Electronic Supplementary Material (ESI) for ChemComm.

Electronic Supplementary Information

Transition Metal Catalyzed Selective B(3)-H or B(4)-H Amination of *o*-Carboranes via Dehydrogenative BH/NH Cross-Coupling

Hairong Lyu 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

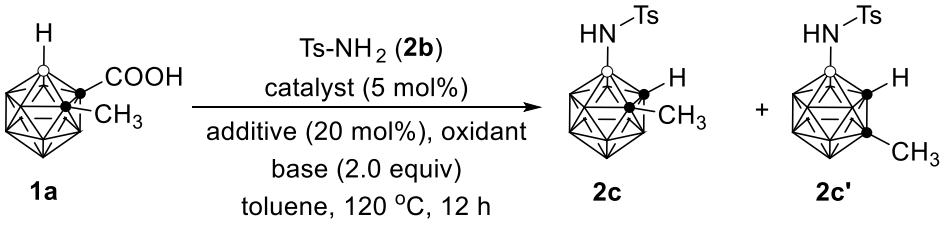
Table of Contents

1. General Procedures

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. All organic solvents were freshly distilled from sodium benzophenone ketyl immediately prior to use. ^{1}H, ^{13}C, ^{11}B and ^{19}F NMR spectra were recorded on a Bruker DPX 400/500 spectrometer at 400/500, 100/125, 128/160 and 376/470 MHz, respectively. All chemical shifts were reported in δ units with references to the residual solvent resonances of the deuterated solvents for proton and carbon chemical shifts, to external $CFCl_3$ (0.00) for fluorine chemical shifts, and to external $BF_3{\cdot}OEt_2$ (0.00 ppm) for boron chemical shifts. 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. Carboranyl carboxylic acids **a**[1] and 3-phenyl-*o*-carborane[2] were prepared according to literature methods. All other chemicals were purchased from either Aldrich or Acros Chemical Co. and used as received unless otherwise specified. Elemental analyses were performed by the Shanghai Institute of Organic Chemistry, CAS, China or MEDAC Ltd., U. K.

2. Experimental Section

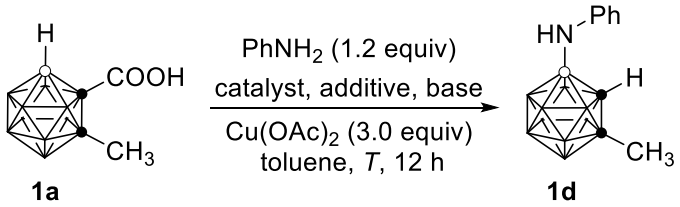
Table S1. Optimization of reaction conditions using tosylamide[a]



entry	catalyst	additive	oxidant (equiv)	base	**2c** (%)[b]	**2c'** (%)[b]
1	[Rh]	$AgSbF_6$	$Cu(OAc)_2$ (2)	K_2HPO_4	25	4
2	[Rh]	$AgNTf_2$	$Cu(OAc)_2$ (2)	K_2HPO_4	49	8
3	[Rh]	$AgNTf_2$	$Cu(OTf)_2$ (2)	K_2HPO_4	trace	trace
4	[Rh]	$AgNTf_2$	Ag_2CO_3 (1)	K_2HPO_4	10	5
5	[Rh]	$AgNTf_2$	$Cu(OAc)_2$ (3)	K_2HPO_4	51	9
6	[Ir]	$AgNTf_2$	$Cu(OAc)_2$ (2)	K_2HPO_4	6	12
7	[Ru]	$AgNTf_2$	$Cu(OAc)_2$ (2)	K_2HPO_4	3	16
8	[Rh]	$AgNTf_2$	$Cu(OAc)_2$ (2) + TEMPO (1)	K_2HPO_4	65	3
9	[Rh]	$AgNTf_2$	$Cu(OAc)_2$ (2) + TEMPO (1)	NaOAc	85	3
10[c]	[Rh]	$AgNTf_2$	$Cu(OAc)_2$ (2) + TEMPO (1)	NaOAc	84	2
11[d]	[Rh]	$AgNTf_2$	$Cu(OAc)_2$ (2) + TEMPO (1)	NaOAc	73	2
12[c,e]	[Rh]	$AgNTf_2$	$Cu(OAc)_2$ (2) + TEMPO (1)	NaOAc	83	2

[a]Reaction conditions: **1a** (0.05 mmol) and tosylamide (0.20 mmol) in of toluene in a closable flask; $AgSbF_6$ = silver hexafluoroantimonate(V); $AgNTf_2$ = silver bis(trifluoromethanesulfonyl)imide; [Rh] = $[Cp^*RhCl_2]_2$; [Ir] = $[Cp^*IrCl_2]_2$; [Ru] = [Ru(*p*-cymene)$Cl_2]_2$; TEMPO = (2,2,6,6-Tetramethylpiperidin-1-yl)oxyl; [b]Yield determined by GC-MS; [c]2.0 equiv of $TsNH_2$ was used; [d]1.2 equiv of $TsNH_2$ was used; [e]3 mol% of $[Cp^*RhCl_2]_2$ was used.

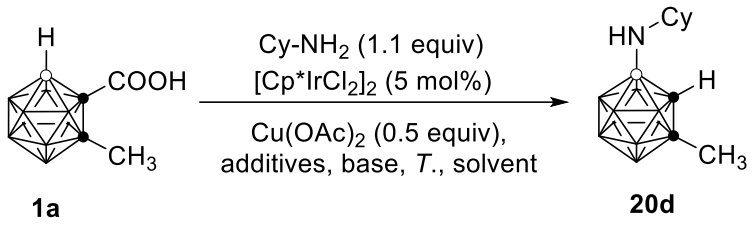
Table S2. Optimization of reaction conditions using aniline[a]



entry	catalyst (5 mol%)	additive (mol%)	base (equiv)	*T* (ºC)	**1d** (%)[b]
1	[Ir]	-	Li_2CO_3 (2)	140	27
2	[Ir]	AgOAc (20)	Li_2CO_3 (2)	140	60
3	[Ir]	$AgNTf_2$ (20)	Li_2CO_3 (2)	140	20
4	[Ir]	AgOAc (10) + $AgNTf_2$ (10)	Li_2CO_3 (2)	140	96
5	[Ir]	AgOAc (10) + $AgNTf_2$ (10)	Li_2CO_3 (2)	120	97
6	[Ir]	AgOAc (10) + $AgNTf_2$ (10)	Li_2CO_3 (2)	100	88
7	[Ir]	AgOAc (10) + $AgNTf_2$ (10)	Li_2CO_3 (2)	80	40
8	[Ir]	AgOAc (10) + $AgNTf_2$ (10)	NaOAc (2)	120	66
9	[Ir]	AgOAc (10) + $AgNTf_2$ (10)	-	120	33
10[c]	[Ir]	AgOAc (10) + $AgNTf_2$ (10)	Li_2CO_3 (2)	120	96
11	[Rh]	AgOAc (10) + $AgNTf_2$ (10)	Li_2CO_3 (2)	120	trace

[a]Reaction conditions: **1a** (0.05 mmol) and aniline (0.06 mmol) in 1.0 mL of toluene in a closed flask; $AgNTf_2$ = silver bis(trifluoromethanesulfonyl)imide; [Ir] = $[Cp^*IrCl_2]_2$; [Rh] = $[Cp^*RhCl_2]_2$; [b]Yields determined by GC-MS. [c]3 mol% of $[Cp^*IrCl_2]_2$ was used.

Table S3. Optimization of reaction conditions using cyclohexylamine[a]

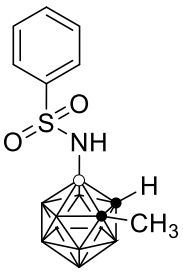


entry	additives (equiv)	base (equiv)	*T* (°C)	solvent	**20d** (%)[b]
1[c]	AgOAc (0.1) + $AgNTf_2$ (0.1)	Li_2CO_3 (2)	120	toluene	0
2[c]	AgOAc (0.1) + $AgNTf_2$ (0.1)	Li_2CO_3 (2)	120	THF	0
3[c]	AgOAc (0.1) + $AgNTf_2$ (0.1)	Li_2CO_3 (2)	120	DCE	trace
4[c]	AgOAc (1) + $AgNTf_2$ (1)	Li_2CO_3 (2)	120	DCE	53
5	AgOAc (1) + $AgNTf_2$ (1)	Li_2CO_3 (2)	120	DCE	70
6[d]	AgOAc (1) + $AgNTf_2$ (1)	Li_2CO_3 (2)	120	DCE	trace
7	AgOAc (2) + $AgNTf_2$ (1)	Li_2CO_3 (2)	120	DCE	77
8	AgOAc (3) + $AgNTf_2$ (1)	Li_2CO_3 (2)	120	DCE	96
9	AgOAc (3) + $AgNTf_2$ (1)	-	120	DCE	94
10	AgOAc (3) + $AgNTf_2$ (1)	-	90	DCE	93
11	AgOAc (3) + $AgNTf_2$ (1)	-	50	DCE	93
12	AgOAc (3) + $AgNTf_2$ (1)	-	r.t.	DCE	50
13	AgOAc (3) + $AgNTf_2$ (0.2)	-	50	DCE	49
14	AgOAc (3) + $AgNTf_2$ (0.5)	-	50.	DCE	71
15	AgOAc (3)	-	50	DCE	trace
16[e]	AgOAc (3) + $AgNTf_2$ (1)	-	50	DCE	82

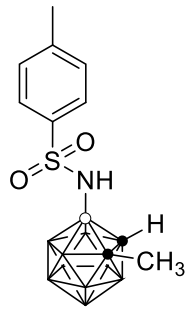
[a]Reaction conditions: **1a** (0.050 mmol) and cyclohexylamine (0.055 mmol) in 1.0 mL of solvent in a closable flask; $AgNTf_2$ = silver bis(trifluoromethanesulfonyl)imide; [b]Yield determined by GC-MS. [c]3.0 equiv $Cu(OAc)_2$ was added. [d]In the absence of $Cu(OAc)_2$. [e]3 mol% of $[Cp^*IrCl_2]_2$ was used.

Preparation of B(3)-sulfonamino-*o*-carboranes (c).

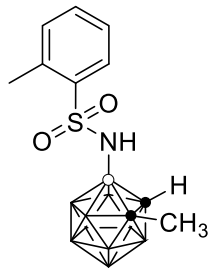
A representative procedure. 1-COOH-*o*-carborane **a** (0.10 mmol), sulfonamides **b** (2.0 equiv, 0.20 mmol), $[Cp^*RhCl_2]_2$ (1.9 mg, 3.0 mol%, 0.003 mmol), $AgNTf_2$ (7.8 mg, 20 mol%, 0.02 mmol), $Cu(OAc)_2$ (36.3 mg, 2.0 equiv, 0.20 mmol), TEMPO (15.6 mg, 1.0 equiv, 0.10 mmol), and NaOAc (16.4 mg, 2.0 equiv, 0.20 mmol) were mixed in dry toluene (2.0 mL). The resulting mixture was heated in a closed flask at 120 °C for 12 h under argon. Then, the reaction solution was filtered through a pad of Celite and washed with diethyl ether. The organic portions were combined. After removal of organic solvents under reduced pressure, the residue was subjected to flash column chromatography on silica gel (230-400 mesh) using a mixture of *n*-hexane, dichloromethane and ethyl acetate (20/10/1 in v/v) as eluent to give the product **c**.



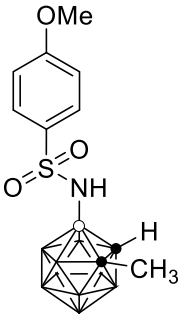
1c: Yield 71% (22.2 mg). White solid. ^{1}H NMR (400 MHz, $CDCl_3$): 7.94 (d, *J* = 7.6 Hz, 2H), 7.64 (t, *J* = 7.4 Hz, 1H), 7.57 (m, 2H) (aryl C*H*), 5.63 (s, 1H) (N*H*), 4.21 (s, 1H) (cage C*H*), 2.07 (s, 3H) (CH_3). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 141.2, 133.3, 129.4, 126.8 (aryl *C*), 71.8, 60.9 (cage *C*), 23.0 (*C*H_3). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -4.0 (1B), -5.0 (2B) (*B*N and *B*H), -8.9 (1B), -10.2 (3B), -13.0 (2B), -14.0 (1B). HRMS: *m*/*z* calcd for $C_9H_{18}{}^{10}B_2{}^{11}B_8NO_2S^-$ [M-H]$^-$: 312.2072. Found: 312.2068.



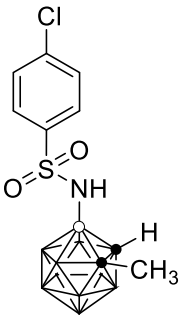
2c: Yield 81% (26.5 mg). Colorless crystals. ^{1}H NMR (400 MHz, $CDCl_3$): 7.81 (d, *J* = 8.4 Hz, 2H), 7.35 (d, *J* = 8.4 Hz, 2H) (aryl C*H*), 5.31 (s, 1H) (N*H*), 4.24 (s, 1H) (cage C*H*), 2.46 (s, 3H), 2.10 (s, 3H) (CH_3). ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 144.2, 138.3, 130.0, 126.8 (aryl *C*), 71.8, 60.8 (cage *C*), 23.0, 21.8 (*C*H_3). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -5.3 (1B), -6.2 (2B) (*B*N and *B*H), -10.2 (1B), -11.6 (3B), -14.4 (2B), -15.2 (1B). HRMS: *m*/*z* calcd for $C_{10}H_{20}{}^{10}B_2{}^{11}B_8NO_2S^-$ [M-H]$^-$: 326.2229. Found: 326.2223.



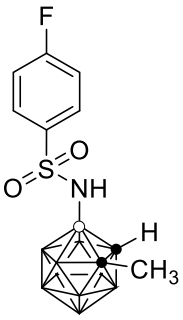
3c: Yield 67% (22.0 mg). White solid. ^{1}H NMR (400 MHz, $CDCl_3$): 7.99 (d, *J* = 7.6 Hz, 1H), 7.51 (m, 1H), 7.36 (m, 2H) (aryl C*H*), 5.69 (s, 1H) (N*H*), 4.15 (s, 1H) (cage C*H*), 2.74 (s, 3H), 2.09 (s, 3H), (CH_3). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 139.1, 136.7, 133.4, 132.8, 128.7, 126.5 (aryl *C*), 61.8, 60.8 (cage *C*), 22.9, 20.3 (*C*H_3). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -5.3 (1B), -6.4 (2B) (*B*N and *B*H), -10.2 (1B), -12.1 (3B), -14.7 (3B). HRMS: *m*/*z* calcd for $C_{10}H_{20}{}^{10}B_2{}^{11}B_8NO_2S^-$ [M-H]$^-$: 326.2229. Found: 326.2224.



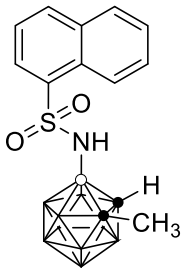
4c: Yield 82% (28.0 mg). White solid. ^{1}H NMR (400 MHz, $CDCl_3$): 7.86 (d, *J* = 8.8 Hz, 2H), 7.01 (d, *J* = 8.8 Hz, 2H) (aryl C*H*), 5.39 (s, 1H) (N*H*), 4.22 (s, 1H) (cage C*H*), 3.90 (s, 3H) (OCH_3), 2.09 (s, 3H) (CH_3). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 163.3, 132.8, 129.0, 114.5 (aryl *C*), 71.8, 60.8 (cage *C*), 55.8 (O*C*H_3), 23.0 (*C*H_3). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -5.7 (1B), -6.5 (2B) (*B*N and *B*H), -10.4 (1B), -12.5 (3B), -14.6 (2B), -15.6 (1B). HRMS: *m*/*z* calcd for $C_{10}H_{20}{}^{10}B_2{}^{11}B_8NO_3S^-$ [M-H]$^-$: 342.2178. Found: 342.2173.



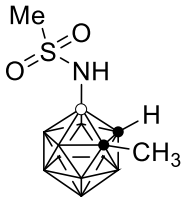
5c: Yield 48% (16.5 mg). White solid. ^{1}H NMR (400 MHz, $CDCl_3$): 7.88 (d, *J* = 8.8 Hz, 2H), 7.54 (d, *J* = 8.8 Hz, 2H) (aryl C*H*), 5.71 (s, 1H) (N*H*), 4.17 (s, 1H) (cage C*H*), 2.08 (s, 3H) (CH_3). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 139.9, 139.6, 129.7, 128.3 (aryl *C*), 71.7, 61.0 (cage *C*), 23.0 (*C*H_3). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -5.7 (1B), -6.5 (2B) (*B*N and *B*H), -10.4 (1B), -12.5 (3B), -14.6 (2B), -15.6 (1B). HRMS: *m*/*z* calcd for $C_9H_{17}{}^{10}B_2{}^{11}B_8ClNO_2S^-$ [M-H]$^-$: 346.1686. Found: 346.1678.



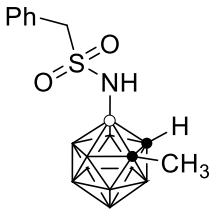
6c: Yield 58% (19.2 mg). White solid. ^{1}H NMR (400 MHz, $CDCl_3$): 7.96 (m, 2H), 7.25 (m, 2H) (aryl C*H*), 5.53 (s, 1H) (N*H*), 4.19 (s, 1H) (cage *H*), 2.09 (s, 3H) (CH_3). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): 165.4 (d, $^{1}J_{C\text{-}F}$ = 255 Hz), 137.2 (d, $^{4}J_{C\text{-}F}$ = 3 Hz), 129.6 (d, $^{3}J_{C\text{-}F}$ = 9 Hz), 116.7 (d, $^{2}J_{C\text{-}F}$ = 22 Hz), 71.9, 61.0 (cage *C*), 23.0 (*C*H_3) ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -5.3 (1B), -6.5 (2B) (*B*N and *B*H), -10.3 (1B), -11.6 (3B), -14.2 (1B), -15.3 (2B). HRMS: *m*/*z* calcd for $C_9H_{17}{}^{10}B_2{}^{11}B_8FNO_2S^-$ [M-H]$^-$: 330.1978. Found: 330.1975.



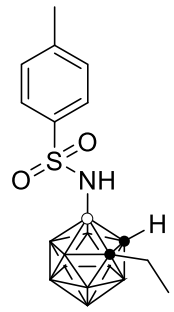
7c: Yield 44% (16.0 mg). White solid. ^{1}H NMR (400 MHz, $CDCl_3$): 8.49 (d, *J* = 8.8 Hz, 1H), 7.94 (m, 4H), 7.66 (m, 2H) (aryl C*H*), 5.69 (s, 1H) (N*H*), 4.27 (s, 1H) (cage C*H*), 2.11 (s, 3H) (CH_3). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 138.0, 135.1, 132.2, 129.9, 129.5, 129.3, 128.2, 128.2, 127.9, 121.8 (aryl *C*), 71.8, 60.9 (cage *C*), 23.0 (*C*H_3). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -5.3 (1B), -6.3 (2B) (*B*N and *B*H), -10.2 (1B), -11.6 (3B), -14.3 (3B). HRMS: *m*/*z* calcd for $C_{13}H_{21}{}^{10}B_2{}^{11}B_8NO_2SNa^+$ $[M+Na]^+$: 386.2196. Found: 386.2189.



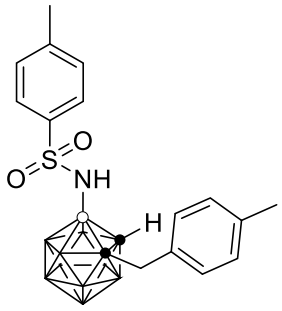
8c: Yield 60% (15.0 mg). White solid. ^{1}H NMR (400 MHz, $CDCl_3$): 5.19 (s, 1H) (N*H*), 4.21 (s, 1H) (cage C*H*), 2.23 (s, 3H), 2.04 (s, 3H) (CH_3). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 71.7, 61.1 (cage *C*), 42.9, 22.9 (*C*H_3). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -4.5 (1B), -5.7 (2B) (*B*N and *B*H), -9.4 (1B), -10.8 (2B), -11.7 (1B), -14.0 (3B). HRMS: *m*/*z* calcd for $C_4H_{16}{}^{10}B_2{}^{11}B_8NO_2S^-$ $[M-H]^-$: 250.1913. Found: 250.1911.



9c: Yield 69% (22.5 mg). White solid. ^{1}H NMR (400 MHz, $CDCl_3$): 7.50 (m, 2H), 7.45 (m, 3H) (aryl C*H*), 4.97 (s, 1H) (N*H*), 4.41 (m, 2H) (CH_2), 4.29 (s, 1H) (cage C*H*), 1.99 (s, 3H) (CH_3). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 131.2, 129.3, 129.2, 128.8 (aryl *C*), 71.8, 61.0 (cage *C*), 60.4 (*C*H_2), 22.8 (*C*H_3). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -5.5 (1B), -6.4 (2B) (*B*N and *B*H), -10.2 (1B), -11.6 (3B), -14.4 (3B). HRMS: *m*/*z* calcd for $C_{10}H_{20}{}^{10}B_2{}^{11}B_8NO_2S^-$ $[M-H]^-$: 326.2229. Found: 326.2223.



10c: Yield 82% (28.0 mg). White solid. ^{1}H NMR (400 MHz, $CDCl_3$): 7.81 (d, *J* = 8.0 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 2H) (aryl C*H*), 5.51 (s, 1H) (N*H*), 4.20 (s, 1H) (cage C*H*), 2.49 (m, 4H), 2.29 (m, 1H) (CH_3 and CH_2), 1.14 (t, *J* = 7.6 Hz, 3H) (CH_3). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 144.2, 138.3, 130.0, 126.8 (aryl *C*), 60.1 (cage *C*), 28.4, 21.8, 13.2 (*C*H_3 and *C*H_2). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -6.0 (3B) (*B*N and *B*H), -10.3 (1B), -13.4 (3B), -14.6 (3B). HRMS: *m*/*z* calcd for $C_{11}H_{22}{}^{10}B_2{}^{11}B_8NO_2S^-$ $[M-H]^-$: 340.2386. Found: 340.2380.



11c: Yield 70% (29.0 mg). Colorless crystals. ^{1}H NMR (400 MHz, $CDCl_3$): 7.86 (d, *J* = 8.4 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 2H), 7.13 (m, 4H) (aryl C*H*), 5.68 (s, 1H) (N*H*), 4.14 (s, 1H) (cage C*H*), 3.57 (m, 2H) (CH_2), 2.45 (s, 3H), 2.34 (s, 3H) (CH_3). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 144.2, 138.3, 138.1, 131.8, 130.3, 130.0, 129.6, 126.8 (aryl *C*), 76.3, 59.1 (cage *C*H), 40.0, 21.7, 21.3 (CH_3 and CH_2). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -5.3 (3B) (*B*N and *B*H), -10.1 (1B), -13.1 (2B), -14.6 (4B). HRMS: *m*/*z* calcd for $C_{17}H_{27}{}^{10}B_2{}^{11}B_8NO_2SNa^+$ $[M+Na]^-$: 440.2667. Found: 440.2664.

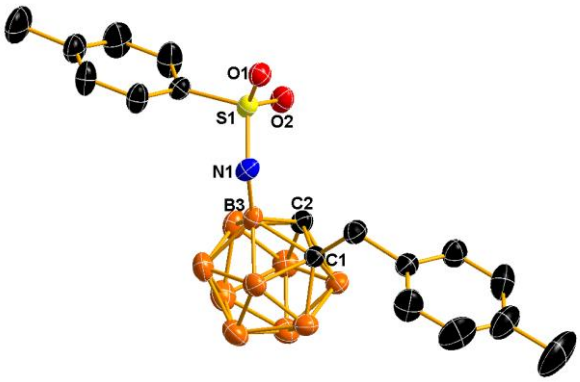
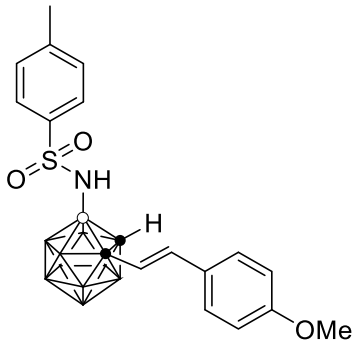


Figure S1. Molecular Structure of **11c**



12c: Yield 57% (25.5 mg). Colorless crystals. ^{1}H NMR (400 MHz, $CDCl_3$): 7.79 (d, *J* = 8.0 Hz, 2H), 7.30 (m, 4H), 6.87 (d, *J* = 8.4 Hz, 2H), 6.81 (d, *J* = 16.0 Hz, 1H), 6.19 (d, *J* = 16.0 Hz, 1H) (aryl C*H* and alkenyl C*H*), 5.40 (s, 1H) (N*H*), 4.35 (s, 1H) (cage C*H*), 3.83 (s, 3H) (OCH_3), 2.43 (s, 3H) (CH_3). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 160.8, 144.0, 138.4, 138.1, 129.9, 128.7, 127.1, 126.8, 117.1, 114.4 (aryl *C*), 75.7, 60.0 (cage *C*), 55.5 (OCH_3), 21.7 (CH_3). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -5.6 (3B) (*B*N and *B*H), -12.2 (4B), -14.3 (3B). HRMS: *m*/*z* calcd for $C_{18}H_{27}{}^{10}B_2{}^{11}B_8NO_3SNa^+$ $[M+Na]^+$: 468.2617. Found: 468.2608.

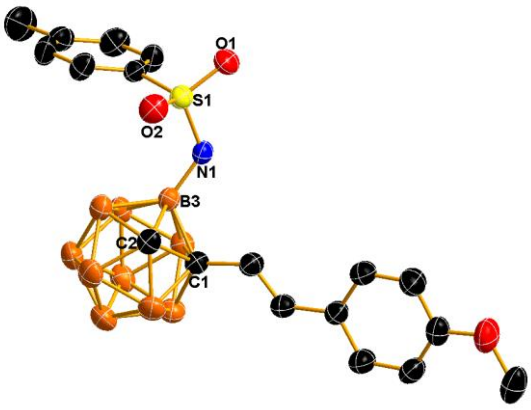
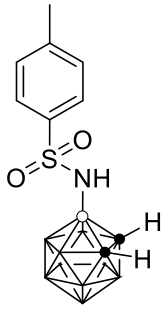


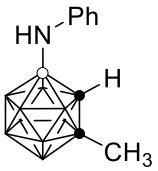
Figure S2. Molecular Structure of **12c**



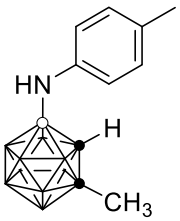
13c: Yield 45% (14.0 mg). White solid. ^{1}H NMR (400 MHz, $CDCl_3$): 7.78 (d, *J* = 7.6 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 2H) (aryl C*H*), 5.36 (s, 1H) (N*H*), 4.09 (s, 2H) (cage C*H*), 2.46 (s, 3H) (CH_3). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 144.3, 138.2, 130.1, 126.6 (aryl *C*), 56.5 (cage *C*), 21.7 (*C*H_3 and *C*H_2). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -3.9 (2B), -6.9 (1B) (*B*N), -10.4 (1B), -14.5 (6B). HRMS: *m*/*z* calcd for $C_9H_{18}{}^{10}B_2{}^{11}B_8NO_2S^-$ [M-H]$^-$: 312.2072. Found: 312.2067.

Preparation of B(4)-arylamino-*o*-carboranes (d).

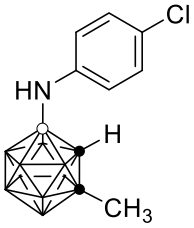
A representative procedure. 1-COOH-*o*-carborane **a** (0.10 mmol), aniline (1.2 equiv, 0.12 mmol), $[Cp^*IrCl_2]_2$ (2.4 mg, 3 mol %, 0.003 mmol), AgOAc (16.7 mg, 10 mol%, 0.01 mmol), $AgNTf_2$ (3.9 mg, 10 mol%, 0.01 mmol), $Cu(OAc)_2$ (54.5 mg, 3.0 equiv, 0.30 mmol) and Li_2CO_3 (14.8 mg, 2.0 equiv, 0.20 mmol) were mixed in dry toluene (2 mL). The resulting mixture was heated in a closed flask at 120 °C or 140 °C for 12 h under argon. Then, the reaction solution was filtered through a pad of Celite and washed with diethyl ether. The organic portions were combined. After removal of organic solvents under reduced pressure, the residue was subjected to flash column chromatography on silica gel (230-400 mesh) using a mixture of *n*-hexane and ethyl acetate (20/1 in v/v) as eluent to give the product **d**.



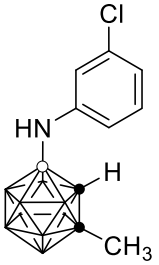
1d: Yield 92% (23.0 mg). Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.19 (t, *J* = 8.4 Hz, 2H), 6.96 (d, *J* = 7.6 Hz, 2H), 6.80 (t, *J* = 7.6 Hz, 1H) (aryl C*H*), 3.74 (s, 1H) (cage C*H*), 2.03 (s, 3H) (C*H*$_3$). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 146.7, 129.3, 119.1, 116.2 (aryl *C*), 68.2, 63.4 (cage *C*), 25.9 (*C*H$_3$). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -0.8 (1B) (*B*N), -2.3 (1B), -7.1 (1B), -10.8 (1B), -12.5 (2B), -13.4 (3B), -17.8 (1B). HRMS: *m*/*z* calcd for $C_9H_{20}{}^{10}B_2{}^{11}B_8N^+$ $[M+H]^+$: 250.2593. Found: 250.2590.



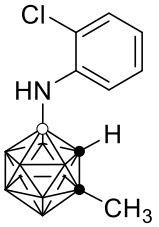
2d: Yield 81% (21.3 mg). Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.99 (d, *J* = 8.0 Hz, 2H), 6.85 (d, *J* = 8.0 Hz, 2H) (aryl C*H*), 3.83 (s, 1H) (N*H*), 3.70 (s, 1H) (cage C*H*), 2.25 (s, 3H), 2.03 (s, 3H) (C*H*$_3$). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 144.4, 129.7, 128.2, 116.2 (aryl *C*), 68.1, 63.4 (cage *C*), 26.0, 20.6 (*C*H$_3$). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -0.2 (1B) (*B*N), -2.8 (1B), -6.9 (1B), -10.6 (1B), -12.4 (2B), -13.3 (3B), -17.9 (1B). HRMS: *m*/*z* calcd for $C_{10}H_{22}{}^{10}B_2{}^{11}B_8N^+$ $[M+H]^+$: 264.2750. Found: 264.2747.



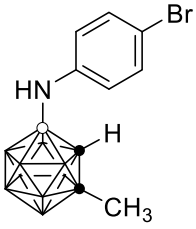
3d: Yield 93% (26.3 mg). Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.12 (d, *J* = 8.4 Hz, 2H), 6.89 (d, *J* = 8.8 Hz, 2H) (aryl C*H*), 3.82 (s, 1H) (cage C*H*), 2.04 (s, 3H) (C*H*$_3$). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 144.7, 129.1, 124.3, 117.8 (aryl *C*), 68.6, 63.3 (cage *C*), 26.0 (*C*H$_3$). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -1.2 (1B) (*B*N), -2.8 (1B), -7.3 (1B), -10.8 (1B), -13.2 (5B), -17.6 (1B). HRMS: *m*/*z* calcd for $C_9H_{19}{}^{10}B_2{}^{11}B_8ClN^+$ $[M+H]^+$: 284.2204. Found: 284.2201.



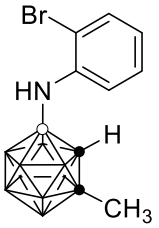
4d: Yield 92% (26.0 mg). Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.08 (t, *J* = 8.0 Hz, 1H), 6.93 (s, 1H), 6.81 (d, *J* = 8.0 Hz, 1H), 6.74 (d, *J* = 7.6 Hz, 1H) (aryl C*H*), 3.98 (s, 1H) (N*H*), 3.73 (s, 1H) (cage C*H*), 2.04 (s, 3H) (C*H*$_3$). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 148.2, 134.8, 130.2, 118.9, 115.9, 114.3 (aryl *C*), 68.4, 63.4 (cage *C*), 26.0 (*C*H$_3$). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -1.1 (1B) (*B*N), -2.8 (1B), -7.1 (1B), -10.8 (1B), -12.3 (1B), -13.2 (4B), -17.6 (1B). HRMS: *m*/*z* calcd for $C_9H_{19}{}^{10}B_2{}^{11}B_8ClN^+$ $[M+H]^+$: 284.2204. Found: 284.2203.



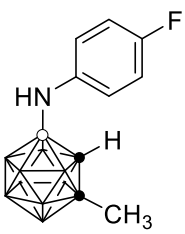
5d: Yield 83% (23.5 mg). Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43 (m, 1H), 7.26 (m, 1H), 7.13 (m, 1H), 6.71 (m, 1H) (aryl C*H*), 4.63 (s, 1H) (N*H*), 3.77 (s, 1H) (cage C*H*), 2.05 (s, 3H) (CH_3). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 143.3, 129.3, 127.6, 121.1, 119.2, 115.4 (aryl *C*), 68.4, 63.7 (cage *C*), 26.0 (*C*H_3). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -0.7 (1B) (*B*N), -2.4 (1B), -6.8 (1B), -10.3 (1B), -12.8 (5B), -17.1 (1B). HRMS: *m*/*z* calcd for $C_9H_{19}{}^{10}B_2{}^{11}B_8ClN^+$ $[M+H]^+$: 284.2204. Found: 284.2200.



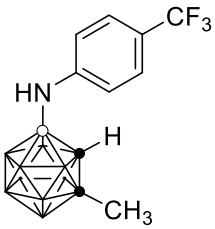
6d: Yield 91% (30.0 mg). Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.24 (d, *J* = 8.8 Hz, 2H), 6.82 (d, *J* = 8.8 Hz, 2H) (aryl C*H*), 3.94 (s, 1H) (N*H*), 3.73 (s, 1H) (cage C*H*), 2.04 (s, 3H) (CH_3). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 146.1, 131.9, 117.7, 110.6 (aryl *C*), 68.4, 63.4 (cage *C*), 26.0 (*C*H_3). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -1.0 (1B) (*B*N), -2.8 (1B), -7.2 (1B), -10.9 (1B), -13.2 (5B), -17.7 (1B). HRMS: *m*/*z* calcd for $C_9H_{19}{}^{10}B_2{}^{11}B_8BrN^+$ $[M+H]^+$: 330.1680. Found: 330.1678.



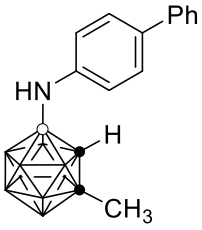
7d: Yield 63% (20.7 mg). Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43 (m, 2H), 7.17 (m, 1H), 6.65 (m, 1H) (aryl C*H*), 4.63 (s, 1H) (N*H*), 3.79 (s, 1H) (cage C*H*), 2.05 (s, 3H) (CH_3). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 144.4, 132.6, 128.3, 119.8, 115.6, 112.5 (aryl *C*), 68.4, 63.7 (cage *C*), 26.0 (*C*H_3). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -1.0 (1B) (*B*N), -2.7 (1B), -7.2 (1B), -10.7 (1B), -12.2 (2B), -13.0 (3B), -17.5 (1B). HRMS: *m*/*z* calcd for $C_9H_{18}{}^{10}B_2{}^{11}B_8BrN^+$ $[M]^+$: 328.1611. Found: 328.1617.



8d: Yield 90% (33.0 mg). Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.87 (m, 4H) (aryl C*H*), 3.84 (s, 1H) (N*H*), 3.72 (s, 1H) (cage C*H*), 2.04 (s, 3H) (CH_3). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 156.5 (d, $^{1}J_{C\text{-}F}$ = 235 Hz), 143.0, 117.1 (d, $^{3}J_{C\text{-}F}$ = 8 Hz), 115.6 (d, $^{2}J_{C\text{-}F}$ = 22 Hz) (aryl *C*), 68.3, 63.4 (cage *C*), 26.0 (*C*H_3). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ 7.4 (1B) (*B*N), 5.1 (1B), 0.8 (1B), -3.0 (1B), -5.4 (5B), -10.0 (1B). ^{19}F NMR (376 MHz, $CDCl_3$): δ -125.8 (m, 1F). HRMS: *m*/*z* calcd for $C_9H_{19}{}^{10}B_2{}^{11}B_8FN^+$ $[M+H]^+$: 268.2499. Found: 268.2496.



9d: Yield 90% (28.5 mg). Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41 (d, *J* = 8.8 Hz, 2H), 6.98 (d, *J* = 8.4 Hz, 2H) (aryl C*H*), 4.20 (s, 1H) (N*H*), 3.76 (s, 1H) (cage C*H*), 2.05 (s, 3H) (C*H*$_3$). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ150.0, 126.6 (q, $^{3}J_{C\text{-}F}$ = 4 Hz) (aryl *C*), 124.9 (q, $^{1}J_{C\text{-}F}$ = 269 Hz) (*C*F$_3$), 120.6 (q, $^{2}J_{C\text{-}F}$ = 33 Hz), 115.4 (aryl *C*), 68.5, 63.4 (cage *C*), 26.0 (*C*H$_3$). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -1.2 (1B) (*B*N), -2.5 (1B), -7.0 (1B), -10.6 (1B), -12.9 (5B), -17.2 (1B). ^{19}F NMR (376 MHz, $CDCl_3$): δ -61.0 (m, 3F). HRMS: *m*/*z* calcd for $C_{10}H_{18}{}^{10}B_2{}^{11}B_8F_2N^+$ [M-F]$^+$: 298.2405. Found: 298.2400.



10d: Yield 92% (30.0 mg). Colorless crystals. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.56 (d, *J* = 7.2 Hz, 2H), 7.44 (m, 4H), 7.29 (t, *J* = 7.2 Hz, 1H), 7.03 (d, *J* = 8.4 Hz, 2H) (aryl C*H*), 4.03 (s, 1H) (N*H*), 3.71 (s, 1H) (cage C*H*), 2.03 (s, 3H) (C*H*$_3$). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 146.4, 141.1, 131.8, 128.8, 127.9, 126.5, 126.4, 116.3 (aryl *C*), 68.2, 63.4 (cage *C*), 25.9 (*C*H$_3$). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -0.8 (1B) (*B*N), -2.8 (1B), -7.0 (1B), -10.7 (1B), -13.3 (5B), -17.8 (1B). HRMS: *m*/*z* calcd for $C_{15}H_{24}{}^{10}B_2{}^{11}B_8N^+$ [M+H]$^+$: 326.2907. Found: 326.2902.

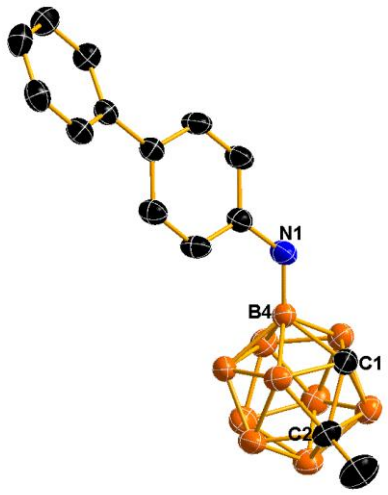
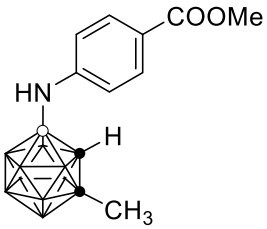


Figure S3. Molecular Structure of **10d**



11d: Yield 94% (29.0 mg). Colorless crystals. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (d, *J* = 8.4 Hz, 2H), 6.92 (d, *J* = 8.8 Hz, 2H) (aryl C*H*), 4.03 (s, 1H) (N*H*), 3.85 (s, 1H) (OCH_3), 3.79 (s, 1H) (cage C*H*), 2.04 (s, 3H) (CH_3). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 167.3 (carbonyl *C*), 151.5, 131.4, 120.2, 115.1 (aryl *C*), 68.5, 63.4 (cage *C*), 51.8 (O*C*H$_3$), 25.9 (*C*H$_3$). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -1.5 (1B) (*B*N), -2.7 (1B), -7.0 (1B), -10.7 (1B), -13.0 (5B), -17.3 (1B). HRMS: *m*/*z* calcd for $C_{11}H_{21}{}^{10}B_2{}^{11}B_8NO_2Na^+$ $[M+Na]^+$: 330.2468. Found: 330.2464.

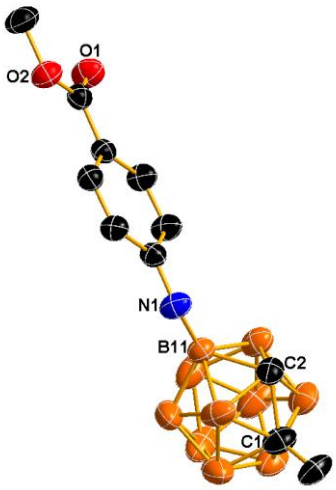
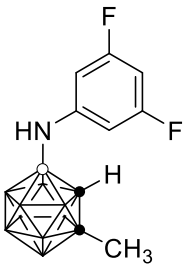
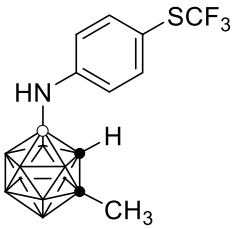


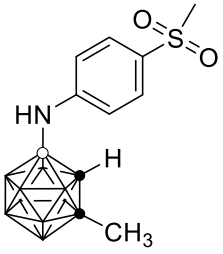
Figure S4. Molecular Structure of **11d**



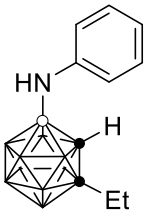
12d: Yield 95% (27.0 mg). Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.44 (m, 2H), 6.21 (m, 1H) (aryl C*H*), 4.10 (s, 1H) (N*H*), 3.75 (s, 1H) (cage C*H*), 2.05 (s, 3H) (CH_3). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 163.9 (dd, $^{3}J_{C\text{-}F}$ = 16 Hz, $^{1}J_{C\text{-}F}$ = 243 Hz), 149.5 (t, $^{3}J_{C\text{-}F}$ = 13 Hz), 98.9 (dd, $^{4}J_{C\text{-}F}$ = 8 Hz, $^{2}J_{C\text{-}F}$ = 20 Hz), 94.1 (t, $^{2}J_{C\text{-}F}$ = 26 Hz) (aryl *C*), 68.6, 63.3 (cage *C*), 26.0 (*C*H$_3$). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ 1.8 (1B) (*B*N), 0.6 (1B), -3.8 (1B), -7.5 (1B), -8.7 (1B), -9.7 (4B), -14.0 (1B). ^{19}F NMR (376 MHz, $CDCl_3$): δ -106.8 (m, 2F). HRMS: *m*/*z* calcd for $C_9H_{18}{}^{10}B_2{}^{11}B_8F_2N^+$ $[M+H]^+$: 286.2405. Found: 286.2404.



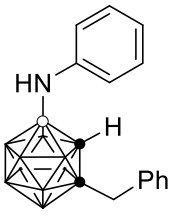
13d: Yield 89% (31.0 mg). Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43 (d, *J* = 8.8 Hz, 2H), 6.95 (d, *J* = 8.8 Hz, 2H) (aryl C*H*), 4.19 (s, 1H) (N*H*), 3.76 (s, 1H) (cage C*H*), 2.05 (s, 3H) (CH_3). ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 149.7, 138.1 (aryl *C*), 129.8 (q, $^{1}J_{C\text{-}F}$ = 306 Hz) (CF_3), 116.5, 111.8 (aryl *C*), 68.5, 63.4 (cage *C*), 26.0 (CH_3). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -1.4 (1B) (*B*N), -2.6 (1B), -7.0 (1B), -10.6 (1B), -12.0 (1B), -12.9 (4B), -17.3 (1B). ^{19}F NMR (376 MHz, $CDCl_3$): δ -44.1 (m, 3F). HRMS: *m*/*z* calcd for $C_{10}H_{19}{}^{10}B_2{}^{11}B_8F_3NS^+$ $[M+H]^+$: 350.2189. Found: 350.2183.



14d: Yield 67% (22.0 mg). White solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64 (d, *J* = 8.4 Hz, 2H), 6.98 (d, *J* = 8.8 Hz, 2H) (aryl C*H*), 4.53 (s, 1H) (N*H*), 3.94 (s, 1H) (cage C*H*), 2.99 (s, 3H), 2.06 (s, 3H) (CH_3). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 152.3, 129.1, 129.0, 115.5 (aryl *C*), 68.8, 63.4 (cage *C*), 45.1, 25.9 (CH_3). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -1.7 (1B) (*B*N), -2.6 (1B), -7.1 (1B), -10.6 (1B), -11.7 (1B), -12.7 (4B), -16.9 (1B). HRMS: *m*/*z* calcd for $C_{10}H_{21}{}^{10}B_2{}^{11}B_8NO_2SNa^+$ $[M+Na]^+$: 350.2189. Found: 350.2185.

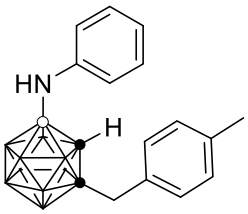


15d: Yield 87% (23.0 mg). Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.18 (t, *J* = 7.6 Hz, 2H), 6.95 (d, *J* = 7.6 Hz, 2H) (aryl C*H*), 6.78 (t, *J* = 7.2 Hz, 1H), 3.94 (s, 1H) (N*H*), 3.71 (s, 1H) (cage C*H*), 2.29 (q, *J* = 7.6 Hz, 2H), 1.10 (t, *J* = 7.6 Hz, 3H) (Et). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 146.9, 129.3, 118.8, 116.0 (aryl *C*), 74.0, 62.7 (cage *C*), 31.7, 13.6 (Et). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -0.9 (1B) (*B*N), -3.3 (1B), -6.1 (1B), -10.8 (1B), -12.4 (1B), -13.9 (4B), -18.5 (1B). HRMS: *m*/*z* calcd for $C_{10}H_{22}{}^{10}B_2{}^{11}B_8N^+$ $[M+H]^+$: 264.2750. Found: 264.2748.

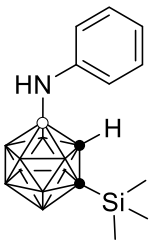


16d: Yield 91% (29.6 mg). Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36 (m, 3H), 7.15 (m, 4H), 6.89 (d, *J* = 7.6 Hz, 2H), 6.77 (t, *J* = 7.2 Hz, 1H) (aryl C*H*), 3.86 (s, 1H) (N*H*), 3.53 (m, 2H) (CH_2), 3.41 (s, 1H) (cage C*H*). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 146.8, 134.4, 129.9, 129.3, 129.2, 128.7, 118.8, 115.8 (aryl *C*), 72.5, 61.1 (cage *C*), 43.6 (CH_2). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -1.0 (1B)

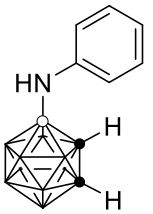
(*B*N), -3.6 (1B), -6.1 (1B), -10.6 (1B), -12.2 (1B), -13.9 (4B), -17.9 (1B). HRMS: *m*/*z* calcd for $C_{15}H_{24}{}^{10}B_2{}^{11}B_8N^+$ $[M+H]^+$: 326.2907. Found: 326.2903.



17d: Yield 94% (32.0 mg). Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.17 (m, 4H), 7.03 (d, *J* = 8.0 Hz, 2H), 6.90 (d, *J* = 7.6 Hz, 2H), 6.78 (t, *J* = 7.6 Hz, 1H) (aryl C*H*), 3.87 (s, 1H) (N*H*), 3.50 (m, 2H) (CH_2), 3.40 (s, 1H) (cage C*H*), 2.37 (s, 3H) (CH_3). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 146.9, 139.5, 131.3, 129.9, 129.7, 129.2, 118.7, 115.8 (aryl *C*), 72.7, 61.0 (cage *C*), 43.2 (*C*H_2), 21.3 (*C*H_3). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -1.1 (1B) (*B*N), -3.7 (1B), -6.1 (1B), -10.6 (1B), -12.2 (1B), -13.9 (4B), -17.9 (1B). HRMS: *m*/*z* calcd for $C_{16}H_{26}{}^{10}B_2{}^{11}B_8N^+$ $[M+H]^+$: 340.3063. Found: 340.3057.



18d: Yield 49% (15.0 mg). Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.19 (t, *J* = 8.0 Hz, 2H), 6.97 (d, *J* = 8.0 Hz, 2H), 6.78 (t, *J* = 7.6 Hz, 1H) (aryl C*H*), 3.98 (s, 1H) (N*H*), 3.52 (s, 1H) (cage C*H*), 0.25 (s, 9H) (CH_3). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 147.0, 129.3, 118.7, 115.9 (aryl *C*), 64.1, 61.7 (cage *C*), -1.3 (*C*H_3). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -0.5 (2B) (*B*N and *B*H), -2.7 (1B), -9.2 (1B), -10.2 (1B), -13.0 (1B), -14.1 (1B), -15.1 (2B), -18.4 (1B). HRMS: *m*/*z* calcd for $C_{11}H_{26}{}^{10}B_2{}^{11}B_8NSi^+$ $[M+H]^+$: 308.2833. Found: 308.2829.

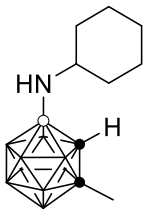


19d: Yield 26% (6.1 mg). Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.18 (t, *J* = 8.0 Hz, 2H), 6.95 (d, *J* = 8.0 Hz, 2H), 6.78 (t, *J* = 7.2 Hz, 1H) (aryl C*H*), 3.94 (s, 1H) (N*H*), 3.72 (s, 1H), 3.56 (s, 1H) (cage C*H*). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 146.8, 129.3, 119.0, 116.0 (aryl *C*), 56.1, 52.3 (cage *C*). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ -1.3 (1B) (*B*N), -2.6 (2B), -10.8 (1B), -12.3 (1B), -14.2 (1B), -16.3 (3B), -21.0 (1B). HRMS: *m*/*z* calcd for $C_8H_{18}{}^{10}B_2{}^{11}B_8N^+$ $[M+H]^+$: 236.2437. Found: 236.2436.

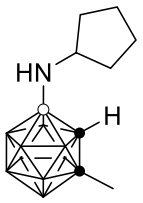
Preparation of B(4)-alkylamino-*o*-carboranes (d).

A representative procedure. 1-COOH-2-Me-*o*-carborane **1a** (20.2 mg, 0.10 mmol), alkylamine **b** (1.1 equiv, 0.11 mmol), $[Cp^*IrCl_2]_2$ (4.0 mg, 5 mol %, 0.005 mmol), AgOAc (50.1 mg, 3.0 equiv, 0.30 mmol), $AgNTf_2$ (38.8 mg, 1.0 equiv, 0.10 mmol) and $Cu(OAc)_2$ (9.1

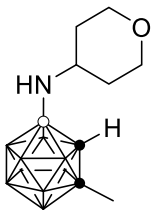
mg, 0.5 equiv, 0.05 mmol) were mixed in 1,2-dichloroethane (2.0 mL). The resulting mixture was heated in a closed flask at 50 ^{o}C for 12 h under argon. Then, the reaction solution was filtered through a pad of Celite and washed with diethyl ether. The organic portions were combined. After removal of organic solvents under reduced pressure, the residue was subjected to flash column chromatography on silica gel (230-400 mesh) using a mixture of *n*-hexane and ethyl acetate (20/1 in v/v) as eluent to give the product **d**.



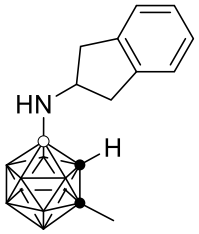
20d: Yield 78% (20.0 mg). Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): 3.53 (s, 1H) (cage C*H*), 2.68 (m, 1H) (C*H*), 2.01 (s, 3H) (CH_3), 1.89 (m, 2H), 1.67 (m, 2H), 1.55 (m, 1H), 1.25 (m, 3H), 1.11 (m, 1H), 0.98 (m, 1H) (CH_2). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 68.1, 63.1 (cage *C*), 56.0 (*C*H), 37.0, 37.0, 26.0, 25.9, 25.6 (*C*H_2 and *C*H_3). ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ 3.0 (1B) (*B*N), -3.1 (1B), -7.3 (1B), -11.1 (1B), -13.3 (3B), -14.3 (2B), -19.6 (1B). HRMS: *m*/*z* calcd for $C_9H_{25}{}^{10}B_2{}^{11}B_8N^+$ $[M]^+$: 255.2985. Found: 255.2987.



21d: Yield 71% (17.0 mg). Colorless oil. ^{1}H NMR (400 MHz, C_6D_6): 3.33 (m, 1H) (C*H*), 2.54 (s, 1H) (cage C*H*), 1.84 (m, 2H), 1.54 (m, 2H), 1.41 (m, 2H) (CH_2), 1.21 (s, 3H) (CH_3), 1.11 (m, 2H) (CH_2). ^{13}C{^{1}H} NMR (100 MHz, C_6D_6): δ 67.9, 63.0 (cage *C*), 56.3 (*C*H), 36.4, 36.3, 25.2, 23.8 (*C*H_2 and *C*H_3). ^{11}B{^{1}H} NMR (128 MHz, C_6D_6): δ 3.3 (1B) (*B*N), -2.5 (1B), -6.7 (1B), -10.6 (1B), -12.9 (3B), -13.9 (2B), -19.2 (1B). HRMS: *m*/*z* calcd for $C_8H_{24}{}^{10}B_2{}^{11}B_8N^+$ $[M+H]^+$: 242.2911. Found: 242.2908.



22d: Yield 62% (16.0 mg). Colorless oil. ^{1}H NMR (400 MHz, C_6D_6): 3.84 (m, 2H), 3.19 (m, 2H) (OCH_2), 2.78 (m, 1H) (C*H*), 2.48 (s, 1H) (cage C*H*), 1.65 (m, 2H) (CH_2), 1.20 (s, 3H) (CH_3), 1.14 (m, 2H) (CH_2). ^{13}C{^{1}H} NMR (100 MHz, C_6D_6): δ 67.9 (cage *C*), 67.2 (O*C*H_2), 63.2 (cage *C*), 53.4 (*C*H), 37.6, 37.5 (*C*H_2), 25.2 (*C*H_3). ^{11}B{^{1}H} NMR (128 MHz, C_6D_6): δ 3.0 (1B) (*B*N), -2.6 (1B), -6.9 (1B), -10.8 (1B), -13.0 (3B), -14.1 (2B), -19.3 (1B). HRMS: *m*/*z* calcd for $C_8H_{23}{}^{10}B_2{}^{11}B_8NO^+$ $[M]^+$: 257.2777. Found: 257.2775.



23d: Yield 42% (12.0 mg). Colorless crystals. ^{1}H NMR (400 MHz, C_6D_6): 7.11 (m, 4H) (aryl C*H*), 3.76 (m, 1H) (C*H*), 3.07 (m, 2H), 2.49 (m, 3H) (CH_2 and cage C*H*), 1.19 (s, 3H) (CH_3). ^{13}C{^{1}H} NMR (100 MHz, C_6D_6): δ 142.1, 126.8, 125.0, 124.9 (aryl *C*), 67.9, 63.1 (cage *C*), 59.4 (*C*H), 43.3, 43.3 (*C*H_2), 25.2 (*C*H_3). ^{11}B{^{1}H} NMR (128 MHz, C_6D_6): δ 2.9 (1B) (*B*N), -2.8 (1B), -7.0 (1B), -10.8 (1B), -13.0 (3B), -14.1 (2B), -19.3 (1B). HRMS: *m*/*z* calcd for $C_{12}H_{23}{}^{10}B_2{}^{11}B_8N^+$ $[M]^+$: 289.2828. Found: 289.2824.

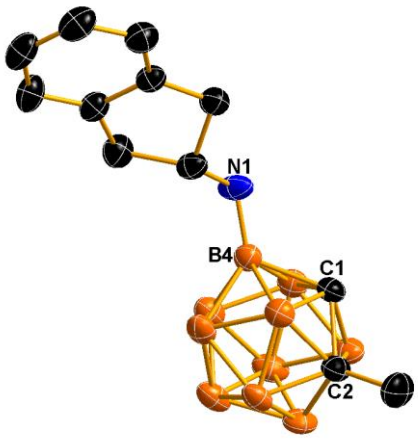
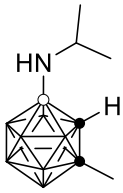


Figure S5. Molecular Structure of **23d**

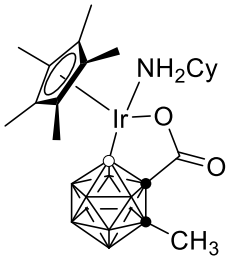


24d: Yield 56% (12.2 mg). Colorless oil. ^{1}H NMR (400 MHz, C_6D_6): 3.07 (m, 1H) (C*H*), 2.50 (s, 1H) (cage C*H*), 1.19 (s, 3H) (CH_3), 0.98 (m, 6H) (CH_3). ^{13}C{^{1}H} NMR (100 MHz, C_6D_6): δ 67.8, 63.1 (cage *C*), 58.6 (*C*H), 26.5, 26.4, 25.2 (*C*H_3). ^{11}B{^{1}H} NMR (128 MHz, C_6D_6): δ 3.1 (1B) (*B*N), -2.7 (1B), -7.0 (1B), -10.9 (1B), -13.2 (3B), -14.3 (2B), -19.5 (1B). HRMS: *m*/*z* calcd for $C_6H_{22}{}^{10}B_2{}^{11}B_8N^+$ $[M+H]^+$: 216.2750. Found: 216.2752.

Mechanistic study.

Synthesis of B(4)-H iridation intermediate III. 1-COOH-2-Me-*o*-carborane **1a** (20.2 mg, 0.10 mmol) and Cp*Ir(OAc)$_2$ (44.6 mg, 1.0 equiv, 0.10 mmol) were dissolved in toluene (0.5 mL). To the resultant clear orange solution was slowly added cyclohexamine (12.6 μL, 1.1

equiv, 0.11 mmol). The resulting mixture was kept at room temperature for 6 hours to precipitate the complex **III** as light yellow crystals (45.0 mg, 72%).



III. ^{1}H NMR (400 MHz, CD_2Cl_2): δ 3.55 (br, 1H), 3.19 (br, 1H) (N*H*$_2$), 2.52 (m, 1H) (C*H*), 2.05 (s, 3H) (C*H*$_3$), 2.02 (m, 2H) (C*H*$_2$), 1.76 (m, 3H) (C*H*$_2$), 1.57 (m, 15H) (C*H*$_3$), 1.30 (m, 2H), 1.17 (m, 3H) (C*H*$_2$). ^{13}C{^{1}H} NMR (125 MHz, CD_2Cl_2): δ 172.0 (*C*=O), 87.4, 87.3 (Cp* *C*), 80.4 (cage *C*), 55.3, 36.0, 34.8, 25.8, 25.5, 22.8 (Cyclohexyl *C* and *C*H$_3$), 9.4, 9.4 (Cp* *C*H$_3$). ^{11}B{^{1}H} NMR (128 MHz, CD_2Cl_2): δ -3.9 (1B), -6.5 (1B), -9.8 (6B), -13.0 (2B) (*B*-Ir and *B*H). HRMS: *m*/*z* calcd for $C_{20}H_{41}{}^{10}B_2{}^{11}B_8IrNO_2^+$ $[M+H]^+$: 628.3757. Found: 628.3767.

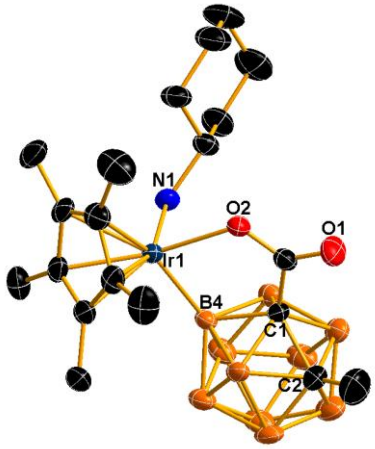


Figure S6. Molecular Structure of **III**

Control experiments.

Transformation of III. Complex **III** (31.4 mg, 0.05 mmol), AgOAc (25.0 mg, 3.0 equiv, 0.15 mmol), $AgNTf_2$ (19.4 mg, 1.0 equiv, 0.05 mmol) and $Cu(OAc)_2$ (4.5 mg, 0.5 equiv, 0.025 mmol) were mixed in 1,2-dichloroethane (2.0 mL). The resulting mixture was heated in a closed flask at 50 °C for 12 h under argon. Subsequently, the reaction mixture was acidified by 1M HCl (1 mL). The organic layer was washed with sat. $NaHCO_3$ (3 x 3 mL) and extracted by dichloromethane. The organic layers were combined and dried over $MgSO_4$. After filtration and evaporation of volatiles under reduced pressure, the residue was subjected to flash column chromatography on silica gel (230-400 mesh) using *n*-hexane and ethyl acetate (20/1 in v/v) as eluent to give the product **20d** (9.0 mg, 71%).

III catalyzed B(4)-H amination. 1-COOH-2-Me-*o*-carborane **1a** (20.2 mg, 0.10 mmol), cyclohexamine (12.6 μL, 1.1 equiv, 0.11 mmol), **III** (6.3 mg, 10 mol %, 0.01 mmol), AgOAc

(50.1 mg, 3.0 equiv, 0.30 mmol), $AgNTf_2$ (38.8 mg, 1.0 equiv, 0.10 mmol) and $Cu(OAc)_2$ (9.1 mg, 0.5 equiv, 0.05 mmol) were mixed in 1,2-dichloroethane (2.0 mL). The resulting mixture was heated in a closed flask at 50 ^oC for 12 h under argon. Then, the reaction solution was filtered through a pad of Celite and washed with diethyl ether. The organic portions were combined. After removal of organic solvents under reduced pressure, the residue was subjected to flash column chromatography on silica gel (230-400 mesh) using *n*-hexane and ethyl acetate (20/1 in v/v) as eluent to give the product **20d** (19.1 mg, 75%).

X-ray Structure Determination. Single-crystal X-ray data of **11c**, **12c**, **10d**, **11d**, **23d** and **III** were collected at 293 K 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.

