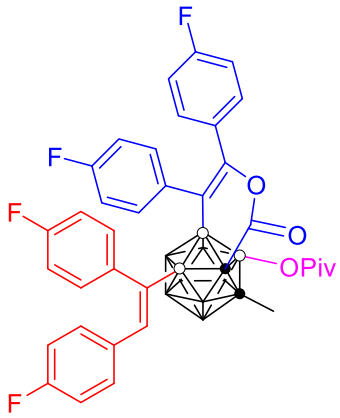
Empirical formula	C45 H56 B10 O4	
Formula weight	768.99	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 26.398(2) Å	α= 90°.
	b = 12.3769(8) Å	β= 104.828(6)°.
	c = 28.5896(18) Å	γ = 90°.
Volume	9029.9(11) $Å^3$	
Z	8	
Density (calculated)	1.131 Mg/m^3	
Absorption coefficient	0.066 mm^{-1}	

F(000)	3264
Crystal size	0.500 x 0.400 x 0.300 mm^3
Theta range for data collection	1.474 to 25.250°.
Index ranges	-31<=h<=31, -14<=k<=14, -34<=l<=33
Reflections collected	67963
Independent reflections	8192 [R(int) = 0.0518]
Completeness to theta = 25.242°	100.0 %
Absorption correction	multi-scan
Max. and min. transmission	0.7456 and 0.6867
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	8192 / 0 / 544
Goodness-of-fit on F^2	1.035
Final R indices [I>2sigma(I)]	R1 = 0.0542, wR2 = 0.1487
R indices (all data)	R1 = 0.0705, wR2 = 0.1617
Extinction coefficient	n/a
Largest diff. peak and hole	0.429 and -0.288 e.$Å^{-3}$



4h: 32.6 mg, 42% yield. Colorless oil. The crude product was purified by column chromatography on silica gel (230-400 mesh) using *n*-hexane and ethyl acetate (20/1 in V/V) as eluent. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.25 (d, *J* = 8.0 Hz, 1H), 7.10 (m, 2H), 7.01 (d, *J* = 8.0 Hz, 1H), 6.86 (m, 3H), 6.76 (m, 3H), 6.72 (s, 1H), 6.69 (s, 2H), 6.65 (d, *J* = 8.0 Hz, 1H), 6.60 (d, *J* = 8.0 Hz, 1H), 6.53 (d, *J* = 8.0 Hz, 1H), 6.39 (s, 1H) (aryl & alkenyl C*H*), 3.73 (s, 3H), 3.65 (s, 3H), 3.50 (s, 3H), 3.47 (s, 3H) (OCH_3), 2.21 (s, 3H) (cage CH_3), 1.18 (s, 9H) (^{t}Bu). ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 175.7 (*C*=O), 160.0, 159.7, 159.0, 158.9 (aryl *C*), 153.4 (*C*=O), 151.9, 142.4, 141.1, 139.6, 138.1, 133.9, 129.9, 129.5, 129.0, 128.9, 122.8, 122.2, 121.8, 121.5, 115.9, 114.7, 114.6, 114.5, 114.4, 113.7, 113.5, 112.7 (aryl & alkenyl *C*), 76.2, 67.3 (cage *C*), 55.4, 55.3, 55.1, 54.9 (O*C*H$_3$), 39.9, 27.2 (^{t}Bu), 21.0 (cage *C*H$_3$). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -3.55

(1B), -5.84 (4B), -14.02 (5B). HRMS (ESI) *m/z*: $[M+Na]^+$ Calcd for $C_{41}H_{48}B_{10}O_8Na$ 800.4239; Found 800.4230.



4i: 47.5 mg, 65% yield. White solid. Mp: 195–197 °C. The crude product was purified by column chromatography on silica gel (230-400 mesh) using *n*-hexane and ethyl acetate (100/1 in V/V) as eluent. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.11-7.06 (m, 2H), 7.03 (d, J = 7.0 Hz, 4H), 6.94-6.81 (m, 6H), 6.79-6.70 (m, 5H) (aryl & alkenyl C*H*), 2.20 (s, 3H) (cage C*H*$_3$), 1.13 (s, 9H) (^{t}Bu). ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 175.5 (*C*=O), 162.7 (d, $^1J_{C\text{-}F}$ = 250 Hz), 162.0 (d, $^1J_{C\text{-}F}$ = 245 Hz), 161.9 (d, $^1J_{C\text{-}F}$ = 246 Hz), 161.8 (d, $^1J_{C\text{-}F}$ = 247 Hz) (aryl *C*), 153.0 (*C*=O), 150.9, 140.3 (alkenyl *C*), 136.1 (d, $^4J_{C\text{-}F}$ = 3.4 Hz), 133.6 (d, $^4J_{C\text{-}F}$ = 3.4 Hz), 132.6 (d, $^4J_{C\text{-}F}$ = 3.3 Hz), 131.3 (d, $^3J_{C\text{-}F}$ = 8.5 Hz), 131.2 (d, $^3J_{C\text{-}F}$ = 7.8 Hz), 131.1 (d, $^3J_{C\text{-}F}$ = 7.6 Hz), 130.6 (d, $^3J_{C\text{-}F}$ = 6.8 Hz), 128.3 (d, $^4J_{C\text{-}F}$ = 3.5 Hz), 115.8 (d, $^2J_{C\text{-}F}$ = 18.5 Hz), 115.6 (d, $^2J_{C\text{-}F}$ = 18.8 Hz), 115.2 (d, $^2J_{C\text{-}F}$ = 21.6 Hz), 115.0 (d, $^2J_{C\text{-}F}$ = 21.3 Hz) (aryl *C*), 76.1, 67.2 (cage *C*), 39.8, 27.0 (^{t}Bu), 20.8 (cage *C*H$_3$). ^{11}B{^{1}H} NMR (160 MHz, $CDCl_3$): δ -3.53 (1B), -5.35 (2B), -6.94 (2B), -13.87 (5B). HRMS (APCI) *m/z*: $[M]^-$ Calcd for $C_{37}H_{36}B_{10}F_4O_4$ 728.3576; Found 728.3553.

Single crystals of **4i** were grown from slow evaporation of *n*-hexane solution at room temperature over 3 days. The data were collected on a Bruker AXS Kappa ApexII Duo Diffractometer.

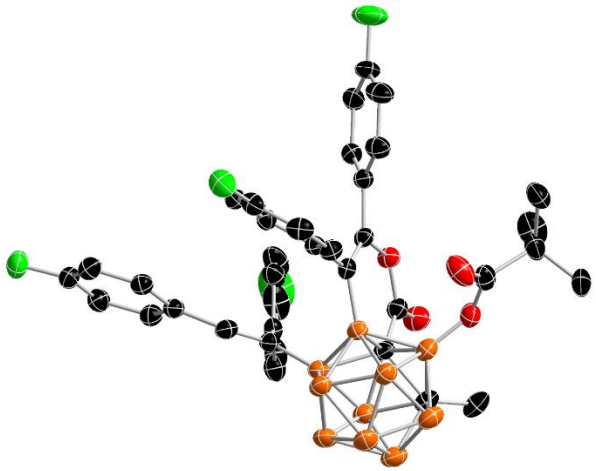
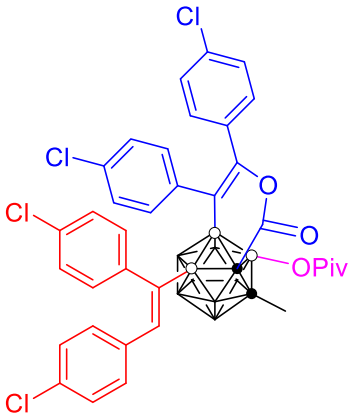


Figure S3. Molecular Structure of **4i**. The thermal ellipsoids are drawn at the 40% probability level.

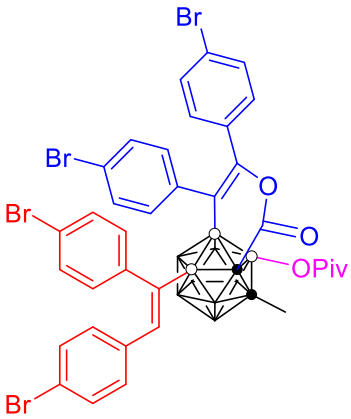
Table S3. Crystal data and structure refinement for **4i**.

Empirical formula	C37 H36 B10 F4 O4	
Formula weight	728.76	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	Cc	
Unit cell dimensions	a = 10.3780(8) Å	α= 90°.
	b = 21.2751(15) Å	β= 93.702(2)°.
	c = 17.0225(12) Å	γ = 90°.
Volume	3750.6(5) $Å^3$	
Z	4	
Density (calculated)	1.291 Mg/m^3	
Absorption coefficient	0.091 mm^{-1}	
F(000)	1504	
Crystal size	0.400 x 0.300 x 0.200 mm^3	
Theta range for data collection	2.187 to 25.249°.	
Index ranges	-12<=h<=12, -25<=k<=25, -20<=l<=20	
Reflections collected	43605	
Independent reflections	6811 [R(int) = 0.1017]	
Completeness to theta = 25.242°	100.0 %	
Absorption correction	multi-scan	
Max. and min. transmission	0.7456 and 0.6723	
Refinement method	Full-matrix least-squares on F^2	

Data / restraints / parameters	6811 / 7 / 525
Goodness-of-fit on F^2	1.049
Final R indices [I>2sigma(I)]	R1 = 0.0431, wR2 = 0.0949
R indices (all data)	R1 = 0.0694, wR2 = 0.1114
Absolute structure parameter	-0.5(3)
Extinction coefficient	0.0043(5)
Largest diff. peak and hole	0.164 and -0.162 e.Å^{-3}

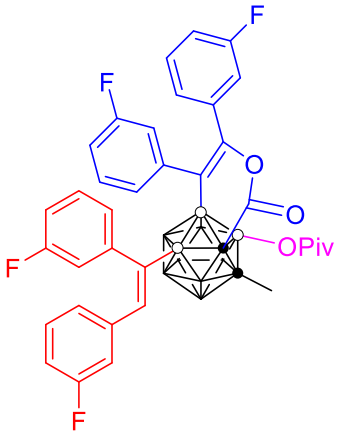


4j: 43.2 mg, 54% yield. Colorless oil. The crude product was purified by column chromatography on silica gel (230-400 mesh) using *n*-hexane and ethyl acetate (100/1 in V/V) as eluent. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.30 (d, *J* = 7.5 Hz, 2H), 7.20-7.12 (m, 4H), 7.07-6.95 (m, 6H), 6.81-6.75 (m, 3H), 6.68 (d, *J* = 8.5 Hz, 2H) (aryl & alkenyl C*H*), 2.20 (s, 3H) (cage C*H*$_3$), 1.13 (s, 9H) (^{t}Bu). ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 175.5, 152.9 (*C*=O), 150.8, 140.1 (alkenyl *C*), 138.6, 136.0, 135.5, 134.7, 133.5, 133.4, 133.3, 130.8, 130.7, 130.5, 130.5, 130.4, 129.0, 128.9, 128.5, 128.3 (aryl *C*), 76.2, 67.2 (cage *C*), 39.8, 27.0 (^{t}Bu), 20.8 (cage *C*H$_3$). ^{11}B{^{1}H} NMR (160 MHz, $CDCl_3$): δ -3.42 (1B), -5.35 (1B), -7.07 (3B), -13.89 (5B). HRMS (APCI) *m/z*: $[M]^-$ Calcd for $C_{37}H_{36}B_{10}Cl_4O_4$ 794.2363; Found 794.2333.



4k: 49.3 mg, 51% yield. Colorless oil. The crude product was purified by column chromatography on silica gel (230-400 mesh) using *n*-hexane and ethyl acetate (100/1 in V/V) as eluent. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.44 (d, *J* = 7.5 Hz, 2H), 7.35-7.28 (m, 4H), 7.20 (d, *J* = 7.5 Hz, 2H), 6.96-6.88 (m, 4H), 6.76-6.67 (m, 3H), 6.62 (d, *J* = 8.0 Hz, 2H) (aryl & alkenyl C*H*), 2.20 (s, 3H) (cage C*H*$_3$), 1.13 (s, 9H) (^{t}Bu). ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$):

δ 175.5, 152.9 (*C*=O), 150.8, 140.1 (alkenyl *C*), 139.1, 136.4, 135.0, 132.0, 131.9, 131.4, 131.3, 131.0, 130.9, 130.8, 130.6, 123.9, 121.8, 121.6, 121.4 (aryl *C*), 76.2, 67.2 (cage *C*), 39.8, 27.0 (^{t}Bu), 20.8 (cage *C*H$_3$). ^{11}B{^{1}H} NMR (160 MHz, CDCl$_3$): δ -3.42 (1B), -5.46 (1B), -7.23 (3B), -14.08 (5B). HRMS (APCI) *m/z*: [M]$^-$ Calcd for $C_{37}H_{36}B_{10}Br_4O_4$ 972.0331; Found 972.0311.



4l: 34.9 mg, 48% yield. White solid. Mp: 170–172 °C. The crude product was purified by column chromatography on silica gel (230-400 mesh) using *n*-hexane and ethyl acetate (100/1 in V/V) as eluent. ^{1}H NMR (500 MHz, CDCl$_3$): δ 7.33 (dd, *J* = 8.0, 14.0 Hz, 1H), 7.18 (dd, *J* = 8.0, 14.0 Hz, 1H), 7.11 (dd, *J* = 8.0, 14.0 Hz, 1H), 7.08-7.01 (m, 2H), 6.96-6.90 (m, 2H), 6.89-6.84 (m, 3H), 6.82-6.75 (m, 3H), 6.71 (d, *J* = 7.5 Hz, 1H), 6.65-6.57 (m, 2H), 6.43 (d, *J* = 10.5 Hz, 1H) (aryl & alkenyl C*H*), 2.20 (s, 3H) (cage C*H*$_3$), 1.14 (s, 9H) (^{t}Bu). ^{13}C{^{1}H} NMR (125 MHz, CDCl$_3$): δ 175.5 (*C*=O), 163.0 (d, $^1J_{C\text{-}F}$ = 246 Hz), 162.7 (d, $^1J_{C\text{-}F}$ = 245 Hz), 162.3 (d, $^1J_{C\text{-}F}$ = 244 Hz), 162.1 (d, $^1J_{C\text{-}F}$ = 245 Hz) (aryl *C*), 152.7 (*C*=O), 150.7 (d, $^4J_{C\text{-}F}$ = 2.6 Hz) (alkenyl *C*), 142.1 (d, $^3J_{C\text{-}F}$ = 7.3 Hz) (aryl *C*), 140.3 (d, $^4J_{C\text{-}F}$ = 2.3 Hz) (alkenyl *C*), 139.6 (d, $^3J_{C\text{-}F}$ = 8.0 Hz), 138.3 (d, $^3J_{C\text{-}F}$ = 7.5 Hz), 133.9 (d, $^3J_{C\text{-}F}$ = 8.0 Hz), 130.5 (d, $^3J_{C\text{-}F}$ = 8.4 Hz), 130.2 (d, $^3J_{C\text{-}F}$ = 8.5 Hz), 129.6 (d, $^3J_{C\text{-}F}$ = 8.1 Hz), 129.4 (d, $^3J_{C\text{-}F}$ = 8.3 Hz), 125.4 (d, $^4J_{C\text{-}F}$ = 2.8 Hz), 125.2 (d, $^4J_{C\text{-}F}$ = 2.8 Hz), 125.1 (d, $^4J_{C\text{-}F}$ = 2.8 Hz), 124.7, 116.6 (d, $^2J_{C\text{-}F}$ = 21.0 Hz), 116.3 (d, $^2J_{C\text{-}F}$ = 22.0 Hz), 116.1 (d, $^2J_{C\text{-}F}$ = 24.9 Hz), 115.9 (d, $^2J_{C\text{-}F}$ = 22.4 Hz), 114.6 (d, $^2J_{C\text{-}F}$ = 21.1 Hz), 114.5 (d, $^2J_{C\text{-}F}$ = 22.9 Hz), 114.4 (d, $^2J_{C\text{-}F}$ = 22.8 Hz), 76.2, 67.1 (cage *C*), 39.8, 27.0 (^{t}Bu), 20.8 (cage *C*H$_3$). ^{11}B{^{1}H} NMR (160 MHz, CDCl$_3$): δ -3.41 (1B), -5.34 (1B), -6.97 (3B), -13.75 (5B). HRMS (APCI) *m/z*: [M]$^-$ Calcd for $C_{37}H_{36}B_{10}F_4O_4$ 728.3576; Found 728.3546.

Single crystals of **4l** were grown from slow evaporation of *n*-hexane solution at room temperature over 3 days. The data were collected on a Bruker AXS Kappa ApexII Duo Diffractometer.

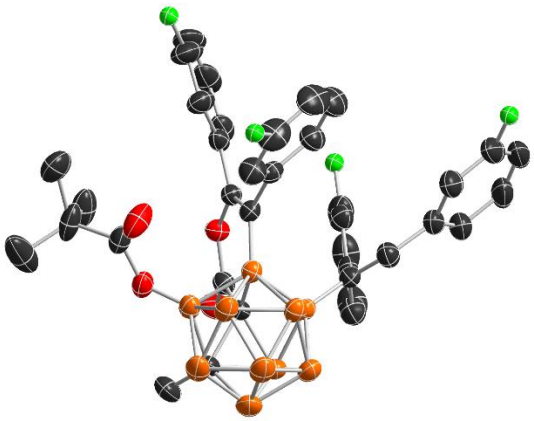
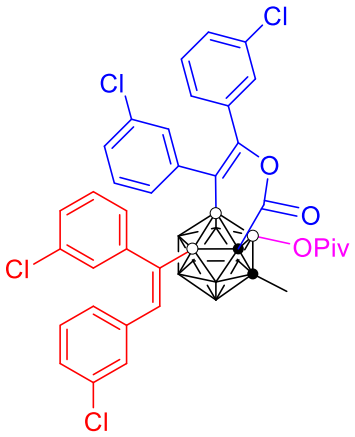


Figure S4. Molecular Structure of **4l**. The thermal ellipsoids are drawn at the 40% probability level.

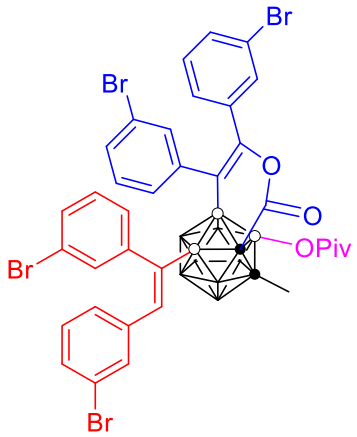
Table S4. Crystal data and structure refinement for **4l**.

Empirical formula	C37 H36 B10 F4 O4	
Formula weight	728.76	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 10.4650(12) Å	α= 96.643(4)°.
	b = 13.5333(16) Å	β= 111.103(4)°.
	c = 14.4641(17) Å	γ = 93.586(4)°.
Volume	1886.2(4) $Å^3$	
Z	2	
Density (calculated)	1.283 Mg/m^3	
Absorption coefficient	0.090 mm^{-1}	
F(000)	752	
Crystal size	0.400 x 0.300 x 0.200 mm^3	
Theta range for data collection	2.452 to 25.248°.	
Index ranges	-12<=h<=12, -16<=k<=16, -17<=l<=17	
Reflections collected	50988	
Independent reflections	6808 [R(int) = 0.1097]	
Completeness to theta = 25.242°	99.6 %	
Absorption correction	multi-scan	

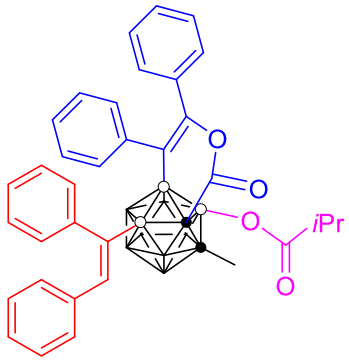
Max. and min. transmission	0.7456 and 0.6989
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6808 / 24 / 554
Goodness-of-fit on F^2	1.044
Final R indices [I>2sigma(I)]	R1 = 0.0685, wR2 = 0.1857
R indices (all data)	R1 = 0.0840, wR2 = 0.2034
Extinction coefficient	0.069(7)
Largest diff. peak and hole	0.755 and -0.333 e.Å^{-3}



4m: 38.8 mg, 49% yield. Colorless oil. The crude product was purified by column chromatography on silica gel (230-400 mesh) using *n*-hexane and ethyl acetate (100/1 in V/V) as eluent. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.39-7.28 (m, 2H), 7.24-7.12 (m, 4H), 7.09-7.02 (m, 3H), 7.01-6.95 (m, 2H), 6.92 (d, *J* = 8.0 Hz, 1H), 6.84-6.75 (m, 3H), 6.69 (s, 1H), 6.63 (d, *J* = 8.0 Hz, 1H) (aryl & alkenyl C*H*), 2.21 (s, 3H) (cage C*H*$_3$), 1.15 (s, 9H) (^{t}Bu). ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 175.4, 152.7 (*C*=O), 150.6, 141.7 (alkenyl *C*), 140.0, 139.1, 137.7, 134.7, 134.4, 134.2, 133.9, 133.4, 130.1, 129.9, 129.7, 129.6, 129.2, 129.1, 129.0, 128.7, 127.9, 127.8, 127.7, 127.6, 127.6, 127.5, 127.3 (aryl *C*), 76.2, 67.2 (cage *C*), 39.8, 27.0 (^{t}Bu), 20.8 (cage *C*H$_3$). ^{11}B{^{1}H} NMR (160 MHz, $CDCl_3$): δ -3.42 (1B), -5.30 (1B), -6.97 (3B), -13.58 (5B). HRMS (APCI) *m/z*: [M]$^-$ Calcd for $C_{37}H_{36}B_{10}Cl_4O_4$ 794.2363; Found 794.2334.

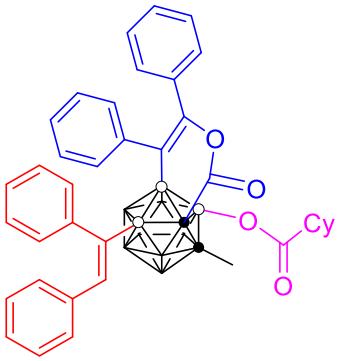


4n: 44.6 mg, 46% yield. Colorless oil. The crude product was purified by column chromatography on silica gel (230-400 mesh) using *n*-hexane and ethyl acetate (100/1 in V/V) as eluent. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.51 (d, *J* = 7.5 Hz, 1H), 7.40-7.33 (m, 3H), 7.25-7.17 (m, 3H), 7.11-6.90 (m, 6H), 6.84-6.74 (m, 3H), 6.68 (d, *J* = 7.5 Hz, 1H) (aryl & alkenyl C*H*), 2.22 (s, 3H) (cage C*H*$_3$), 1.16 (s, 9H) (^{t}Bu). ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 175.4, 152.7 (*C*=O), 150.5, 142.0 (alkenyl *C*), 139.8, 139.3, 138.0, 133.6, 132.7, 132.6, 131.9, 131.8, 131.5, 130.7, 130.5, 130.3, 130.2, 129.5, 129.4, 128.3, 128.0, 127.9, 127.8, 122.9, 122.6, 122.2, 122.1 (aryl *C*), 76.3, 67.3 (cage *C*), 39.8, 27.0 (^{t}Bu), 20.8 (cage *C*H$_3$). ^{11}B{^{1}H} NMR (160 MHz, $CDCl_3$): δ -3.44 (1B), -5.41 (1B), -7.23 (3B), -13.87 (5B). HRMS (APCI) *m/z*: $[M]^-$ Calcd for $C_{37}H_{36}B_{10}Br_4O_4$ 972.0331; Found 972.0295.



4o: 29.2 mg, 45% yield. White solid. Mp: 208–209 °C. The crude product was purified by column chromatography on silica gel (230-400 mesh) using *n*-hexane and ethyl acetate (100/1 in V/V) as eluent. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.36-7.29 (m, 3H), 7.20-7.09 (m, 10H), 7.07-7.00 (m, 3H), 6.96-6.90 (m, 2H) (aryl C*H*), 6.85 (s, 1H) (alkenyl C*H*), 6.81-6.76 (m, 2H) (aryl C*H*), 2.60-2.50 (m, 1H) (^{i}Pr), 2.17 (s, 3H) (cage C*H*$_3$), 1.13 (d, *J* = 7.0 Hz, 3H), 1.08 (d, *J* = 7.0 Hz, 3H) (^{i}Pr). ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 173.9, 153.2 (*C*=O), 151.8, 141.0, 140.7, 138.0, 136.8, 132.6, 129.7, 129.6, 129.4, 129.0, 128.9, 128.6, 128.3, 127.8, 127.3, 127.0, 126.9 (aryl & alkenyl *C*), 75.8, 67.3 (cage *C*), 35.2 (^{i}Pr), 20.8 (cage *C*H$_3$), 18.9, 18.7 (^{i}Pr). ^{11}B{^{1}H} NMR (160 MHz, $CDCl_3$): δ -3.68 (1B), -5.60 (1B), -

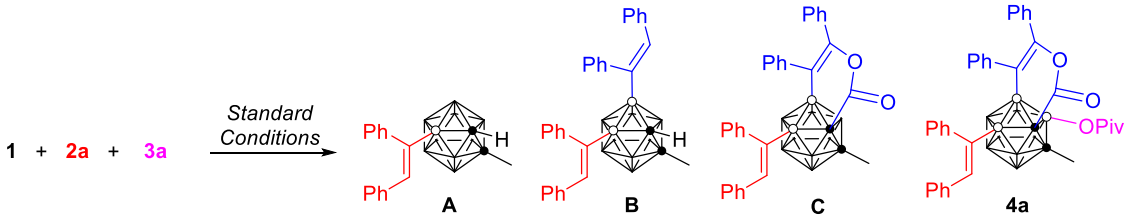
6.87 (3B), -13.97 (5B). HRMS (ESI) *m/z*: $[M]^-$ Calcd for $C_{36}H_{38}B_{10}O_4$ 642.3796; Found 642.3782.



4p: 21.0 mg, 31% yield. Colorless oil. The crude product was purified by column chromatography on silica gel (230-400 mesh) using *n*-hexane and ethyl acetate (100/1 in V/V) as eluent. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.36-7.28 (m, 3H), 7.21-7.09 (m, 10H), 7.08-7.01 (m, 3H), 6.93-6.84 (m, 3H), 6.82-6.77 (m, 2H) (aryl & alkenyl C*H*), 2.33-2.25 (m, 1H) (Cy), 2.17 (s, 3H) (cage C*H*$_3$), 1.91-1.80 (m, 2H), 1.70-1.60 (m, 2H), 1.42-1.02 (m, 6H) (Cy). ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 172.9, 153.3 (*C*=O), 151.8, 140.9, 140.7, 137.9, 136.8, 132.6, 129.7, 129.6, 129.4, 129.1, 128.9, 128.6, 128.3, 127.8, 127.7, 127.3, 127.0, 126.9 (aryl & alkenyl *C*), 75.8, 67.4 (cage *C*), 44.1, 29.0, 28.8, 25.4, 25.2, 25.1 (Cy), 20.8 (cage *C*H$_3$). ^{11}B{^{1}H} NMR (160 MHz, $CDCl_3$): δ -3.71 (1B), -5.61 (1B), -7.02 (3B), -12.40 (1B), -14.11 (4B). HRMS (ESI) *m/z*: $[M]^-$ Calcd for $C_{39}H_{42}B_{10}O_4$ 682.4110; Found 682.4098.

Preliminary mechanistic study

Production distributions at different reaction times

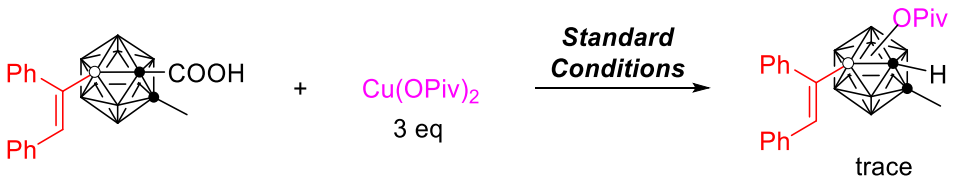


Each Schlenk flask was charged with a $PhCF_3$ (5 mL) suspension of 1-COOH-2-CH$_3$-*o*-$C_2B_{10}H_{10}$ (**1**; 10.1 mg, 0.05 mmol), diphenylacetylene (**2a**; 26.7 mg, 0.15 mmol), $Cu(OPiv)_2$ (**3a**; 39.9 mg, 0.15 mmol), $[Cp^*RhCl_2]_2$ (1.5 mg, 0.0025 mmol), $AgSbF_6$ (3.4 mg, 0.01 mmol) and Li_2CO_3 (22.2 mg, 0.3 mmol). The mixture was then heated at 160 °C (bath temperature) for 10 min, 15 min, 30 min, 45 min, and 60 min, respectively. After

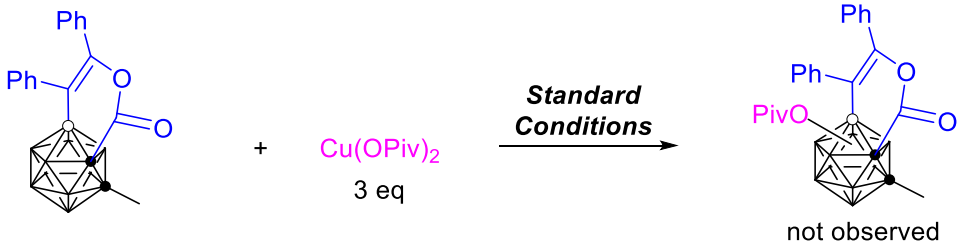
cooling to room temperature, the reaction mixture was quenched with diluted hydrochloric acid. The organic portion was analyzed by GC-MS.

time	**1** (%)	**A** (%)	**B** (%)	**C** (%)	**4a** (%)
10 min	89	1	2	2	6
15 min	79	2	2	2	15
30 min	4	2	4	6	84
45 min	0	3	6	4	87
60 min	0	3	3	6	88

Control experiments



A $PhCF_3$ (5 mL) suspension of 1-COOH-2-Me-4-[(Ph)C=CH(Ph)]-*o*-$C_2B_{10}H_9$ (19.0 mg, 0.05 mmol), $Cu(OPiv)_2$ (39.9 mg, 0.15 mmol), $[Cp^*RhCl_2]_2$ (1.5 mg, 0.0025 mmol), $AgSbF_6$ (3.4 mg, 0.01 mmol) and Li_2CO_3 (22.2 mg, 0.3 mmol) in a closed Schlenk flask was heated at 160 °C (bath temperature) for 2 h. After cooling to room temperature, the reaction mixture was analyzed by GC-MS. Only a trace amount of the expected acyloxylated species was observed.



A $PhCF_3$ (5 mL) suspension of *o*-carborane-fused isocoumarin (18.9 mg, 0.05 mmol), $Cu(OPiv)_2$ (39.9 mg, 0.15 mmol), $[Cp^*RhCl_2]_2$ (1.5 mg, 0.0025 mmol), $AgSbF_6$ (3.4 mg, 0.01 mmol) and Li_2CO_3 (22.2 mg, 0.3 mmol) in a closed Schlenk flask was heated at 160 °C (bath temperature) for 2 h. After cooling to room temperature, the reaction mixture was analyzed by GC-MS. No acyloxylated species was observed.

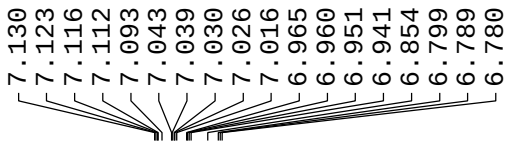
X-ray Structure Determination. X-ray data of **4a**, **4g, 4i** and **4l** were collected on a Bruker SMART 1000 CCD diffractometer using Mo-Kα radiation. An empirical absorption correction was applied using the SADABS program.[3] All structures were solved by direct methods and subsequent Fourier difference techniques and refined anisotropically for all non-hydrogen atoms by full-matrix least squares calculations on F^2 using the SHELXTL program package.[4] All hydrogen atoms were geometrically fixed using the riding model.

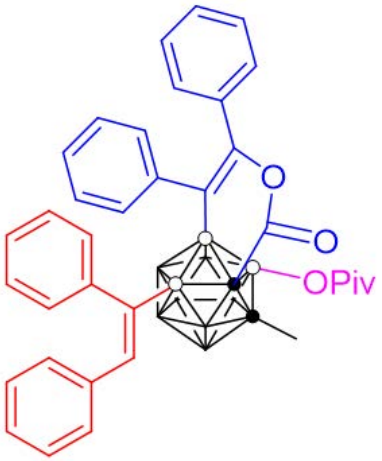
CCDC (**4a**: 2087978, **4g**: 2087980, **4i**: 2087981 and **4l**: 2087983) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

References

1. Y. Quan and Z. Xie, *J. Am. Chem. Soc.*, 2014, **136**, 15513−15516.
2. B. Cheng, Y. Chen and Z. Xie, *J. Org. Chem.* 2021, **86**, 12412-12418.
3. G. M. Sheldrick, S*ADABS: Program for Empirical Absorption Correction of Area Detector Data*. (University of Göttingen: Germany, 1996).
4. G. M. Sheldrick, *SHELXTL 5.10 for Windows NT: Structure Determination Software Programs*. (Bruker Analytical X-ray Systems, Inc., Madison, Wisconsin, USA, 1997).

CY-H-A-80p





4a

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PROCNO                 1

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PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                   203
DW                62.400 usec
DE                 6.50 usec
TE                295.5 K
D1             1.00000000 sec
TD0                    1
SFO1         400.1324708 MHz
NUC1                  1H
P1                15.00 usec
PLW1           8.31000042 W

F2 - Processing parameters
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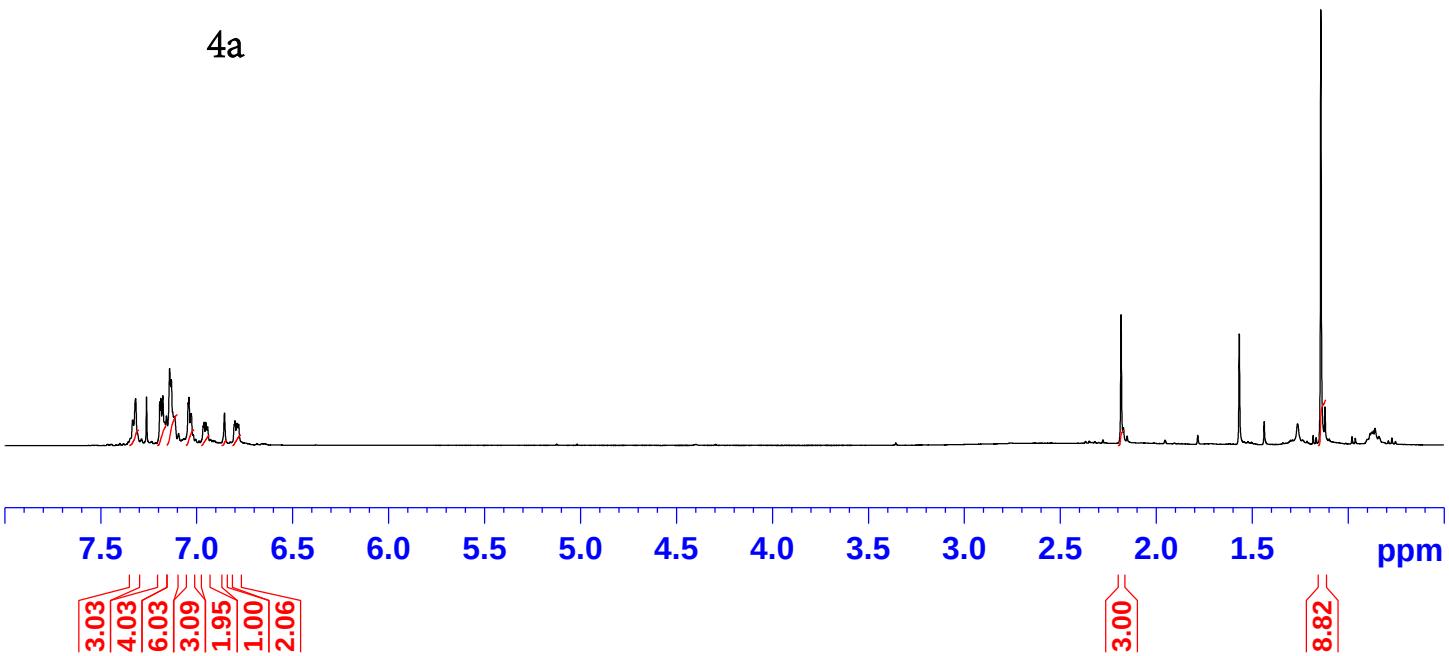
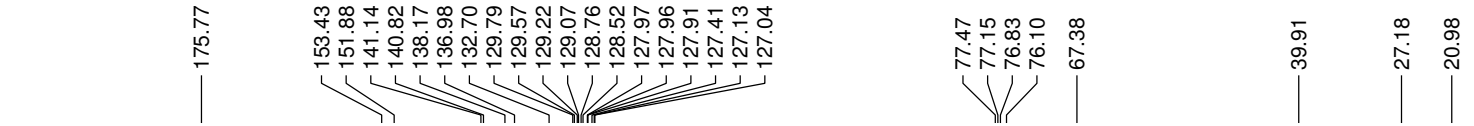
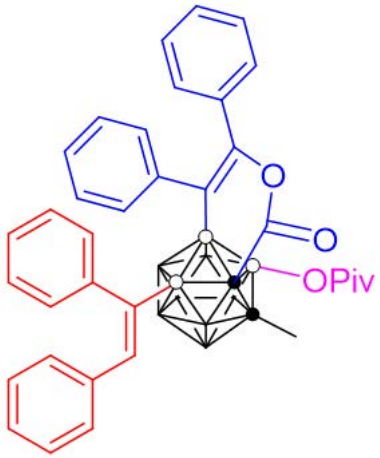
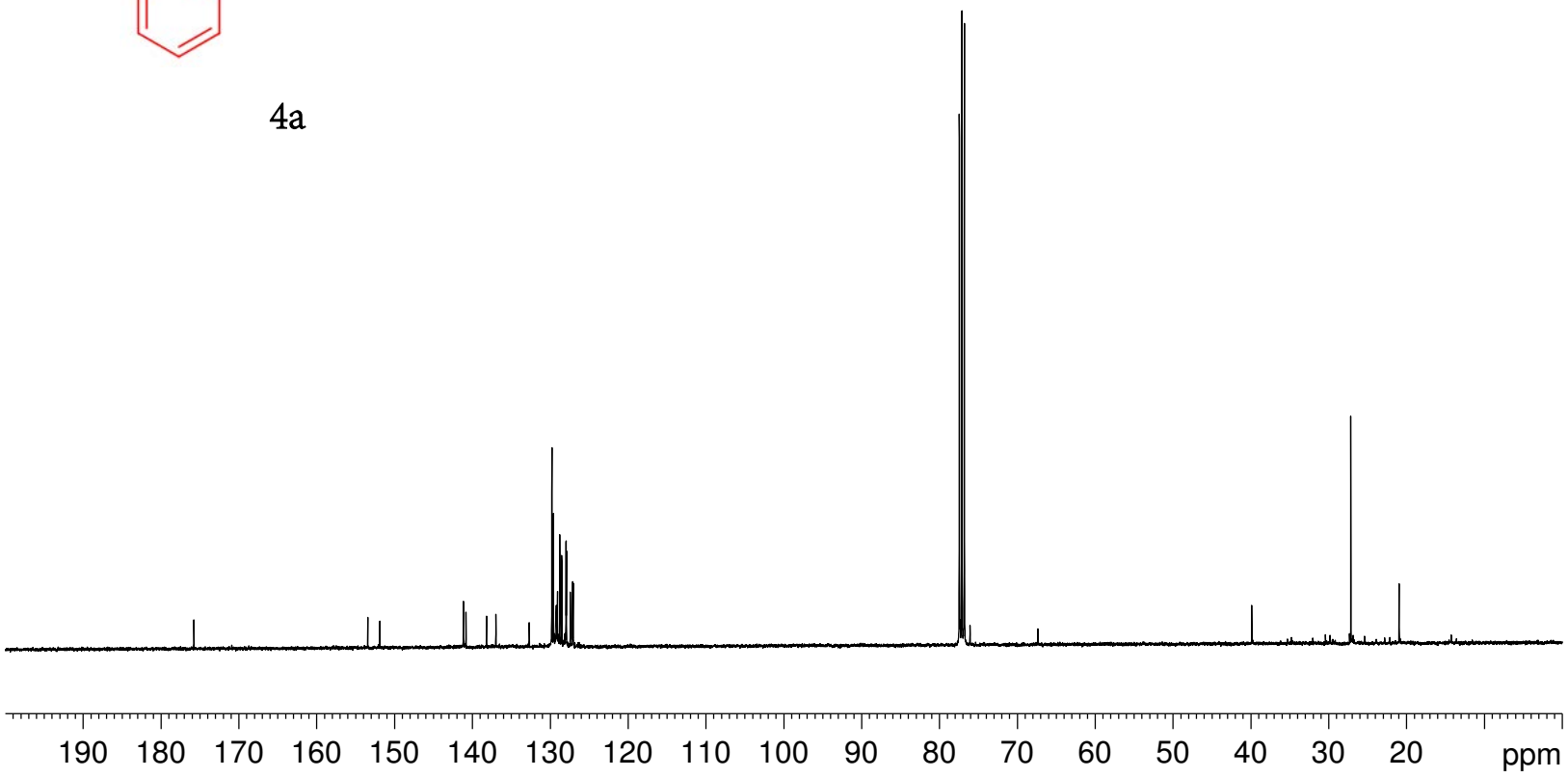