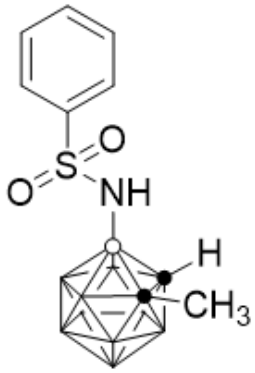
CCDC 2162465-2162470 for **10d**, **11c**, **11d**, **12c**, **23d** and **III** contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

3. References

1. (a) Y. Quan and Z. Xie, *J. Am. Chem. Soc.* 2014, **136**, 15513-15516. (b) H. Lyu, Y. Quan and Z. Xie, *Angew. Chem., Int. Ed.* 2015, **54**, 10623-10626. (c) H. Lyu, Y. Quan and Z. Xie, *Angew. Chem., Int. Ed.* 2016, **55**, 11840-11844.
2. D. Zhao, J. J. Zhang and Z. Xie, *Angew. Chem. Int. Ed.* 2014, **53**, 8488–8491.
3. G. M. Sheldrick, SADABS: 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.

lhr-H-0554-CDCl3

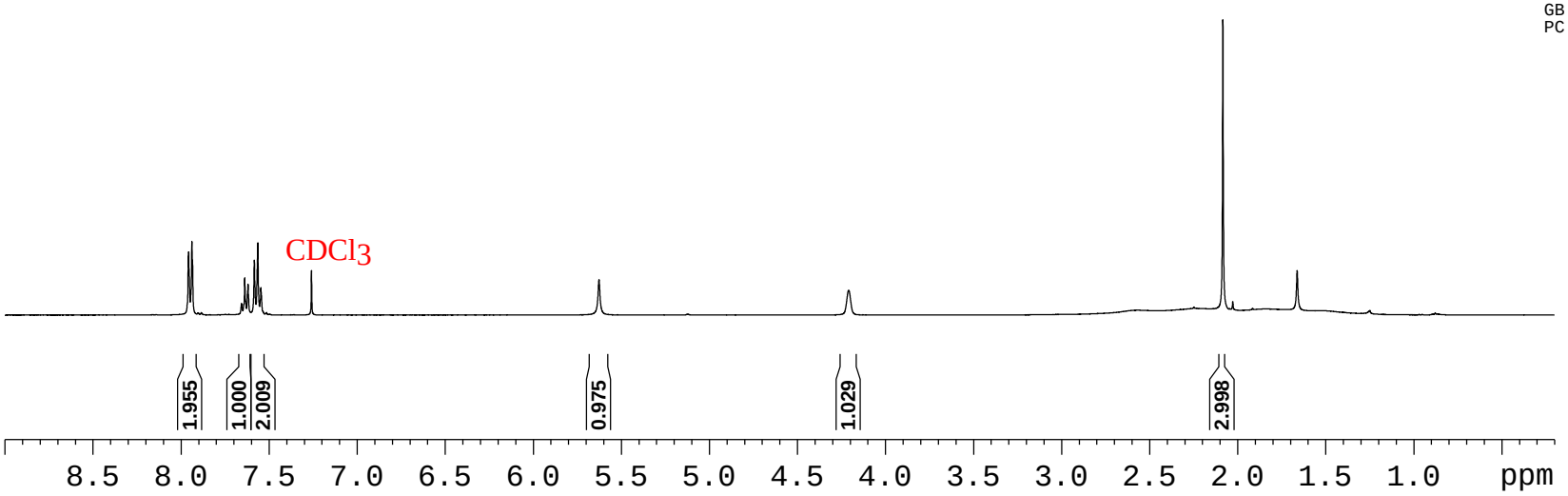
Bruker Advance III 400



1c

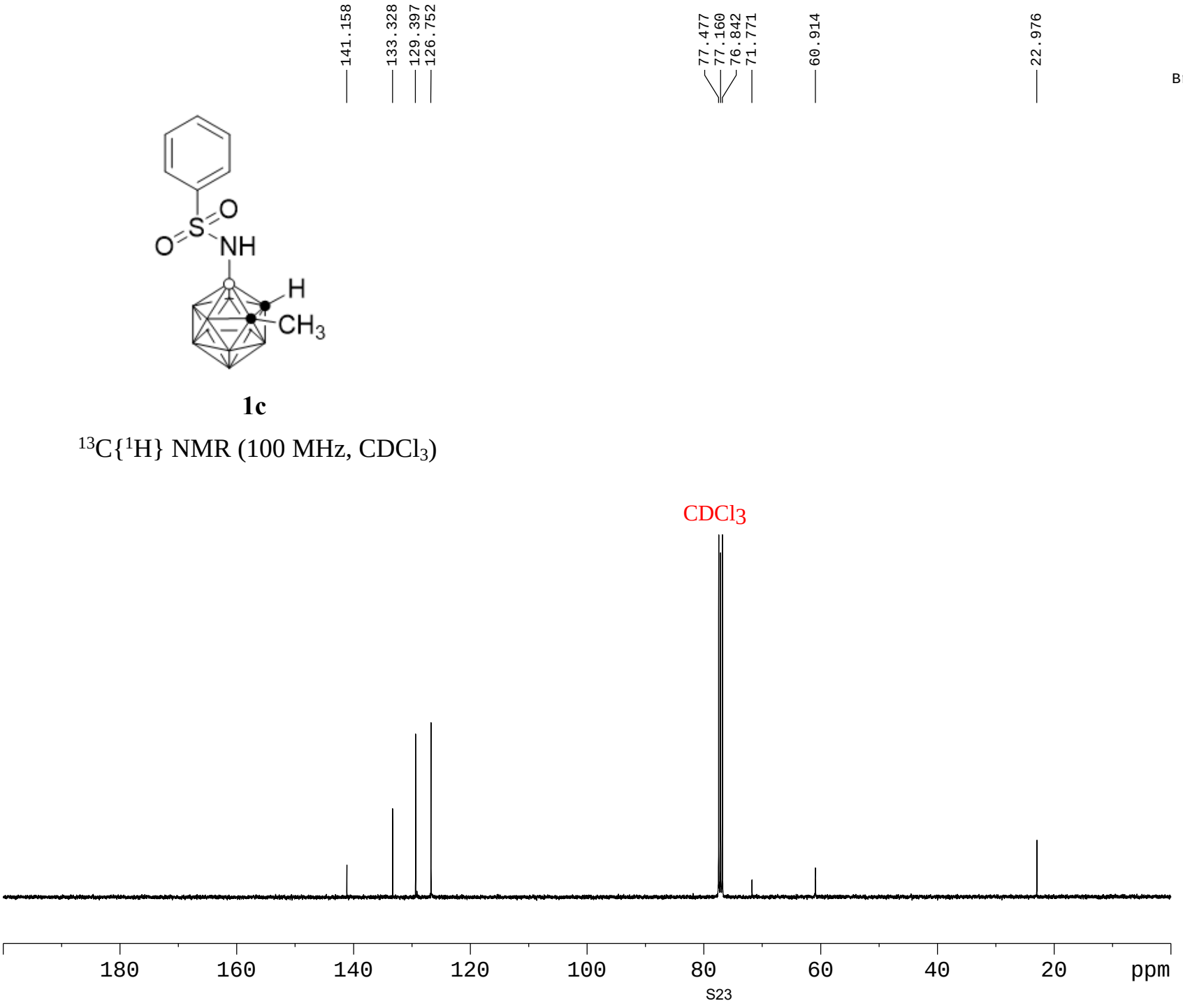
^{1}H NMR (400 MHz, $CDCl_3$)

```
Current Data Parameters
NAME      lhr-H-0554-CDCl3
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20161203
Time              20.51 h
INSTRUM             spect
PROBHD   Z824601_0021 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    12
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                294.2 K
D1           1.00000000 sec
TD0                    1
SFO1        400.1324708 MHz
NUC1                  1H
P1                15.00 usec
PLW1         8.31000042 W

F2 - Processing parameters
SI                 65536
SF          400.1300098 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



1c

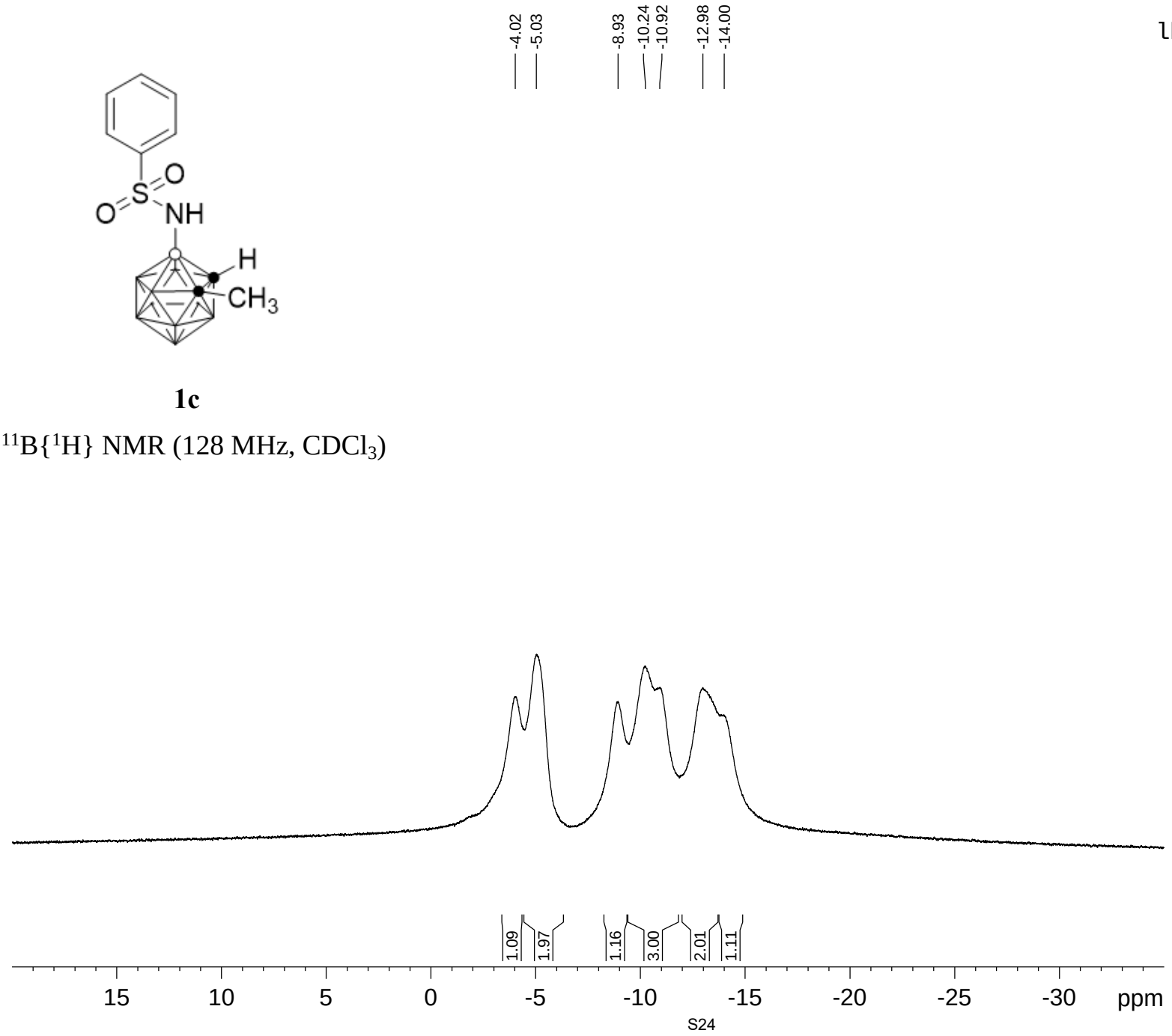
$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)




```
lhr-C-0554-CDCl3

Bruker Advance III 400

Current Data Parameters
NAME      lhr-C-0554-CDCl3
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20161203
Time              21.22 h
INSTRUM             spect
PROBHD   Z824601_0021 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                  766
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                  203
DW               20.800 usec
DE                6.50 usec
TE               294.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P1                 9.50 usec
PLW1        41.25000000 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         8.31000042 W
PLW12        0.23083000 W
PLW13        0.11611000 W

F2 - Processing parameters
SI                32768
SF          100.6127564 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

1c

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)

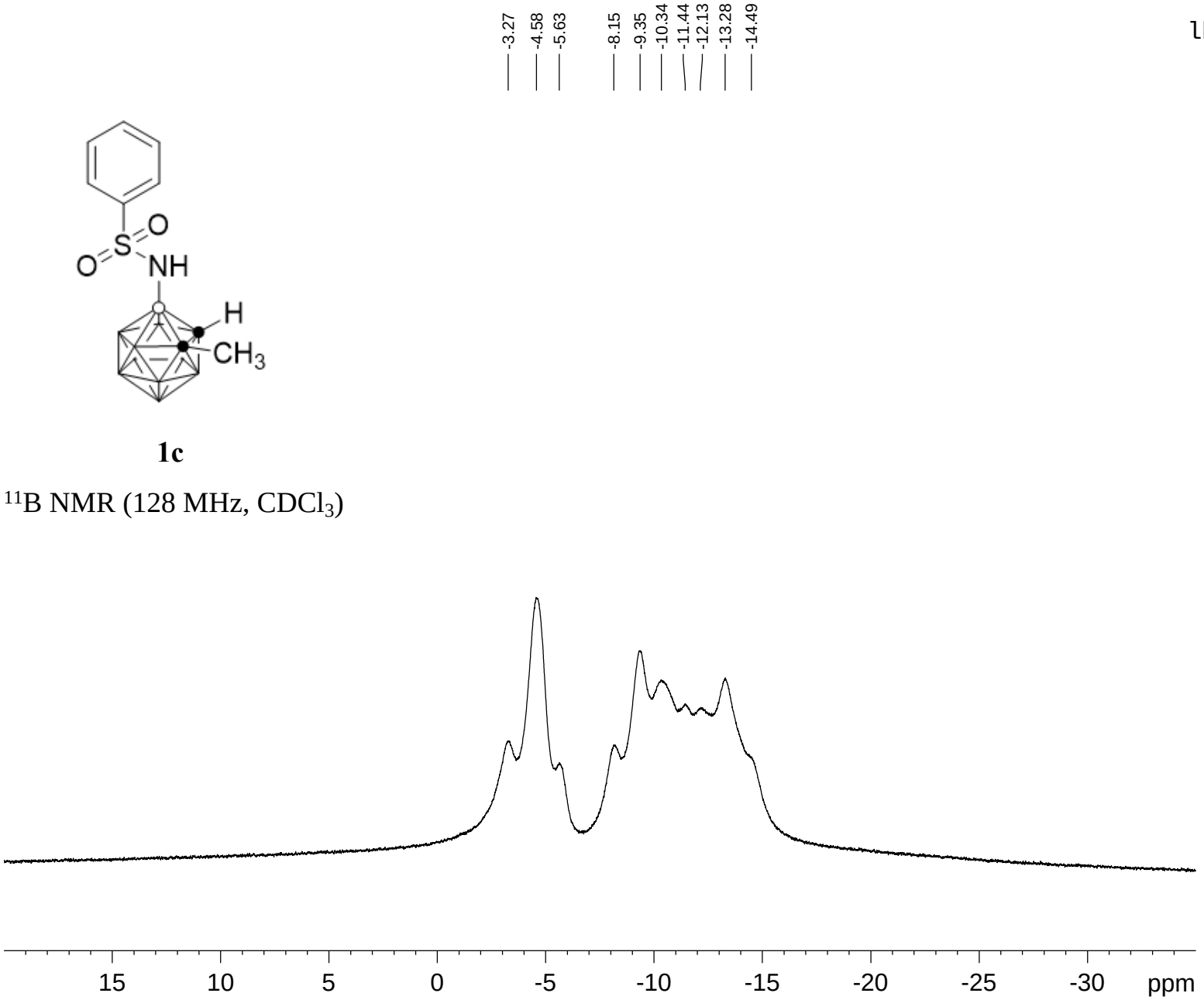


lhr-B-0554-CDCl3

```
Current Data Parameters
NAME      lhr-B-0554-CDCl3
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20161204
Time                19.40 h
INSTRUM              spect
PROBHD   Z108618_0257 (
PULPROG              zgdc
TD                   65536
SOLVENT              CDCl3
NS                      28
DS                       4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ              1.3631488 sec
RG                     287
DW                  20.800 usec
DE                   6.50 usec
TE                  295.5 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
```

1c

^{11}B NMR (128 MHz, $CDCl_3$)



lhr-B-0554-CDCl3(C)

```
Current Data Parameters
NAME      lhr-B-0554-CDCl3(C)
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20161204
Time                  19.43 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                     362
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
```

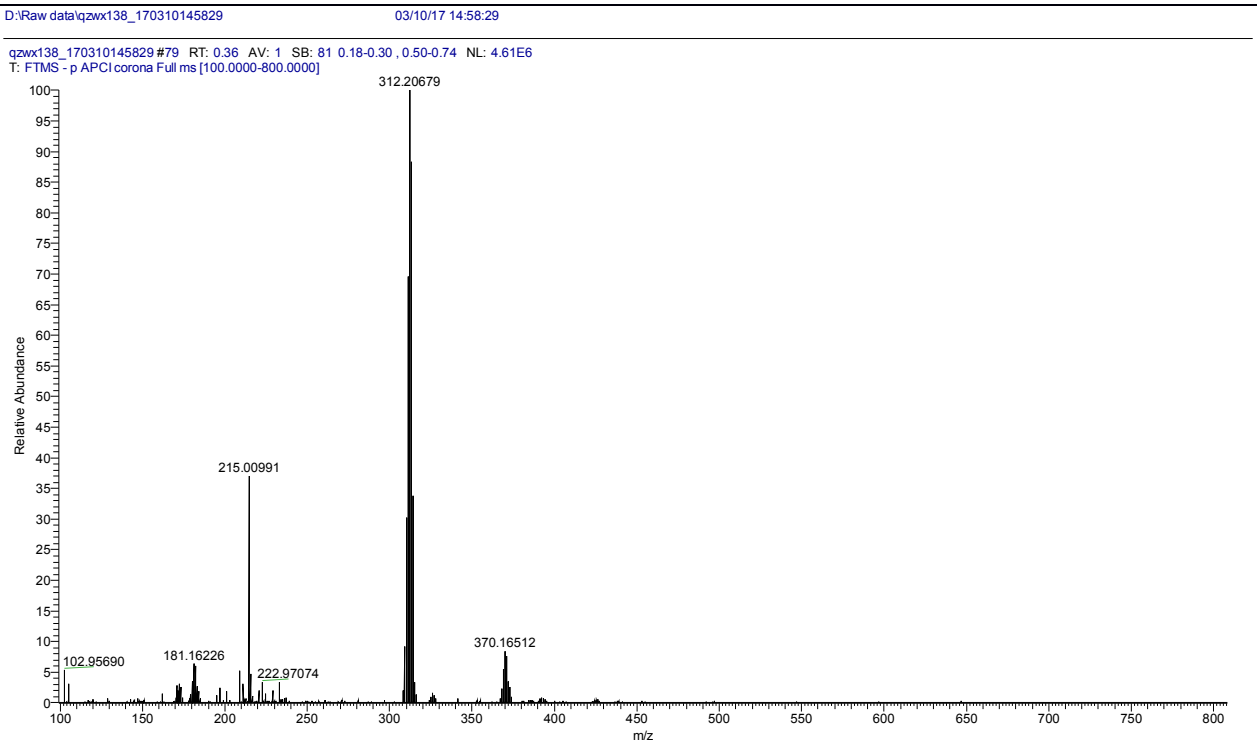
Thermo QEFMS Analysis Report

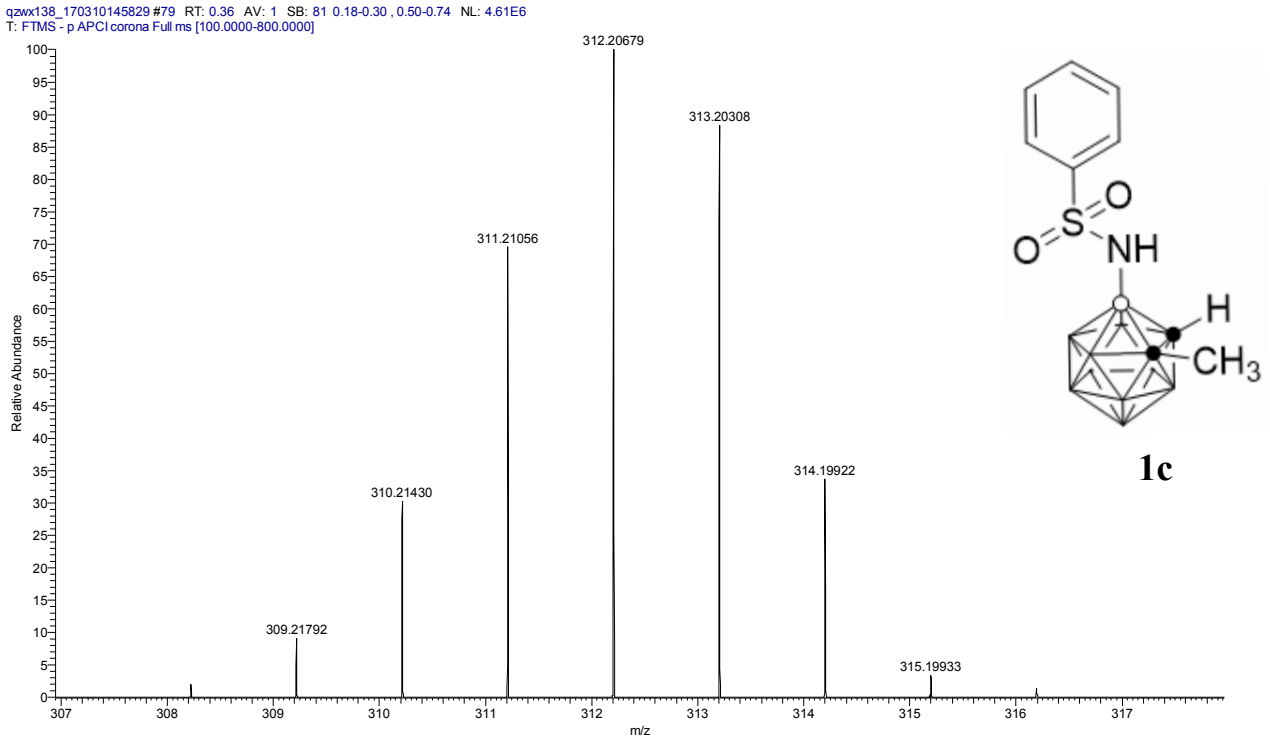
Analysis Info

Sample Name :	Lhr-0554	Reference No.:	Qzwx138
Instrument :	Q Exactive Focus Orbitrap		
Source :	APCI	Polarity :	Negative
Comment :	APCI neg, 5.0uA, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_9H_{19}B_{10}NO_2S$
Experimental Mass $[M-H]^-$:	312.20679
Theoretical Mass $[M-H]^-$:	312.20721
Error (ppm) :	1.3

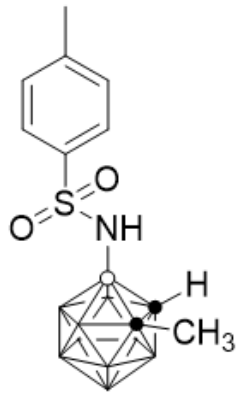




1c

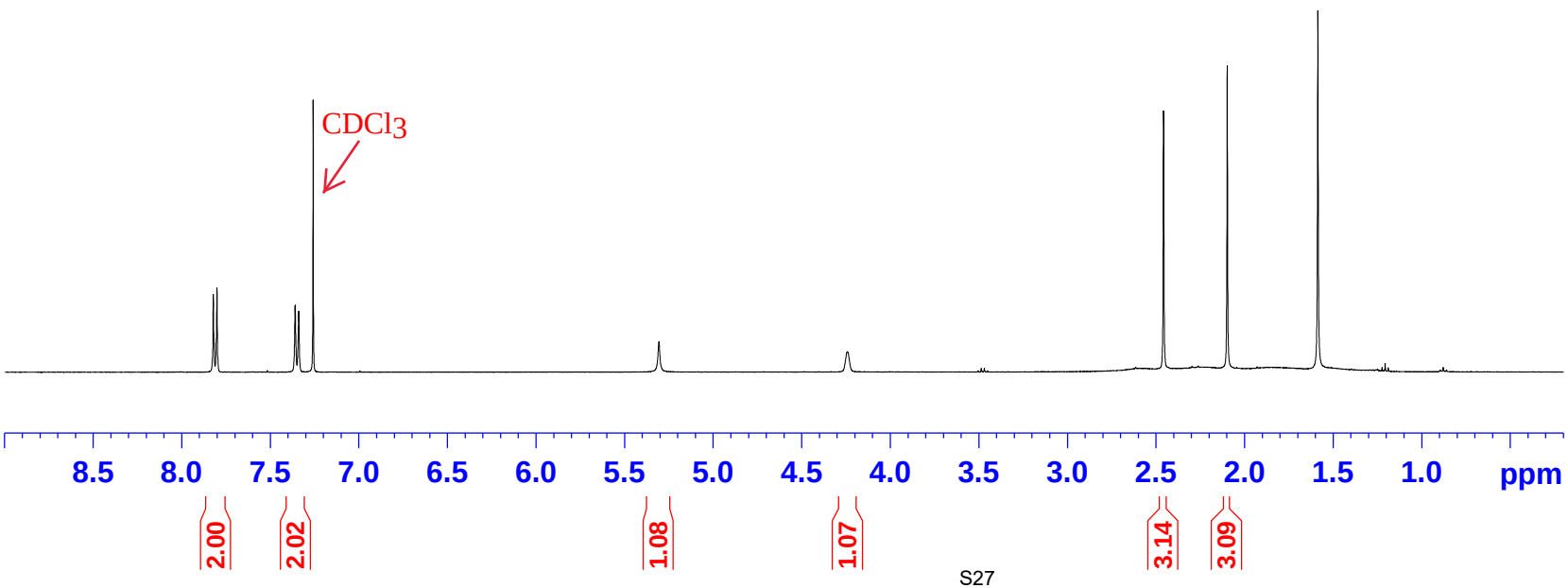
lhr-H-0551-re-cdcl3

7.824 7.803 7.362 7.341 7.260 5.308 4.243 2.459 2.099 1.587



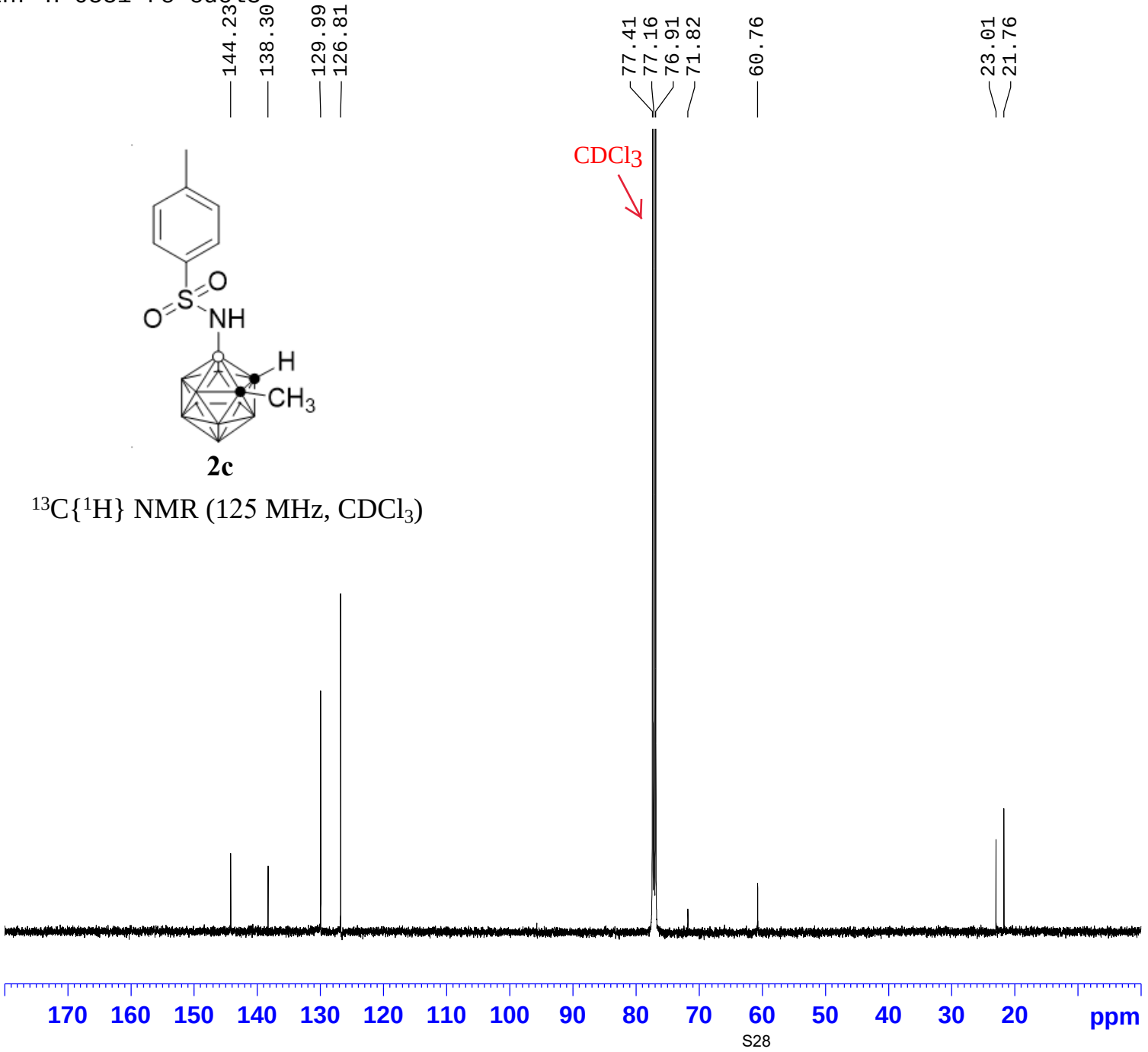
2c

^{1}H NMR (400 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      lhr-H-0551-re-cdcl3
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_            20190402
Time                22.11 h
INSTRUM             spect
PROBHD   Z824601_0021 (
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     12
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                 294.7 K
D1            1.00000000 sec
TD0                     1
SFO1         400.1324708 MHz
NUC1                   1H
P1                 15.00 usec
PLW1          8.31000042 W

F2 - Processing parameters
SI                  65536
SF           400.1300098 MHz
WDW                    EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

lhr-H-0551-re-cdcl3



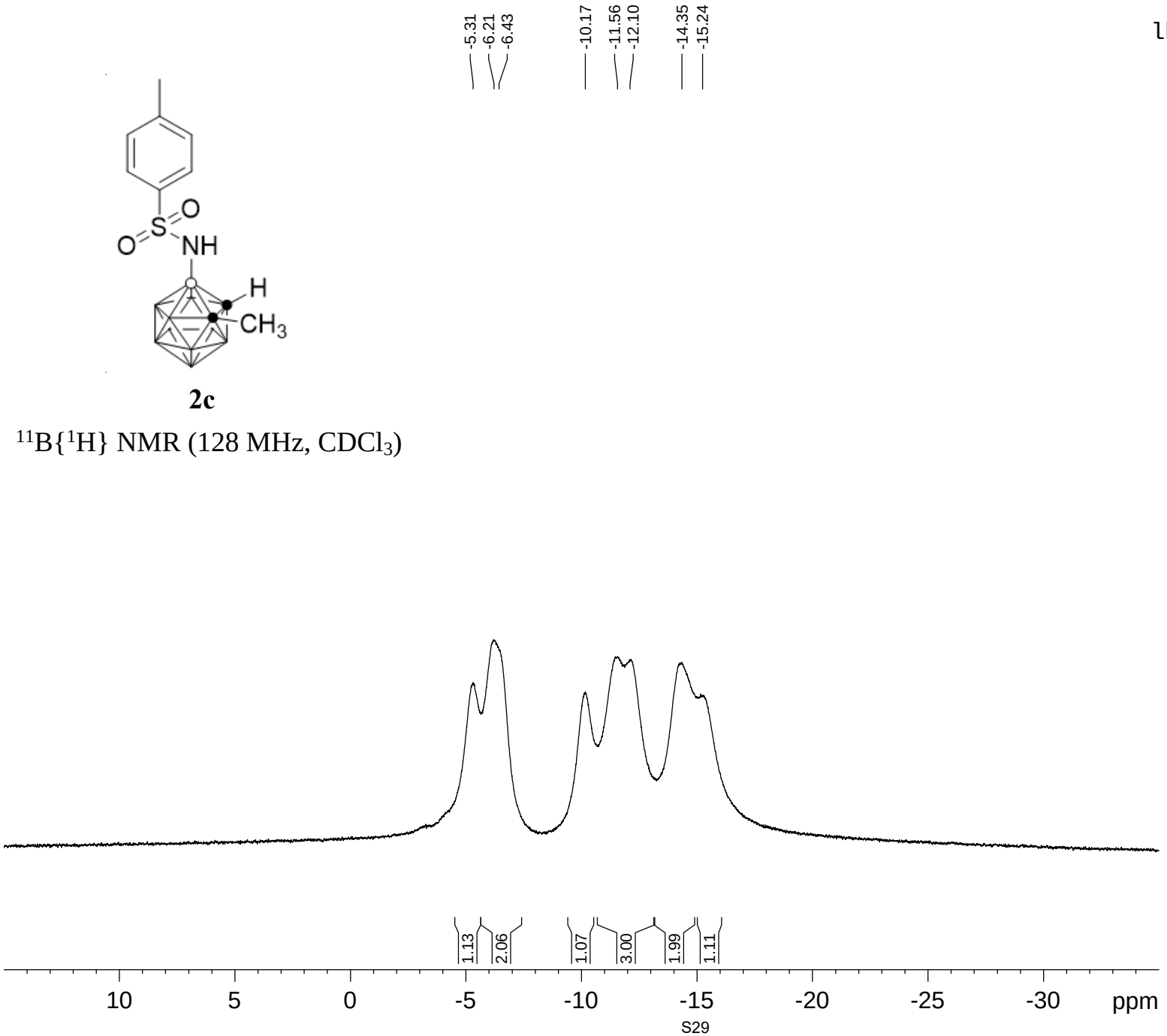
2c

$^{13}C\{^{1}H\}$ NMR (125 MHz, $CDCl_3$)

```
Current Data Parameters
NAME      lhr-H-0551-re-cdcl3
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20190403
Time                 10.04 h
INSTRUM              spect
PROBHD    Z149001_0010 (
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    600
DS                      4
SWH             29761.904 Hz
FIDRES           0.908261 Hz
AQ               1.1010048 sec
RG                  206.72
DW                  16.800 usec
DE                  18.00 usec
TE                  298.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1
SFO1          125.7703643 MHz
NUC1                  13C
P1                  10.00 usec
PLW1          61.00000000 W
SFO2          500.1320005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2               80.00 usec
PLW2          15.00000000 W
PLW12          0.29663000 W
PLW13          0.14920001 W

F2 - Processing parameters
SI                  32768
SF            125.7577714 MHz
WDW                   EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

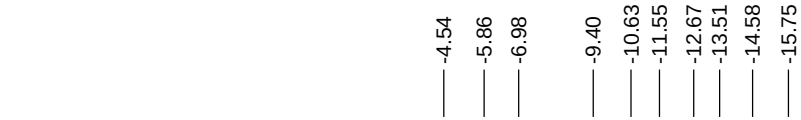
2c

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)

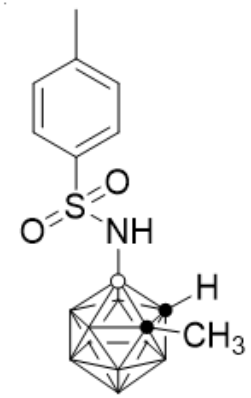


lhr-B-0551-24-CDCl3

```
Current Data Parameters
NAME      lhr-B-0551-24-CDCl3
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20161130
Time                  21.15 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                       287
DW                    20.800 usec
DE                     6.50 usec
TE                    296.2 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
```



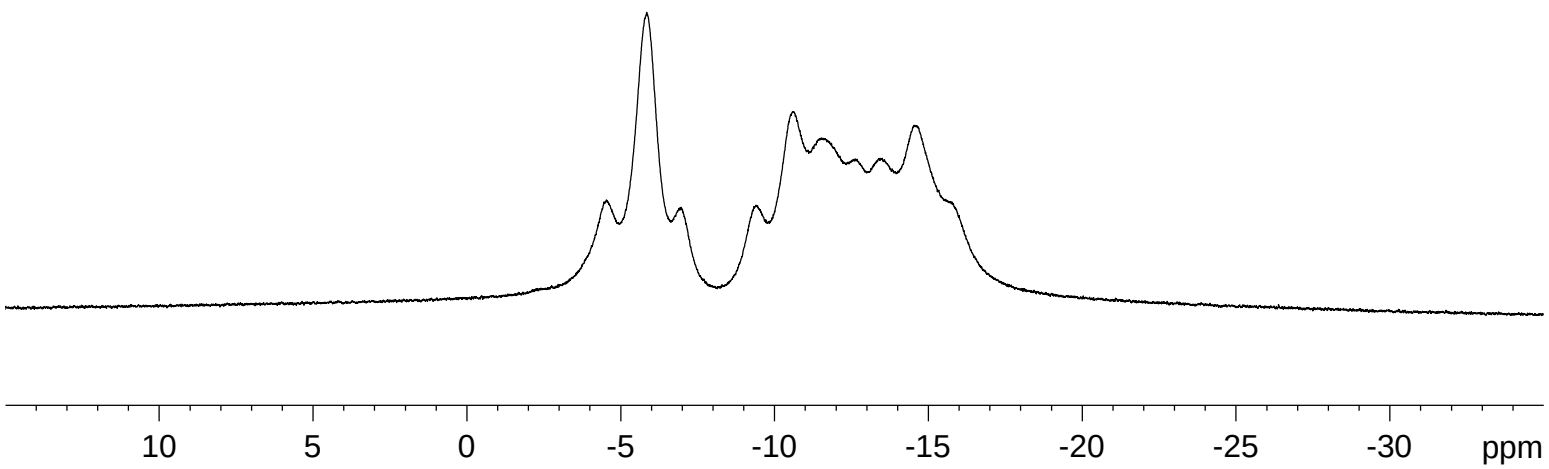
lhr-B-0551-24-CDCl3(C)



2c

^{11}B NMR (128 MHz, $CDCl_3$)

```
Current Data Parameters
NAME      lhr-B-0551-24-CDCl3(C)
EXPNO                          1
PROCNO                         1

F2 - Acquisition Parameters
Date_               20161130
Time                   21.18 h
INSTRUM                spect
PROBHD   Z108618_0257 (
PULPROG                   zg
TD                     65536
SOLVENT                CDCl3
NS                        25
DS                         2
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                 1.3631488 sec
RG                       203
DW                    20.800 usec
DE                     6.50 usec
TE                    296.0 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
```



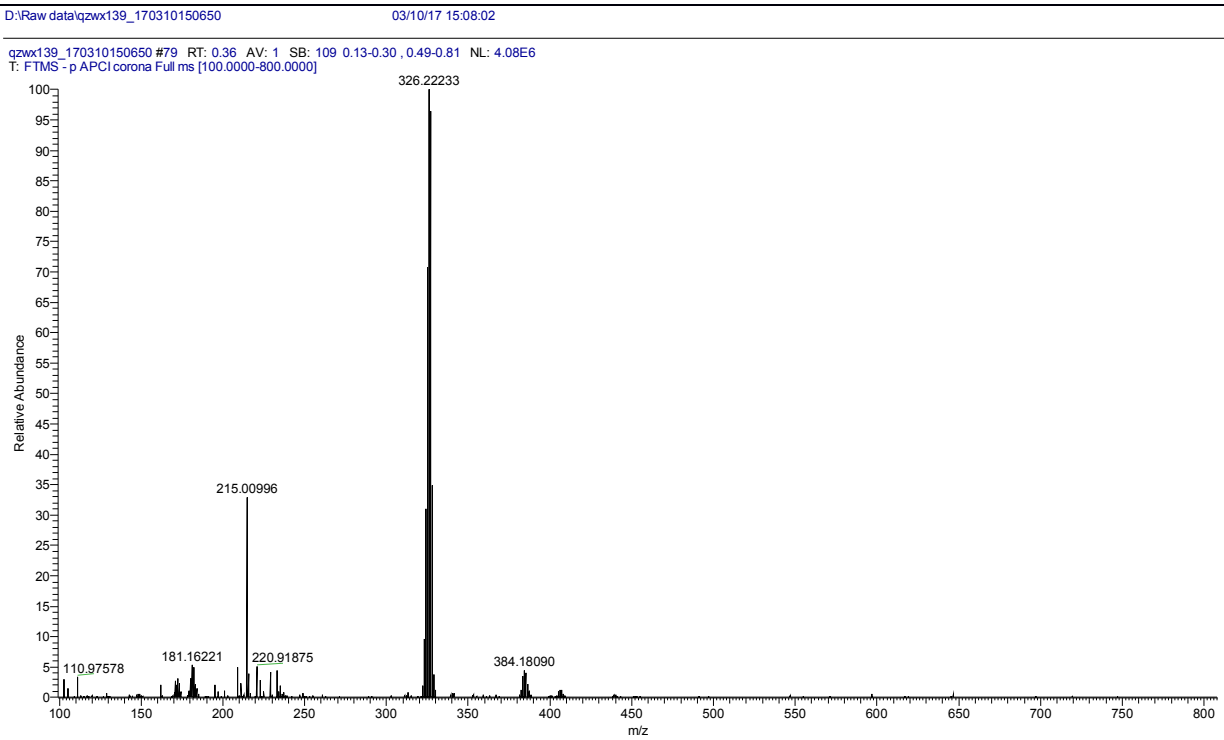
Thermo QEFMS Analysis Report

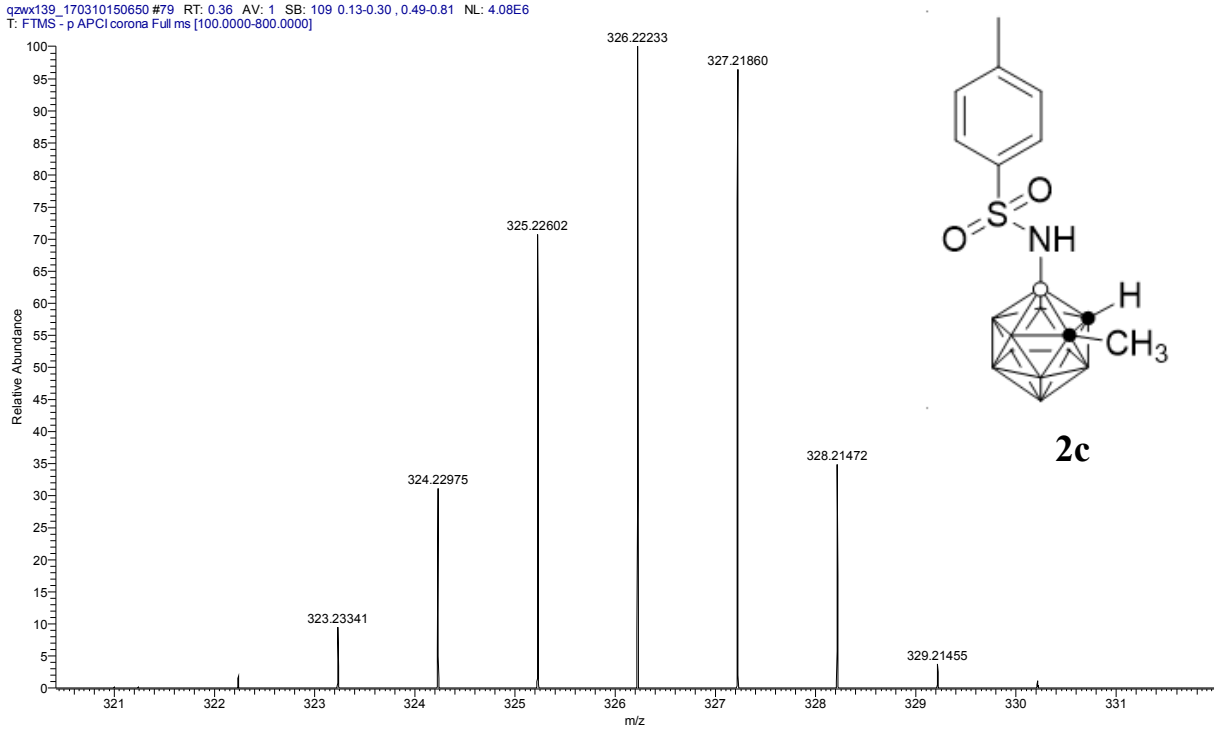
Analysis Info

Sample Name :	Lhr-0551	Reference No.:	Qzwx139
Instrument :	Q Exactive Focus Orbitrap		
Source :	APCI	Polarity :	Negative
Comment :	APCI neg, 5.0uA, by LC, with sheath gas		

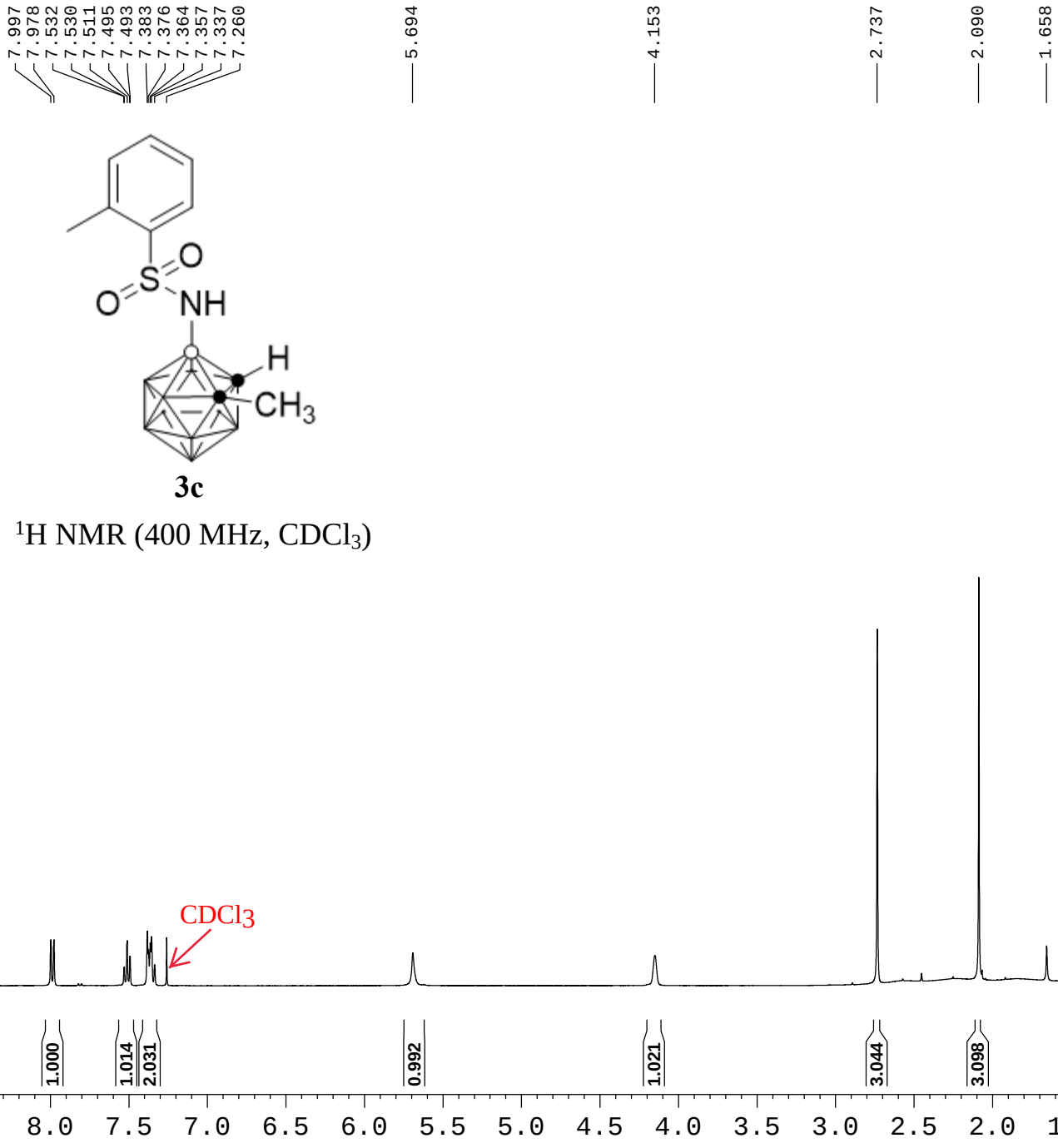
Accurate Mass Measurement

Molecular formula :	$C_{10}H_{21}B_{10}NO_2S$
Experimental Mass $[M-H]^-$:	326.22233
Theoretical Mass $[M-H]^-$:	326.22291
Error (ppm) :	1.7



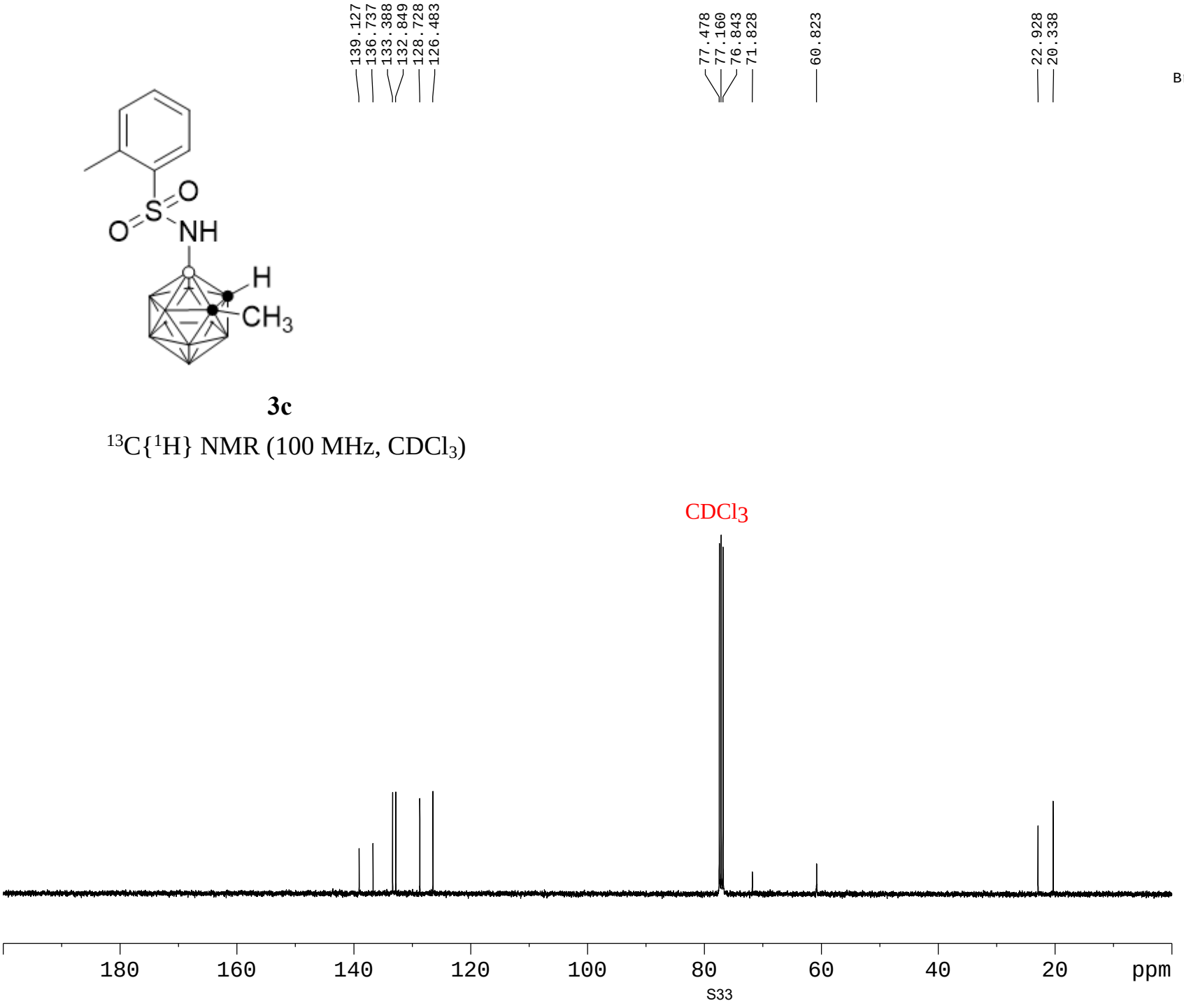


2c


7.997
7.978
7.532
7.530
7.511
7.495
7.493
7.383
7.376
7.364
7.357
7.337
7.260
5.694
4.153
2.737
2.090
1.658
lhr-H-0577-cc-CDCl3
Bruker Advance III 400
Current Data Parameters
NAME lhr-H-0577-cc-CDCl3
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161230
Time 14.35 h
INSTRUM spect
PROBHD Z824601_0021 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 12
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 295.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 15.00 usec
PLW1 8.31000042 W
F2 - Processing parameters
SI 65536
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
S
O
NH
H
CH3
3c
1H NMR (400 MHz, CDCl3)
CDCl3
1.000
1.014
2.031
0.992
1.021
3.044
3.098
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

3c

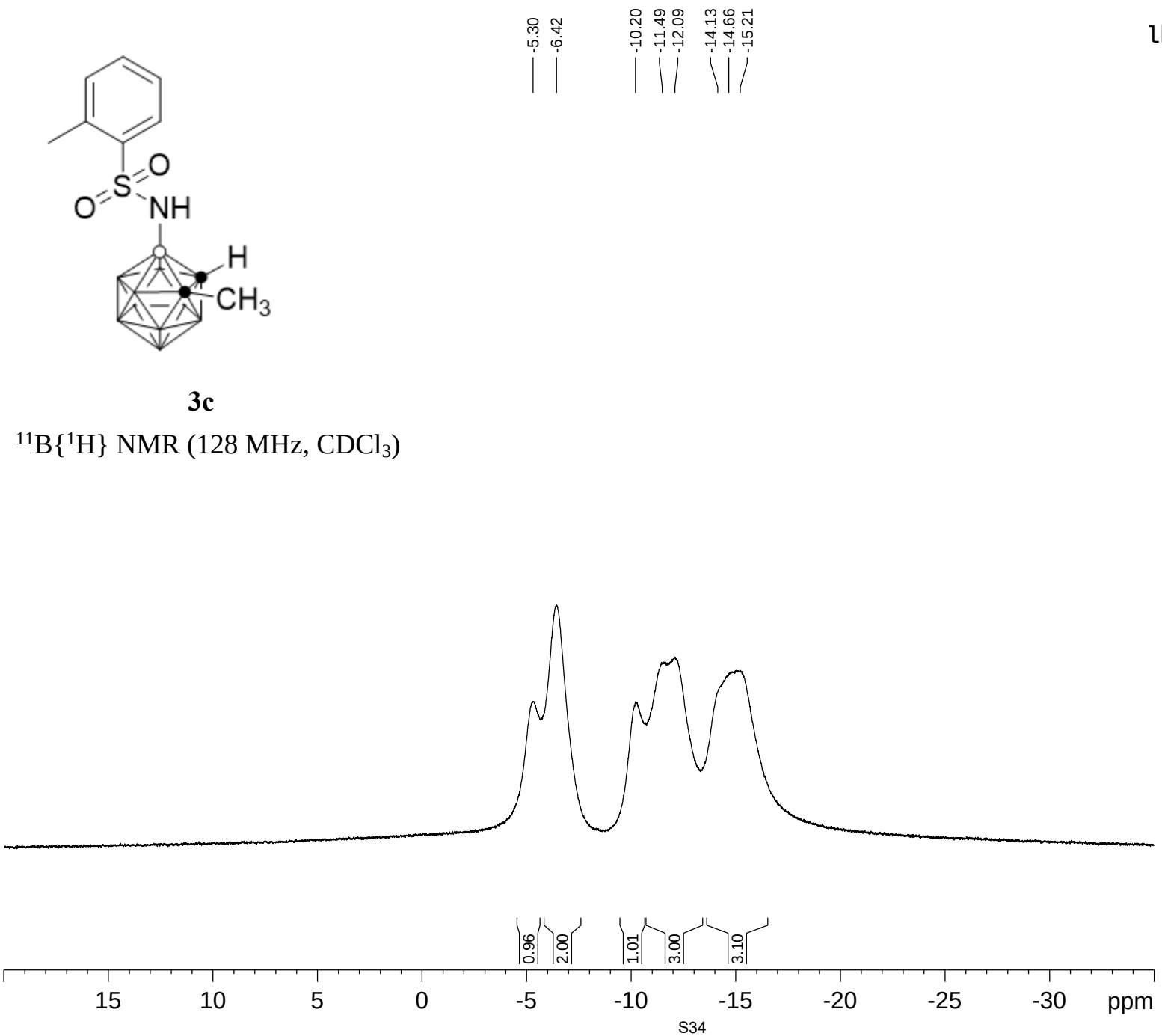
$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)




```
lhr-C-0577-cc-CDCl3

Bruker Advance III 400

Current Data Parameters
NAME      lhr-C-0577-cc-CDCl3
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_           20161230
Time              14.39 h
INSTRUM             spect
PROBHD   Z824601_0021 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   394
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                   203
DW                20.800 usec
DE                 6.50 usec
TE                295.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1
SFO1         100.6228298 MHz
NUC1                 13C
P1                 9.50 usec
PLW1         41.25000000 W
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2          8.31000042 W
PLW12         0.23083000 W
PLW13         0.11611000 W

F2 - Processing parameters
SI                 32768
SF           100.6127569 MHz
WDW                   EM
SSB      0
LB                 1.00 Hz
GB       0
PC                 1.40
```

3c

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)

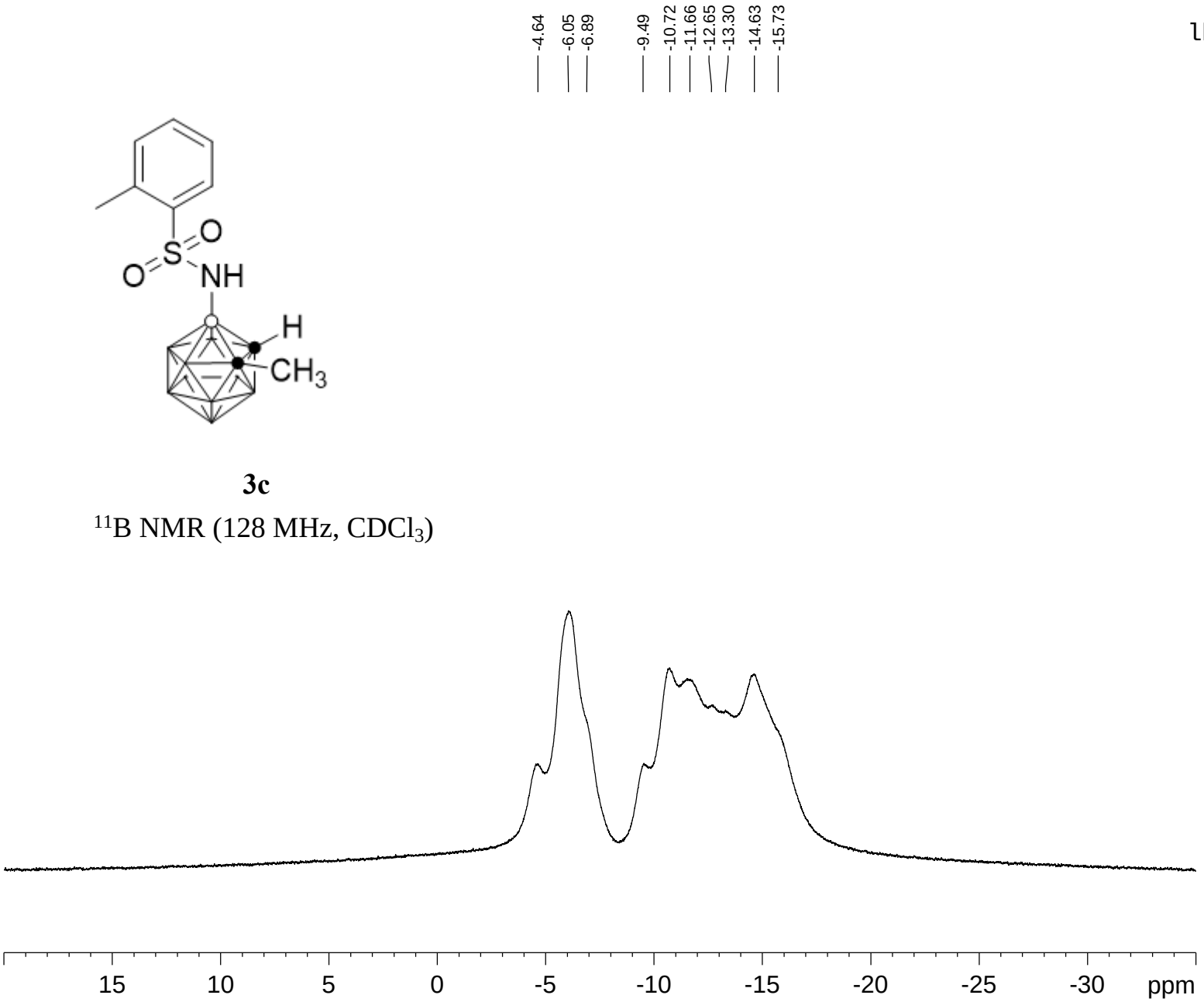


lhr-B-0577-cc-CDCl3

```
Current Data Parameters
NAME      lhr-B-0577-cc-CDCl3
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20161230
Time                  14.25 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                       287
DW                    20.800 usec
DE                     6.50 usec
TE                    295.3 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
```

3c

^{11}B NMR (128 MHz, $CDCl_3$)



lhr-B-0577-cc-CDCl3(C)

```
Current Data Parameters
NAME      lhr-B-0577-cc-CDCl3(C)
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161230
Time               14.28 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG               zg
TD                 65536
SOLVENT            CDCl3
NS                    25
DS                     2
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                   203
DW                20.800 usec
DE                 6.50 usec
TE                295.1 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           128.4097504 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

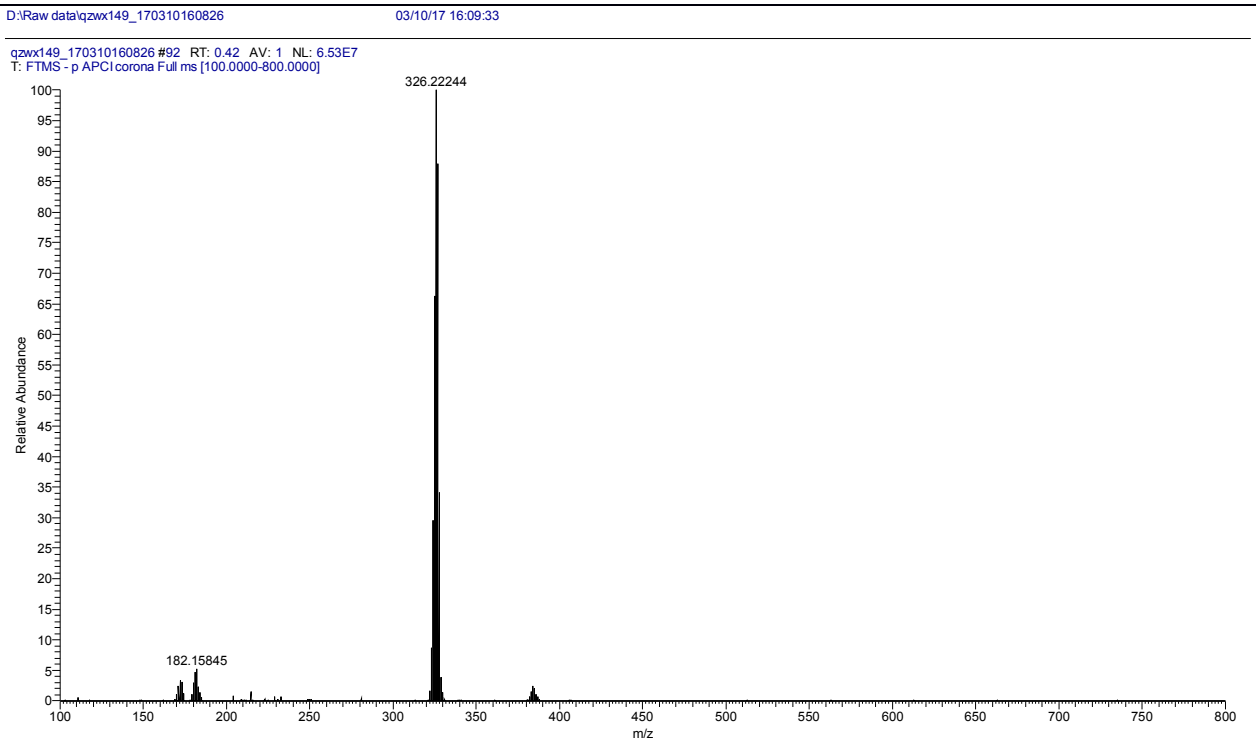
Thermo QEFMS Analysis Report

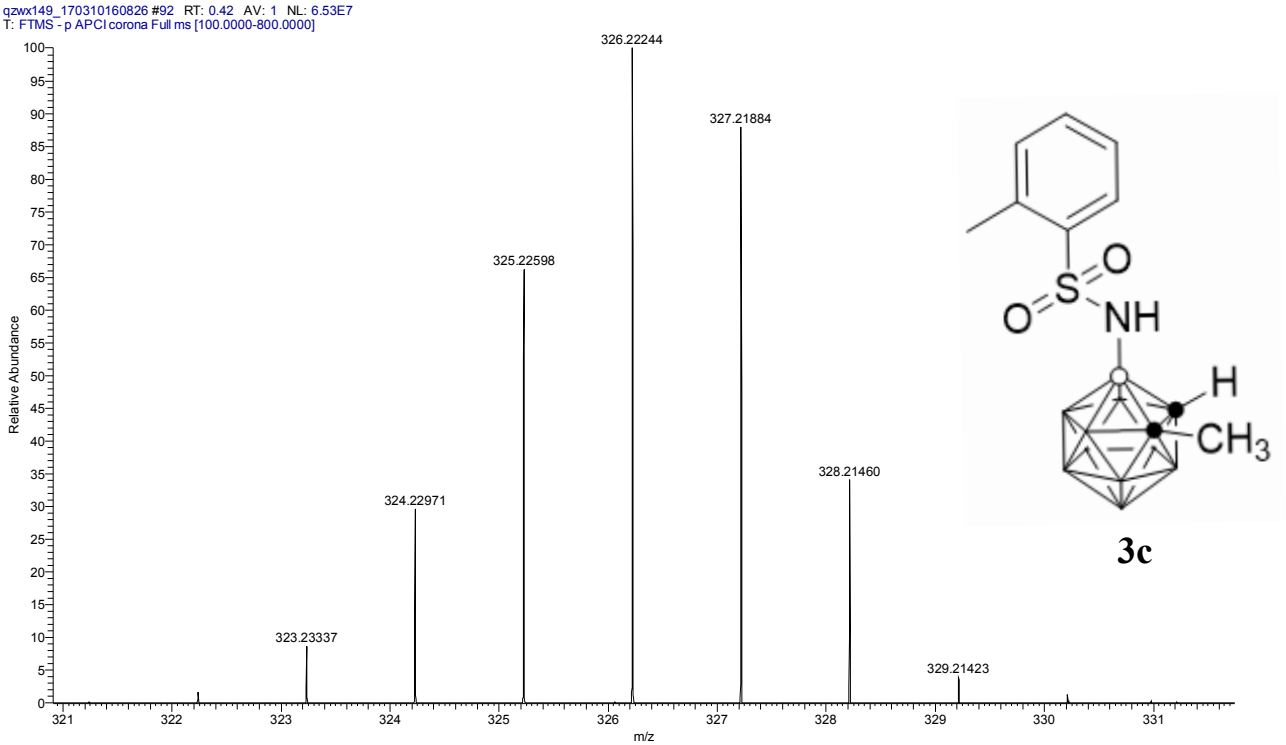
Analysis Info

Sample Name :	Lhr-0577	Reference No.:	Qzwx149
Instrument :	Q Exactive Focus Orbitrap		
Source :	APCI	Polarity :	Negative
Comment :	APCI neg, 5.0uA, by LC, with sheath gas		

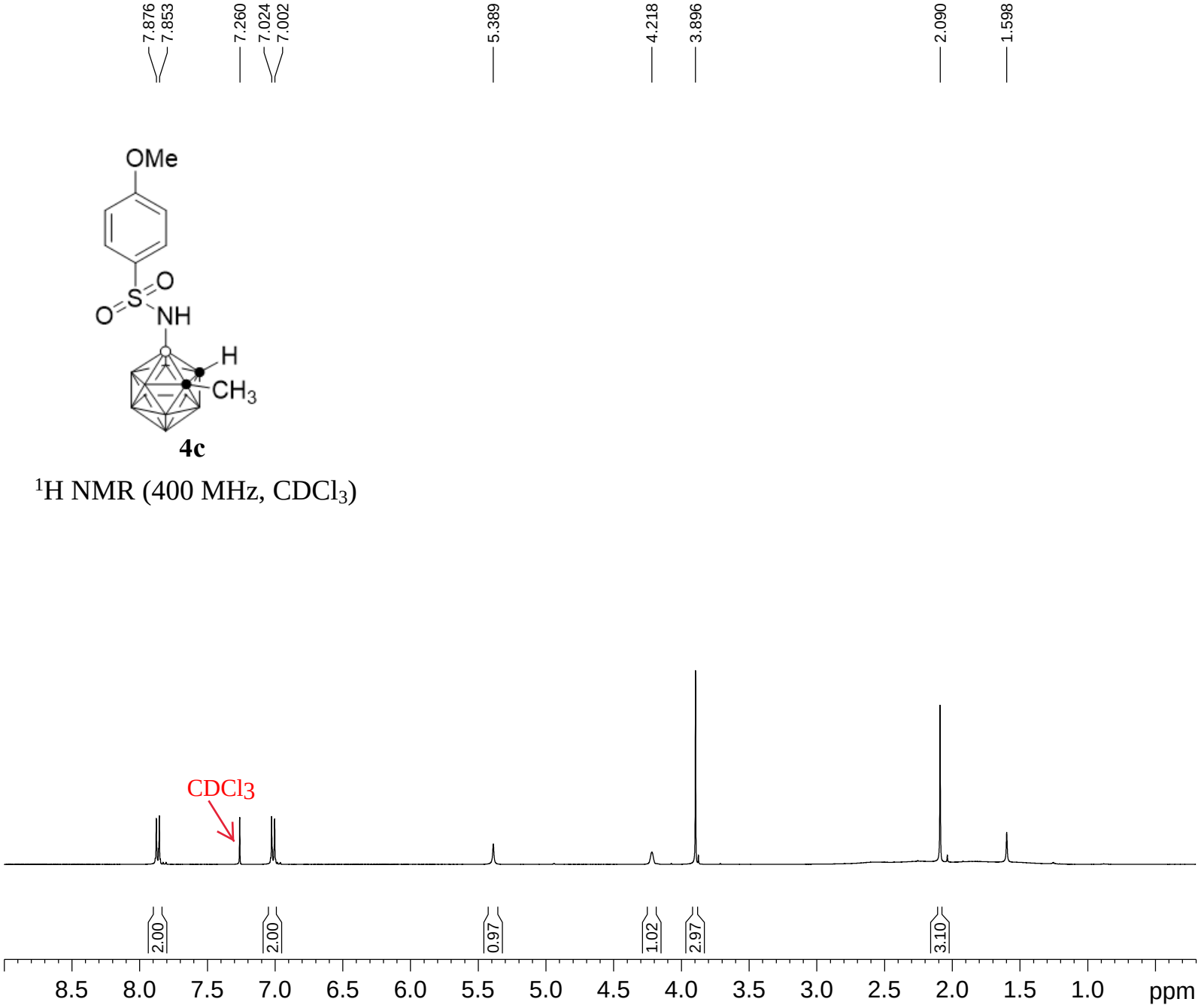
Accurate Mass Measurement

Molecular formula :	$C_{10}H_{21}B_{10}NO_2S$
Experimental Mass $[M-H]^-$:	326.22244
Theoretical Mass $[M-H]^-$:	326.22291
Error (ppm) :	1.4





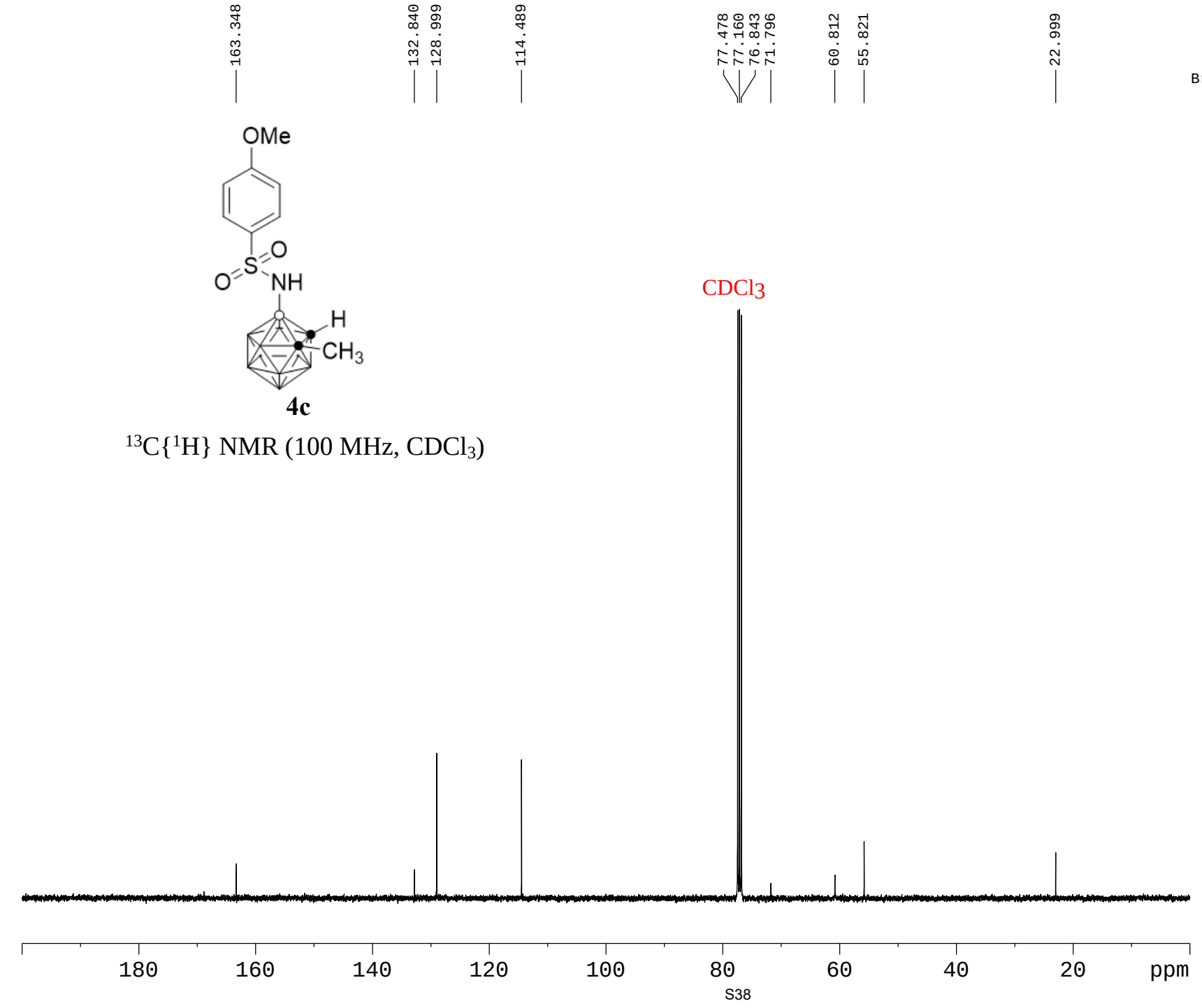
3c



Current Data Parameters
NAME lhr-H-0559-2-cc-CDCl3
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161215
Time 11.02 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 128
DW 62.400 usec
DE 6.50 usec
TE 295.7 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 400.2300106 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



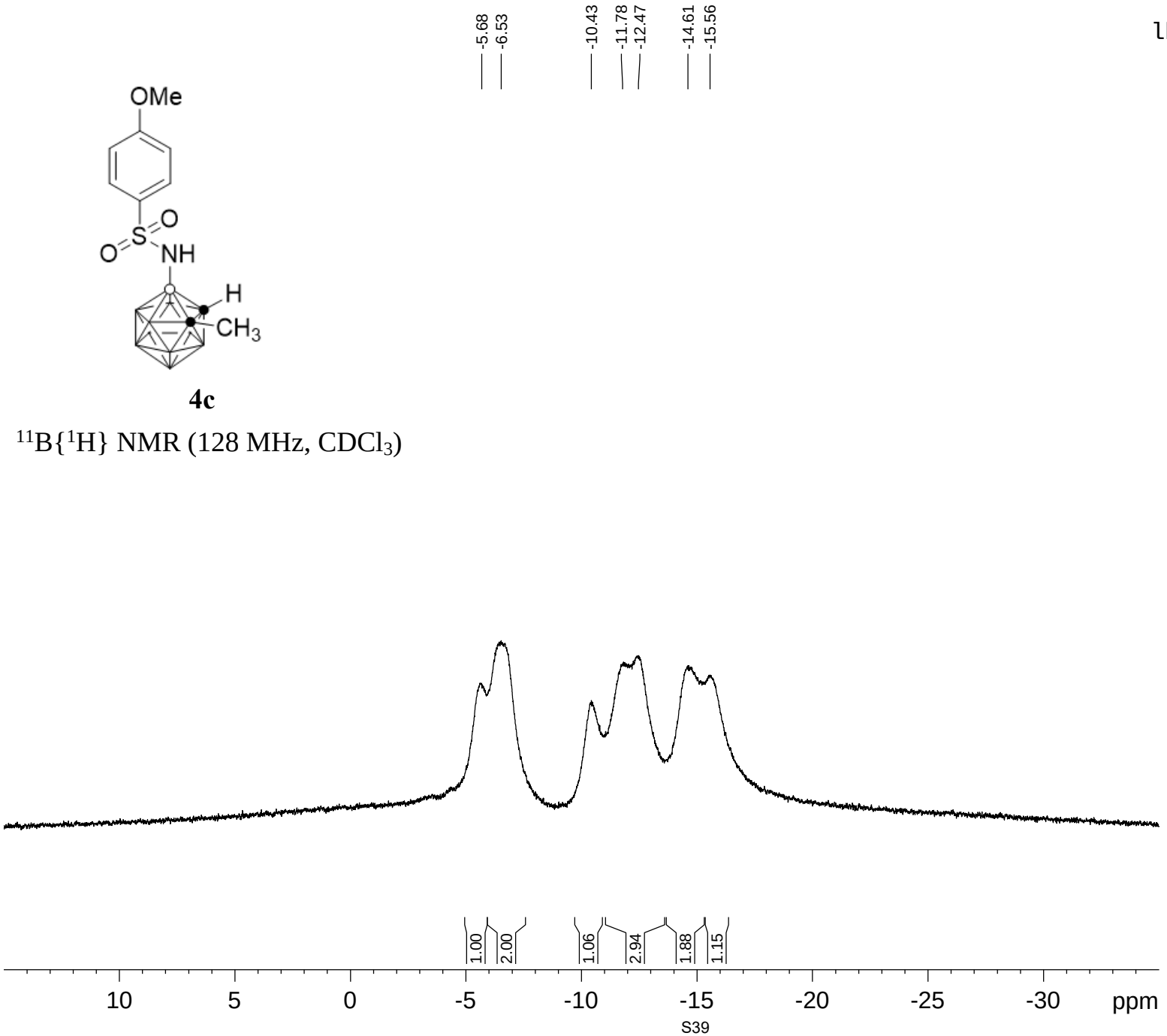
4c

$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)

```
lhr-C-0559-2-cc-CDCl3

Bruker Advance III 400

Current Data Parameters
NAME      lhr-C-0559-2-cc-CDCl3
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_           20161215
Time              11.48 h
INSTRUM             spect
PROBHD   Z824601_0021 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   880
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631488 sec
RG                   203
DW                20.800 usec
DE                 6.50 usec
TE                295.3 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1         100.6228298 MHz
NUC1                13C
P1                 9.50 usec
PLW1         41.25000000 W
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2          8.31000042 W
PLW12         0.23083000 W
PLW13         0.11611000 W

F2 - Processing parameters
SI                 32768
SF           100.6127556 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

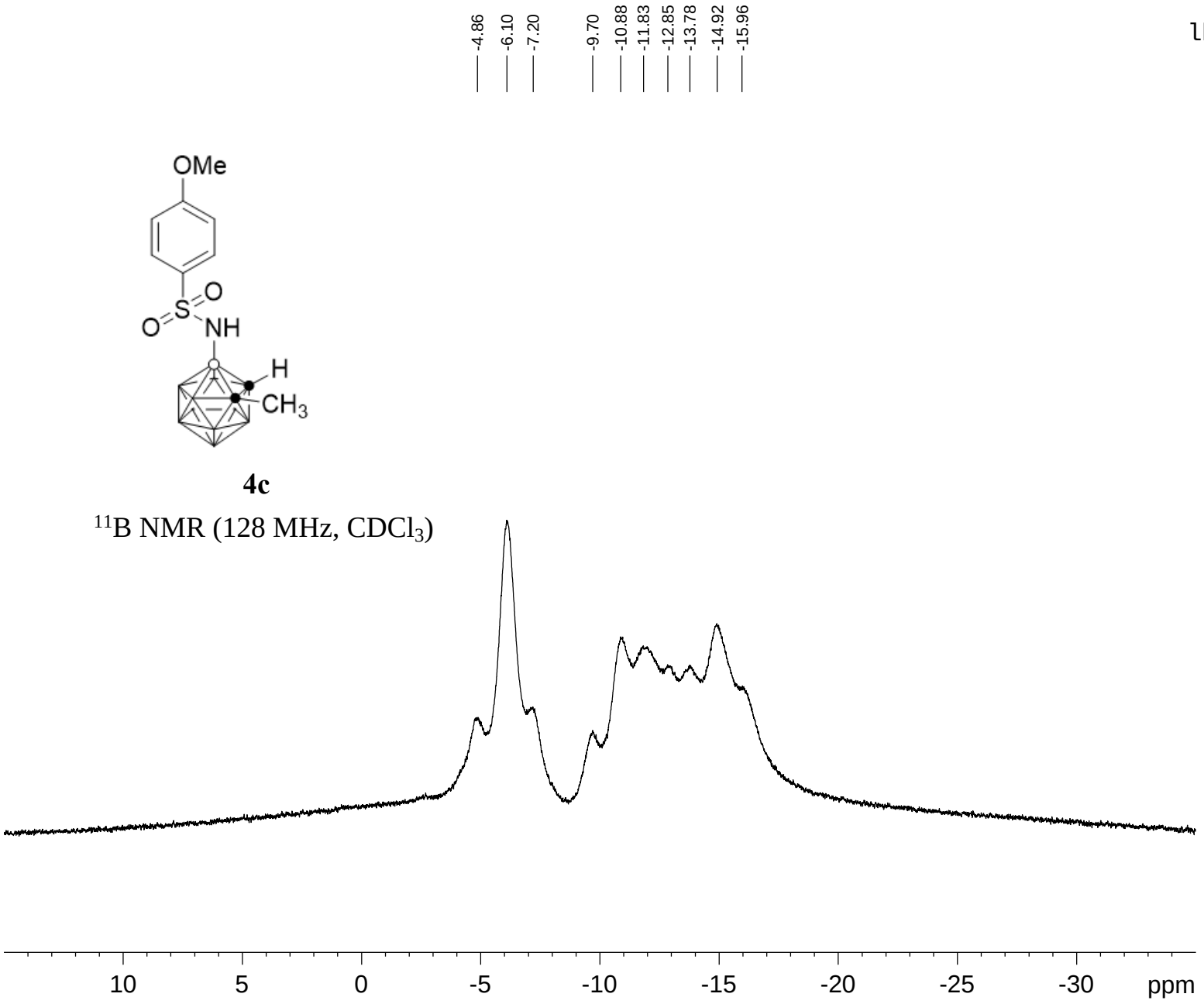
4c

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)



lhr-B-0559-cc-CDCl3

```
Current Data Parameters
NAME      lhr-B-0559-cc-CDCl3
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20161215
Time                  16.30 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                       287
DW                    20.800 usec
DE                     6.50 usec
TE                    296.7 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
```



4c

^{11}B NMR (128 MHz, $CDCl_3$)

lhr-B-0559-cc-CDCl3(C)

```
Current Data Parameters
NAME      lhr-B-0559-cc-CDCl3(C)
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161215
Time               16.32 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG              zg
TD                65536
SOLVENT           CDCl3
NS                   33
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                296.4 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
```

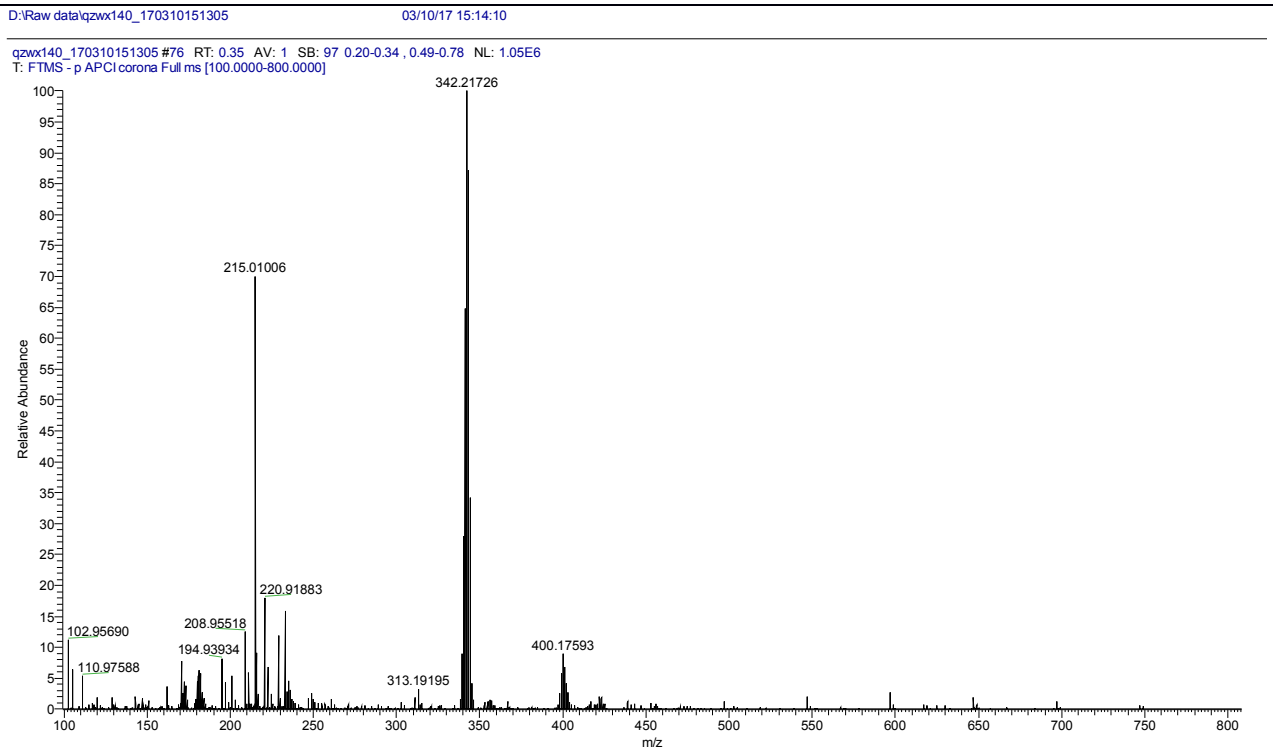
Thermo QEFMS Analysis Report

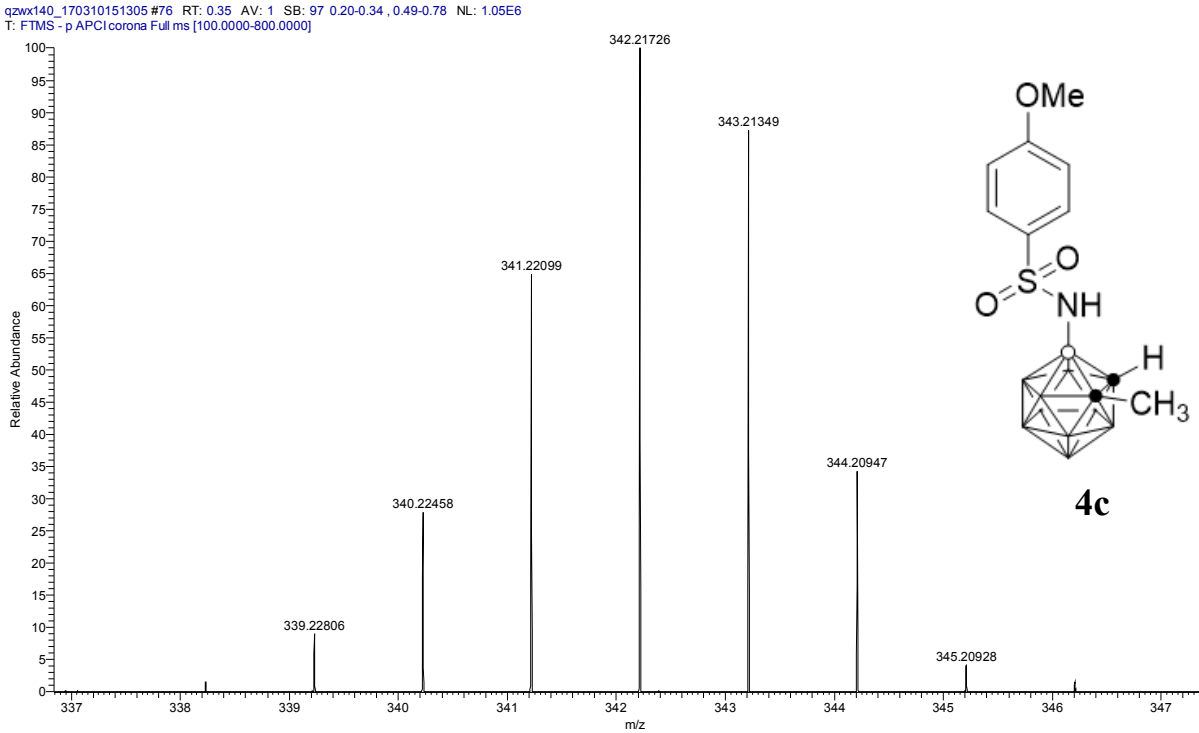
Analysis Info

Sample Name :	Lhr-0559	Reference No.:	Qzwx140
Instrument :	Q Exactive Focus Orbitrap		
Source :	APCI	Polarity :	Negative
Comment :	APCI neg, 5.0uA, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{10}H_{21}B_{10}NO_3S$
Experimental Mass $[M-H]^-$:	342.21726
Theoretical Mass $[M-H]^-$:	342.21783
Error (ppm) :	1.6

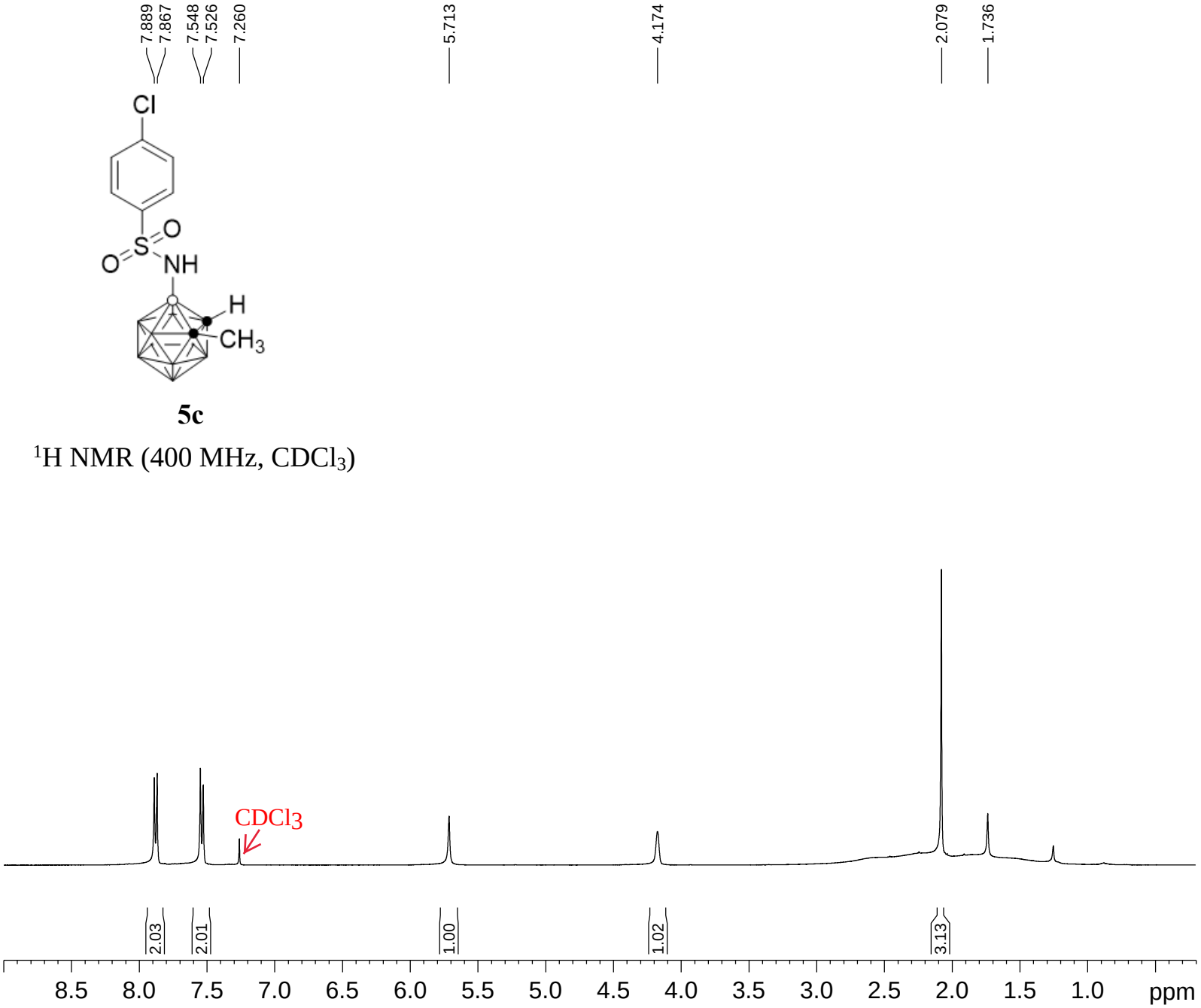




4c

5c

^{1}H NMR (400 MHz, $CDCl_3$)

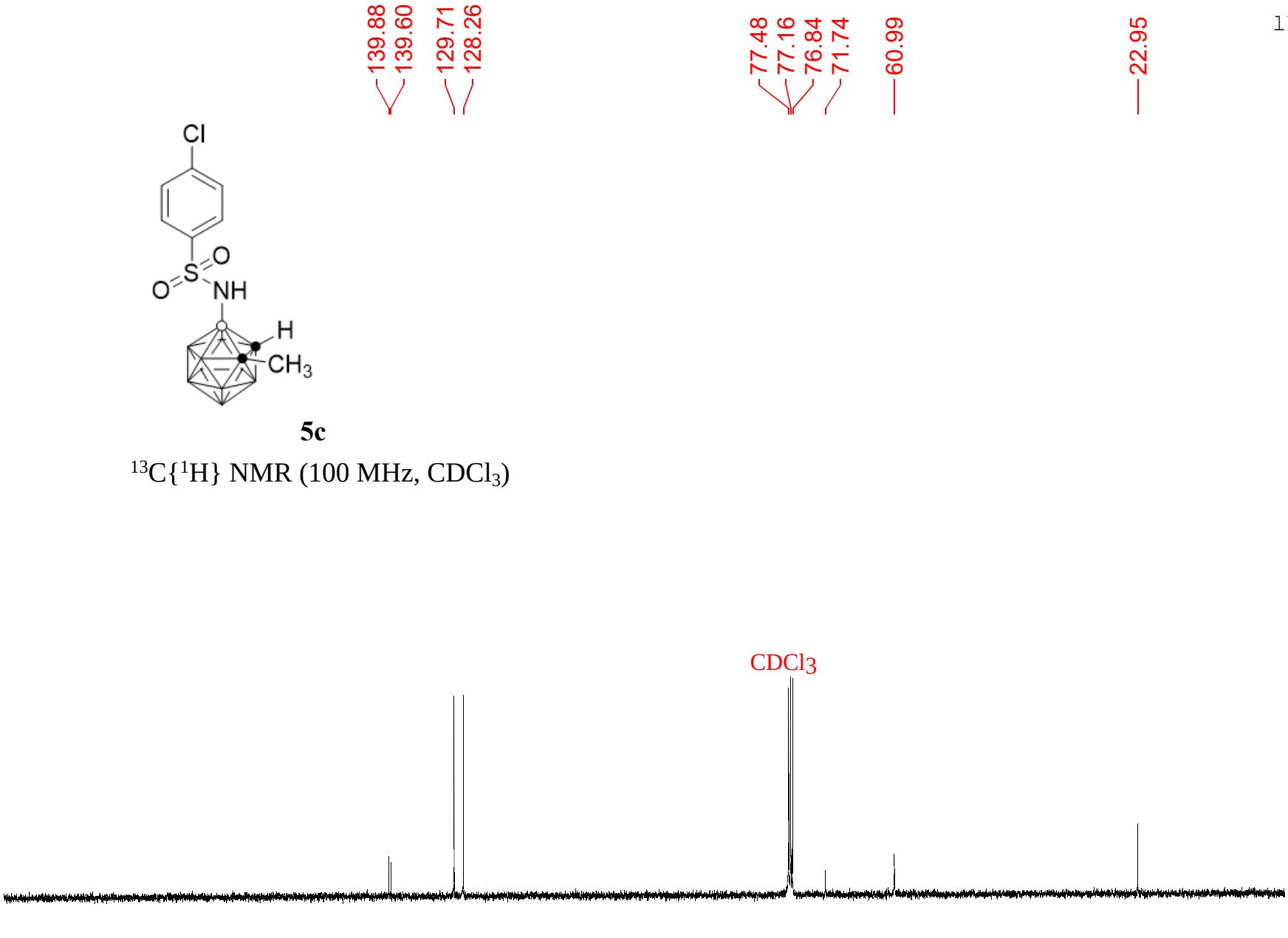


lhr-H-0557-10-CDCl3

Current Data Parameters
NAME lhr-H-0557-10-CDC
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161221
Time 13.07 h
INSTRUM spect
PROBHD Z108618_0257 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 90.5
DW 62.400 usec
DE 6.50 usec
TE 294.8 K
D1 1.00000000 sec
TD0 1
SFO1 400.2324714 MH
NUC1 1H
P1 12.80 usec
PLW1 13.56000042 W

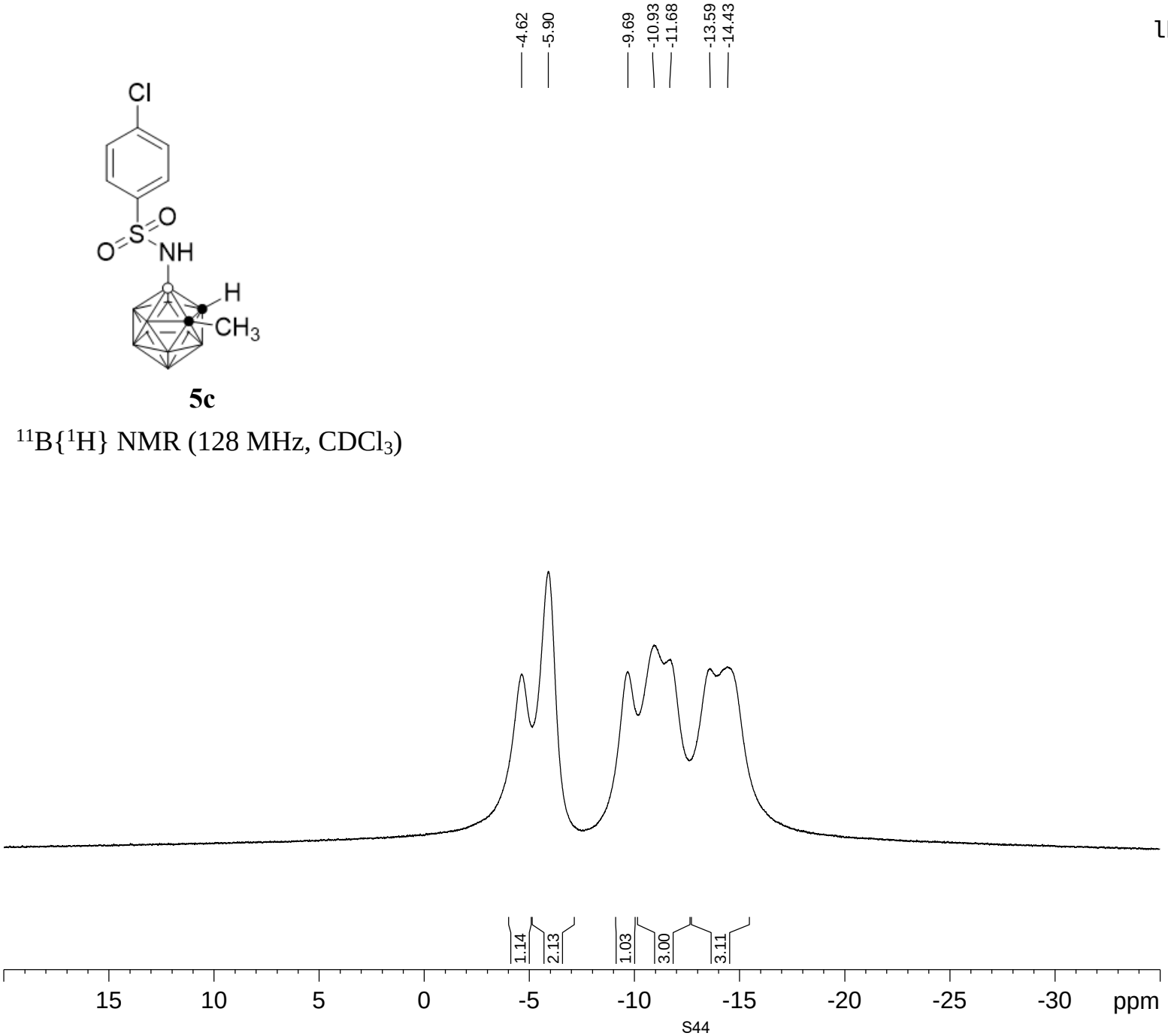
F2 - Processing parameters
SI 65536
SF 400.2300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00




```
Current Data Parameters
NAME      lhr-C-0557-10-cc-CDCl3
EXPNO                          1
PROCNO                         1

F2 - Acquisition Parameters
Date_              20161221
Time_                 13.10 h
INSTRUM               spect
PROBHD    Z108618_0257 (
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     120
DS                       4
SWH              29761.904 Hz
FIDRES            0.454131 Hz
AQ                1.1010048 sec
RG                     203
DW                  16.800 usec
DE                   6.50 usec
TE                  295.2 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             100.6379021 MHz
WDW                    EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

5c

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)

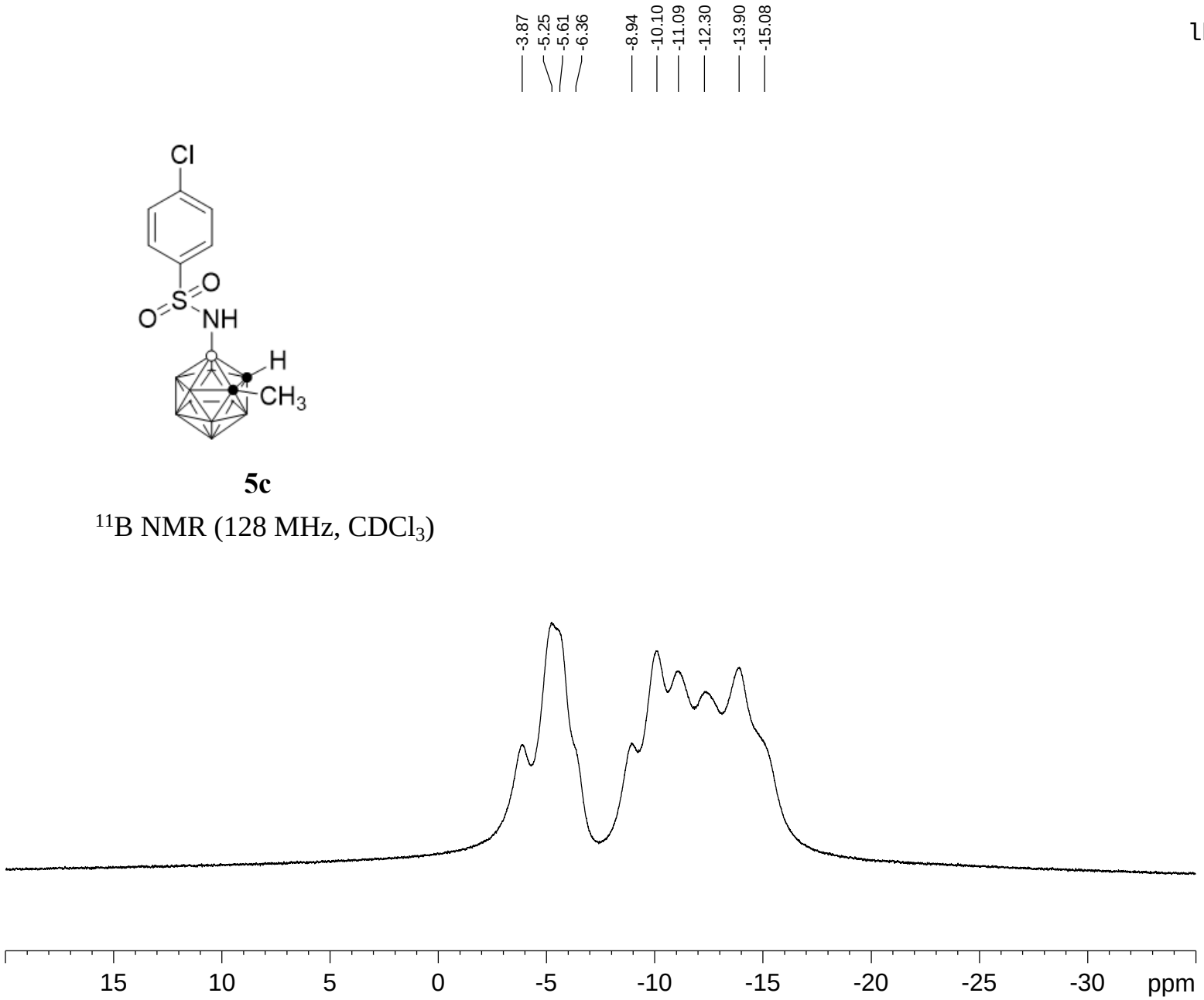


lhr-B-0557-10-cc-CDCl3

```
Current Data Parameters
NAME      lhr-B-0557-10-cc-CDCl3
EXPNO                          1
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20161221
Time                       13.00 h
INSTRUM                    spect
PROBHD   Z108618_0257 (
PULPROG                     zgdc
TD                         65536
SOLVENT                    CDCl3
NS                            39
DS                             4
SWH                    24038.461 Hz
FIDRES                  0.366798 Hz
AQ                     1.3631488 sec
RG                           287
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
```

5c

^{11}B NMR (128 MHz, $CDCl_3$)



lhr-B-0557-10-cc-CDCl3(C

```
Current Data Parameters
NAME      lhr-B-0557-10-cc-CDCl3(C)
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161221
Time              13.02 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG               zg
TD                 65536
SOLVENT            CDCl3
NS                    24
DS                     2
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                   203
DW                20.800 usec
DE                 6.50 usec
TE                295.0 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
```

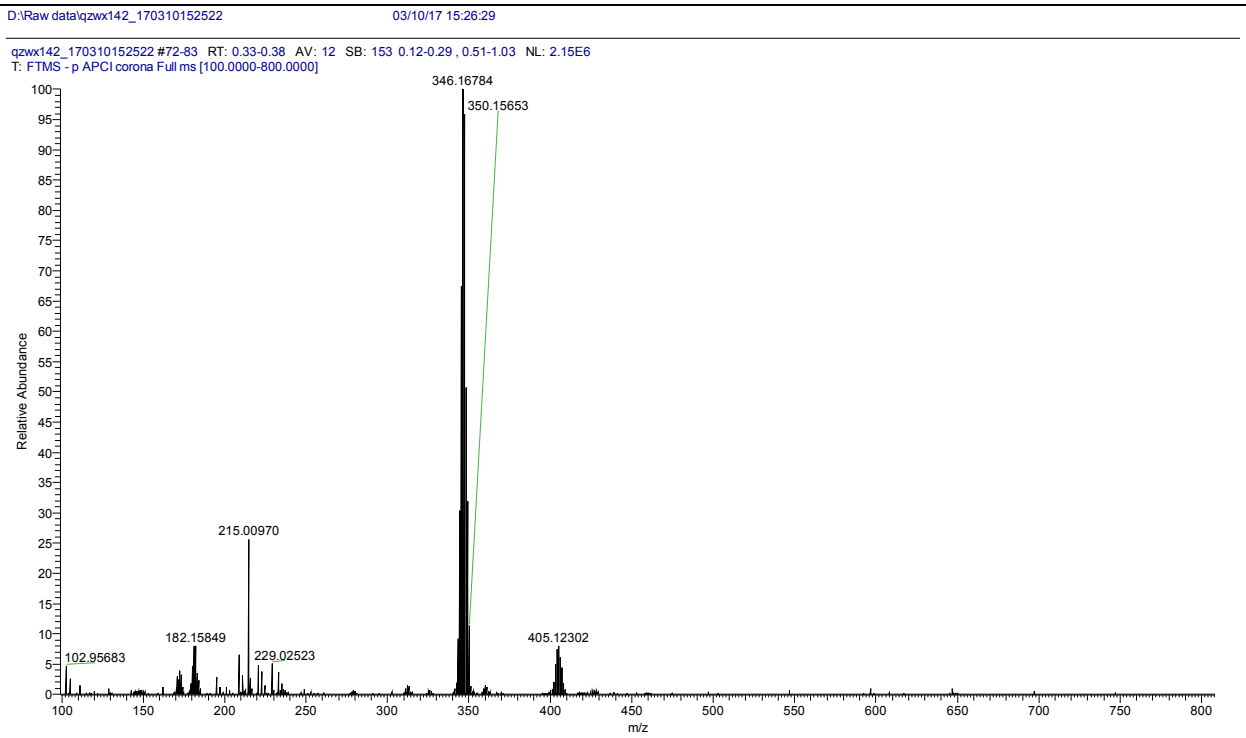
Thermo QEFMS Analysis Report

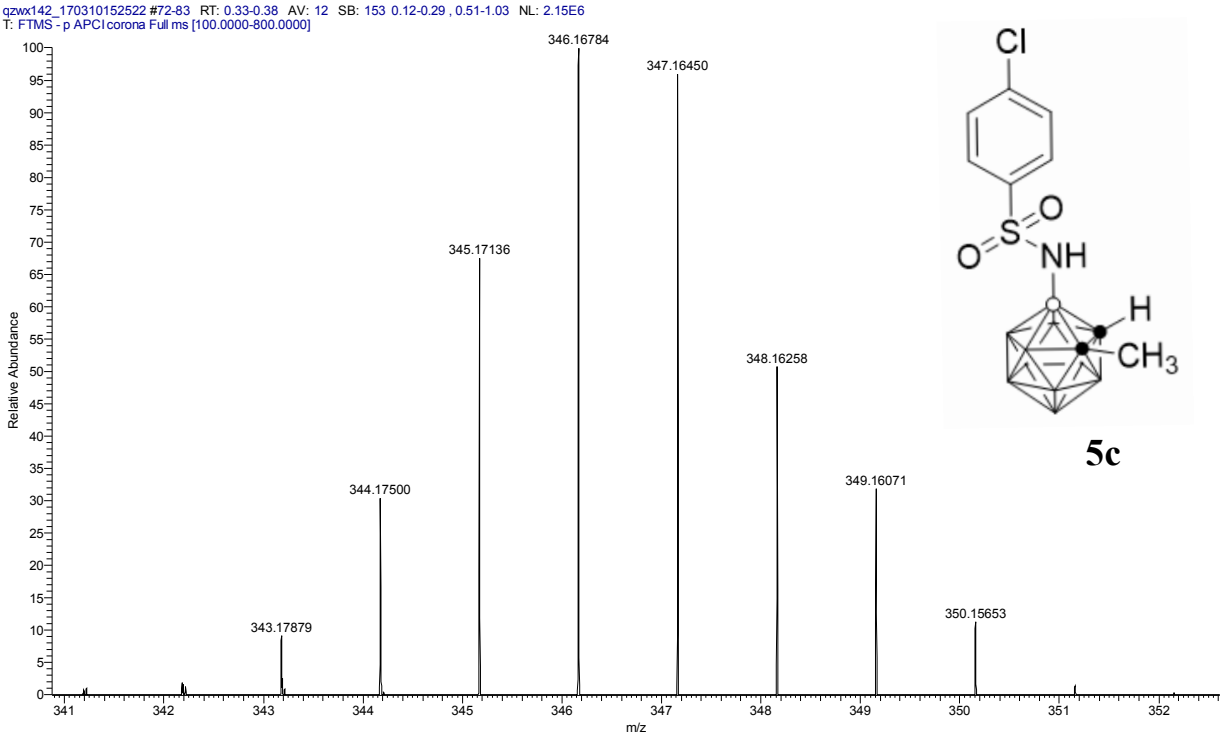
Analysis Info

Sample Name :	Lhr-0557	Reference No.:	Qzwx142
Instrument :	Q Exactive Focus Orbitrap		
Source :	APCI	Polarity :	Negative
Comment :	APCI neg, 5.0uA, by LC, with sheath gas		

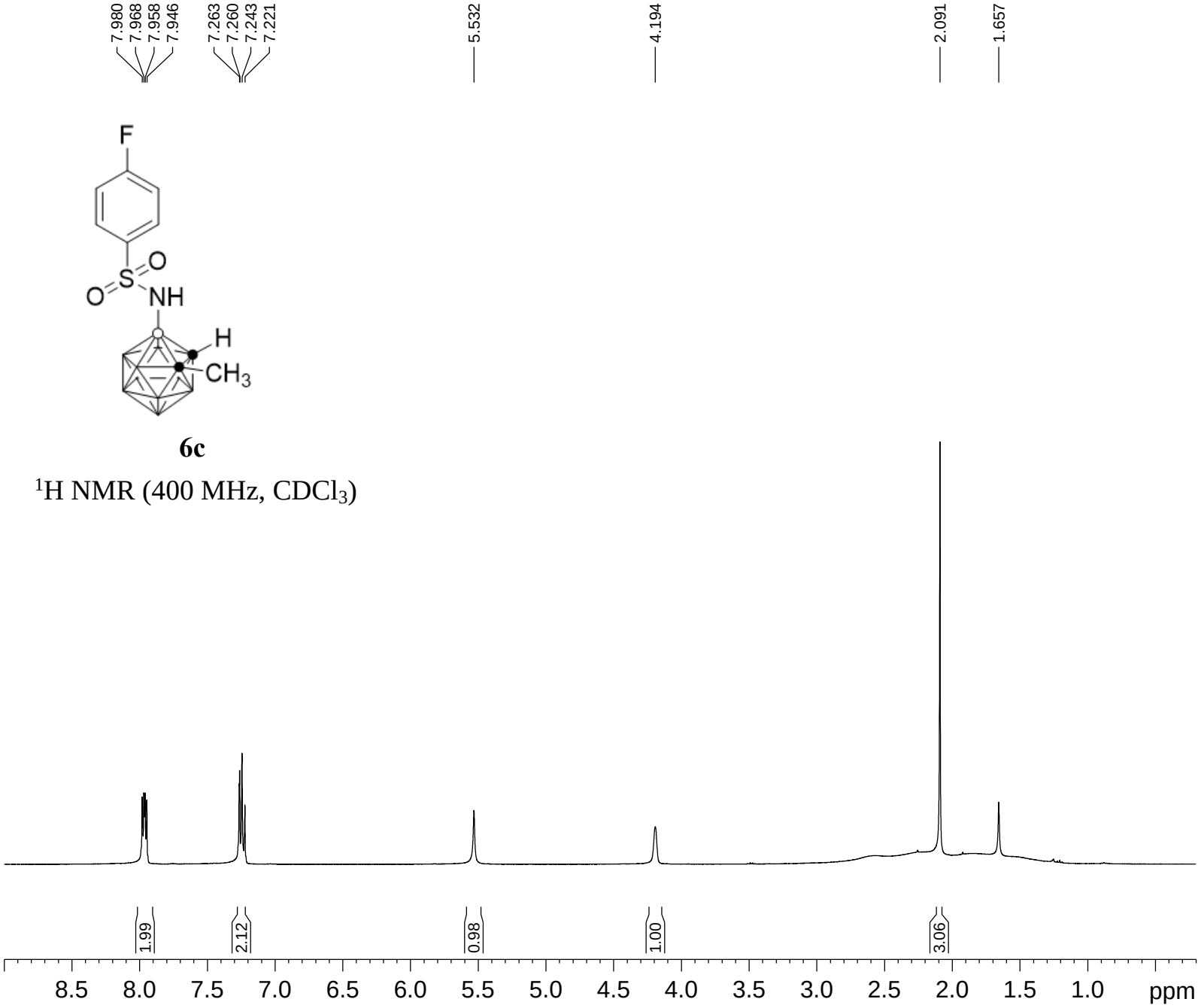
Accurate Mass Measurement

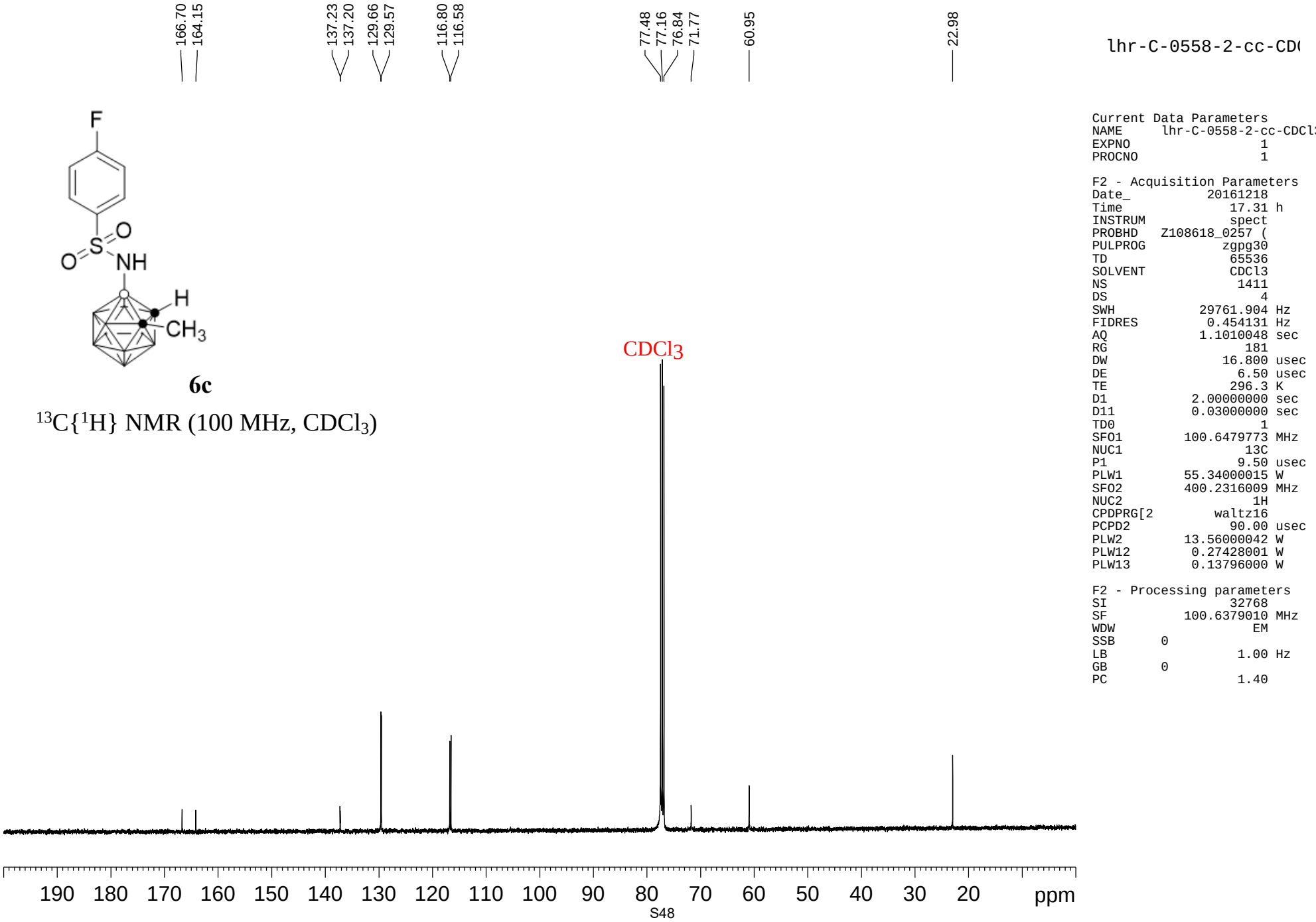
Molecular formula :	$C_9H_{18}B_{10}ClNO_2S$
Experimental Mass $[M-H]^-$:	346.16784
Theoretical Mass $[M-H]^-$:	346.16856
Error (ppm) :	2.0





5c


7.980
7.968
7.958
7.946
7.263
7.260
7.243
7.221
5.532
4.194
2.091
1.657
lhr-H-0558-2-cc-CDCl
F
O
S
O
NH
H
CH3
6c
1H NMR (400 MHz, CDCl3)
Current Data Parameters
NAME lhr-H-0558-2-cc-CDCl3
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161218
Time 17.27 h
INSTRUM spect
PROBHD Z108618_0257 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 10
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 80.6
DW 62.400 usec
DE 6.50 usec
TE 295.9 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 400.2300105 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.99
2.12
0.98
1.00
3.06
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

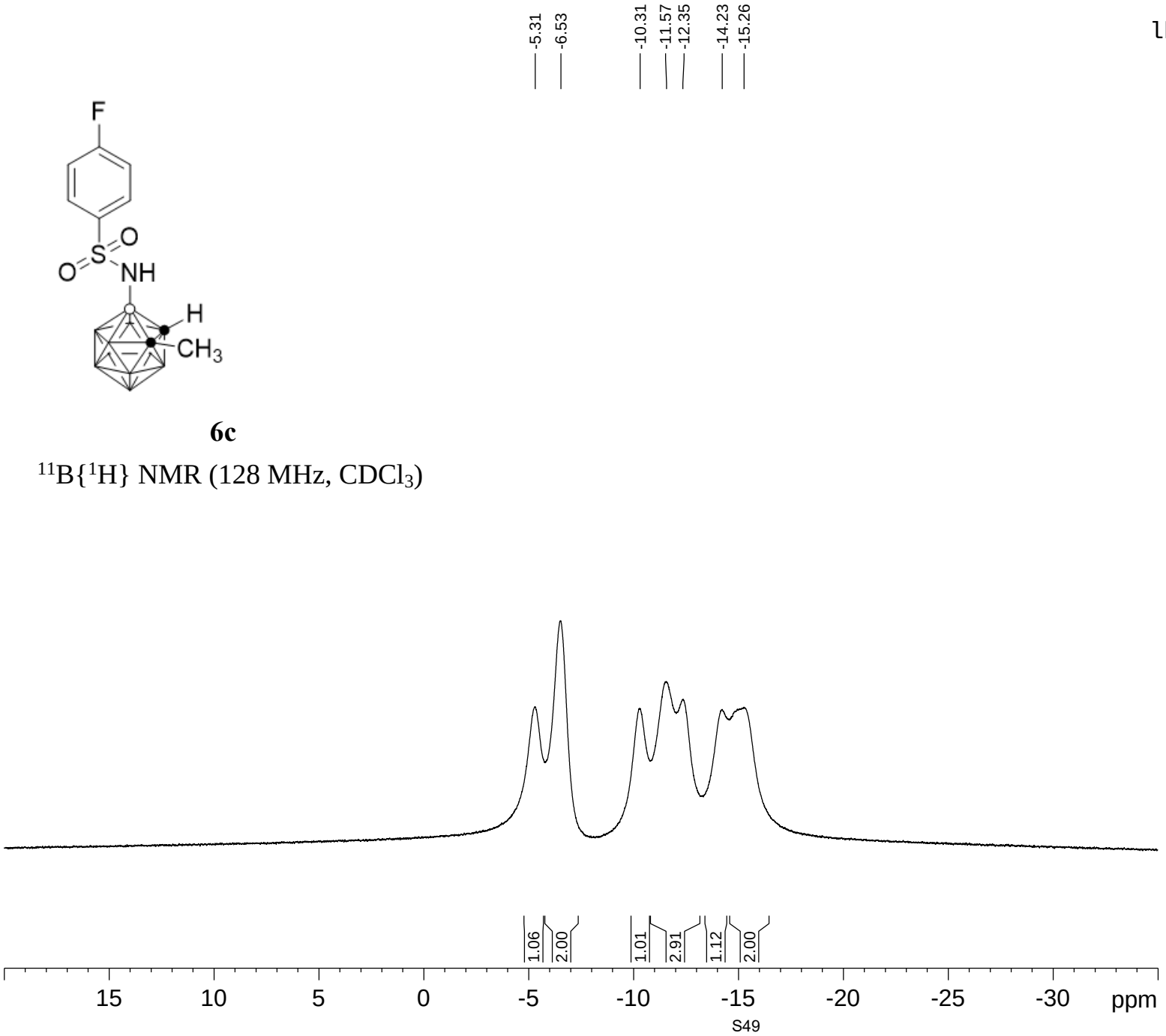


6c

$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)

6c

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)

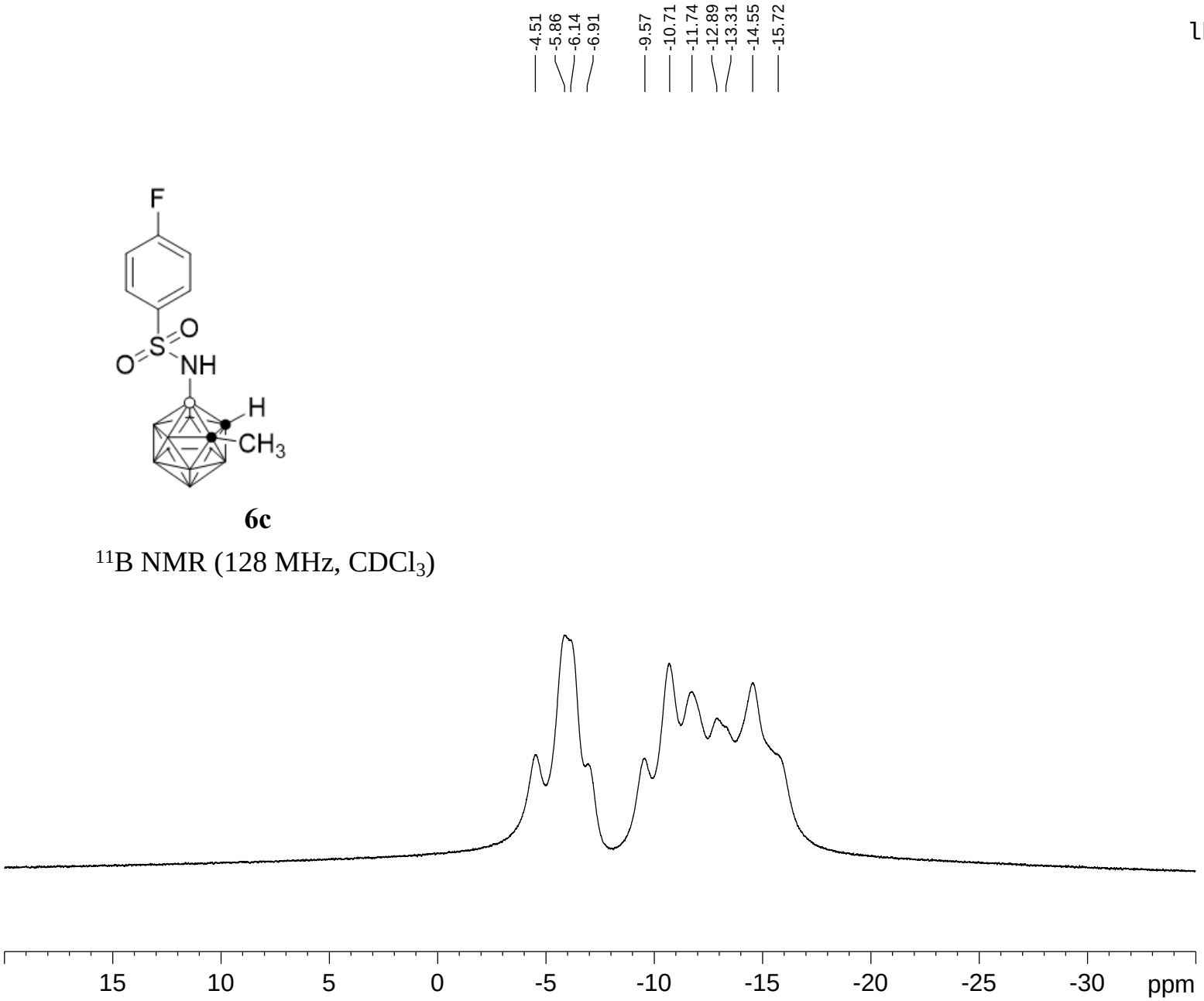


lhr-B-0558-2-cc-CDCl3

```
Current Data Parameters
NAME      lhr-B-0558-2-cc-CDCl3
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_            20161218
Time                18.47 h
INSTRUM             spect
PROBHD    Z108618_0257 (
PULPROG              zgdc
TD                  65536
SOLVENT             CDCl3
NS                     36
DS                      4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631488 sec
RG                    287
DW                  20.800 usec
DE                   6.50 usec
TE                  296.7 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
```

6c

^{11}B NMR (128 MHz, $CDCl_3$)



lhr-B-0558-2-cc-CDCl3(C)

```
Current Data Parameters
NAME      lhr-B-0558-2-cc-CDCl3(C)
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161218
Time              18.51 h
INSTRUM           spect
PROBHD   Z108618_0257 (
PULPROG              zg
TD                65536
SOLVENT           CDCl3
NS                   62
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                296.1 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
```

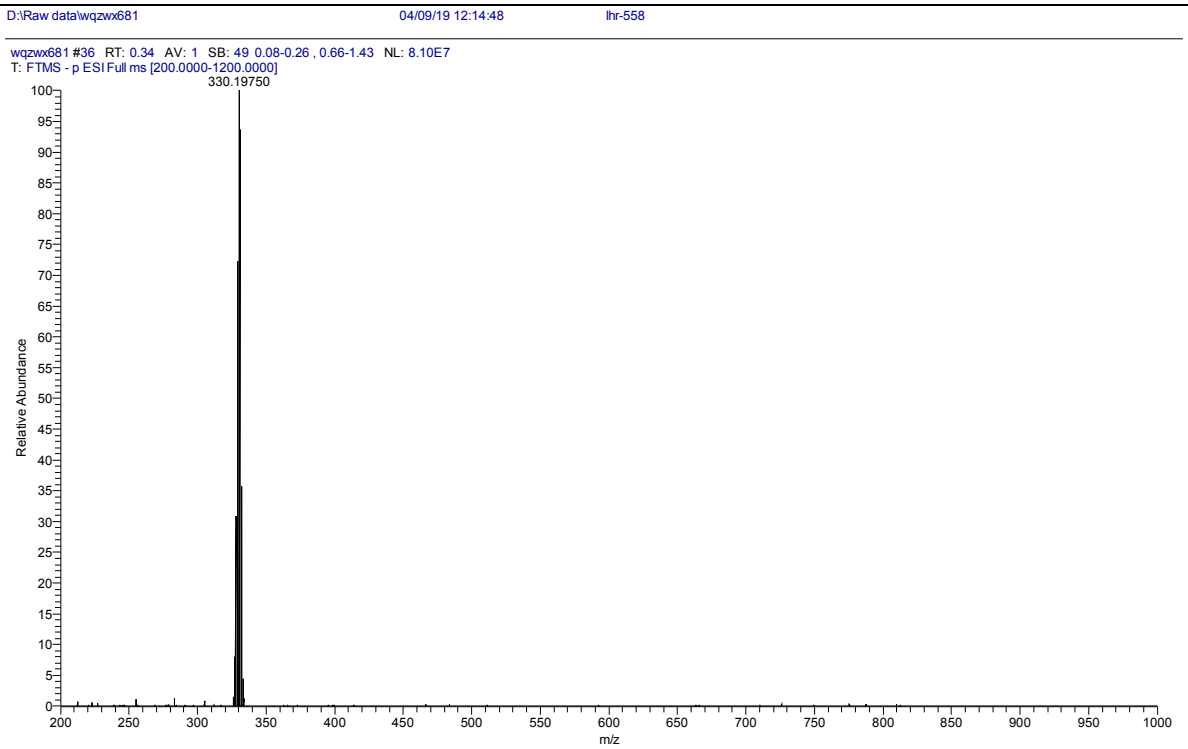
Thermo QEFMS Analysis Report

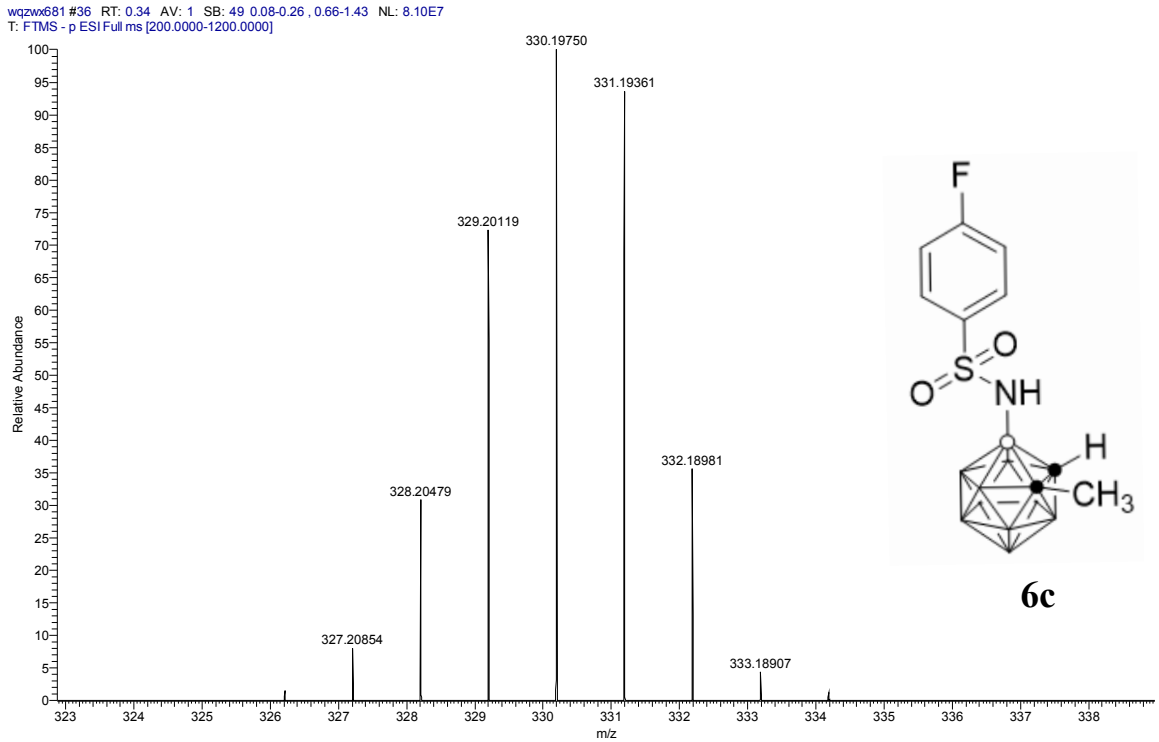
Analysis Info

Sample Name :	Lhr-558	Reference No.:	Wqzwx681
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Negative
Comment :	ESI, 3.0kV, by LC, with sheath gas		

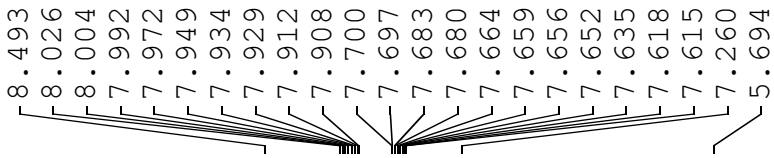
Accurate Mass Measurement

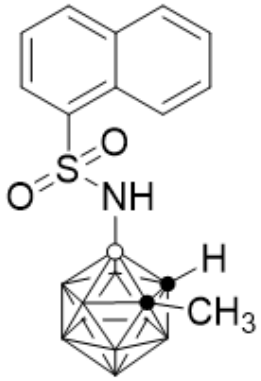
Molecular formula :	$C_9B_{10}H_{18}NO_2SF$
Experimental Mass $[M-H]^-$:	330.19750
Theoretical Mass $[M-H]^-$:	330.19779
Error (ppm) :	-0.9





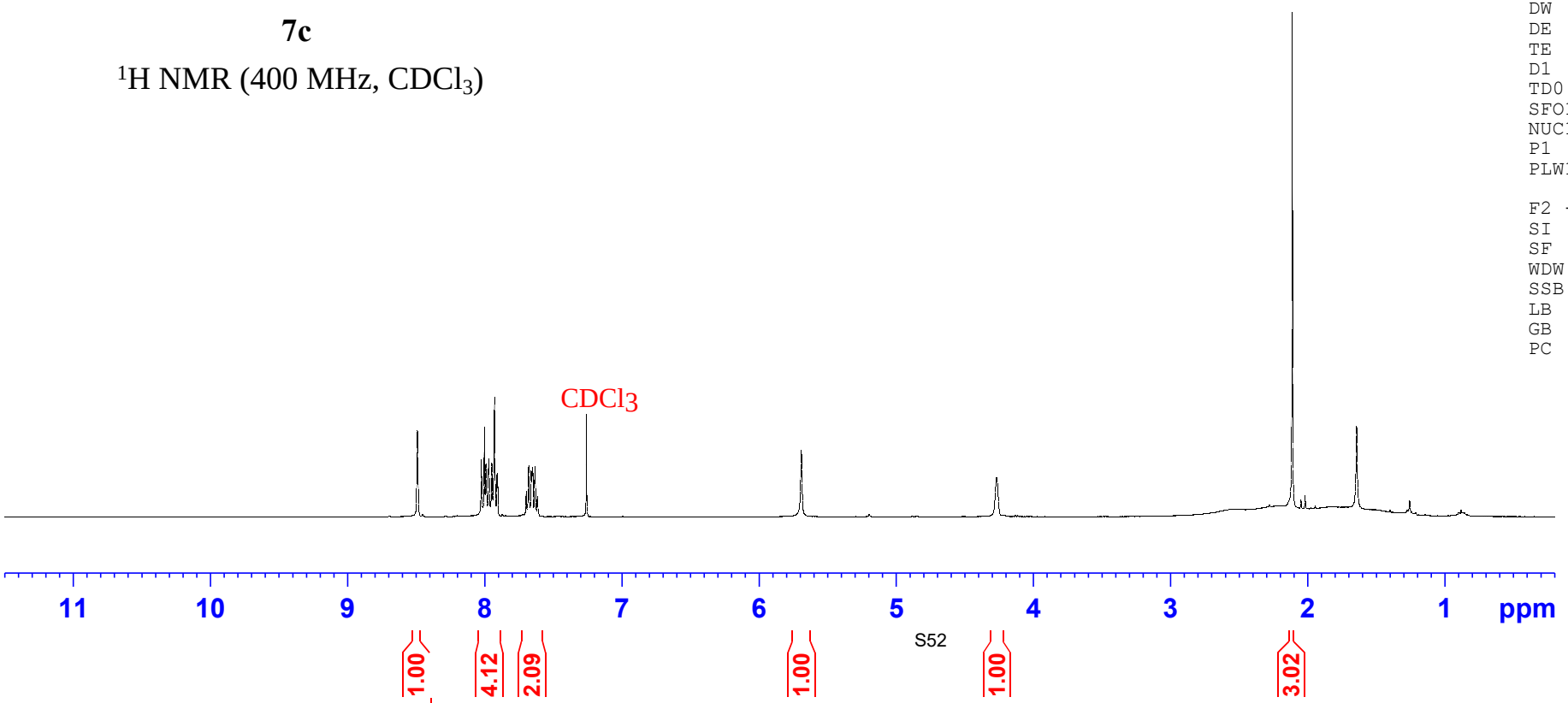
6c





7c

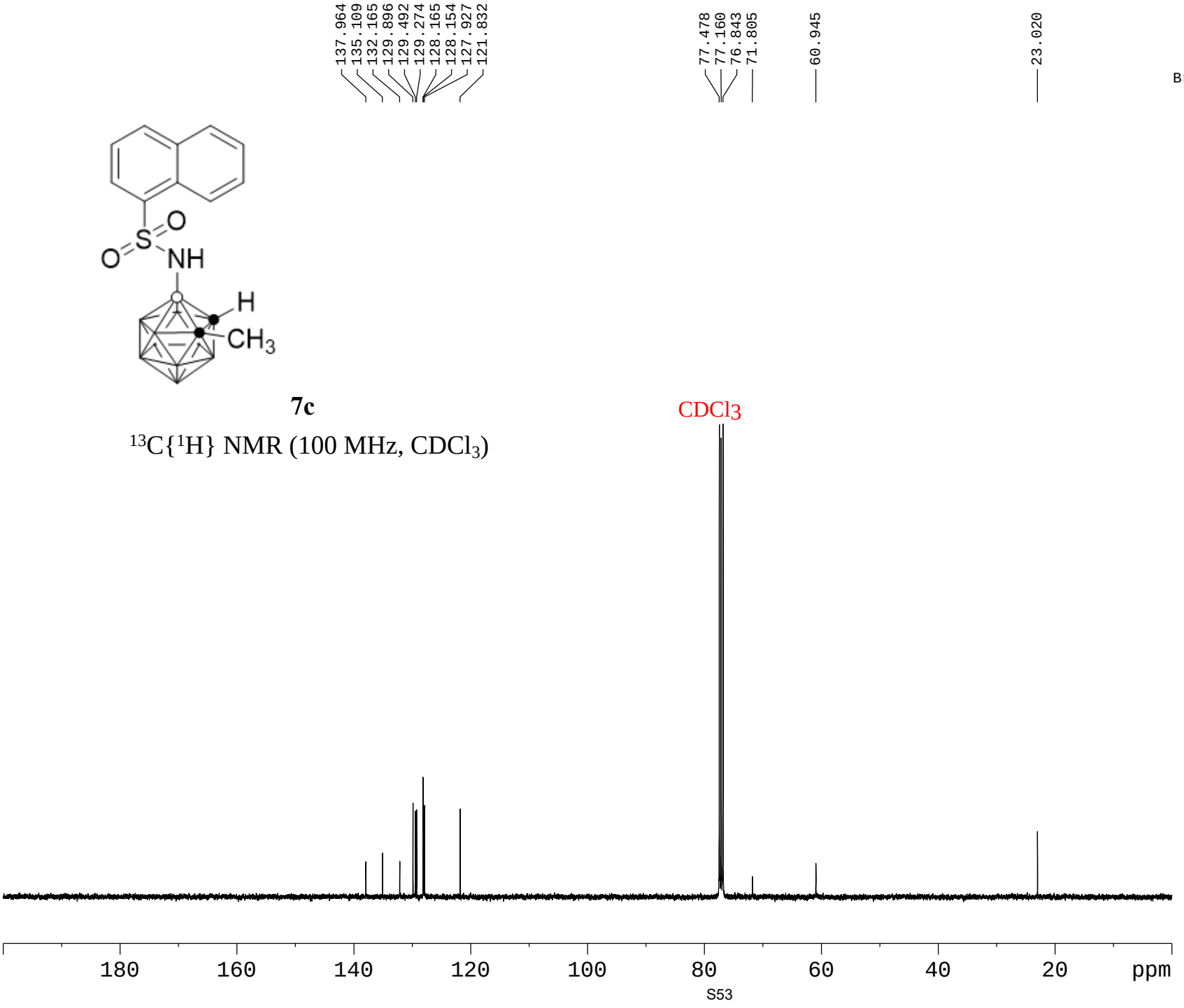
^{1}H NMR (400 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      lhr-H-0563-cc-CDCl3
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20161222
Time                 11.46 h
INSTRUM                spect
PROBHD   Z824601_0021 (
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       12
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                   295.8 K
D1              1.00000000 sec
TD0                       1
SFO1           400.1324708 MHz
NUC1                     1H
P1                   15.00 usec
PLW1            8.31000042 W

F2 - Processing parameters
SI                    65536
SF             400.1300097 MHz
WDW                      EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```

7c

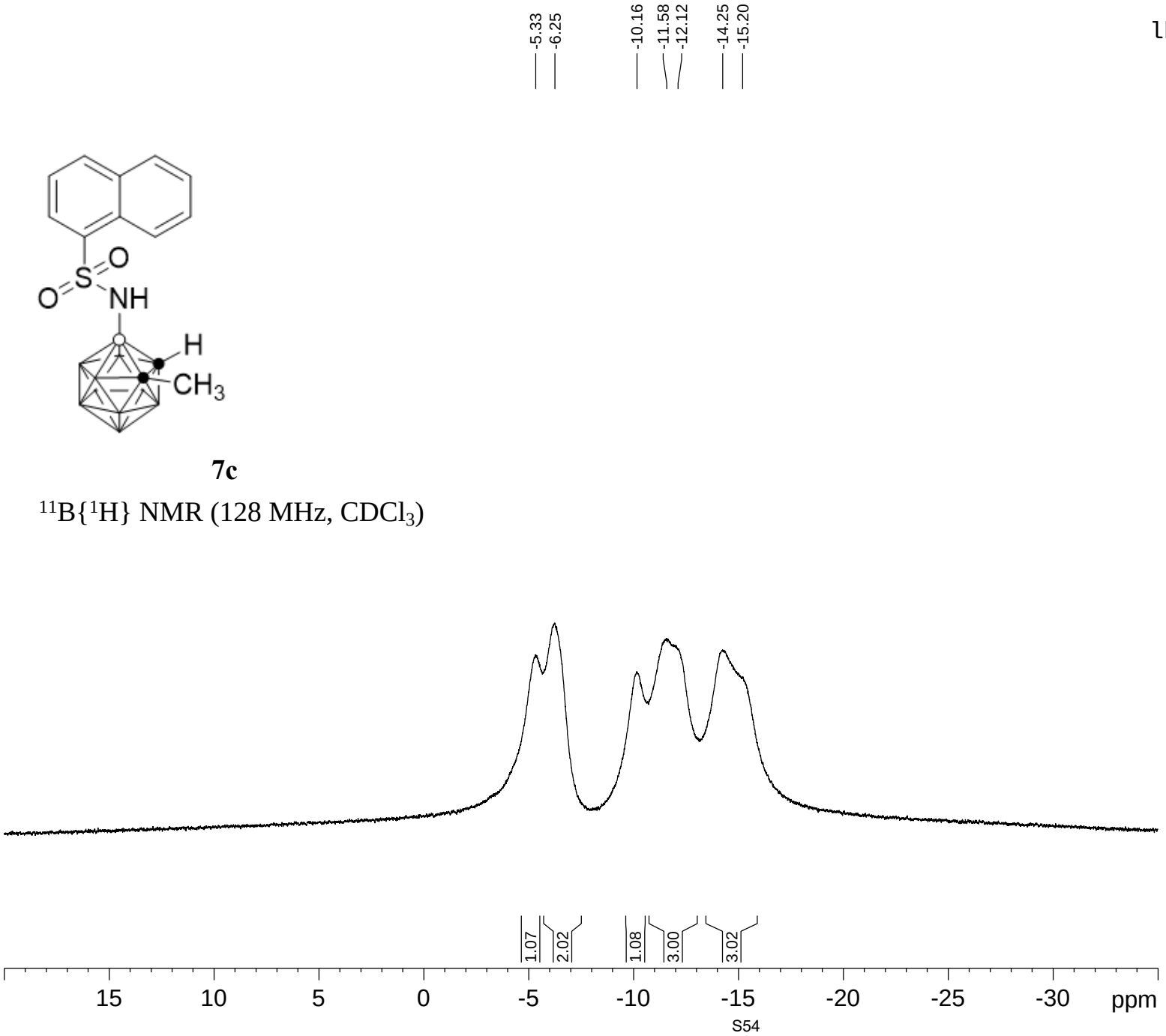
$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)




```
lhr-C-0563-cc-CDCl3

Bruker Advance III 400

Current Data Parameters
NAME      lhr-C-0563-cc-CDCl3
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_           20161222
Time              11.48 h
INSTRUM             spect
PROBHD   Z824601_0021 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   970
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631488 sec
RG                   203
DW                20.800 usec
DE                 6.50 usec
TE                295.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1
SFO1        100.6228298 MHz
NUC1                13C
P1                 9.50 usec
PLW1        41.25000000 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         8.31000042 W
PLW12        0.23083000 W
PLW13        0.11611000 W

F2 - Processing parameters
SI                 32768
SF          100.6127556 MHz
WDW                   EM
SSB      0
LB                 1.00 Hz
GB       0
PC                 1.40
```

7c

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)

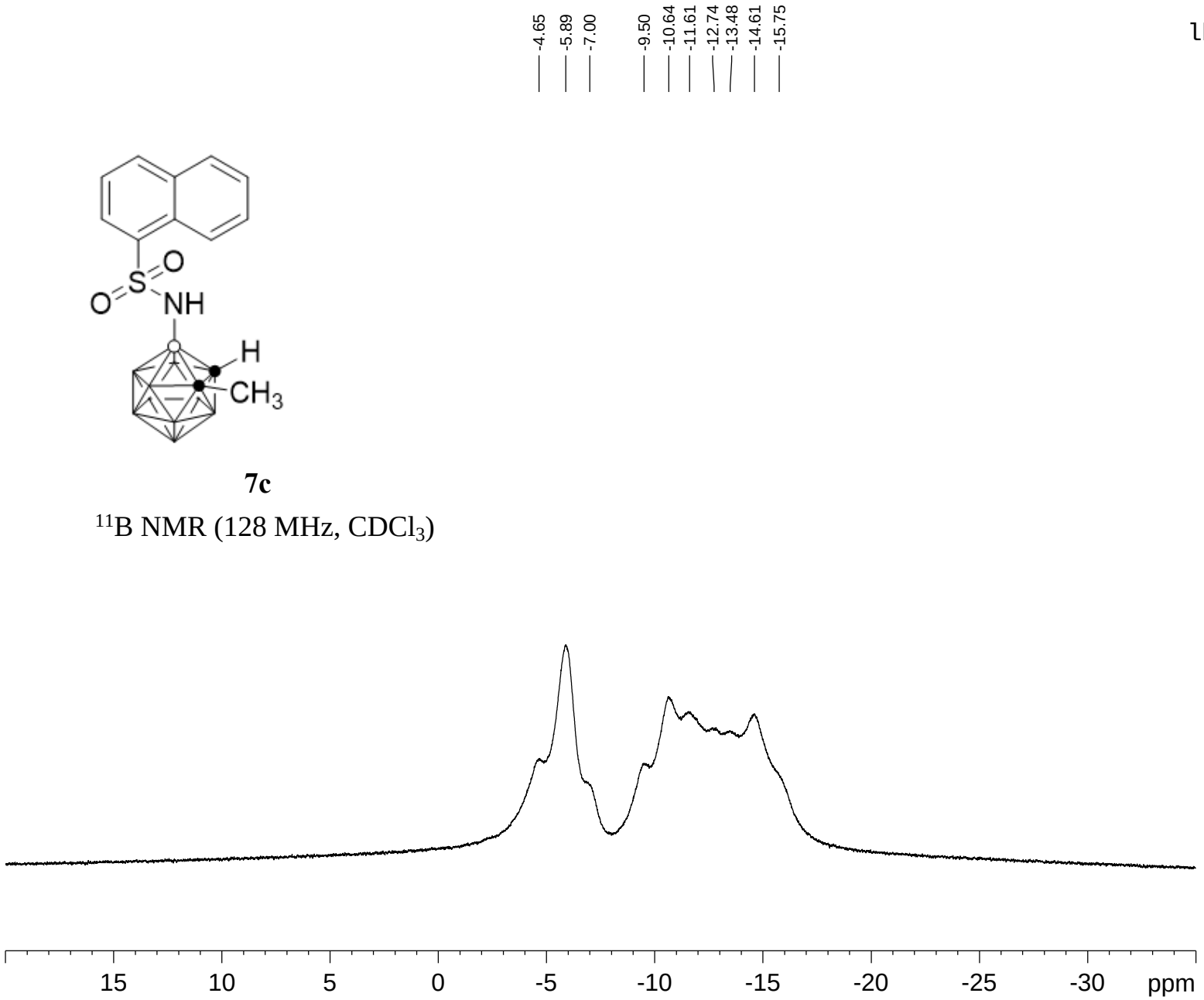


lhr-B-0563-cc-CDCl3

```
Current Data Parameters
NAME      lhr-B-0563-cc-CDCl3
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20161222
Time                  16.33 h
INSTRUM               spect
PROBHD   Z108618_0257 (
PULPROG                zgdc
TD                    65536
SOLVENT               CDCl3
NS                       27
DS                        4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631488 sec
RG                      287
DW                  20.800 usec
DE                   6.50 usec
TE                  295.4 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
```

7c

^{11}B NMR (128 MHz, $CDCl_3$)



lhr-B-0563-cc-CDCl3(C)

```
Current Data Parameters
NAME      lhr-B-0563-cc-CDCl3(C)
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_              20161222
Time                  16.35 h
INSTRUM               spect
PROBHD   Z108618_0257 (
PULPROG                    zg
TD                      65536
SOLVENT                 CDCl3
NS                         34
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                     295.0 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               128.4097504 MHz
WDW                        EM
SSB       0
LB                      1.00 Hz
GB        0
PC                      1.40
```