CY-C-A-81p



4a

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TD                65536
SOLVENT           CDCl3
NS                 3072
DS                    4
SWH           40760.871 Hz
FIDRES         0.621962 Hz
AQ            0.8039083 sec
RG                  161
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DE                6.50 usec
TE                294.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
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NUC1                13C
P1                9.50 usec
PLW1        55.34000015 W
SFO2        400.2316009 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        13.56000042 W
PLW12        0.27428001 W
PLW13        0.13796000 W

F2 - Processing parameters
SI                32768
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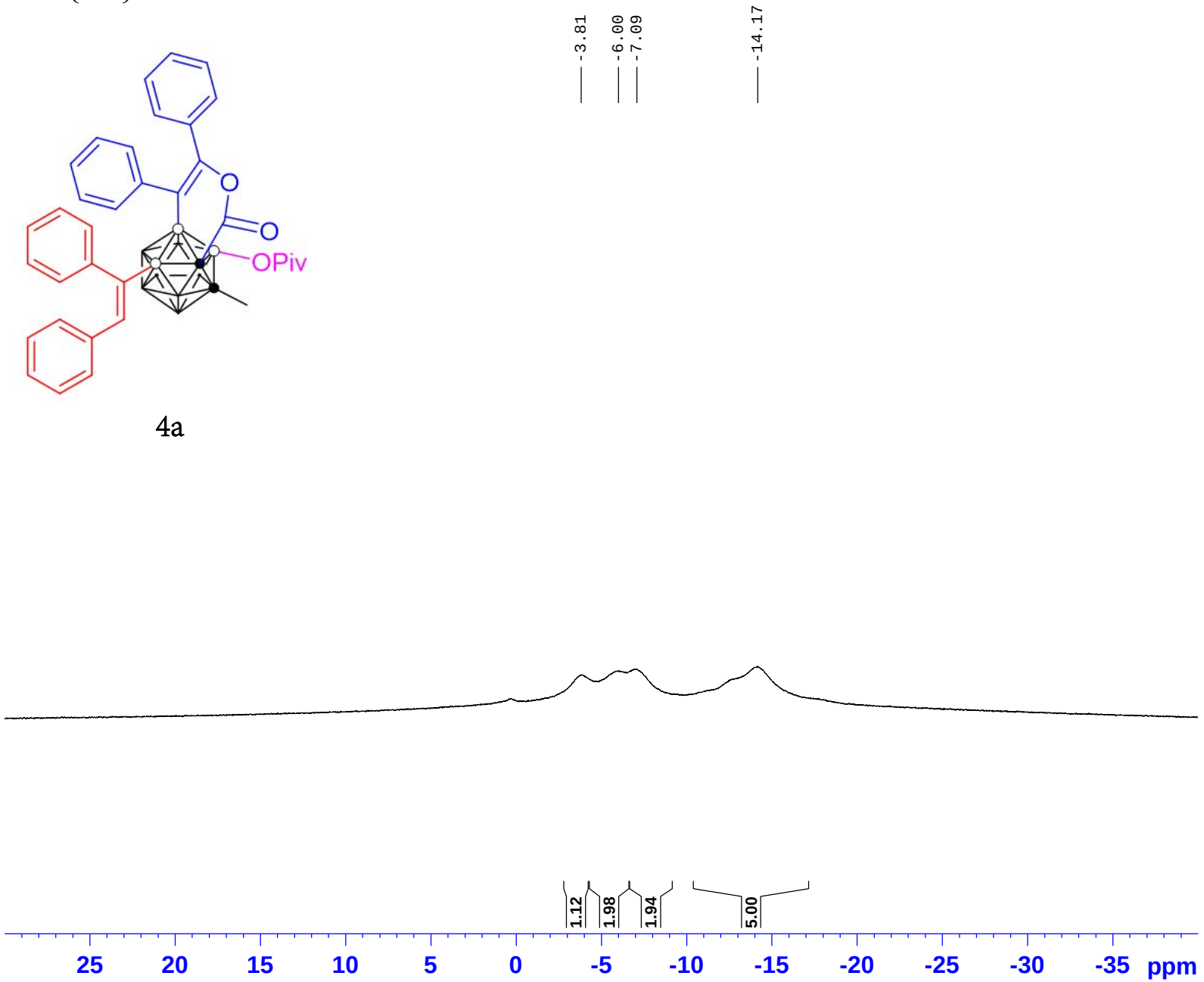
$^{11}B\{^{1}H\}$ NMR

4a



CY-B-A-81p

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EXPNO                  1
PROCNO                 1

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TD                 65536
SOLVENT            CDCl3
NS                    64
DS                     4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
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RG                   406
DW                20.800 usec
DE                 6.50 usec
TE                295.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1
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P1                 7.50 usec
PLW1        55.09999847 W
SFO2        400.2316009 MHz
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CPDPRG[2         waltz16
PCPD2             90.00 usec
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PLW12        0.27428001 W

F2 - Processing parameters
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LB                 1.00 Hz
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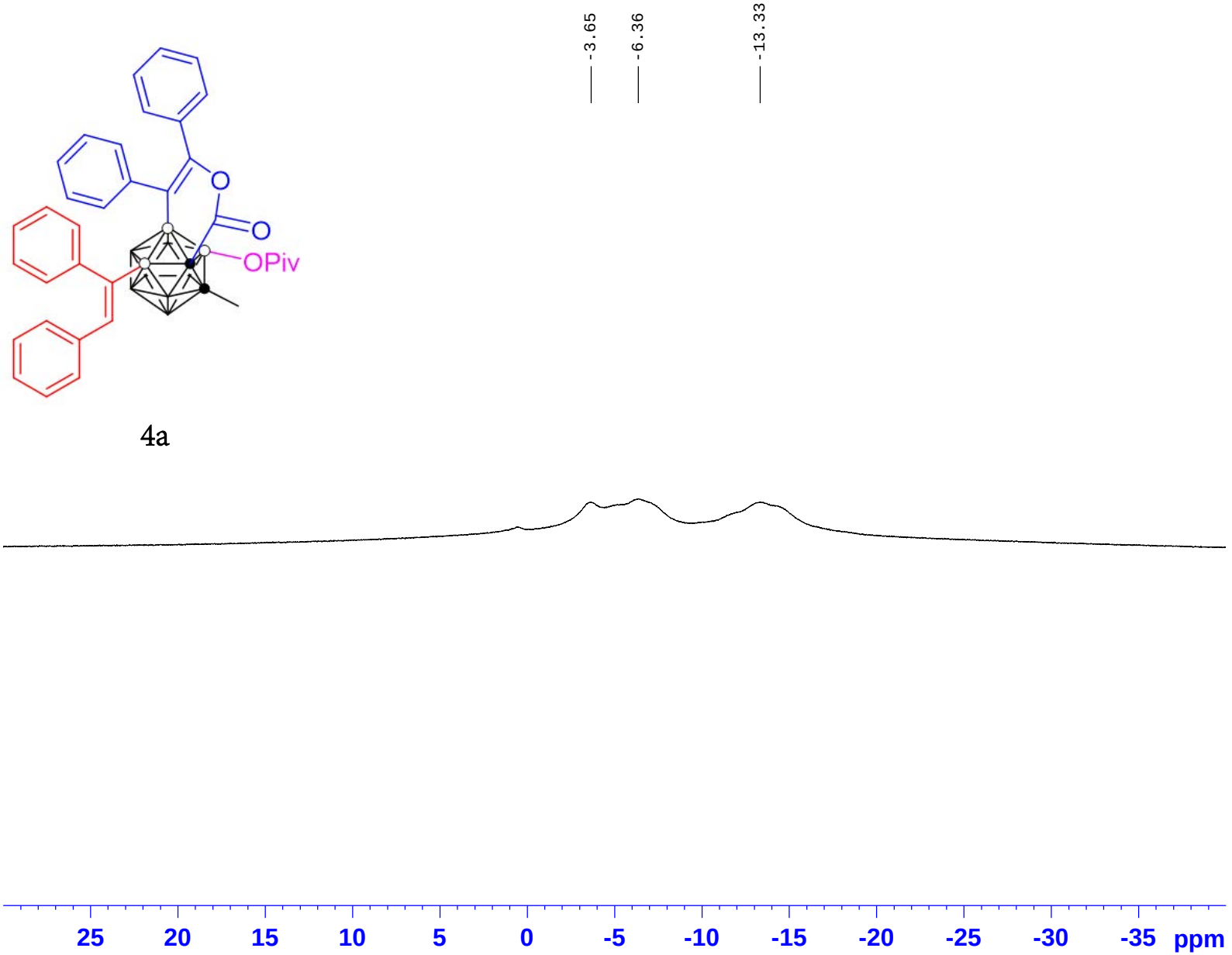
^{11}B NMR

4a



CY-B-A-81p-(C)

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PROCNO                1

F2 - Acquisition Parameters
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PULPROG             zg
TD                65536
SOLVENT           CDCl3
NS                   88
DS                    2
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                  362
DW                20.800 usec
DE                 6.50 usec
TE                294.7 K
D1           2.00000000 sec
TD0                   1
SFO1        128.4096890 MHz
NUC1                11B
P1                 7.50 usec
PLW1        55.09999847 W

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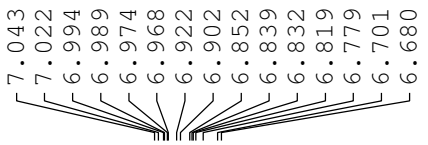

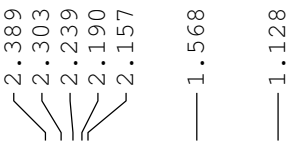

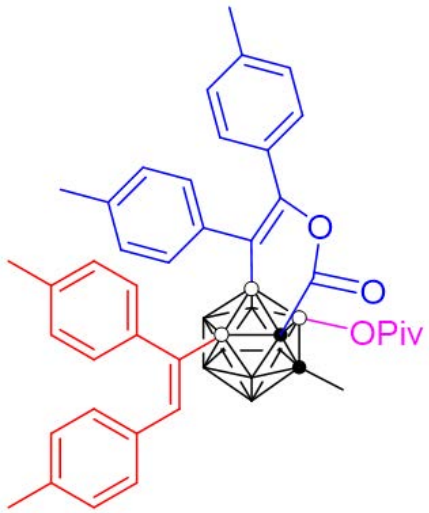

4b

```
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NAME          CY-H-A-96p
EXPNO                  1
PROCNO                 1

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Date_           20170318
Time               9.50 h
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PROBHD   Z824601_0021 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                  203
DW                62.400 usec
DE                 6.50 usec
TE                295.4 K
D1            1.00000000 sec
TD0                   1
SFO1         400.1324708 MHz
NUC1                 1H
P1                15.00 usec
PLW1          8.31000042 W

F2 - Processing parameters
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WDW                  EM
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LB                0.30 Hz
GB        0
PC                1.00
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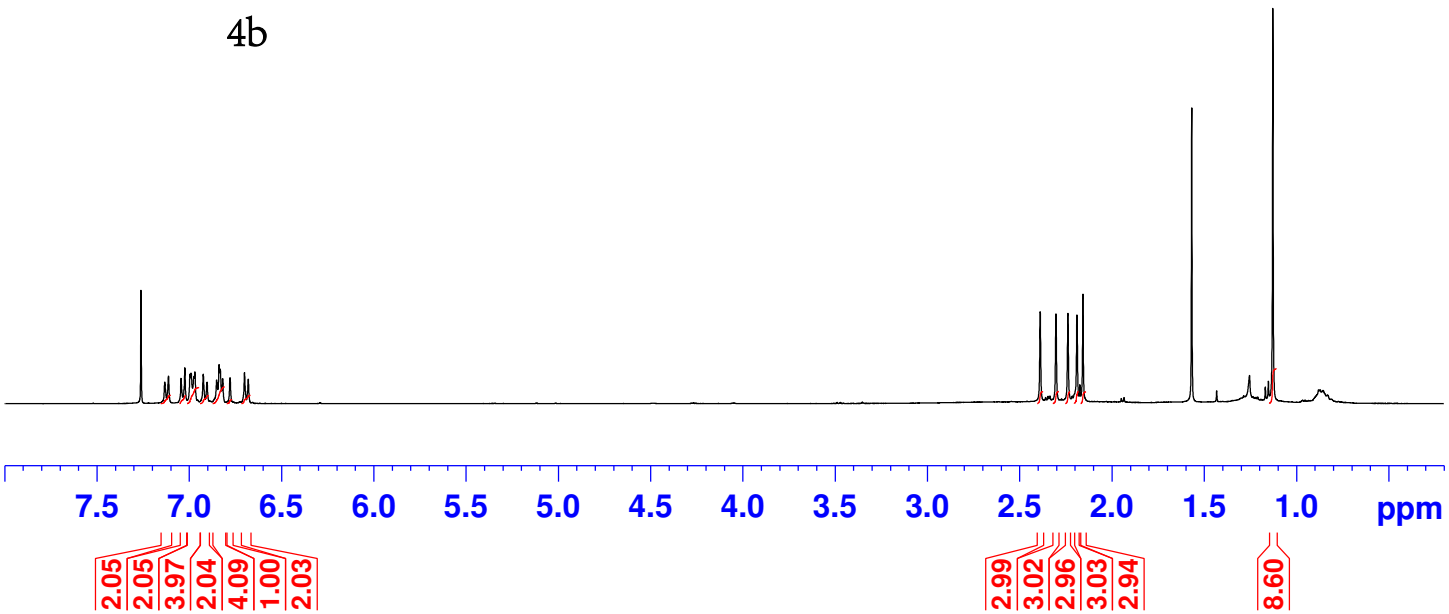

CY-C-A-96Rh-4-Me

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151.80
140.86
138.94
138.02
137.15
136.49
136.46
135.35
134.37
130.01
129.72
129.63
129.42
129.41
129.20
129.05
128.64
128.58
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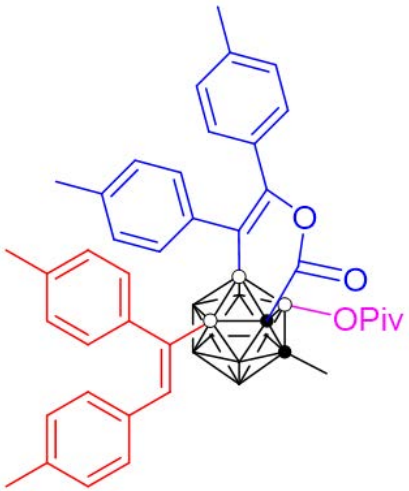
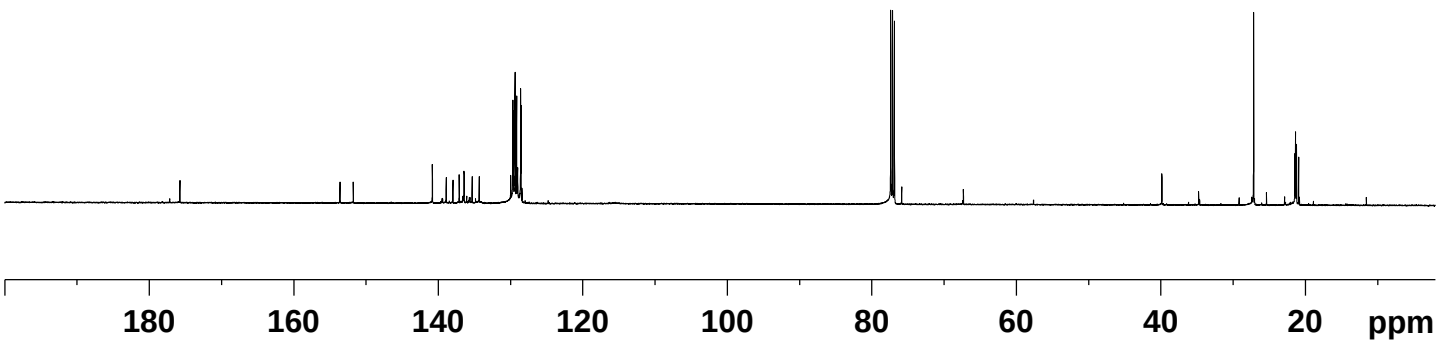
76.90
75.89
67.36
39.88
27.18
21.48
21.39
21.35
21.25
20.95



4b

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EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
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PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  4000
DS                     4
SWH            29761.904 Hz
FIDRES          0.908261 Hz
AQ             1.1010048 sec
RG                206.72
DW                16.800 usec
DE                 6.50 usec
TE                295.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1
SFO1         125.7703643 MHz
NUC1                13C
P1                 9.75 usec
PLW1         94.00000000 W
SFO2         500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2         25.00000000 W
PLW12         0.39063001 W
PLW13         0.19648001 W

F2 - Processing parameters
SI                 32768
SF           125.7577781 MHz
WDW                   EM
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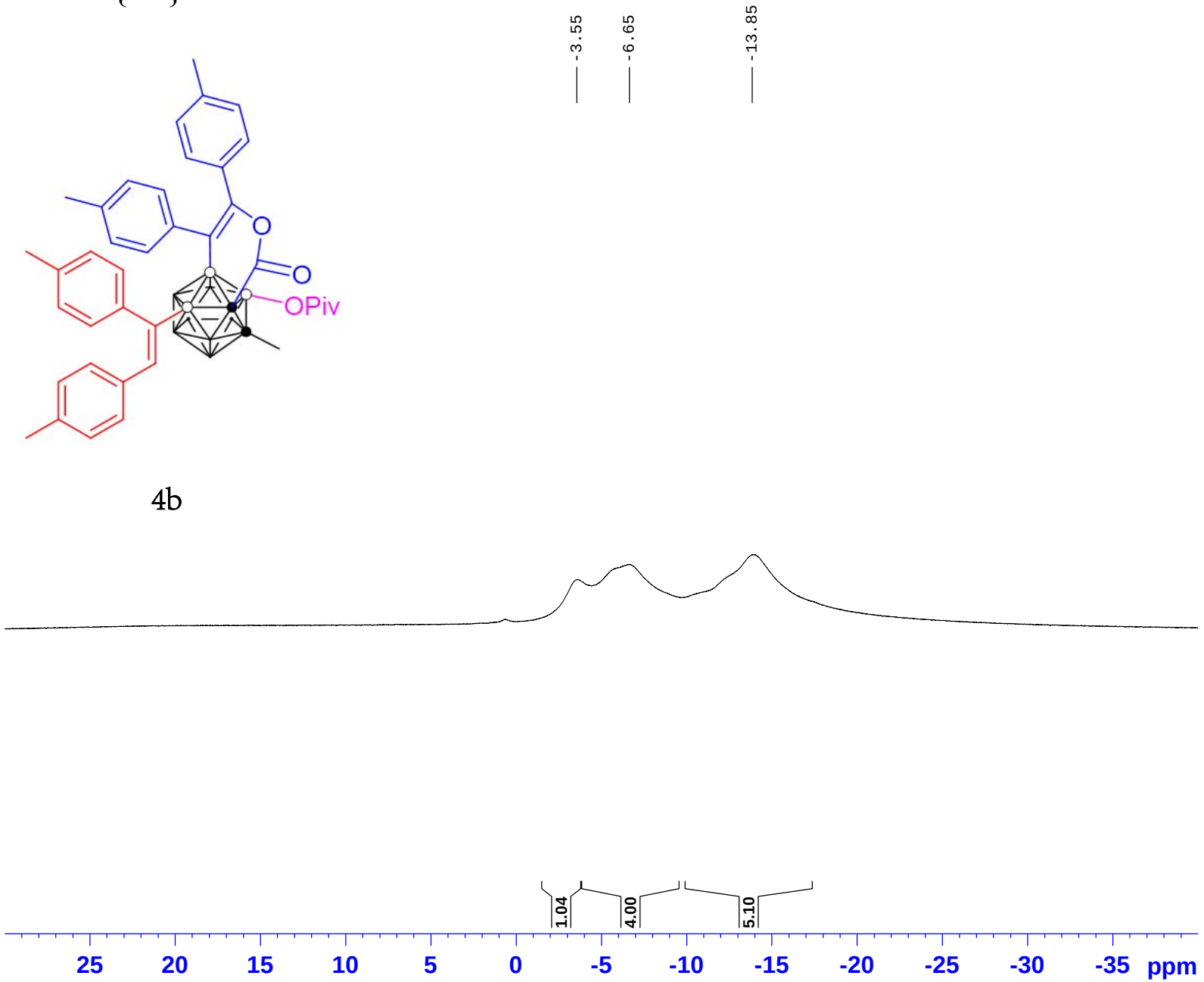
$^{11}B\{^{1}H\}$ NMR

CY-B-A-96Rh-4-Me

4b

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EXPNO                    1
PROCNO                   1

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TD                 32768
SOLVENT            CDCl3
NS                   160
DS                     4
SWH            24038.461 Hz
FIDRES          1.467191 Hz
AQ             0.6815744 sec
RG                206.72
DW                20.800 usec
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TE                295.1 K
D1             1.00000000 sec
D11            0.03000000 sec
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P1                16.00 usec
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SFO2         500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
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PLW13         0.19648001 W

F2 - Processing parameters
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PC                 1.40
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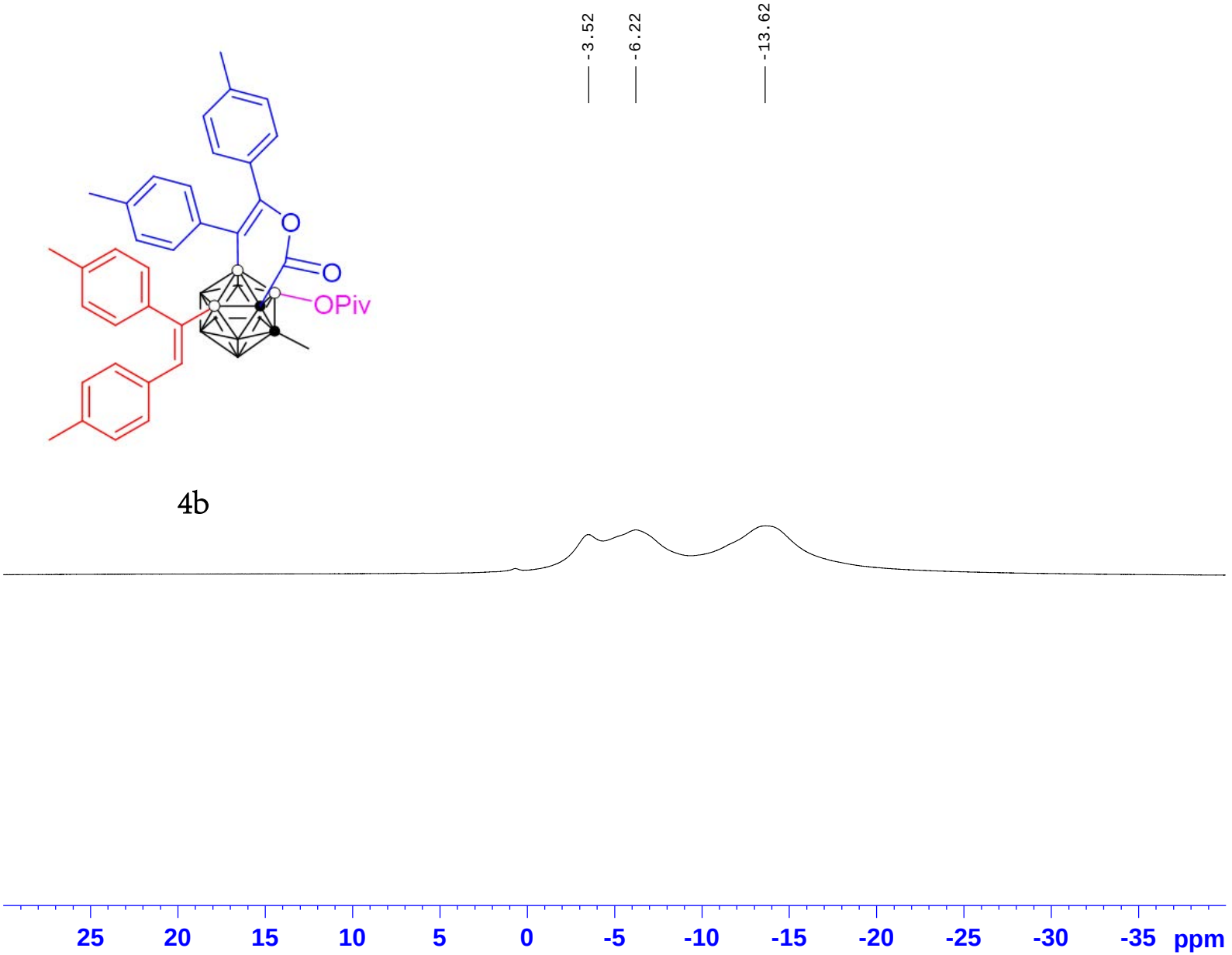
^{11}B NMR

CY-B-A-96Rh-4-Me-(C)

4b

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Current Data Parameters
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EXPNO                    1
PROCNO                   1

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Time                2.20 h
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PULPROG                 zg
TD                   32768
SOLVENT              CDCl3
NS                     320
DS                       4
SWH             24038.461 Hz
FIDRES           1.467191 Hz
AQ               0.6815744 sec
RG                  206.72
DW                  20.800 usec
DE                    6.50 usec
TE                   295.1 K
D1              1.00000000 sec
TD0                      1
SFO1          160.4615792 MHz
NUC1                  11B
P1                  16.00 usec
PLW1          50.00000000 W

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LB                   1.00 Hz
GB        0
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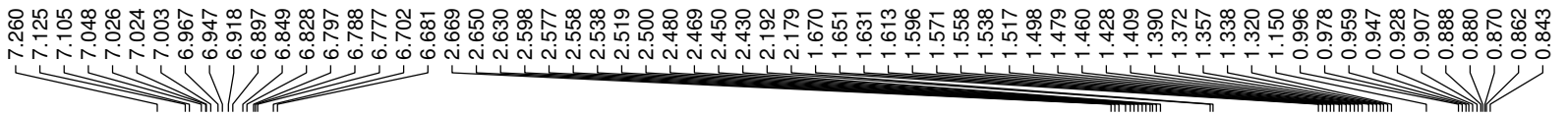
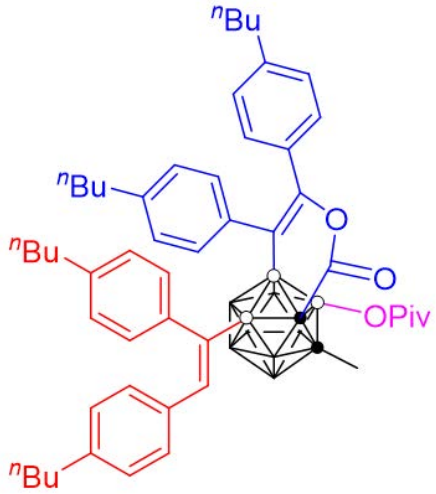
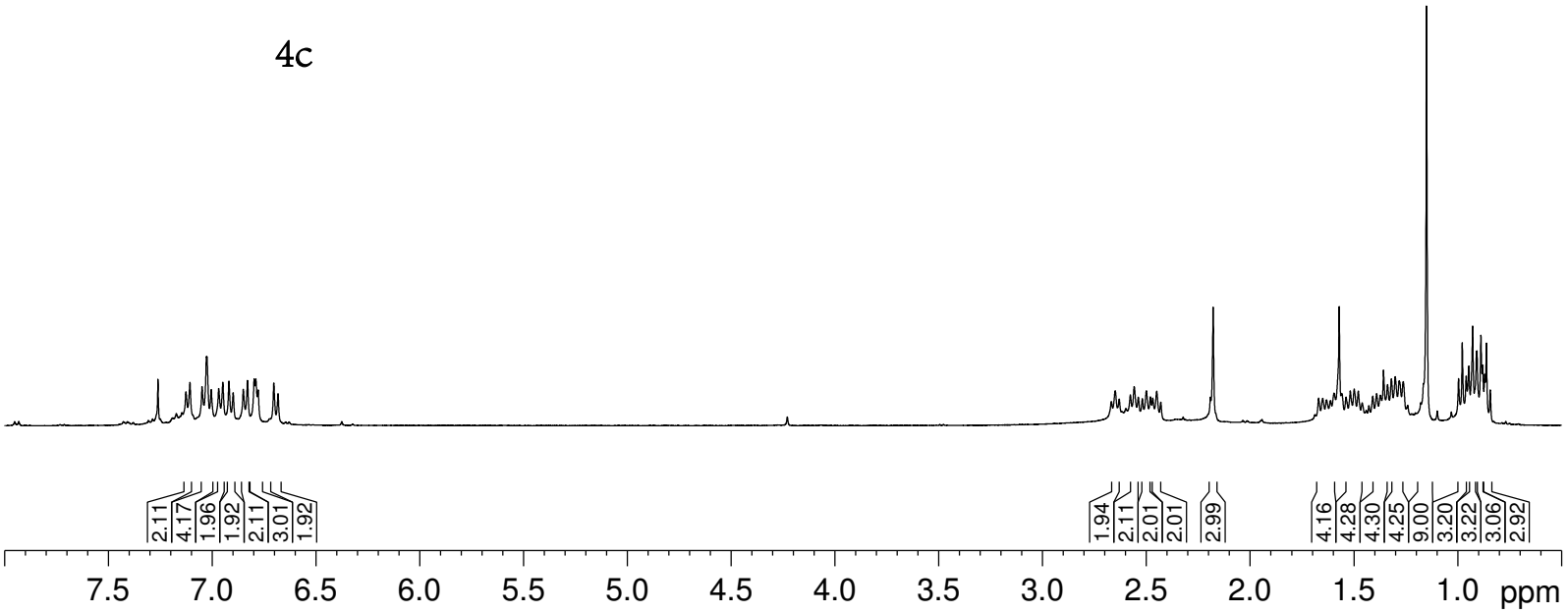


CY-H-A-116p-p



4c



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PROCNO 1

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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 12
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 50.8
DW 62.400 usec
DE 6.50 usec
TE 294.7 K
D1 1.00000000 sec
TD0 1
SFO1 400.2324714 MHz
NUC1 1H
P1 12.80 usec
PLW1 13.56000042 W

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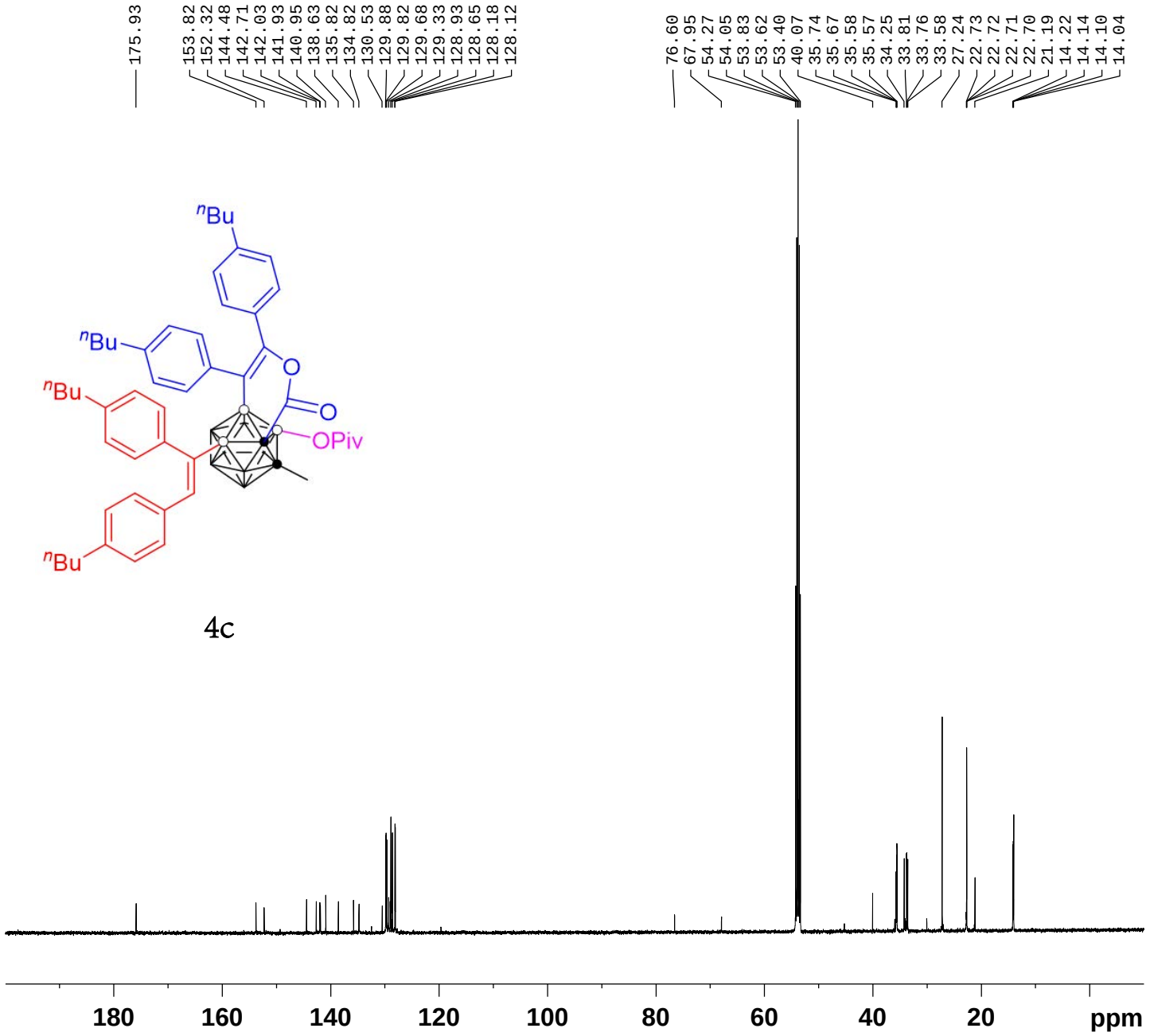
CY-C-A-116P-Rh

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Current Data Parameters
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PROCNO                 1

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PULPROG           zgpg30
TD                65536
SOLVENT           CD2Cl2
NS                  2100
DS                     4
SWH           29761.904 Hz
FIDRES         0.908261 Hz
AQ            1.1010048 sec
RG               206.72
DW               16.800 usec
DE                6.50 usec
TE               295.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1
SFO1        125.7703643 MHz
NUC1                13C
P1                 9.75 usec
PLW1        94.00000000 W
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        25.00000000 W
PLW12        0.39063001 W
PLW13        0.19648001 W

F2 - Processing parameters
SI                 32768
SF          125.7577393 MHz
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SSB       0
LB                1.00 Hz
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PC                1.40
```
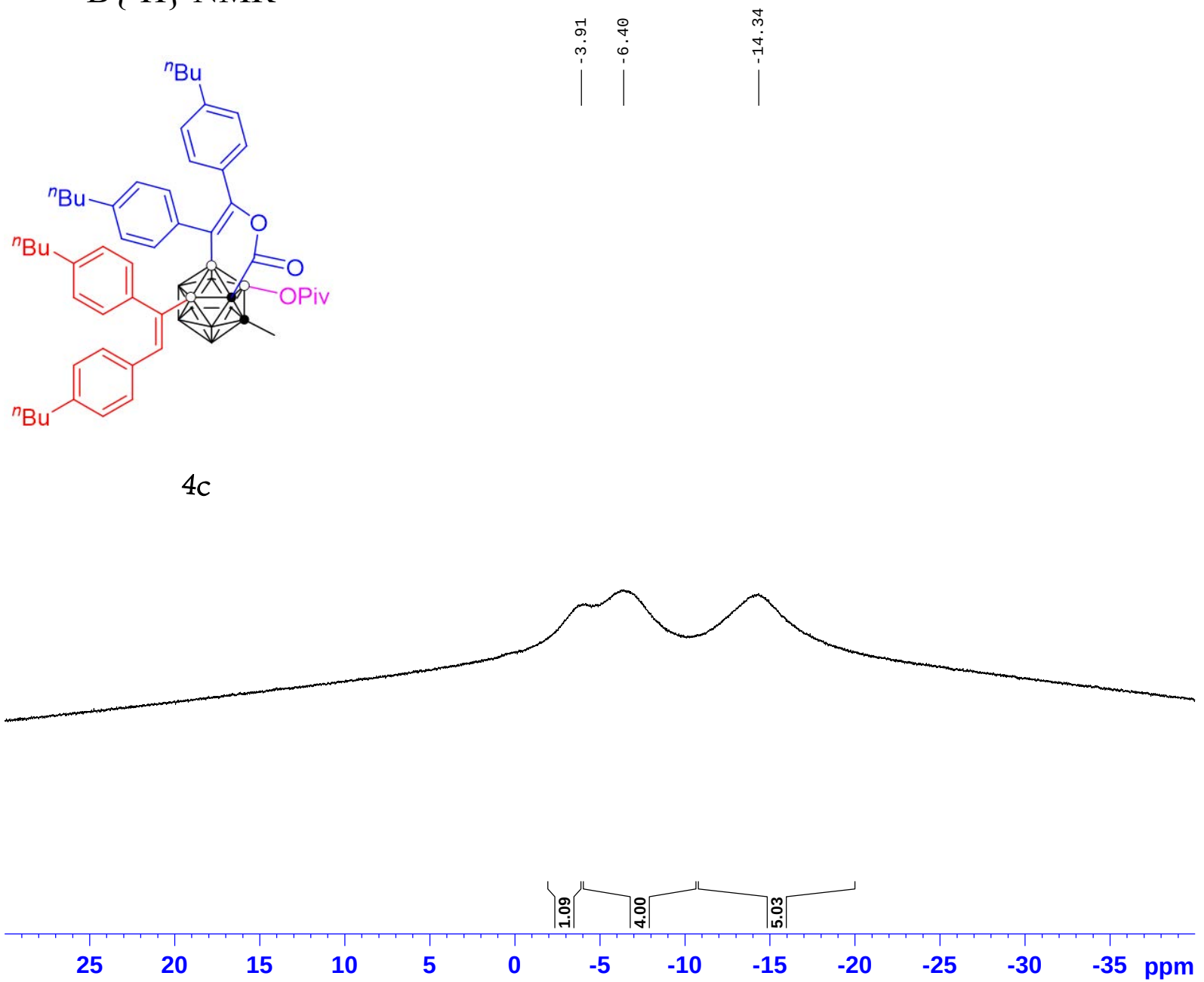