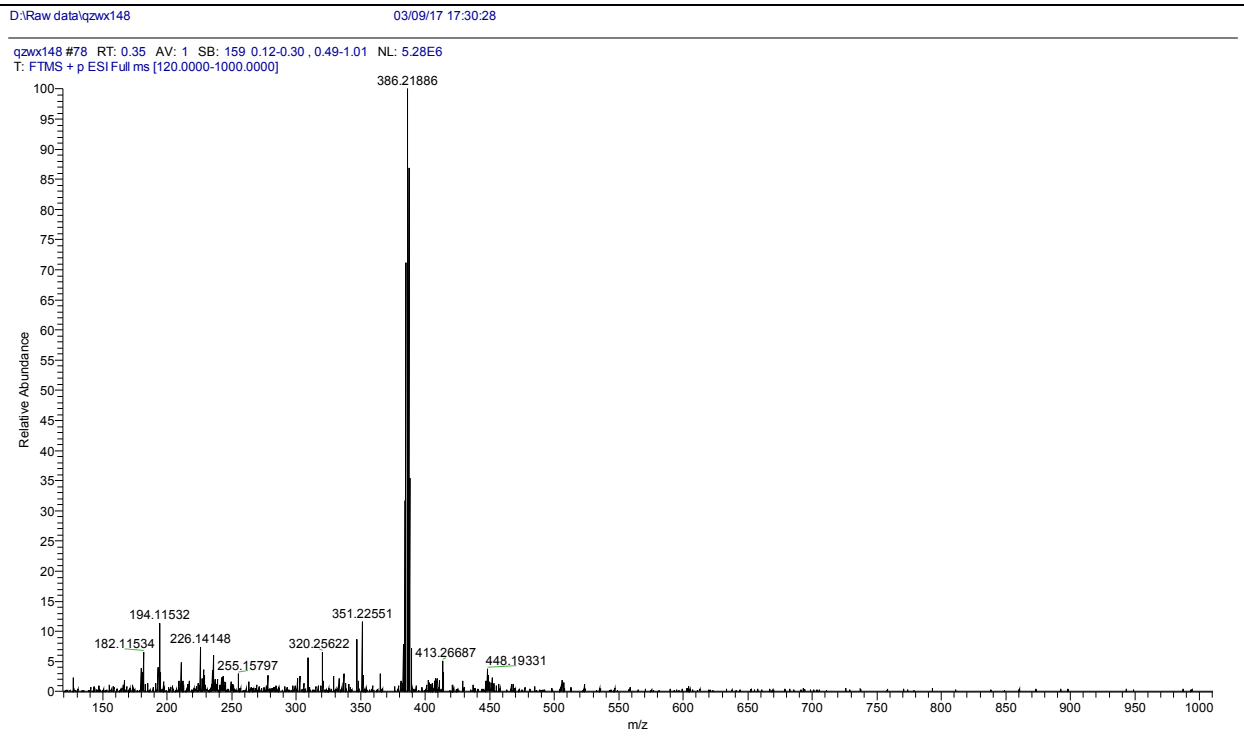
Thermo QEFMS Analysis Report

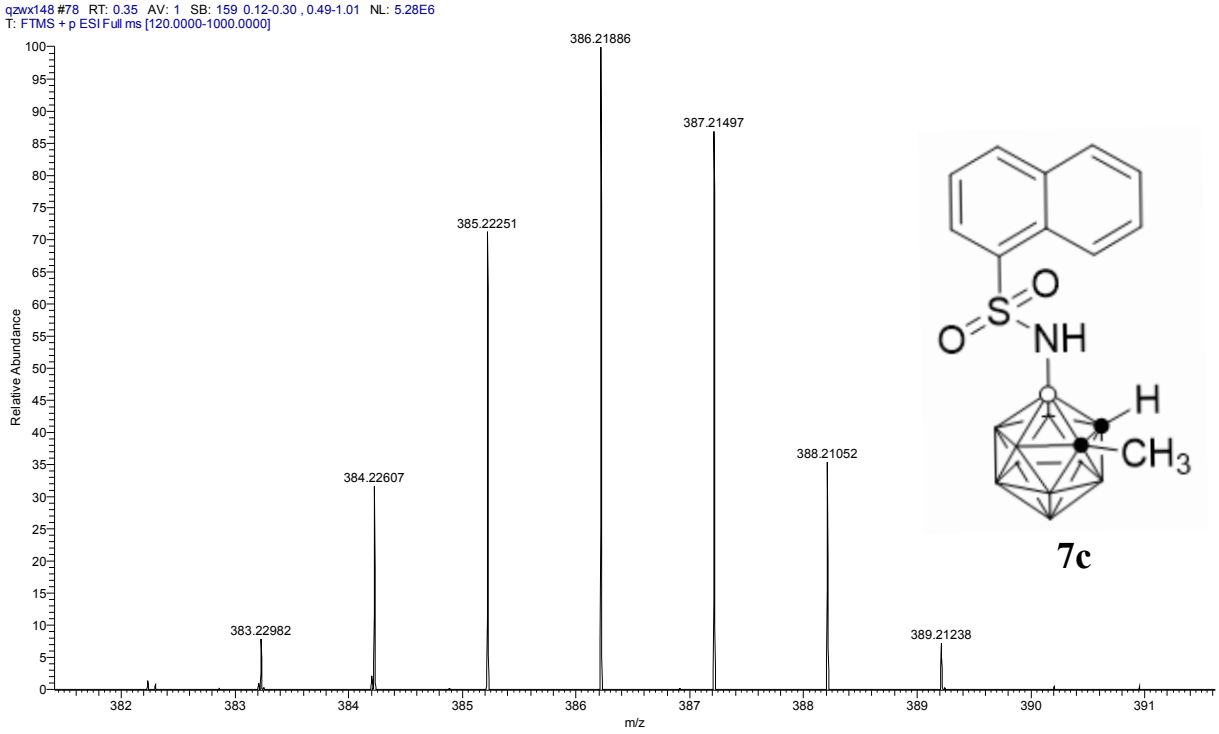
Analysis Info

Sample Name :	Lhr-0577	Reference No.:	Qzwx148
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.5kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{13}H_{21}B_{10}NO_2S$
Experimental Mass $[M+Na]^+$:	386.21886
Theoretical Mass $[M+Na]^+$:	386.21955
Error (ppm) :	1.7

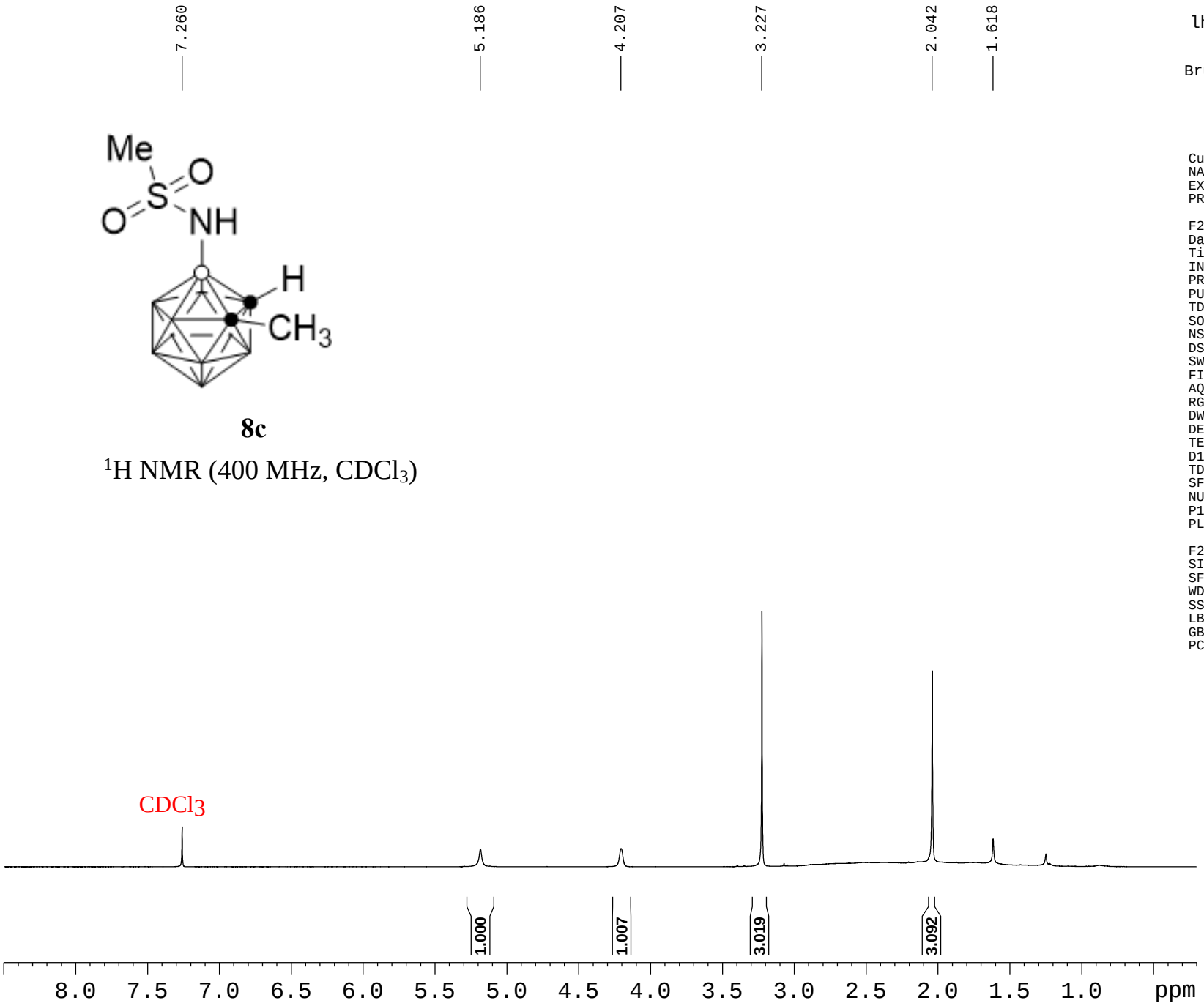




7c

8c

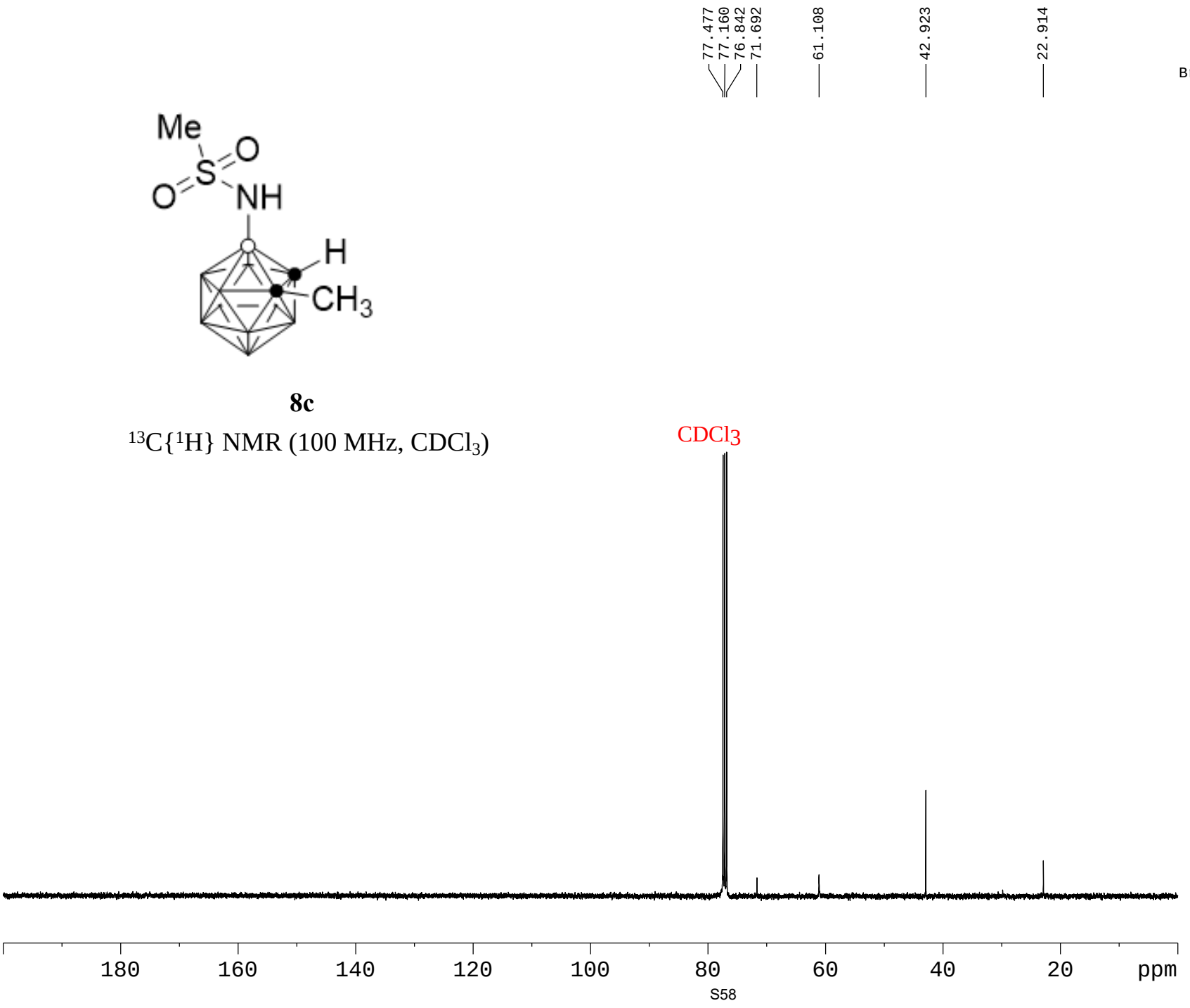
^{1}H NMR (400 MHz, $CDCl_3$)




```
lhr-H-0579-cc-CDCl3

Bruker Advance III 400

Current Data Parameters
NAME      lhr-H-0579-cc-CDCl3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170104
Time              12.33 h
INSTRUM            spect
PROBHD   Z824601_0021 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    12
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                295.1 K
D1           1.00000000 sec
TD0                    1
SFO1        400.1324708 MHz
NUC1                  1H
P1                15.00 usec
PLW1          8.31000042 W

F2 - Processing parameters
SI                 65536
SF          400.1300096 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

8c

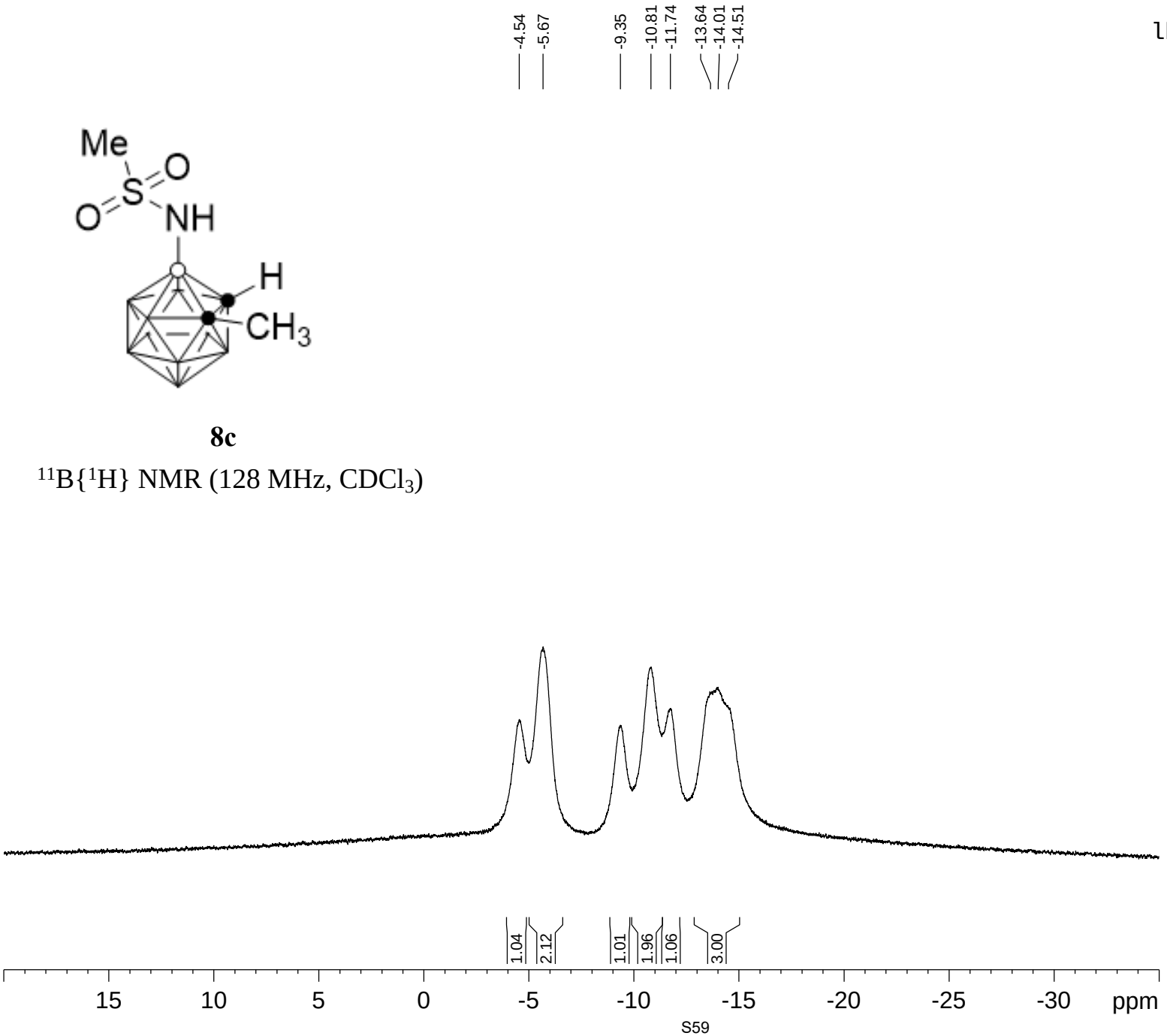
$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)




```
lhr-C-0579-cc-CDCl3

Bruker Advance III 400

Current Data Parameters
NAME      lhr-C-0579-cc-CDCl3
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_           20170105
Time              12.35 h
INSTRUM             spect
PROBHD    Z824601_0021 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1000
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                   203
DW                20.800 usec
DE                 6.50 usec
TE                296.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1
SFO1         100.6228298 MHz
NUC1                13C
P1                 9.50 usec
PLW1         41.25000000 W
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2          8.31000042 W
PLW12         0.23083000 W
PLW13         0.11611000 W

F2 - Processing parameters
SI                 32768
SF           100.6127558 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

8c

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)

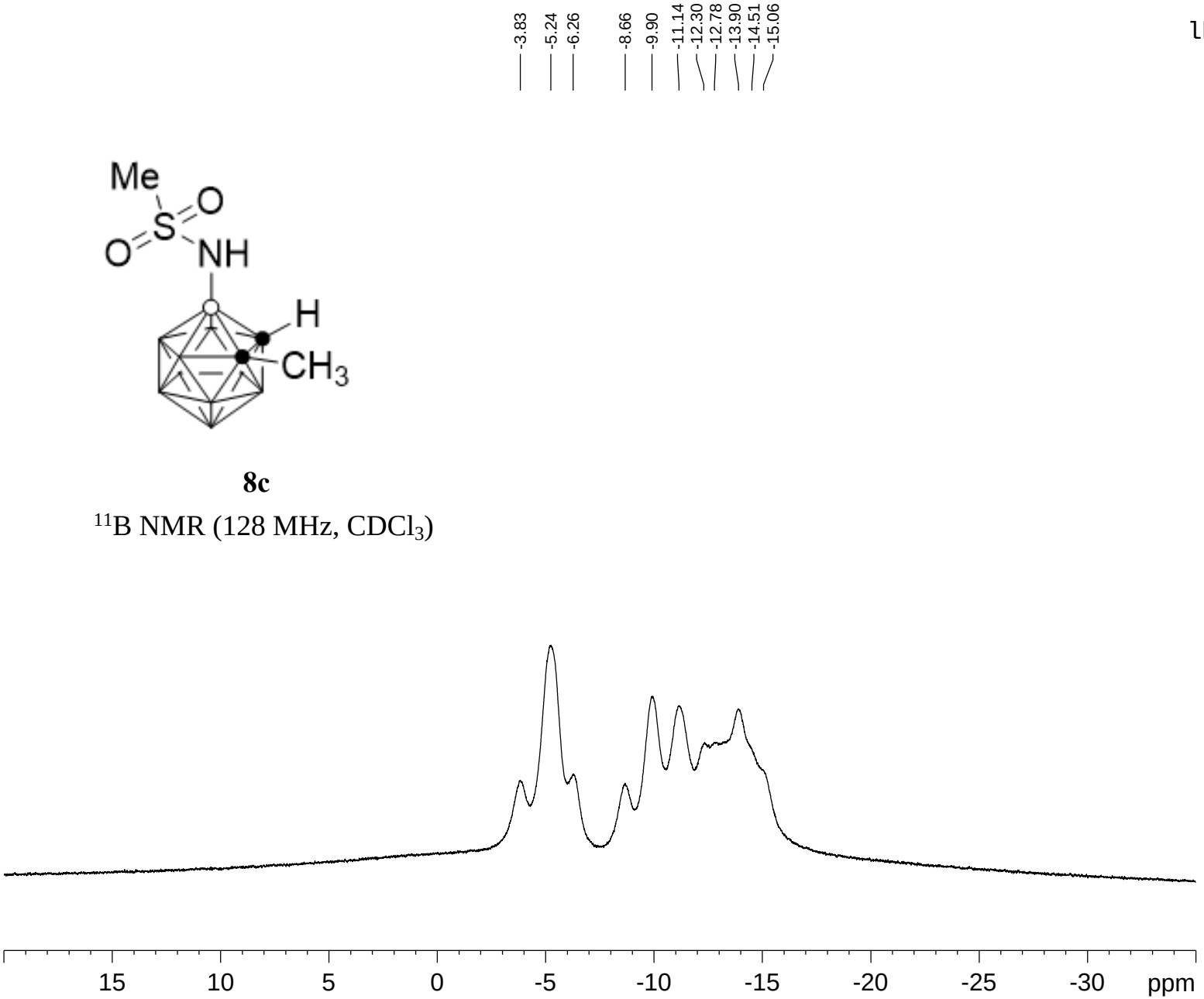


lhr-B-0579-cc-CDCl3

```
Current Data Parameters
NAME      lhr-B-0579-cc-CDCl3
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20170103
Time                  22.01 h
INSTRUM               spect
PROBHD   Z108618_0257 (
PULPROG                zgdc
TD                    65536
SOLVENT               CDCl3
NS                       12
DS                        4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631488 sec
RG                      203
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
```

8c

^{11}B NMR (128 MHz, $CDCl_3$)



lhr-B-0579-cc-CDCl3(C)

```
Current Data Parameters
NAME      lhr-B-0579-cc-CDCl3(C)
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_               20170103
Time                   22.04 h
INSTRUM                spect
PROBHD   Z108618_0257 (
PULPROG                   zg
TD                     65536
SOLVENT                CDCl3
NS                        34
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                    294.6 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                128.4097504 MHz
WDW                       EM
SSB       0
LB                     1.00 Hz
GB        0
PC                     1.40
```

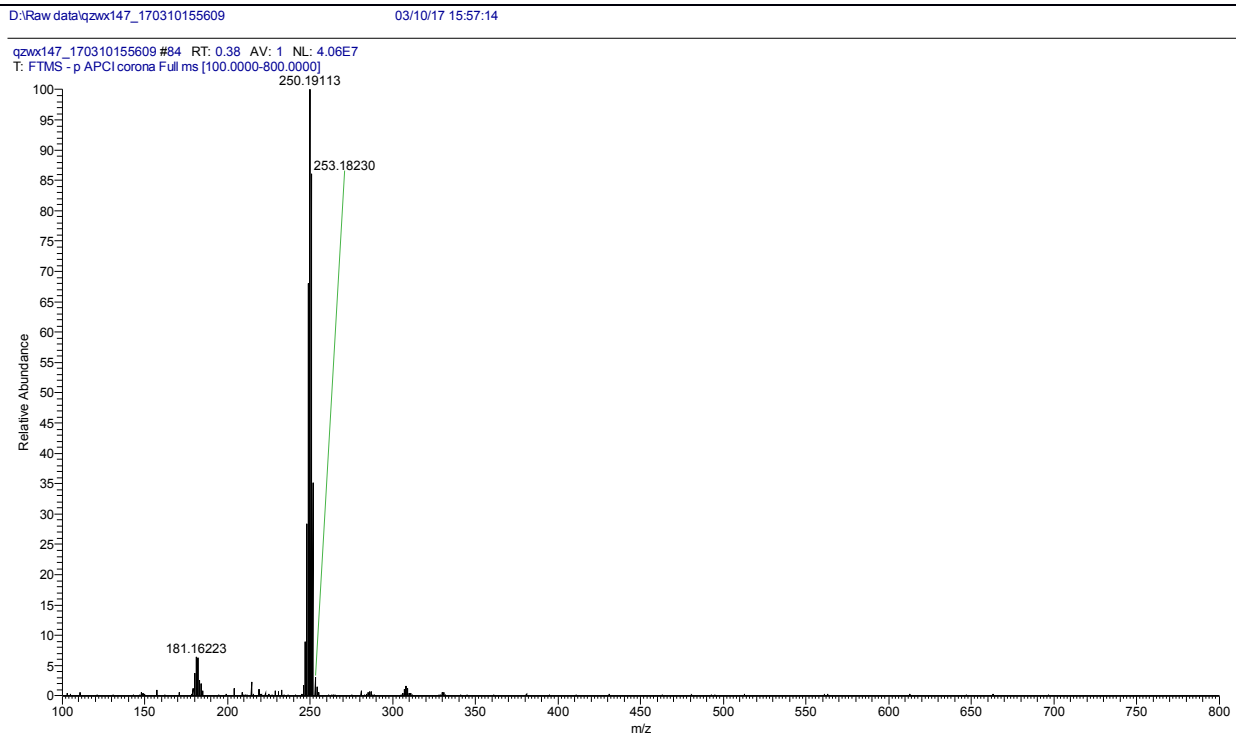
Thermo QEFMS Analysis Report

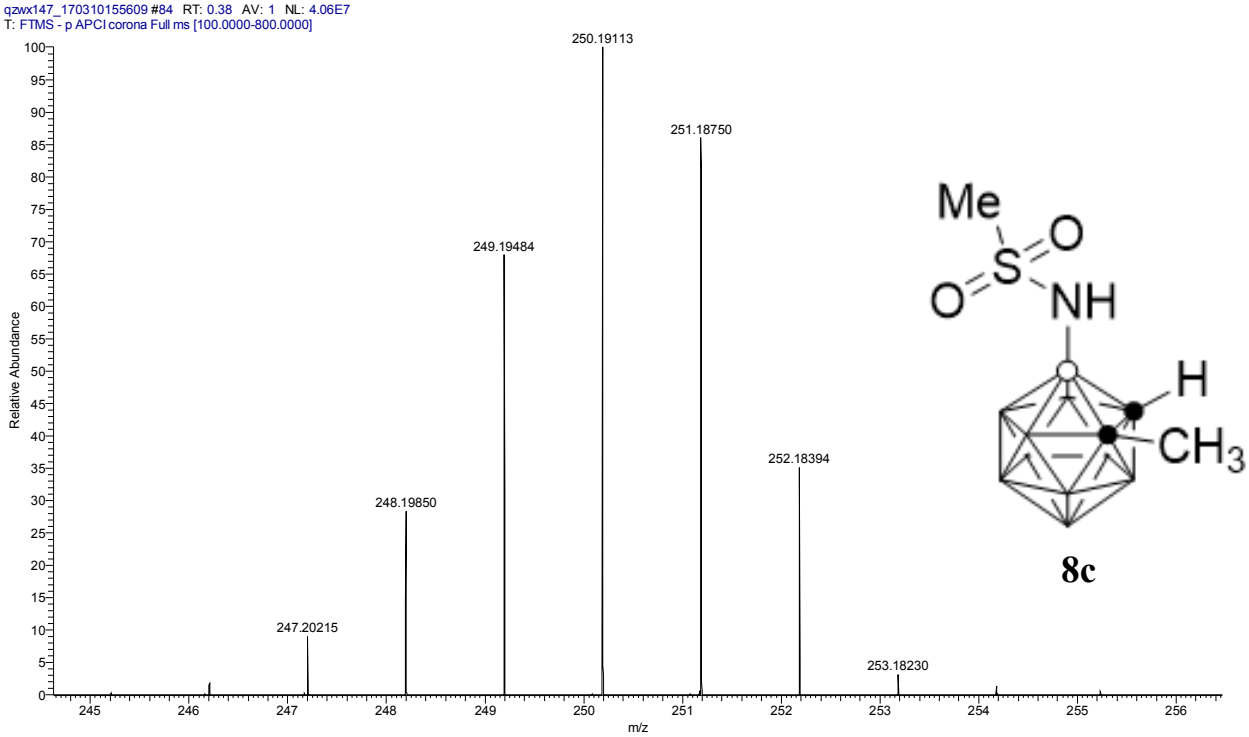
Analysis Info

Sample Name :	Lhr-0579	Reference No.:	Qzwx147
Instrument :	Q Exactive Focus Orbitrap		
Source :	APCI	Polarity :	Negative
Comment :	APCI neg, 5.0uA, by LC, with sheath gas		

Accurate Mass Measurement

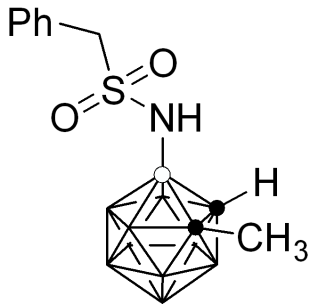
Molecular formula :	$C_4H_{17}B_{10}NO_2S$
Experimental Mass $[M-H]^-$:	250.19113
Theoretical Mass $[M-H]^-$:	250.19132
Error (ppm) :	0.7





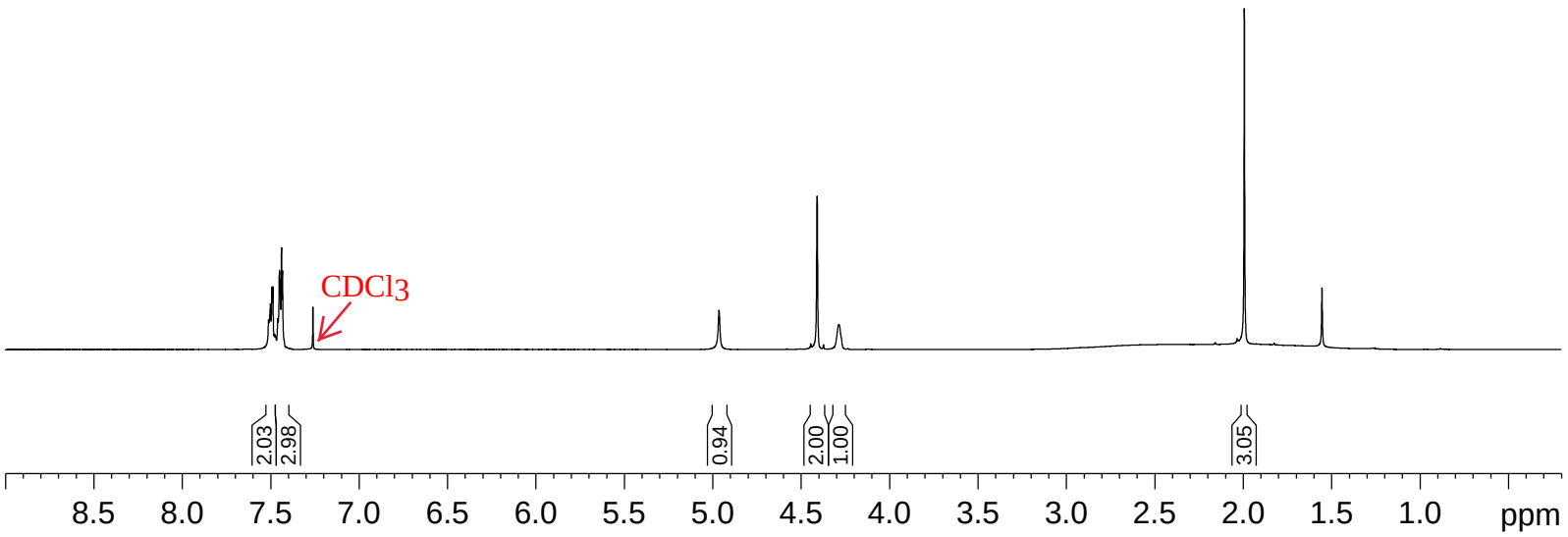
8c

lhr-H-0870-cdcl3



9c

^{1}H NMR (400 MHz, $CDCl_3$)



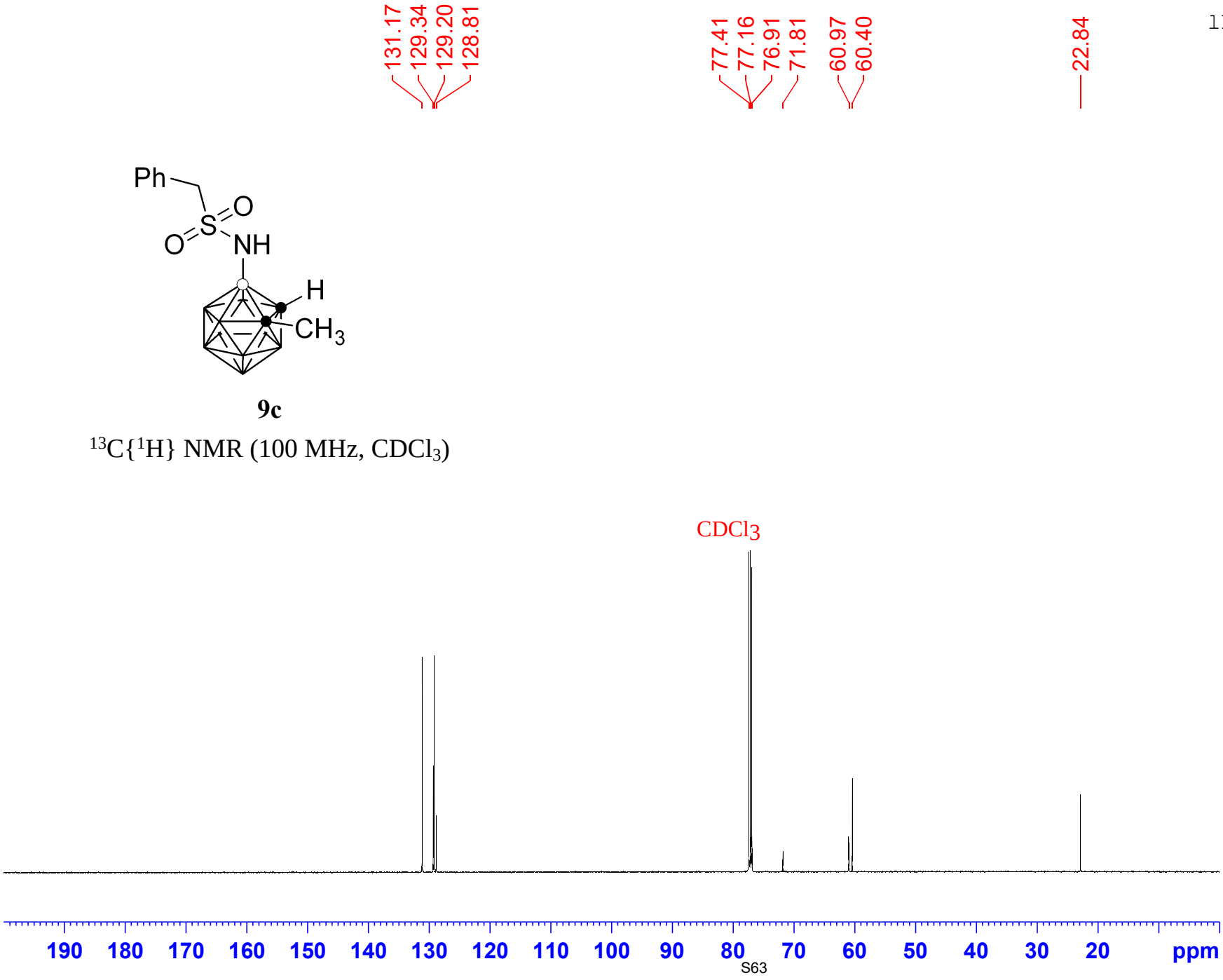
Current Data Parameters
NAME lhr-H-0870-cdcl3
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180423
Time 17.23 h
INSTRUM spect
PROBHD Z108618_0257 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 64
DW 62.400 usec
DE 6.50 usec
TE 294.3 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 400.2300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

9c

$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)

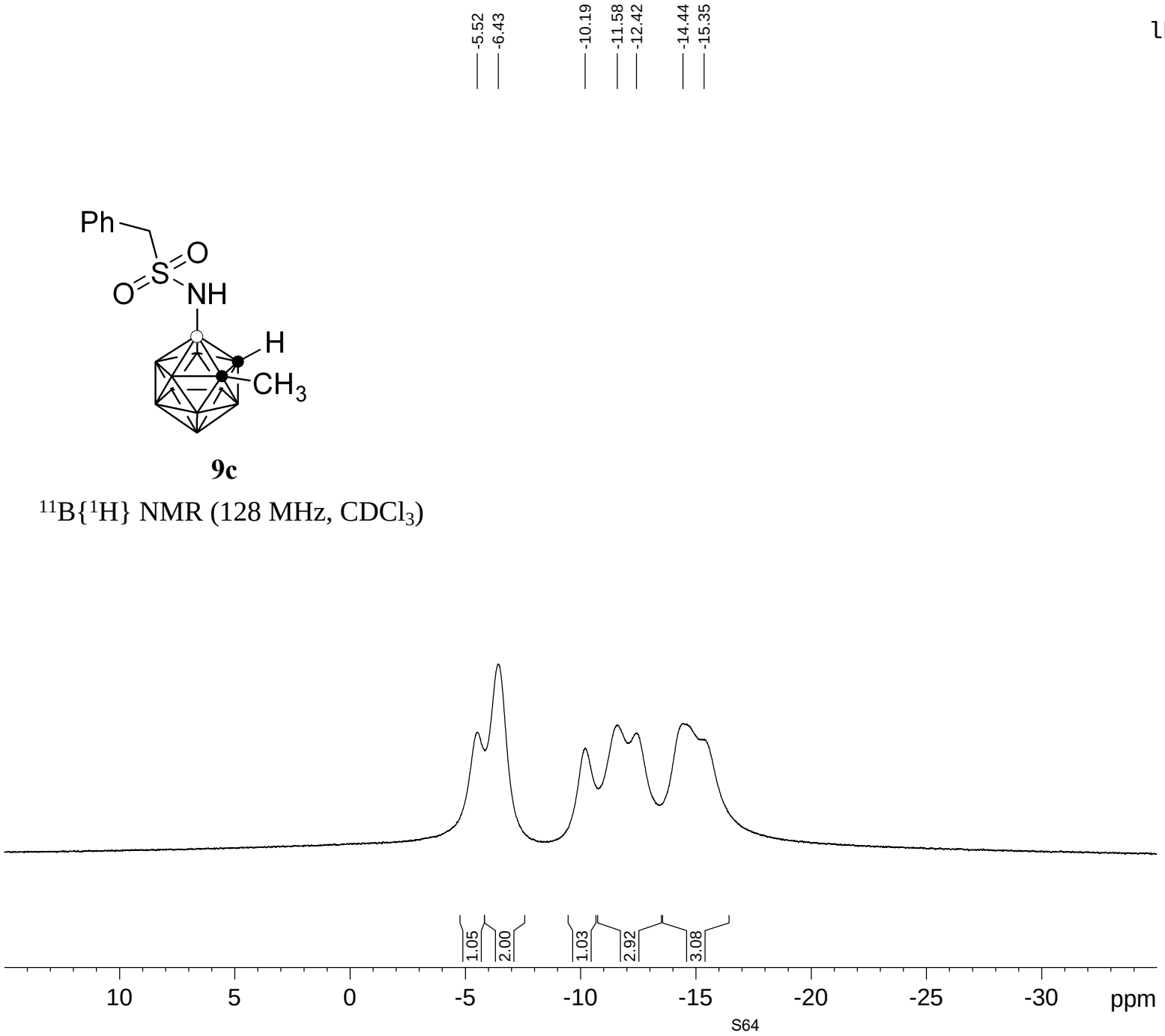


lhr-C-0870-cdcl3

```
Current Data Parameters
NAME      lhr-C-0870-cdcl3
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20180423
Time              18.58 h
INSTRUM            spect
PROBHD   Z149001_0010 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  500
DS                    4
SWH           29761.904 Hz
FIDRES         0.908261 Hz
AQ            1.1010048 sec
RG               206.72
DW               16.800 usec
DE               18.00 usec
TE               298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        125.7703643 MHz
NUC1               13C
P1               10.00 usec
PLW1        61.00000000 W
SFO2        500.1320005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            80.00 usec
PLW2        15.00000000 W
PLW12        0.29663000 W
PLW13        0.14920001 W

F2 - Processing parameters
SI                32768
SF          125.7577738 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

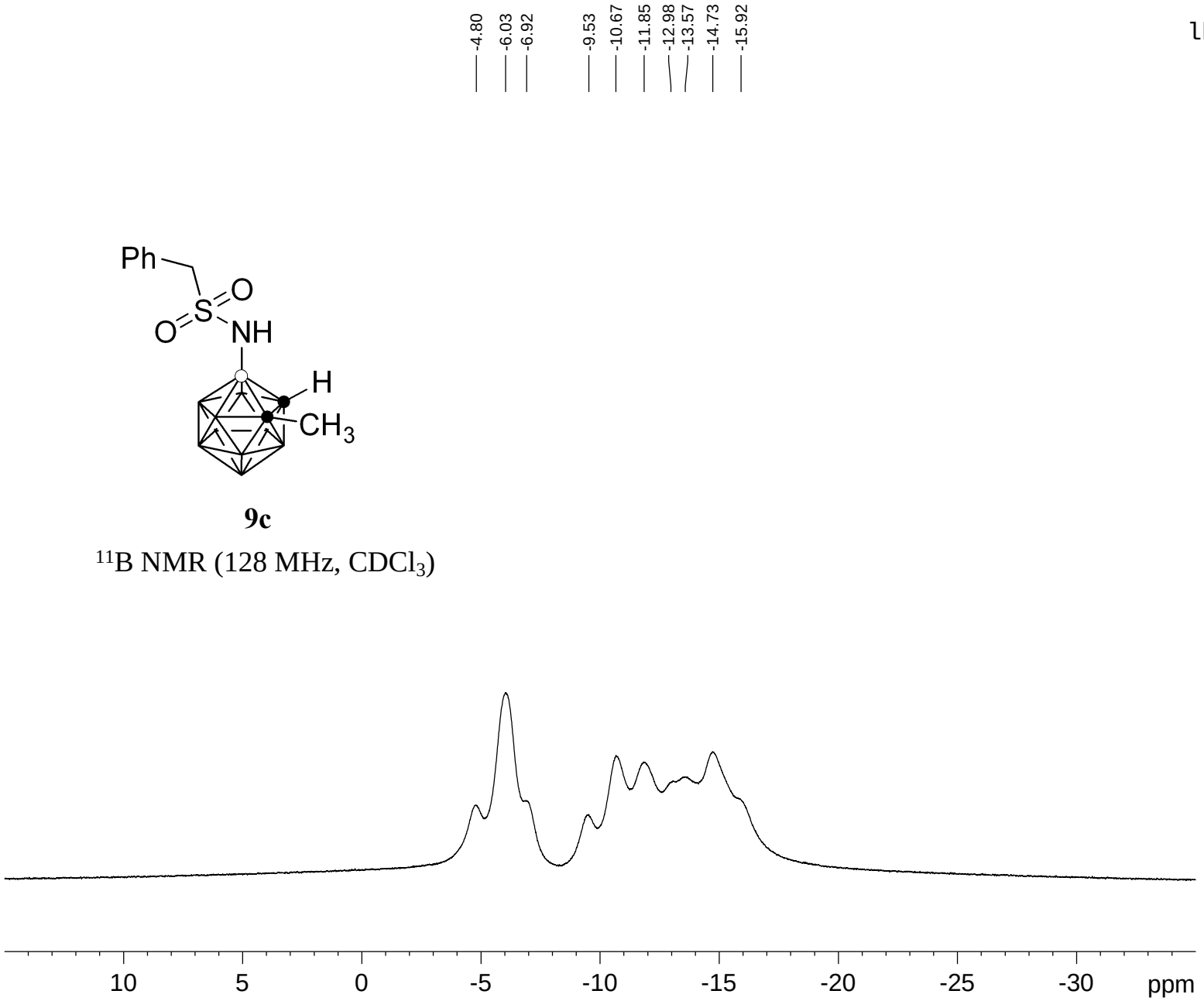
9c

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)



lhr-B-0870-cdcl3

```
Current Data Parameters
NAME      lhr-B-0870-cdcl3
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20180423
Time                17.27 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG              zgdc
TD                  65536
SOLVENT             CDCl3
NS                     26
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                 294.8 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
```



9c

^{11}B NMR (128 MHz, $CDCl_3$)

lhr-B-0870-cdcl3(C)

```
Current Data Parameters
NAME      lhr-B-0870-cdcl3(C)
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20180423
Time                  17.30 h
INSTRUM               spect
PROBHD   Z108618_0257 (
PULPROG                   zg
TD                     65536
SOLVENT               CDCl3
NS                        31
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                  294.4 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
```

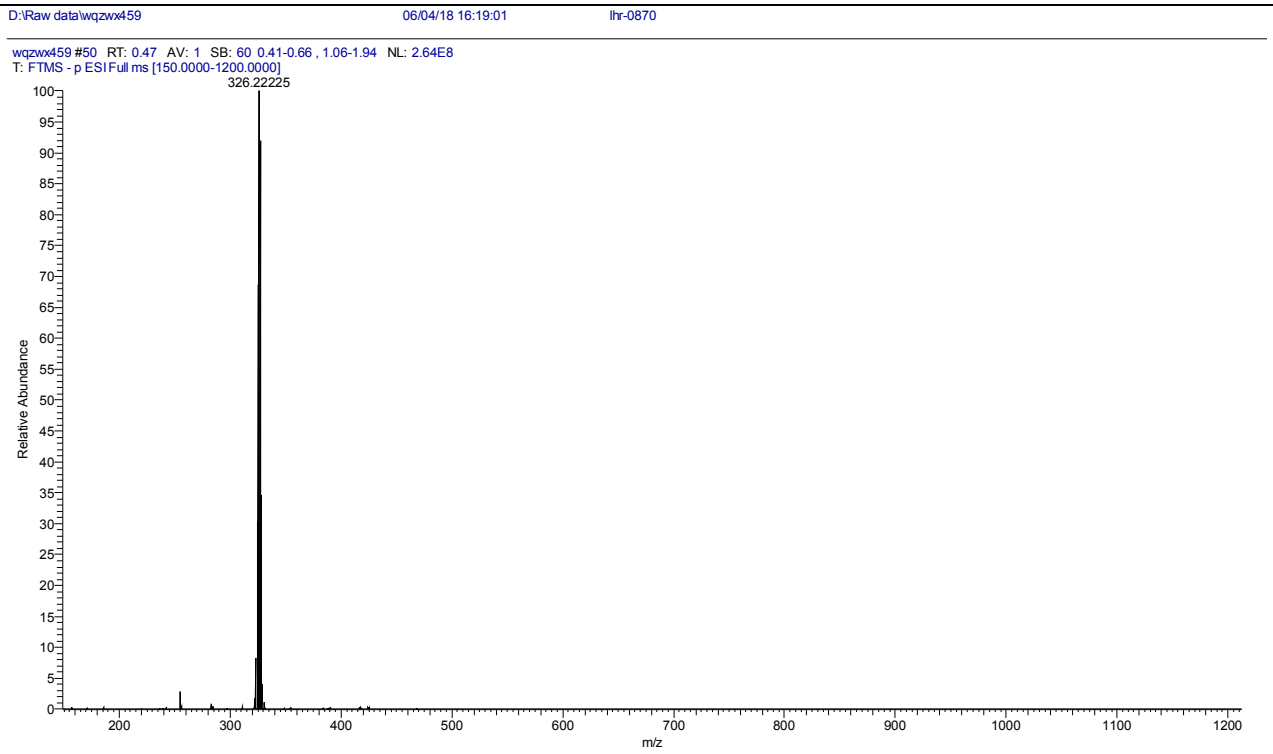
Thermo QEFMS Analysis Report

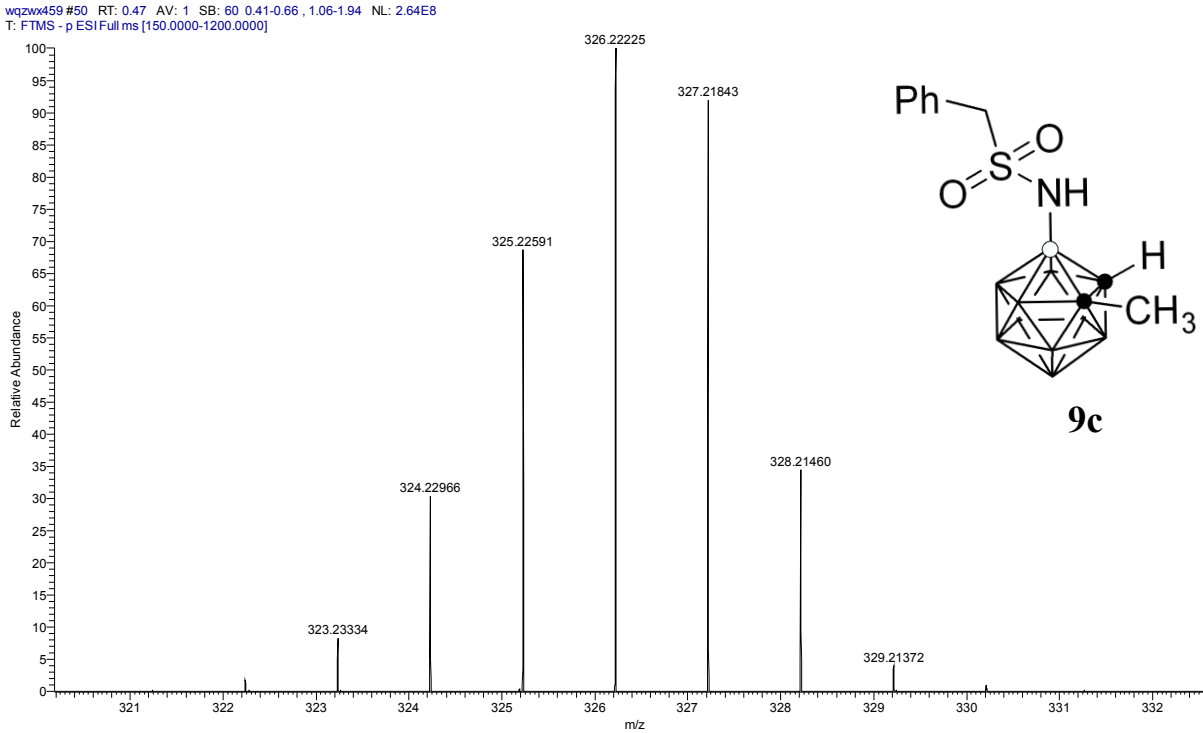
Analysis Info

Sample Name :	Lhr-0870	Reference No.:	Wqzwx459
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Negative
Comment :	ESI neg, 3.0kV, by LC, with sheath gas		

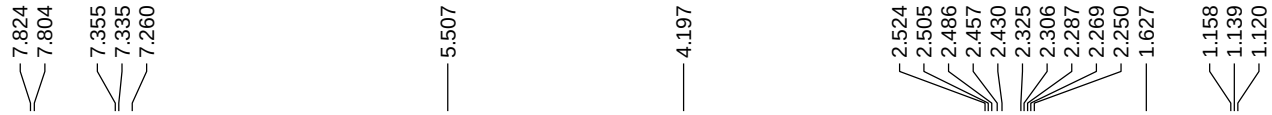
Accurate Mass Measurement

Molecular formula :	$C_{10}H_{21}B_{10}NO_2S$
Experimental Mass $[M-H]^-$:	326.22225
Theoretical Mass $[M-H]^-$:	326.22291
Error (ppm) :	2.0

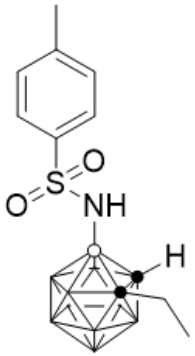




9c

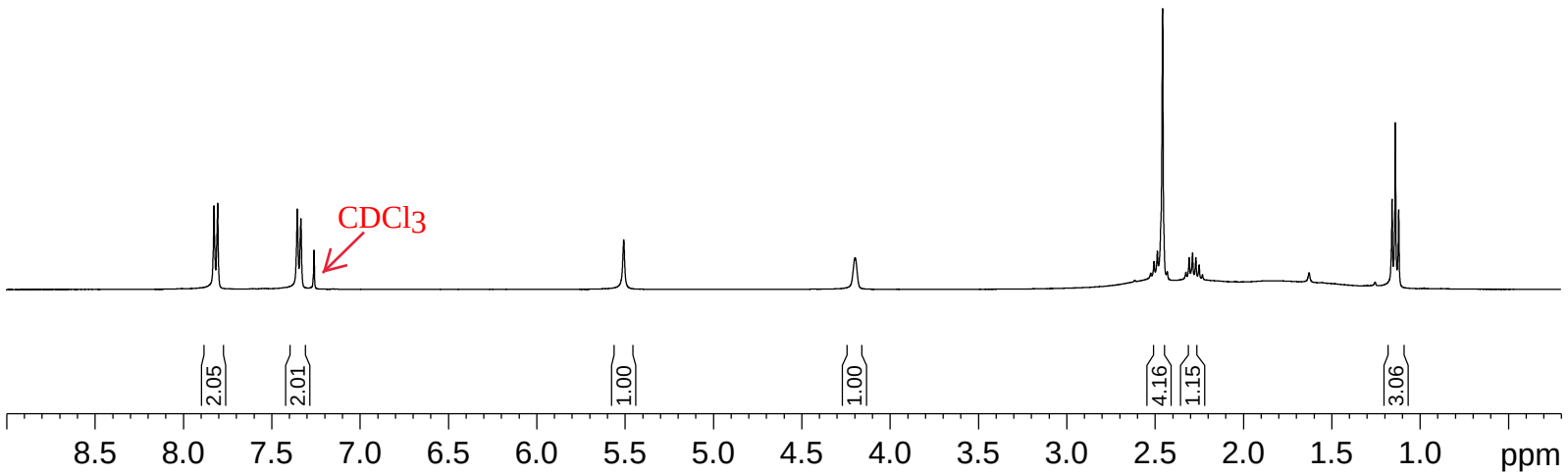


lhr-H-0582-cc-CDCl3



10c

^{1}H NMR (400 MHz, $CDCl_3$)



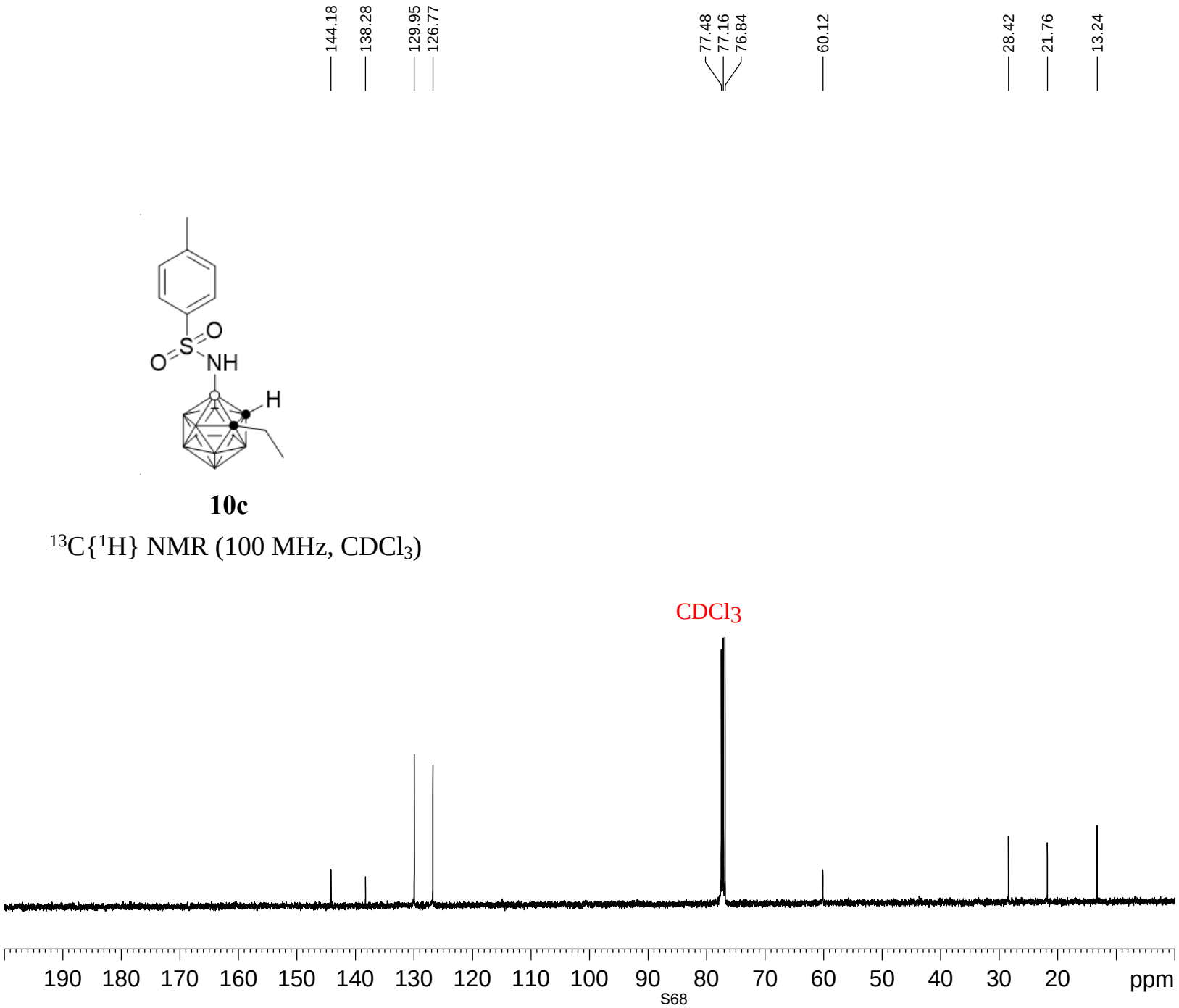
Current Data Parameters
NAME lhr-H-0582-cc-CDC
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170101
Time 20.07 h
INSTRUM spect
PROBHD Z108618_0257 (
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 295.1 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 400.2300105 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

10c

$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)

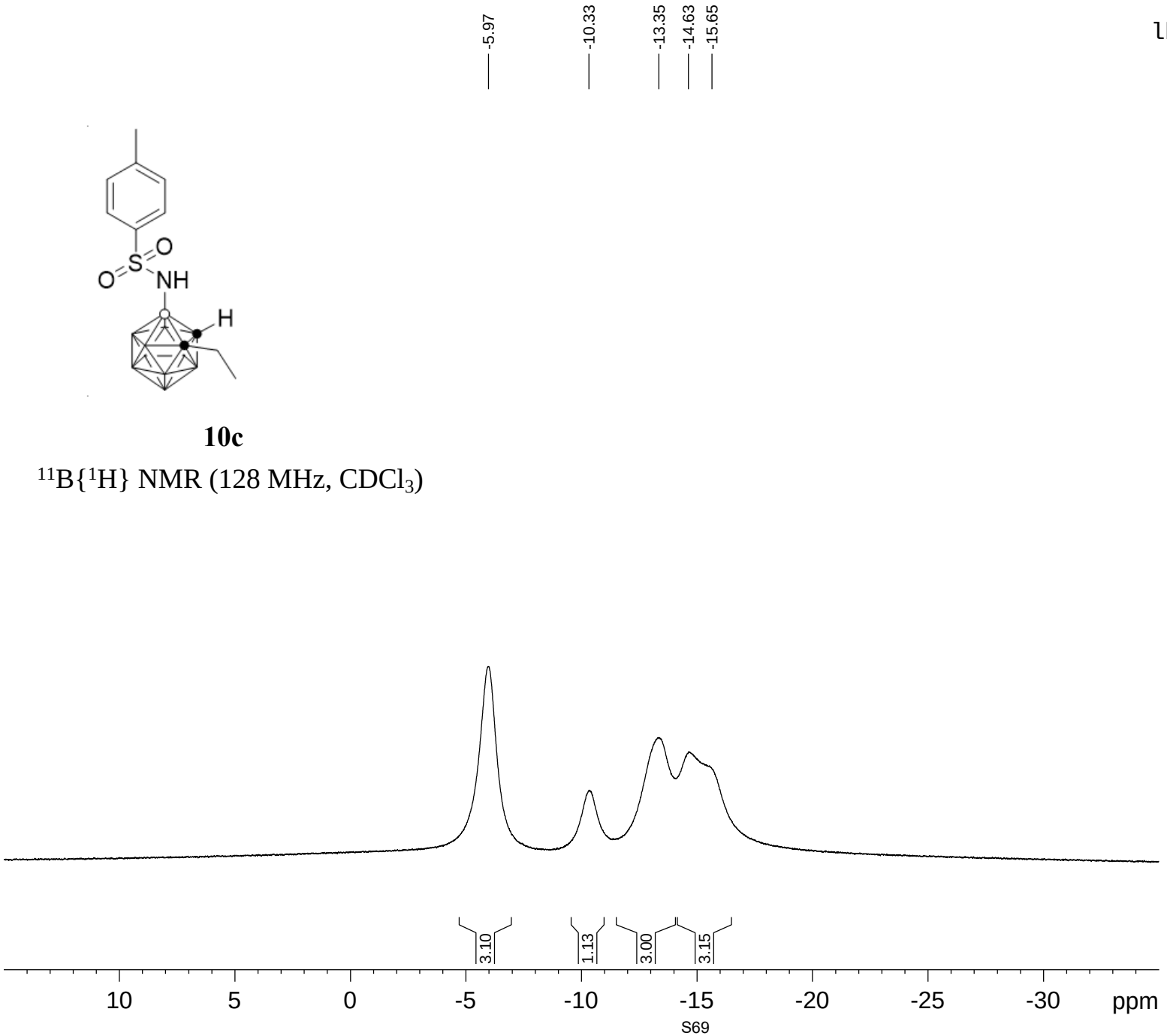


lhr-C-0582-cc-CDCl3

```
Current Data Parameters
NAME      lhr-C-0582-cc-CDCl3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170101
Time              20.13 h
INSTRUM             spect
PROBHD    Z108618_0257 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                  271
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG                  203
DW                16.800 usec
DE                 6.50 usec
TE                295.3 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          100.6379014 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