$^{11}B\{^{1}H\}$ NMR

4c



CY-B-A-116p-p

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Current Data Parameters
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EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20170825
Time             21.18 h
INSTRUM            spect
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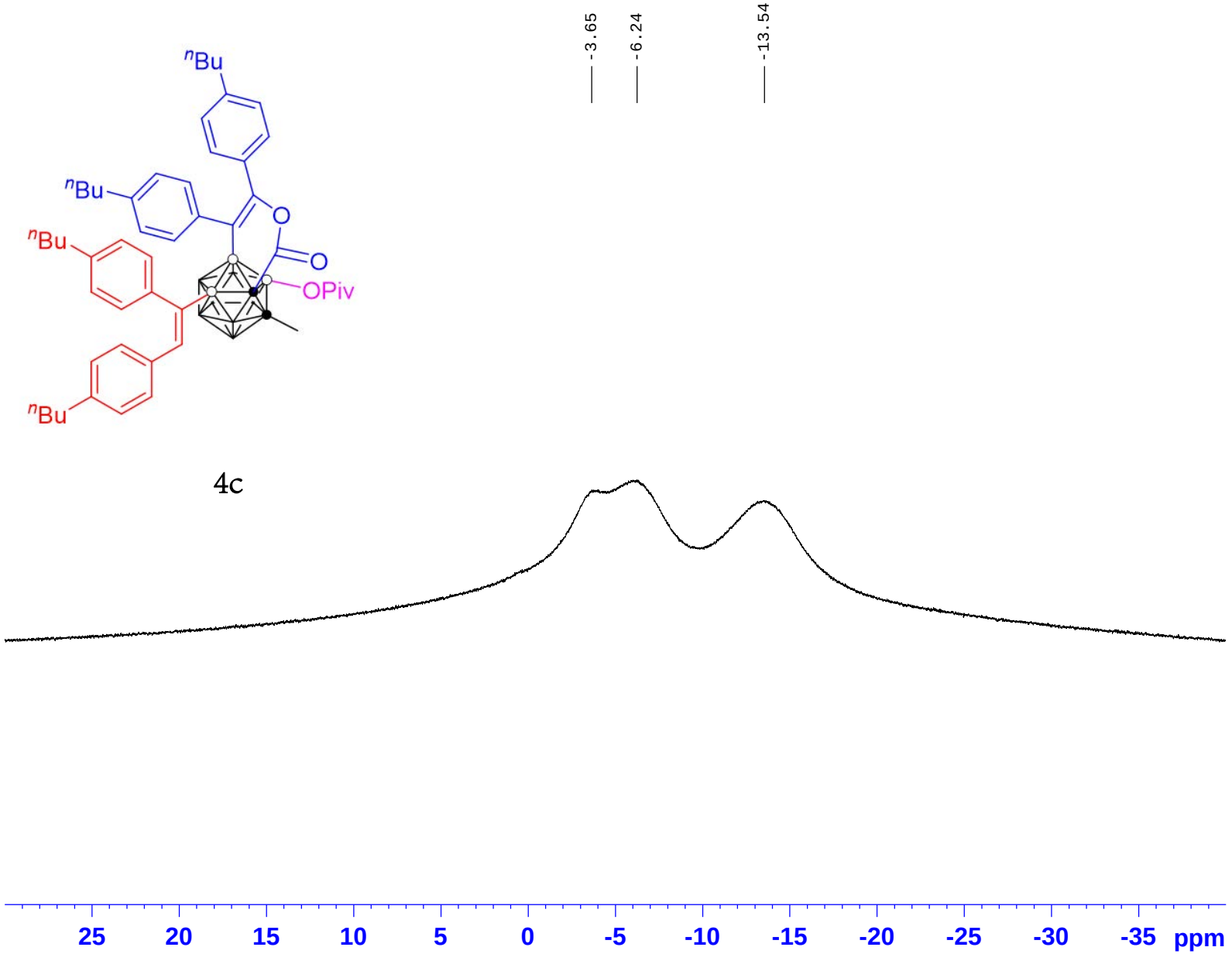
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                   64
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                  406
DW                20.800 usec
DE                 6.50 usec
TE                295.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        128.4096890 MHz
NUC1                11B
P1                 7.50 usec
PLW1        55.09999847 W
SFO2        400.2316009 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2        13.56000042 W
PLW12        0.27428001 W

F2 - Processing parameters
SI                 32768
SF          128.4097615 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{11}B NMR

4c



CY-B-A-116p-p-(C)

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Current Data Parameters
NAME      CY-B-A-116p-p-(C)
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
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Time                21.24 h
INSTRUM             spect
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PULPROG                 zg
TD                   65536
SOLVENT              CDCl3
NS                     128
DS                       2
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ              1.3631488 sec
RG                     406
DW                  20.800 usec
DE                   6.50 usec
TE                  294.8 K
D1             2.00000000 sec
TD0                      1
SFO1          128.4096890 MHz
NUC1                  11B
P1                   7.50 usec
PLW1          55.09999847 W

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WDW                    EM
SSB       0
LB                   1.00 Hz
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PC                   1.40
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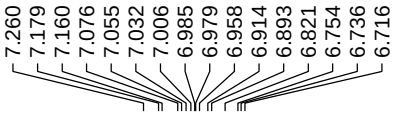
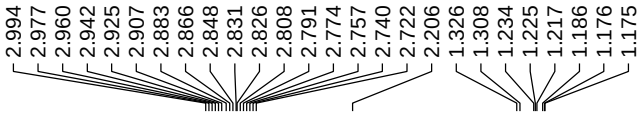
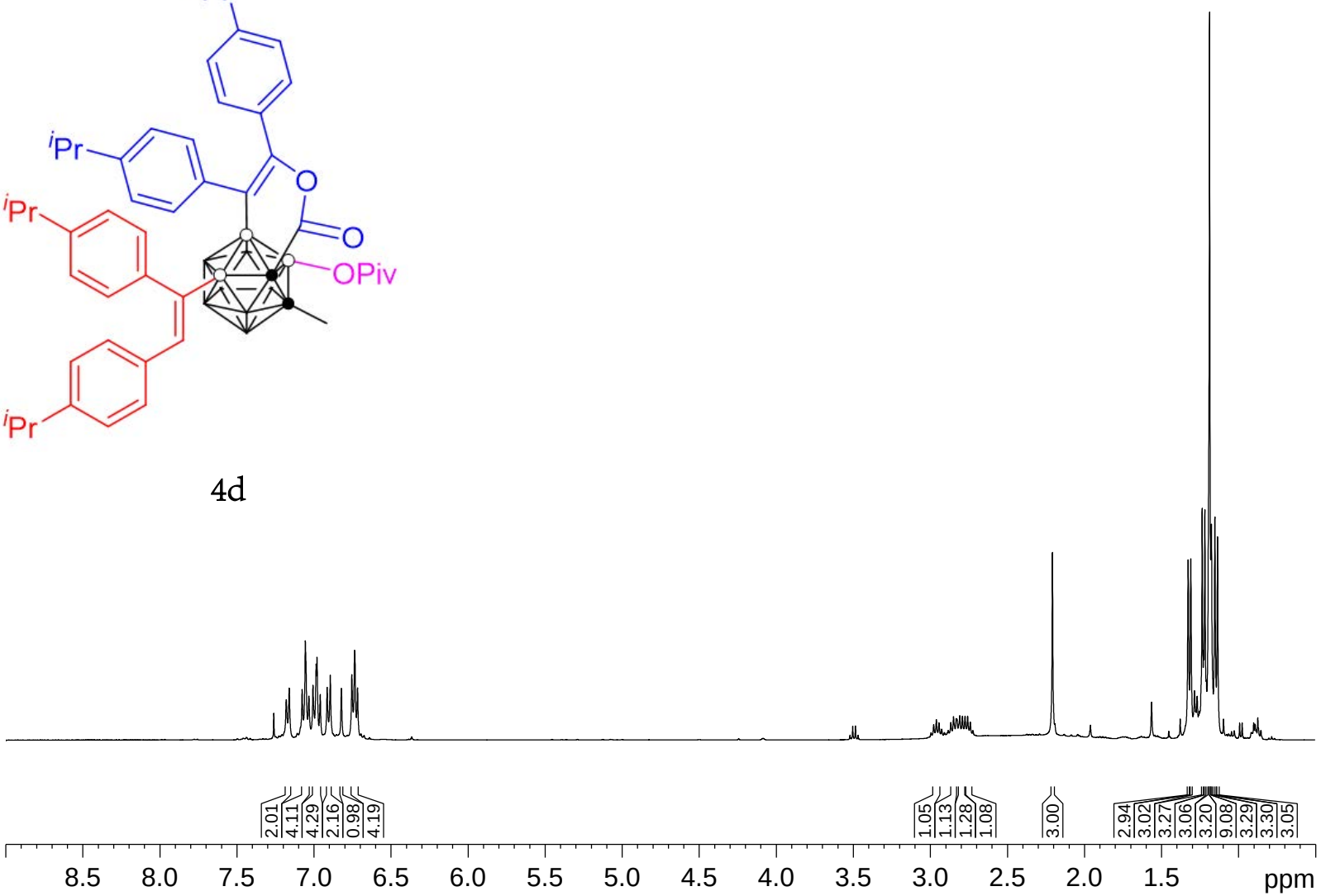




CY-H-A-91Rh

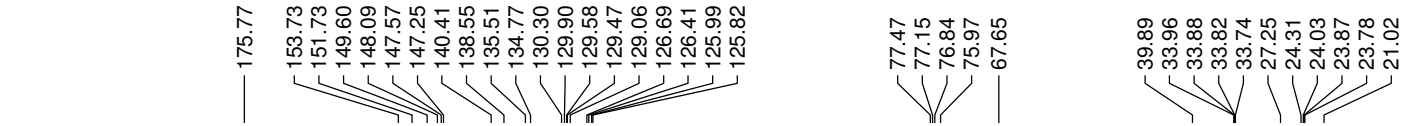
4d



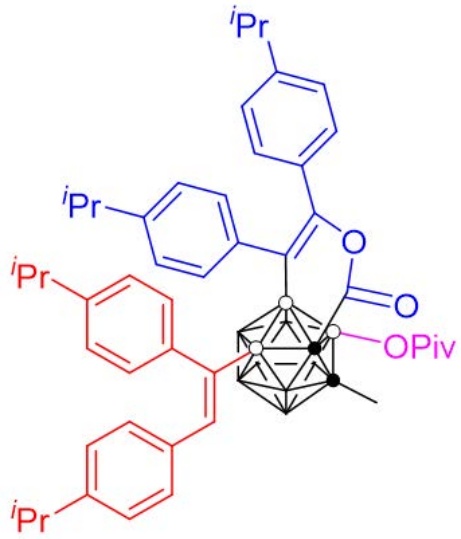
Current Data Parameters
NAME CY-H-A-91Rh
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170703
Time 17.11 h
INSTRUM spect
PROBHD Z108618_0257 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 12
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 32
DW 62.400 usec
DE 6.50 usec
TE 295.4 K
D1 1.00000000 sec
TD0 1
SFO1 400.2324714 MH
NUC1 1H
P1 12.80 usec
PLW1 13.56000042 W

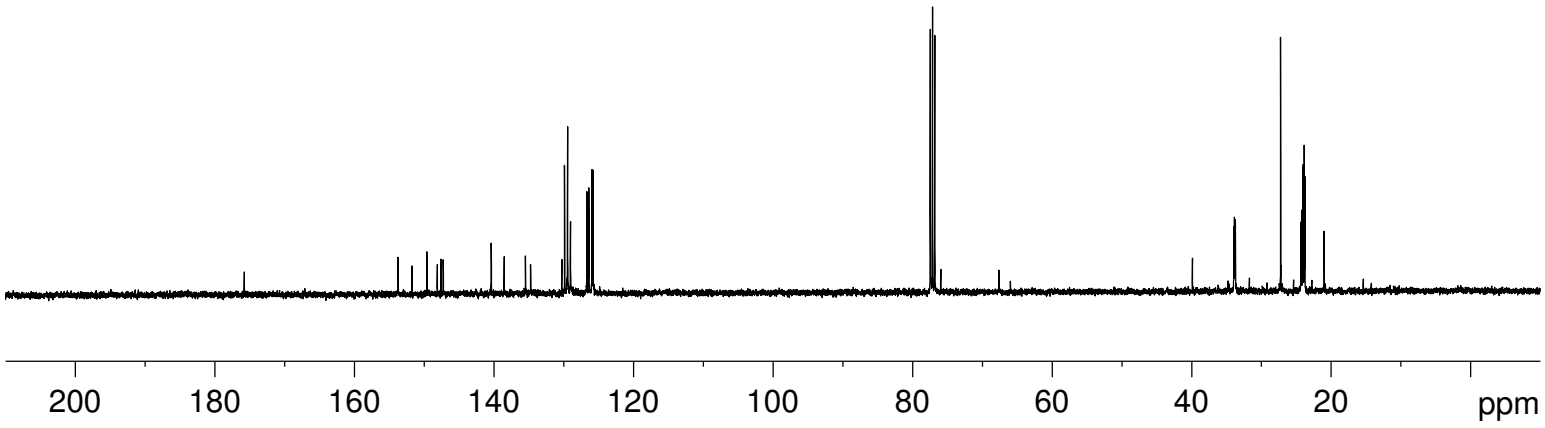
F2 - Processing parameters
SI 65536
SF 400.2300108 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



CY-C-A-91Rh



4d

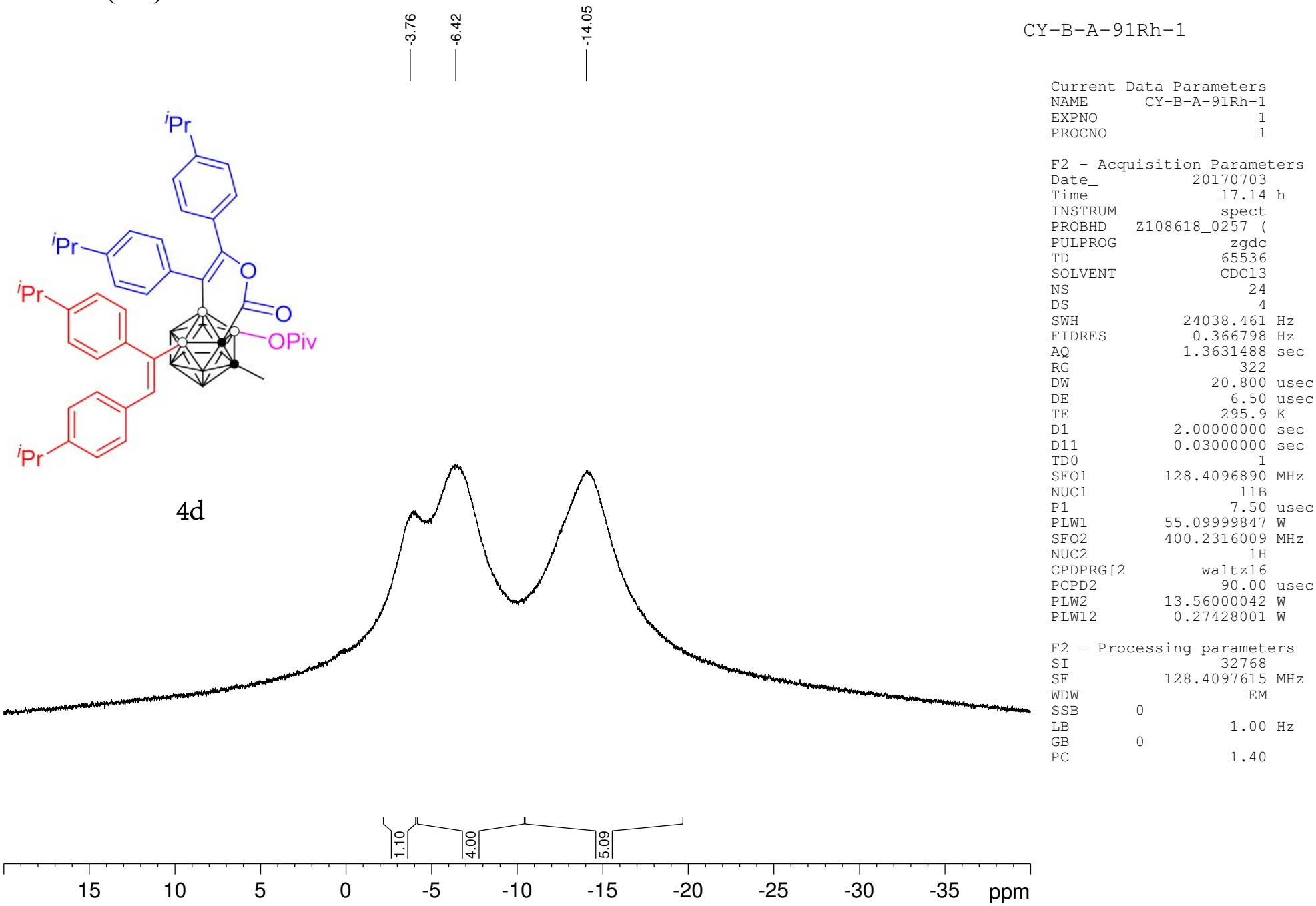



```
Current Data Parameters
NAME         CY-C-A-91Rh
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170703
Time              17.23 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  152
DS                    4
SWH           40760.871 Hz
FIDRES         0.621962 Hz
AQ             0.8039083 sec
RG                  203
DW                12.267 usec
DE                 6.50 usec
TE                295.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1        100.6479773 MHz
NUC1               13C
P1                9.50 usec
PLW1        55.34000015 W
SFO2        400.2316009 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        13.56000042 W
PLW12        0.27428001 W
PLW13        0.13796000 W

F2 - Processing parameters
SI                32768
SF          100.6379034 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

$^{11}B\{^{1}H\}$ NMR



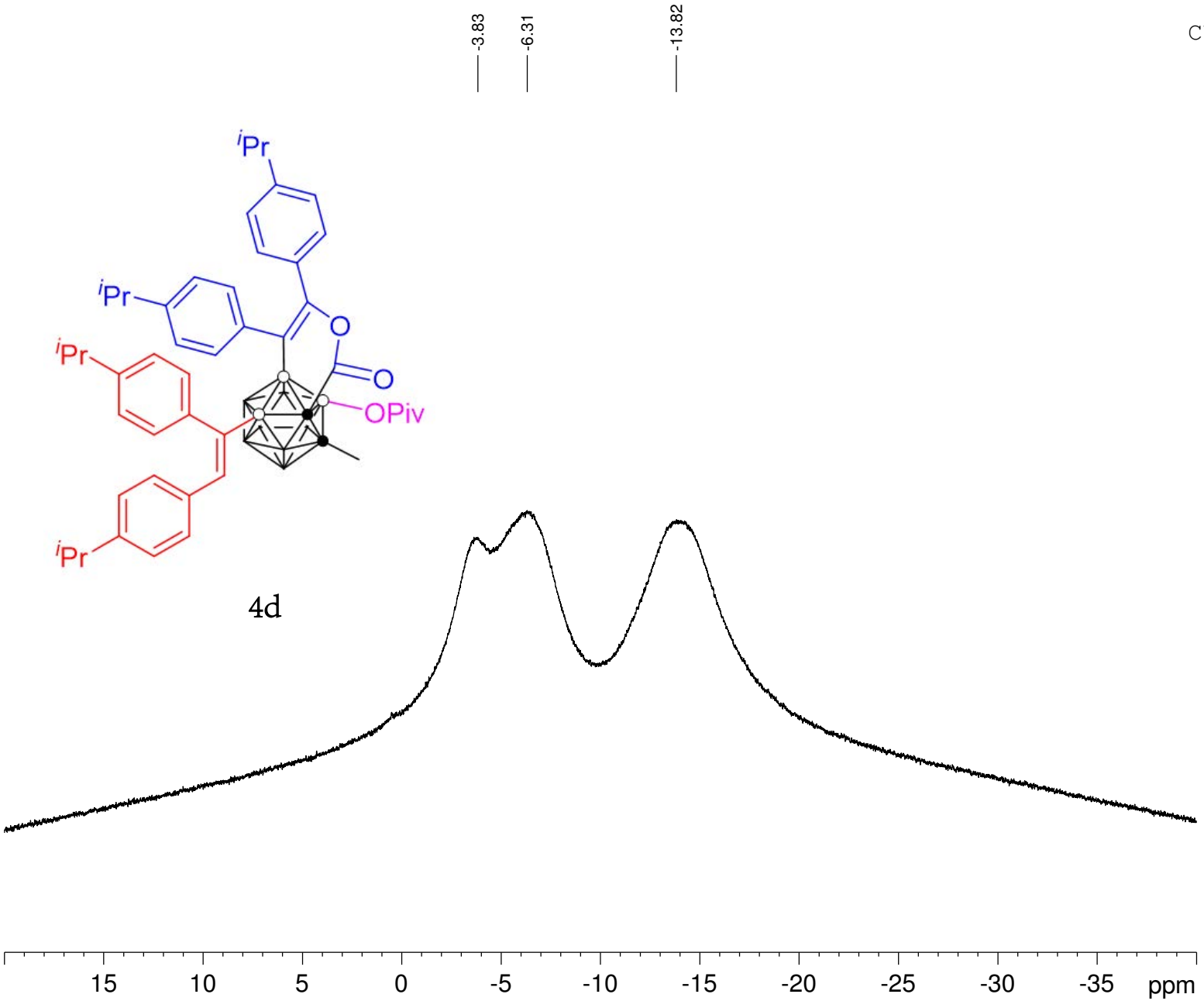
CY-B-A-91Rh-1

```
Current Data Parameters
NAME       CY-B-A-91Rh-1
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170703
Time               17.14 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                    24
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                   322
DW                20.800 usec
DE                 6.50 usec
TE                295.9 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1         128.4096890 MHz
NUC1                11B
P1                 7.50 usec
PLW1         55.09999847 W
SFO2         400.2316009 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         13.56000042 W
PLW12         0.27428001 W

F2 - Processing parameters
SI                 32768
SF           128.4097615 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{11}B NMR

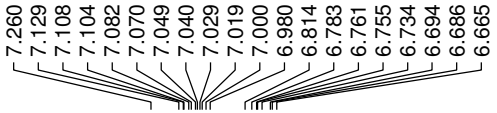


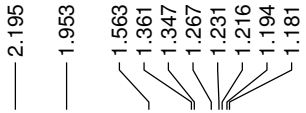
CY-B-A-91Rh-1-(C)

```
Current Data Parameters
NAME      CY-B-A-91Rh-1-(C)
EXPNO                     1
PROCNO                    1

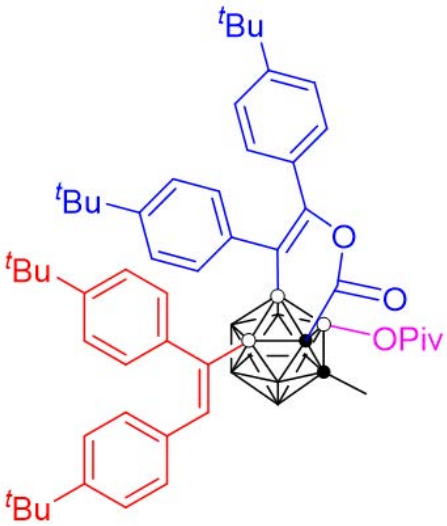
F2 - Acquisition Parameters
Date_           20170703
Time                17.16 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG                zg
TD                  65536
SOLVENT             CDCl3
NS                     32
DS                      2
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                    256
DW                20.800 usec
DE                 6.50 usec
TE                 295.4 K
D1            2.00000000 sec
TD0                     1
SFO1         128.4096890 MHz
NUC1                 11B
P1                 7.50 usec
PLW1         55.09999847 W

F2 - Processing parameters
SI                  32768
SF           128.4097430 MHz
WDW                    EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

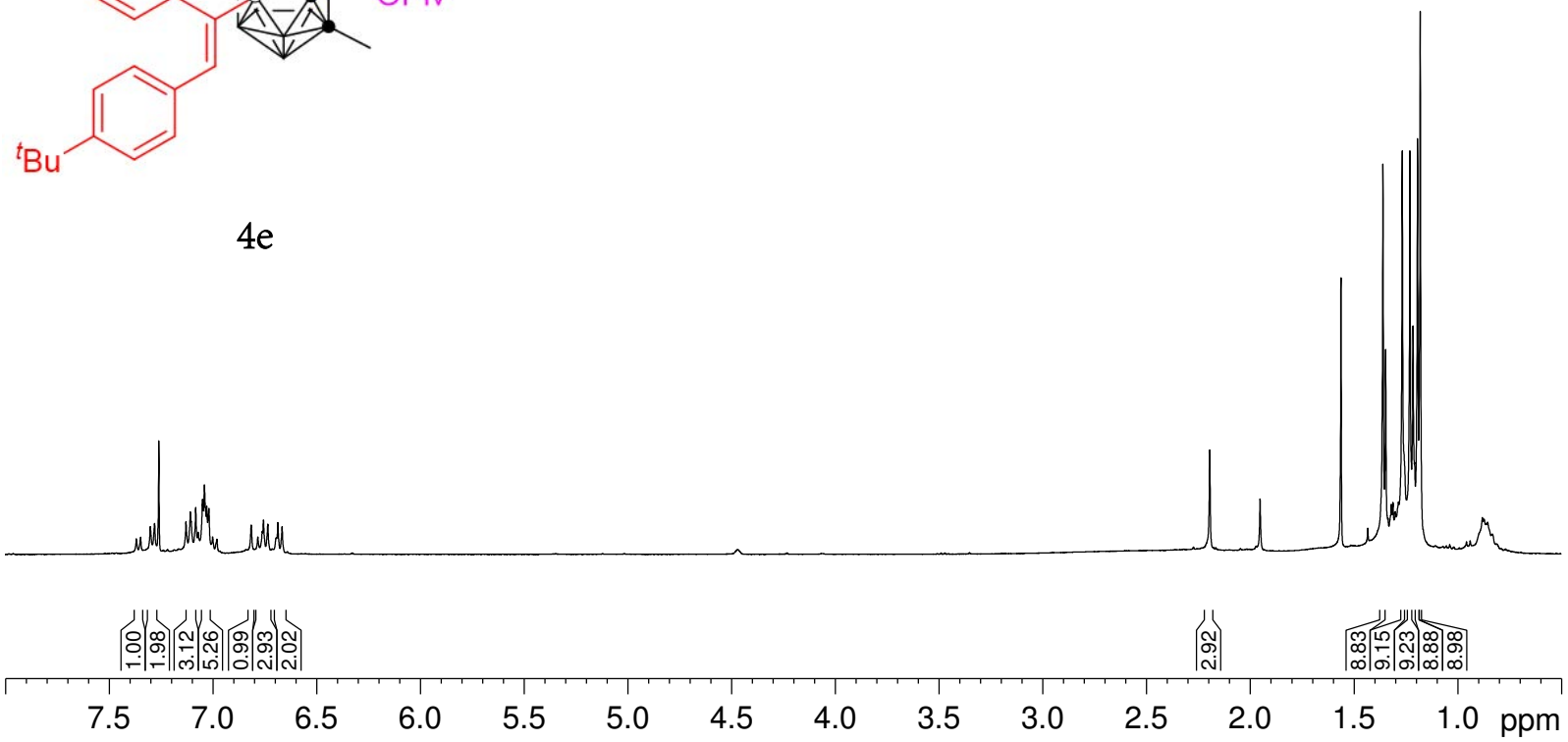




CY-H-A-95p



4e



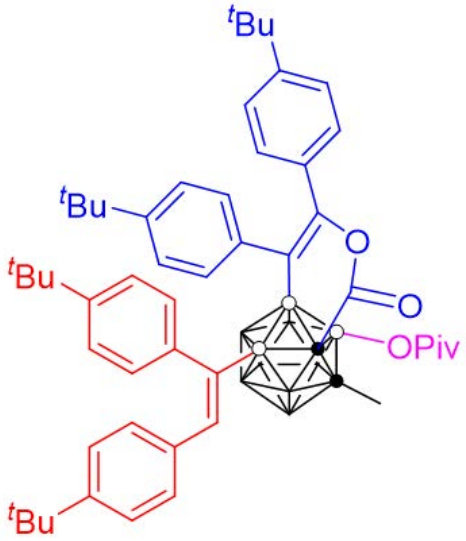
Current Data Parameters
NAME CY-H-A-95p
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170317
Time 18.54 h
INSTRUM spect
PROBHD Z108618_0257 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 12
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 71.8
DW 62.400 usec
DE 6.50 usec
TE 294.6 K
D1 1.00000000 sec
TD0 1
SFO1 400.2324714 MHz
NUC1 1H
P1 12.80 usec
PLW1 13.56000042 W

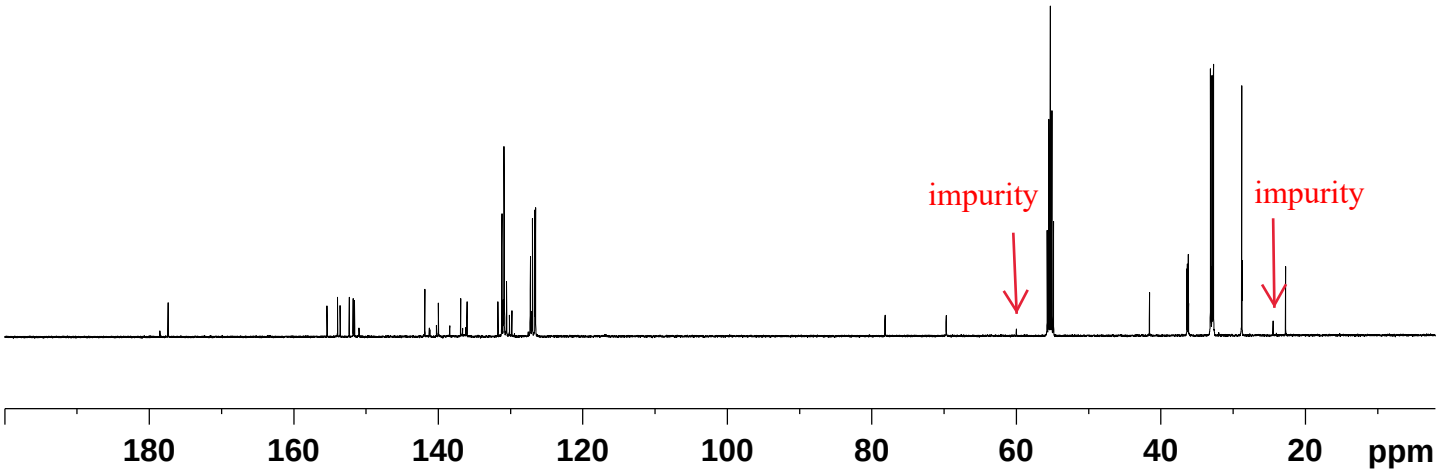
F2 - Processing parameters
SI 65536
SF 400.2300103 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

CY-C-A-95Rh-tBu

177.44
155.44
153.98
153.62
152.36
151.82
151.63
141.90
140.03
136.93
136.07
131.79
131.24
131.05
130.94
130.59
127.29
127.00
126.66
126.58
78.20
69.73
55.75
55.54
55.32
55.10
54.89
41.60
36.42
36.36
36.23
36.20
33.16
32.94
32.77
32.73
28.83
22.76



4e




```
Current Data Parameters
NAME      CY-C-A-95Rh-tBu
EXPNO                   1
PROCNO                  1

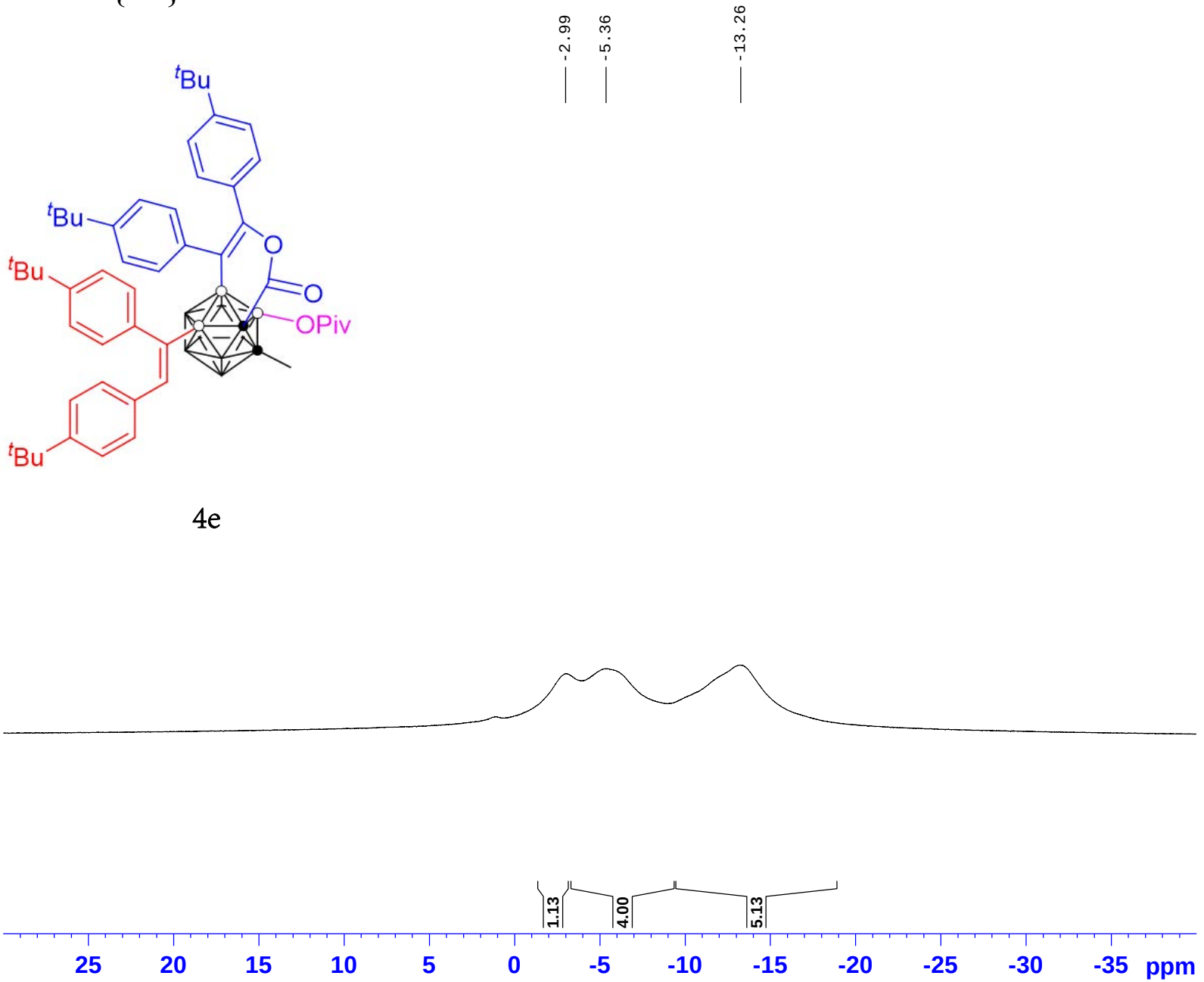
F2 - Acquisition Parameters
Date_           20200615
Time              23.27 h
INSTRUM             spect
PROBHD   Z119470_0283 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           29761.904 Hz
FIDRES         0.908261 Hz
AQ            1.1010048 sec
RG               206.72
DW               16.800 usec
DE                6.50 usec
TE               295.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        125.7703643 MHz
NUC1                13C
P1                 9.75 usec
PLW1        94.00000000 W
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        25.00000000 W
PLW12        0.39063001 W
PLW13        0.19648001 W

F2 - Processing parameters
SI                32768
SF          125.7577927 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

$^{11}B\{^{1}H\}$ NMR

CY-B-A-95Rh-1

4e

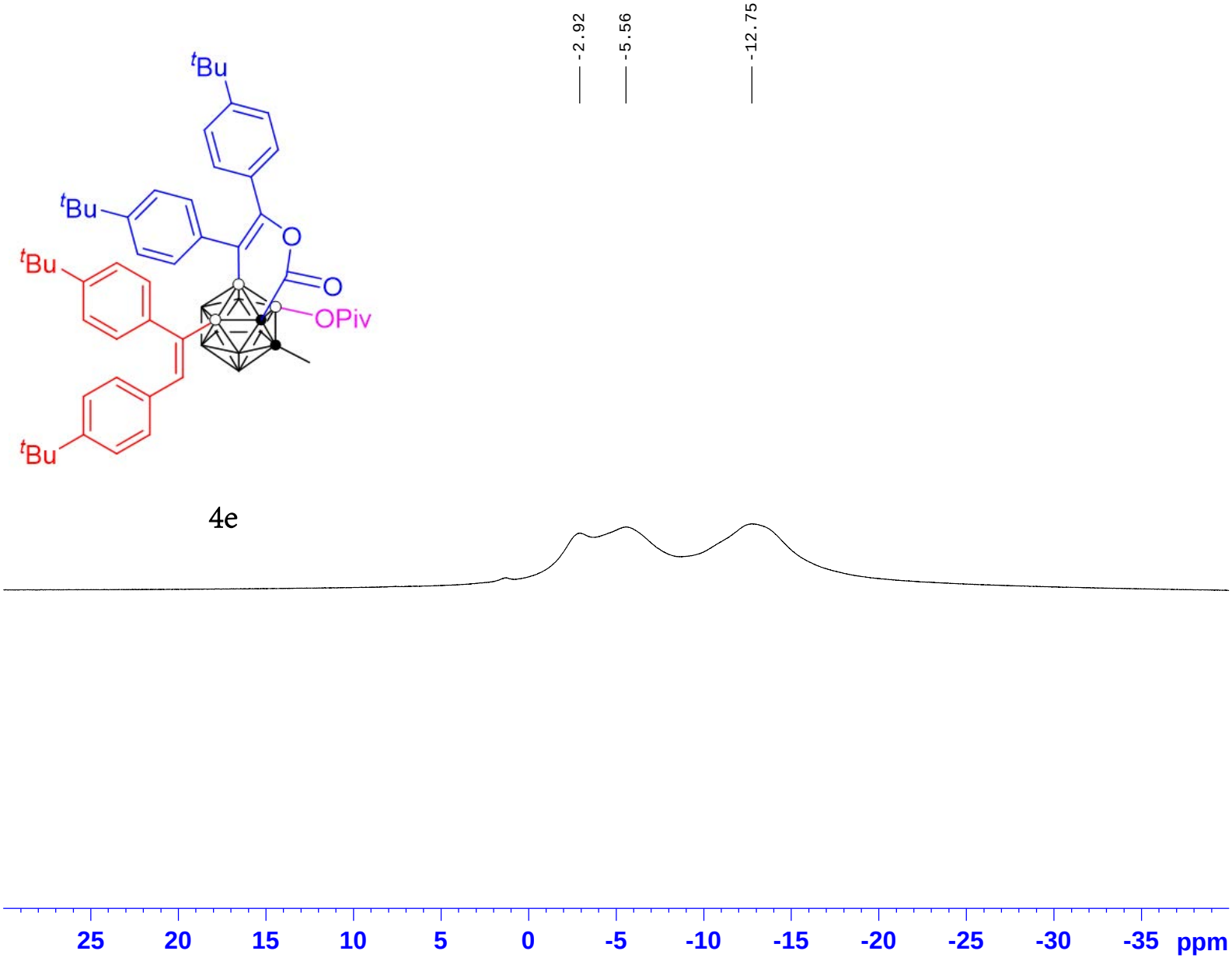



```
Current Data Parameters
NAME      CY-B-A-95Rh-1
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20200610
Time             18.59 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                   57
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                  406
DW                20.800 usec
DE                 6.50 usec
TE                295.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1
SFO1        128.4096890 MHz
NUC1               11B
P1                7.50 usec
PLW1        55.09999847 W
SFO2        400.2316009 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        13.56000042 W
PLW12        0.27428001 W

F2 - Processing parameters
SI                32768
SF          128.4097615 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

^{11}B NMR

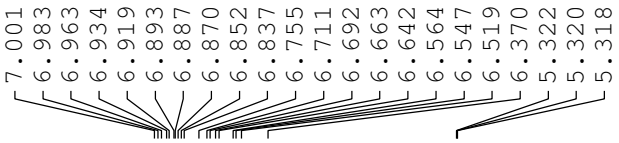


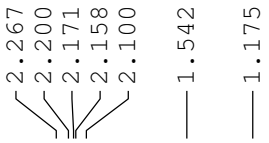
CY-B-A-95Rh-1-(C)

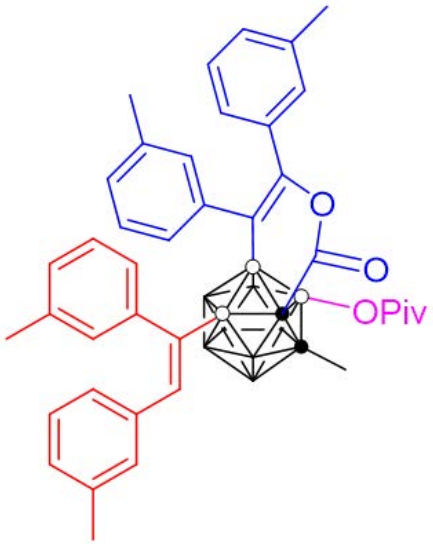
```
Current Data Parameters
NAME      CY-B-A-95Rh-1-(C)
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_            20200610
Time               19.07 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG                zg
TD                  65536
SOLVENT             CDCl3
NS                    126
DS                      2
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                    406
DW                20.800 usec
DE                 6.50 usec
TE                294.5 K
D1            2.00000000 sec
TD0                     1
SFO1         128.4096890 MHz
NUC1                  11B
P1                 7.50 usec
PLW1         55.09999847 W

F2 - Processing parameters
SI                  32768
SF           128.4097430 MHz
WDW                    EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```





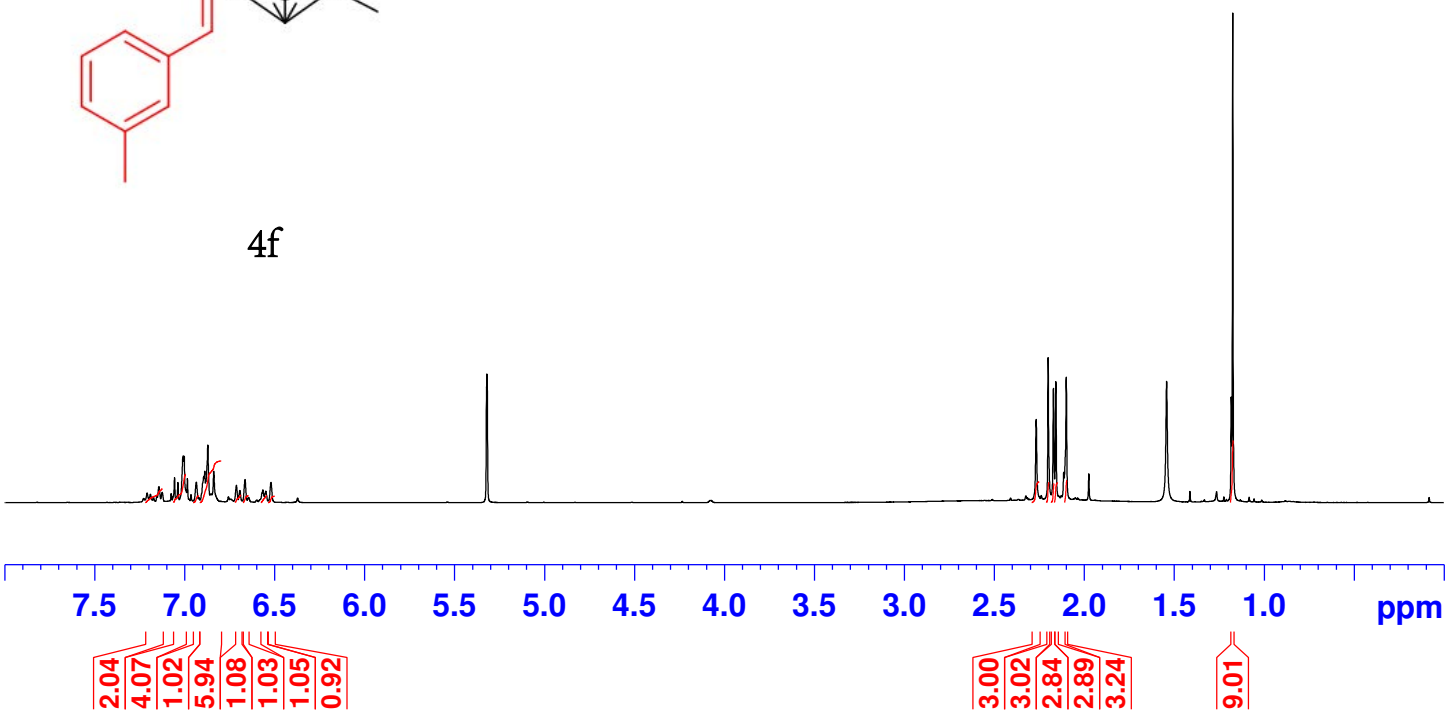


4f

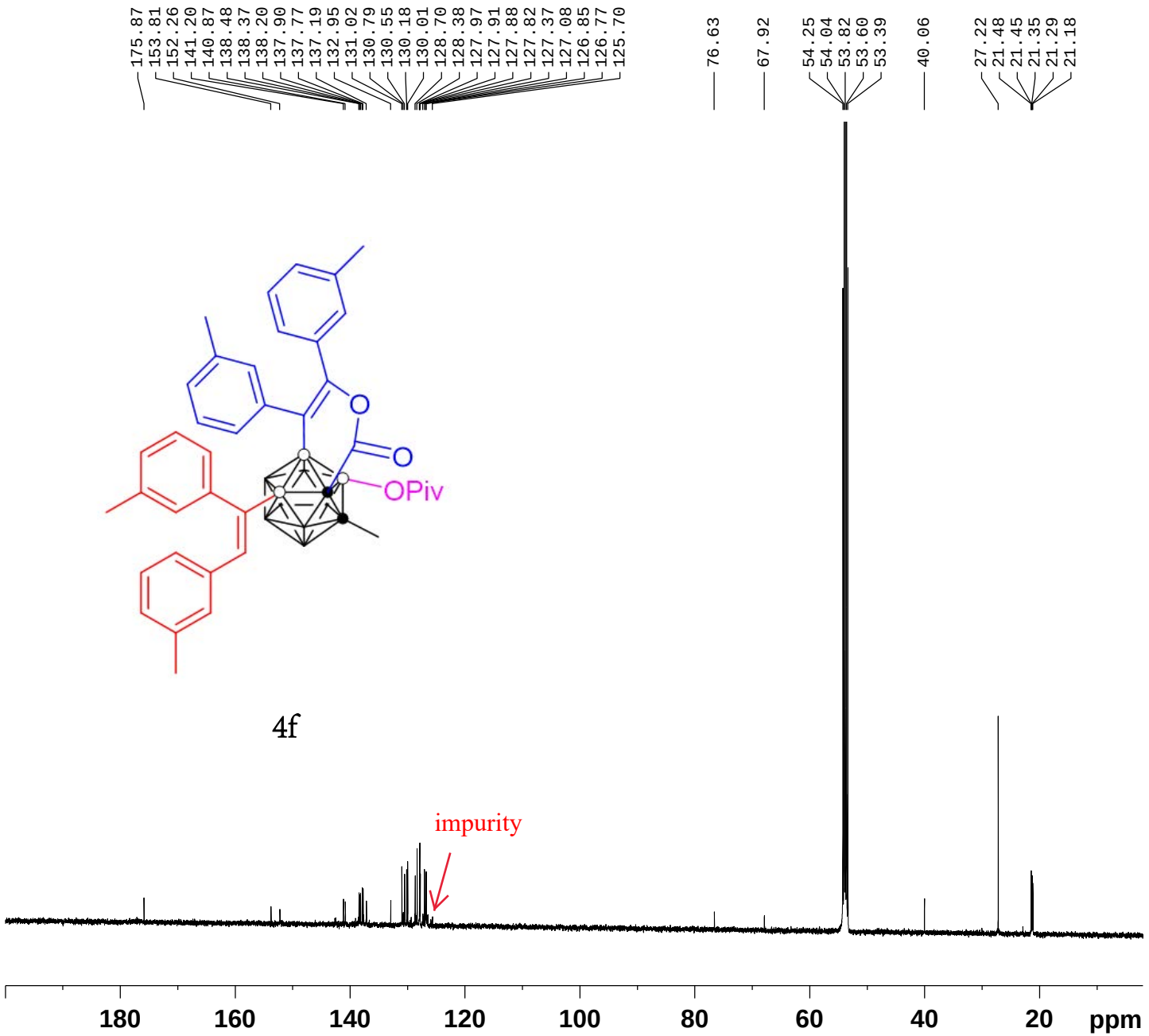
```
Current Data Parameters
NAME       CY-H-A-97P-Rh
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200624
Time              19.04 h
INSTRUM             spect
PROBHD   Z820201_0170 (
PULPROG             zg30
TD                 65536
SOLVENT          CD2Cl2
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ             4.0894465 sec
RG                   203
DW                62.400 usec
DE                 6.50 usec
TE                296.9 K
D1           1.00000000 sec
TD0                    1
SFO1        400.1324708 MHz
NUC1                  1H
P1                 6.75 usec
PLW1        13.17700005 W

F2 - Processing parameters
SI                 65536
SF          400.1300154 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



CY-C-A-97P-Rh



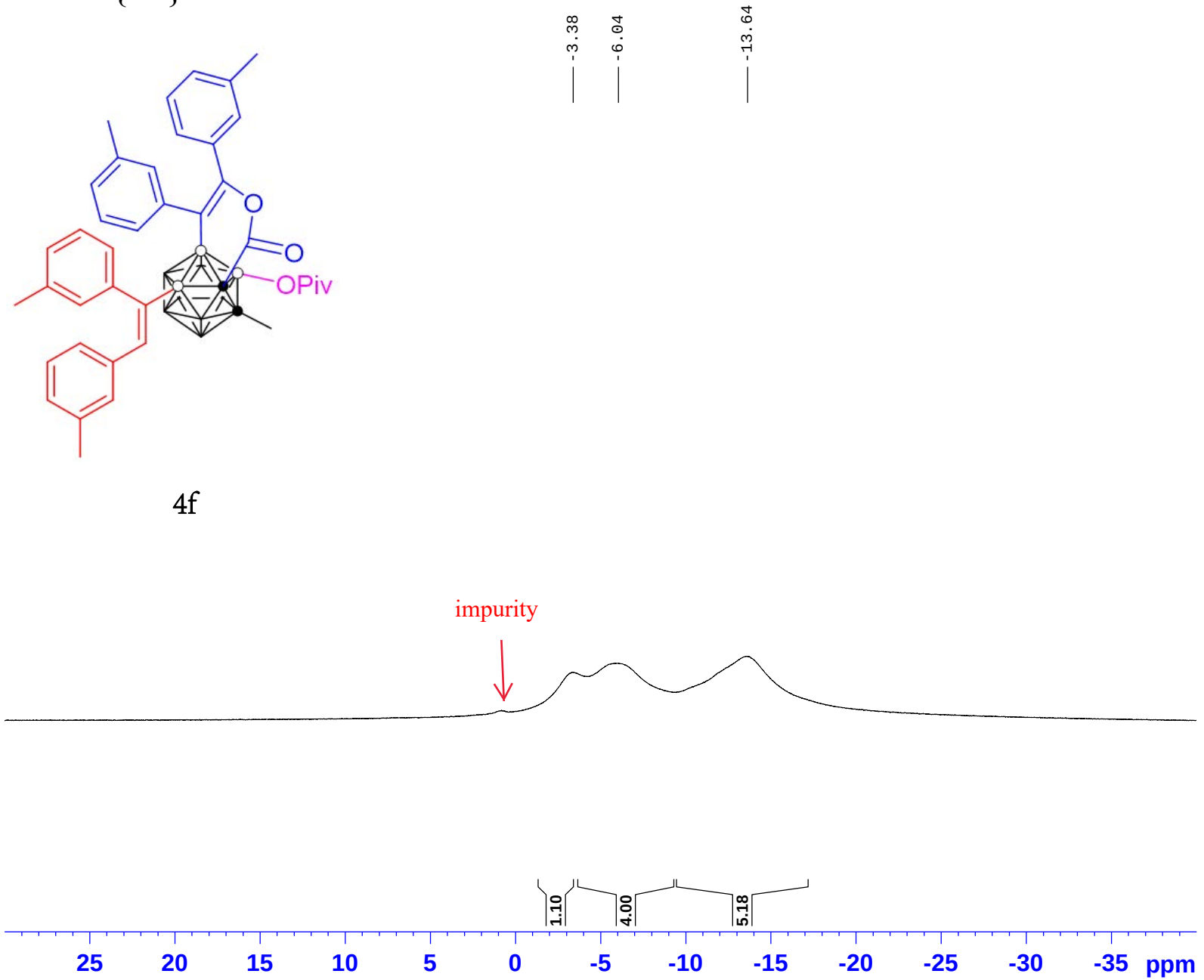

```
Current Data Parameters
NAME       CY-C-A-97P-Rh
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200630
Time               0.22 h
INSTRUM           spect
PROBHD   Z119470_0283 (
PULPROG          zgpg30
TD                65536
SOLVENT          CD2Cl2
NS                 3200
DS                    4
SWH           29761.904 Hz
FIDRES         0.908261 Hz
AQ            1.1010048 sec
RG               206.72
DW               16.800 usec
DE                6.50 usec
TE                295.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        125.7703643 MHz
NUC1               13C
P1                9.75 usec
PLW1        94.00000000 W
SFO2        500.1320005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2             80.00 usec
PLW2        25.00000000 W
PLW12        0.39063001 W
PLW13        0.19648001 W

F2 - Processing parameters
SI                32768
SF          125.7577417 MHz
WDW                  EM
SSB      0
LB                1.00 Hz
GB       0
PC                1.40
```

$^{11}B\{^{1}H\}$ NMR

4f



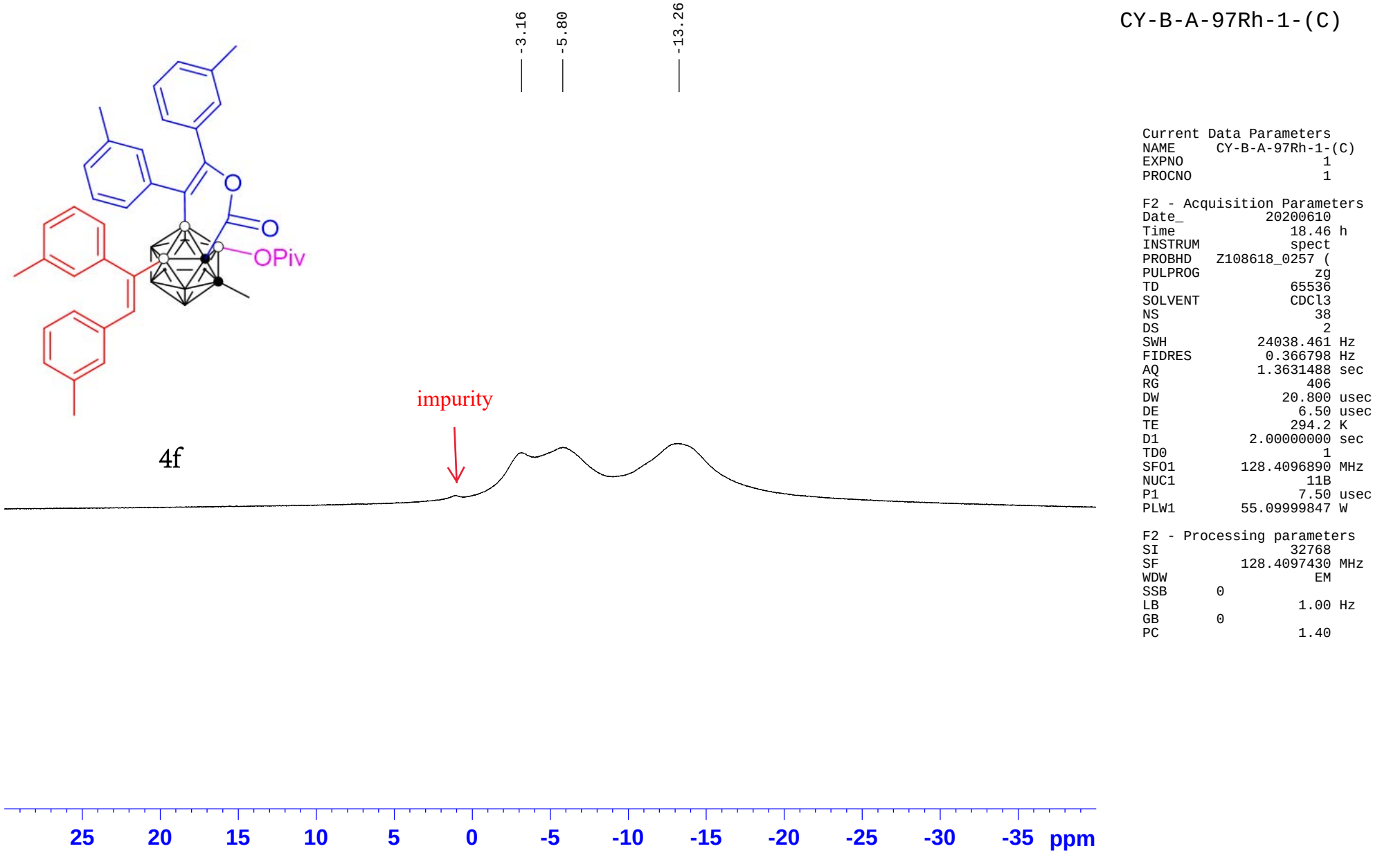
CY-B-A-97Rh-1

```
Current Data Parameters
NAME      CY-B-A-97Rh-1
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20200610
Time             18.39 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                   35
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                  406
DW               20.800 usec
DE                6.50 usec
TE               294.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1
SFO1        128.4096890 MHz
NUC1               11B
P1                7.50 usec
PLW1        55.09999847 W
SFO2        400.2316009 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        13.56000042 W
PLW12        0.27428001 W

F2 - Processing parameters
SI                32768
SF          128.4097615 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

^{11}B NMR

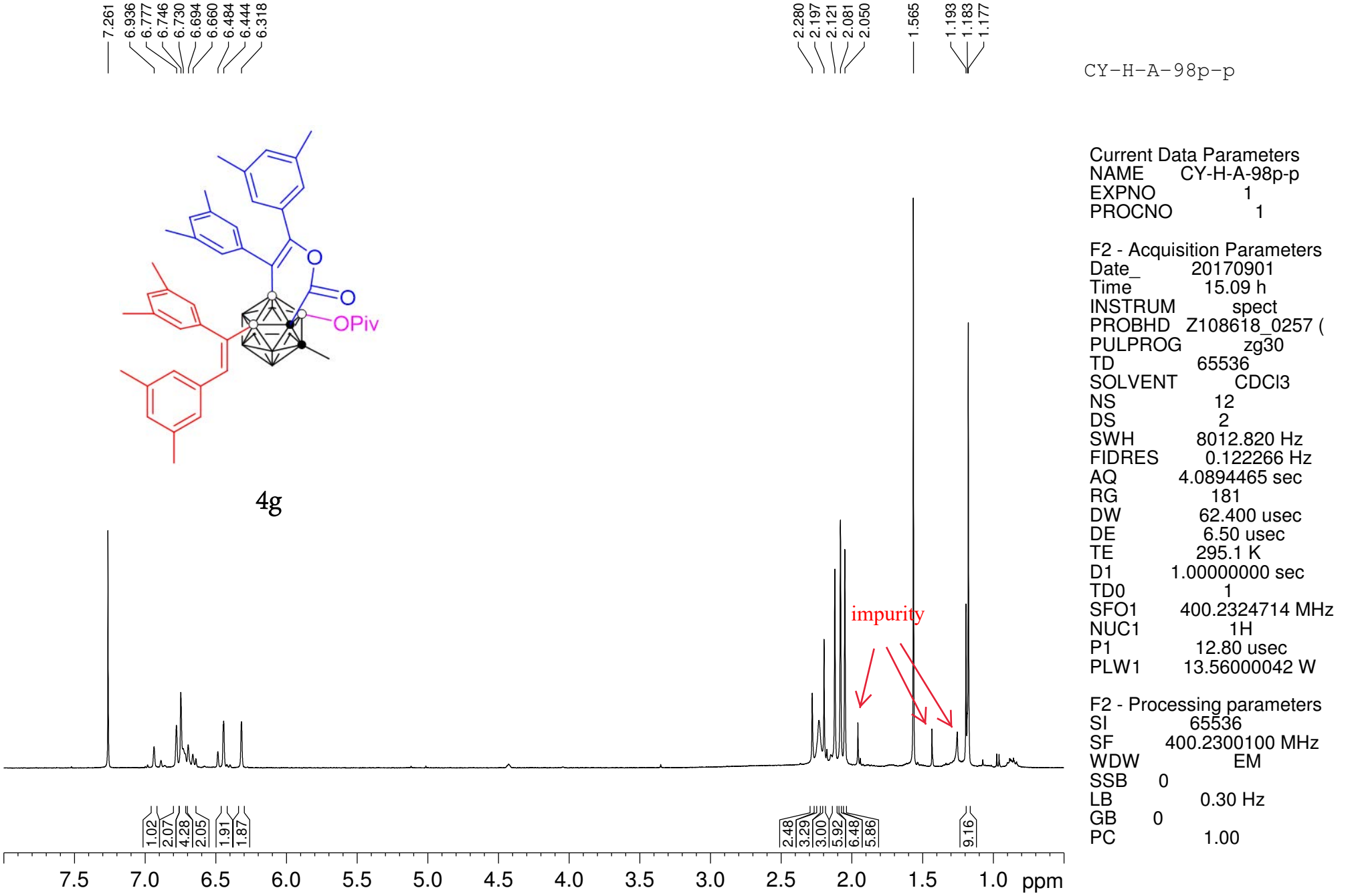


CY-B-A-97Rh-1-(C)

```
Current Data Parameters
NAME      CY-B-A-97Rh-1-(C)
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20200610
Time                18.46 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG                 zg
TD                   65536
SOLVENT              CDCl3
NS                      38
DS                       2
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631488 sec
RG                     406
DW                  20.800 usec
DE                   6.50 usec
TE                  294.2 K
D1              2.00000000 sec
TD0                      1
SFO1          128.4096890 MHz
NUC1                  11B
P1                   7.50 usec
PLW1          55.09999847 W

F2 - Processing parameters
SI                   32768
SF            128.4097430 MHz
WDW                     EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.40
```

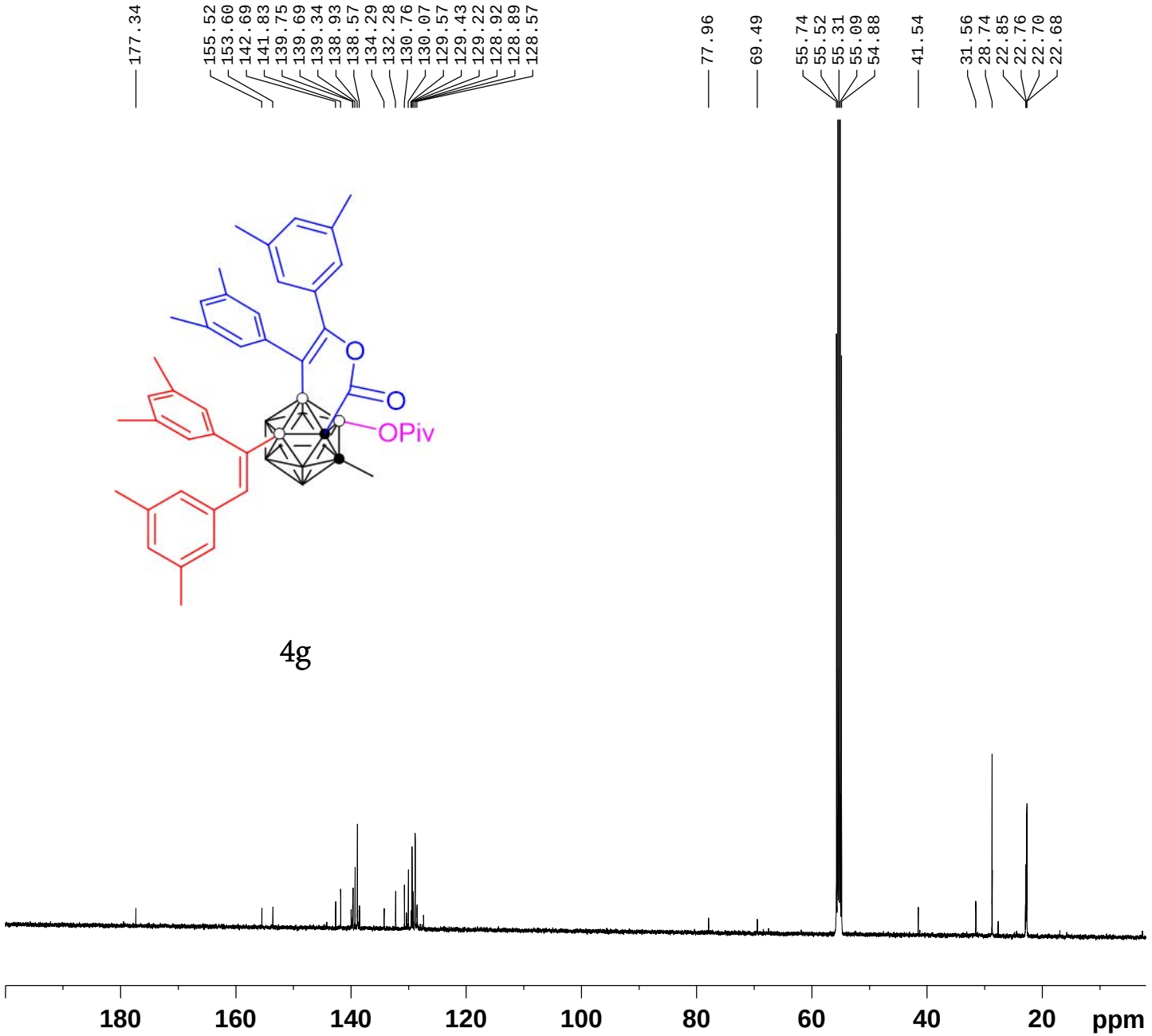


Current Data Parameters
NAME CY-H-A-98p-p
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170901
Time 15.09 h
INSTRUM spect
PROBHD Z108618_0257 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 12
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 181
DW 62.400 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1
SFO1 400.2324714 MHz
NUC1 1H
P1 12.80 usec
PLW1 13.56000042 W

F2 - Processing parameters
SI 65536
SF 400.2300100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

CY-C-A-98Rh



4g

```
Current Data Parameters
NAME         CY-C-A-98Rh
EXPNO                  1
PROCNO                 1

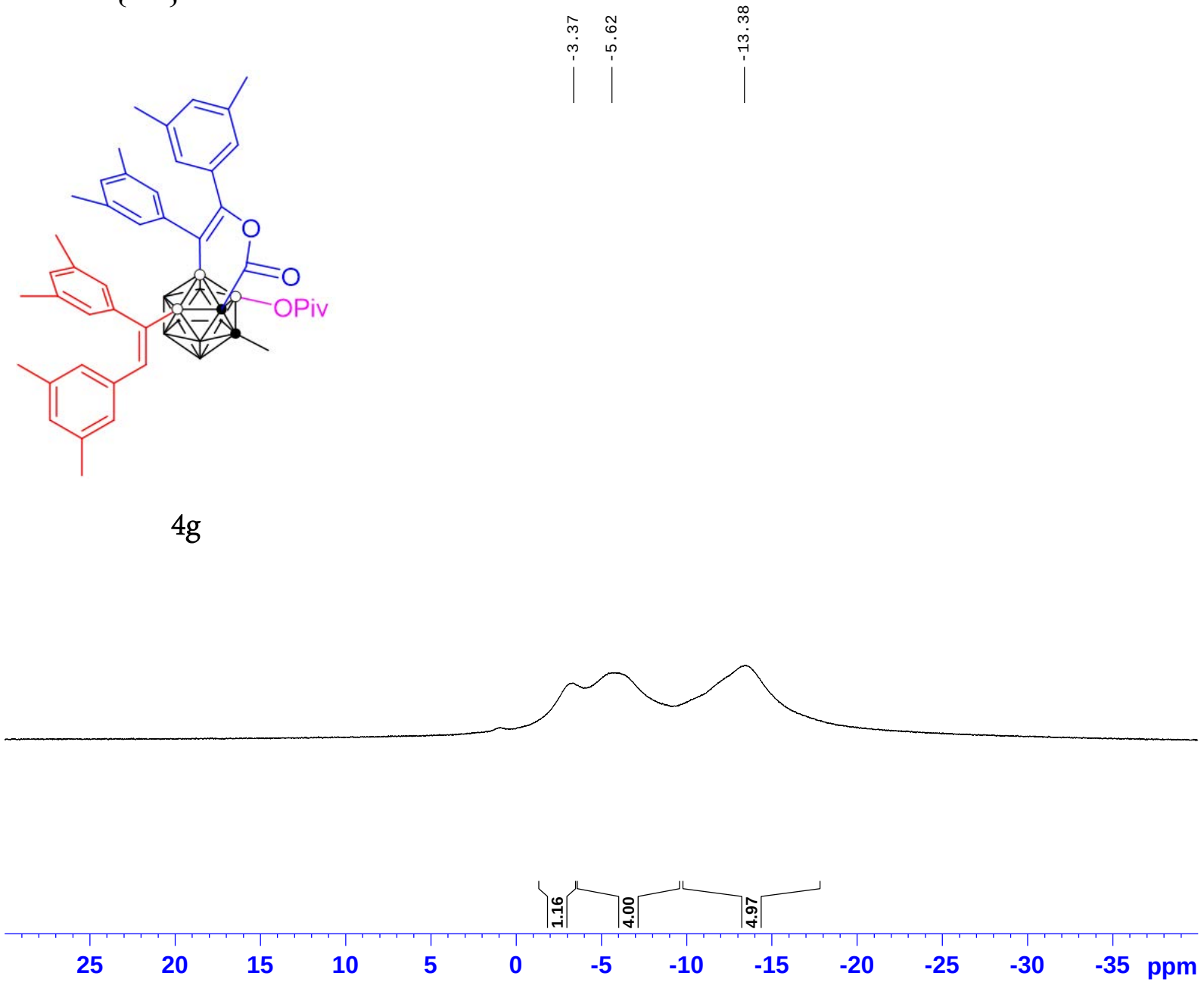
F2 - Acquisition Parameters
Date_           20200616
Time               3.54 h
INSTRUM            spect
PROBHD   Z119470_0283 (
PULPROG          zgpg30
TD                65536
SOLVENT          CD2Cl2
NS                 5000
DS                    4
SWH           29761.904 Hz
FIDRES         0.908261 Hz
AQ            1.1010048 sec
RG               206.72
DW               16.800 usec
DE                6.50 usec
TE                295.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        125.7703643 MHz
NUC1                13C
P1                 9.75 usec
PLW1        94.00000000 W
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        25.00000000 W
PLW12        0.39063001 W
PLW13        0.19648001 W

F2 - Processing parameters
SI                32768
SF          125.7575539 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

$^{11}B\{^{1}H\}$ NMR

CY-B-A-98Rh-1

4g



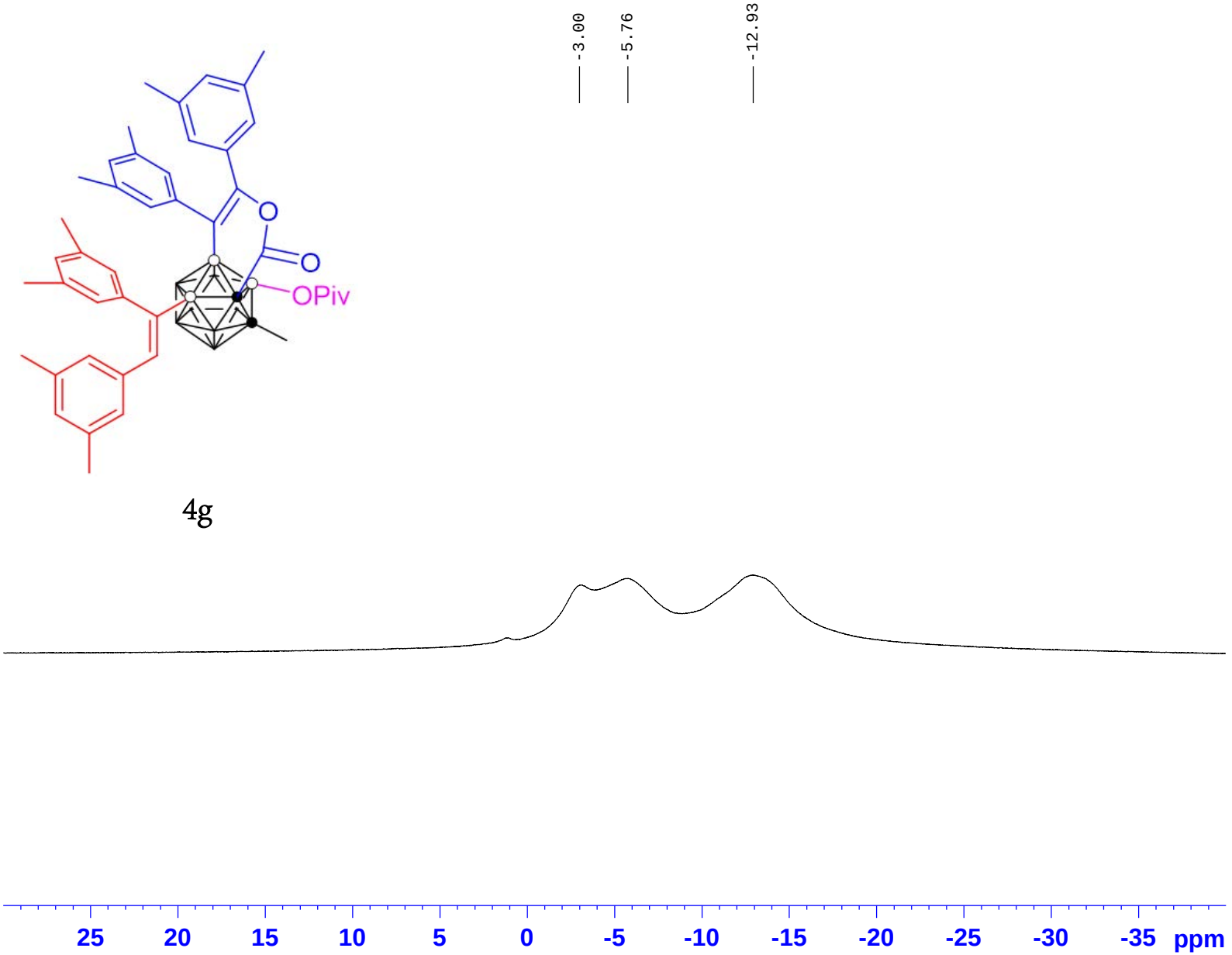

```
Current Data Parameters
NAME      CY-B-A-98Rh-1
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20200610
Time              18.47 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                  406
DW                20.800 usec
DE                 6.50 usec
TE                294.7 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        128.4096890 MHz
NUC1               11B
P1                7.50 usec
PLW1        55.09999847 W
SFO2        400.2316009 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        13.56000042 W
PLW12        0.27428001 W

F2 - Processing parameters
SI                32768
SF          128.4097615 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