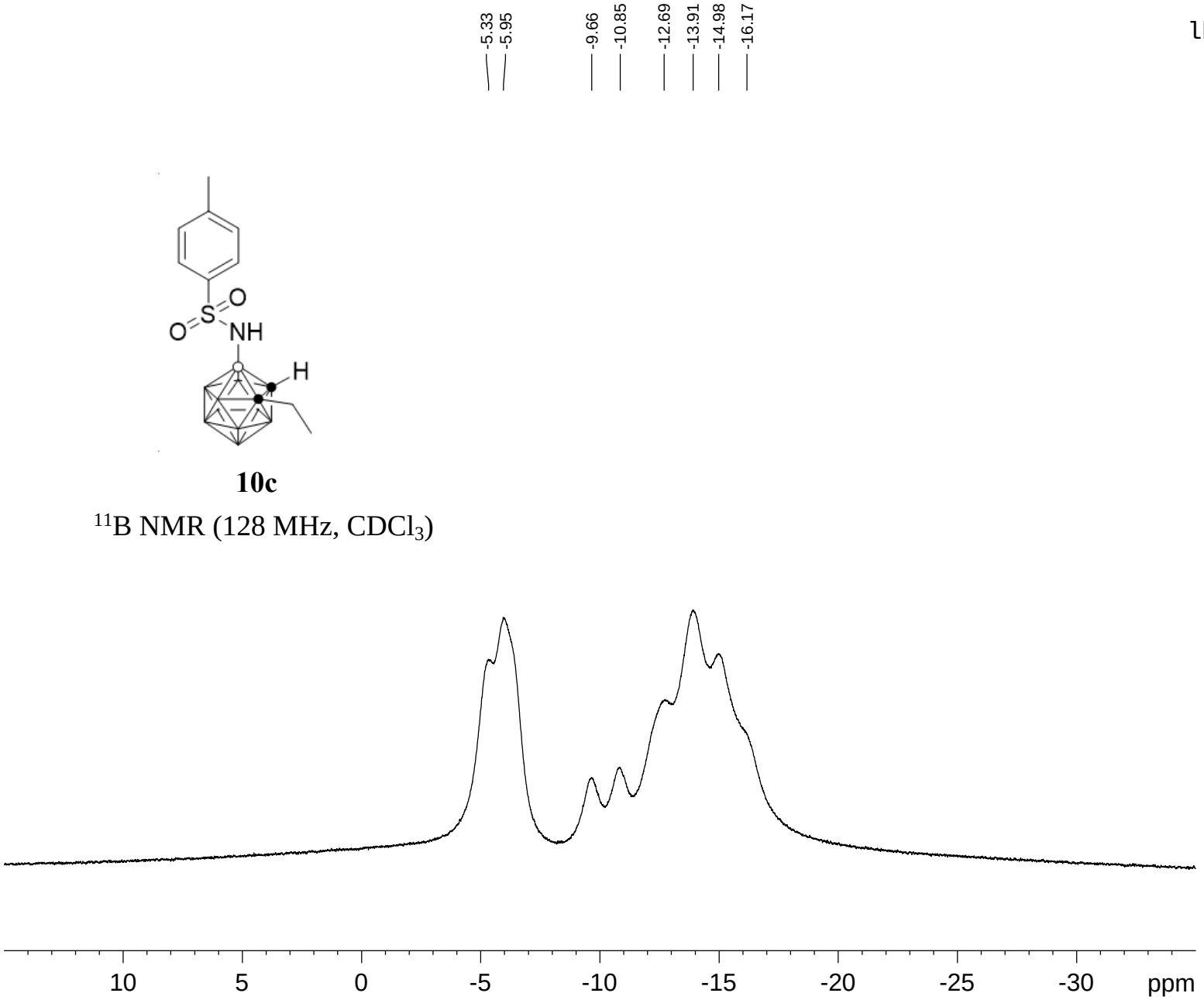
10c

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)



lhr-B-0582-cc-CDCl3

```
Current Data Parameters
NAME      lhr-B-0582-cc-CDCl3
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20170101
Time                  20.27 h
INSTRUM               spect
PROBHD   Z108618_0257 (
PULPROG                zgdc
TD                    65536
SOLVENT               CDCl3
NS                       26
DS                        4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                1.3631488 sec
RG                      287
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
```



lhr-B-0582-cc-CDCl3(C)

```
Current Data Parameters
NAME      lhr-B-0582-cc-CDCl3(C)
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_              20170101
Time                  20.28 h
INSTRUM               spect
PROBHD   Z108618_0257 (
PULPROG                  zg
TD                    65536
SOLVENT               CDCl3
NS                       37
DS                        2
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631488 sec
RG                      203
DW                   20.800 usec
DE                    6.50 usec
TE                   295.2 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             128.4097504 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```


10c

^{11}B NMR (128 MHz, $CDCl_3$)

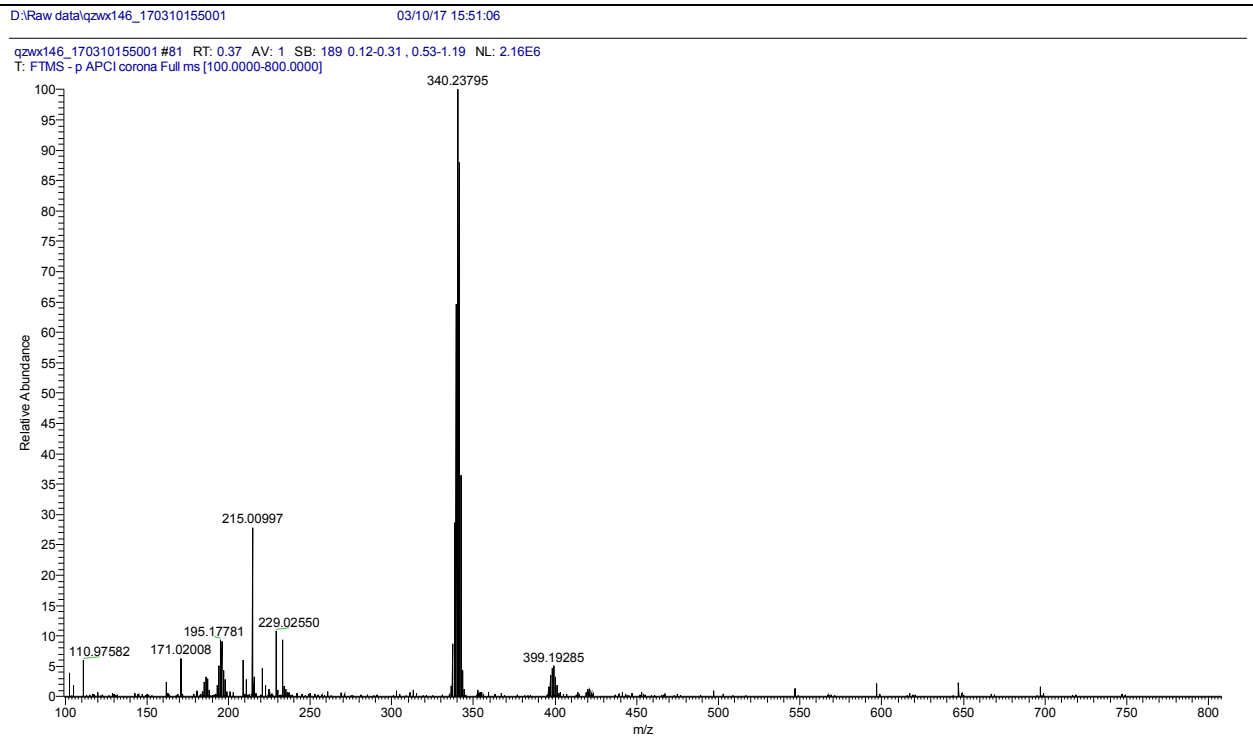
Thermo QEFMS Analysis Report

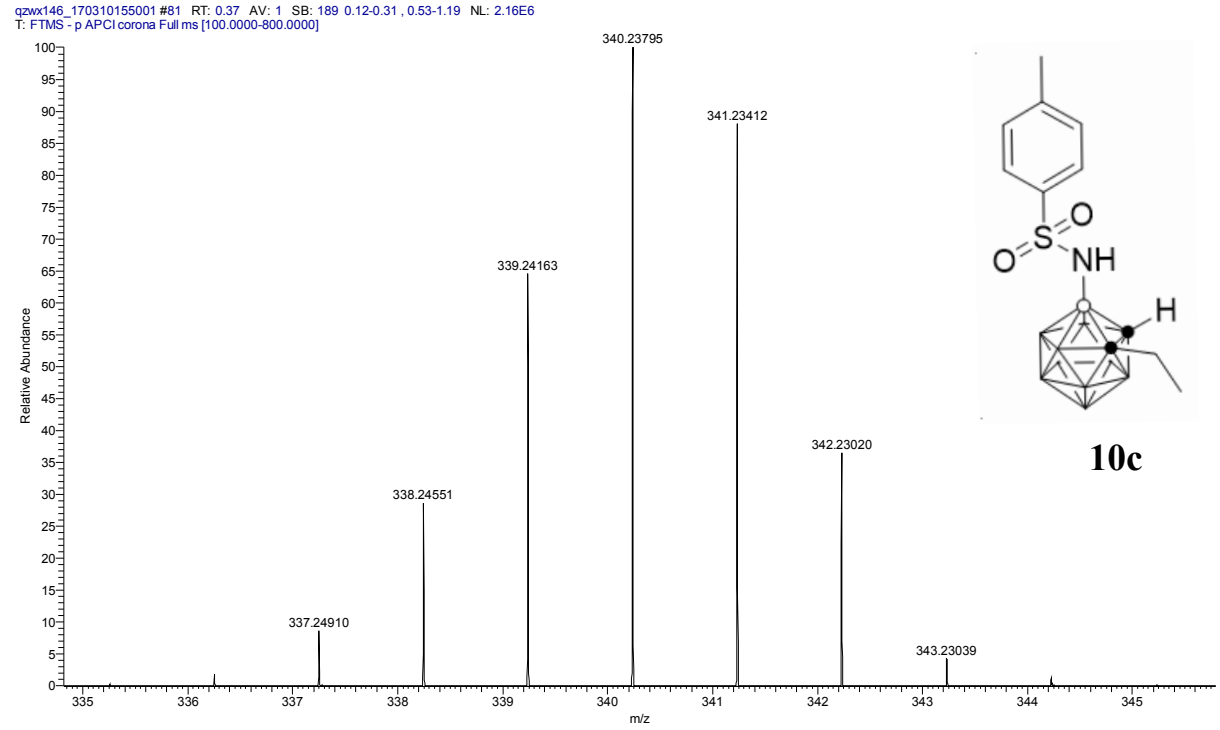
Analysis Info

Sample Name :	Lhr-0582	Reference No.:	Qzwx146
Instrument :	Q Exactive Focus Orbitrap		
Source :	APCI	Polarity :	Negative
Comment :	APCI neg, 5.0uA, by LC, with sheath gas		

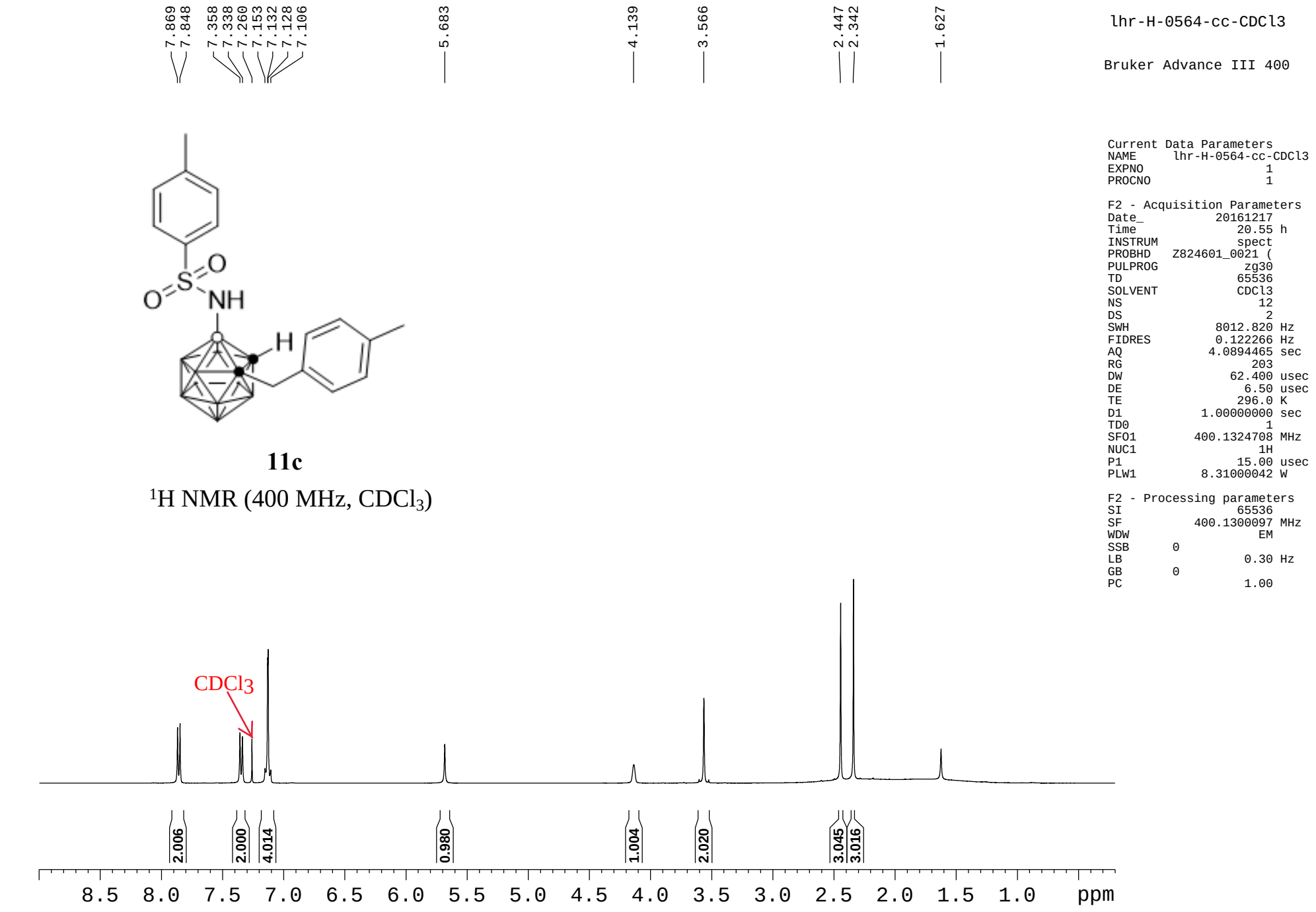
Accurate Mass Measurement

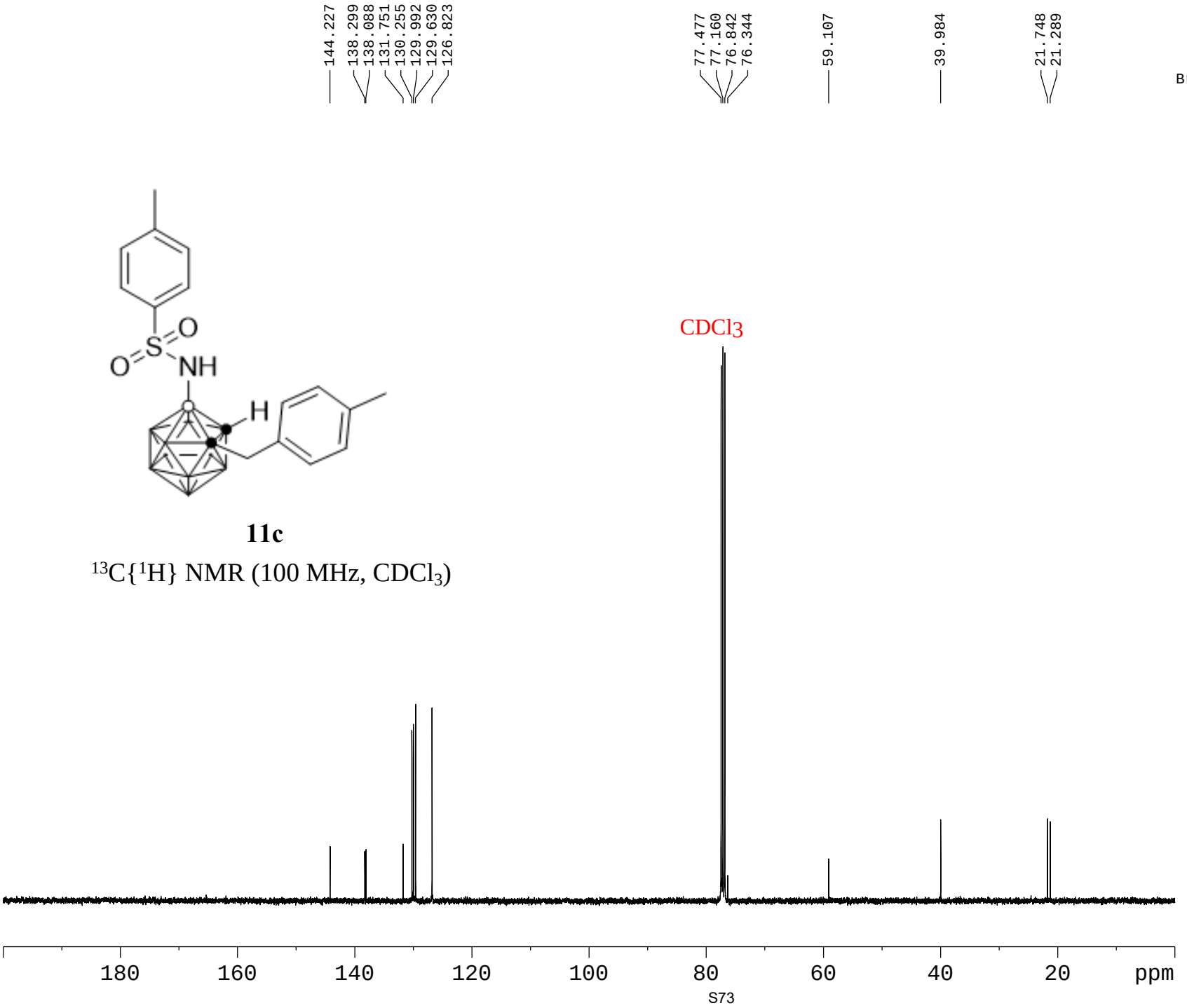
Molecular formula :	$C_{11}H_{23}B_{10}NO_2S$
Experimental Mass $[M-H]^-$:	340.23795
Theoretical Mass $[M-H]^-$:	340.23861
Error (ppm) :	1.9





10c


7.869
7.848
7.358
7.338
7.260
7.153
7.132
7.128
7.106
5.683
4.139
3.566
2.447
2.342
1.627
lhr-H-0564-cc-CDCl3
Bruker Advance III 400
Current Data Parameters
NAME lhr-H-0564-cc-CDCl3
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161217
Time 20.55 h
INSTRUM spect
PROBHD Z824601_0021 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 12
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 296.0 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 15.00 usec
PLW1 8.31000042 W
F2 - Processing parameters
SI 65536
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
S
O
O
NH
H
11c
1H NMR (400 MHz, CDCl3)
CDCl3
2.006
2.000
4.014
0.980
1.004
2.020
3.045
3.016
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm



11c

$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)

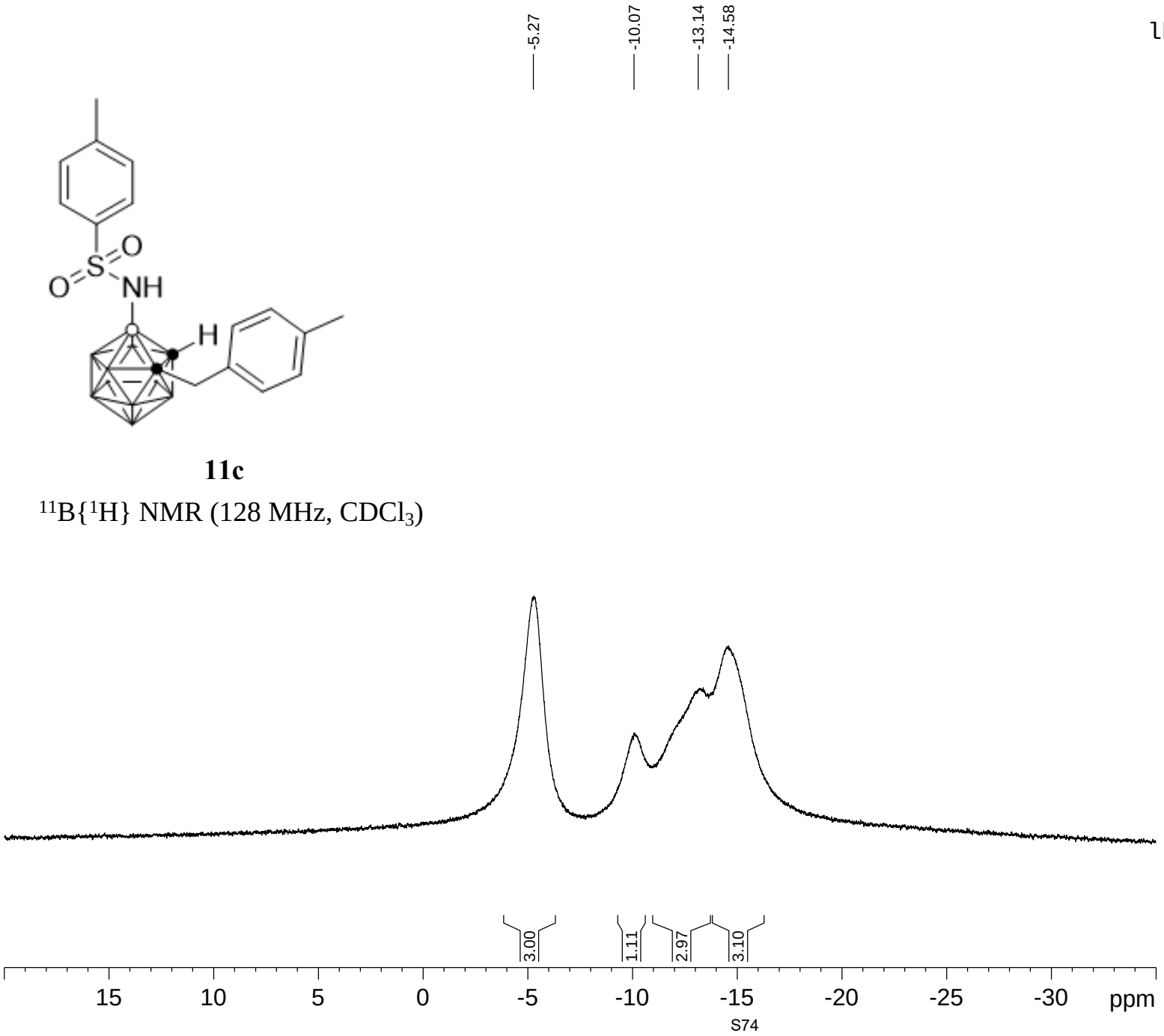
lhr-C-0564-cc-CDCl3

Bruker Advance III 400

```
Current Data Parameters
NAME      lhr-C-0564-cc-CDCl3
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20161217
Time                  20.59 h
INSTRUM                spect
PROBHD    Z824601_0021 (
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      774
DS                        4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                1.3631488 sec
RG                      203
DW                   20.800 usec
DE                    6.50 usec
TE                   296.1 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                       1
SFO1           100.6228298 MHz
NUC1                    13C
P1                    9.50 usec
PLW1           41.25000000 W
SFO2           400.1316005 MHz
NUC2                    1H
CPDPRG[2             waltz16
PCPD2                90.00 usec
PLW2            8.31000042 W
PLW12           0.23083000 W
PLW13           0.11611000 W

F2 - Processing parameters
SI                    32768
SF             100.6127559 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

11c

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)

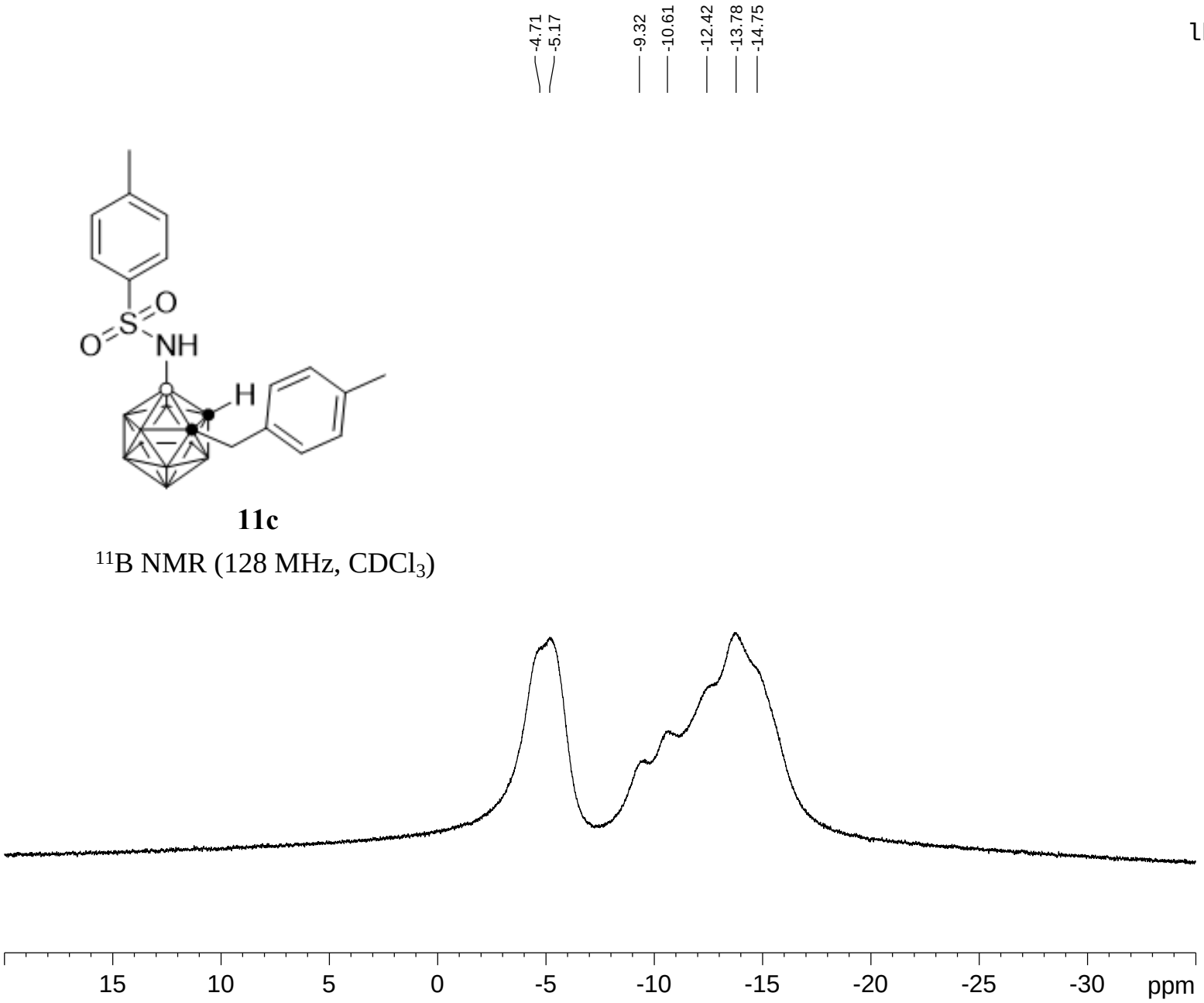


lhr-B-0564-cc-CDCl3

```
Current Data Parameters
NAME      lhr-B-0564-cc-CDCl3
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20161218
Time                  15.29 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                      287
DW                   20.800 usec
DE                    6.50 usec
TE                   296.2 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
```

11c

^{11}B NMR (128 MHz, $CDCl_3$)



lhr-B-0564-cc-CDCl3(C)

```
Current Data Parameters
NAME      lhr-B-0564-cc-CDCl3(C)
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161218
Time               15.31 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG              zg
TD                65536
SOLVENT           CDCl3
NS                   31
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                296.0 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
```

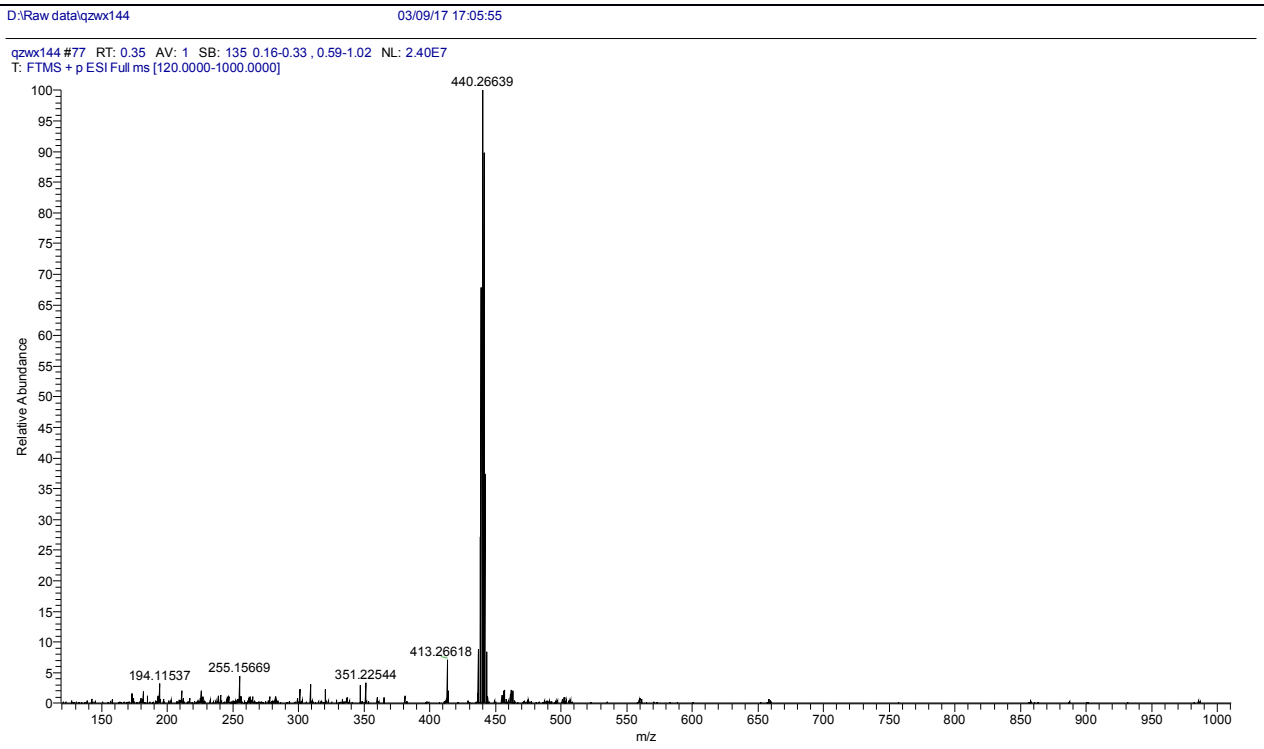
Thermo QEFMS Analysis Report

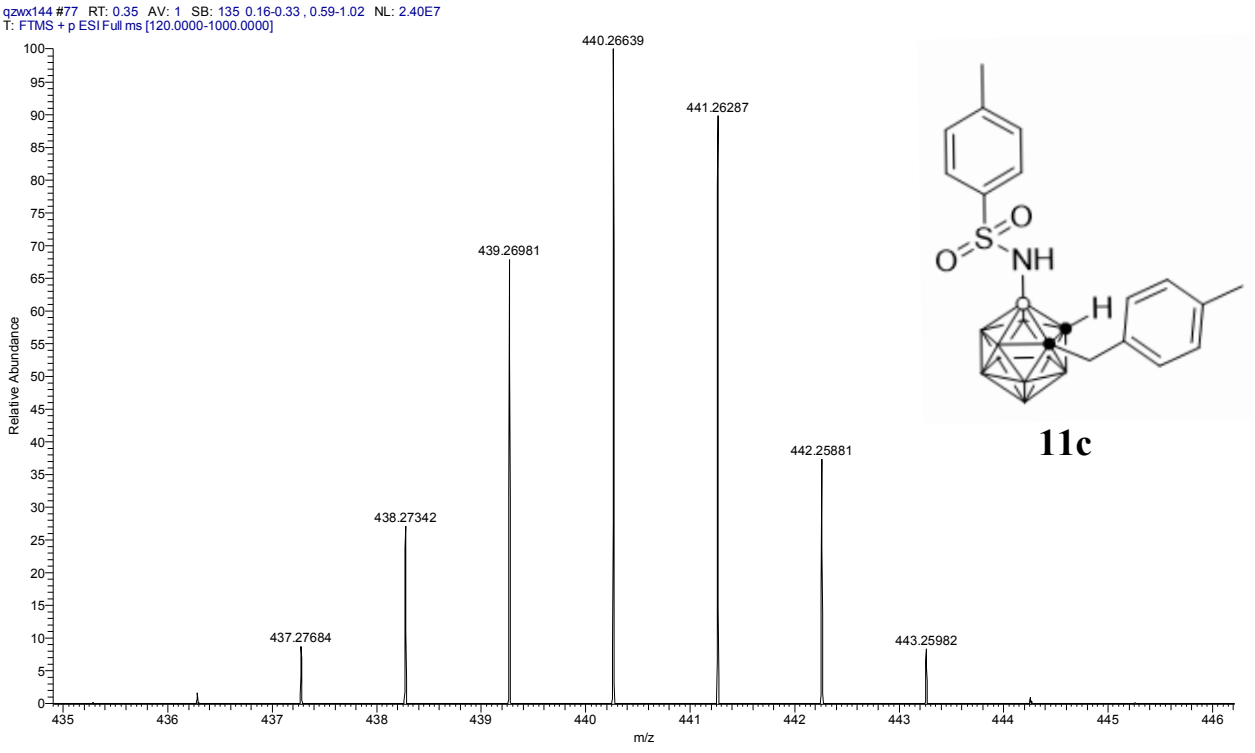
Analysis Info

Sample Name :	Lhr-0564	Reference No.:	Qzwx144
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.5kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{17}H_{27}B_{10}NO_2S$
Experimental Mass $[M+Na]^+$:	440.26639
Theoretical Mass $[M+Na]^+$:	440.26668
Error (ppm) :	0.6

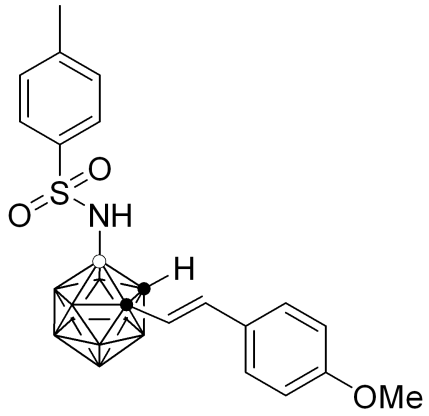




11c

lhr-H-0568-1-cc-CDCl3

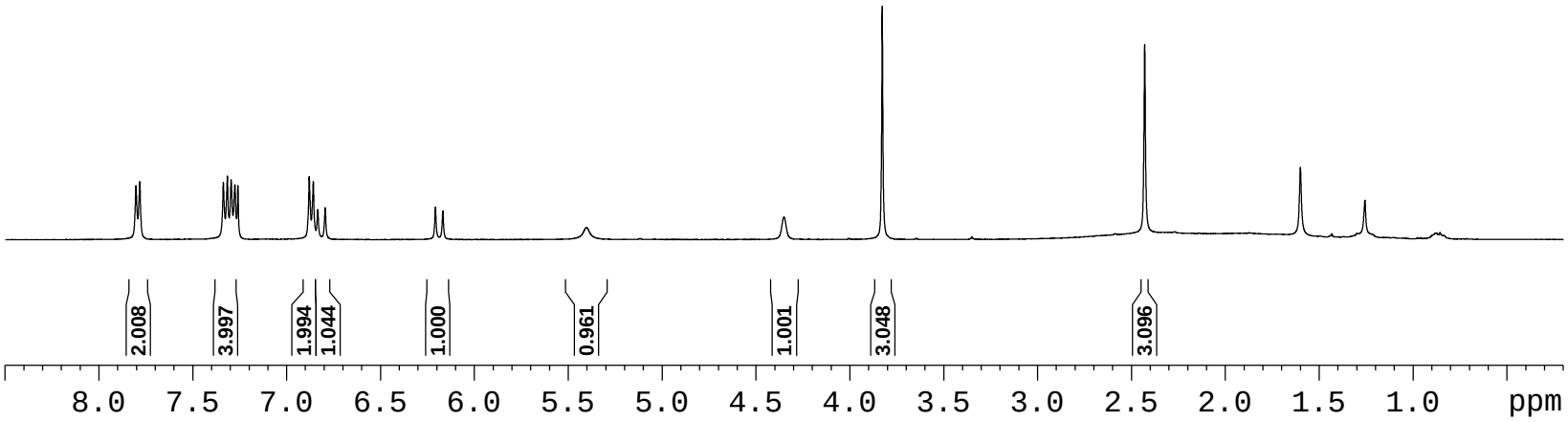
Bruker Advance III 400

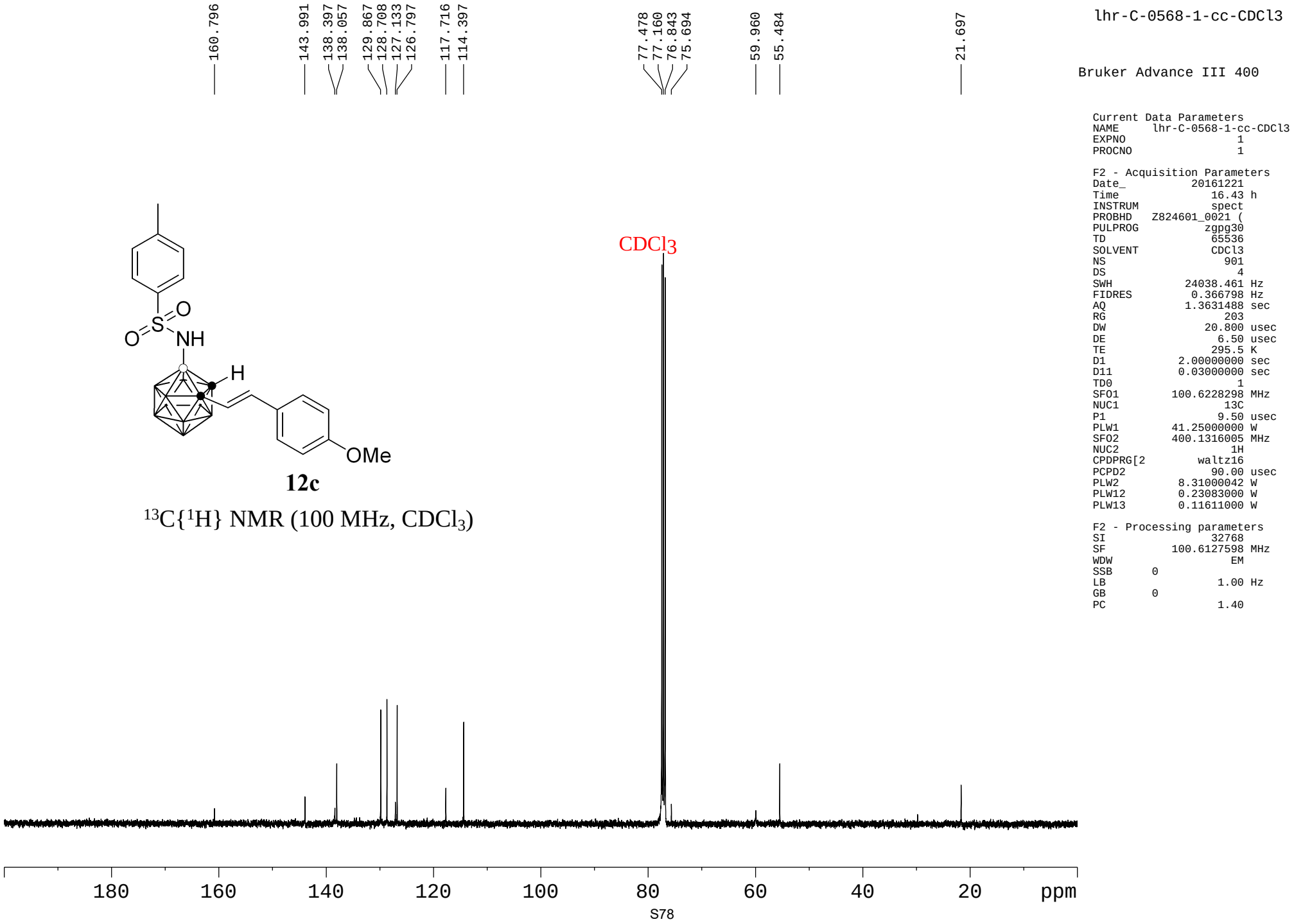


12c

^{1}H NMR (400 MHz, $CDCl_3$)

```
Current Data Parameters
NAME      lhr-H-0568-1-cc-CDCl3
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20161221
Time              10.04 h
INSTRUM           spect
PROBHD   Z824601_0021 (
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                    9
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                295.8 K
D1           1.00000000 sec
TD0                   1
SFO1        400.1324708 MHz
NUC1                1H
P1                15.00 usec
PLW1         8.31000042 W

F2 - Processing parameters
SI                65536
SF          400.1300098 MHz
WDW                 EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```





12c

$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)

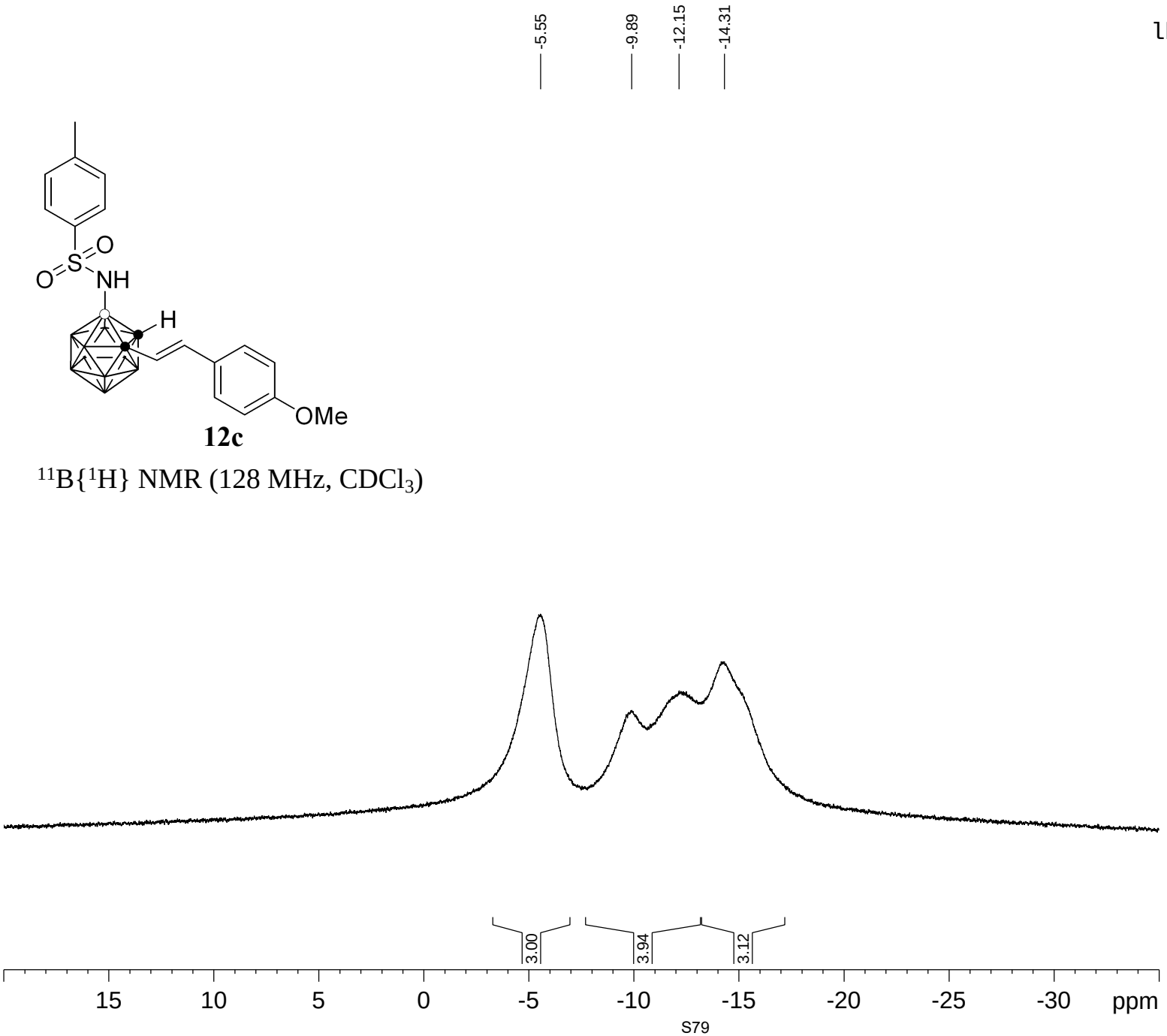
lhr-C-0568-1-cc-CDCl3

Bruker Advance III 400

```
Current Data Parameters
NAME      lhr-C-0568-1-cc-CDCl3
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_              20161221
Time                  16.43 h
INSTRUM                spect
PROBHD    Z824601_0021 (
PULPROG               zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       901
DS                         4
SWH                24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631488 sec
RG                       203
DW                    20.800 usec
DE                     6.50 usec
TE                    295.5 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1
SFO1             100.6228298 MHz
NUC1                     13C
P1                     9.50 usec
PLW1             41.25000000 W
SFO2             400.1316005 MHz
NUC2                     1H
CPDPRG[2              waltz16
PCPD2                 90.00 usec
PLW2              8.31000042 W
PLW12             0.23083000 W
PLW13             0.11611000 W

F2 - Processing parameters
SI                     32768
SF               100.6127598 MHz
WDW                       EM
SSB       0
LB                     1.00 Hz
GB        0
PC                     1.40
```

12c

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)

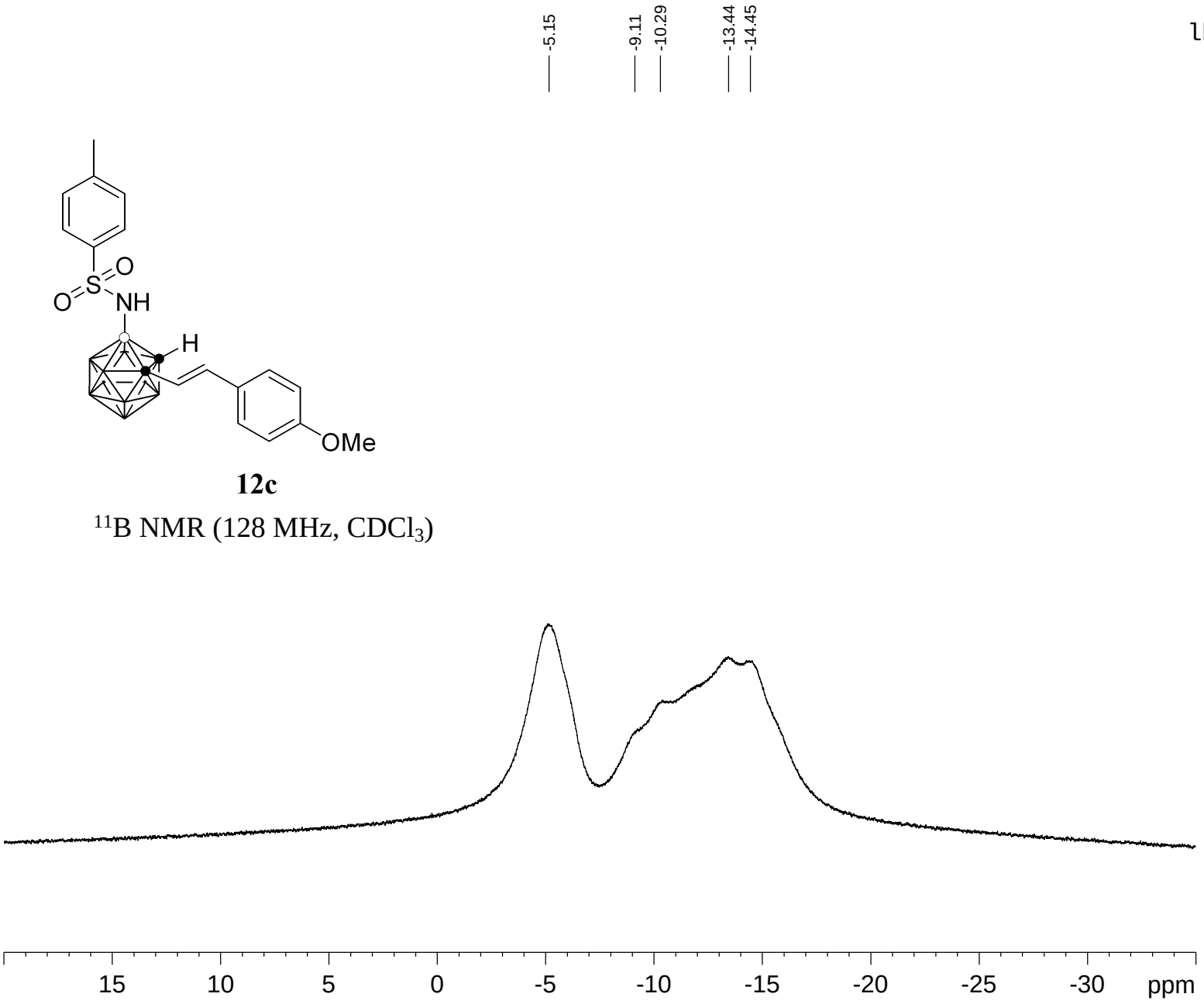


lhr-B-0568-1-cc-CDCl3

```
Current Data Parameters
NAME      lhr-B-0568-1-cc-CDCl3
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_               20161221
Time                   13.19 h
INSTRUM                spect
PROBHD    Z108618_0257 (
PULPROG                 zgdc
TD                     65536
SOLVENT                CDCl3
NS                        39
DS                         4
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                 1.3631488 sec
RG                       287
DW                    20.800 usec
DE                     6.50 usec
TE                    295.3 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
```

12c

^{11}B NMR (128 MHz, $CDCl_3$)



lhr-B-0568-1-cc-CDCl3(C)

```
Current Data Parameters
NAME      lhr-B-0568-1-cc-CDCl3(C)
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20161221
Time              13.25 h
INSTRUM           spect
PROBHD   Z108618_0257 (
PULPROG             zg
TD                65536
SOLVENT           CDCl3
NS                   74
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                294.6 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
```

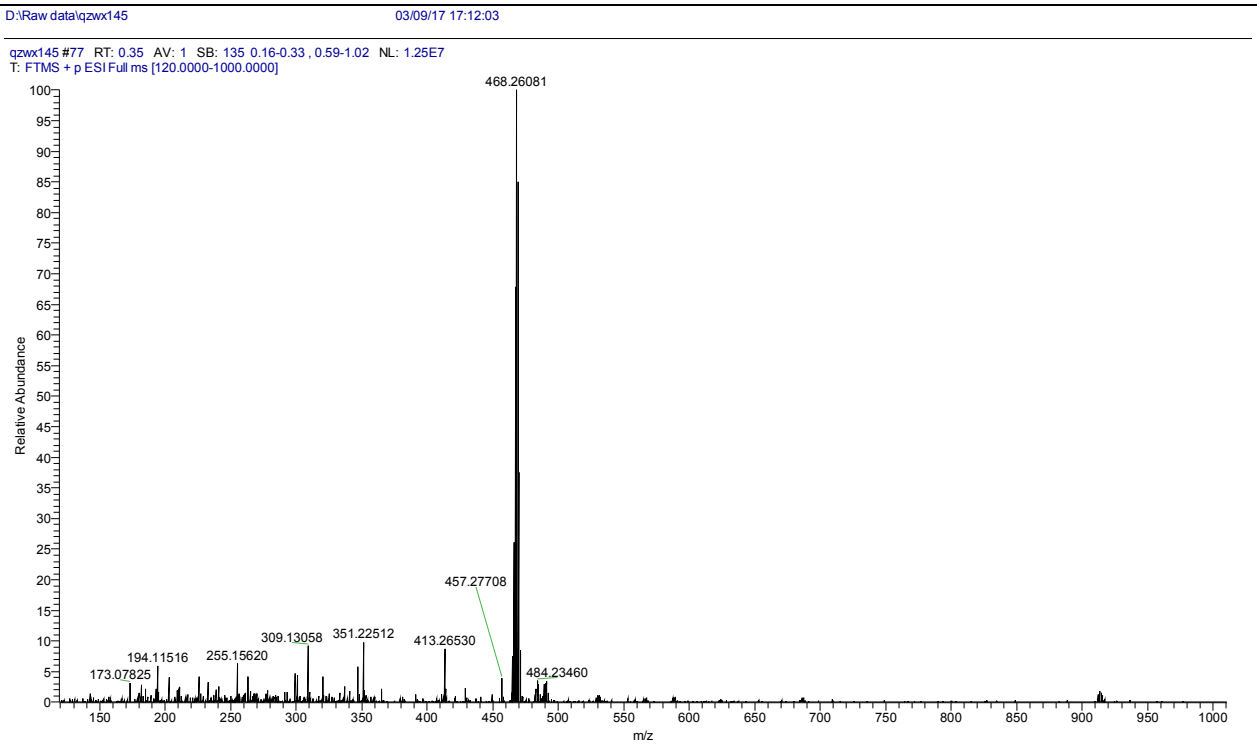
Thermo QEFMS Analysis Report

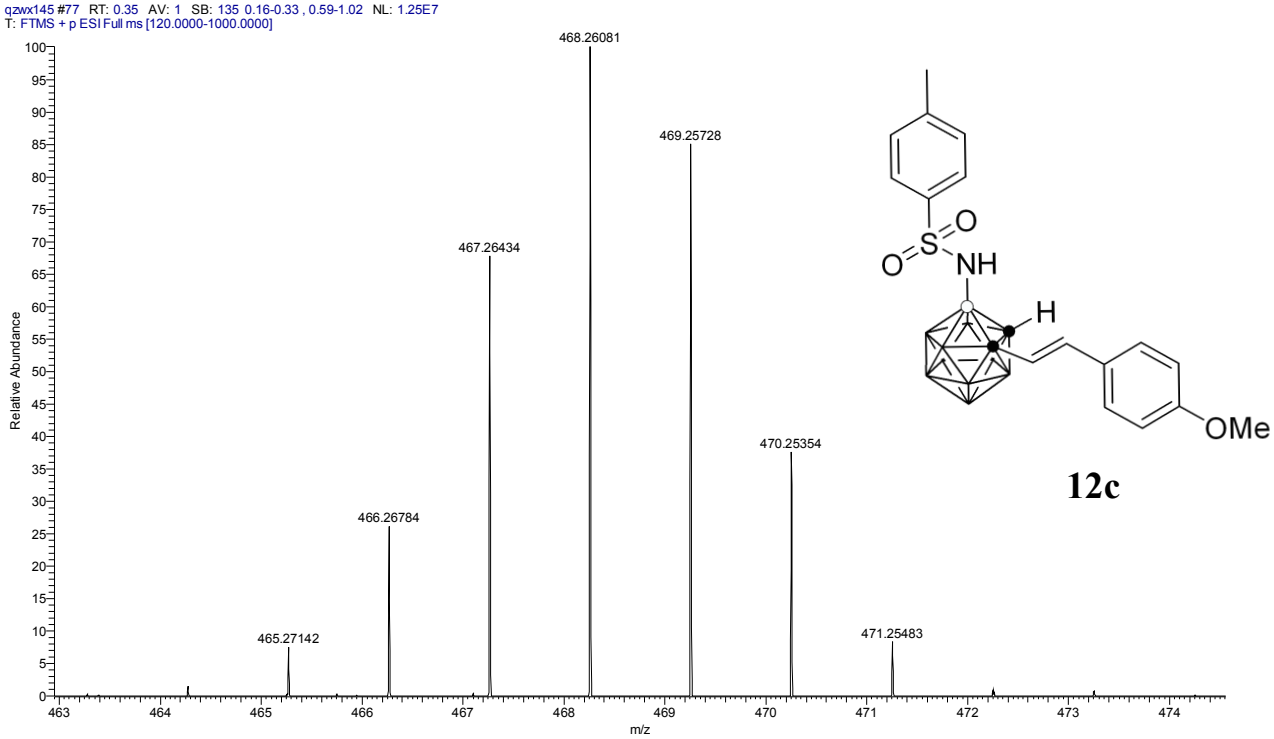
Analysis Info

Sample Name :	Lhr-0568-1	Reference No.:	Qzwx145
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.5kV, by LC, with sheath gas		

Accurate Mass Measurement

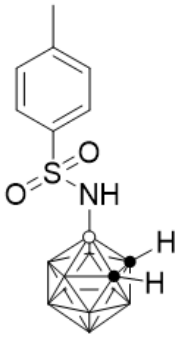
Molecular formula :	$C_{18}H_{27}B_{10}NO_3S$
Experimental Mass $[M+Na]^+$:	468.26081
Theoretical Mass $[M+Na]^+$:	468.26165
Error (ppm) :	1.7





lhr-H-0562-3-cc-CDCl3

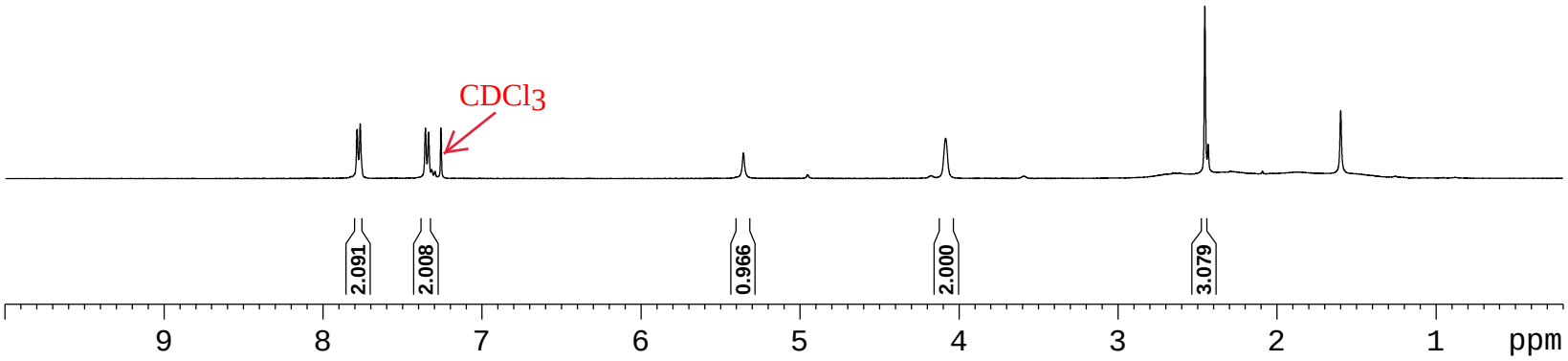
Bruker Advance III 400

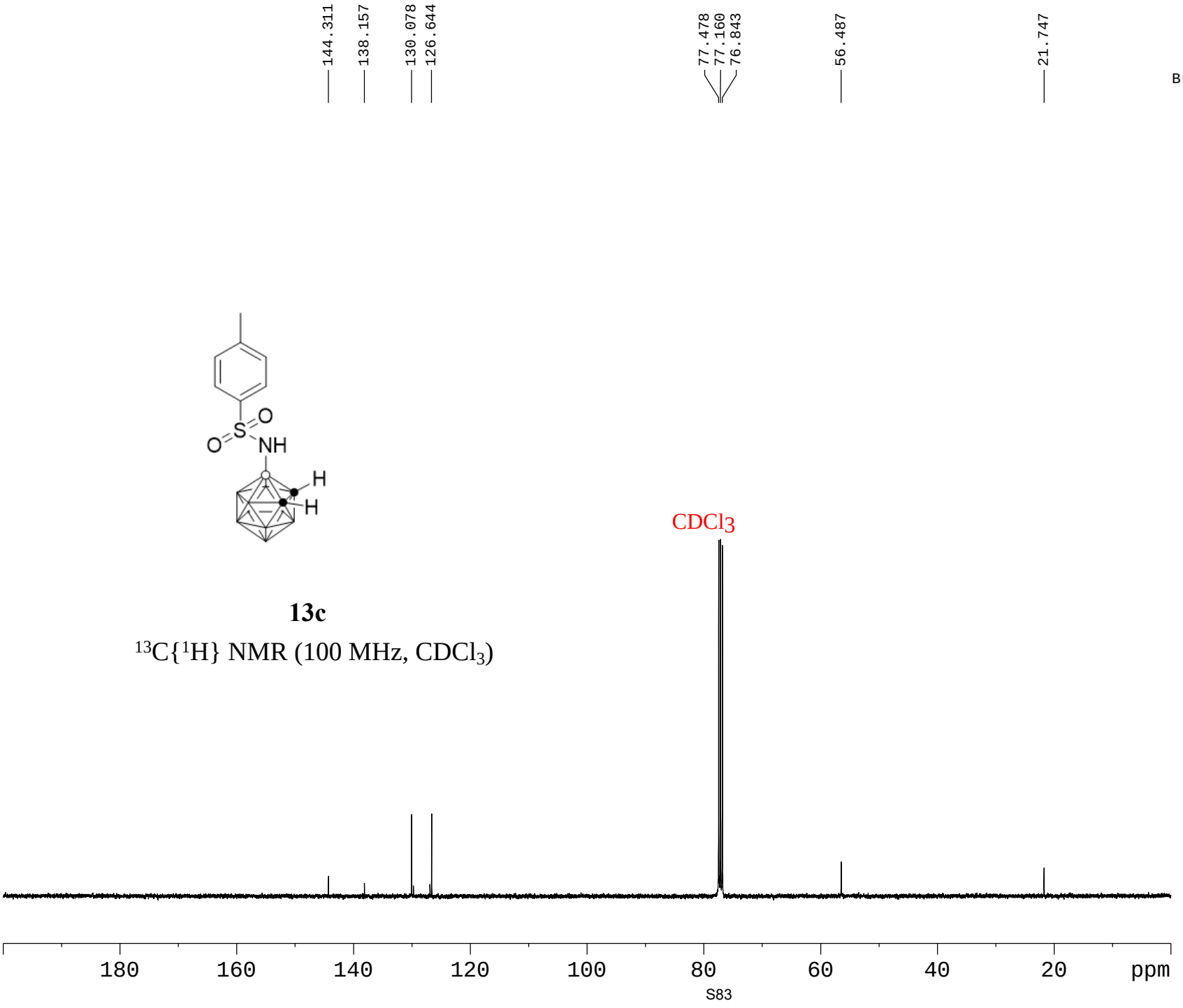


13c

^{1}H NMR (400 MHz, $CDCl_3$)

```
Current Data Parameters
NAME      lhr-H-0562-3-cc-CDCl3
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20161215
Time              17.23 h
INSTRUM           spect
PROBHD   Z824601_0021 (
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                296.1 K
D1           1.00000000 sec
TD0                   1
SFO1        400.1324708 MHz
NUC1                1H
P1                15.00 usec
PLW1         8.31000042 W

F2 - Processing parameters
SI                65536
SF          400.1300100 MHz
WDW                 EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```





lhr-C-0562-3-cc-CDCl3

Bruker Advance III 400

Current Data Parameters
NAME lhr-C-0562-3-cc-CDCl3
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161215
Time 17.26 h
INSTRUM spect
PROBHD Z824601_0021 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1184
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 296.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 9.50 usec
PLW1 41.25000000 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 8.31000042 W
PLW12 0.23083000 W
PLW13 0.11611000 W

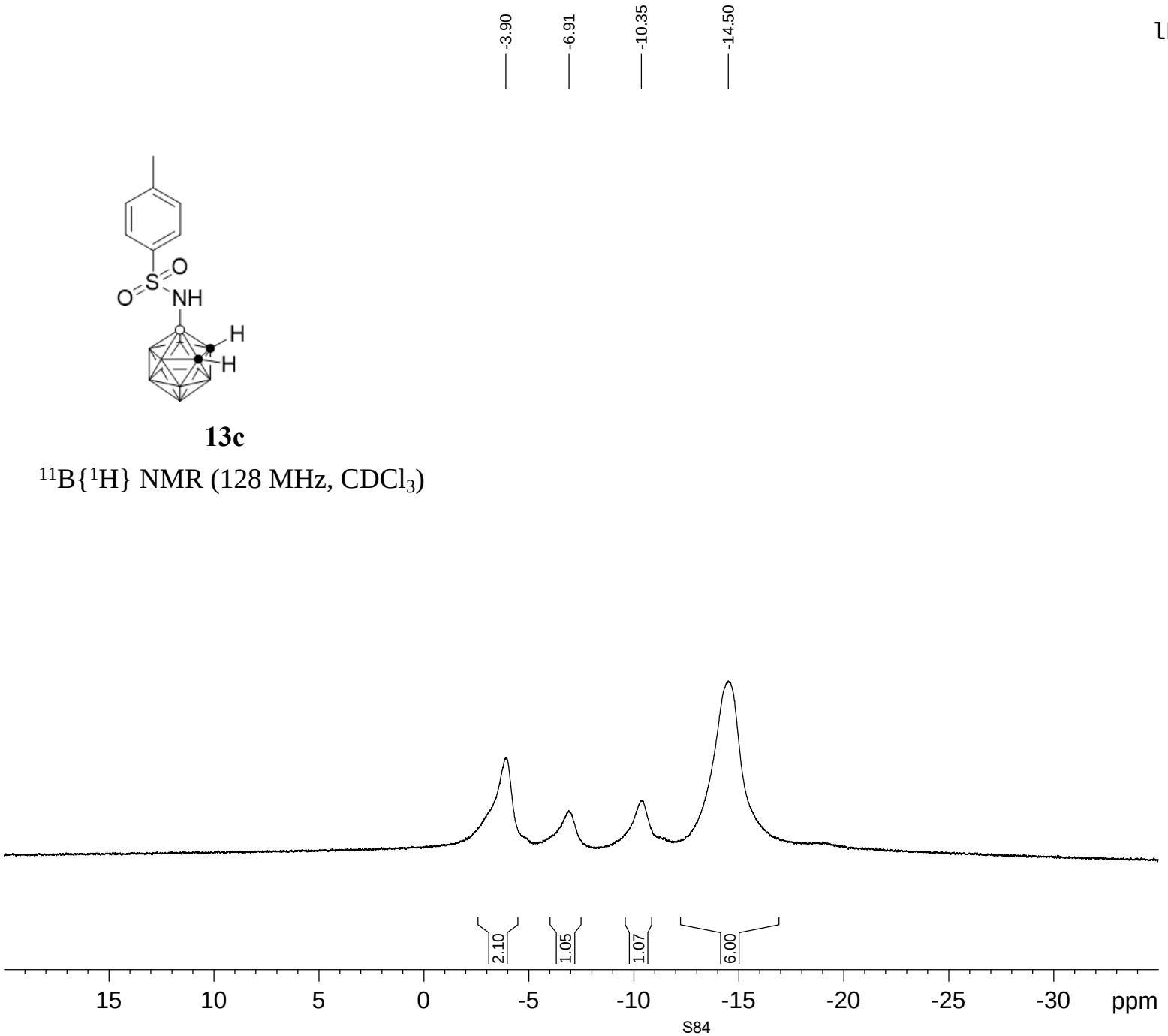
F2 - Processing parameters
SI 32768
SF 100.6127548 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

13c

$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)

13c

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)

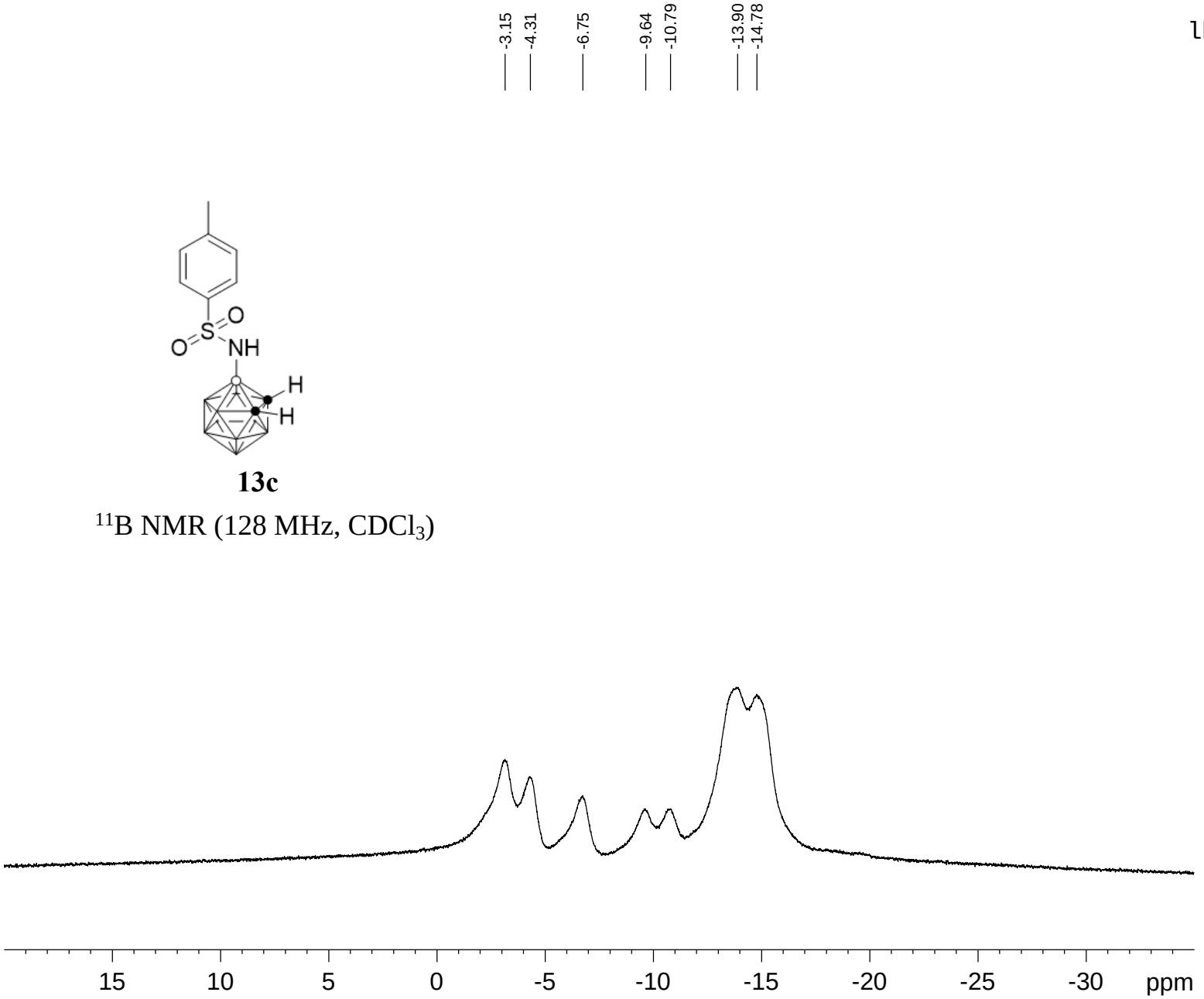


lhr-B-0562-3-cc-CDCl3

```
Current Data Parameters
NAME      lhr-B-0562-3-cc-CDCl3
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_               20161216
Time                   10.32 h
INSTRUM                spect
PROBHD    Z108618_0257 (
PULPROG                 zgdc
TD                     65536
SOLVENT                 C6D6
NS                        21
DS                         4
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                 1.3631488 sec
RG                       287
DW                    20.800 usec
DE                     6.50 usec
TE                    296.3 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
```

13c

^{11}B NMR (128 MHz, $CDCl_3$)



lhr-B-0562-3-cc-CDCl3(C)

```
Current Data Parameters
NAME      lhr-B-0562-3-cc-CDCl3(C)
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161216
Time              10.34 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG               zg
TD                 65536
SOLVENT             C6D6
NS                    34
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                296.0 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
```

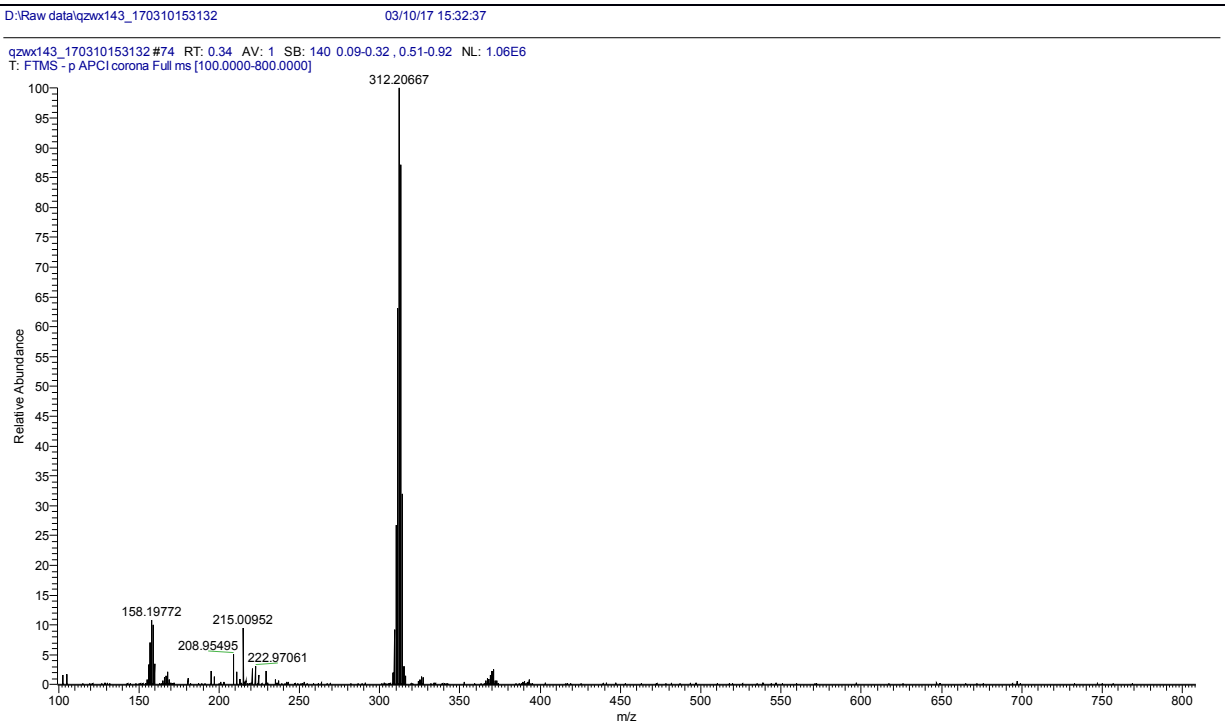
Thermo QEFMS Analysis Report

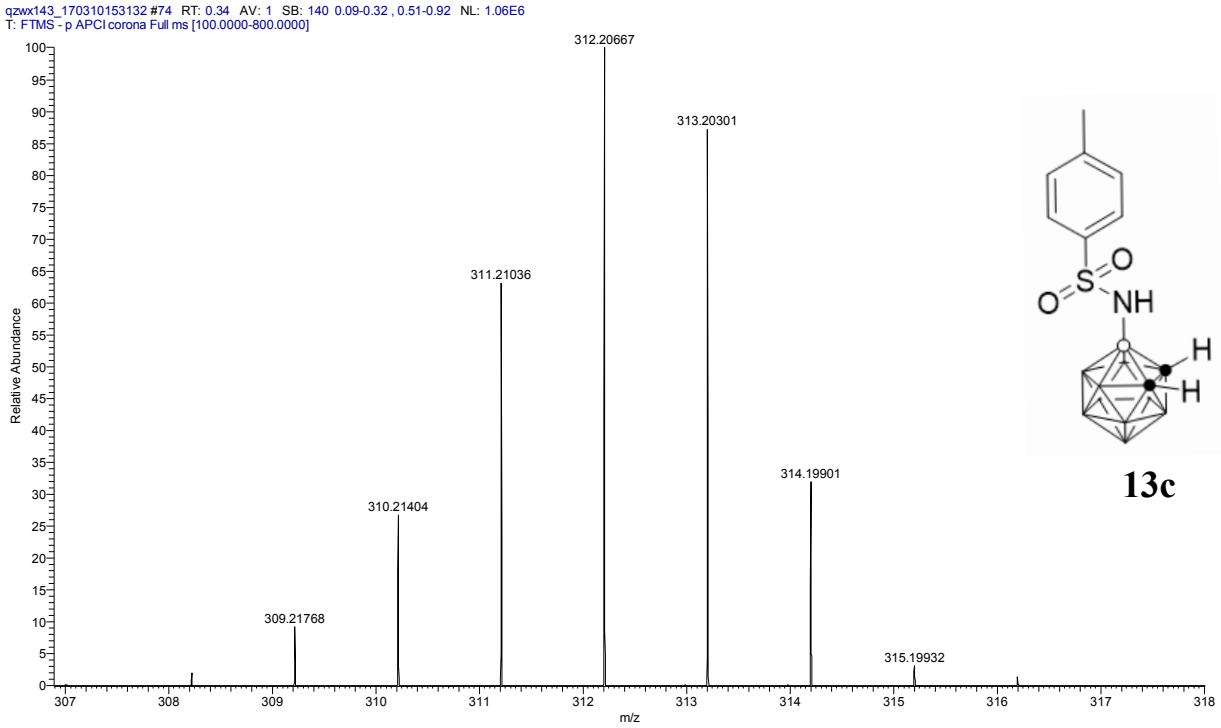
Analysis Info

Sample Name :	Lhr-0562-3	Reference No.:	Qzwx143
Instrument :	Q Exactive Focus Orbitrap		
Source :	APCI	Polarity :	Negative
Comment :	APCI neg, 5.0uA, by LC, with sheath gas		

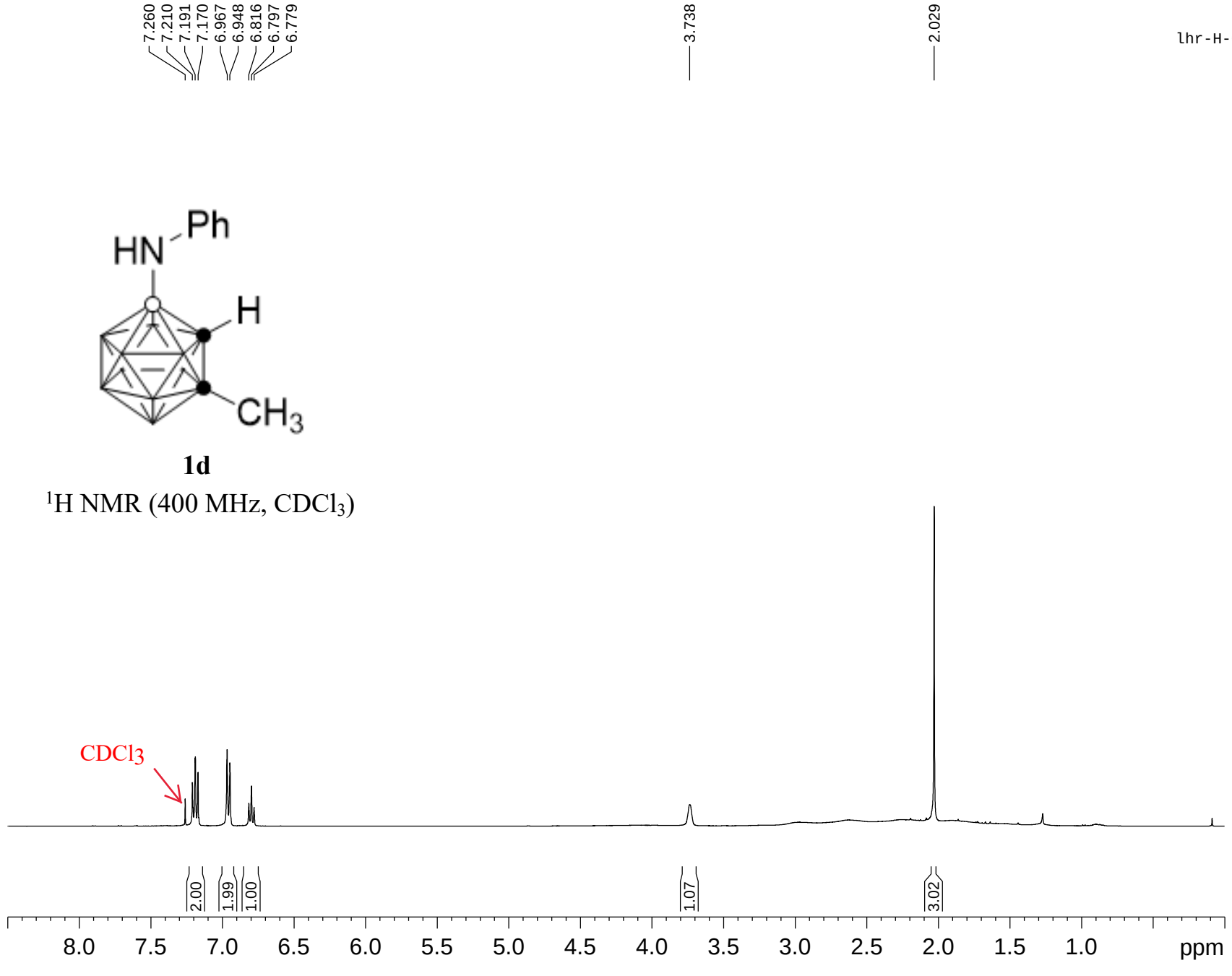
Accurate Mass Measurement

Molecular formula :	$C_9H_{19}B_{10}NO_2S$
Experimental Mass $[M-H]^-$:	312.20667
Theoretical Mass $[M-H]^-$:	312.20721
Error (ppm) :	1.7