^{11}B NMR

4g

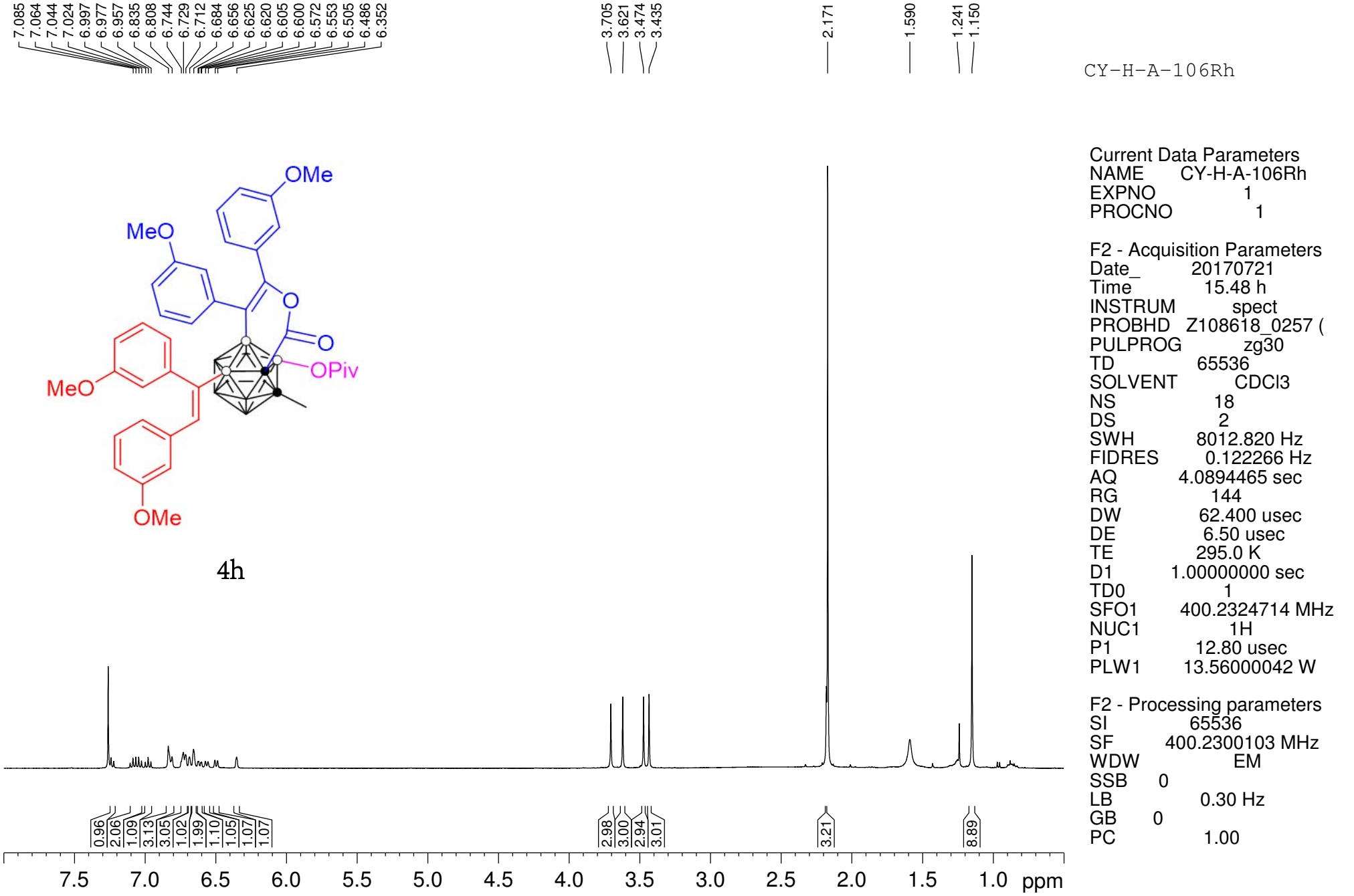


CY-B-A-98Rh-1-(C)

```
Current Data Parameters
NAME      CY-B-A-98Rh-1-(C)
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20200610
Time                18.53 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG                 zg
TD                   65536
SOLVENT              CDCl3
NS                      59
DS                       2
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631488 sec
RG                     406
DW                  20.800 usec
DE                   6.50 usec
TE                  294.1 K
D1               2.00000000 sec
TD0                      1
SFO1           128.4096890 MHz
NUC1                   11B
P1                    7.50 usec
PLW1           55.09999847 W

F2 - Processing parameters
SI                   32768
SF             128.4097430 MHz
WDW                    EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```



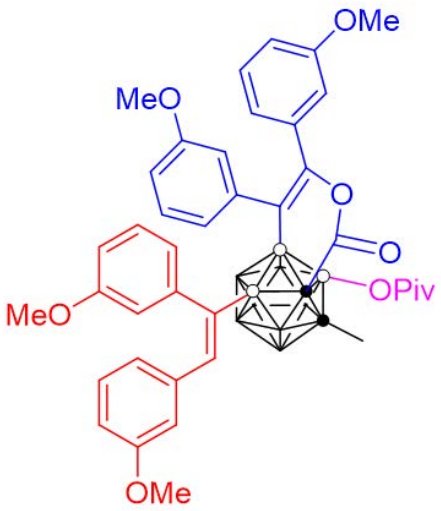
Current Data Parameters
NAME CY-H-A-106Rh
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170721
Time 15.48 h
INSTRUM spect
PROBHD Z108618_0257 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 18
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 144
DW 62.400 usec
DE 6.50 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1
SFO1 400.2324714 MHz
NUC1 1H
P1 12.80 usec
PLW1 13.56000042 W

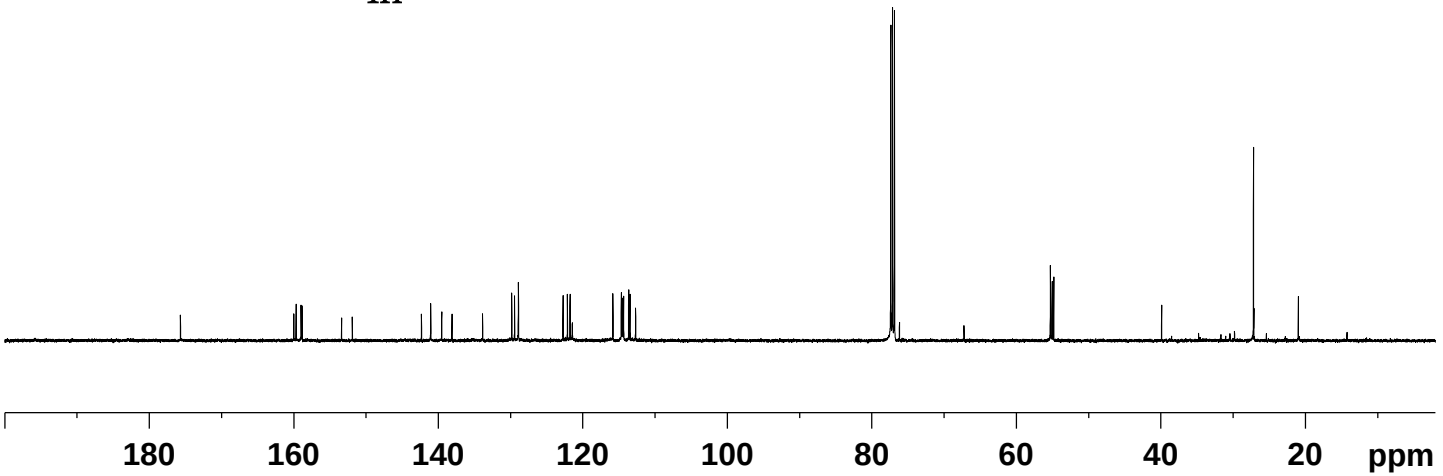
F2 - Processing parameters
SI 65536
SF 400.2300103 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

CY-C-A-106Rh

175.72 160.03 159.71 159.03 158.90 153.41 151.94 142.36 141.09 139.56 138.13 133.90 129.87 129.48 128.96 128.94 122.77 122.16 121.80 121.48 115.87 114.71 114.56 114.50 114.38 113.69 113.50 112.72 77.40 77.15 76.89 76.20 67.27 55.32 55.30 55.07 54.85 39.91 27.20 20.99



4h



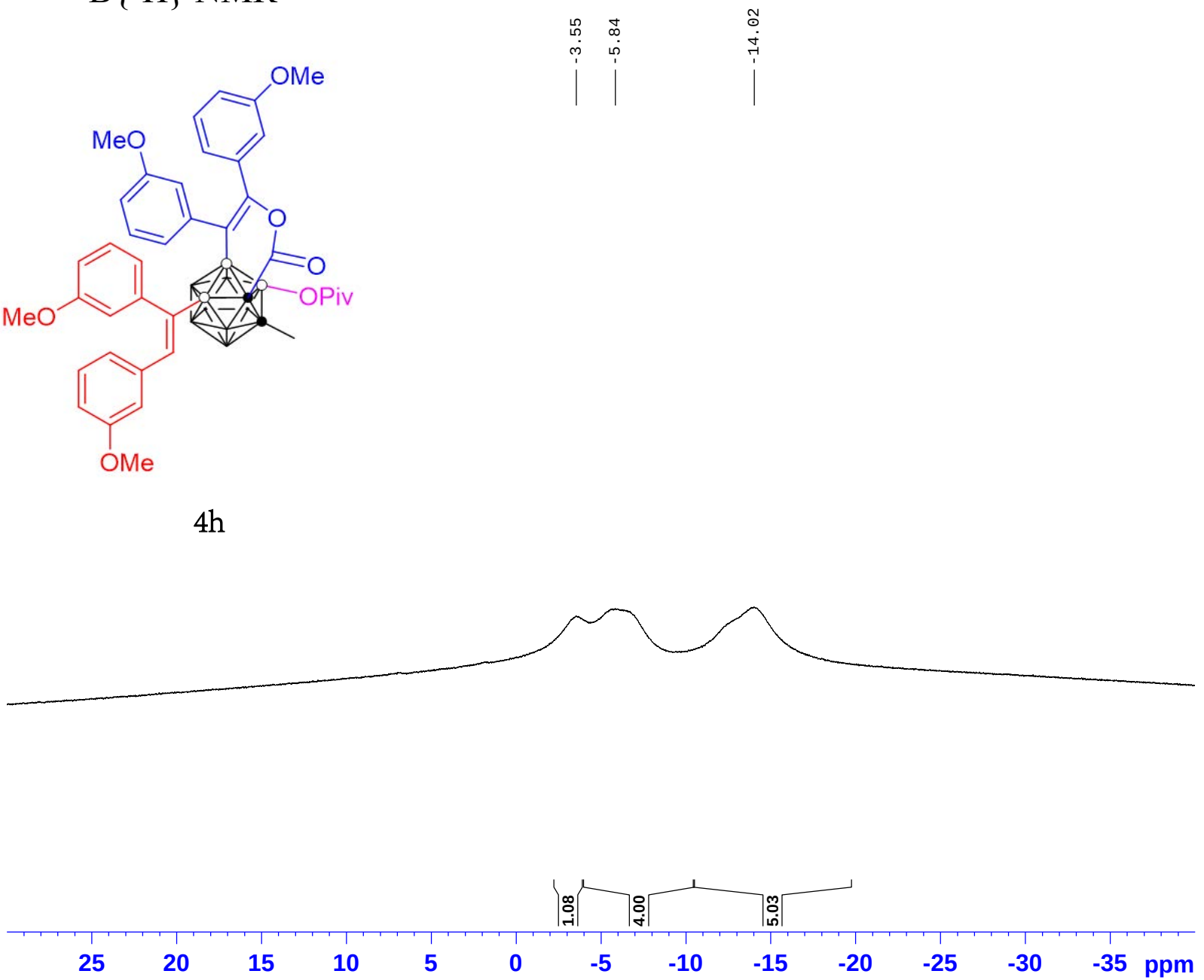

```
Current Data Parameters
NAME        CY-C-A-106Rh
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170722
Time              14.22 h
INSTRUM            spect
PROBHD   Z119470_0283 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                  756
DS                    4
SWH           29761.904 Hz
FIDRES         0.908261 Hz
AQ            1.1010048 sec
RG               206.72
DW               16.800 usec
DE                6.50 usec
TE               297.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        125.7703643 MHz
NUC1                13C
P1                10.00 usec
PLW1        94.00000000 W
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        25.00000000 W
PLW12        0.39063001 W
PLW13        0.19648001 W

F2 - Processing parameters
SI                32768
SF          125.7577748 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

$^{11}B\{^{1}H\}$ NMR

4h



CY-B-A-106Rh

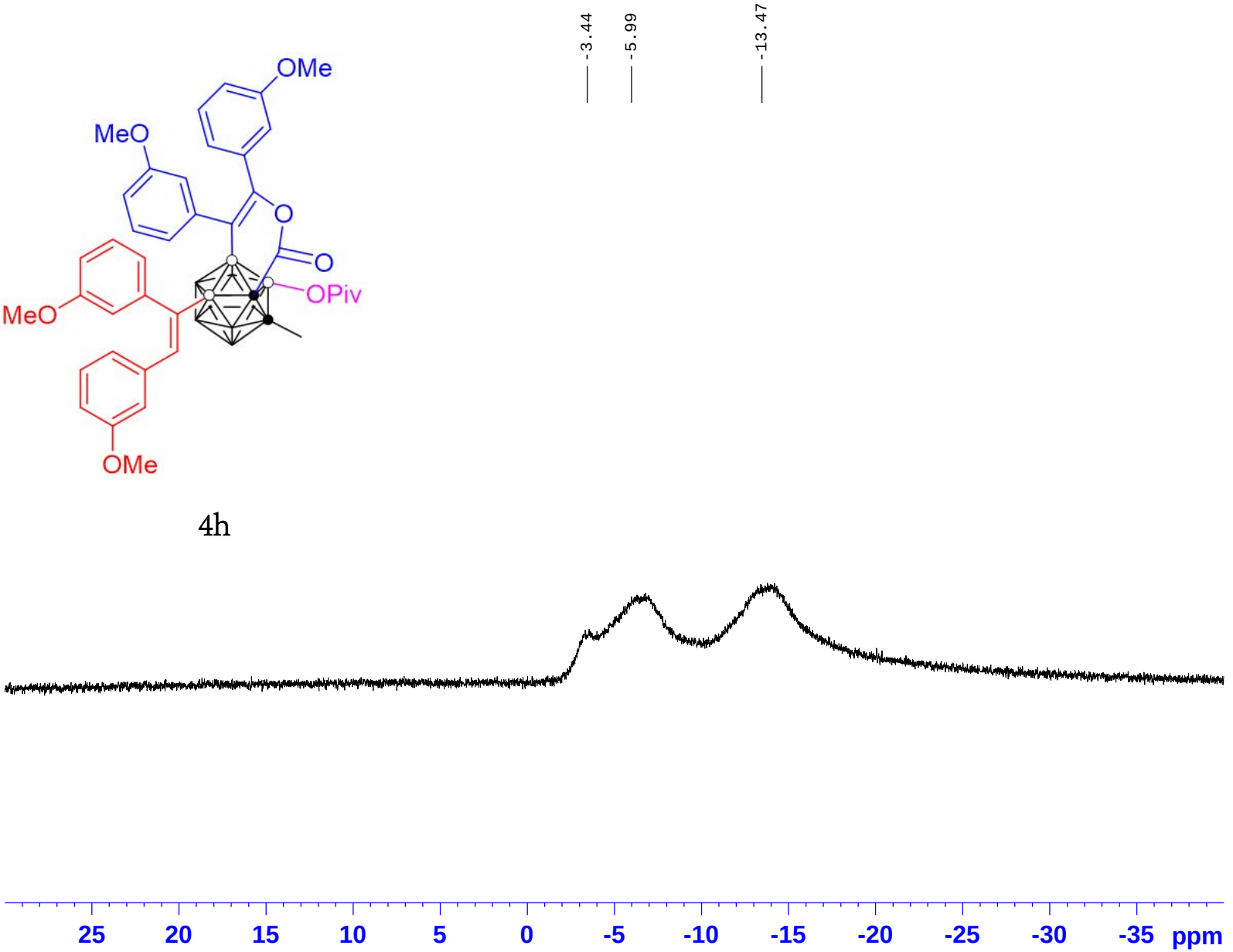
```
Current Data Parameters
NAME       CY-B-A-106Rh
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20170722
Time             16.16 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                   48
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                  322
DW                20.800 usec
DE                 6.50 usec
TE                295.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        128.4096890 MHz
NUC1               11B
P1                7.50 usec
PLW1        55.09999847 W
SFO2        400.2316009 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        13.56000042 W
PLW12        0.27428001 W

F2 - Processing parameters
SI                32768
SF          128.4097615 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

^{11}B NMR

4h



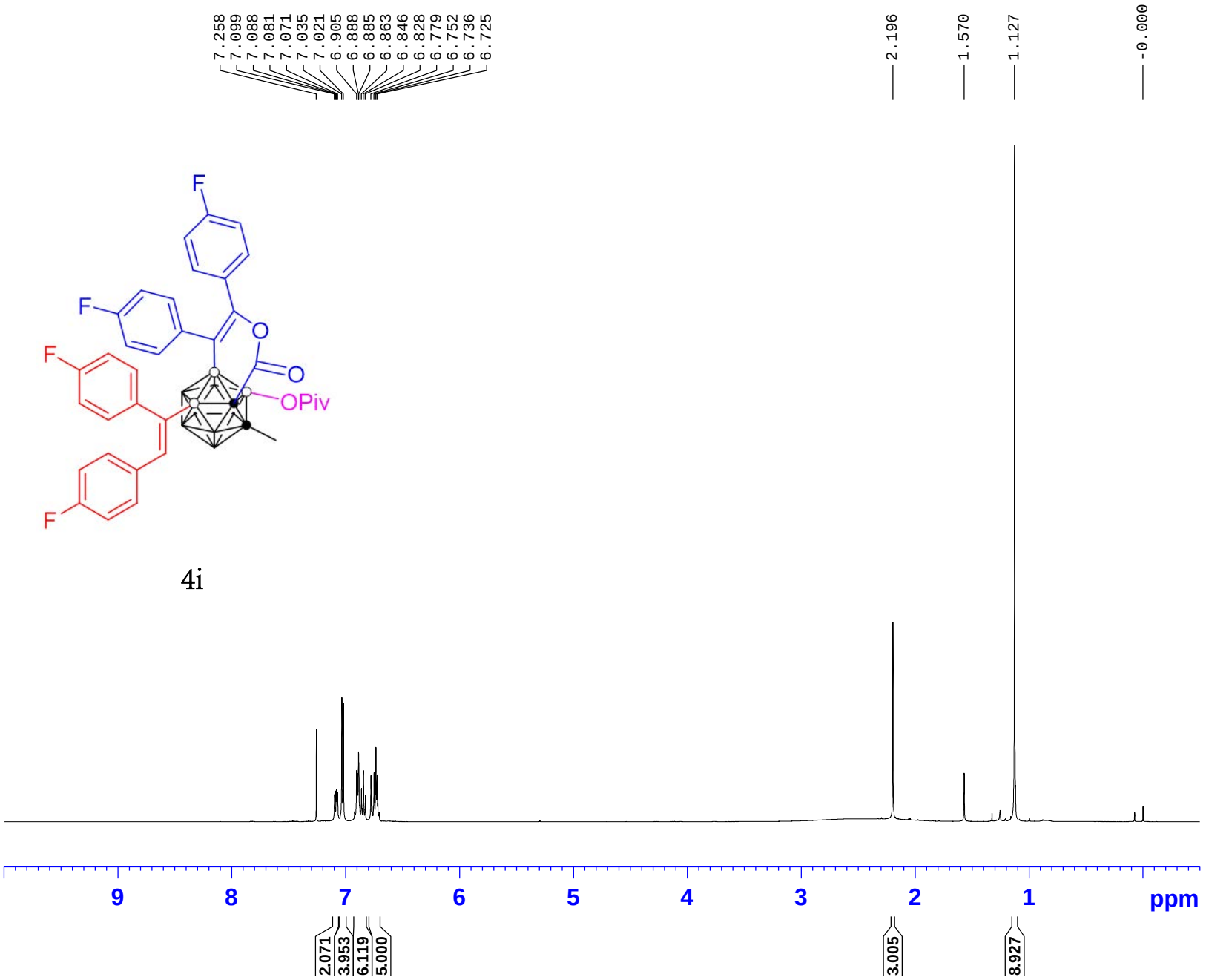
CY-B-A-106Rh-(C)

```
Current Data Parameters
NAME      CY-B-A-106Rh-(C)
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20170722
Time               14.25 h
INSTRUM             spect
PROBHD   Z119470_0283 (
PULPROG                zg
TD                  32768
SOLVENT              None
NS                      2
DS                      4
SWH            24038.461 Hz
FIDRES          1.467191 Hz
AQ             0.6815744 sec
RG                206.72
DW                20.800 usec
DE                 6.50 usec
TE                297.8 K
D1           1.00000000 sec
TD0                     1
SFO1         160.4615792 MHz
NUC1                 11B
P1                 16.00 usec
PLW1        50.00000000 W

F2 - Processing parameters
SI                  16384
SF           160.4615792 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

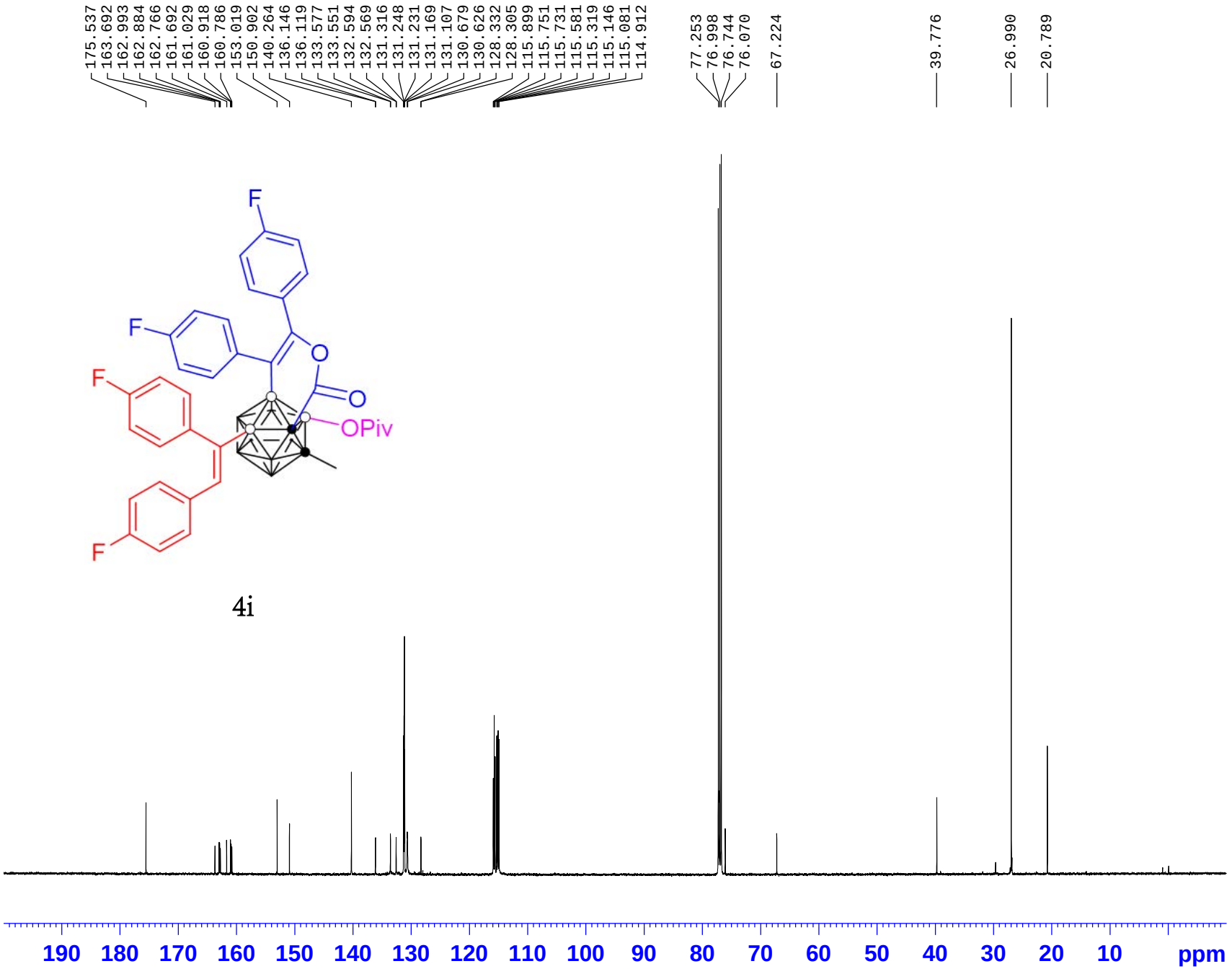
CB5058-H




```
Current Data Parameters
NAME            CB5058-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210124
Time                1.34 h
INSTRUM            spect
PROBHD   Z119470_0283 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            10000.000 Hz
FIDRES          0.305176 Hz
AQ             3.2767999 sec
RG                 56.83
DW                50.000 usec
DE                 6.50 usec
TE                295.2 K
D1             1.00000000 sec
TD0                    1
SFO1          500.1330883 MHz
NUC1                  1H
P1                 10.91 usec
PLW1          25.00000000 W

F2 - Processing parameters
SI                 65536
SF            500.1300132 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```




```
Current Data Parameters
NAME            CB5058-C
EXPNO                  1
PROCNO                 1

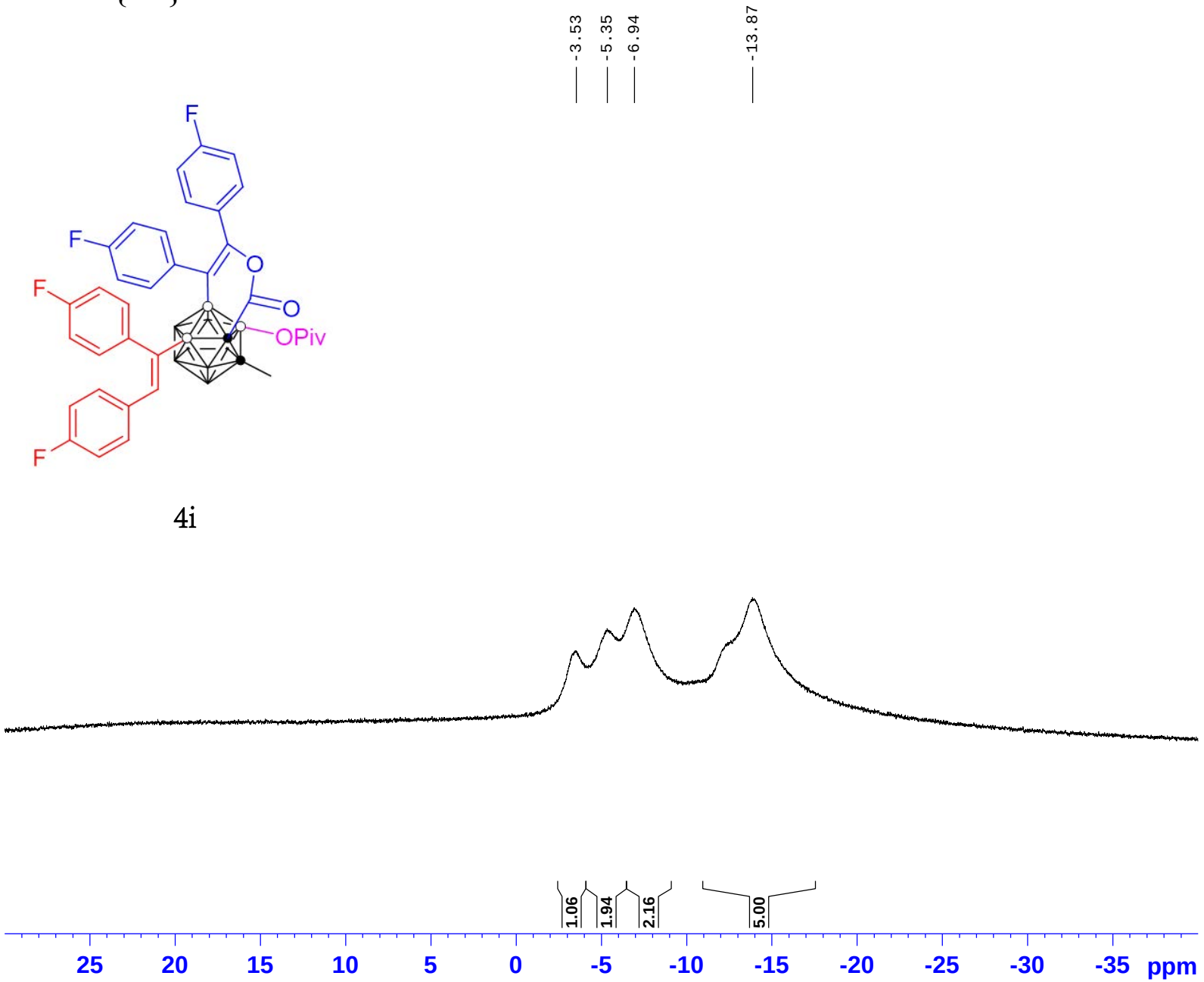
F2 - Acquisition Parameters
Date_           20210124
Time                5.15 h
INSTRUM             spect
PROBHD   Z119470_0283 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  4096
DS                     4
SWH           29761.904 Hz
FIDRES         0.908261 Hz
AQ            1.1010048 sec
RG                206.72
DW               16.800 usec
DE                6.50 usec
TE                295.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1
SFO1        125.7703643 MHz
NUC1                13C
P1                 9.75 usec
PLW1        94.00000000 W
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        25.00000000 W
PLW12        0.46495000 W
PLW13        0.23387000 W

F2 - Processing parameters
SI                 32768
SF          125.7577936 MHz
WDW                   EM
SSB      0
LB                 1.00 Hz
GB       0
PC                 1.40
```

$^{11}B\{^{1}H\}$ NMR

CB5058-B-dc

4i



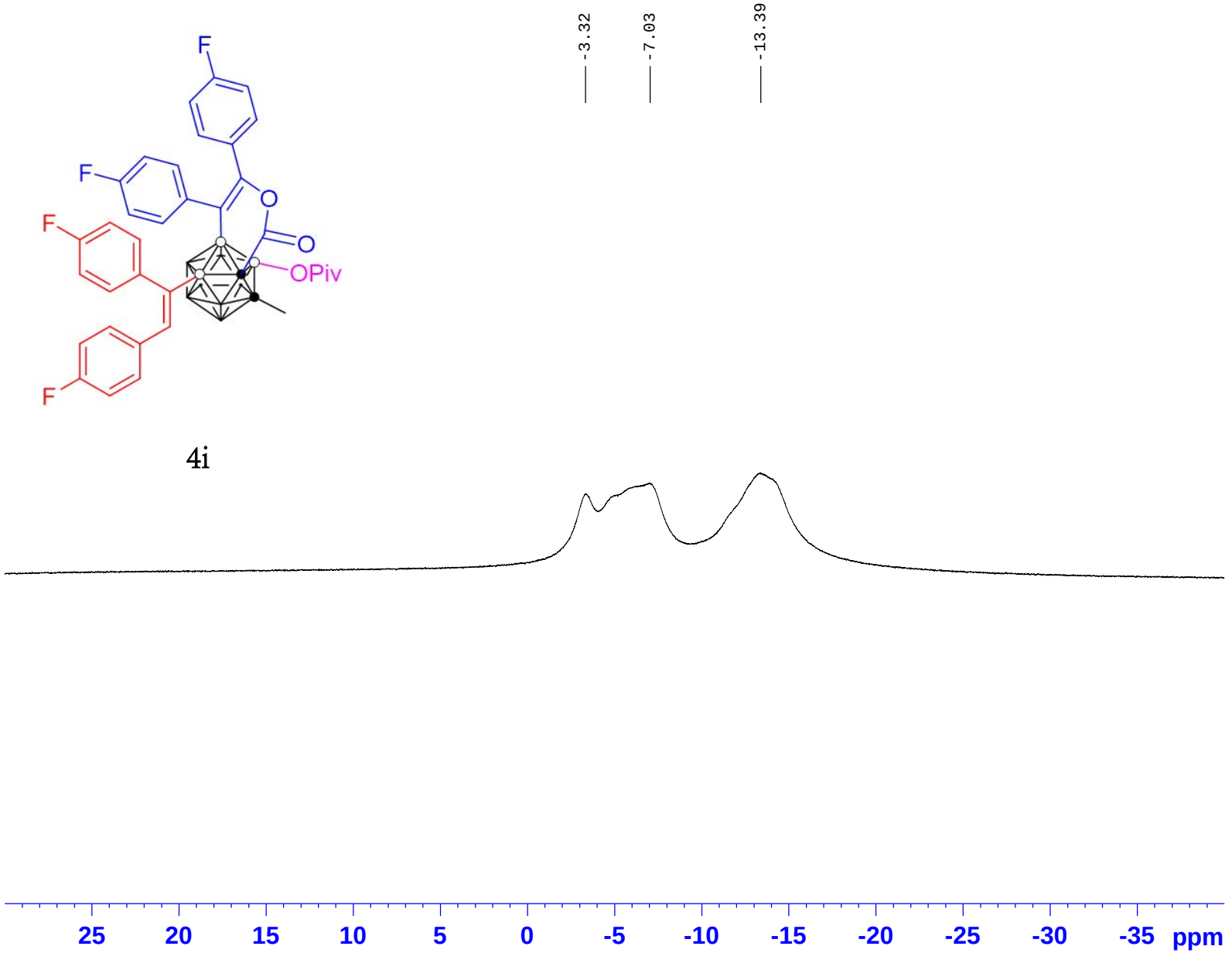

```
Current Data Parameters
NAME         CB5058-B-dc
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210124
Time               1.36 h
INSTRUM            spect
PROBHD   Z119470_0283 (
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                   32
DS                    4
SWH           24038.461 Hz
FIDRES         1.467191 Hz
AQ             0.6815744 sec
RG                206.72
DW                20.800 usec
DE                 6.50 usec
TE                295.1 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1         160.4615790 MHz
NUC1                 11B
P1                16.00 usec
PLW1         50.00000000 W
SFO2         500.1320005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2         25.00000000 W
PLW12         0.46495000 W
PLW13         0.23387000 W

F2 - Processing parameters
SI                16384
SF           160.4615999 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

^{11}B NMR

4i



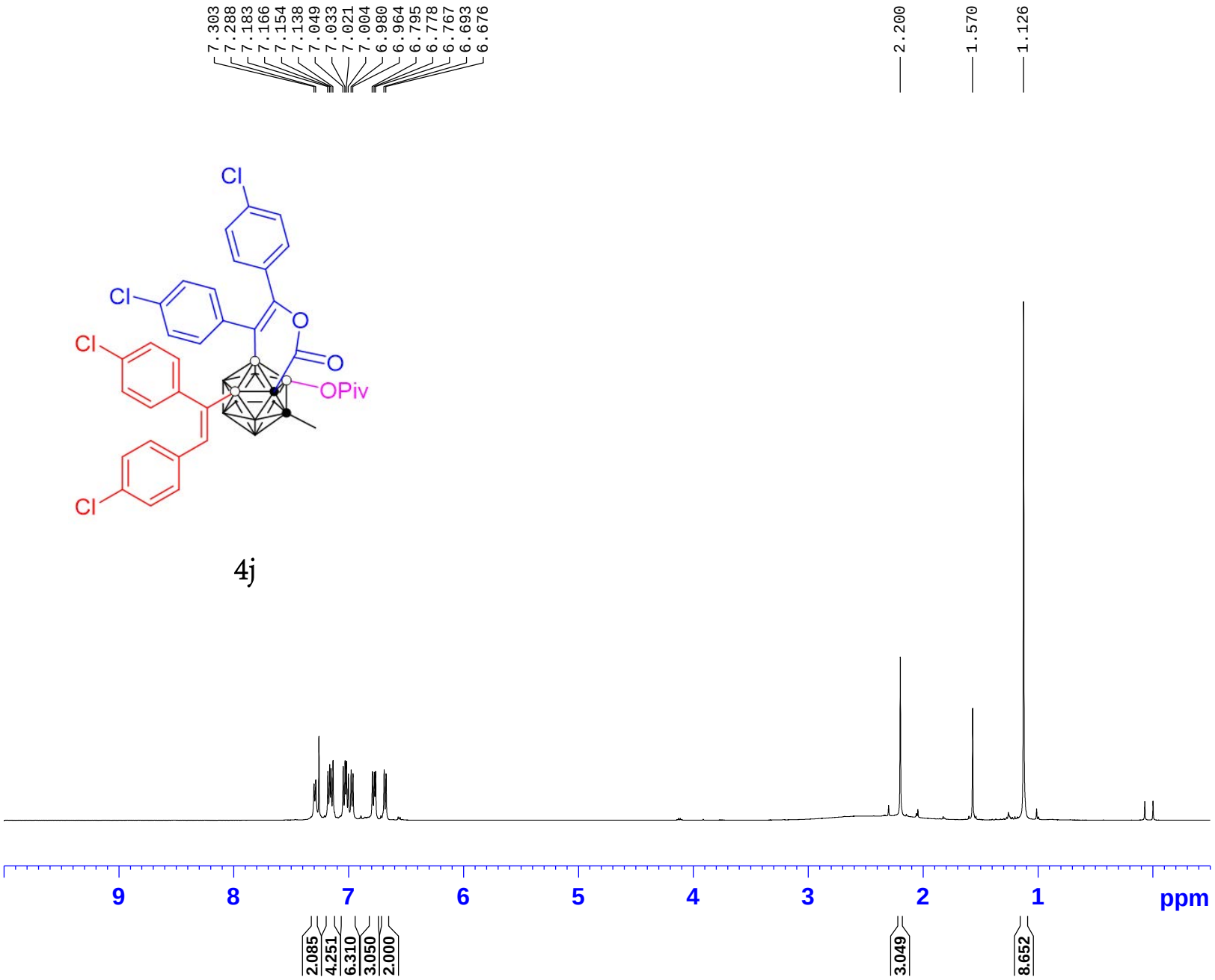
CB5058-B-c

```
Current Data Parameters
NAME          CB5058-B-c
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210124
Time                1.38 h
INSTRUM            spect
PROBHD   Z119470_0283 (
PULPROG               zg
TD                 32768
SOLVENT            CDCl3
NS                    64
DS                     4
SWH            24038.461 Hz
FIDRES          1.467191 Hz
AQ             0.6815744 sec
RG                206.72
DW                20.800 usec
DE                 6.50 usec
TE                295.1 K
D1           1.00000000 sec
TD0                    1
SFO1         160.4615792 MHz
NUC1                11B
P1                16.00 usec
PLW1         50.00000000 W

F2 - Processing parameters
SI                 16384
SF           160.4615997 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

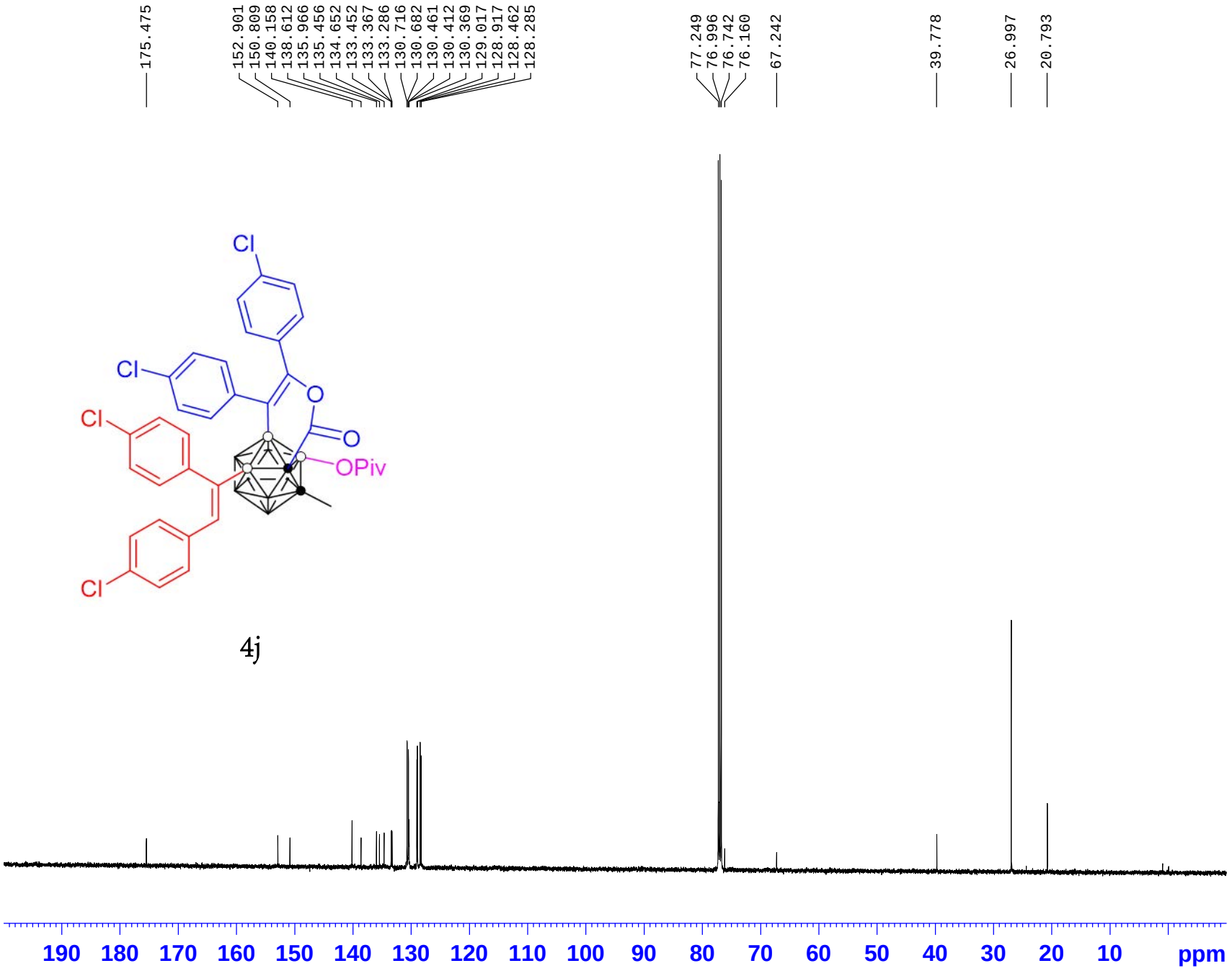
CB5097-p-H




```
Current Data Parameters
NAME          CB5097-p-H
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210307
Time              10.33 h
INSTRUM             spect
PROBHD   Z119470_0283 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                 93.28
DW                50.000 usec
DE                 6.50 usec
TE                 295.1 K
D1            1.00000000 sec
TD0                    1
SFO1          500.1330883 MHz
NUC1                  1H
P1                10.91 usec
PLW1          25.00000000 W

F2 - Processing parameters
SI                 65536
SF            500.1300114 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```




```
Current Data Parameters
NAME          CB5097-p-C
EXPNO                  6
PROCNO                 1

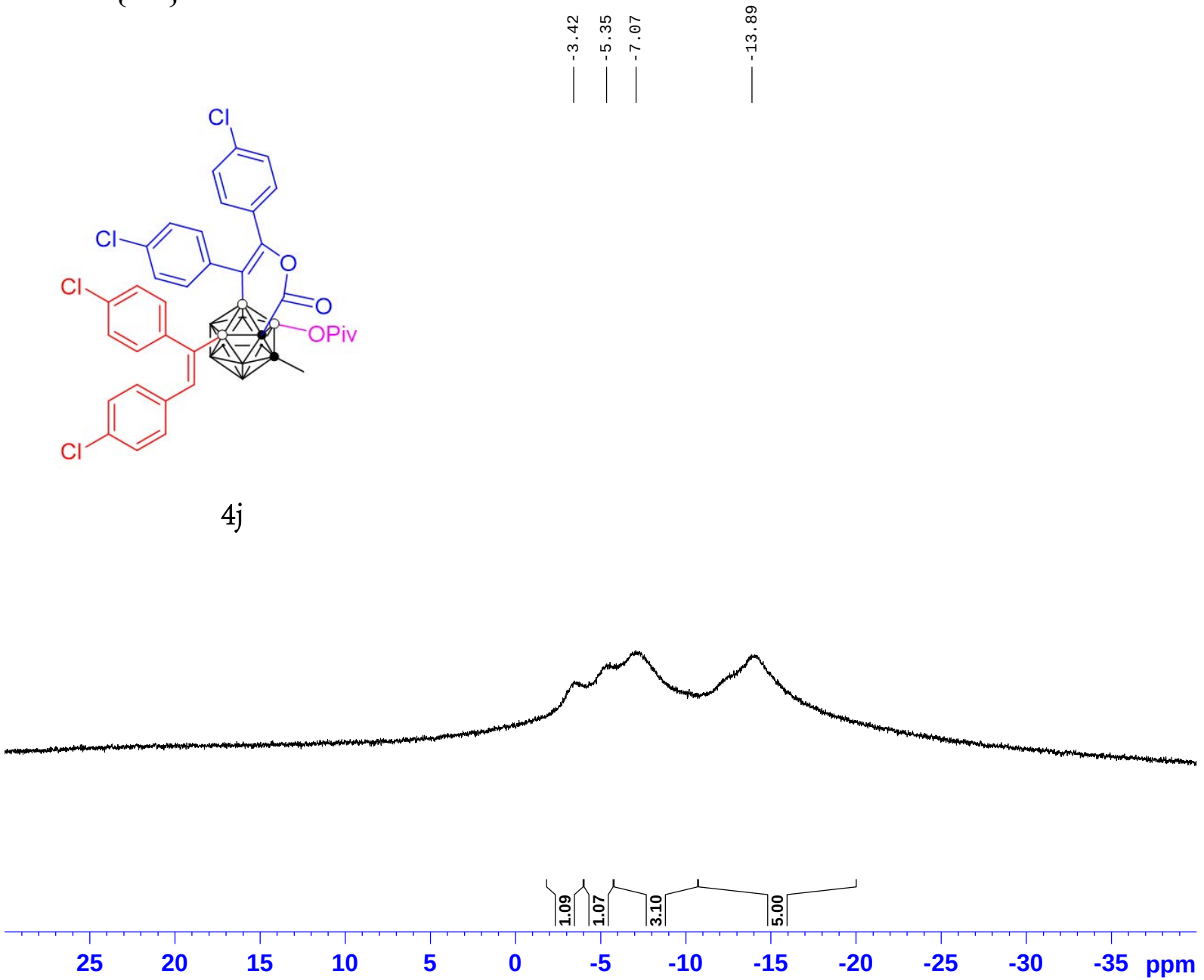
F2 - Acquisition Parameters
Date_           20210307
Time              10.29 h
INSTRUM            spect
PROBHD   Z119470_0283 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           29761.904 Hz
FIDRES         0.908261 Hz
AQ            1.1010048 sec
RG               117.01
DW               16.800 usec
DE                6.50 usec
TE               295.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        125.7703643 MHz
NUC1               13C
P1                9.75 usec
PLW1        94.00000000 W
SFO2        500.1320005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            80.00 usec
PLW2        25.00000000 W
PLW12        0.46495000 W
PLW13        0.23387000 W

F2 - Processing parameters
SI                32768
SF          125.7577936 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

$^{11}B\{^{1}H\}$ NMR

CB5097-p-B-dc

4j



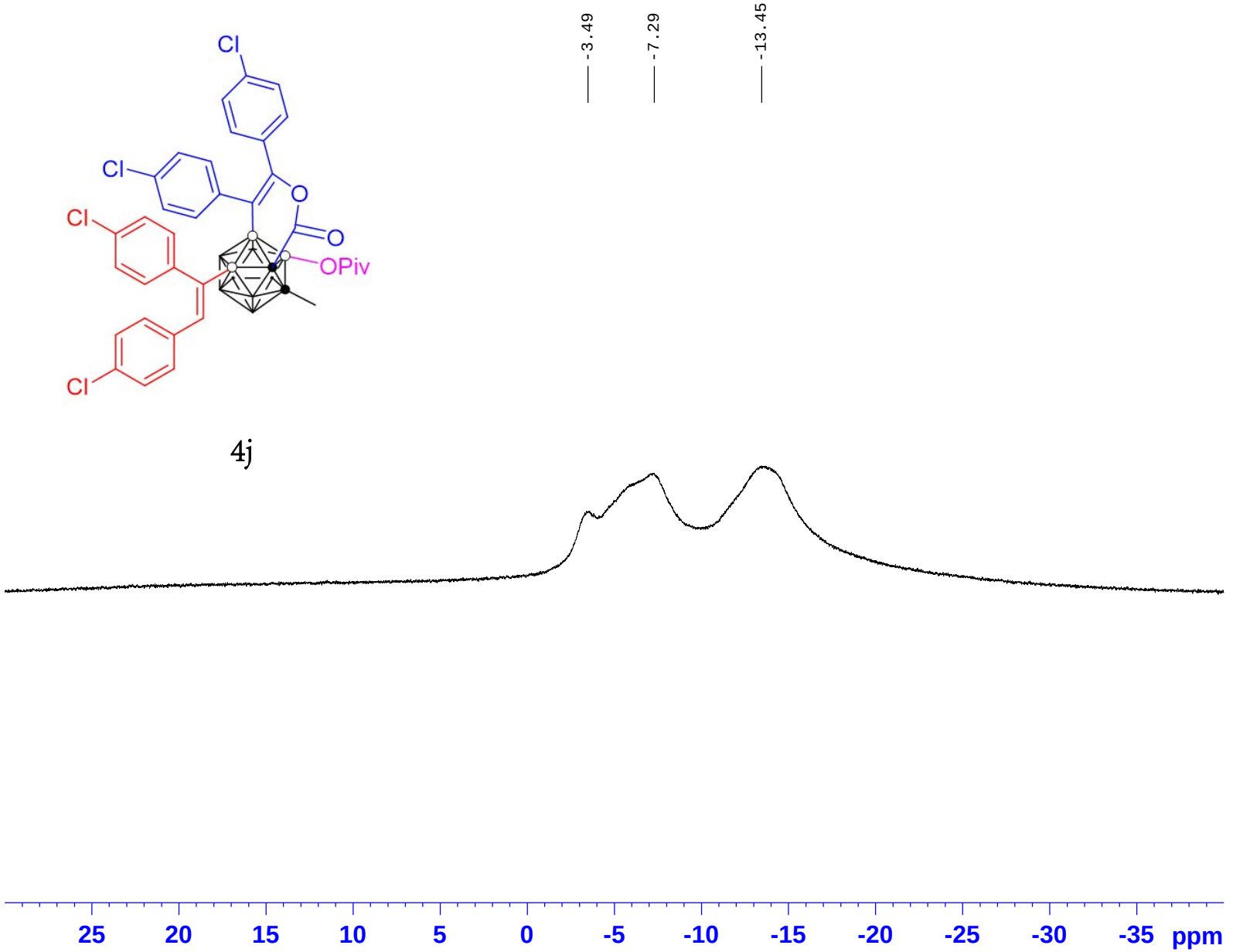

```
Current Data Parameters
NAME      CB5097-p-B-dc
EXPNO                 5
PROCNO                1

F2 - Acquisition Parameters
Date_          20210307
Time             10.36 h
INSTRUM            spect
PROBHD   Z119470_0283 (
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                   32
DS                    4
SWH           24038.461 Hz
FIDRES         1.467191 Hz
AQ            0.6815744 sec
RG               206.72
DW               20.800 usec
DE                6.50 usec
TE               295.1 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        160.4615790 MHz
NUC1                11B
P1                16.00 usec
PLW1        50.00000000 W
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             80.00 usec
PLW2        25.00000000 W
PLW12        0.46495000 W
PLW13        0.23387000 W

F2 - Processing parameters
SI                16384
SF          160.4615999 MHz
WDW                  EM
SSB      0
LB                1.00 Hz
GB       0
PC                1.40
```

^{11}B NMR

4j



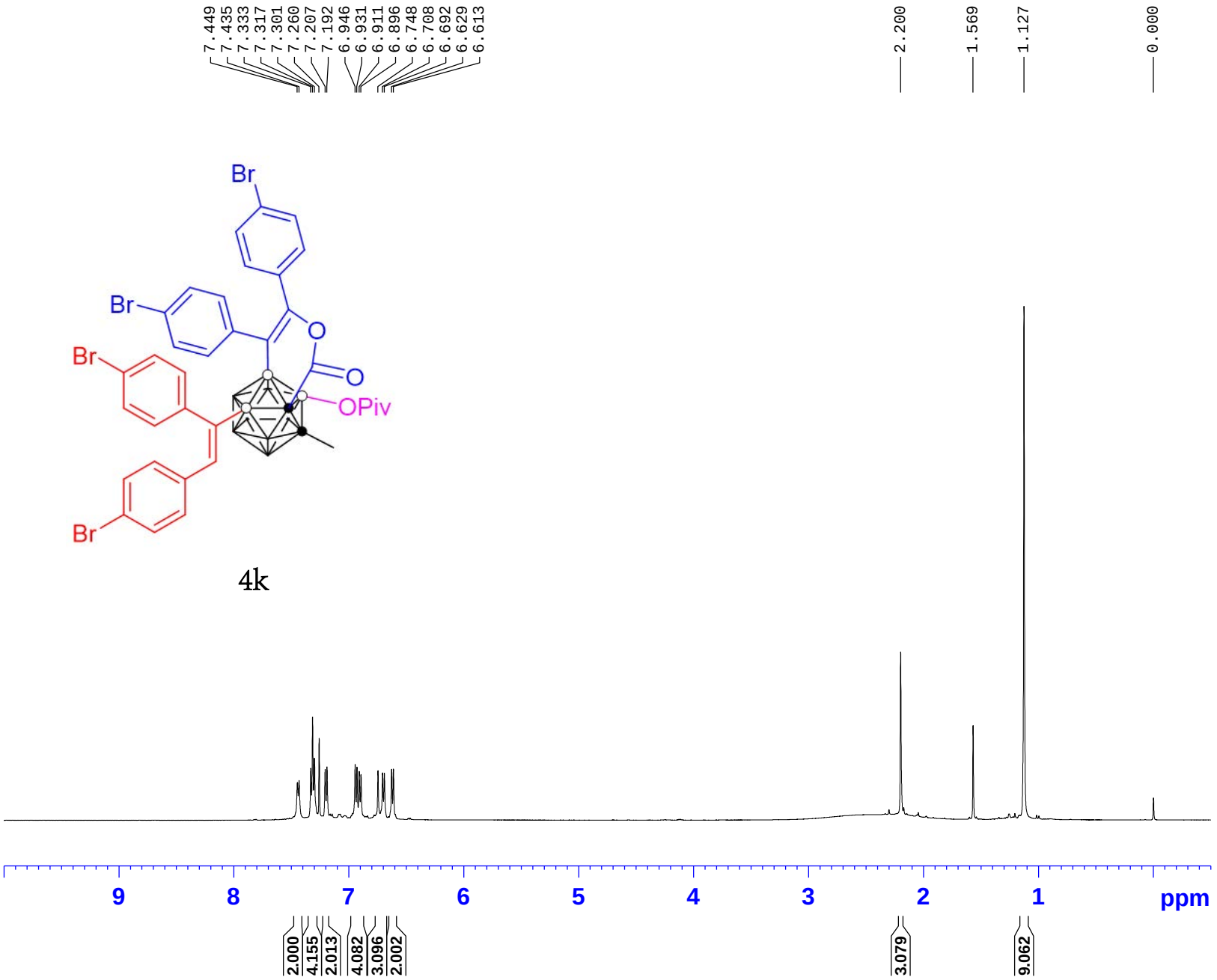
CB5097-p-B-c

```
Current Data Parameters
NAME       CB5097-p-B-c
EXPNO                 6
PROCNO                1

F2 - Acquisition Parameters
Date_          20210307
Time              10.40 h
INSTRUM            spect
PROBHD   Z119470_0283 (
PULPROG              zg
TD                32768
SOLVENT           CDCl3
NS                   64
DS                    4
SWH           24038.461 Hz
FIDRES         1.467191 Hz
AQ            0.6815744 sec
RG               206.72
DW                20.800 usec
DE                 6.50 usec
TE                295.2 K
D1           1.00000000 sec
TD0                   1
SFO1        160.4615792 MHz
NUC1                11B
P1                16.00 usec
PLW1        50.00000000 W

F2 - Processing parameters
SI                16384
SF          160.4615997 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

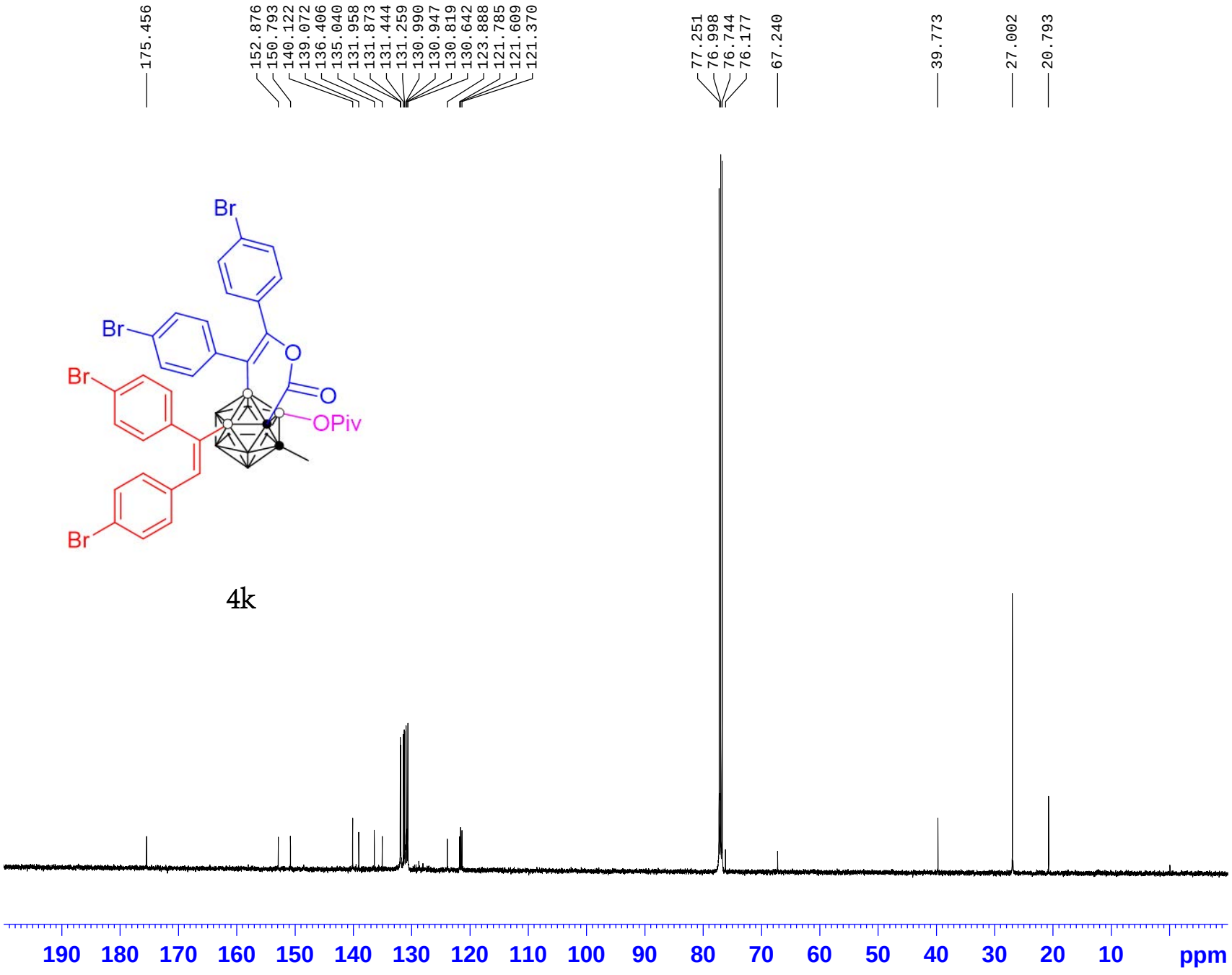
CB5100-p-H




```
Current Data Parameters
NAME          CB5100-p-H
EXPNO                  8
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210307
Time               11.52 h
INSTRUM             spect
PROBHD   Z119470_0283 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            10000.000 Hz
FIDRES          0.305176 Hz
AQ             3.2767999 sec
RG                 56.83
DW                50.000 usec
DE                 6.50 usec
TE                295.2 K
D1             1.00000000 sec
TD0                    1
SFO1          500.1330883 MHz
NUC1                  1H
P1                 10.91 usec
PLW1          25.00000000 W

F2 - Processing parameters
SI                 65536
SF            500.1300128 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



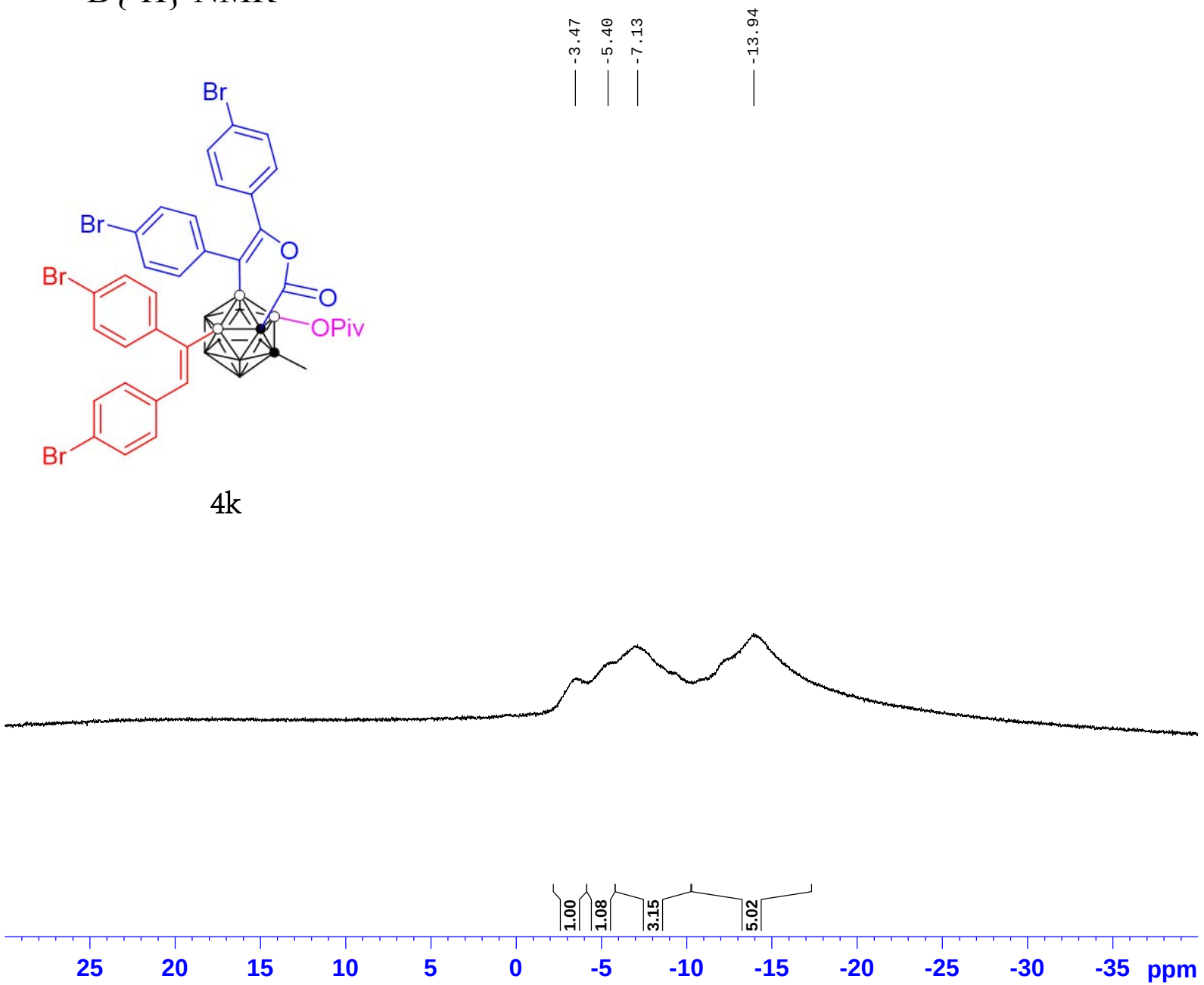

```
Current Data Parameters
NAME          CB5100-p-C
EXPNO                  7
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210307
Time               12.55 h
INSTRUM             spect
PROBHD   Z119470_0283 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            29761.904 Hz
FIDRES          0.908261 Hz
AQ             1.1010048 sec
RG                206.72
DW                16.800 usec
DE                 6.50 usec
TE                295.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1
SFO1         125.7703643 MHz
NUC1                13C
P1                 9.75 usec
PLW1         94.00000000 W
SFO2         500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2         25.00000000 W
PLW12         0.46495000 W
PLW13         0.23387000 W

F2 - Processing parameters
SI                 32768
SF           125.7577945 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

$^{11}B\{^{1}H\}$ NMR

4k



CB5100-B-dc

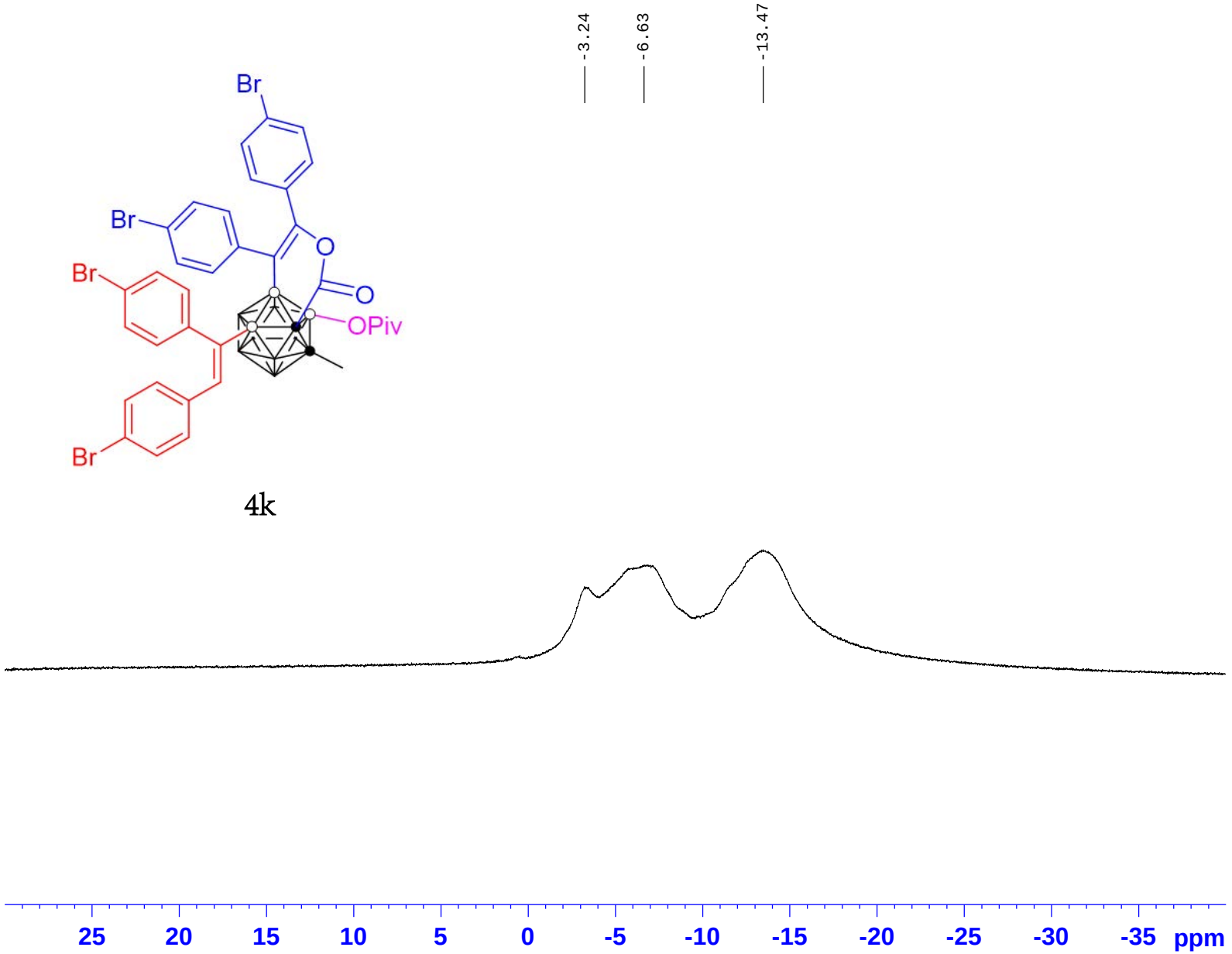
```
Current Data Parameters
NAME         CB5100-B-dc
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210302
Time               2.32 h
INSTRUM            spect
PROBHD   Z119470_0283 (
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                   32
DS                    4
SWH           24038.461 Hz
FIDRES         1.467191 Hz
AQ             0.6815744 sec
RG                206.72
DW                20.800 usec
DE                 6.50 usec
TE                295.1 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1         160.4615790 MHz
NUC1                 11B
P1                16.00 usec
PLW1         50.00000000 W
SFO2         500.1320005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2         25.00000000 W
PLW12         0.46495000 W
PLW13         0.23387000 W

F2 - Processing parameters
SI                16384
SF           160.4615999 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

^{11}B NMR

4k



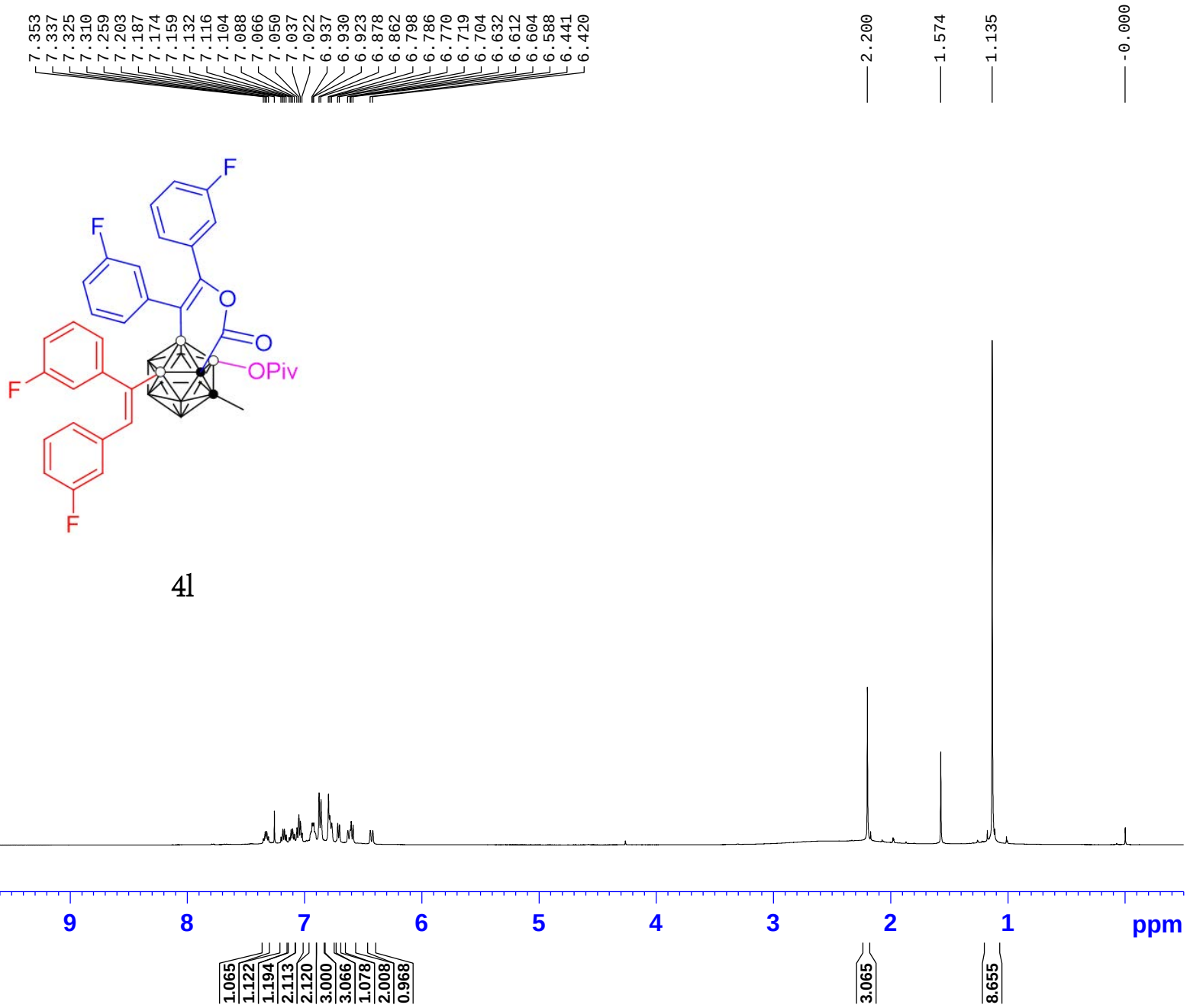
CB5100-B-c

```
Current Data Parameters
NAME         CB5100-B-c
EXPNO                 7
PROCNO                1

F2 - Acquisition Parameters
Date_          20210302
Time              2.36 h
INSTRUM           spect
PROBHD   Z119470_0283 (
PULPROG             zg
TD                32768
SOLVENT           CDCl3
NS                   64
DS                    4
SWH           24038.461 Hz
FIDRES         1.467191 Hz
AQ            0.6815744 sec
RG               206.72
DW                20.800 usec
DE                 6.50 usec
TE                295.1 K
D1           1.00000000 sec
TD0                   1
SFO1        160.4615792 MHz
NUC1                11B
P1                16.00 usec
PLW1        50.00000000 W

F2 - Processing parameters
SI                16384
SF          160.4615997 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

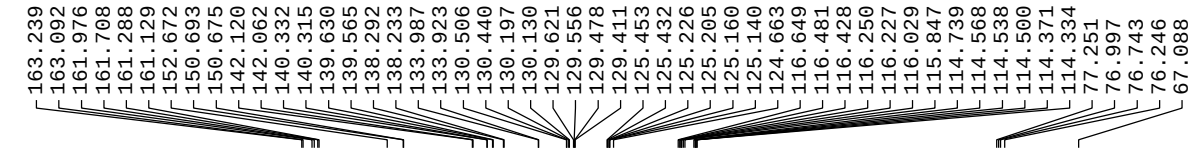
CB5009-p-H




```
Current Data Parameters
NAME          CB5009-p-H
EXPNO                  9
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210307
Time               20.47 h
INSTRUM            spect
PROBHD   Z119470_0283 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                 56.83
DW                50.000 usec
DE                 6.50 usec
TE                295.2 K
D1            1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P1                 10.91 usec
PLW1         25.00000000 W

F2 - Processing parameters
SI                 65536
SF           500.1300131 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

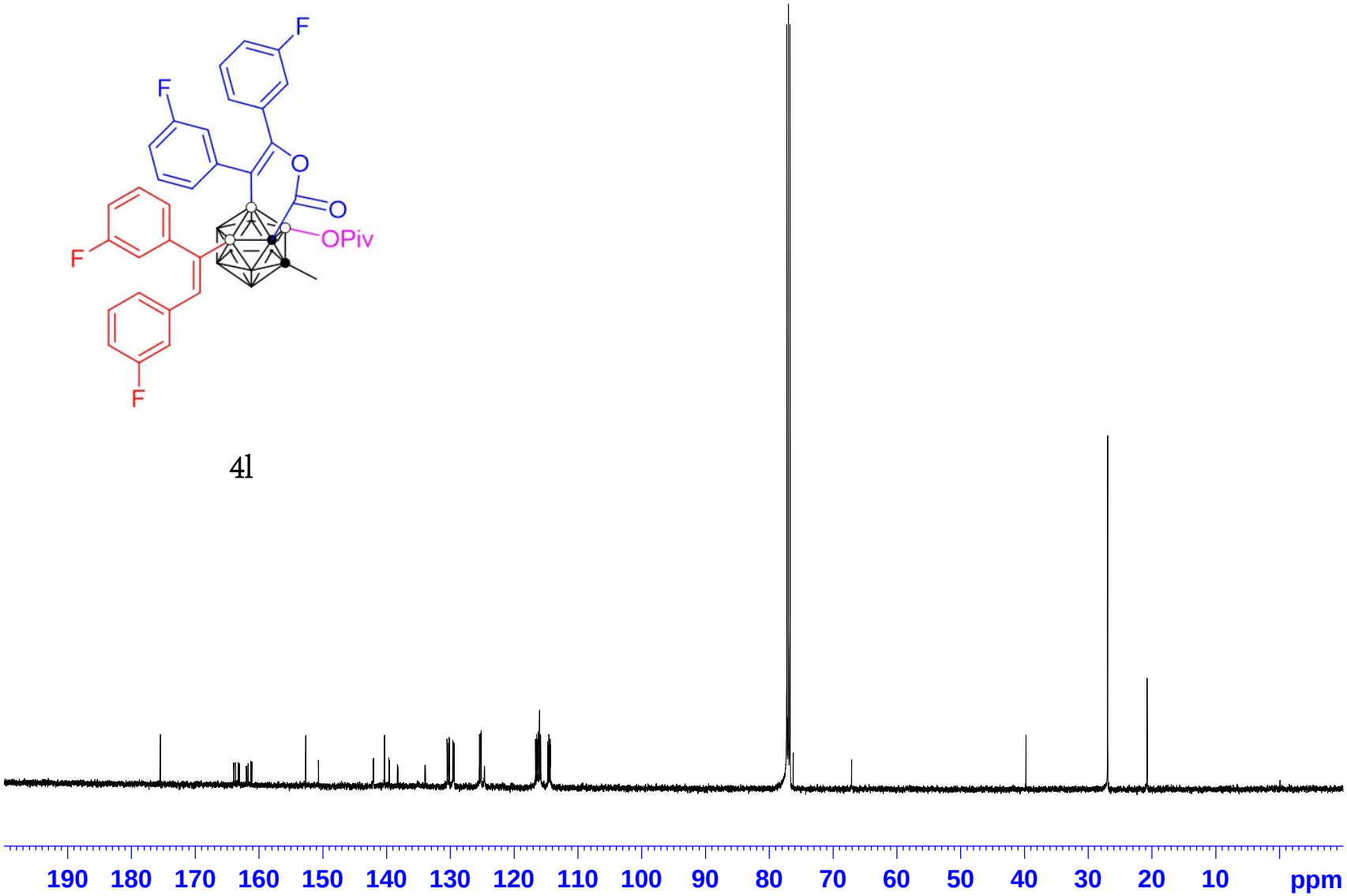


4l

```
Current Data Parameters
NAME          CB5009-p-C
EXPNO                  7
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210307
Time              21.49 h
INSTRUM            spect
PROBHD   Z119470_0283 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           29761.904 Hz
FIDRES         0.908261 Hz
AQ             1.1010048 sec
RG                206.72
DW               16.800 usec
DE                6.50 usec
TE                295.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        125.7703643 MHz
NUC1               13C
P1                9.75 usec
PLW1        94.00000000 W
SFO2        500.1320005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2             80.00 usec
PLW2        25.00000000 W
PLW12        0.46495000 W
PLW13        0.23387000 W