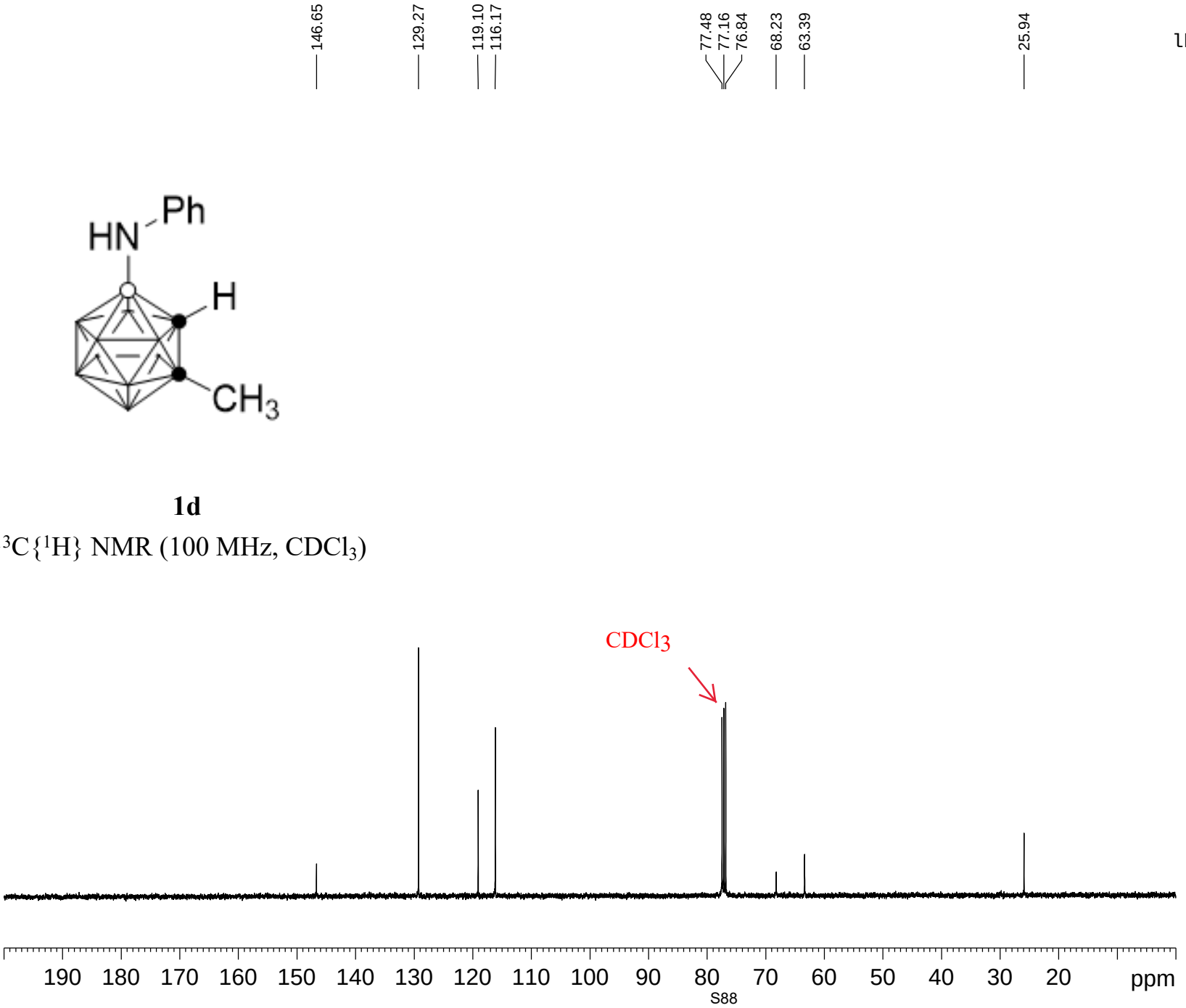


13c


7.260
7.210
7.191
7.170
6.967
6.948
6.816
6.797
6.779
3.738
2.029
lhr-H-0392-CDCl3
Current Data Parameters
NAME lhr-H-0392-CDCl3
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160607
Time 22.03 h
INSTRUM spect
PROBHD Z108618_0257 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 9
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 45.2
DW 62.400 usec
DE 6.50 usec
TE 294.9 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 400.2300104 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
HN
Ph
H
CH3
1d
1H NMR (400 MHz, CDCl3)
CDCl3
2.00
1.99
1.00
1.07
3.02
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

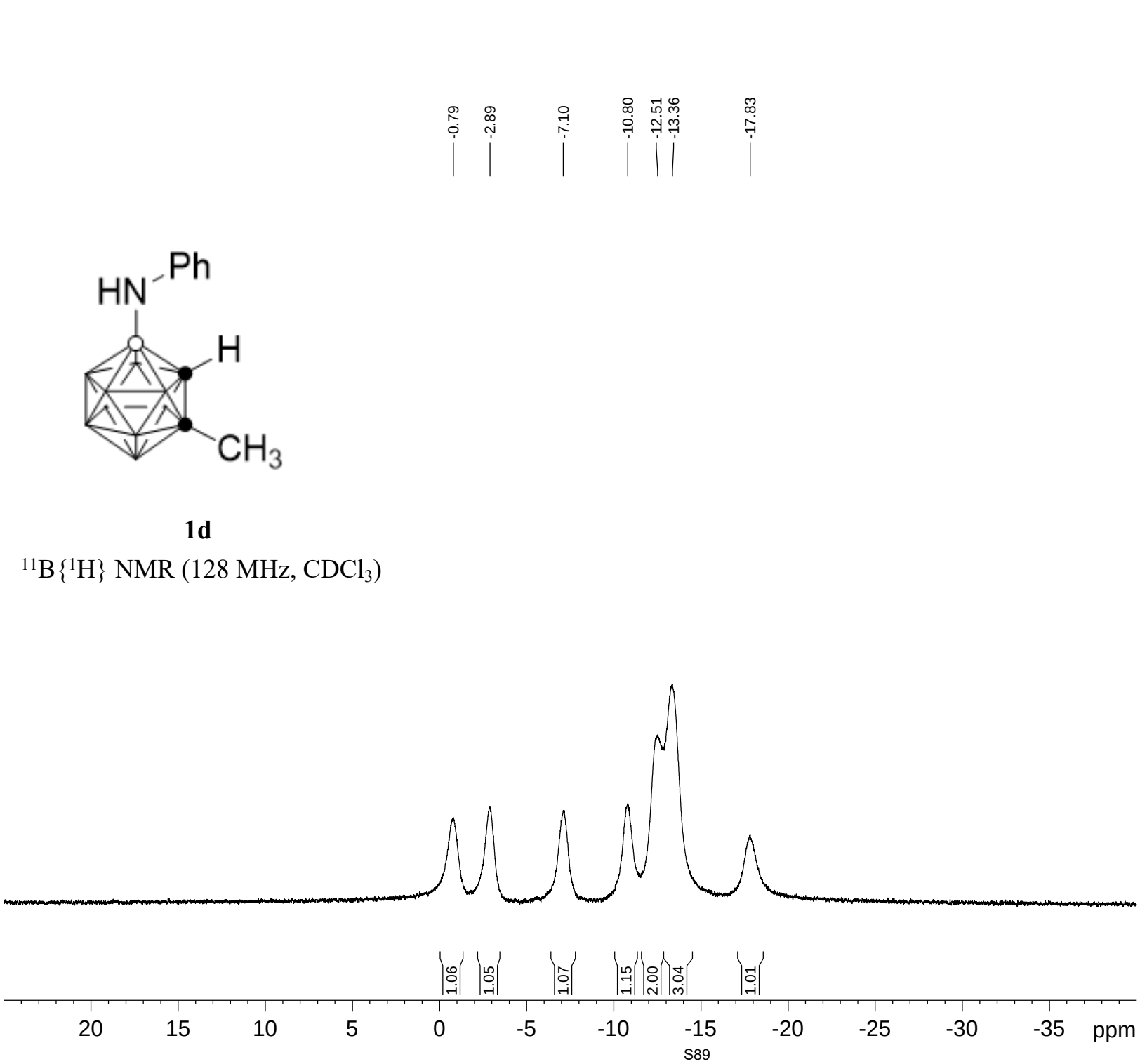
1d

$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)



lhr-C-0392-CDCl3

```
Current Data Parameters
NAME      lhr-C-0392-CDCl3
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20160608
Time              21.07 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                  148
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.1010048 sec
RG                 90.5
DW                16.800 usec
DE                 6.50 usec
TE                297.0 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           100.6379040 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

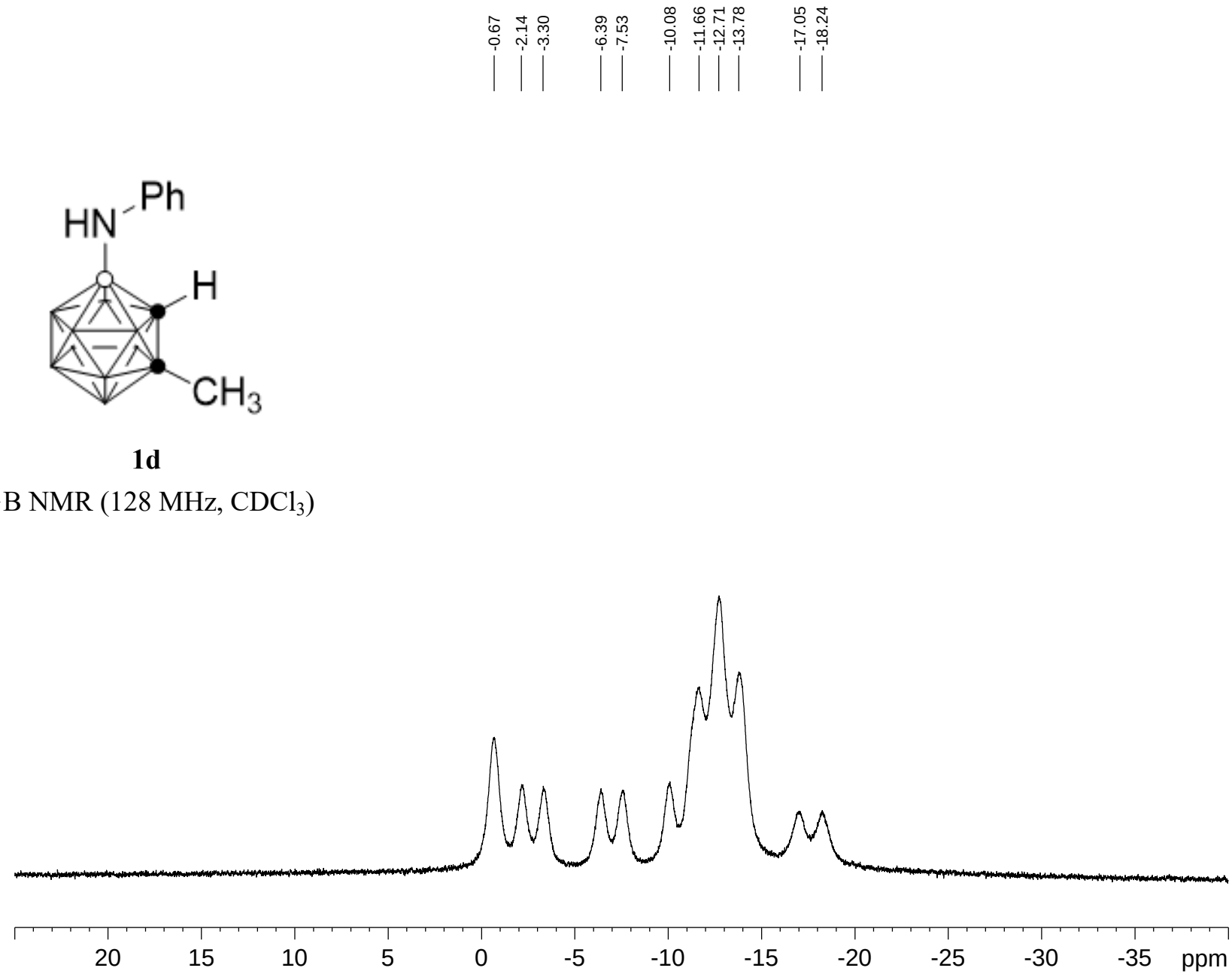


1d

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)

1d

^{11}B NMR (128 MHz, $CDCl_3$)



lhr-B-0392-CDCl3(C)

```
Current Data Parameters
NAME      lhr-B-0392-CDCl3(C)
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20160607
Time               22.13 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG                 zg
TD                   65536
SOLVENT              CDCl3
NS                      24
DS                       2
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ              1.3631488 sec
RG                       1
DW                  20.800 usec
DE                   6.50 usec
TE                  295.3 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
```

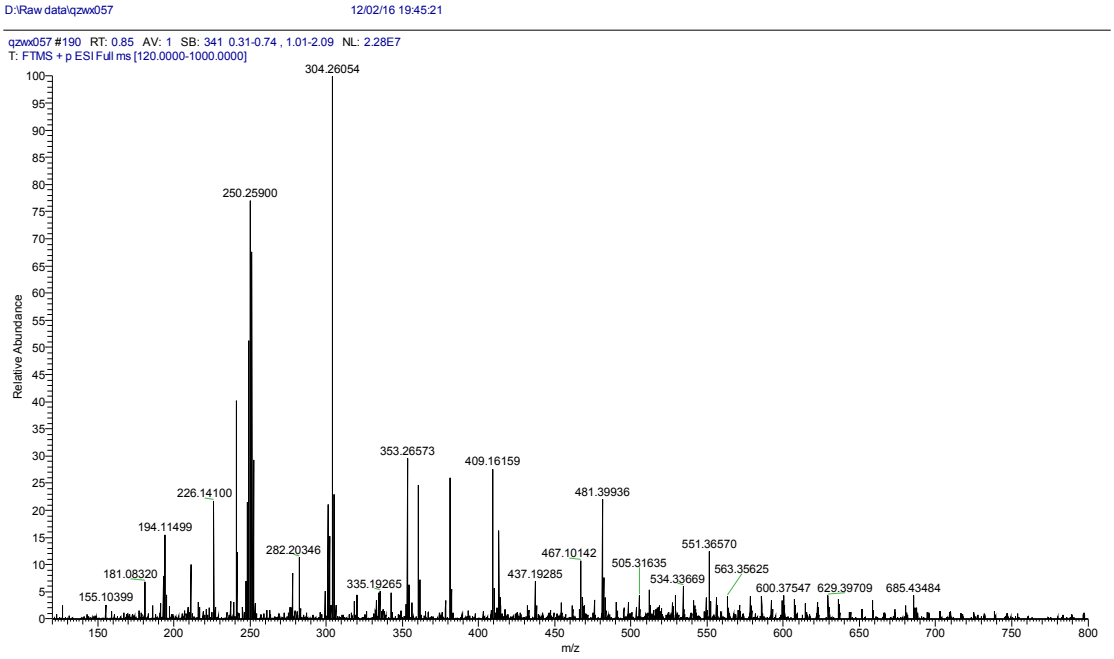
Thermo QEFMS Analysis Report

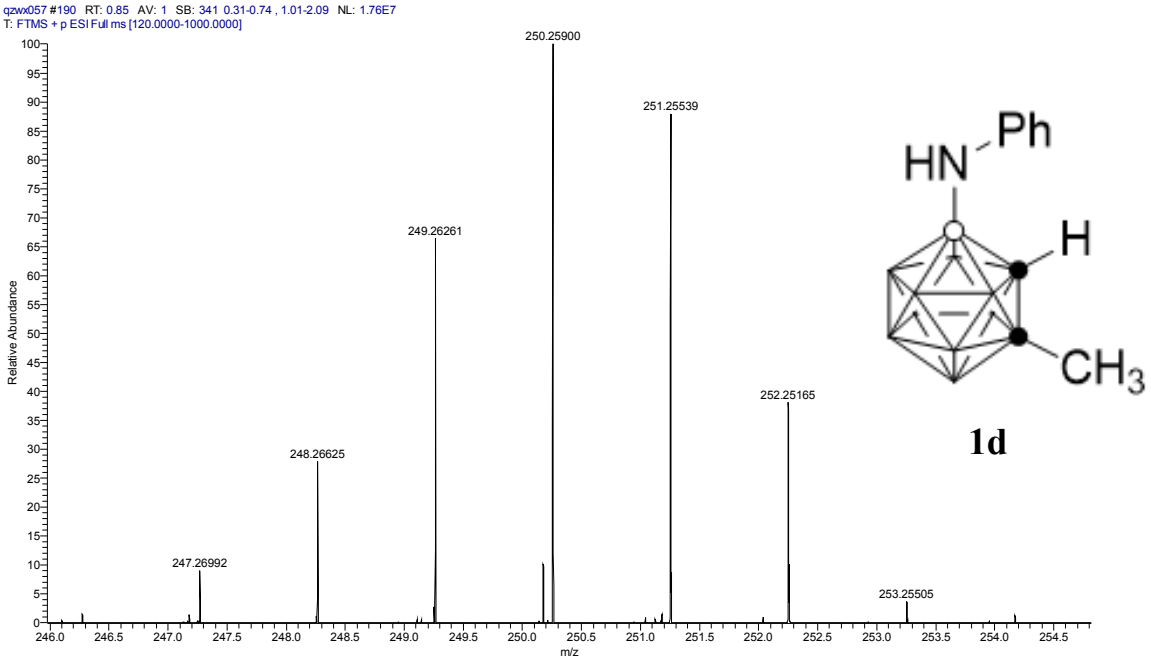
Analysis Info

Sample Name :	Lhr-392	Reference No.:	Qzwx057
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.5kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_9H_{19}B_{10}N$
Experimental Mass $[M+H]^+$:	250.25900
Theoretical Mass $[M+H]^+$:	250.25934
Error (ppm) :	1.3

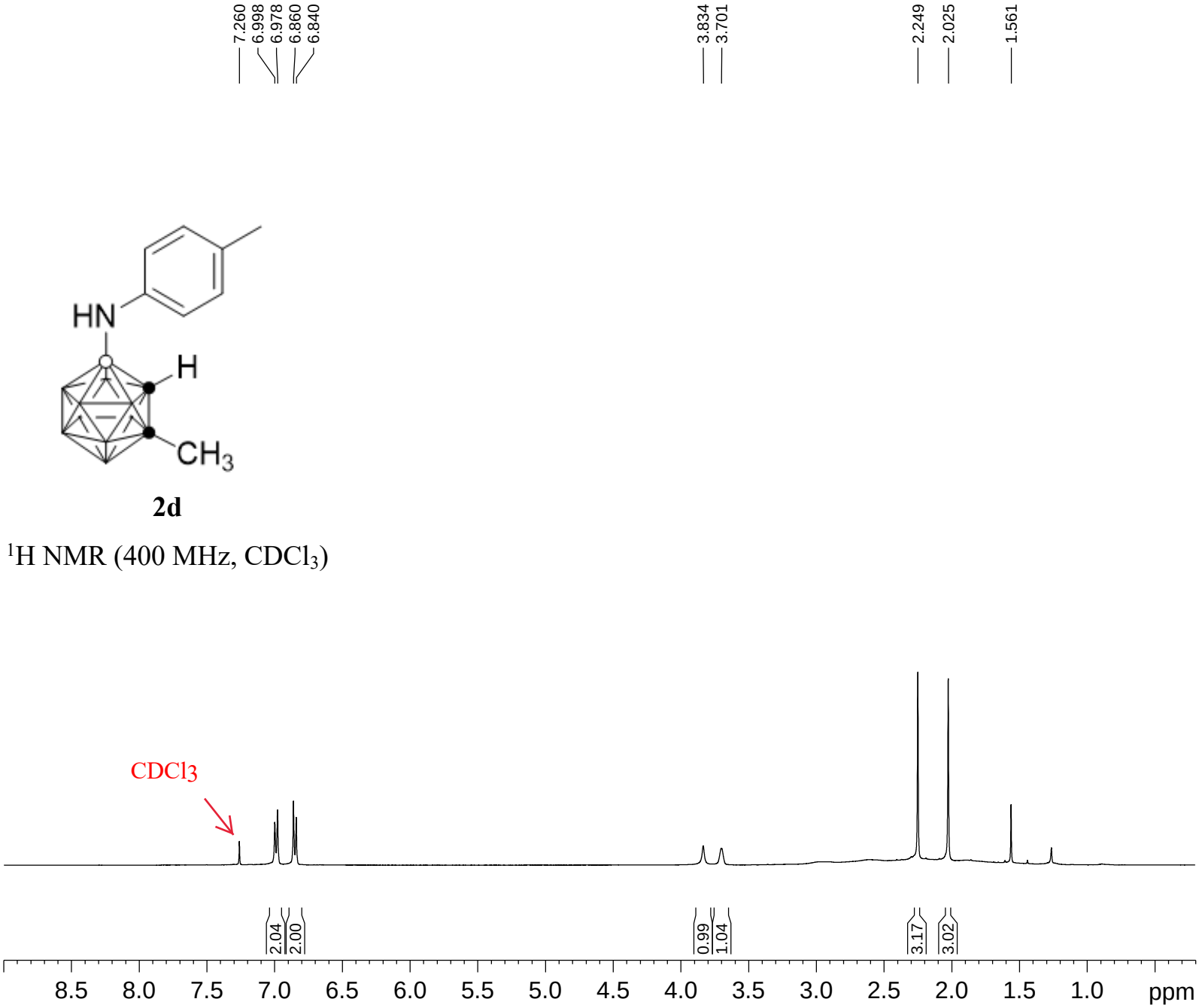




1d

2d

^{1}H NMR (400 MHz, $CDCl_3$)

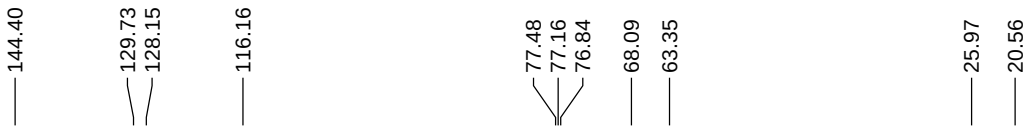


lhr-H-0513-phnhCH3-(

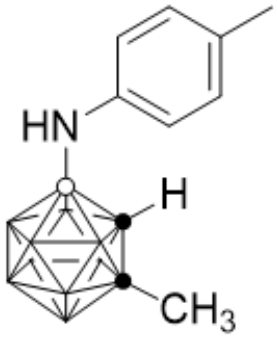
Current Data Parameters
NAME lhr-H-0513-phnhCH3-CD
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161101
Time 9.39 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 90.5
DW 62.400 usec
DE 6.50 usec
TE 296.6 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 400.2300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

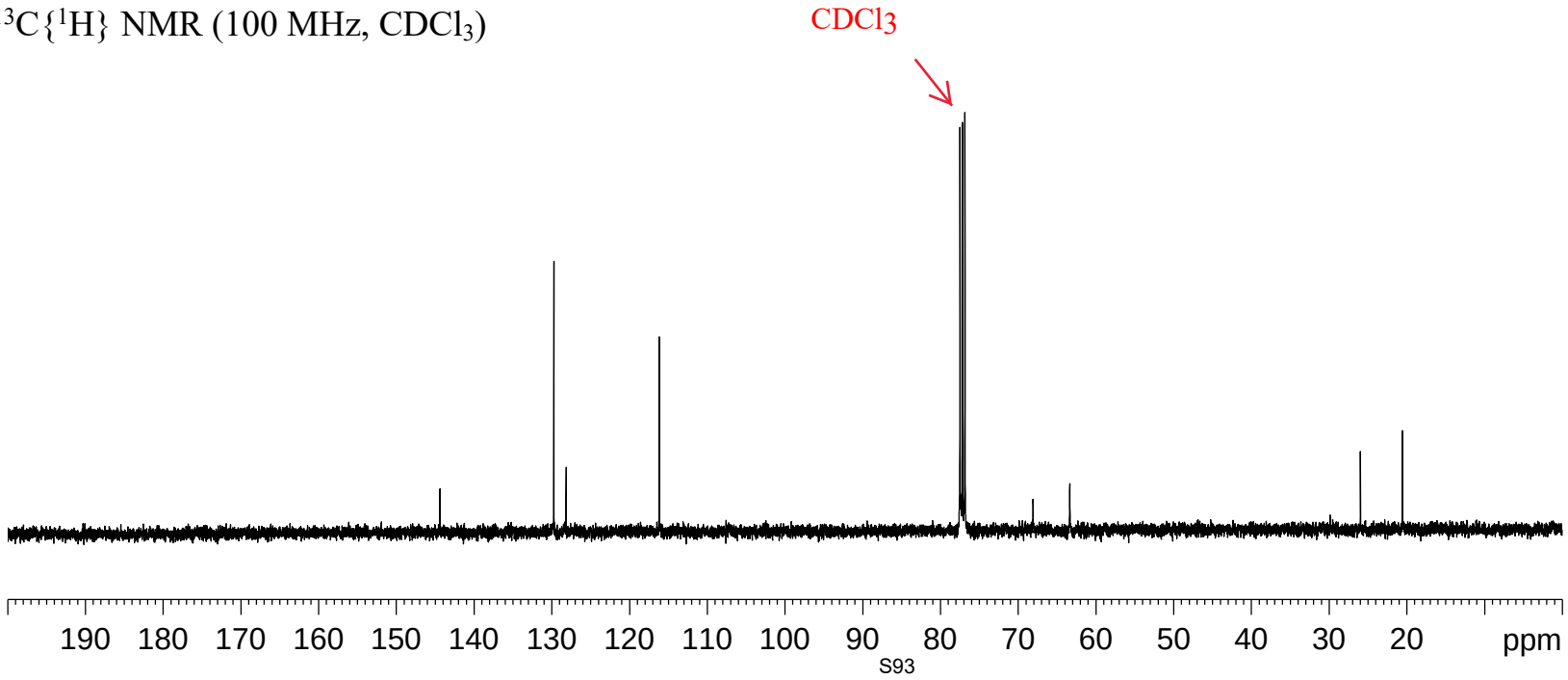


lhr-C-0513-phnhCH3



2d

$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)

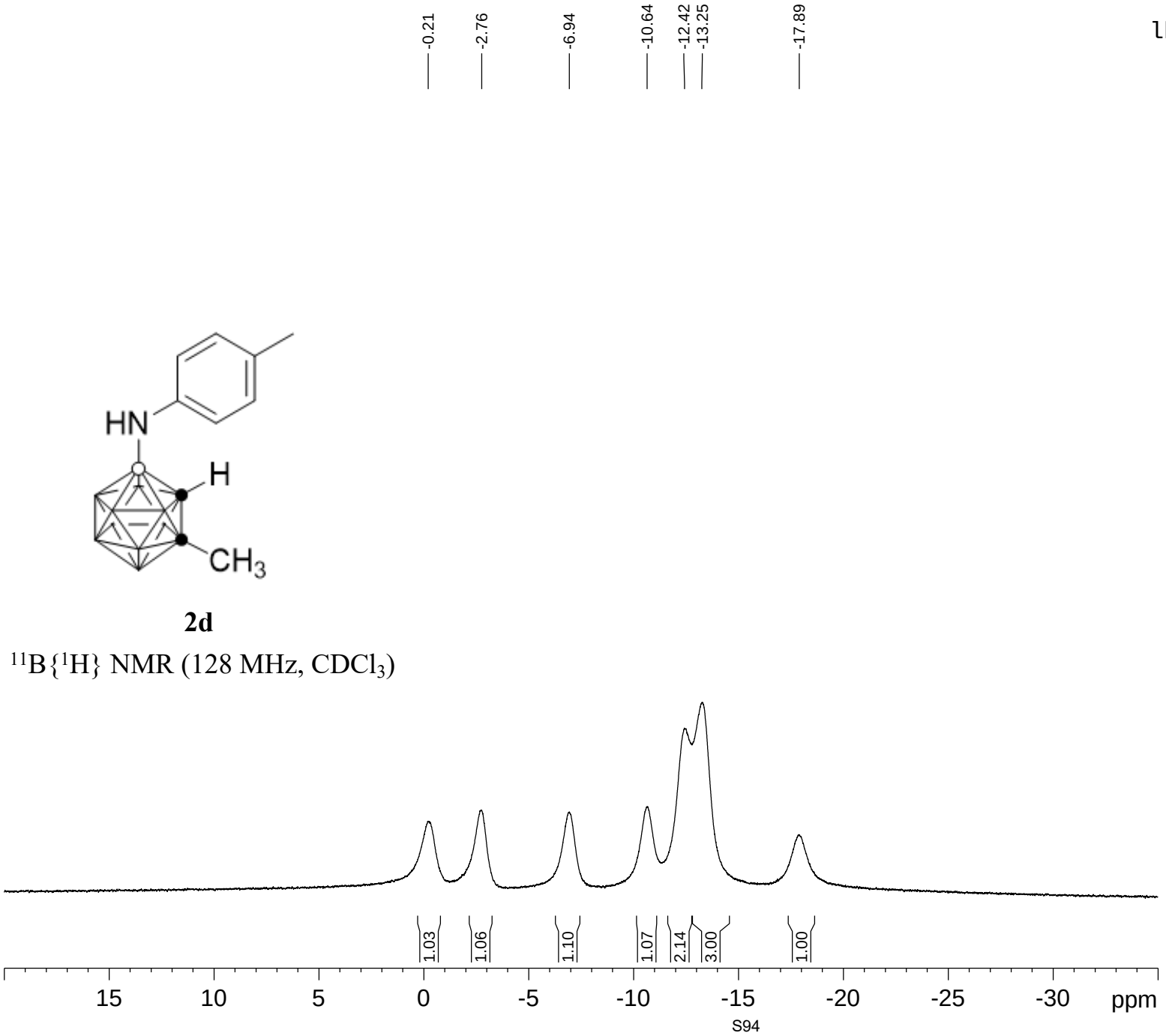



```
Current Data Parameters
NAME      lhr-C-0513-phnhCH3-CDCl3
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20161101
Time              10.08 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   100
DS                     4
SWH            29761.904 Hz
FIDRES          0.454131 Hz
AQ             1.1010048 sec
RG                   161
DW                16.800 usec
DE                 6.50 usec
TE                296.8 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           100.6379014 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

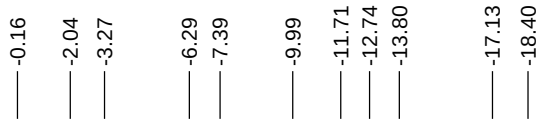
lhr-B-0513-phnhCH3-CDCl3

2d

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)

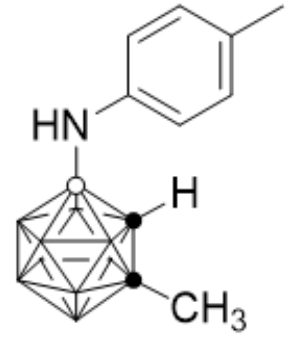



```
Current Data Parameters
NAME      lhr-B-0513-phnhCH3-CDCl3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161101
Time                9.51 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG             zgdc
TD                 65536
SOLVENT             C6D6
NS                    12
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                   287
DW                20.800 usec
DE                 6.50 usec
TE                296.7 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
```



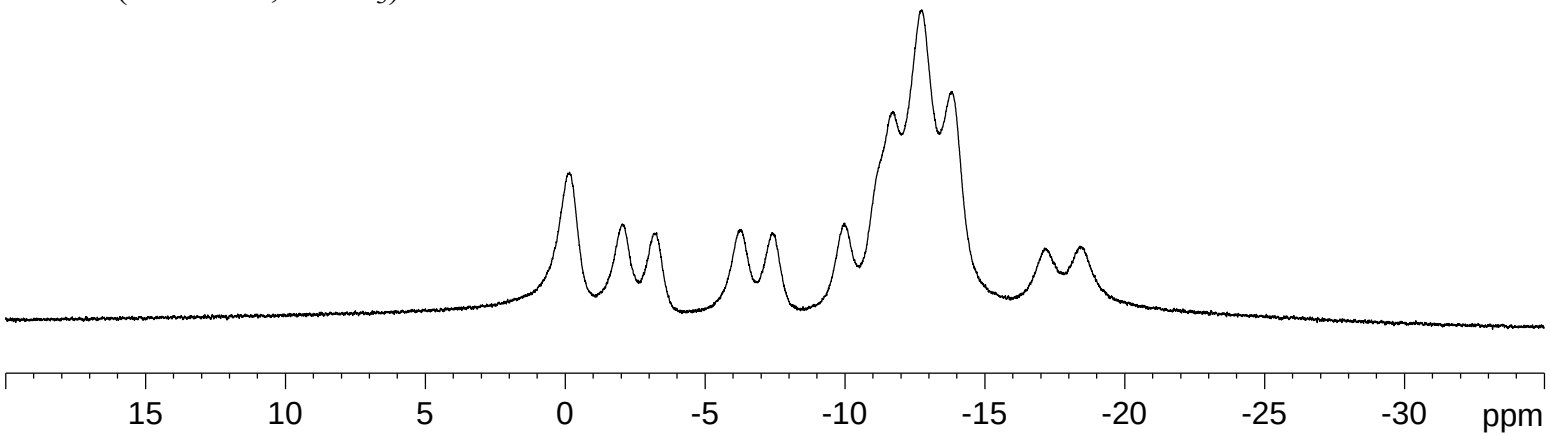
lhr-B-0513-phnhCH3-CDCl3(C)

```
Current Data Parameters
NAME      lhr-B-0513-phnhCH3-CDCl3(C)
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20161101
Time                9.52 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG                zg
TD                  65536
SOLVENT              C6D6
NS                     16
DS                      2
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                    287
DW                20.800 usec
DE                 6.50 usec
TE                296.5 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           128.4097504 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



2d

^{11}B NMR (128 MHz, $CDCl_3$)



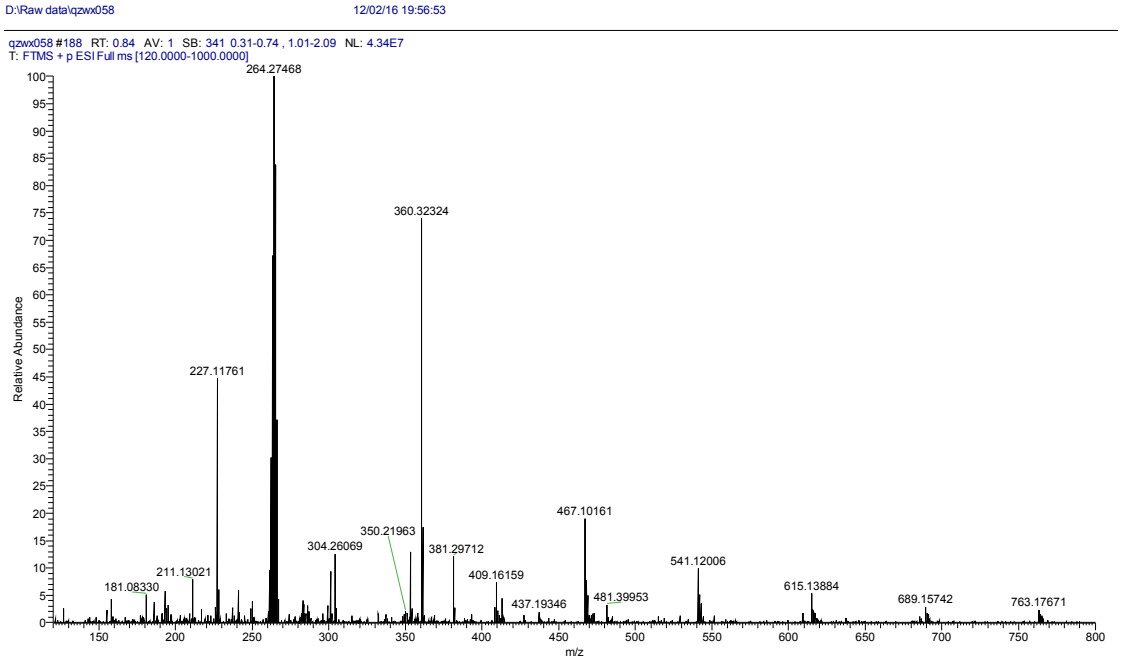
Thermo QEFMS Analysis Report

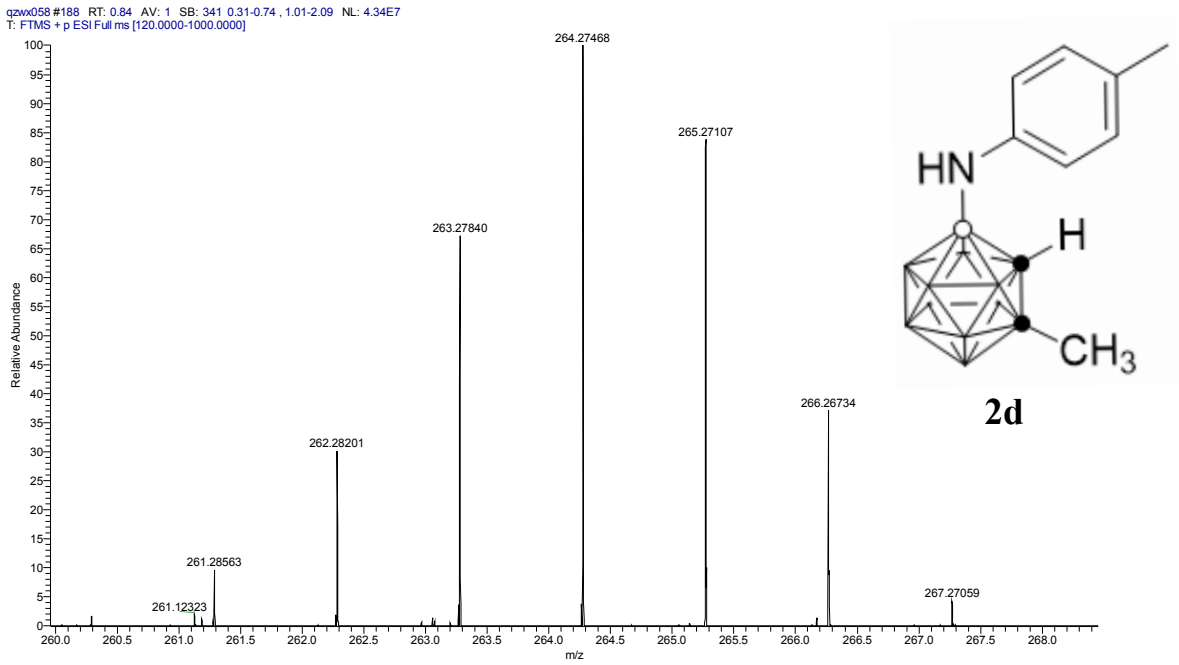
Analysis Info

Sample Name :	Lhr-513	Reference No.:	Qzwx058
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.5kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{10}H_{21}B_{10}N$
Experimental Mass $[M+H]^+$:	264.27468
Theoretical Mass $[M+H]^+$:	264.27500
Error (ppm) :	1.2

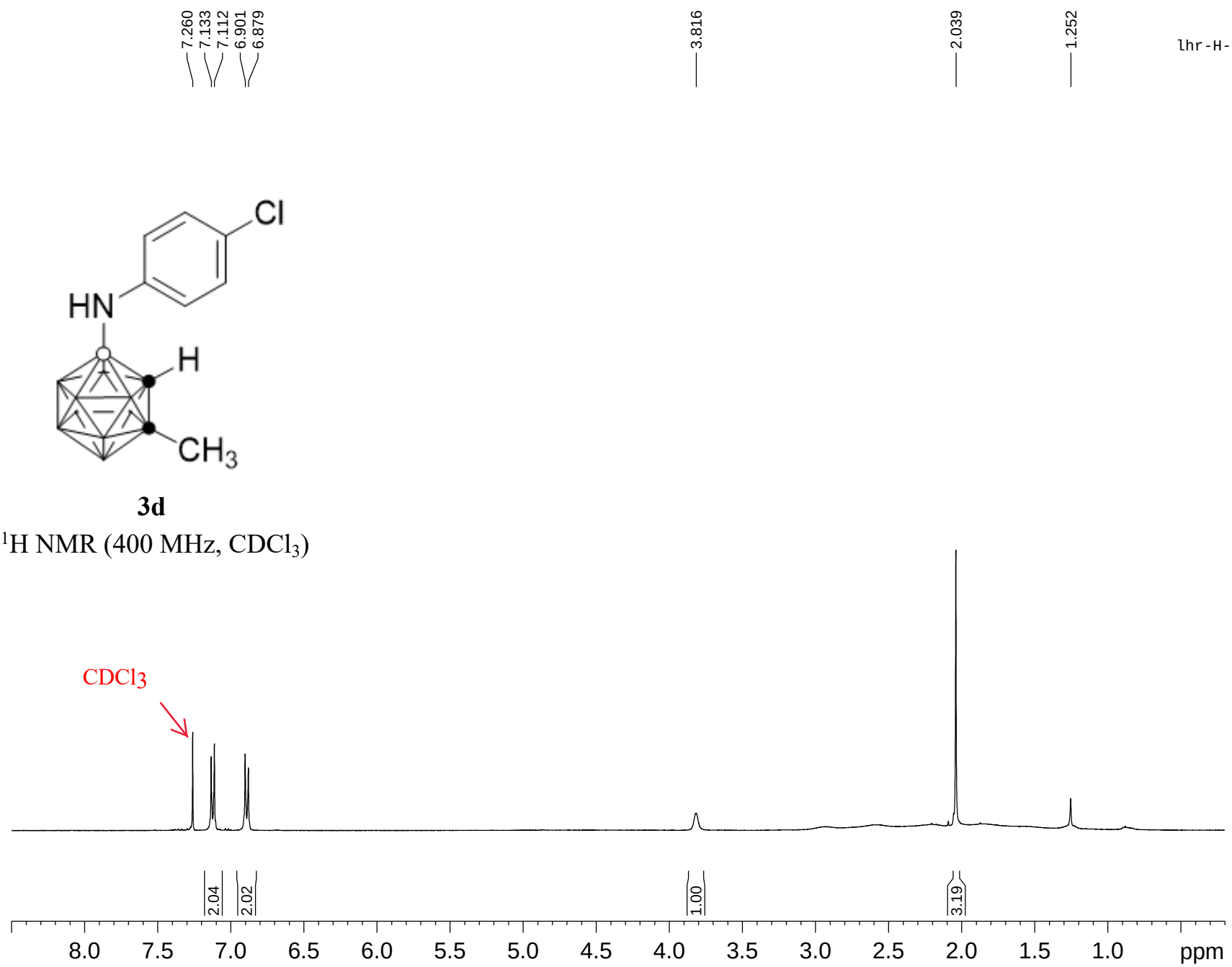




2d

3d

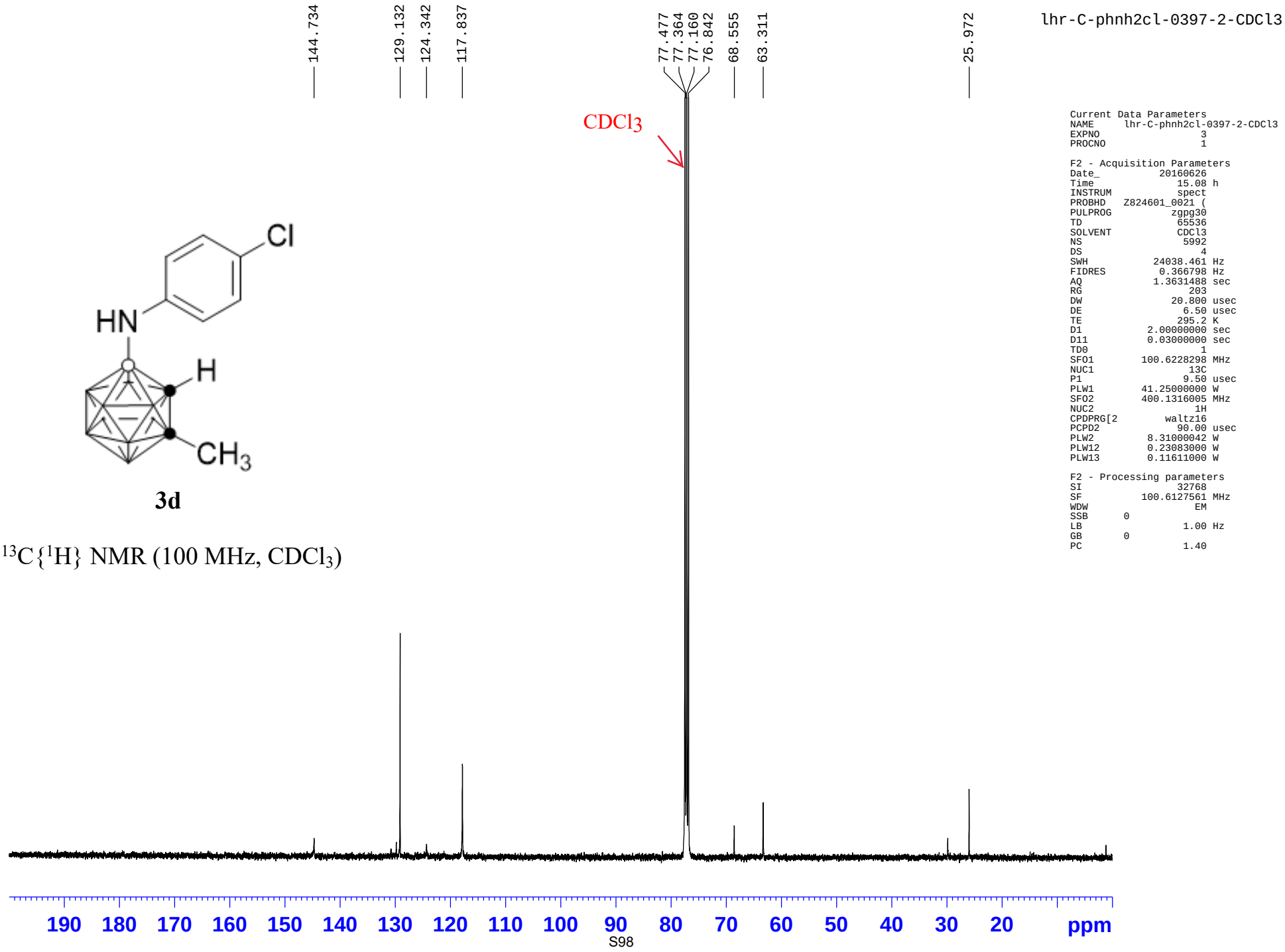
^{1}H NMR (400 MHz, $CDCl_3$)



Current Data Parameters
NAME lhr-H-0397-2-clphnh-CDC
EXPNO 1
PROCNO 1

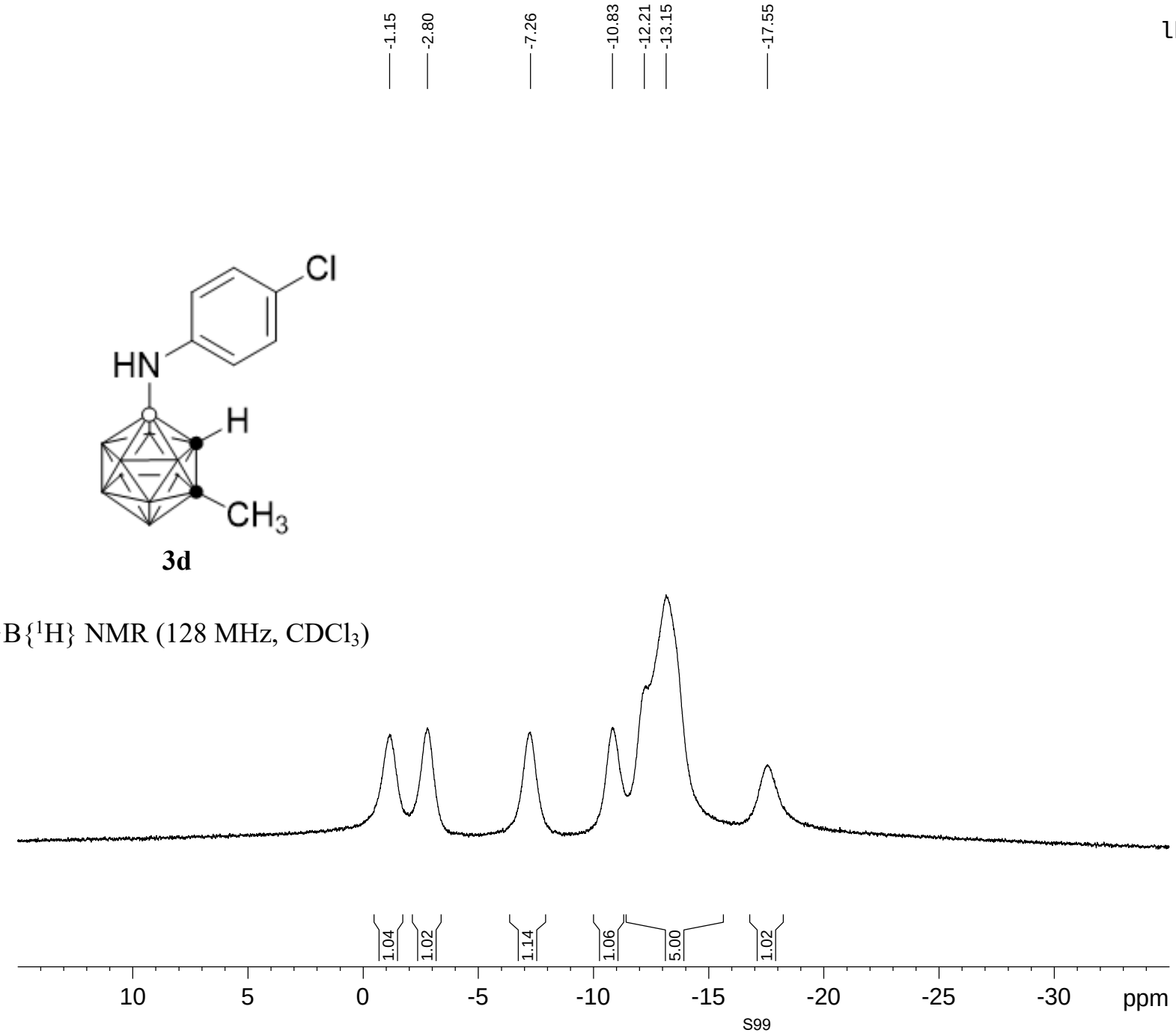
F2 - Acquisition Parameters
Date_ 20160622
Time 14.38 h
INSTRUM spect
PROBHD Z108618_0257 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 128
DW 62.400 usec
DE 6.50 usec
TE 294.7 K
D1 1.00000000 sec
TD0 1
SFO1 400.2324714 MHz
NUC1 1H
P1 12.80 usec
PLW1 13.56000042 W

F2 - Processing parameters
SI 65536
SF 400.2300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



3d

$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)



3d

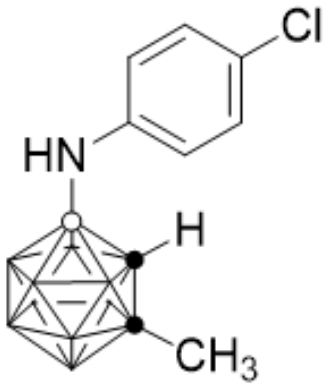
$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)

lhr-B-0397-2-clphnh-CDCl3

```
Current Data Parameters
NAME      lhr-B-0397-2-clphnh-CDCl3
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20160622
Time              14.48 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                    20
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631488 sec
RG                   287
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
```

lhr-B-0397-2-clphnh-CDCl3(C)

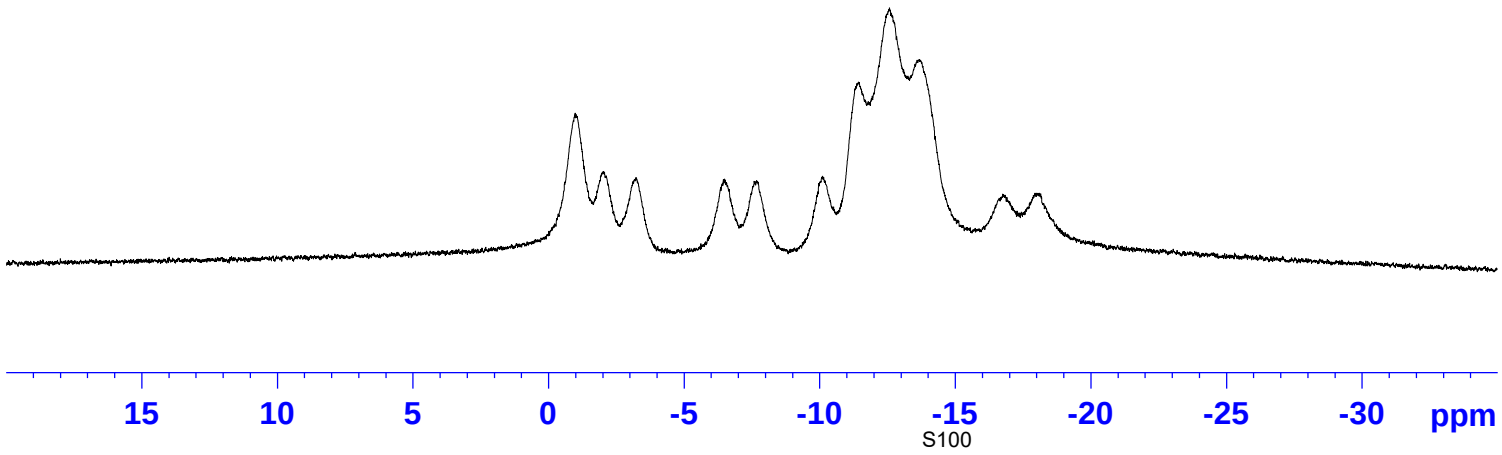
-0.99 -2.04 -3.23 -6.49 -7.68 -10.11 -11.43 -12.58 -13.69 -16.76 -18.09



3d

^{11}B NMR (128 MHz, $CDCl_3$)

```
Current Data Parameters
NAME      lhr-B-0397-2-clphnh-CDCl3(C)
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20160622
Time              14.50 h
INSTRUM           spect
PROBHD   Z108618_0257 (
PULPROG             zg
TD                65536
SOLVENT           CDCl3
NS                   20
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                294.7 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
```



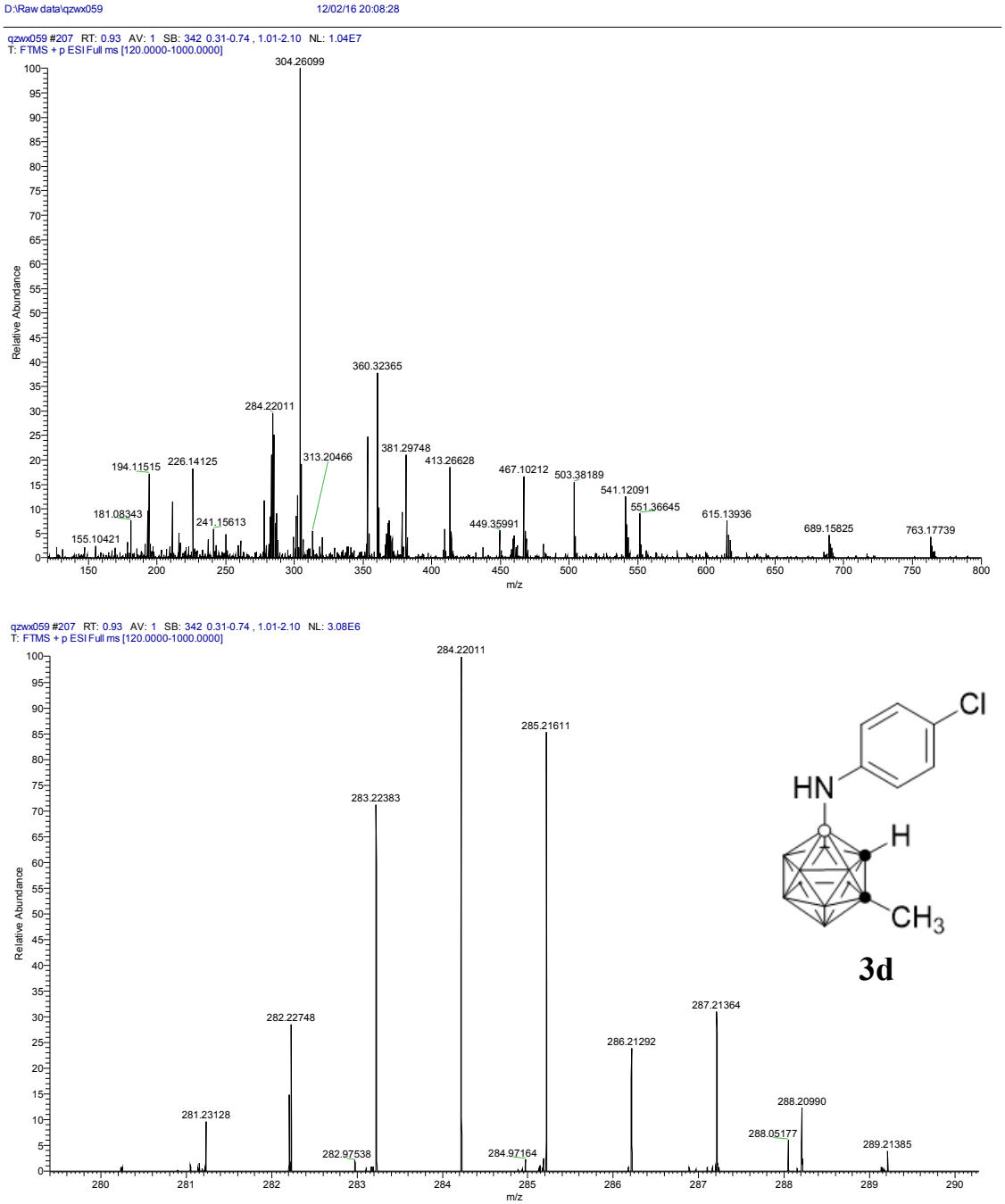
Thermo QEFMS Analysis Report

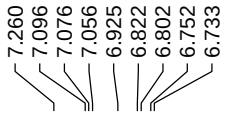
Analysis Info

Sample Name :	Lhr-397	Reference No.:	Qzwx059
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.5kV, by LC, with sheath gas		

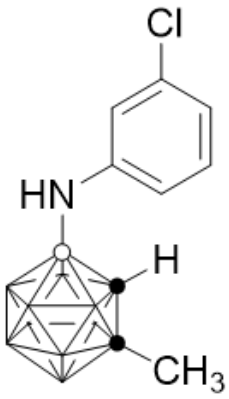
Accurate Mass Measurement

Molecular formula :	$C_9H_{18}B_{10}ClN$
Experimental Mass $[M+H]^+$:	284.22011
Theoretical Mass $[M+H]^+$:	284.22042
Error (ppm) :	1.0



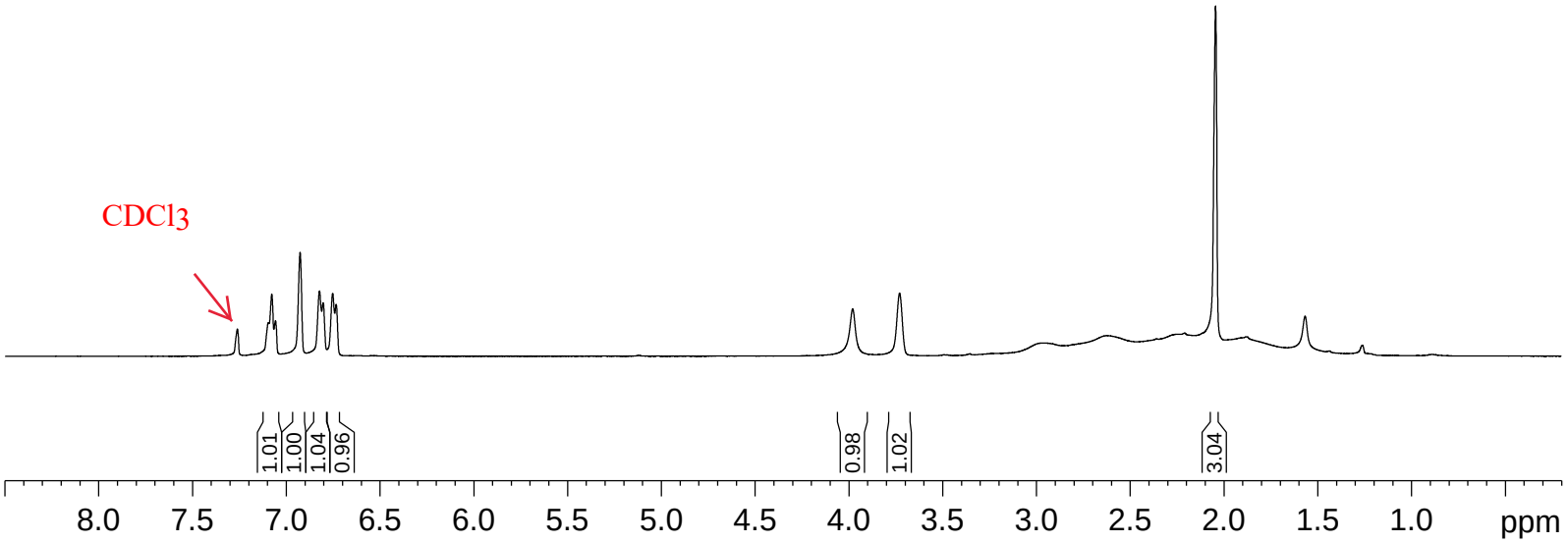


lhr-H-0547-pn3cl-CD



4d

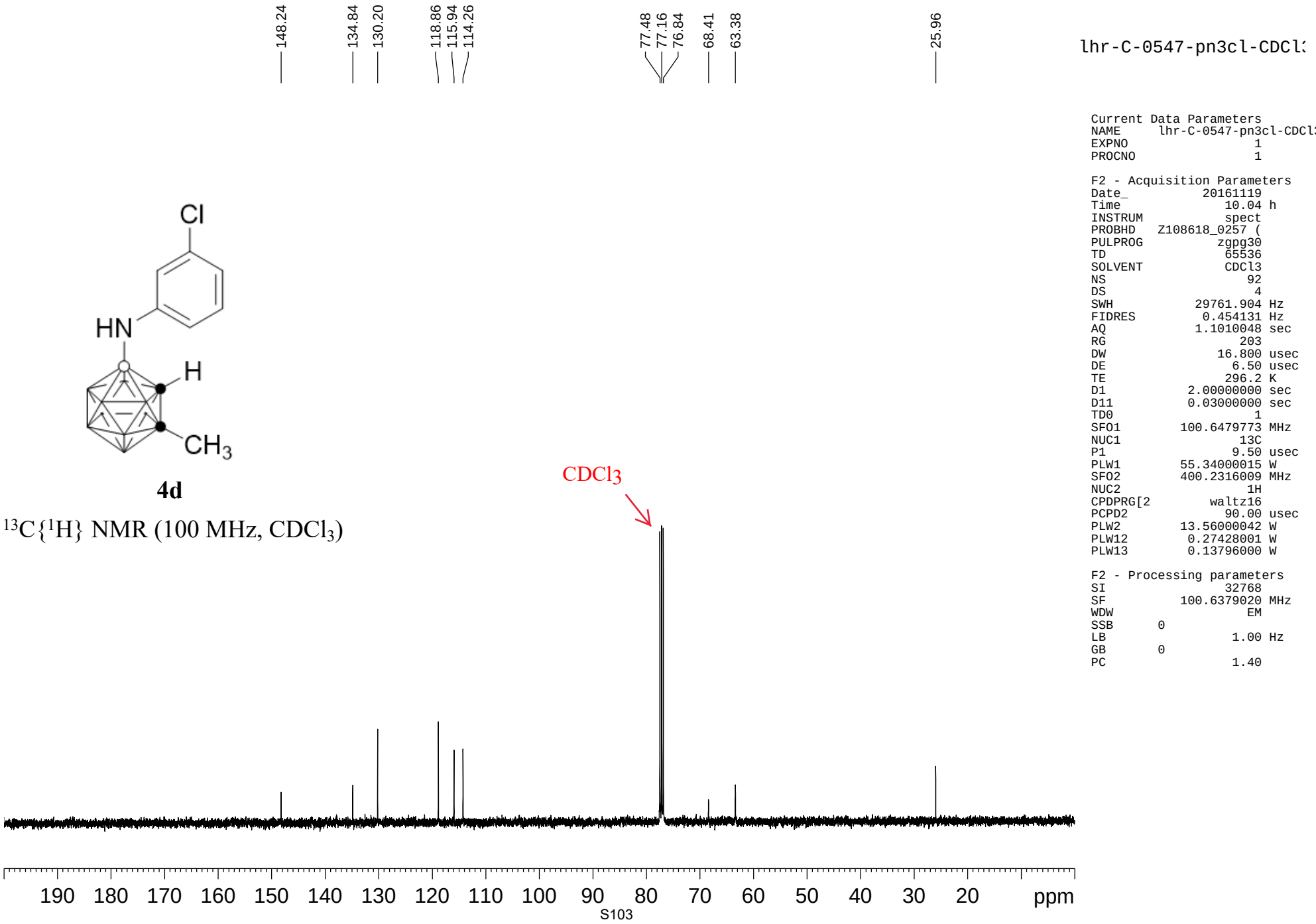
^{1}H NMR (400 MHz, $CDCl_3$)

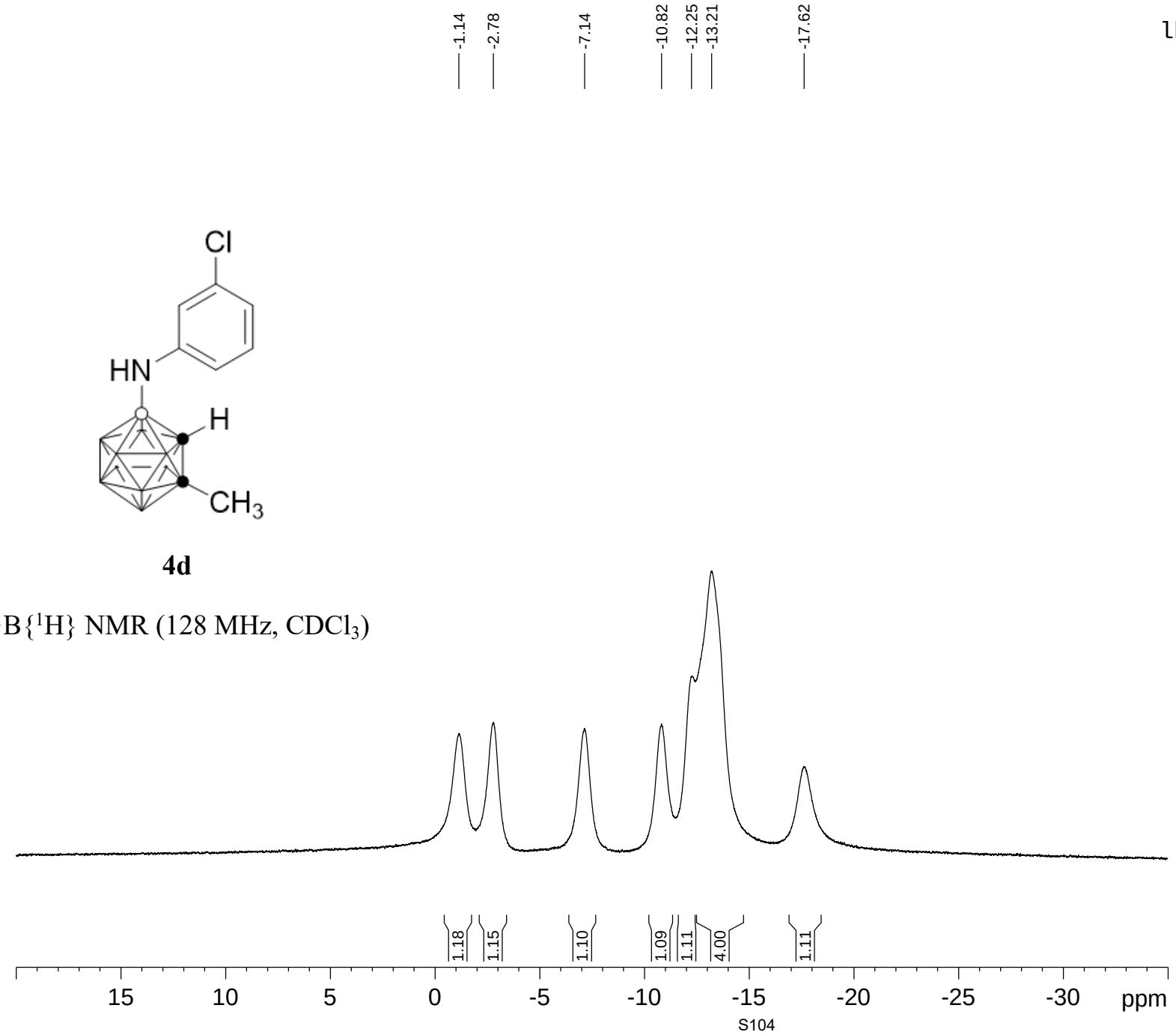


Current Data Parameters
NAME lhr-H-0547-pn3cl-CD
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161119
Time 9.58 h
INSTRUM spect
PROBHD Z108618_0257 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 11
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 64
DW 62.400 usec
DE 6.50 usec
TE 296.0 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 400.2300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


148.24
134.84
130.20
118.86
115.94
114.26
77.48
77.16
76.84
68.41
63.38
25.96
lhr-C-0547-pn3cl-CDCl3
Current Data Parameters
NAME lhr-C-0547-pn3cl-CDCl3
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161119
Time 10.04 h
INSTRUM spect
PROBHD Z108618_0257 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 92
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 296.2 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 100.6379020 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Cl
HN
H
CH3
4d
13C{1H} NMR (100 MHz, CDCl3)
CDCl3
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

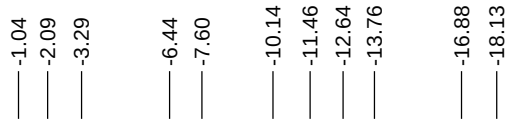


4d

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)

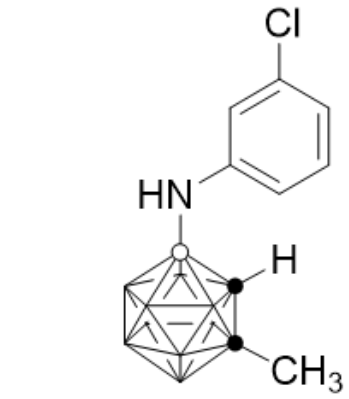
lhr-B-0547-pn3cl-CDCl3

```
Current Data Parameters
NAME      lhr-B-0547-pn3cl-CDCl3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161118
Time               20.26 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                    12
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                   287
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
```



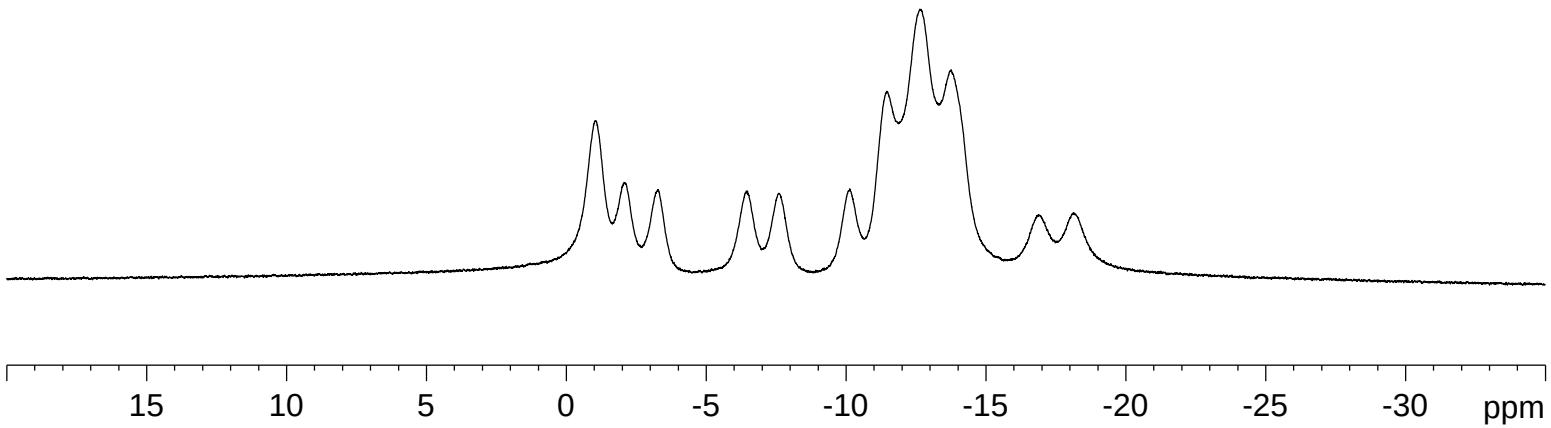
lhr-B-0547-pn3cl-CDCl3(C)