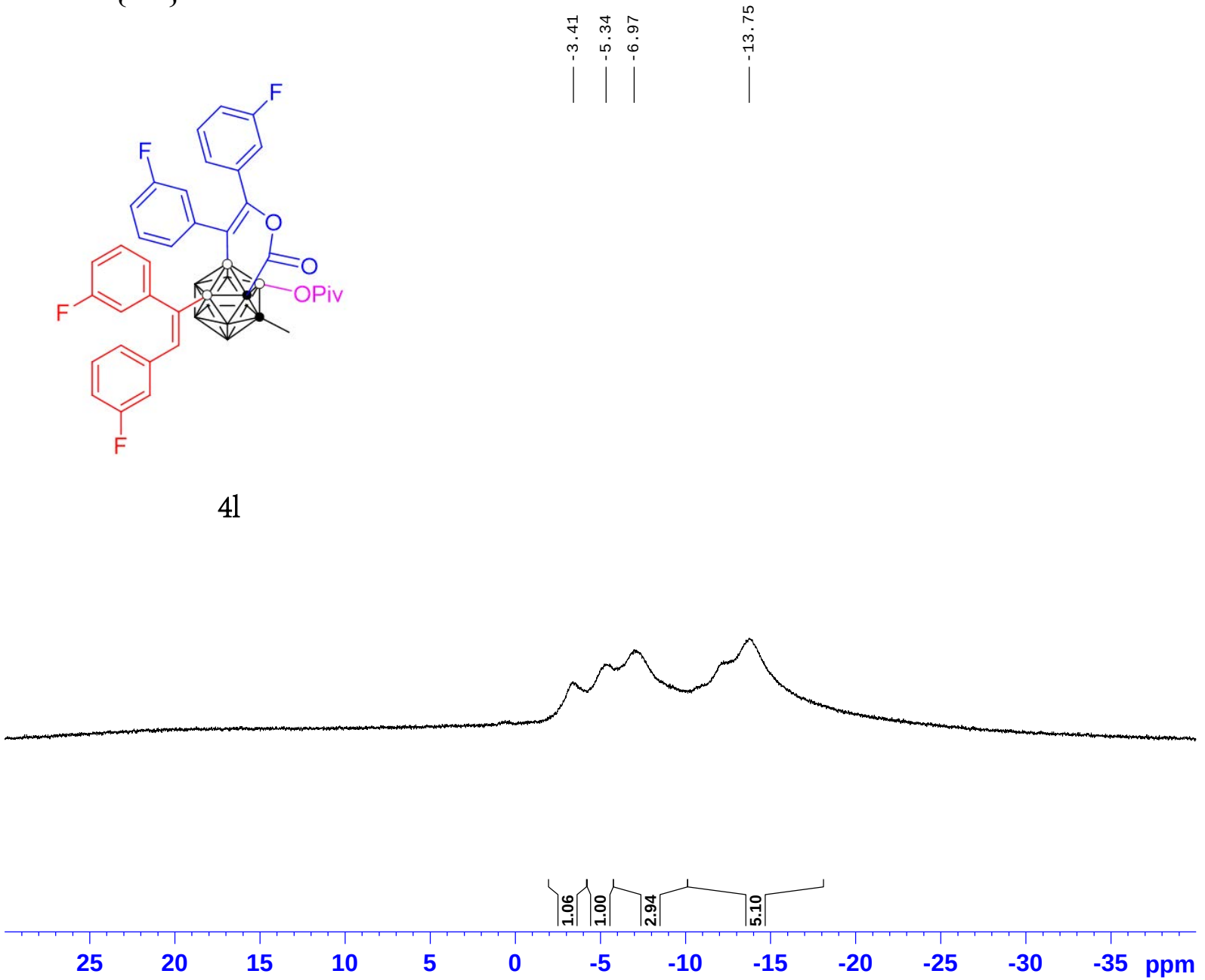
F2 - Processing parameters
SI                32768
SF          125.7577936 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



$^{11}B\{^{1}H\}$ NMR

CB5009-p-B-dc

4l



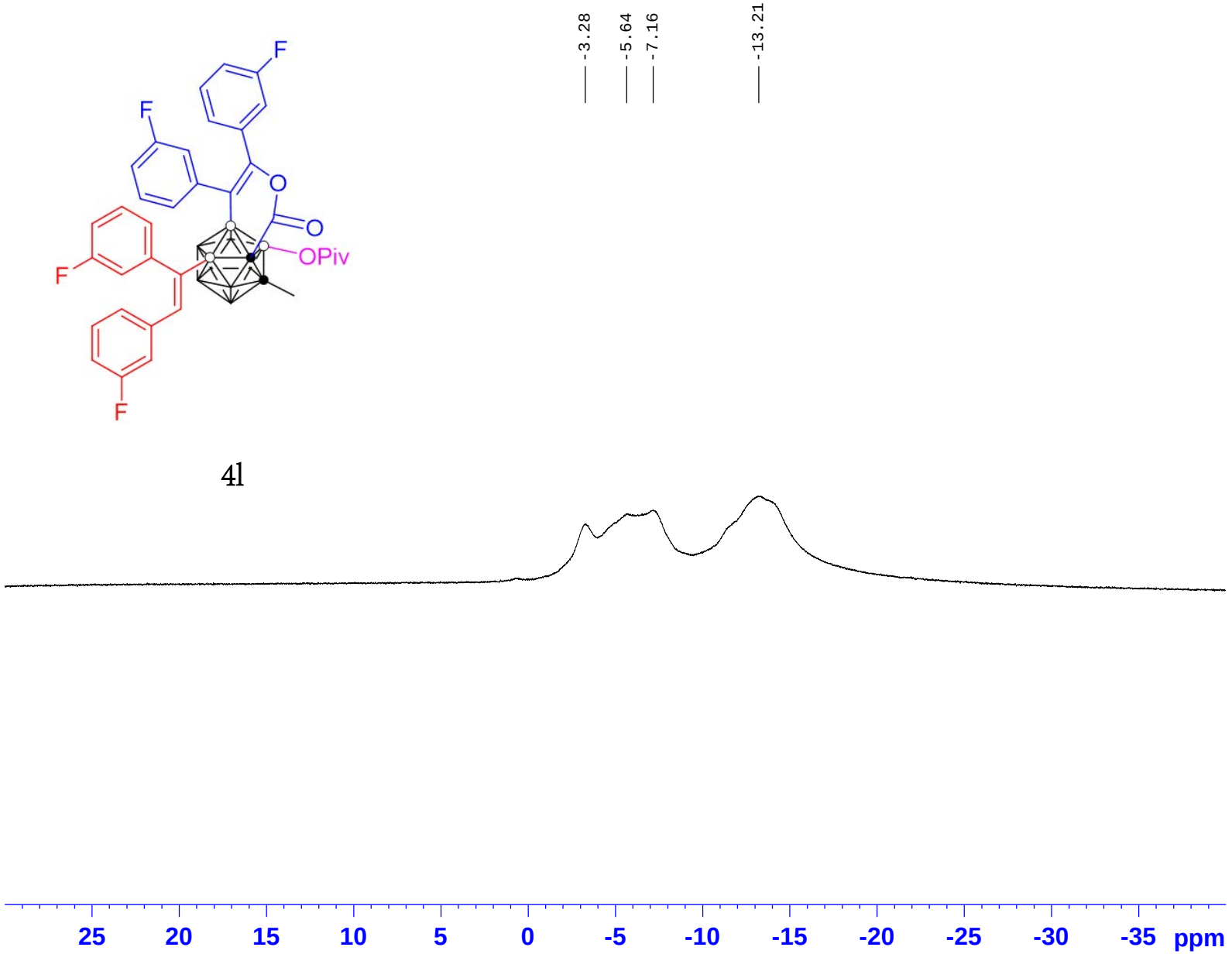

```
Current Data Parameters
NAME      CB5009-p-B-dc
EXPNO                 4
PROCNO                1

F2 - Acquisition Parameters
Date_          20210307
Time             20.50 h
INSTRUM            spect
PROBHD   Z119470_0283 (
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                   32
DS                    4
SWH           24038.461 Hz
FIDRES         1.467191 Hz
AQ            0.6815744 sec
RG               206.72
DW               20.800 usec
DE                6.50 usec
TE               295.1 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        160.4615790 MHz
NUC1                11B
P1                16.00 usec
PLW1        50.00000000 W
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        25.00000000 W
PLW12        0.46495000 W
PLW13        0.23387000 W

F2 - Processing parameters
SI                16384
SF          160.4615999 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

^{11}B NMR

4l



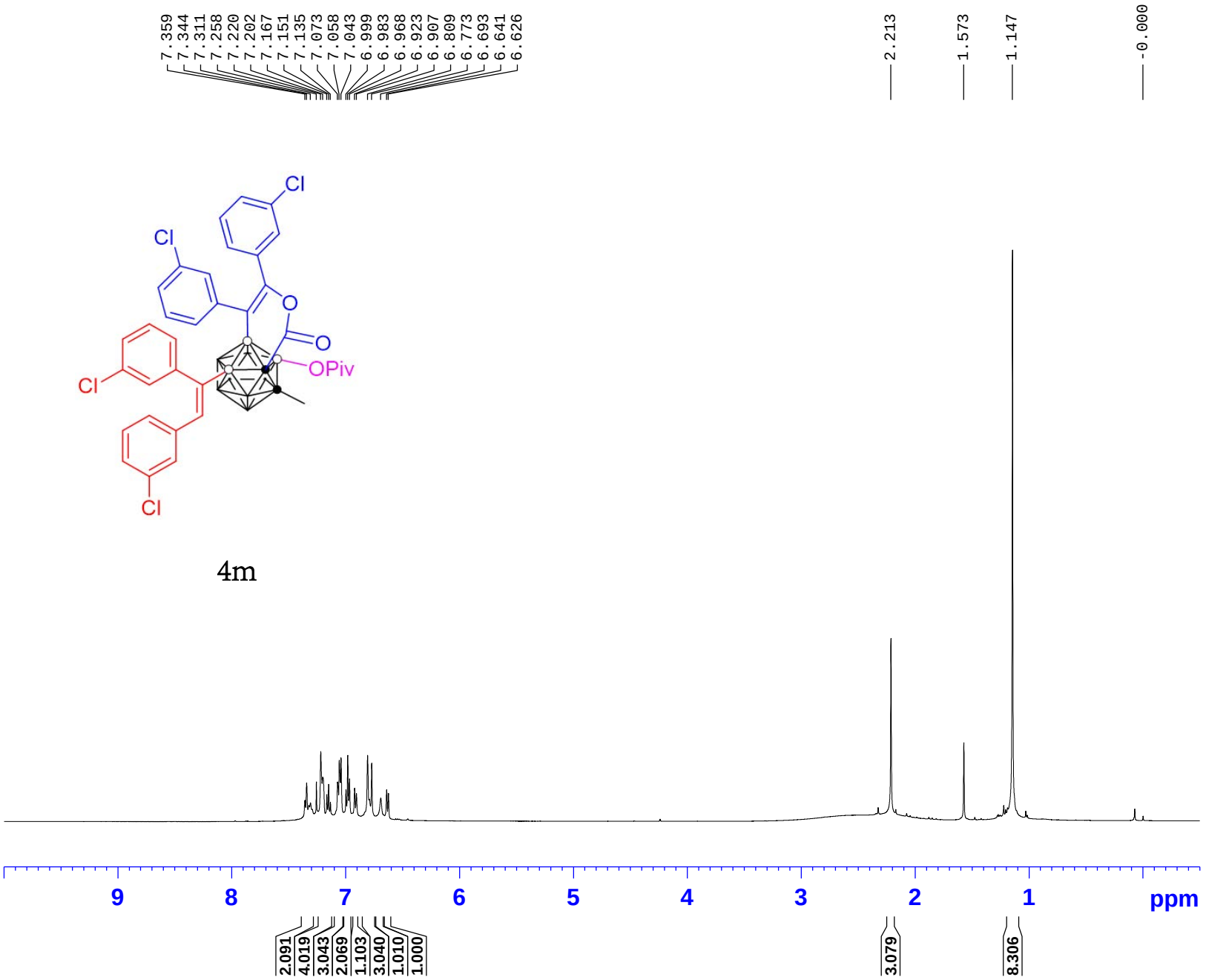
CB5009-p-B-c

```
Current Data Parameters
NAME       CB5009-p-B-c
EXPNO                 9
PROCNO                1

F2 - Acquisition Parameters
Date_          20210307
Time             20.53 h
INSTRUM            spect
PROBHD   Z119470_0283 (
PULPROG              zg
TD                32768
SOLVENT           CDCl3
NS                   64
DS                    4
SWH           24038.461 Hz
FIDRES         1.467191 Hz
AQ            0.6815744 sec
RG               206.72
DW               20.800 usec
DE                6.50 usec
TE               295.2 K
D1           1.00000000 sec
TD0                   1
SFO1        160.4615792 MHz
NUC1                11B
P1                16.00 usec
PLW1        50.00000000 W

F2 - Processing parameters
SI                16384
SF          160.4615997 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

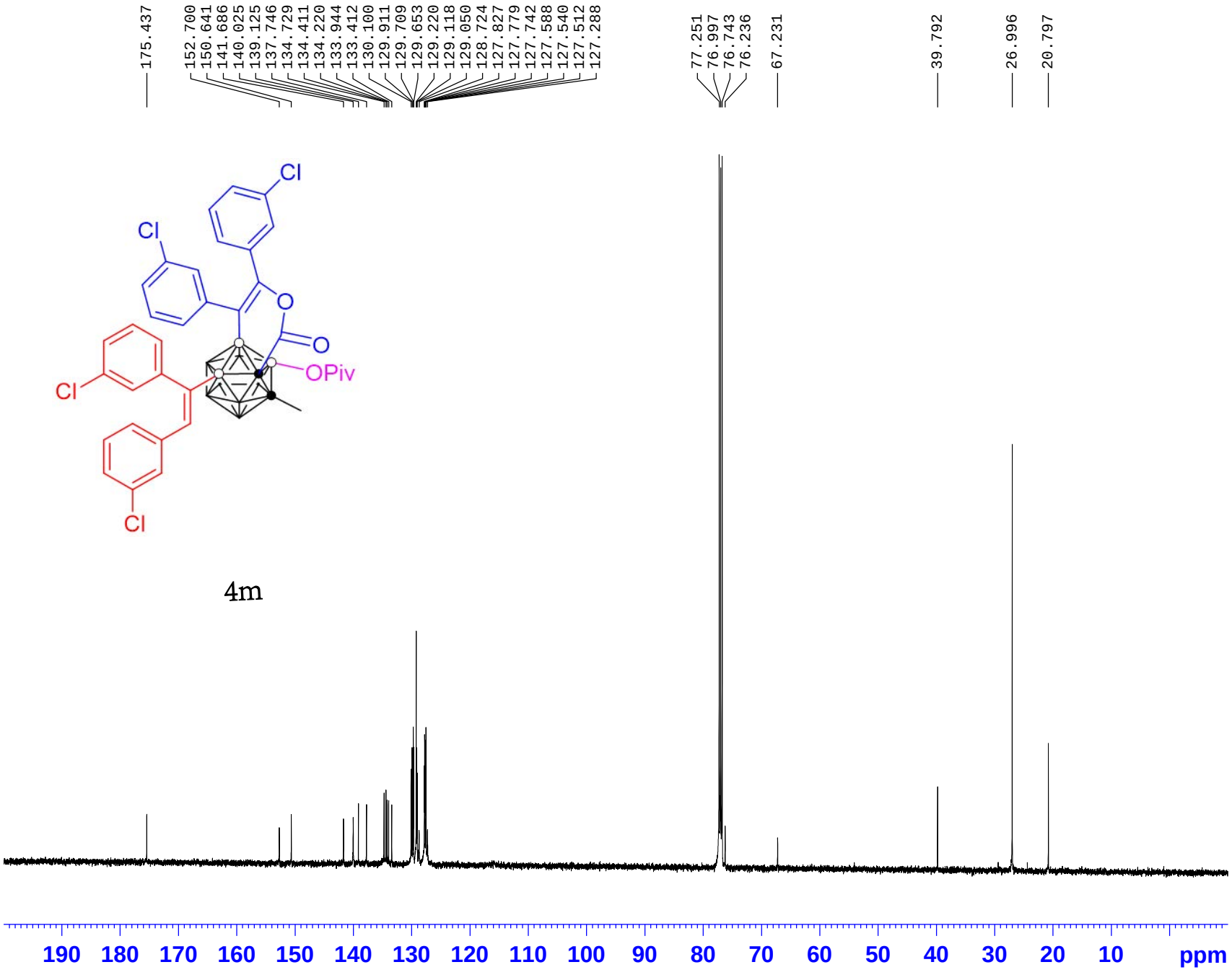
CB5098-p-H




```
Current Data Parameters
NAME          CB5098-p-H
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210307
Time                8.18 h
INSTRUM            spect
PROBHD   Z119470_0283 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                 63.76
DW                50.000 usec
DE                 6.50 usec
TE                295.1 K
D1             1.00000000 sec
TD0                    1
SFO1          500.1330883 MHz
NUC1                  1H
P1                 10.91 usec
PLW1          25.00000000 W

F2 - Processing parameters
SI                 65536
SF            500.1300141 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



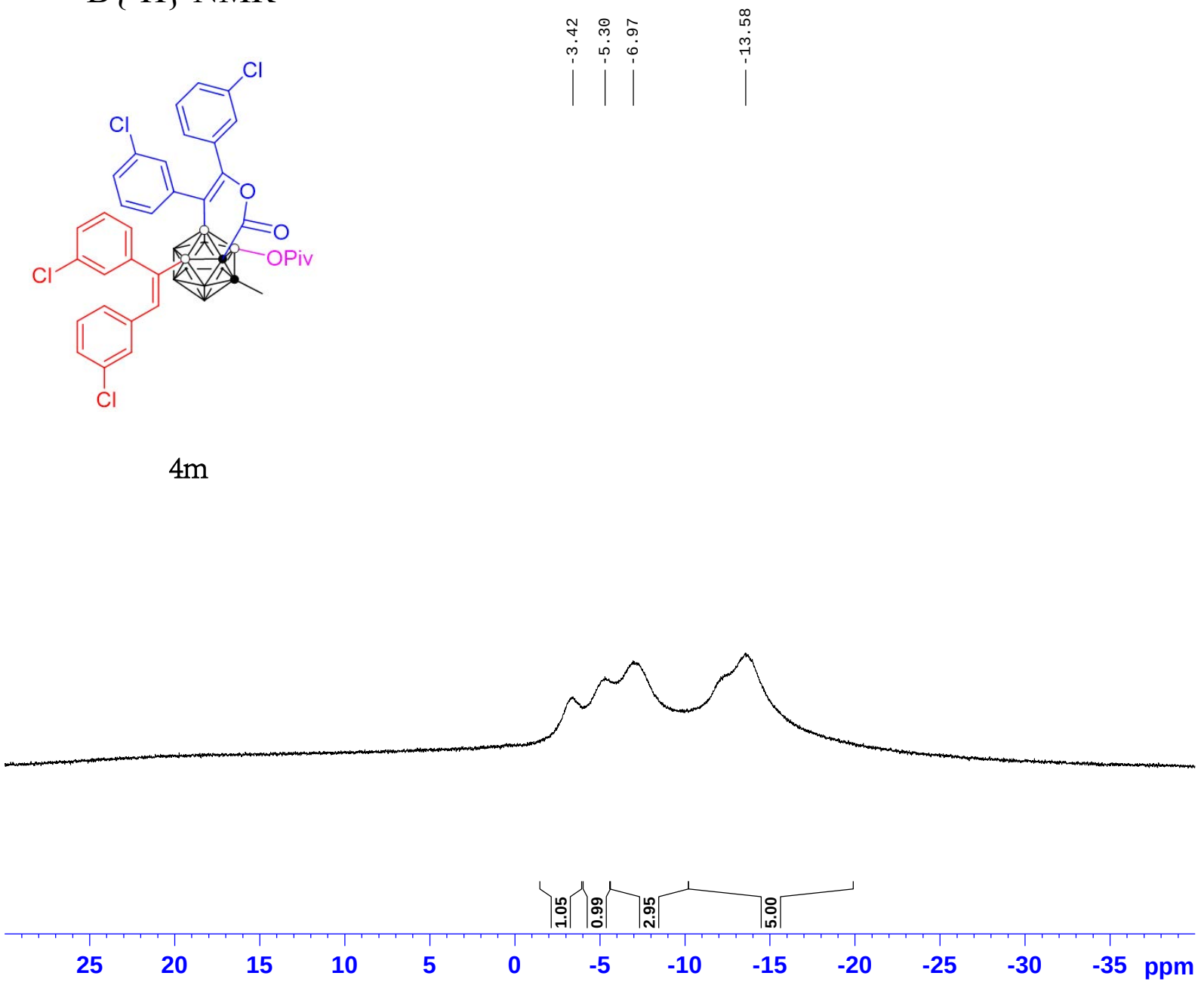

```
Current Data Parameters
NAME          CB5098-p-C
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210307
Time                9.21 h
INSTRUM            spect
PROBHD   Z119470_0283 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH           29761.904 Hz
FIDRES         0.908261 Hz
AQ            1.1010048 sec
RG               206.72
DW               16.800 usec
DE                6.50 usec
TE               295.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1
SFO1        125.7703643 MHz
NUC1               13C
P1                9.75 usec
PLW1        94.00000000 W
SFO2        500.1320005 MHz
NUC2               1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        25.00000000 W
PLW12        0.46495000 W
PLW13        0.23387000 W

F2 - Processing parameters
SI                 32768
SF          125.7577949 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

$^{11}B\{^{1}H\}$ NMR

4m



CB5098-p-B-dc

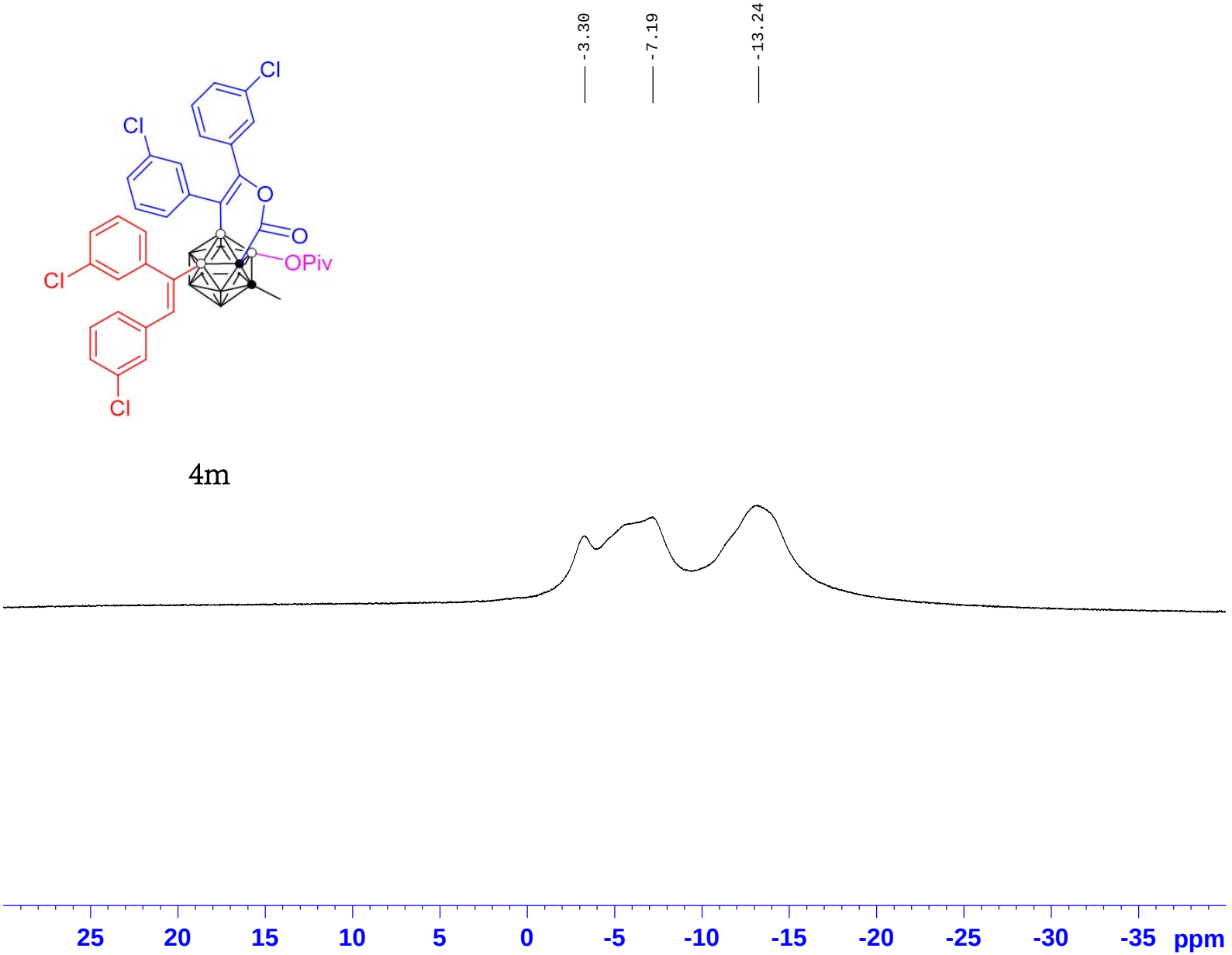
```
Current Data Parameters
NAME      CB5098-p-B-dc
EXPNO                4
PROCNO               1

F2 - Acquisition Parameters
Date_          20210307
Time              8.21 h
INSTRUM           spect
PROBHD   Z119470_0283 (
PULPROG          zgpg30
TD               32768
SOLVENT          CDCl3
NS                  32
DS                   4
SWH          24038.461 Hz
FIDRES        1.467191 Hz
AQ            0.6815744 sec
RG               206.72
DW                20.800 usec
DE                 6.50 usec
TE                295.1 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                  1
SFO1        160.4615790 MHz
NUC1               11B
P1                16.00 usec
PLW1        50.00000000 W
SFO2        500.1320005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2             80.00 usec
PLW2        25.00000000 W
PLW12        0.46495000 W
PLW13        0.23387000 W

F2 - Processing parameters
SI                16384
SF          160.4615999 MHz
WDW                EM
SSB      0
LB                1.00 Hz
GB       0
PC                1.40
```

^{11}B NMR

4m



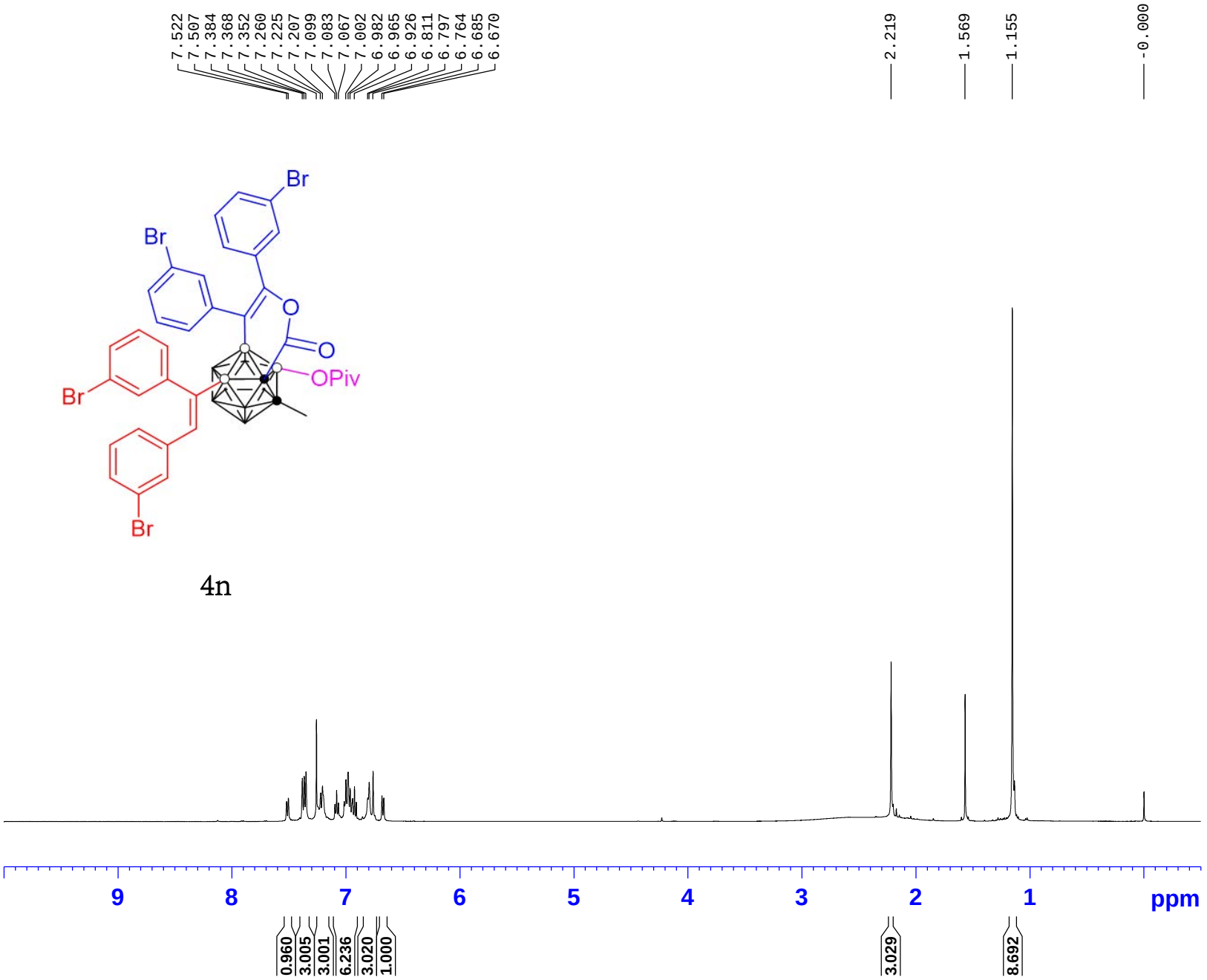
CB5098-p-B-c

```
Current Data Parameters
NAME       CB5098-p-B-c
EXPNO                 5
PROCNO                1

F2 - Acquisition Parameters
Date_          20210307
Time               8.25 h
INSTRUM           spect
PROBHD   Z119470_0283 (
PULPROG              zg
TD                32768
SOLVENT           CDCl3
NS                   64
DS                    4
SWH           24038.461 Hz
FIDRES         1.467191 Hz
AQ            0.6815744 sec
RG               206.72
DW               20.800 usec
DE                6.50 usec
TE               295.2 K
D1           1.00000000 sec
TD0                   1
SFO1        160.4615792 MHz
NUC1               11B
P1               16.00 usec
PLW1        50.00000000 W

F2 - Processing parameters
SI                16384
SF          160.4615997 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

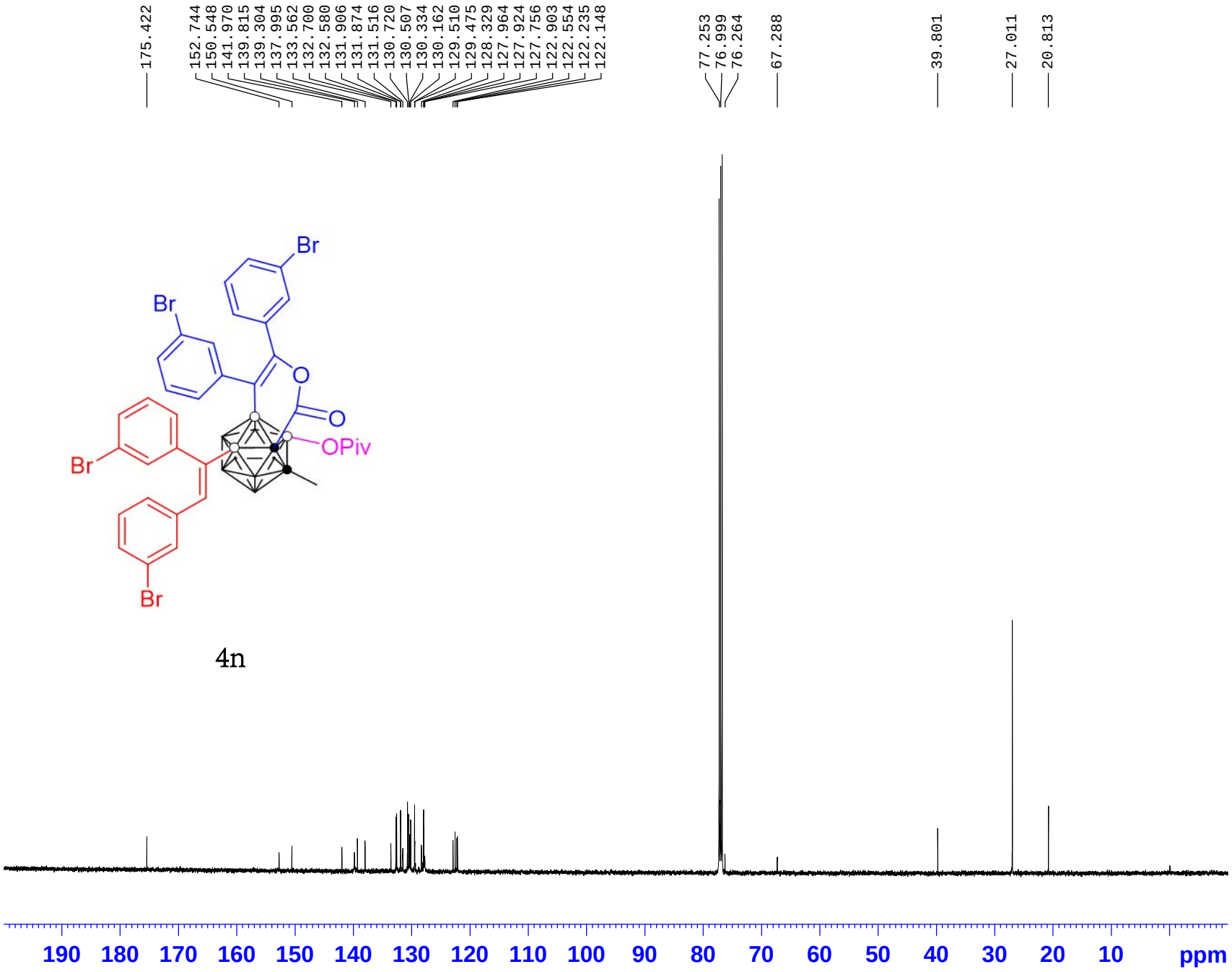
CB5101-p-H




```
Current Data Parameters
NAME          CB5101-p-H
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210307
Time               13.04 h
INSTRUM             spect
PROBHD   Z119470_0283 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            10000.000 Hz
FIDRES          0.305176 Hz
AQ             3.2767999 sec
RG                 56.83
DW                50.000 usec
DE                 6.50 usec
TE                295.2 K
D1             1.00000000 sec
TD0                    1
SFO1          500.1330883 MHz
NUC1                  1H
P1                 10.91 usec
PLW1          25.00000000 W

F2 - Processing parameters
SI                 65536
SF            500.1300117 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



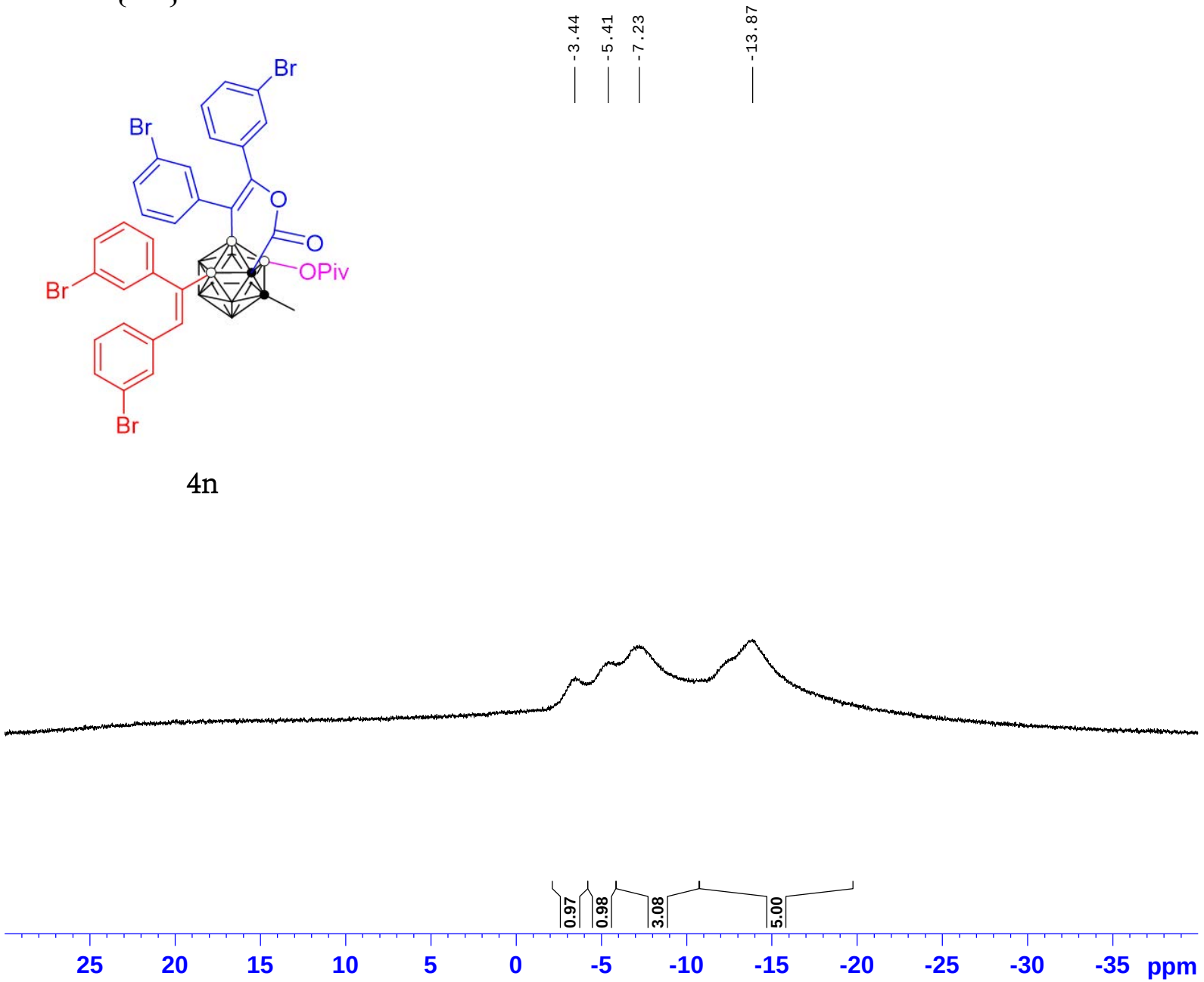

```
Current Data Parameters
NAME          CB5101-p-C
EXPNO                  8
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210307
Time              14.06 h
INSTRUM            spect
PROBHD   Z119470_0283 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           29761.904 Hz
FIDRES         0.908261 Hz
AQ            1.1010048 sec
RG               206.72
DW               16.800 usec
DE                6.50 usec
TE               295.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        125.7703643 MHz
NUC1               13C
P1                9.75 usec
PLW1        94.00000000 W
SFO2        500.1320005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            80.00 usec
PLW2        25.00000000 W
PLW12        0.46495000 W
PLW13        0.23387000 W

F2 - Processing parameters
SI                32768
SF          125.7577936 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

$^{11}B\{^{1}H\}$ NMR

4n



CB5101-p-B-dc

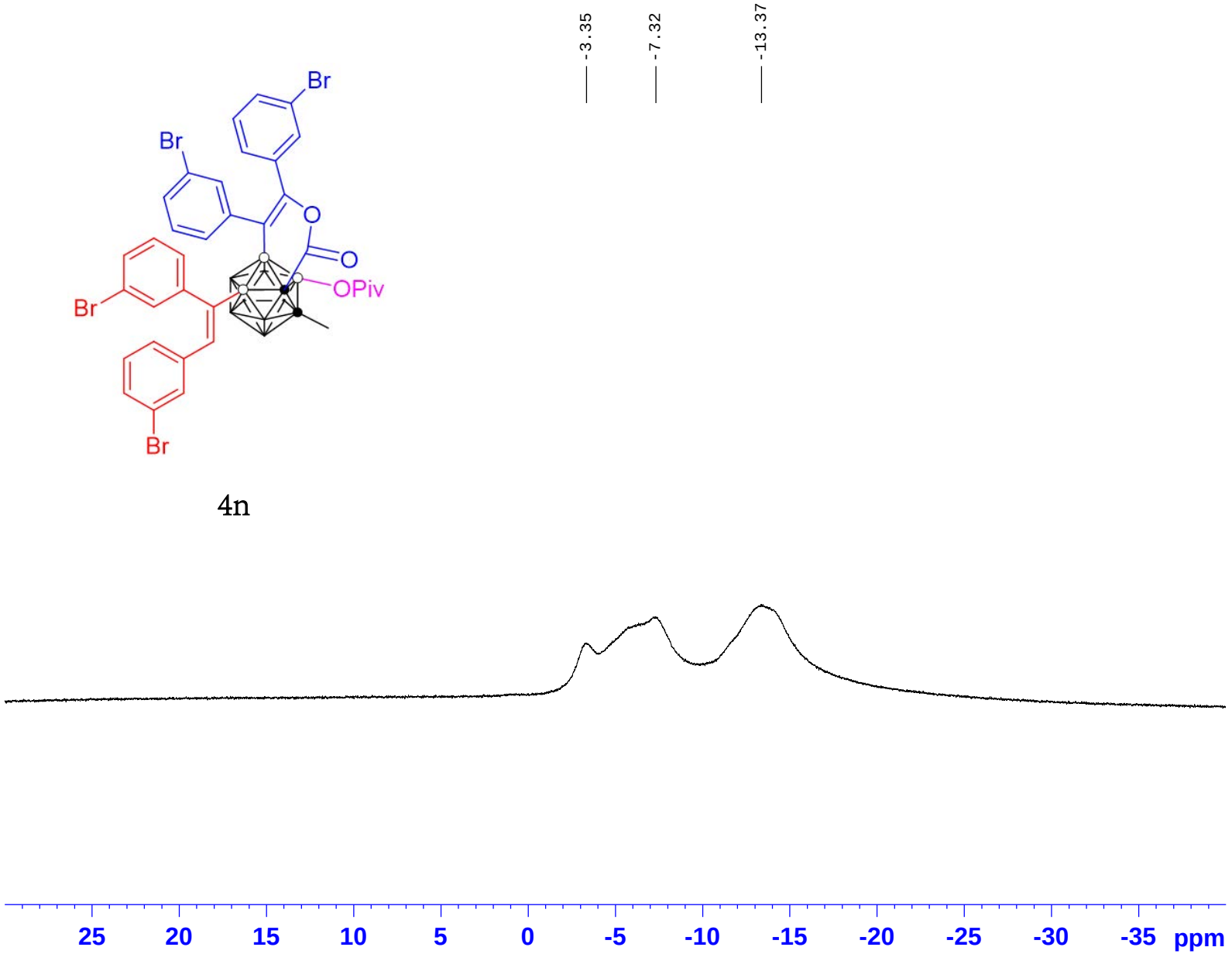
```
Current Data Parameters
NAME      CB5101-p-B-dc
EXPNO                 5
PROCNO                1

F2 - Acquisition Parameters
Date_          20210307
Time             13.07 h
INSTRUM            spect
PROBHD   Z119470_0283 (
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                   32
DS                    4
SWH           24038.461 Hz
FIDRES         1.467191 Hz
AQ            0.6815744 sec
RG               206.72
DW               20.800 usec
DE                6.50 usec
TE               295.1 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        160.4615790 MHz
NUC1                11B
P1                16.00 usec
PLW1        50.00000000 W
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             80.00 usec
PLW2        25.00000000 W
PLW12        0.46495000 W
PLW13        0.23387000 W

F2 - Processing parameters
SI                16384
SF          160.4615999 MHz
WDW                  EM
SSB      0
LB                1.00 Hz
GB       0
PC                1.40
```

^{11}B NMR

4n

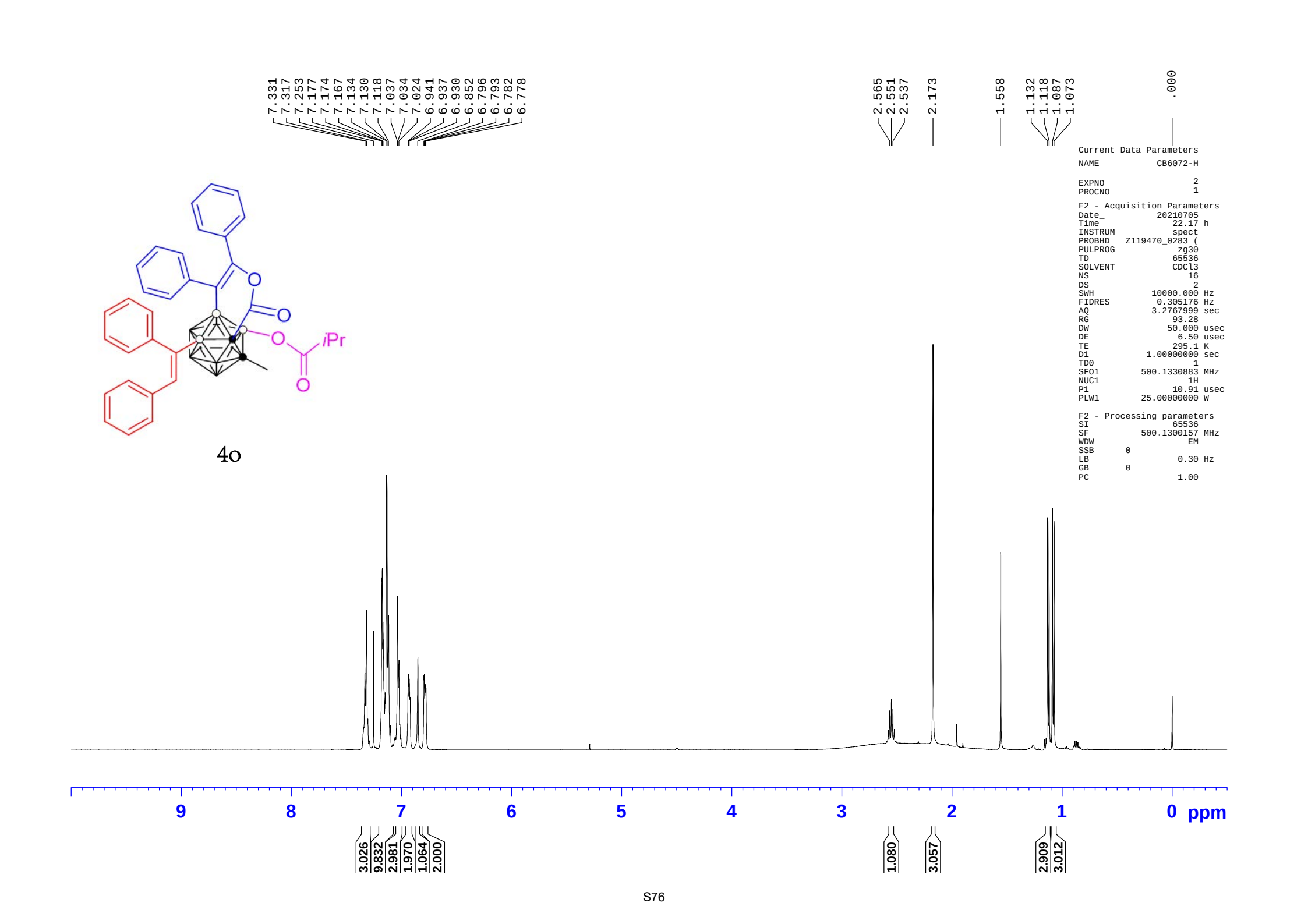


CB5101-p-B-c

```
Current Data Parameters
NAME       CB5101-p-B-c
EXPNO                 9
PROCNO                1

F2 - Acquisition Parameters
Date_          20210307
Time              13.10 h
INSTRUM             spect
PROBHD   Z119470_0283 (
PULPROG               zg
TD                 32768
SOLVENT            CDCl3
NS                    64
DS                     4
SWH           24038.461 Hz
FIDRES         1.467191 Hz
AQ            0.6815744 sec
RG               206.72
DW                20.800 usec
DE                 6.50 usec
TE                295.1 K
D1           1.00000000 sec
TD0                    1
SFO1        160.4615792 MHz
NUC1                11B
P1                16.00 usec
PLW1        50.00000000 W

F2 - Processing parameters
SI                 16384
SF          160.4615997 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```


7.331
7.317
7.253
7.177
7.174
7.167
7.134
7.130
7.118
7.037
7.034
7.024
6.941
6.937
6.930
6.852
6.796
6.793
6.782
6.778
2.565
2.551
2.537
2.173
1.558
1.132
1.118
1.087
1.073
.000
Current Data Parameters
NAME CB6072-H
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210705
Time 22.17 h
INSTRUM spect
PROBHD Z119470_0283 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 93.28
DW 50.000 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P1 10.91 usec
PLW1 25.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300157 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
O
O
O
iPr
9
8
7
6
5
4
3
2
1
0
ppm
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9.832
2.981
1.970
1.064
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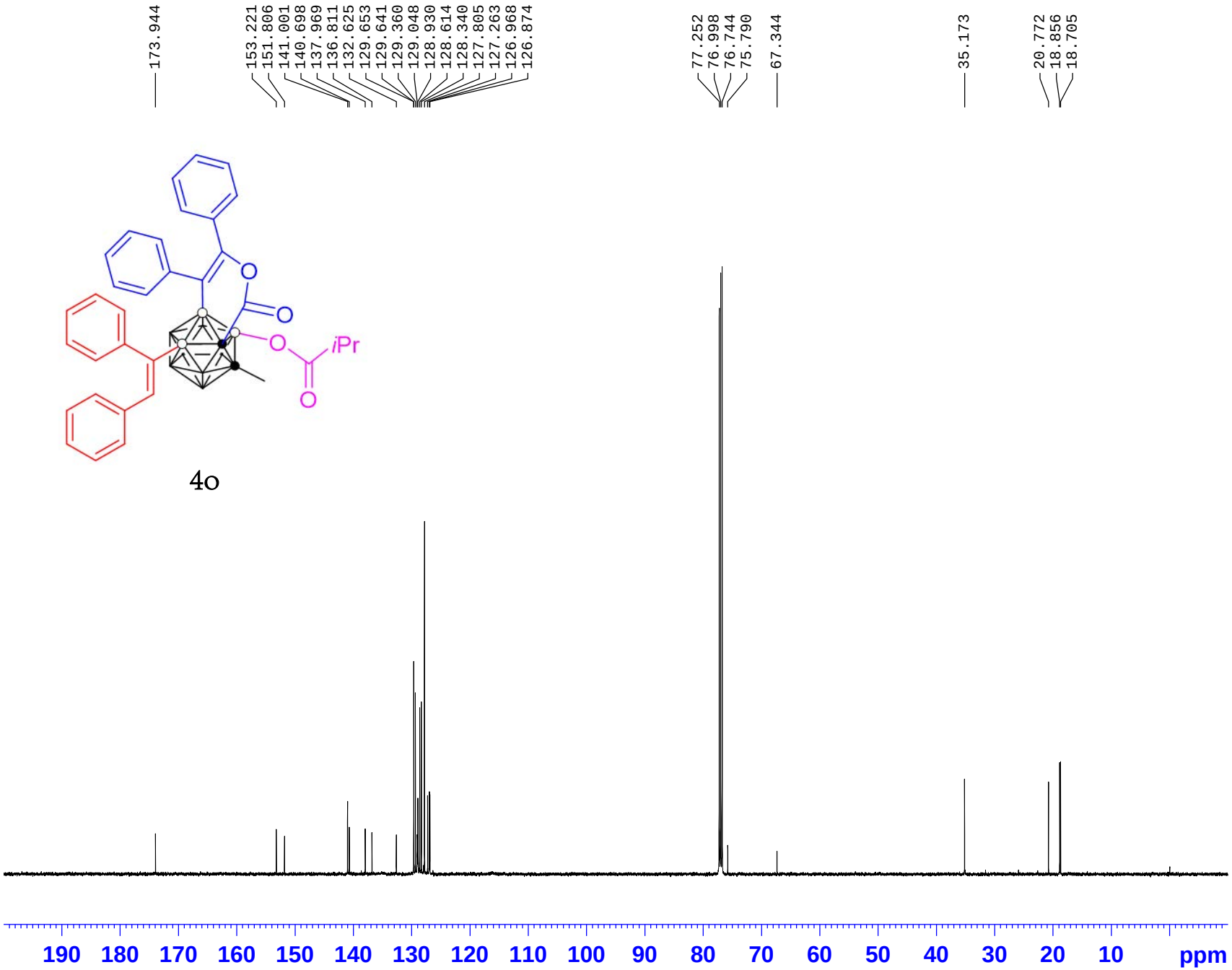
1.080
3.057
2.909
3.012

4o



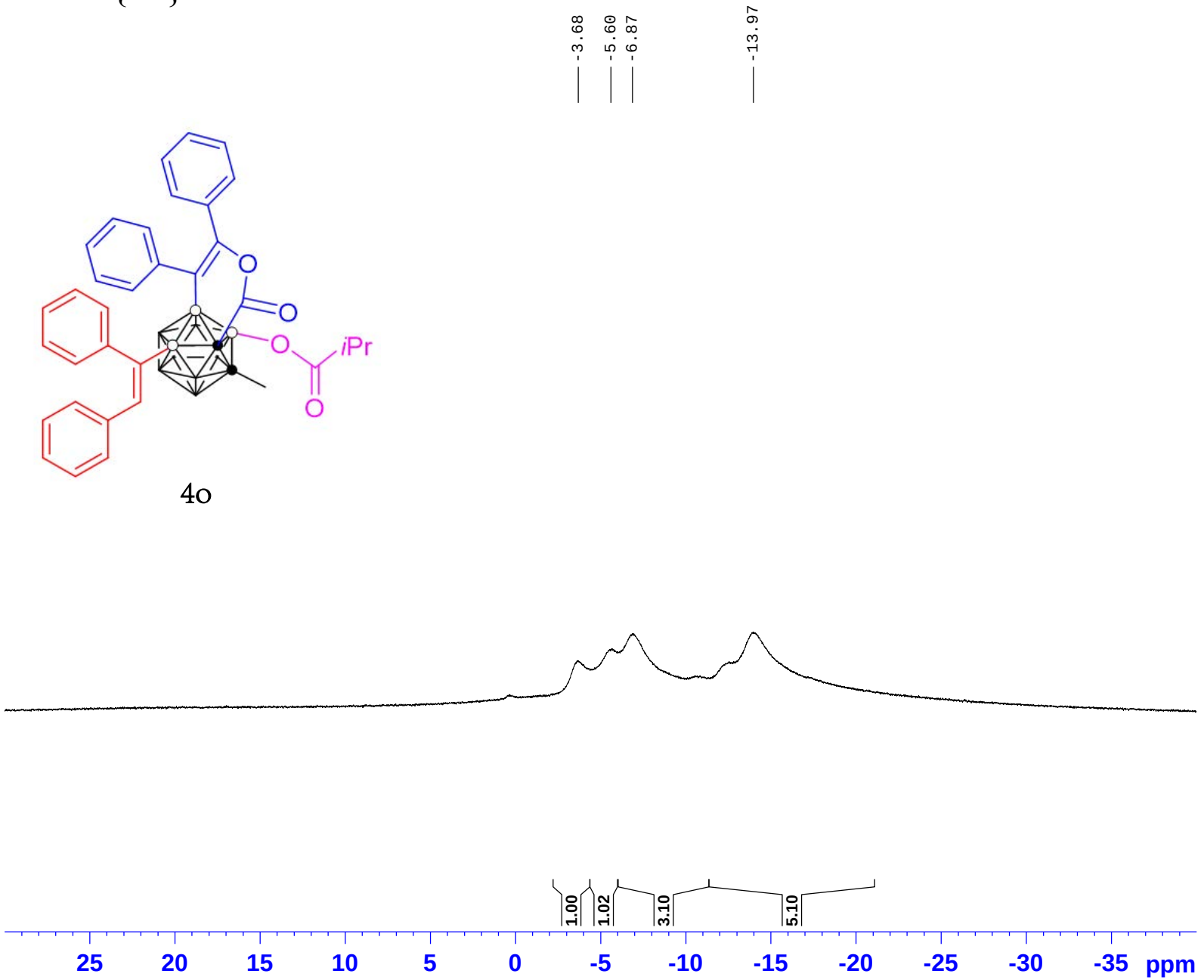

```
Current Data Parameters
NAME            CB6072-C
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210705
Time              23.57 h
INSTRUM            spect
PROBHD   Z119470_0283 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1536
DS                    4
SWH           29761.904 Hz
FIDRES         0.908261 Hz
AQ            1.1010048 sec
RG               206.72
DW               16.800 usec
DE                6.50 usec
TE               295.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        125.7703643 MHz
NUC1               13C
P1                9.75 usec
PLW1        94.00000000 W
SFO2        500.1320005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            80.00 usec
PLW2        25.00000000 W
PLW12        0.46495000 W
PLW13        0.23387000 W

F2 - Processing parameters
SI                32768
SF          125.7577945 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

$^{11}B\{^{1}H\}$ NMR

4o



CB6072-B-dc

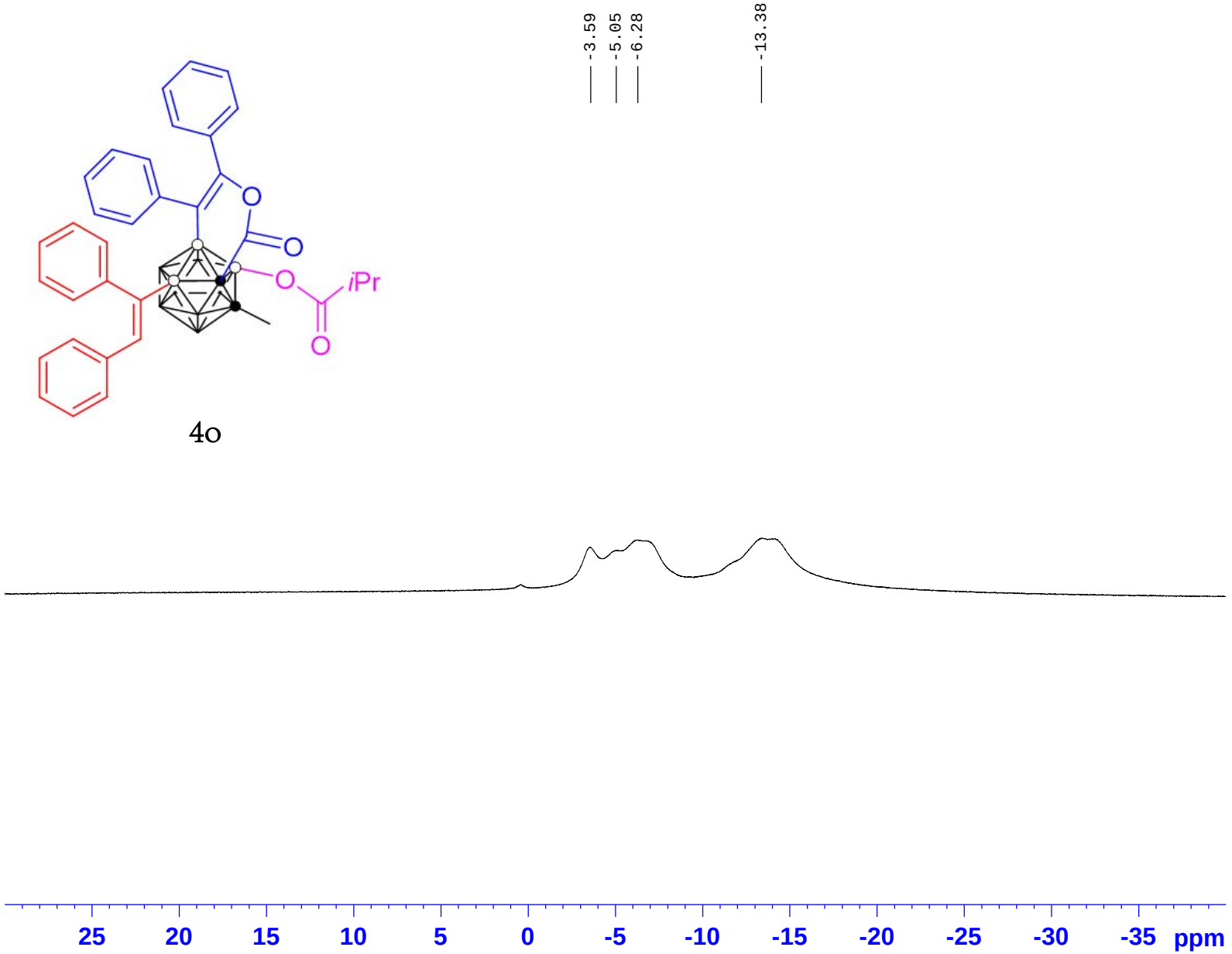
```
Current Data Parameters
NAME         CB6072-B-dc
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210705
Time              22.21 h
INSTRUM             spect
PROBHD   Z119470_0283 (
PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                    64
DS                     4
SWH            24038.461 Hz
FIDRES          1.467191 Hz
AQ              0.6815744 sec
RG                206.72
DW                 20.800 usec
DE                  6.50 usec
TE                295.2 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1         160.4615790 MHz
NUC1                11B
P1                 16.00 usec
PLW1         50.00000000 W
SFO2         500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2              80.00 usec
PLW2         25.00000000 W
PLW12         0.46495000 W
PLW13         0.23387000 W

F2 - Processing parameters
SI                 16384
SF           160.4615999 MHz
WDW                   EM
SSB      0
LB                 1.00 Hz
GB       0
PC                 1.40
```

^{11}B NMR

4o



CB6072-B-c

```
Current Data Parameters
NAME          CB6072-B-c
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210705
Time              22.32 h
INSTRUM            spect
PROBHD   Z119470_0283 (
PULPROG               zg
TD                 32768
SOLVENT            CDCl3
NS                    64
DS                     4
SWH            24038.461 Hz
FIDRES          1.467191 Hz
AQ             0.6815744 sec
RG                206.72
DW                20.800 usec
DE                 6.50 usec
TE                295.1 K
D1           1.00000000 sec
TD0                    1
SFO1         160.4615792 MHz
NUC1                 11B
P1                16.00 usec
PLW1         50.00000000 W

F2 - Processing parameters
SI                 16384
SF           160.4615997 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
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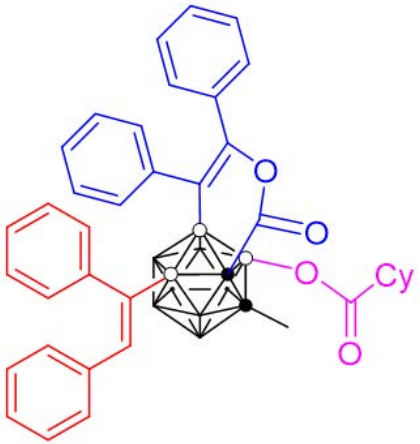


4p

7.326 7.312 7.260 7.173 7.169 7.162 7.133 7.119 7.040 7.037 7.026 6.900 6.896 6.889 6.881 6.866 6.802 6.798 6.788 6.783

2.288 2.281 2.170 1.884 1.880 1.853 1.824 1.820 1.674 1.659 1.644 1.566 1.360 1.355 1.303 1.297 1.279 1.273 1.162 1.138 1.083 .000

Current Data Parameters

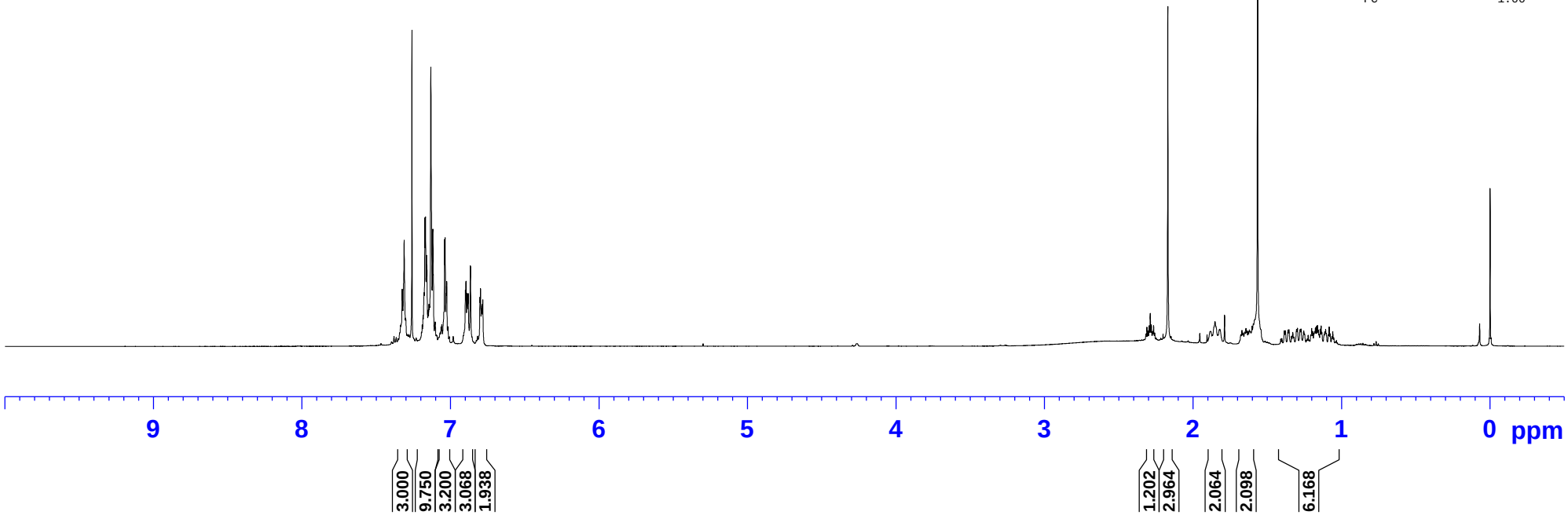
NAME	CB6074-H
EXPNO	3
PROCNO	1

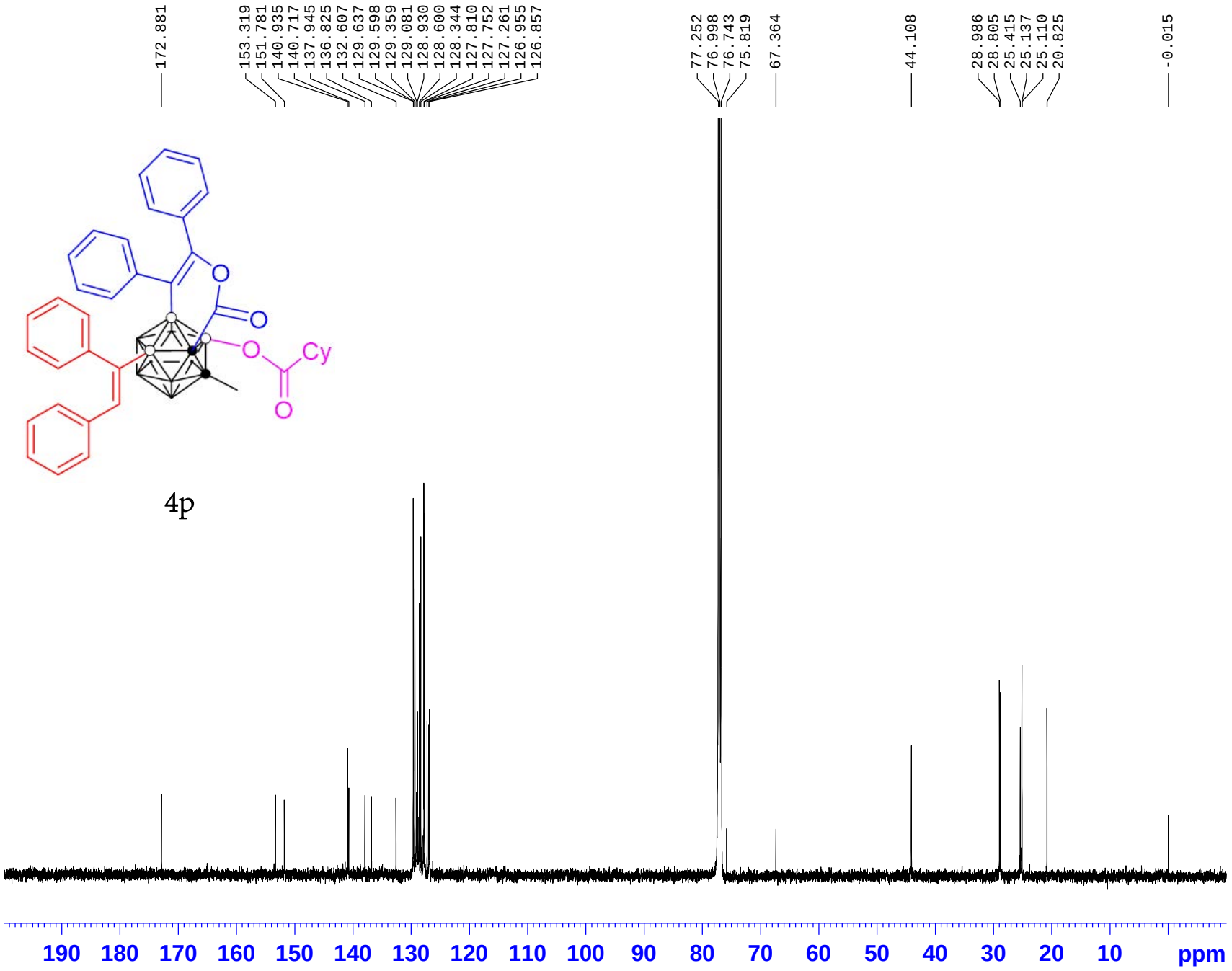
F2 - Acquisition Parameters

Date_	20210706
Time	0.23 h
INSTRUM	spect
PROBHD	Z119470_0283 (
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	32
DS	2
SWH	10000.000 Hz
FIDRES	0.305176 Hz
AQ	3.2767999 sec
RG	163.99
DW	50.000 usec
DE	6.50 usec
TE	295.1 K
D1	1.00000000 sec
TD0	1
SFO1	500.1330883 MHz
NUC1	1H
P1	10.91 usec
PLW1	25.00000000 W

F2 - Processing parameters

SI	65536
SF	500.1300124 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



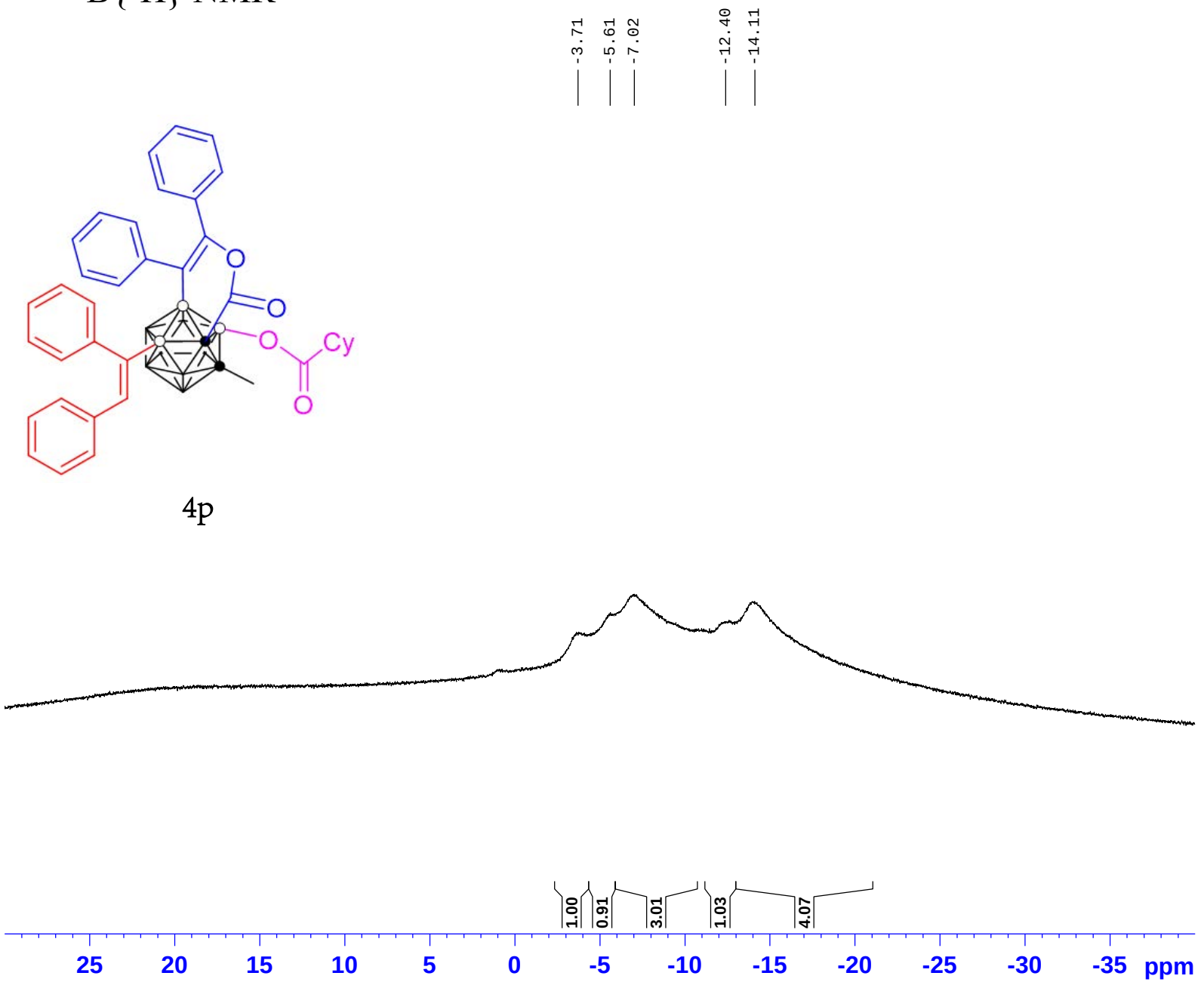



```
Current Data Parameters
NAME            CB6074-C
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210706
Time                7.22 h
INSTRUM             spect
PROBHD   Z119470_0283 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  7168
DS                     4
SWH            29761.904 Hz
FIDRES          0.908261 Hz
AQ              1.1010048 sec
RG                206.72
DW                16.800 usec
DE                 6.50 usec
TE                295.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1          125.7703643 MHz
NUC1                13C
P1                  9.75 usec
PLW1          94.00000000 W
SFO2          500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2              80.00 usec
PLW2          25.00000000 W
PLW12          0.46495000 W
PLW13          0.23387000 W

F2 - Processing parameters
SI                 32768
SF            125.7577931 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

$^{11}B\{^{1}H\}$ NMR

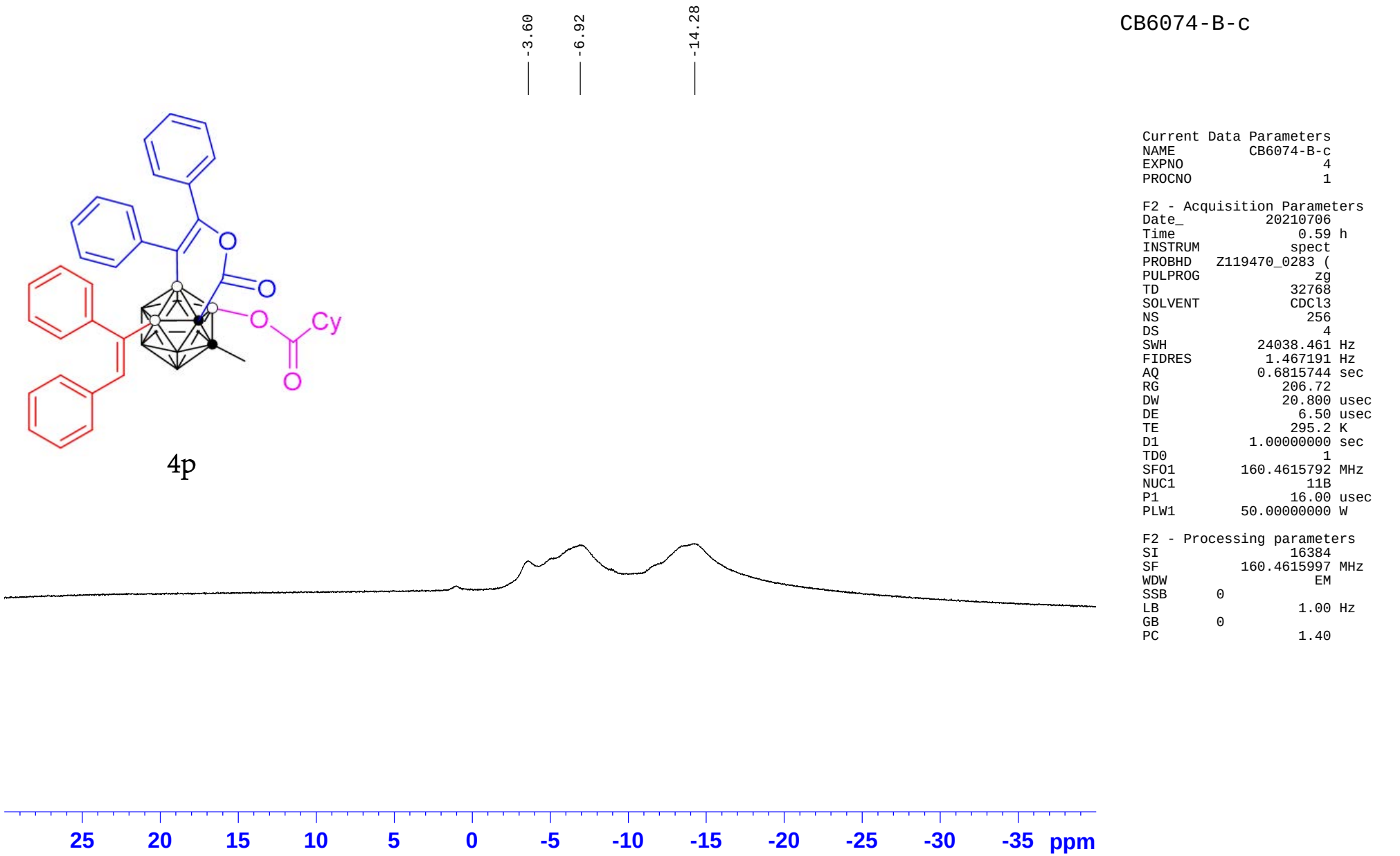
CB6074-B-dc

4p




```
Current Data Parameters
NAME         CB6074-B-dc
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210706
Time                0.45 h
INSTRUM            spect
PROBHD   Z119470_0283 (
PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   256
DS                     4
SWH            24038.461 Hz
FIDRES          1.467191 Hz
AQ              0.6815744 sec
RG                206.72
DW                20.800 usec
DE                 6.50 usec
TE                295.2 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1         160.4615790 MHz
NUC1                11B
P1                16.00 usec
PLW1         50.00000000 W
SFO2         500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2         25.00000000 W
PLW12         0.46495000 W
PLW13         0.23387000 W

F2 - Processing parameters
SI                 16384
SF           160.4615999 MHz
WDW                   EM
SSB      0
LB                 1.00 Hz
GB       0
PC                 1.40
```

^{11}B NMR



4p

CB6074-B-c

```
Current Data Parameters
NAME          CB6074-B-c
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210706
Time                0.59 h
INSTRUM            spect
PROBHD   Z119470_0283 (
PULPROG               zg
TD                 32768
SOLVENT            CDCl3
NS                   256
DS                     4
SWH            24038.461 Hz
FIDRES          1.467191 Hz
AQ             0.6815744 sec
RG                206.72
DW                20.800 usec
DE                 6.50 usec
TE                295.2 K
D1            1.00000000 sec
TD0                    1
SFO1         160.4615792 MHz
NUC1                 11B
P1                 16.00 usec
PLW1         50.00000000 W

F2 - Processing parameters
SI                 16384
SF           160.4615997 MHz
WDW                  EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```