```
Current Data Parameters
NAME      lhr-B-0547-pn3cl-CDCl3(C)
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20161118
Time              20.28 h
INSTRUM           spect
PROBHD   Z108618_0257 (
PULPROG             zg
TD                65536
SOLVENT           CDCl3
NS                   18
DS                    2
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                  203
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          128.4097504 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



4d

^{11}B NMR (128 MHz, $CDCl_3$)



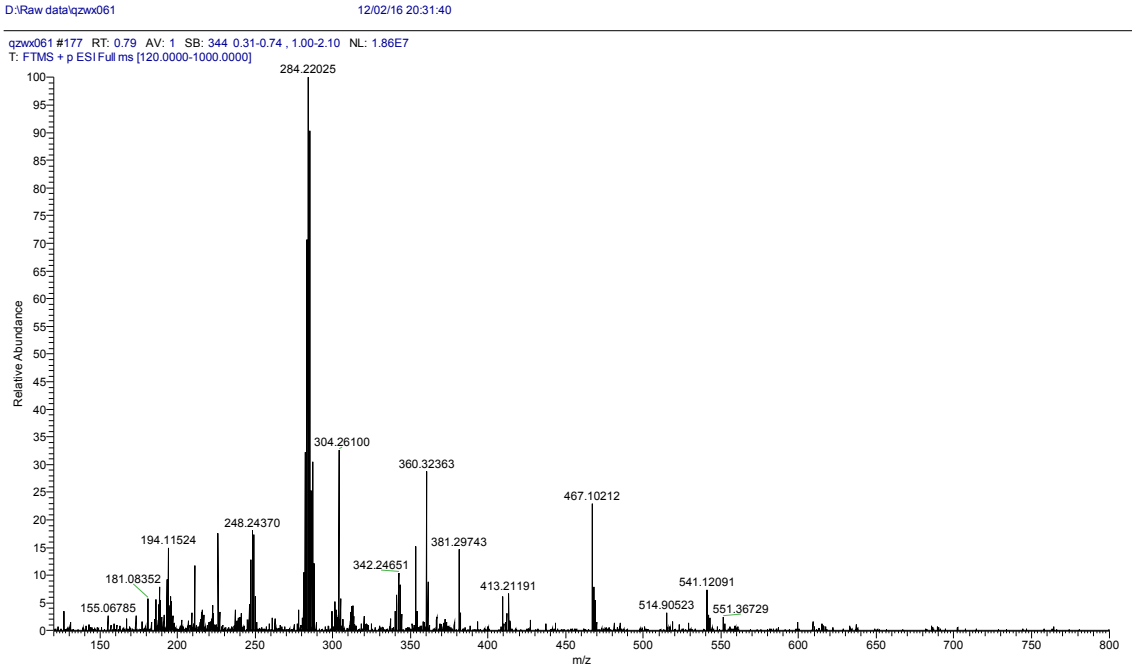
Thermo QEFMS Analysis Report

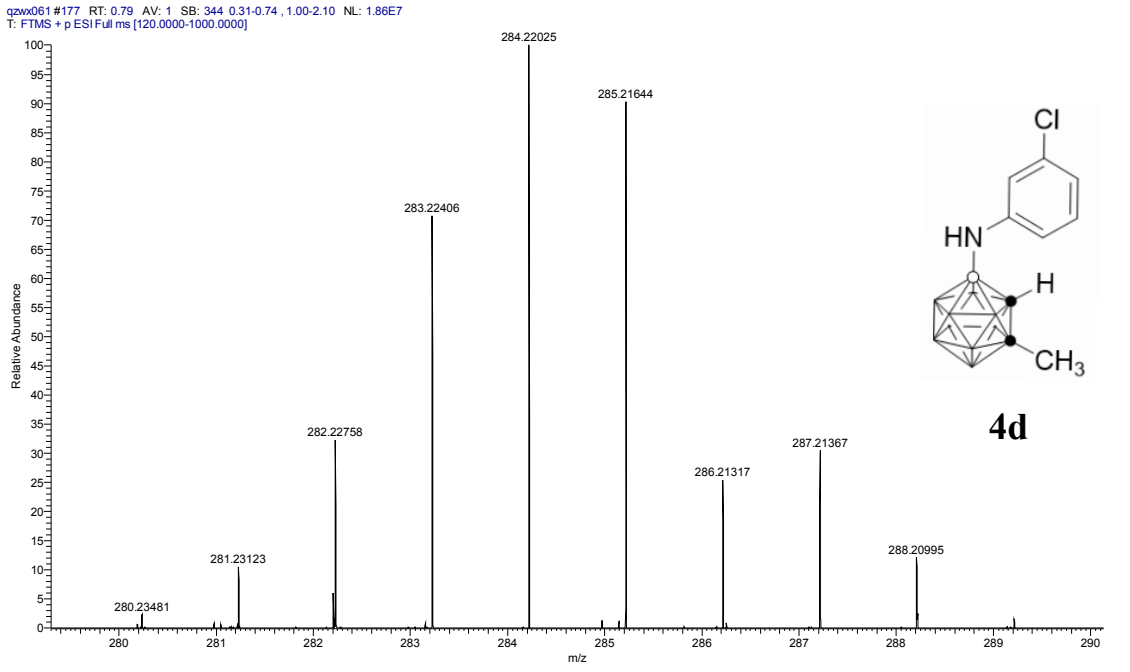
Analysis Info

Sample Name :	Lhr-547	Reference No.:	Qzwx061
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.5kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_9H_{18}B_{10}ClN$
Experimental Mass $[M+H]^+$:	284.22025
Theoretical Mass $[M+H]^+$:	284.22042
Error (ppm) :	0.5



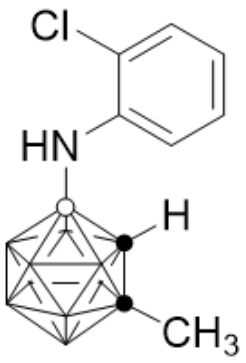


4d

lhr-H-0530-phnh-oCl-CDCl3

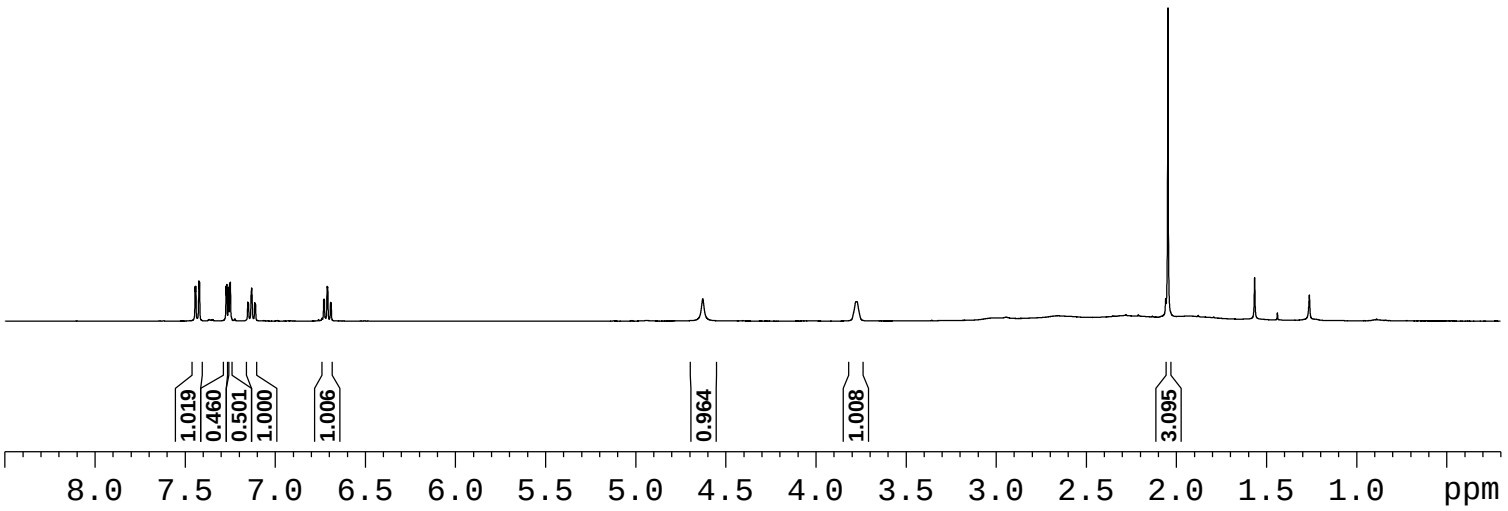
Bruker Advance III 400

```
Current Data Parameters
NAME      lhr-H-0530-phnh-oCl-CDCl3
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20161105
Time              12.44 h
INSTRUM             spect
PROBHD   Z824601_0021 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                   161
DW                62.400 usec
DE                 6.50 usec
TE                294.5 K
D1             1.00000000 sec
TD0                    1
SFO1         400.1324708 MHz
NUC1                  1H
P1                15.00 usec
PLW1          8.31000042 W

F2 - Processing parameters
SI                 65536
SF           400.1300097 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



5d

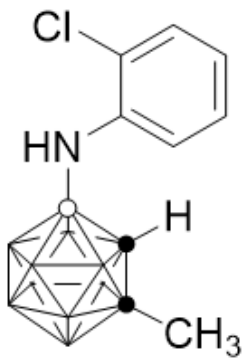
^{1}H NMR (400 MHz, $CDCl_3$)



143.319
129.280
127.597
121.134
119.171
115.371
77.478
77.160
76.843
68.373
63.716
25.951

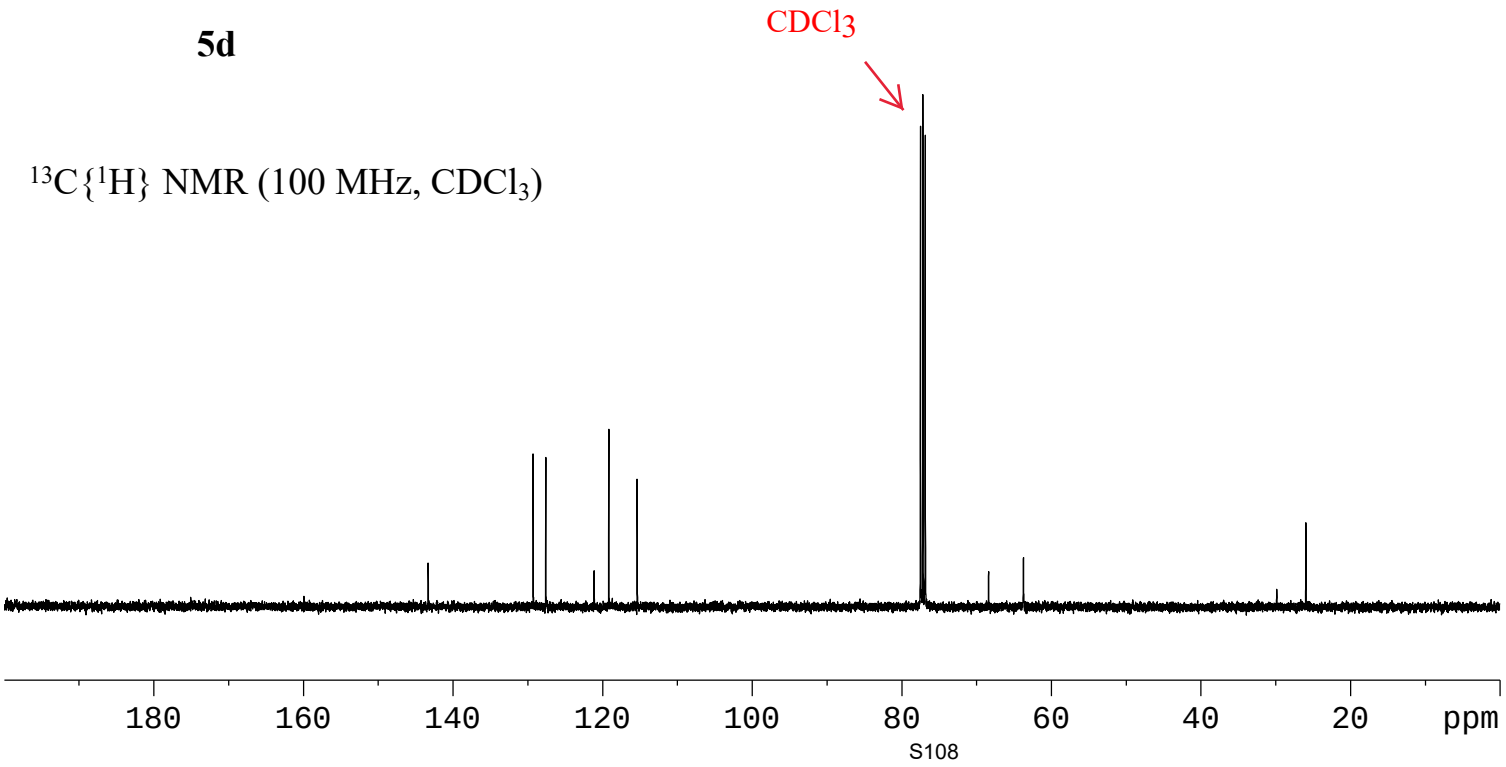
lhr-C-0530-phnh-oCl-CDCl3

Bruker Advance III 400



5d

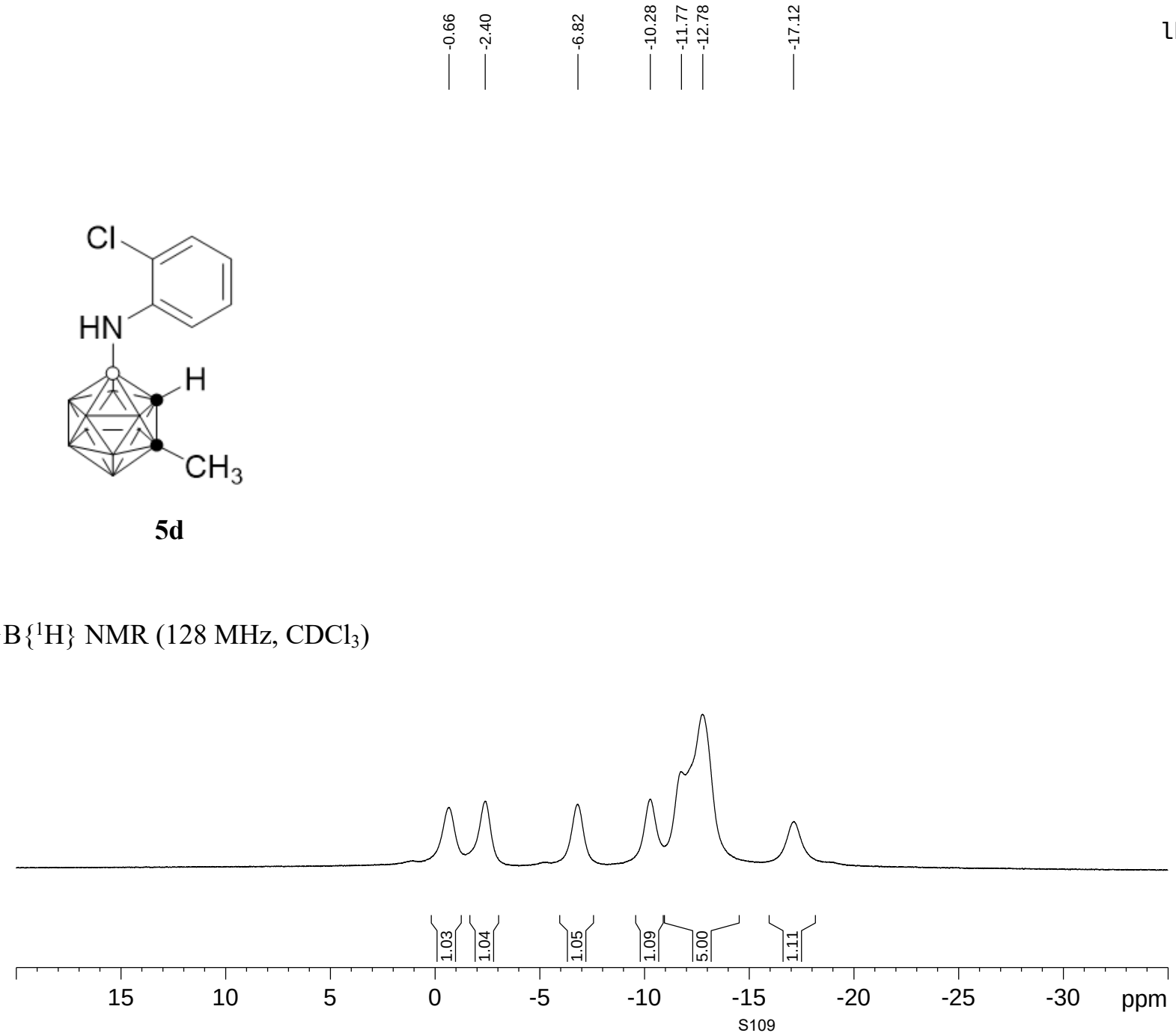
$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      lhr-C-0530-phnh-oCl-CDCl3
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20161105
Time              12.55 h
INSTRUM            spect
PROBHD   Z824601_0021 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                  166
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                  203
DW               20.800 usec
DE                6.50 usec
TE               294.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P1                9.50 usec
PLW1        41.25000000 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2            90.00 usec
PLW2         8.31000042 W
PLW12        0.23083000 W
PLW13        0.11611000 W

F2 - Processing parameters
SI                32768
SF          100.6127576 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

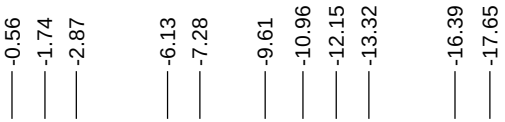
5d

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)



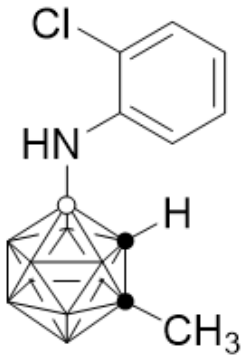
lhr-B-0530-phnh-oCl-CDCl3

```
Current Data Parameters
NAME      lhr-B-0530-phnh-oCl-CDCl3
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20161105
Time              13.34 h
INSTRUM           spect
PROBHD   Z108618_0257 (
PULPROG           zgdc
TD                65536
SOLVENT           CDCl3
NS                   12
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                  203
DW                20.800 usec
DE                 6.50 usec
TE                296.2 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
```



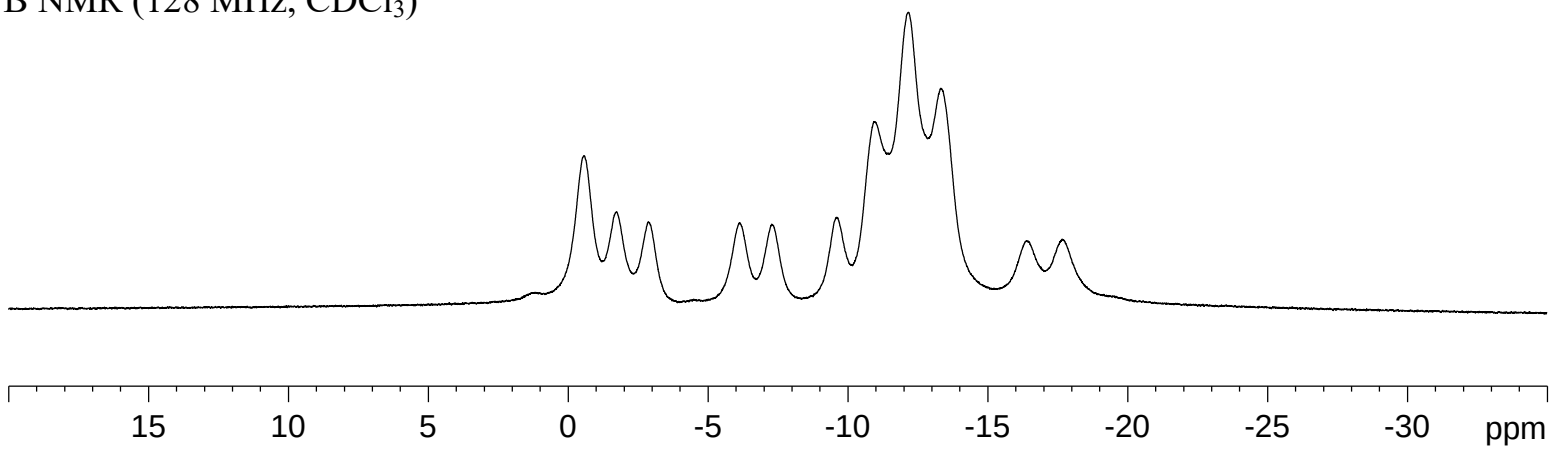
lhr-B-0530-phnh-oCl-CDCl3(C)

```
Current Data Parameters
NAME     lhr-B-0530-phnh-oCl-CDCl3(C)
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20161105
Time              13.36 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG               zg
TD                 65536
SOLVENT            CDCl3
NS                    18
DS                     2
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                   203
DW                20.800 usec
DE                 6.50 usec
TE                296.2 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           128.4097504 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



5d

^{11}B NMR (128 MHz, $CDCl_3$)



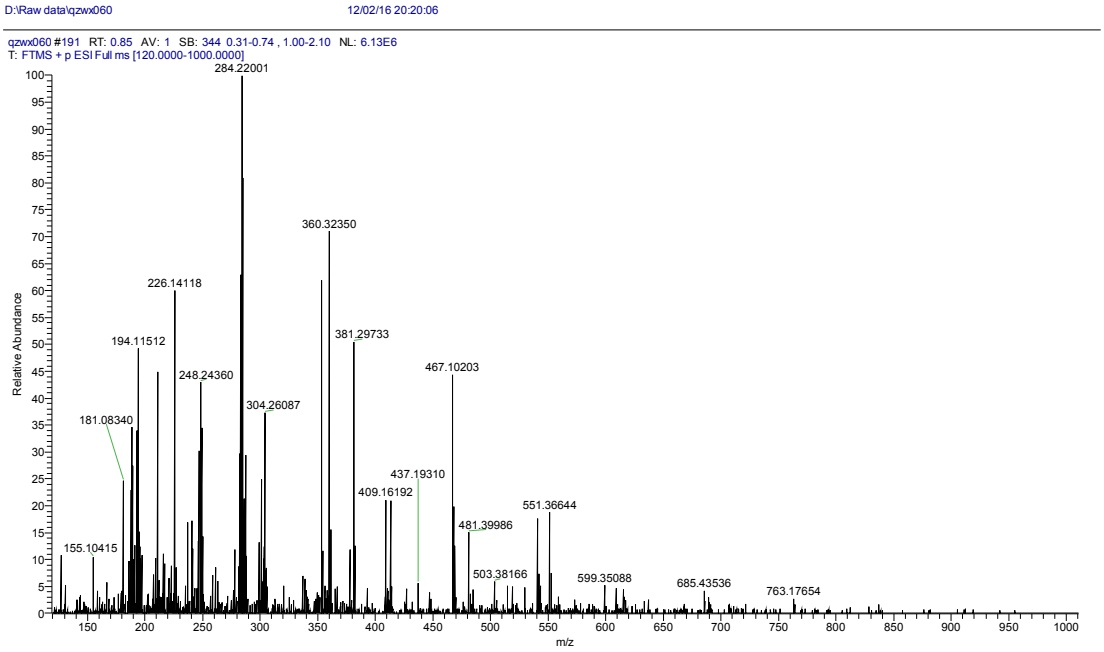
Thermo QEFMS Analysis Report

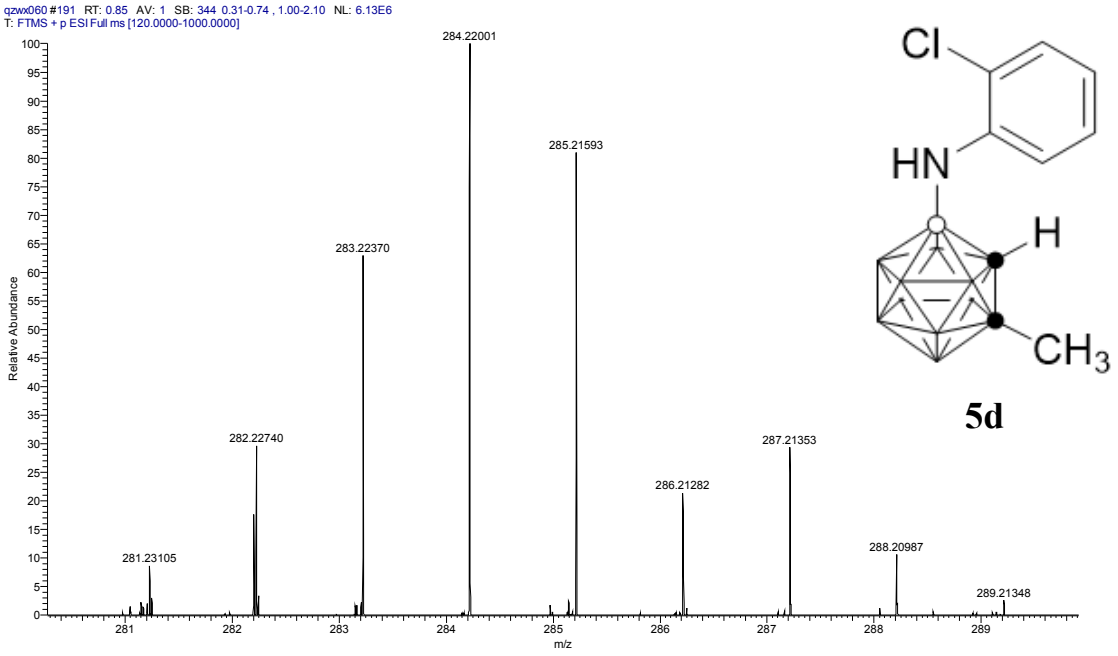
Analysis Info

Sample Name :	Lhr-530	Reference No.:	Qzwx060
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.5kV, by LC, with sheath gas		

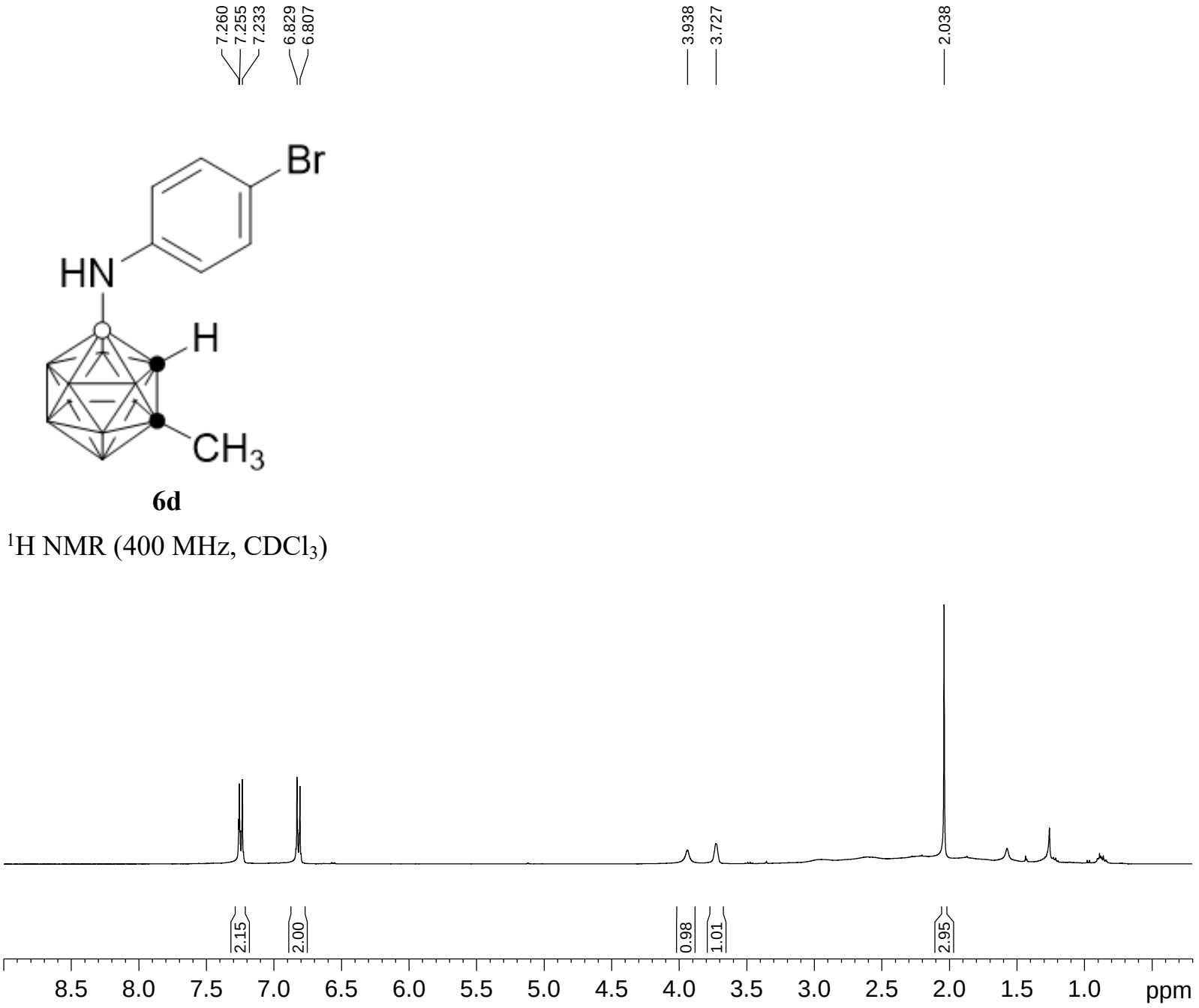
Accurate Mass Measurement

Molecular formula :	$C_9H_{18}B_{10}ClN$
Experimental Mass $[M+H]^+$:	284.22001
Theoretical Mass $[M+H]^+$:	284.22042
Error (ppm) :	1.4





5d



6d

^{1}H NMR (400 MHz, $CDCl_3$)

lhr-H-0529-phnhBr-C

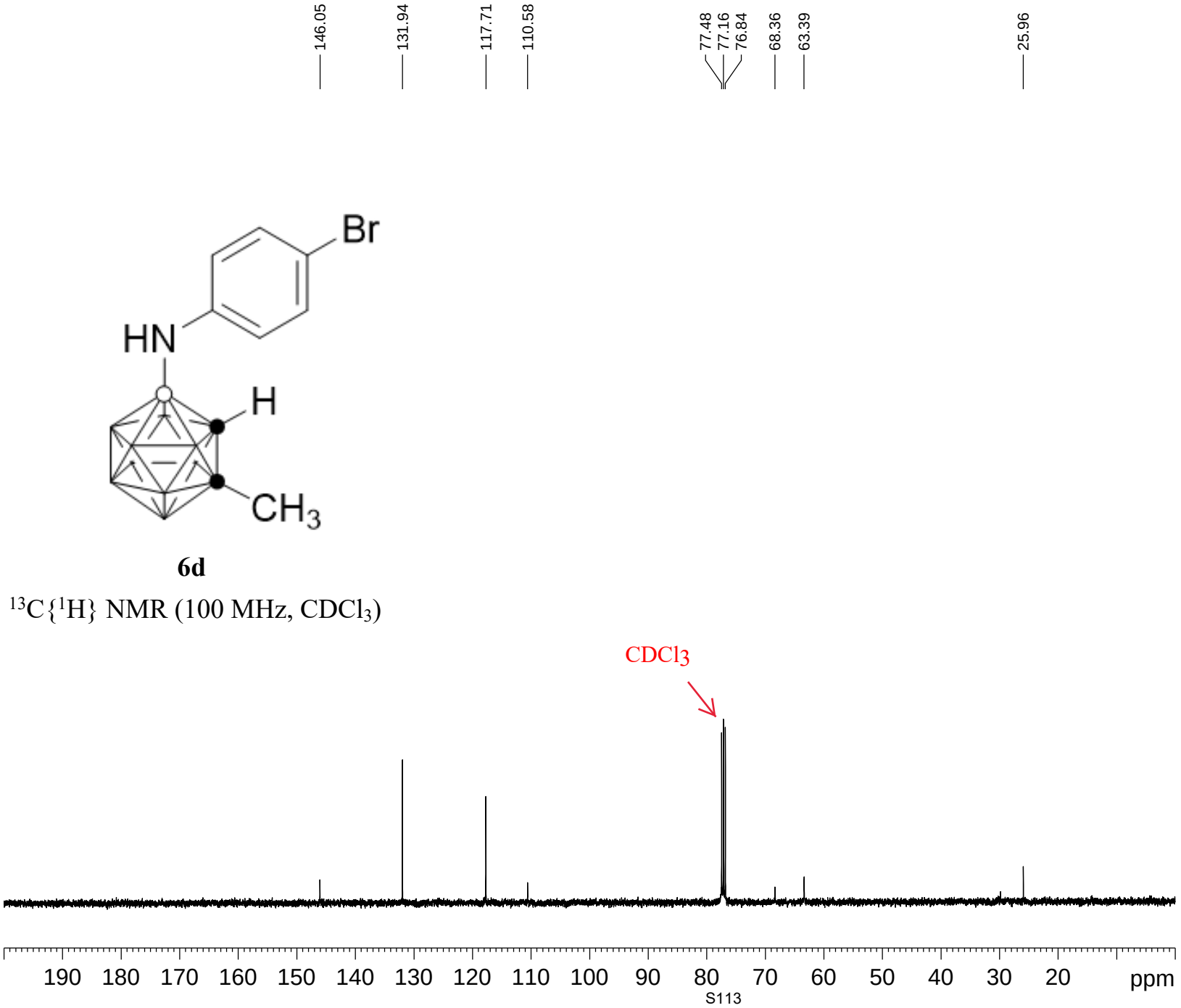
Current Data Parameters
NAME lhr-H-0529-phnhBr-CD
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161107
Time 15.57 h
INSTRUM spect
PROBHD Z108618_0257 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
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 296.0 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 400.2300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

6d

$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)

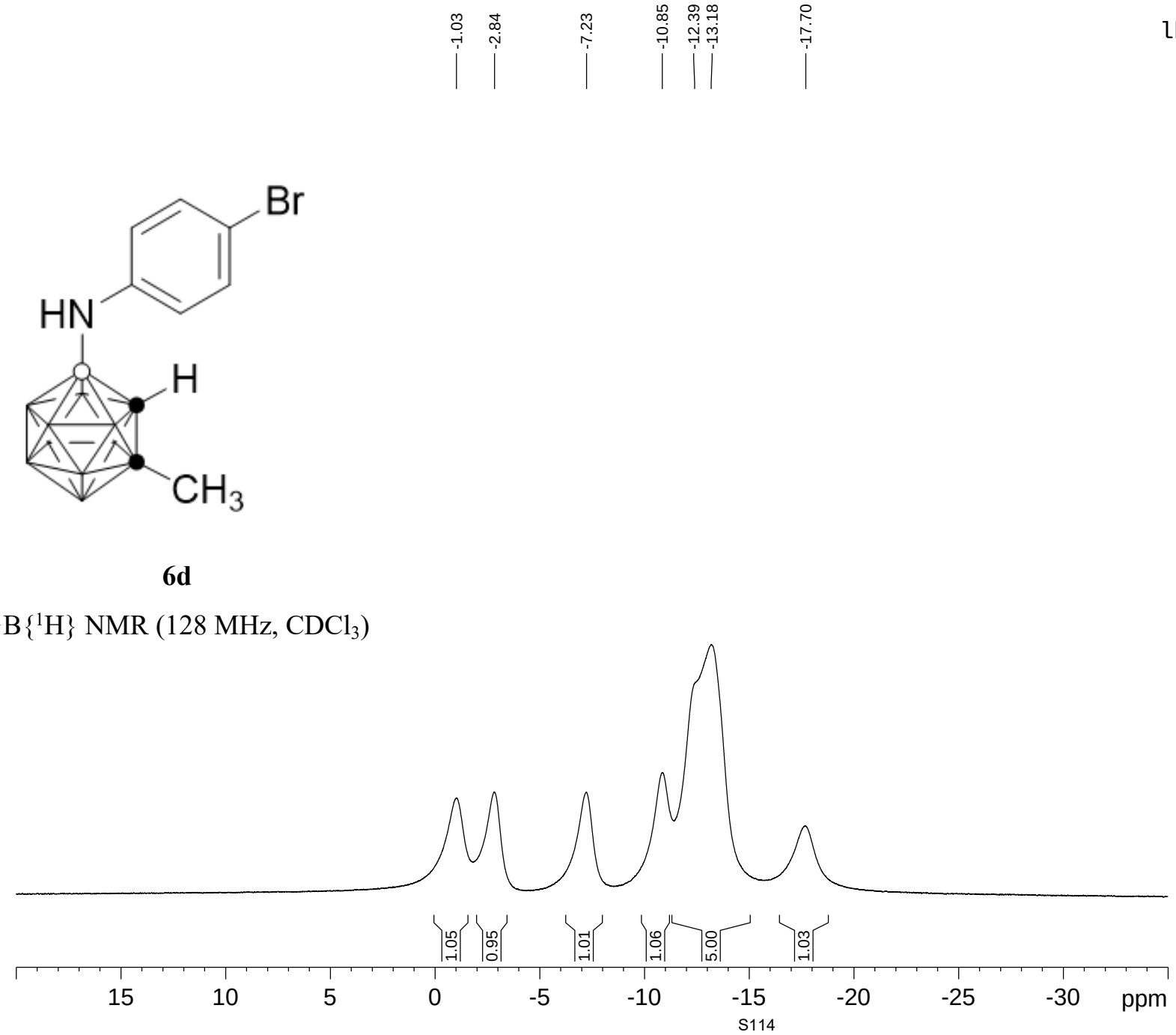


lhr-C-0529-phnhBr-(

```
Current Data Parameters
NAME      lhr-C-0529-phnhBr-CDCl3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161107
Time               16.19 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    100
DS                      4
SWH             29761.904 Hz
FIDRES           0.454131 Hz
AQ              1.1010048 sec
RG                    161
DW                 16.800 usec
DE                  6.50 usec
TE                 296.6 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            100.6379020 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

6d

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)

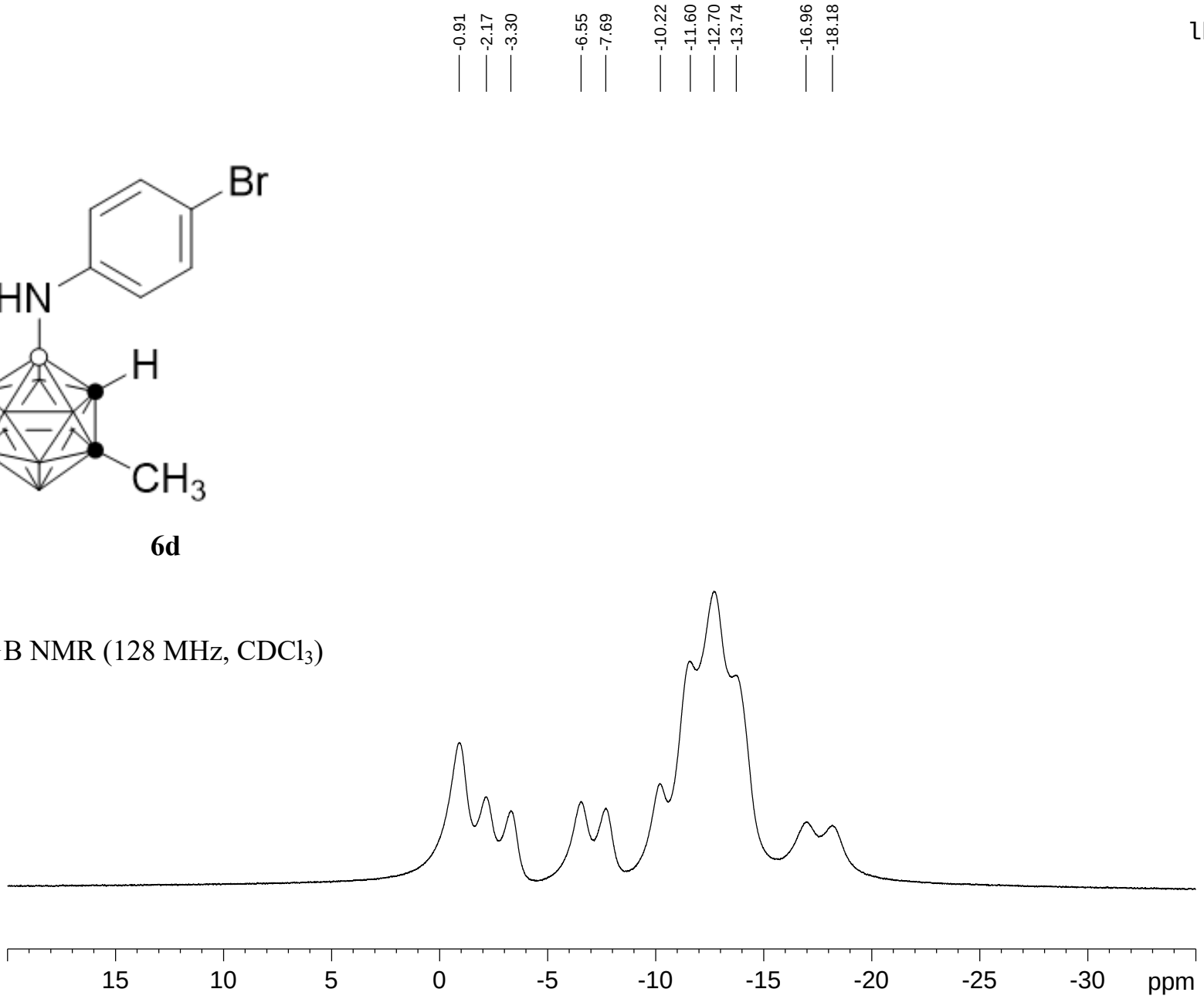


lhr-B-0529-phnhBr-CDCl3

```
Current Data Parameters
NAME      lhr-B-0529-phnhBr-CDCl3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161107
Time                9.30 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG             zgdc
TD                 65536
SOLVENT              THF
NS                    12
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                   203
DW                20.800 usec
DE                 6.50 usec
TE                296.4 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
```

6d

^{11}B NMR (128 MHz, $CDCl_3$)

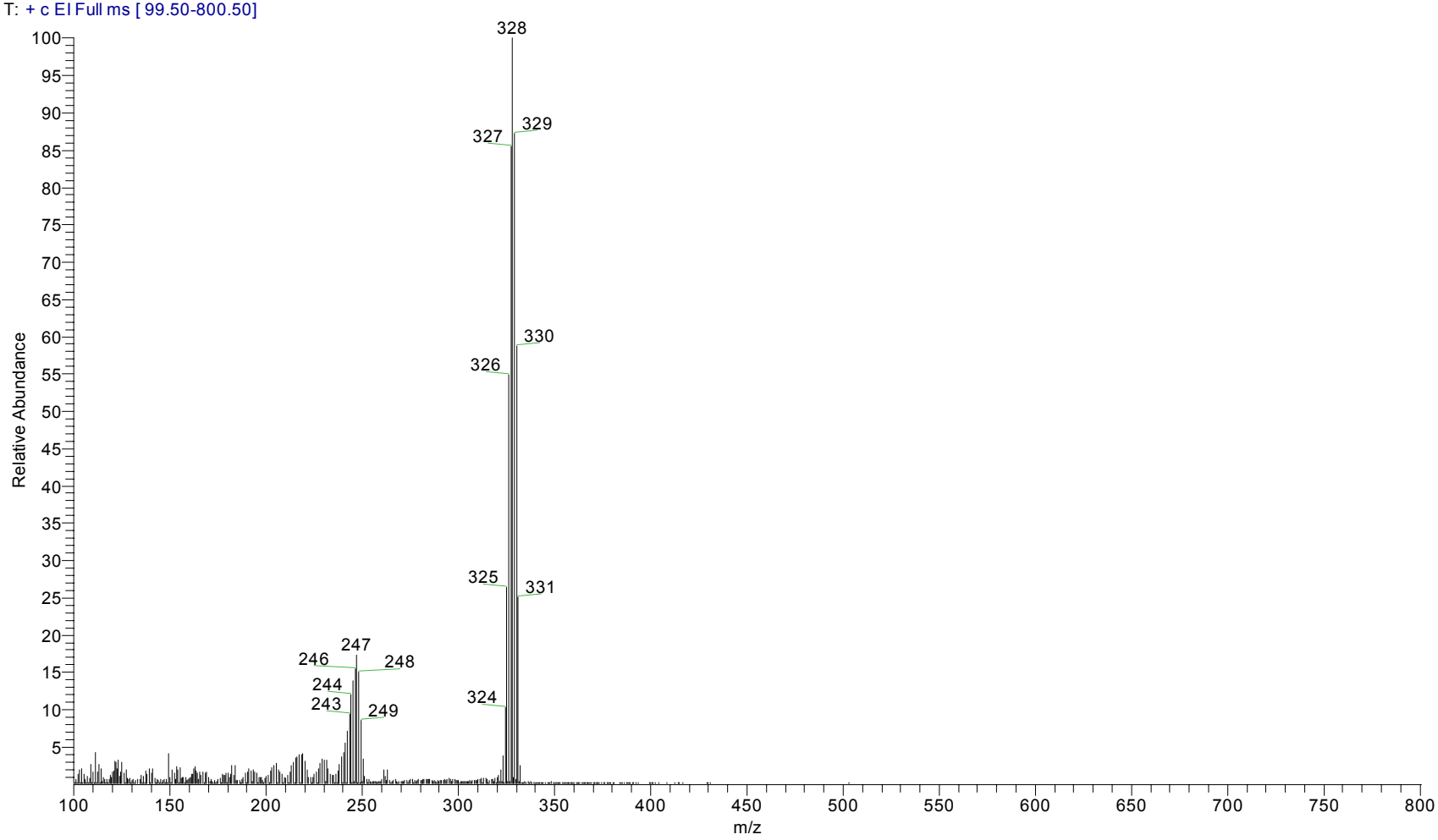


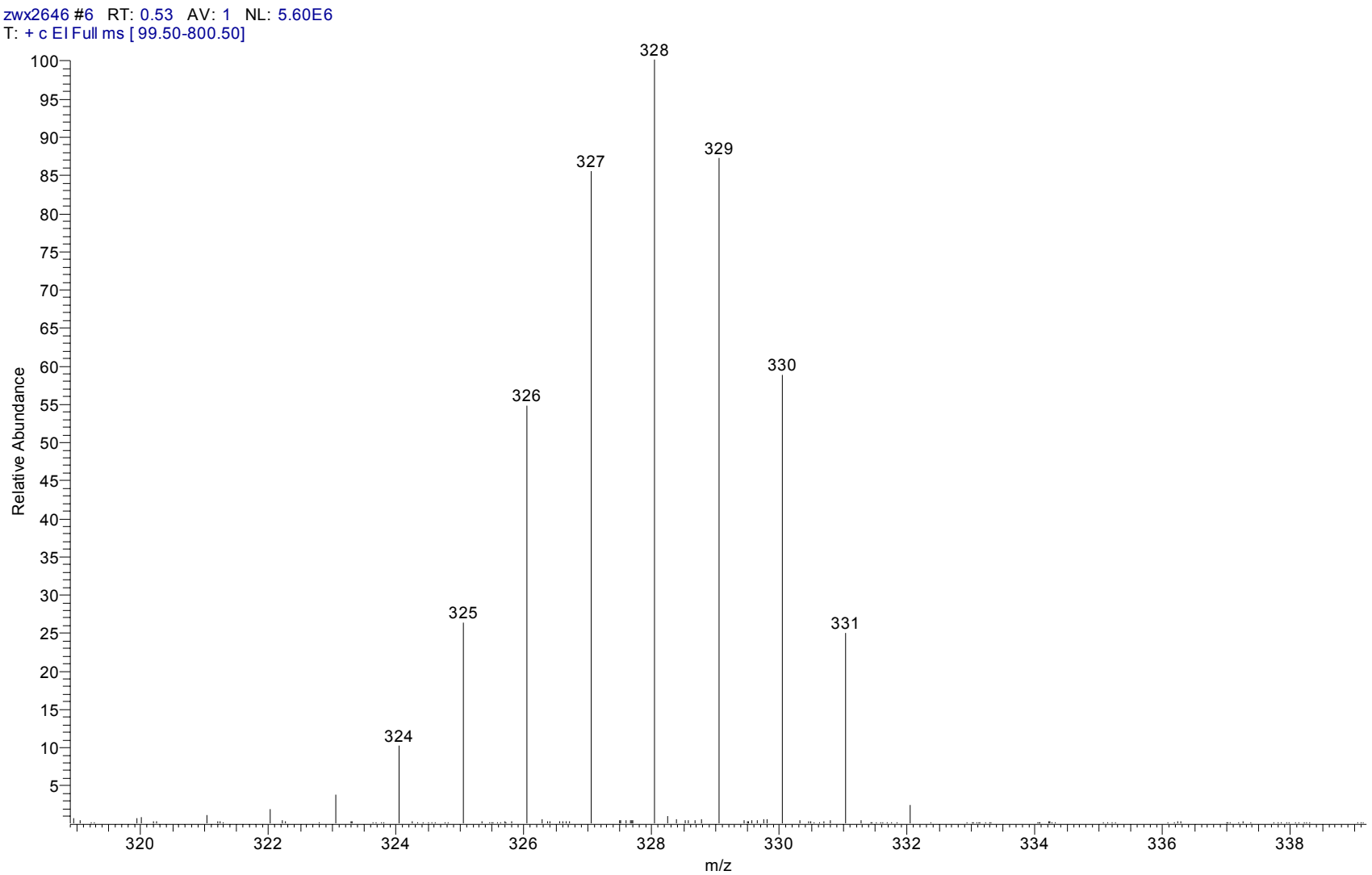
lhr-B-0529-phnhBr-CDCl3(C)

```
Current Data Parameters
NAME      lhr-B-0529-phnhBr-CDCl3(C)
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20161107
Time               9.31 h
INSTRUM           spect
PROBHD   Z108618_0257 (
PULPROG             zg
TD                65536
SOLVENT             THF
NS                   13
DS                    2
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                  203
DW               20.800 usec
DE                6.50 usec
TE               296.1 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          128.4097504 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

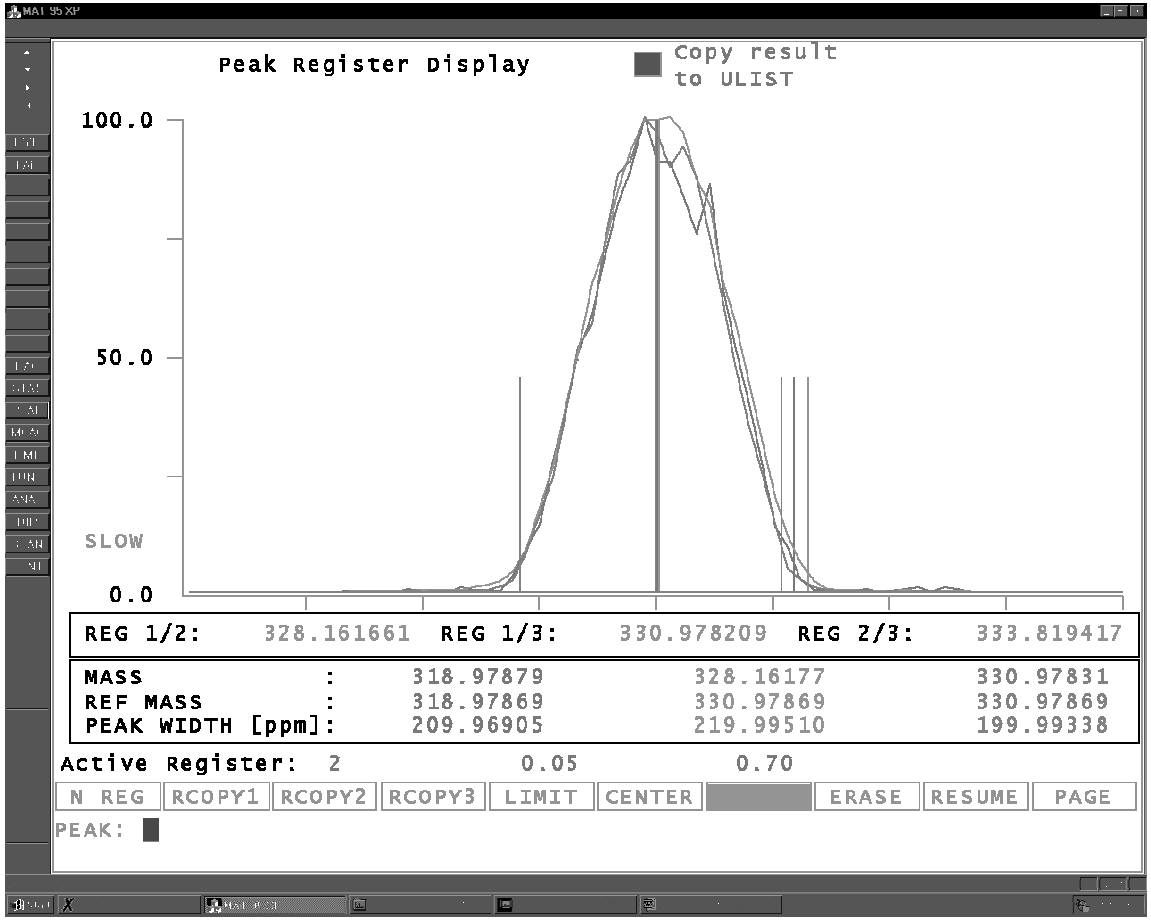
D:\MS_raw_data\zwx2646 03/09/17 04:41:51 PM lhr-0592
ei pos, 50eV, 0.7mA, unknown conc.

zwx2646 #6 RT: 0.53 AV: 1 NL: 5.60E6
T: + c EI Full ms [99.50-800.50]

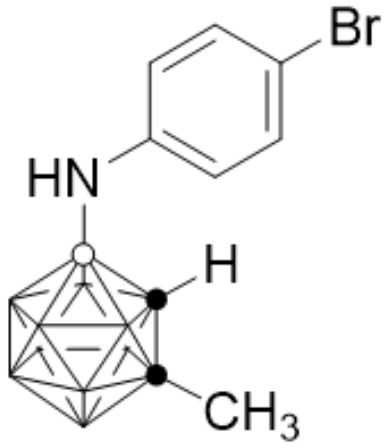



zwx2646 #6 RT: 0.53 AV: 1 NL: 5.60E6
T: + c EI Full ms [99.50-800.50]
Relative Abundance
m/z
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
320
322
324
326
328
330
332
334
336
338
324
325
326
327
328
329
330
331

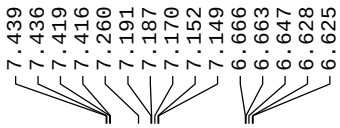
Accurate Mass Measurement



Molecular formula
$C_9H_{18}B_{10}BrN$
[M]+ (theoretical)
= 328.1611

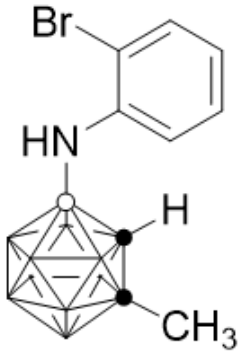


6d



lhr-H-0592-CDCl3

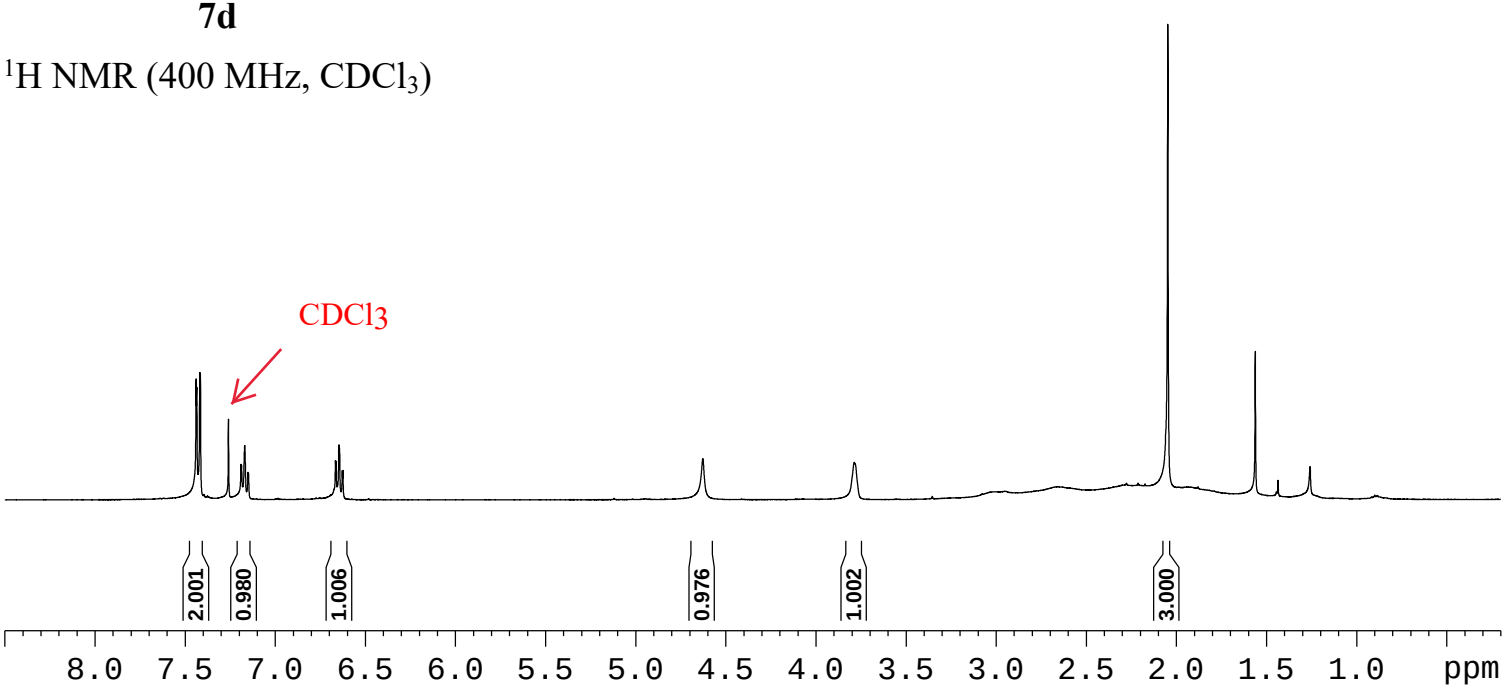
Bruker Advance III 400



7d

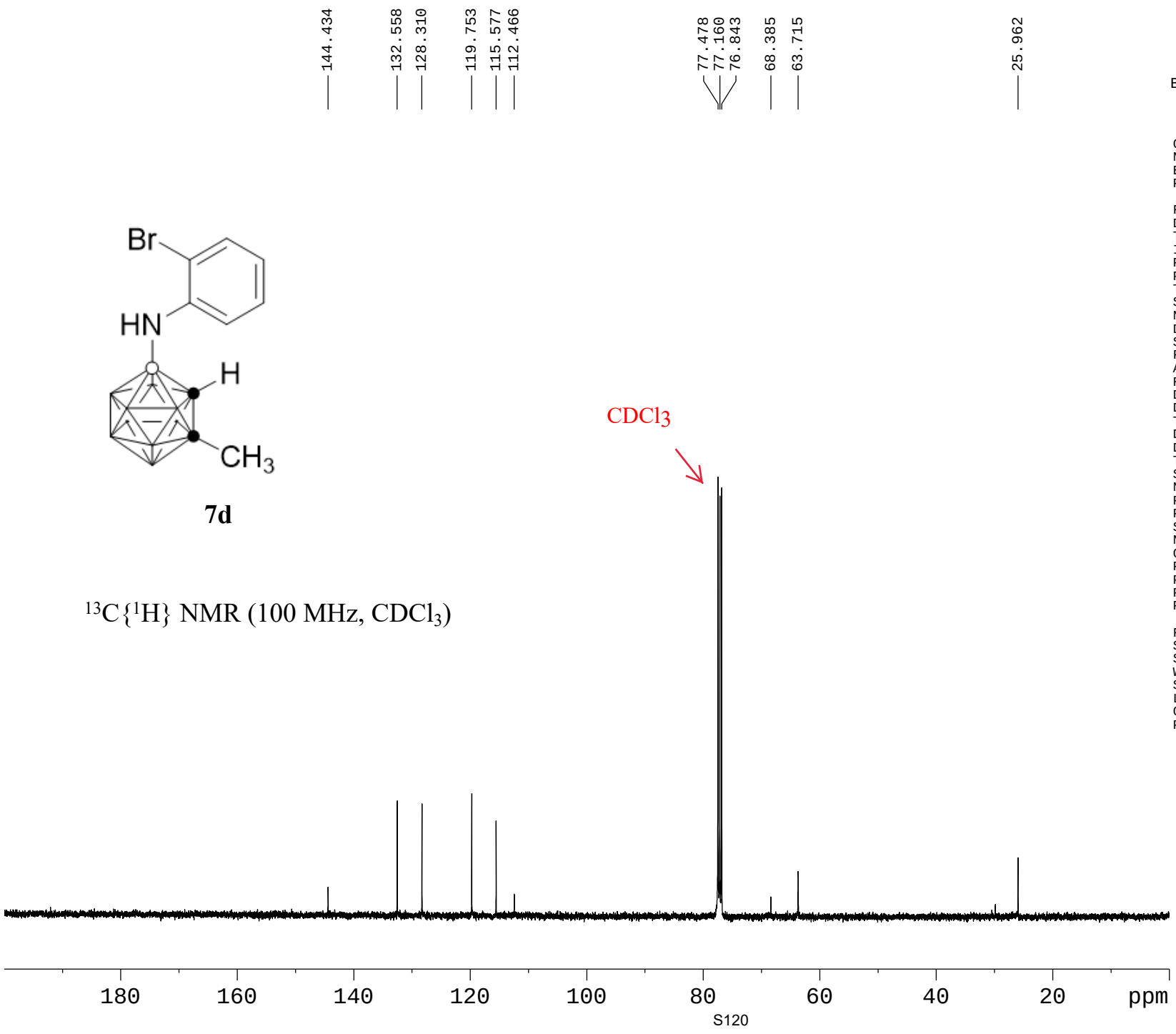
^{1}H NMR (400 MHz, $CDCl_3$)

```
Current Data Parameters
NAME      lhr-H-0592-CDCl3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170114
Time              10.53 h
INSTRUM             spect
PROBHD   Z824601_0021 (
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                295.0 K
D1           1.00000000 sec
TD0                    1
SFO1        400.1324708 MHz
NUC1                  1H
P1                15.00 usec
PLW1          8.31000042 W

F2 - Processing parameters
SI                 65536
SF          400.1300096 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



7d

$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)



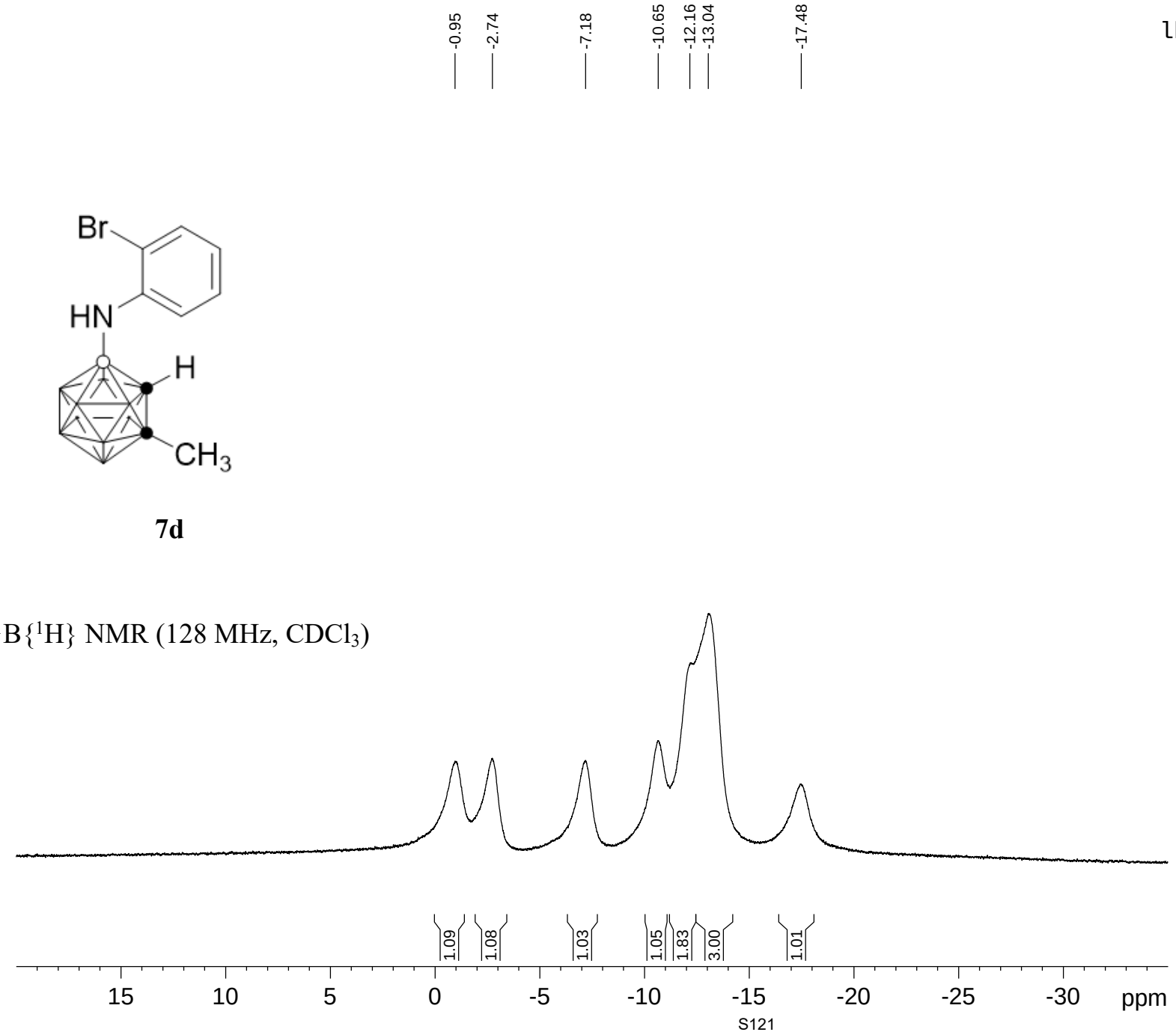
lhr-C-0592-CDCl3

Bruker Advance III 400

```
Current Data Parameters
NAME      lhr-C-0592-CDCl3
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20170114
Time               14.04 h
INSTRUM             spect
PROBHD    Z824601_0021 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   738
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631488 sec
RG                   203
DW                20.800 usec
DE                 6.50 usec
TE                295.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1
SFO1        100.6228298 MHz
NUC1                13C
P1                 9.50 usec
PLW1        41.25000000 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         8.31000042 W
PLW12        0.23083000 W
PLW13        0.11611000 W

F2 - Processing parameters
SI                 32768
SF          100.6127564 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

7d

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)

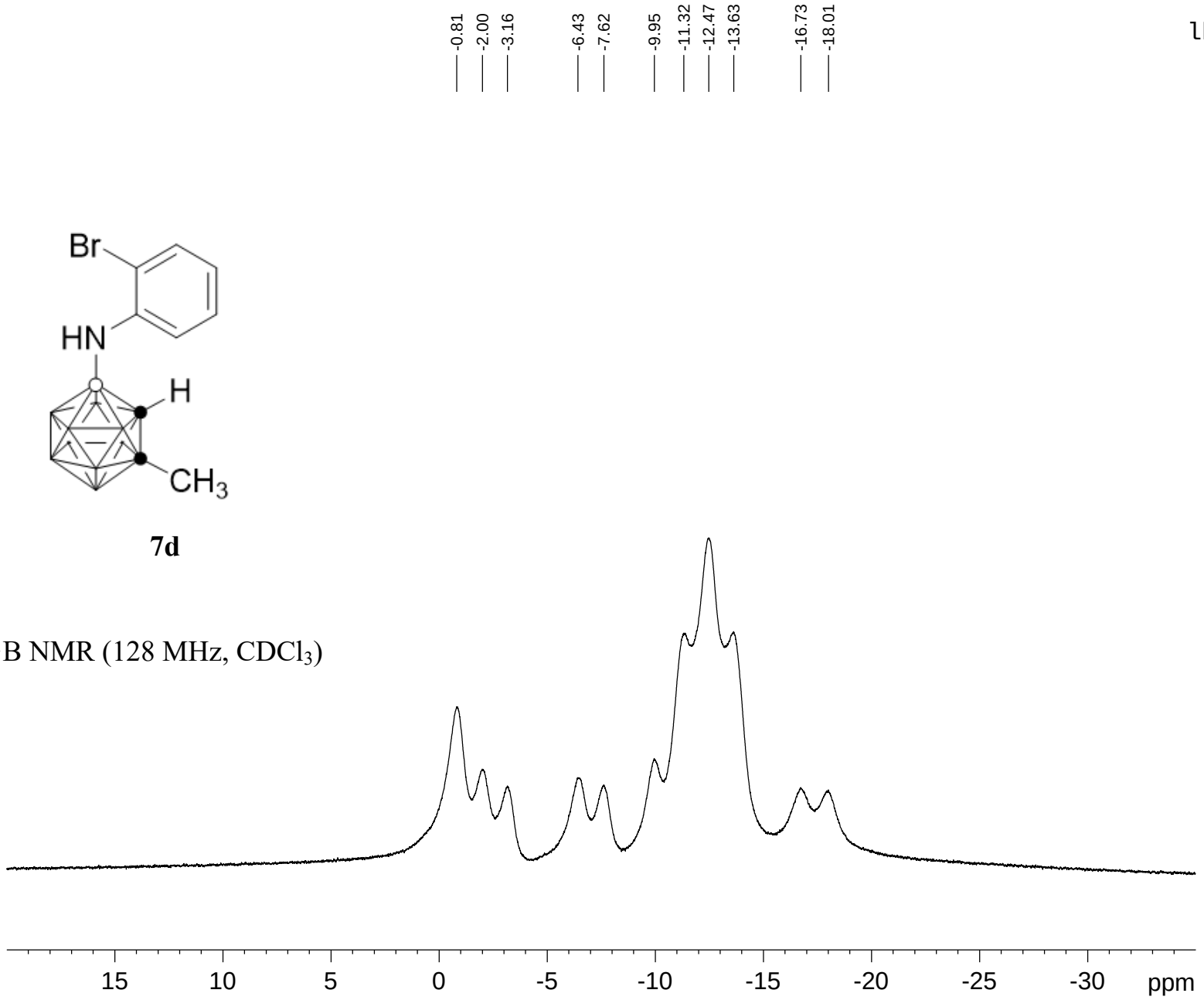


lhr-B-0592-CDCl3

```
Current Data Parameters
NAME      lhr-B-0592-CDCl3
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20170114
Time                 10.43 h
INSTRUM              spect
PROBHD   Z108618_0257 (
PULPROG               zgdc
TD                   65536
SOLVENT              CDCl3
NS                      12
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                  295.5 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
```

7d

^{11}B NMR (128 MHz, $CDCl_3$)

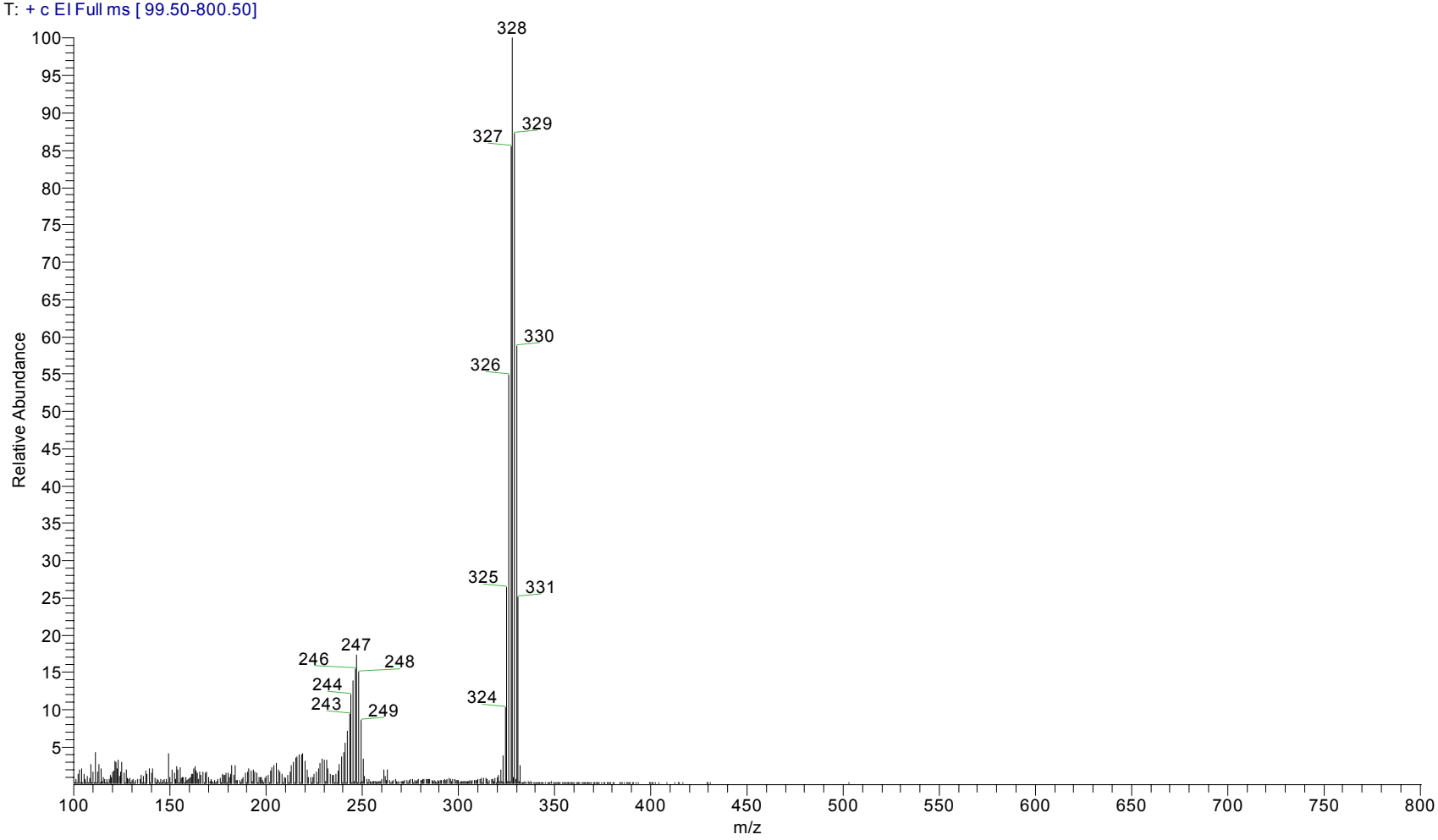


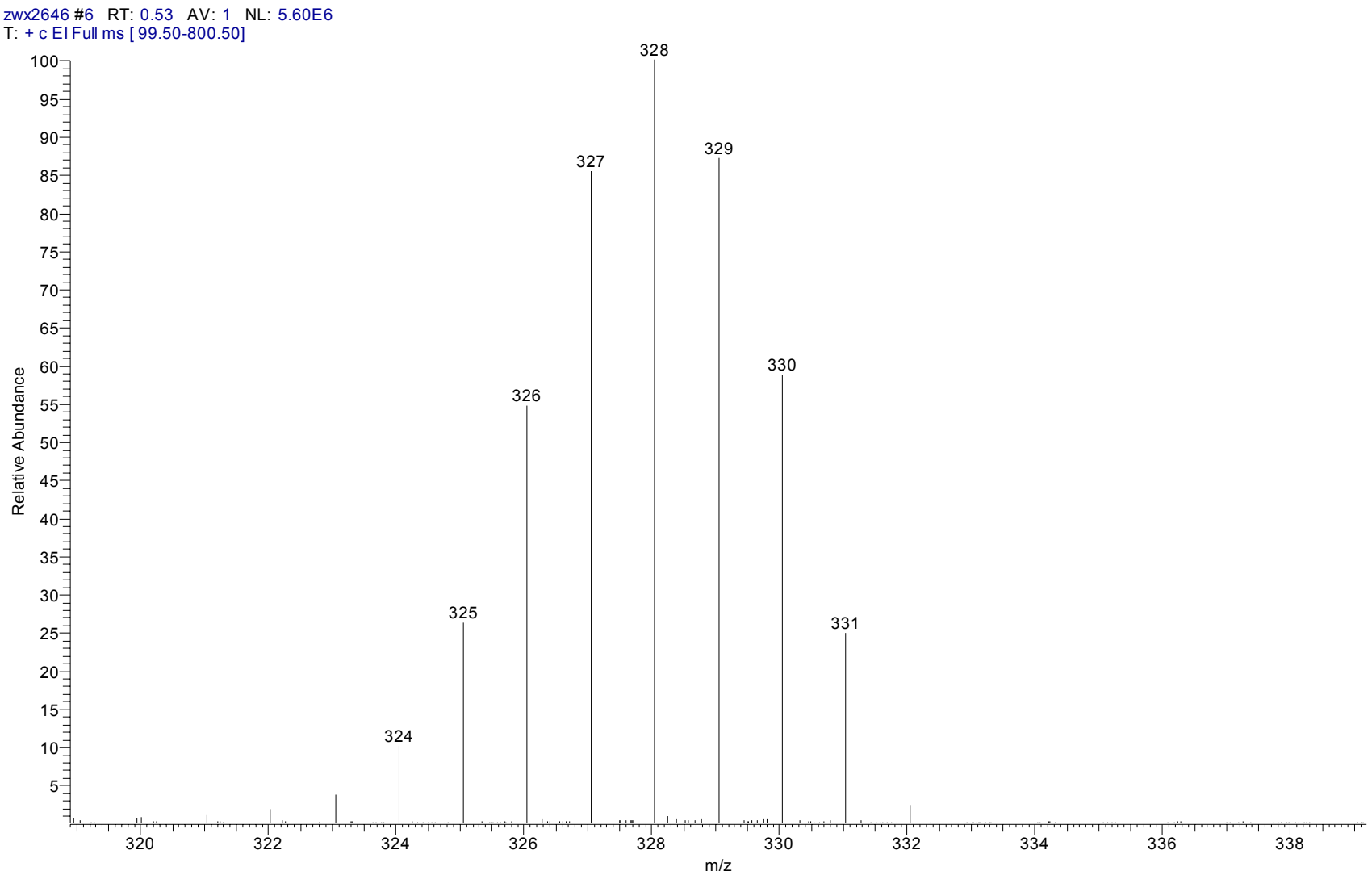
lhr-B-0592-CDCl3(C)

```
Current Data Parameters
NAME      lhr-B-0592-CDCl3(C)
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20170114
Time                  10.46 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                      203
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
```

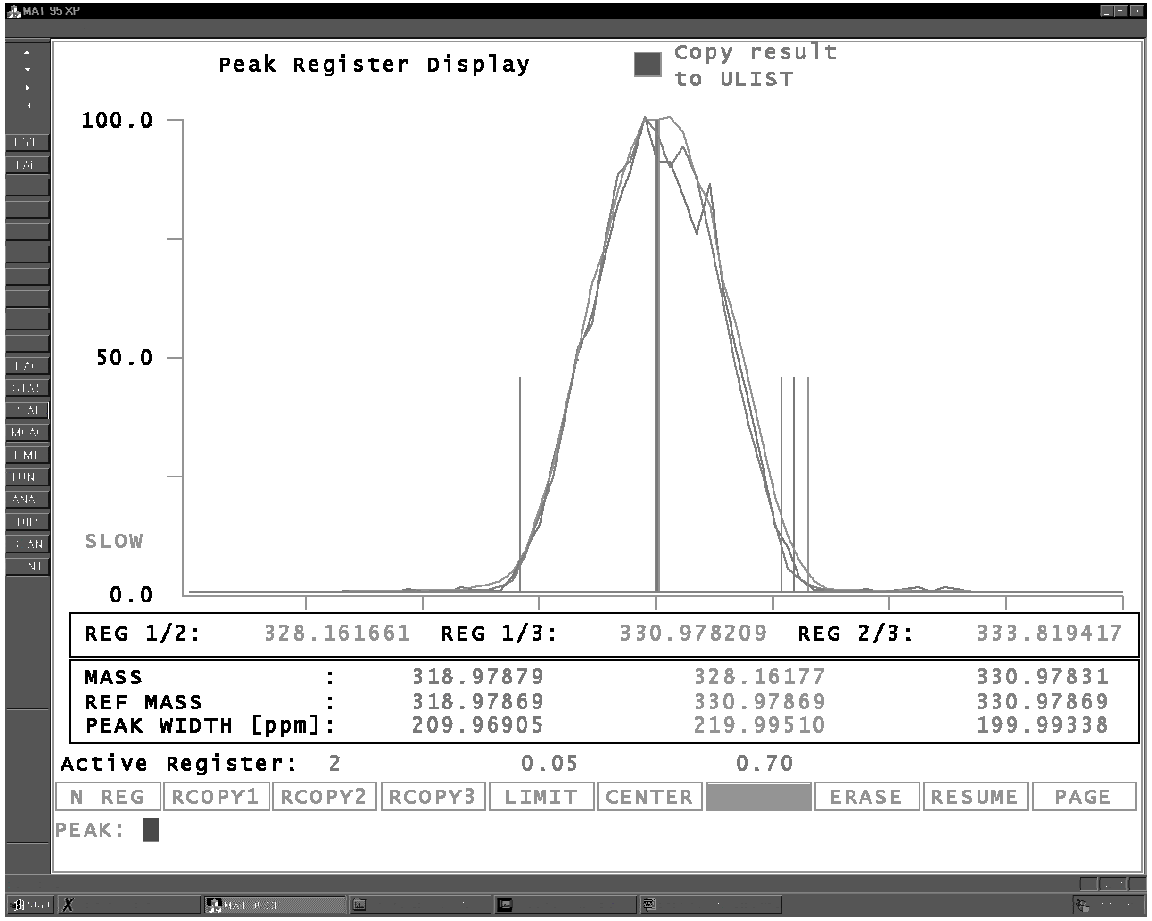
D:\MS_raw_data\zwx2646 03/09/17 04:41:51 PM lhr-0592
ei pos, 50eV, 0.7mA, unknown conc.

zwx2646 #6 RT: 0.53 AV: 1 NL: 5.60E6
T: + c EI Full ms [99.50-800.50]

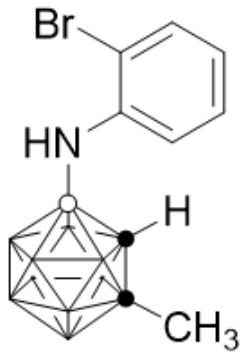



zwx2646 #6 RT: 0.53 AV: 1 NL: 5.60E6
T: + c EI Full ms [99.50-800.50]
Relative Abundance
m/z
328
327
329
326
330
325
331
324

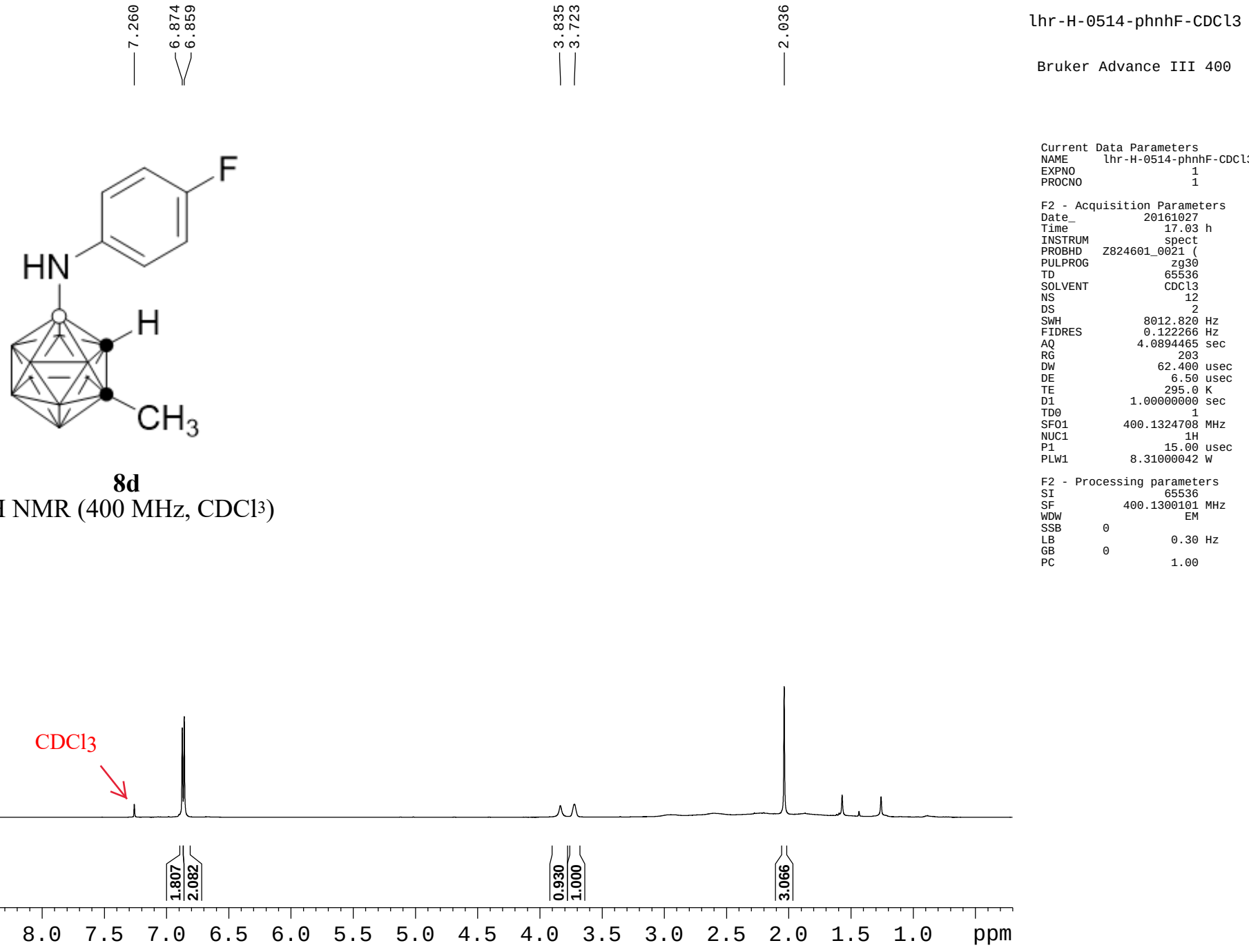
Accurate Mass Measurement



Molecular formula
$C_9H_{18}B_{10}BrN$
[M]+ (theoretical)
= 328.1611

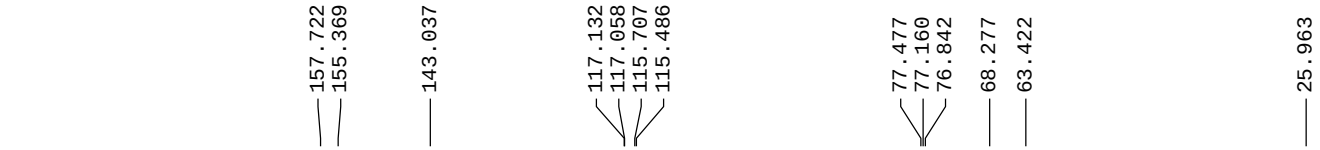


7d



8d

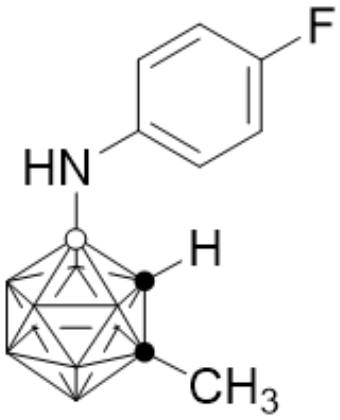
$_1$H NMR (400 MHz, CDCl^3)



lhr-C-0514-phnhF-CDCl3

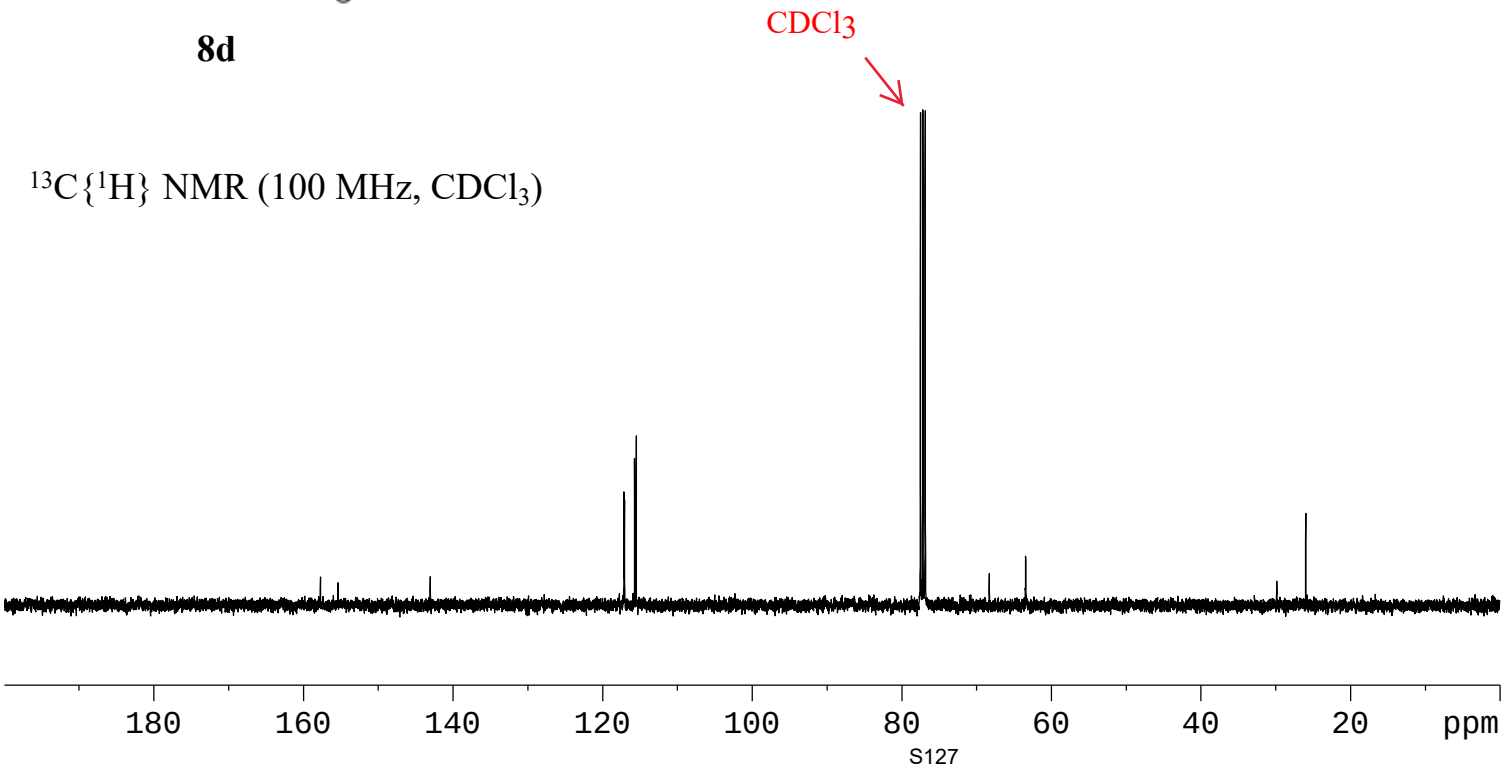
Bruker Advance III 400

```
Current Data Parameters
NAME      lhr-C-0514-phnhF-CDCl3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161027
Time              17.40 h
INSTRUM             spect
PROBHD   Z824601_0021 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   152
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                   203
DW                20.800 usec
DE                 6.50 usec
TE                295.6 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1         100.6228298 MHz
NUC1                 13C
P1                  9.50 usec
PLW1         41.25000000 W
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2          8.31000042 W
PLW12         0.23083000 W
PLW13         0.11611000 W

F2 - Processing parameters
SI                 32768
SF           100.6127566 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



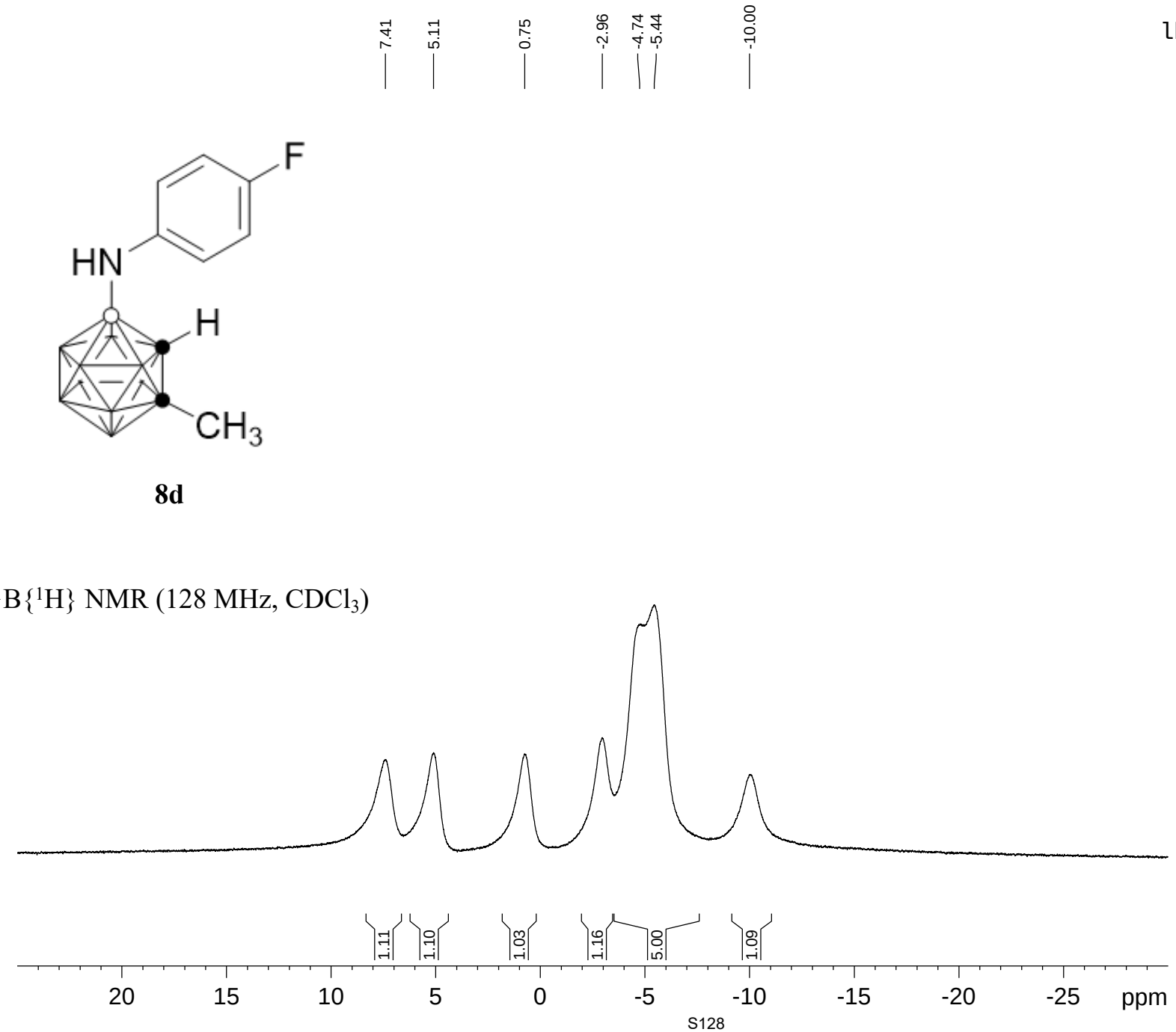
8d

$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)



8d

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)

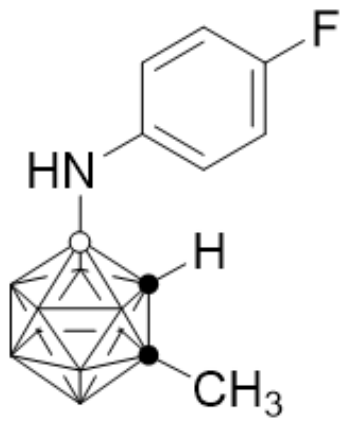


lhr-B-0514-phnh2F-CDCl3

```
Current Data Parameters
NAME      lhr-B-0514-phnh2F-CDCl3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161029
Time               9.17 h
INSTRUM           spect
PROBHD   Z108618_0257 (
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                   12
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                  287
DW               20.800 usec
DE                6.50 usec
TE                294.8 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
```

7.51 5.75 4.60 1.41 0.23 -2.31 -3.87 -4.95 -5.95 -9.34 -10.54

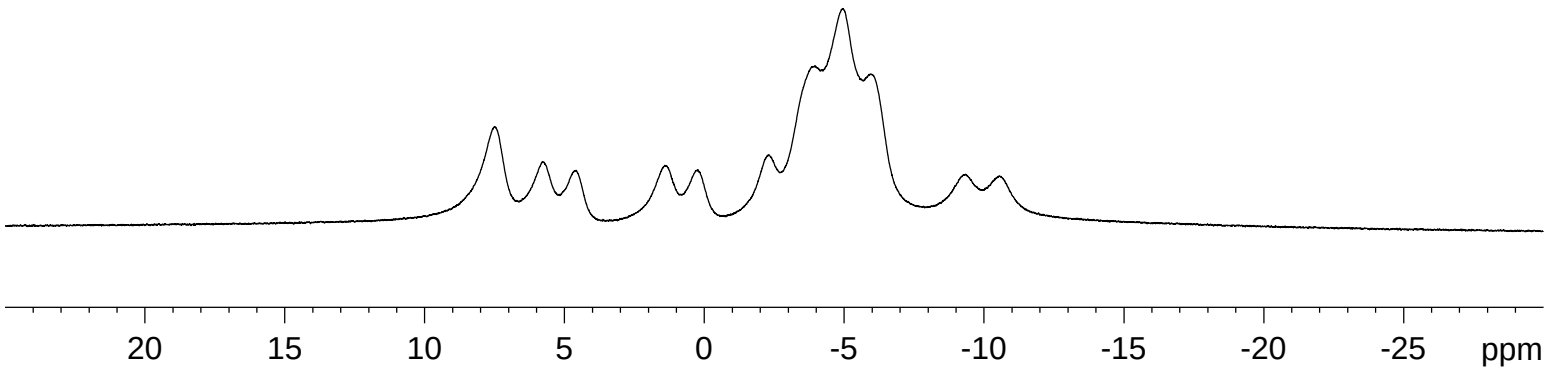
lhr-B-0514-phnh2F-CDCl3(



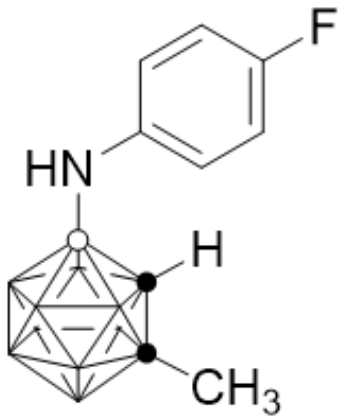
8d

```
Current Data Parameters
NAME      lhr-B-0514-phnh2F-CDCl3(C)
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161029
Time                9.20 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG               zg
TD                 65536
SOLVENT            CDCl3
NS                    26
DS                     2
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                   287
DW                20.800 usec
DE                 6.50 usec
TE                294.4 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           128.4097504 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{11}B NMR (128 MHz, $CDCl_3$)



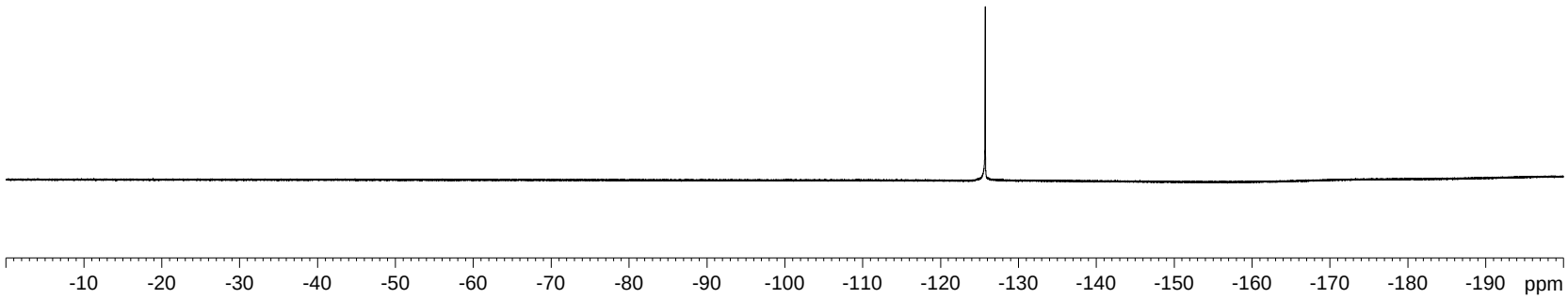
-125.75



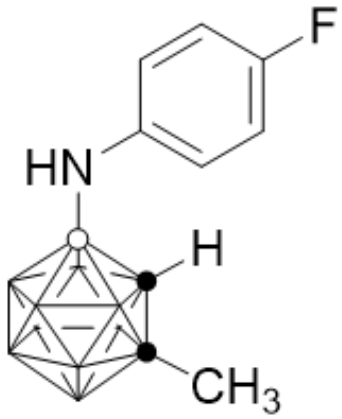
8d

$^{19}F\{^{1}H\}$ NMR (376 MHz, $CDCl_3$)

```
Current Data Parameters
NAME      lhr-F-0514-phnh2F-CDCl3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161029
Time                9.54 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG       zgfhigqn.2
TD                131072
SOLVENT            CDCl3
NS                    16
DS                     4
SWH            89285.711 Hz
FIDRES          0.681196 Hz
AQ              0.7340032 sec
RG                   645
DW                 5.600 usec
DE                 6.50 usec
TE                 294.7 K
D1           1.00000000 sec
D11          0.03000000 sec
D12          0.00002000 sec
TD0                    1
SFO1         376.5548010 MHz
NUC1                 19F
P1                14.70 usec
PLW1         18.36000061 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                 65536
SF           376.5924602 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



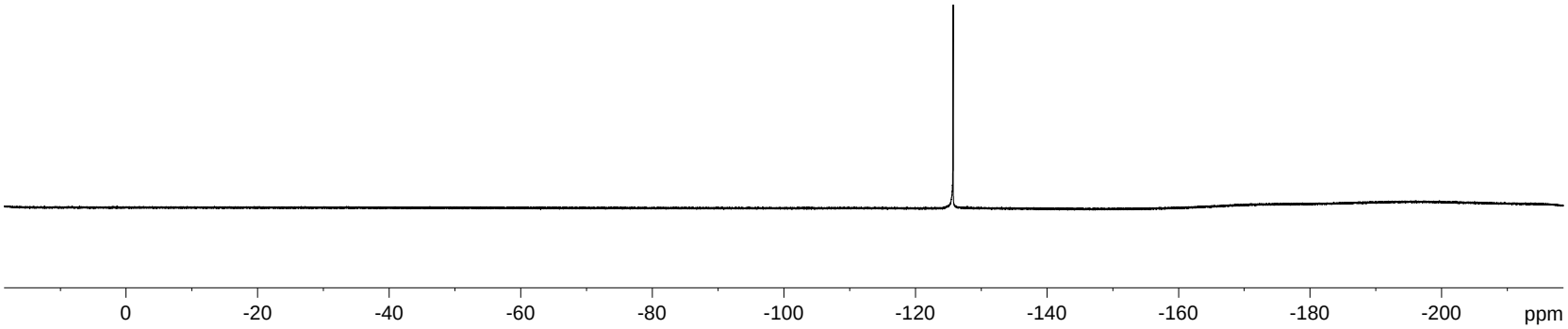
-125.73



8d

^{19}F NMR (376 MHz, $CDCl_3$)

```
Current Data Parameters
NAME      lhr-F-0514-phnh2F-CDCl3(C)
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161029
Time                9.55 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG       zgfhigqn.2
TD                131072
SOLVENT            CDCl3
NS                    16
DS                     4
SWH            89285.711 Hz
FIDRES          0.681196 Hz
AQ              0.7340032 sec
RG                   645
DW                 5.600 usec
DE                 6.50 usec
TE                 294.8 K
D1           1.00000000 sec
D11          0.03000000 sec
D12          0.00002000 sec
TD0                    1
SFO1         376.5548010 MHz
NUC1                 19F
P1                14.70 usec
PLW1         18.36000061 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                 65536
SF           376.5924602 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



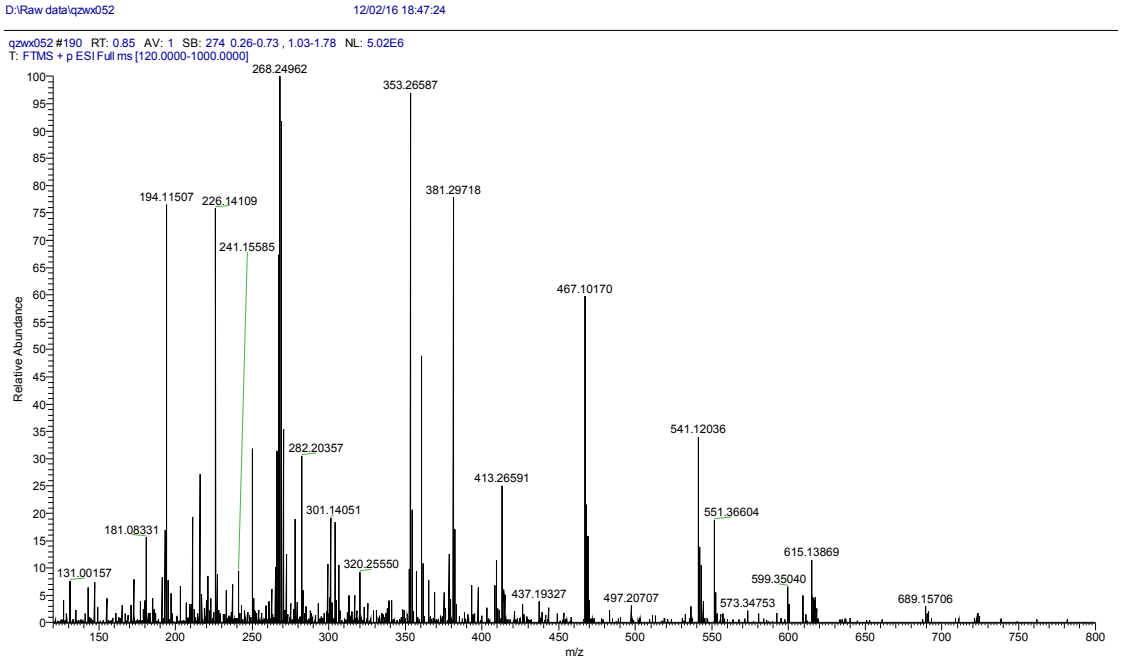
Thermo QEFMS Analysis Report

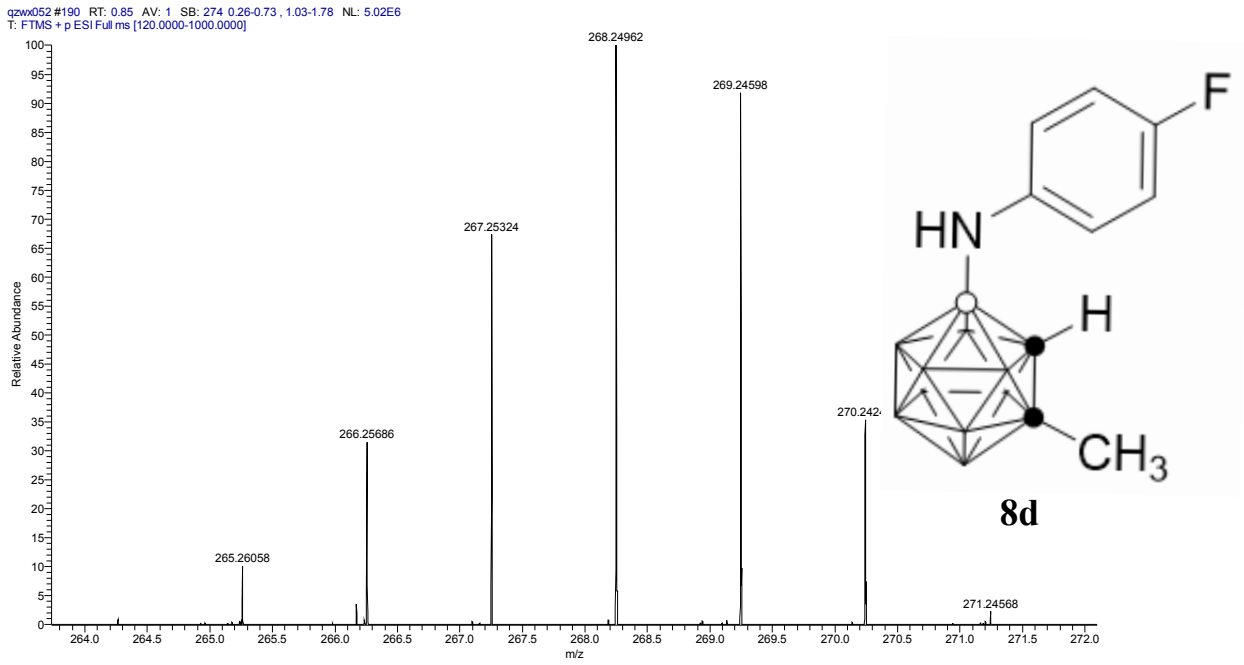
Analysis Info

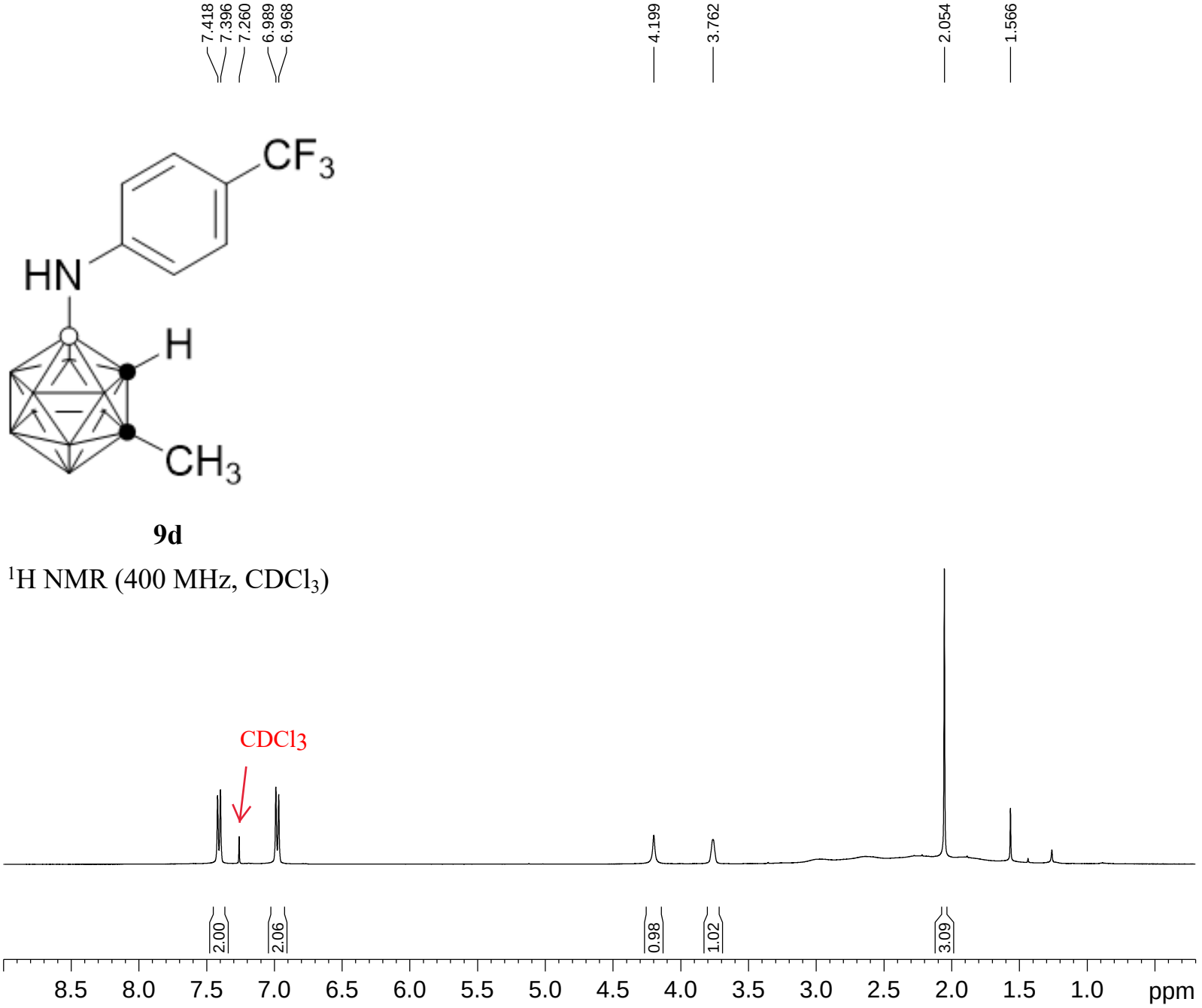
Sample Name :	Lhr-514	Reference No.:	Qzwx052
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.5kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_9H_{18}B_{10}FN$
Experimental Mass $[M+H]^+$:	268.24962
Theoretical Mass $[M+H]^+$:	268.24992
Error (ppm) :	1.1





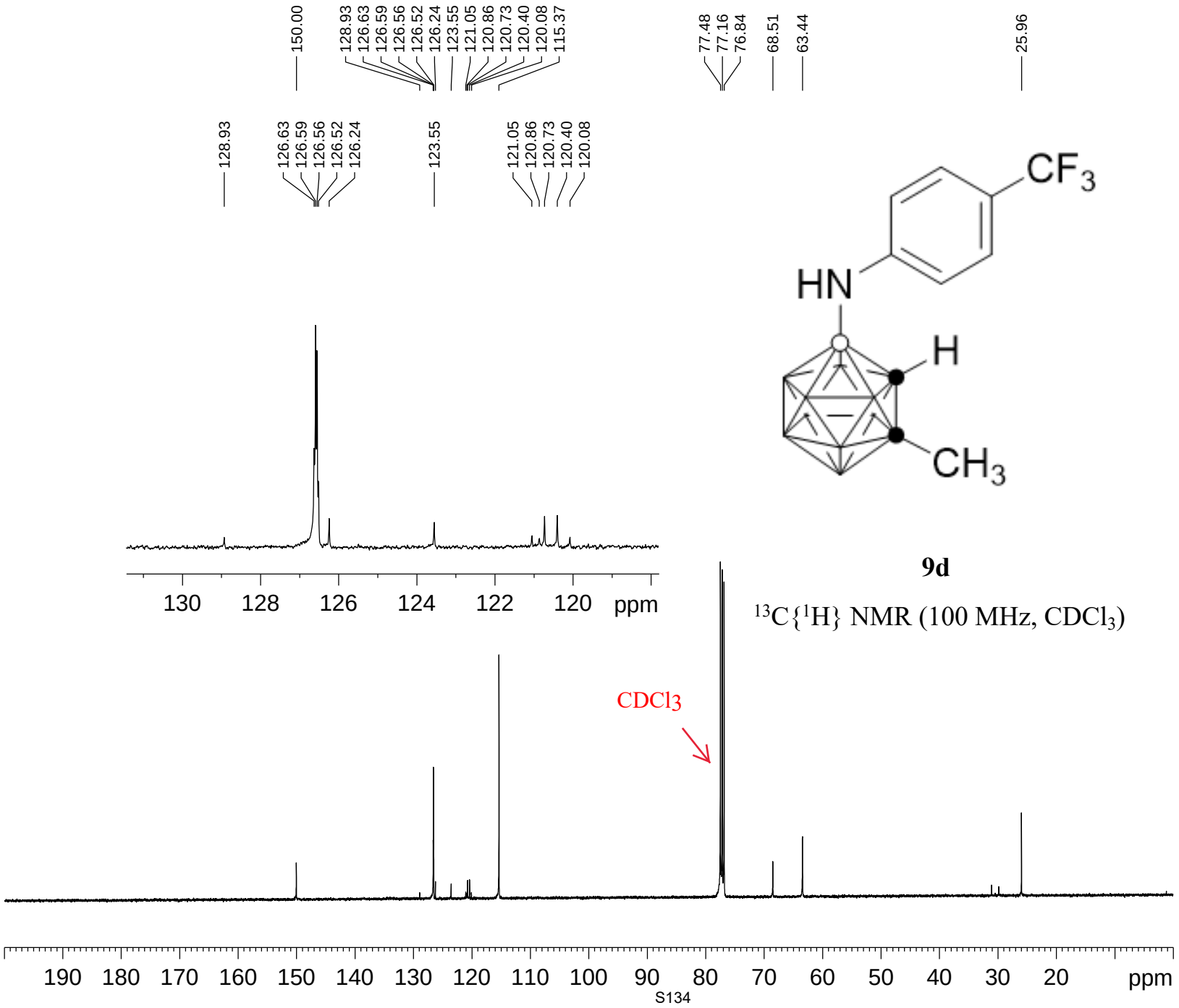


lhr-H-0517-phnh2CF3-

Current Data Parameters
NAME lhr-H-0517-phnh2CF3-CDCl3
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161030
Time 14.08 h
INSTRUM spect
PROBHD Z108618_0257 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 90.5
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 400.2300104 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

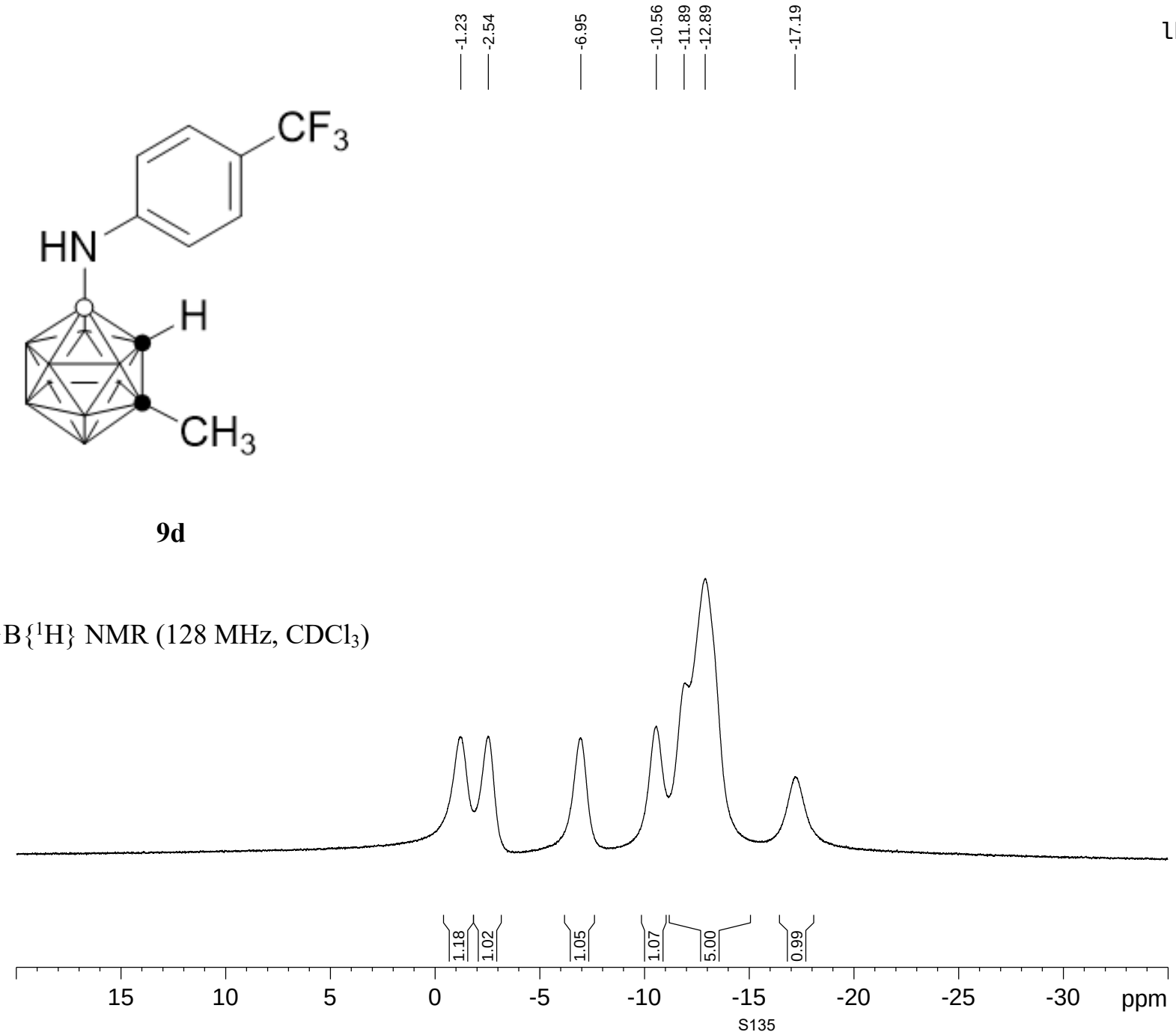


lhr-C-0517-phnh2CF3

```
Current Data Parameters
NAME      lhr-C-0517-phnh2CF3-CDCl3
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_           20161030
Time              19.45 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3439
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG                  161
DW               16.800 usec
DE                6.50 usec
TE                294.8 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           100.6379016 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

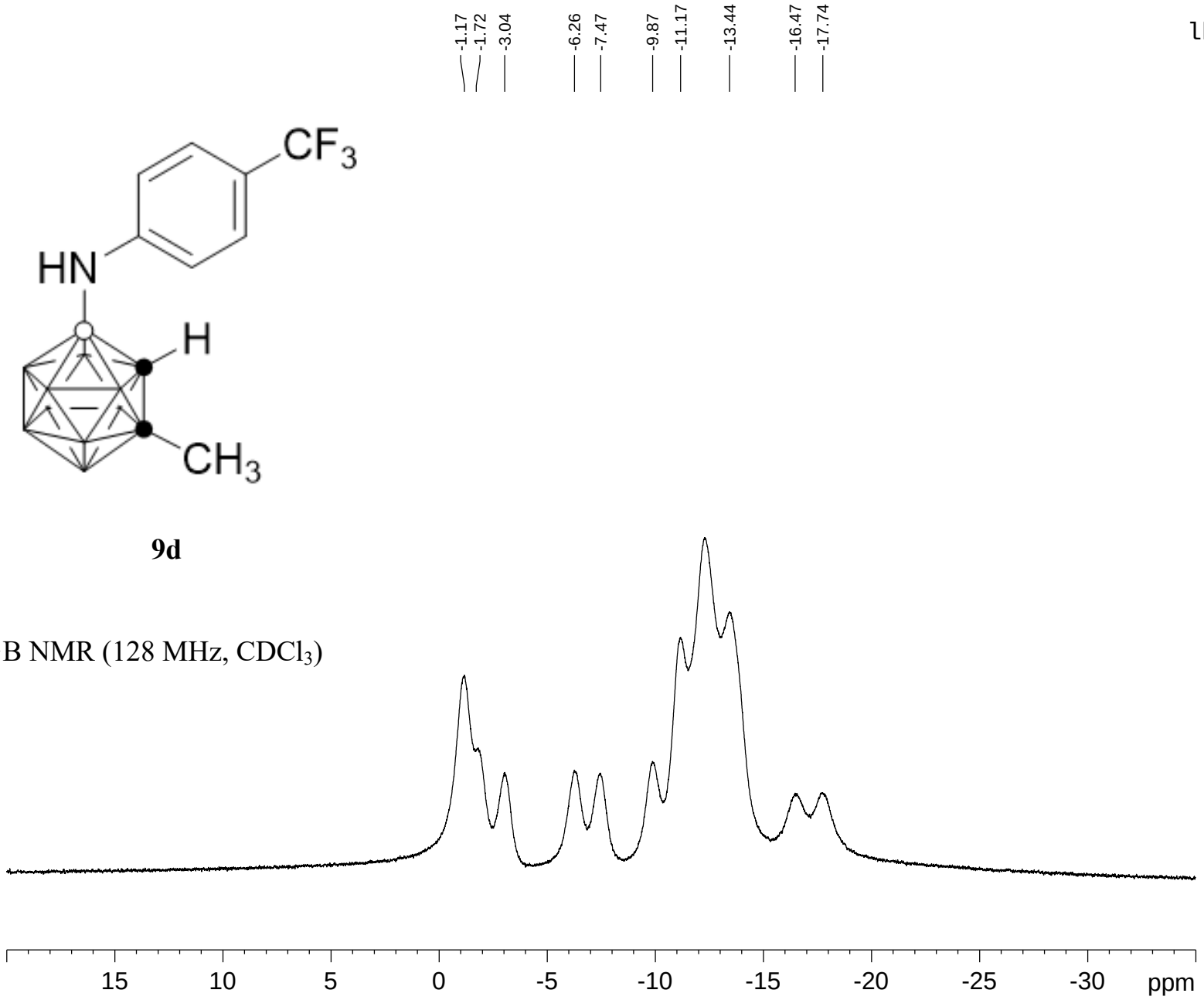
9d

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)



lhr-B-0517-phnh2CF3-CDCl3

```
Current Data Parameters
NAME      lhr-B-0517-phnh2CF3-CDCl3
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20161031
Time               9.52 h
INSTRUM           spect
PROBHD   Z108618_0257 (
PULPROG           zgdc
TD               65536
SOLVENT           C6D6
NS                  16
DS                   4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                 287
DW                20.800 usec
DE                 6.50 usec
TE                295.8 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
```

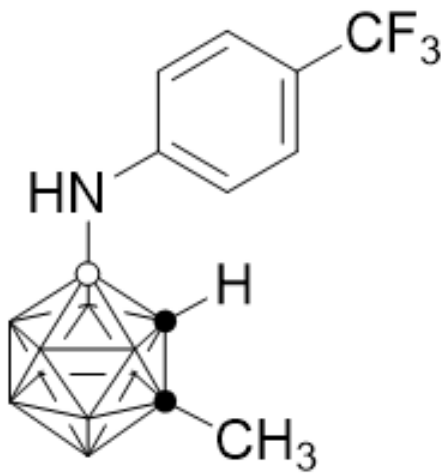


9d

^{11}B NMR (128 MHz, $CDCl_3$)

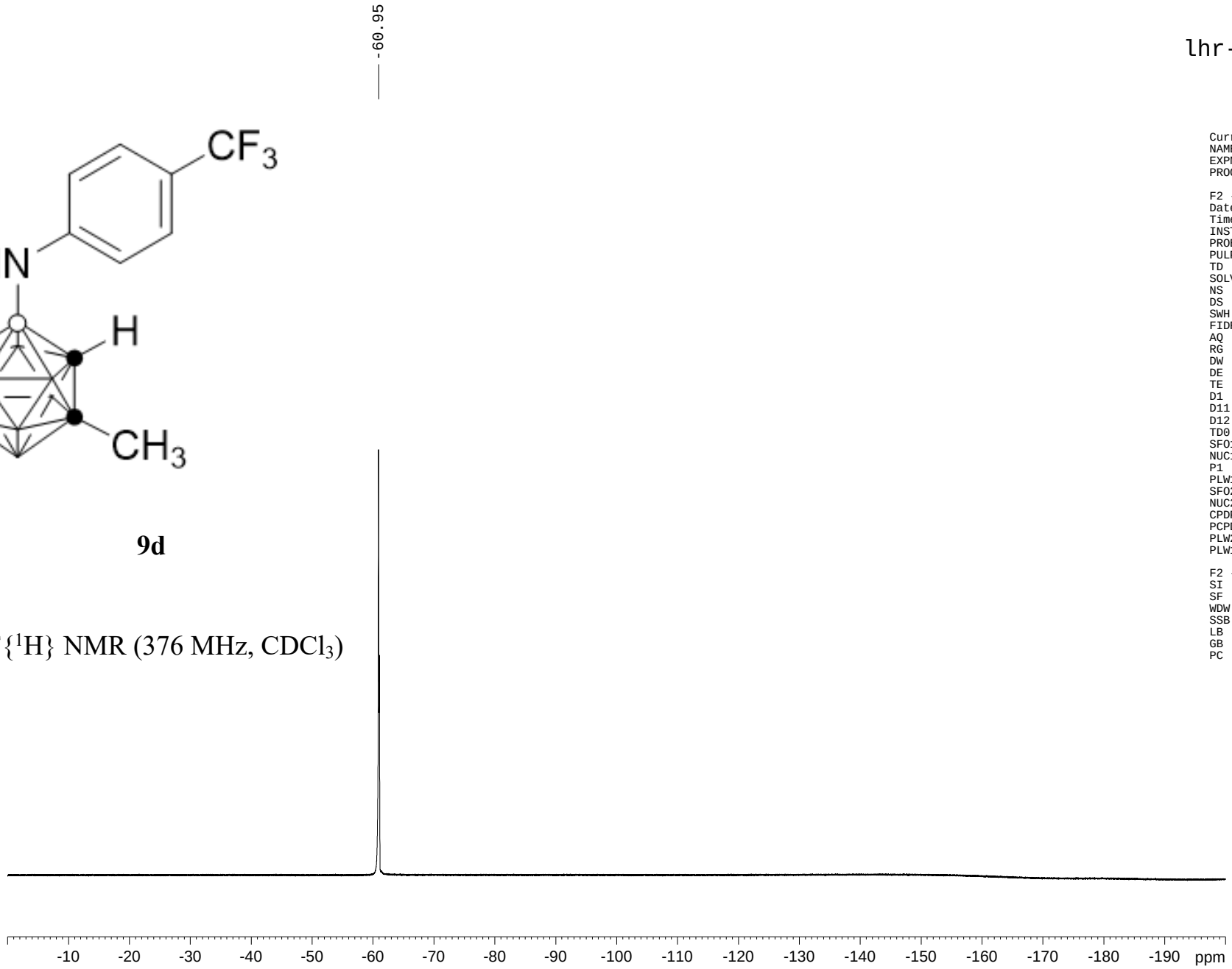
lhr-B-0517-phnh2CF3-CDCl3(C)

```
Current Data Parameters
NAME      lhr-B-0517-phnh2CF3-CDCl3(C)
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20161031
Time               9.53 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG              zg
TD                65536
SOLVENT            C6D6
NS                   12
DS                    2
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                  287
DW               20.800 usec
DE                6.50 usec
TE               295.3 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          128.4097504 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



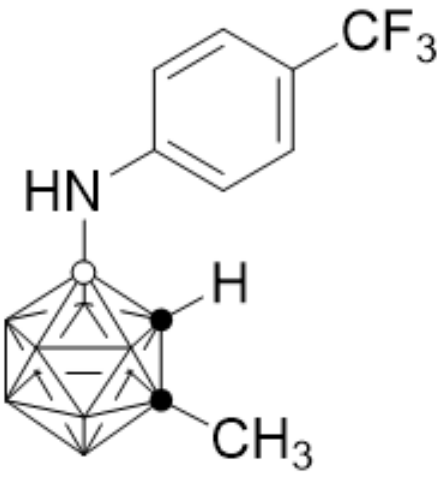
9d

$^{19}F\{^{1}H\}$ NMR (376 MHz, $CDCl_3$)



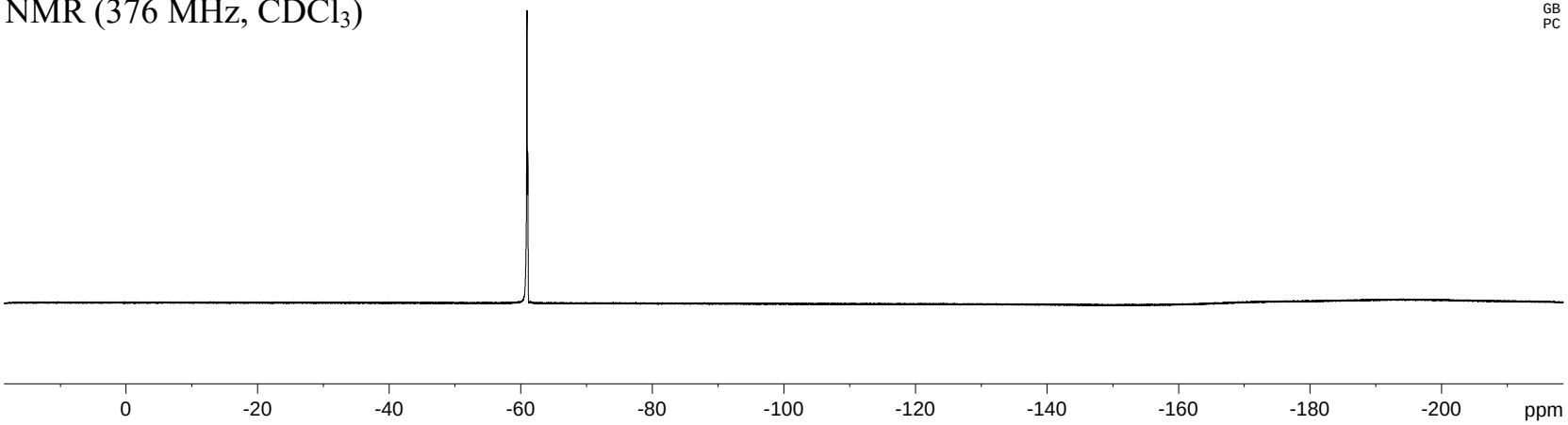
lhr-F-0517-phnh2CF3-(

```
Current Data Parameters
NAME      lhr-F-0517-phnh2CF3-CDCl3
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20161031
Time                9.56 h
INSTRUM           spect
PROBHD   Z108618_0257 (
PULPROG       zgfhigqn.2
TD               131072
SOLVENT            C6D6
NS                   21
DS                    4
SWH           89285.711 Hz
FIDRES         0.681196 Hz
AQ            0.7340032 sec
RG                  645
DW                5.600 usec
DE                6.50 usec
TE                295.3 K
D1           1.00000000 sec
D11          0.03000000 sec
D12          0.00002000 sec
TD0                   1
SFO1         376.5548010 MHz
NUC1                19F
P1                14.70 usec
PLW1         18.36000061 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                65536
SF           376.5924602 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



9d

^{19}F NMR (376 MHz, $CDCl_3$)



lhr-F-0517-phnh2CF3-(

```
Current Data Parameters
NAME     lhr-F-0517-phnh2CF3-CDCl3(C)
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20161031
Time                9.57 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG       zgfhigqn.2
TD                131072
SOLVENT             C6D6
NS                    10
DS                     4
SWH            89285.711 Hz
FIDRES          0.681196 Hz
AQ              0.7340032 sec
RG                   645
DW                 5.600 usec
DE                 6.50 usec
TE                 295.3 K
D1           1.00000000 sec
D11          0.03000000 sec
D12          0.00002000 sec
TD0                    1
SFO1         376.5548010 MHz
NUC1                 19F
P1                14.70 usec
PLW1         18.36000061 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                 65536
SF           376.5924602 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

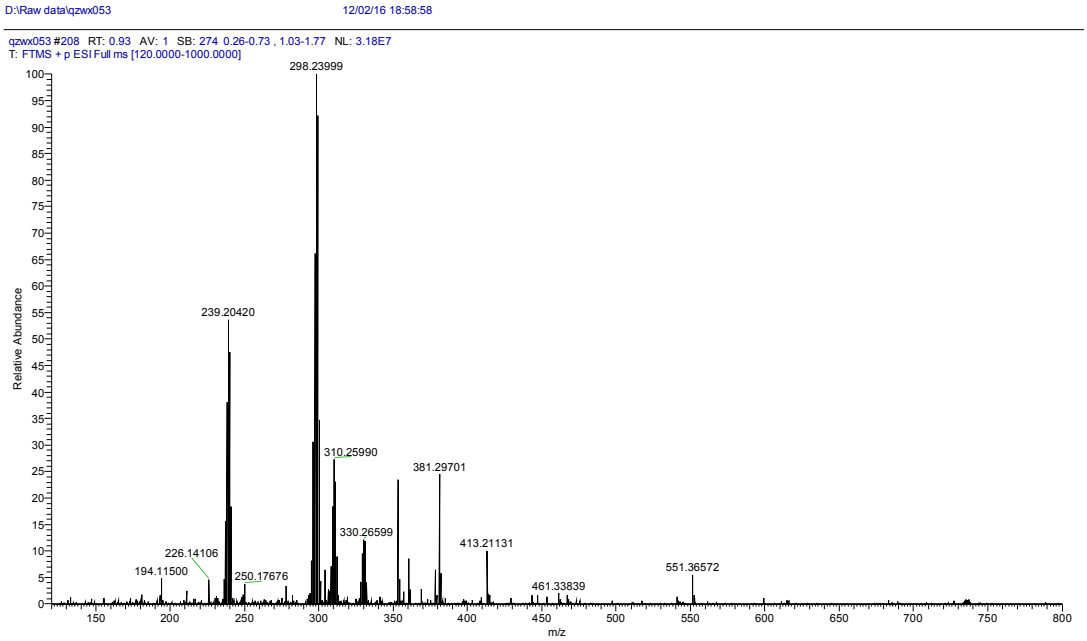
Thermo QEFMS Analysis Report

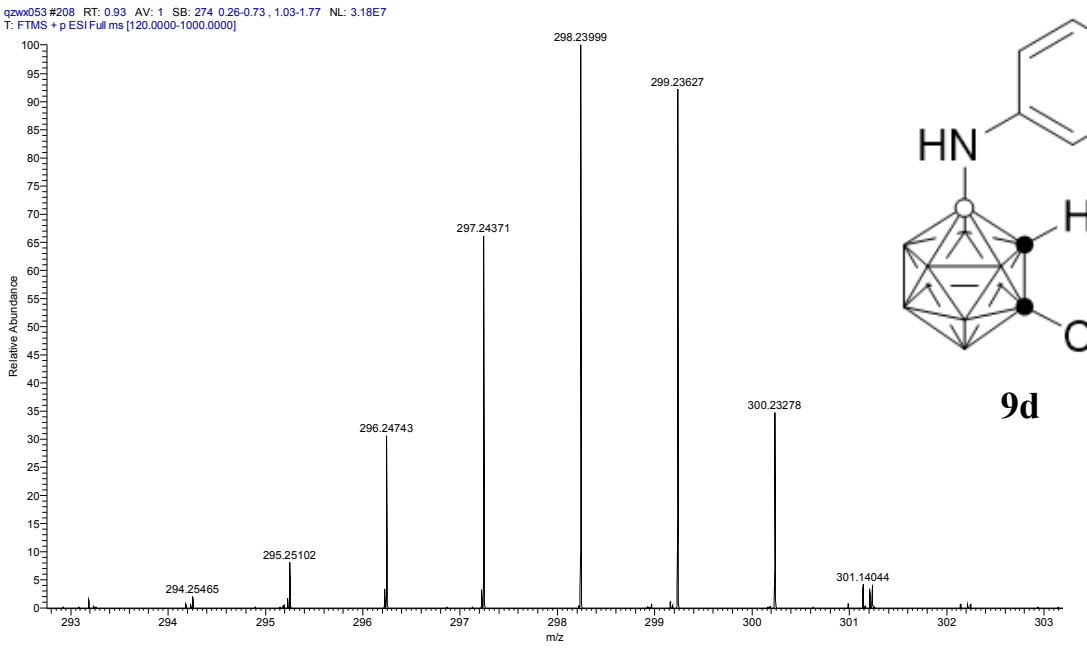
Analysis Info

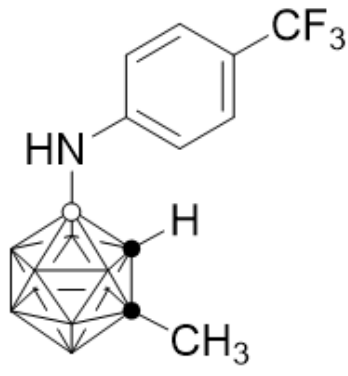
Sample Name :	Lhr-517	Reference No.:	Qzwx053
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.5kV, by LC, with sheath gas		

Accurate Mass Measurement

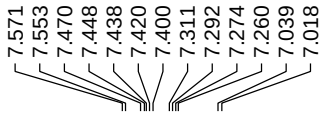
Molecular formula :	$C_{10}H_{18}B_{10}F_3N$
Experimental Mass $[M-F]^+$:	298.23999
Theoretical Mass $[M-F]^+$:	298.24050
Error (ppm) :	1.7



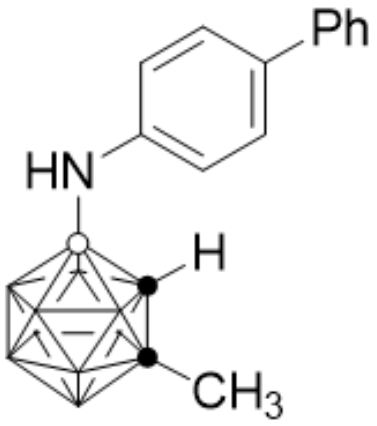




9d

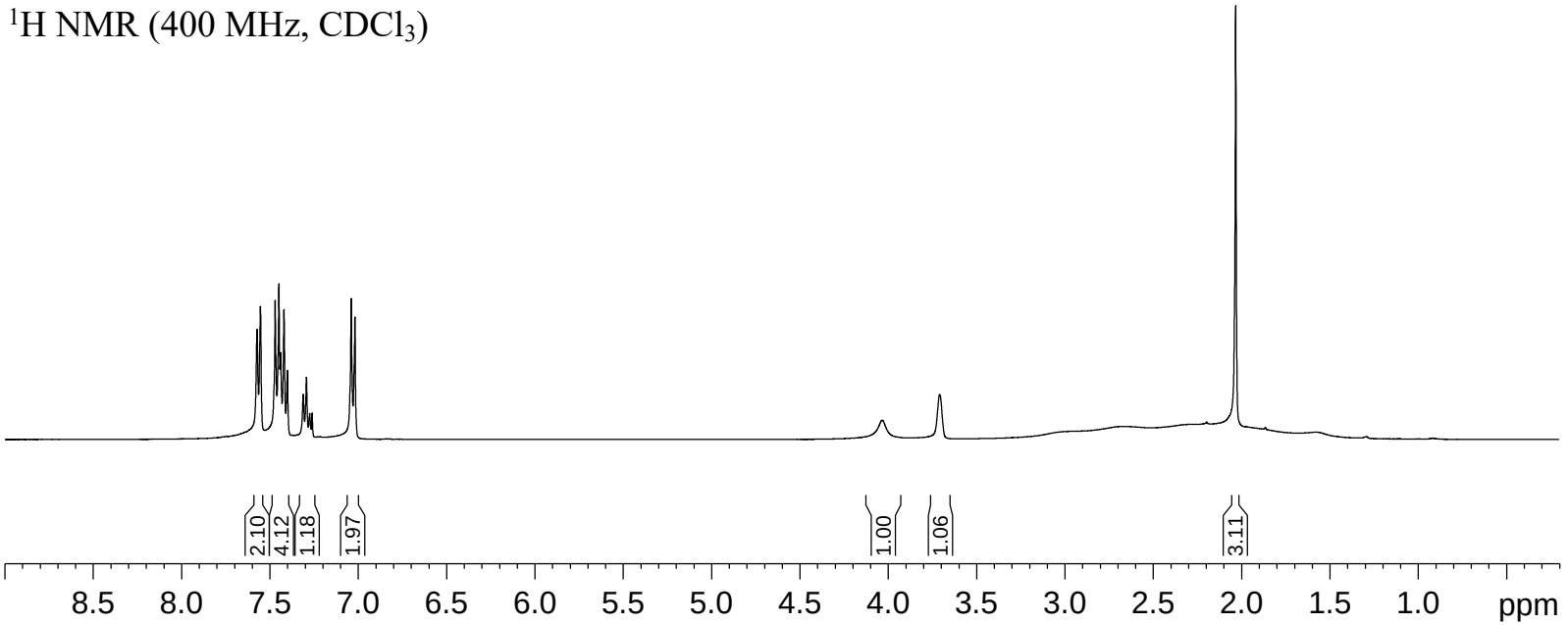


lhr-H-0526-phnhPH-C



10d

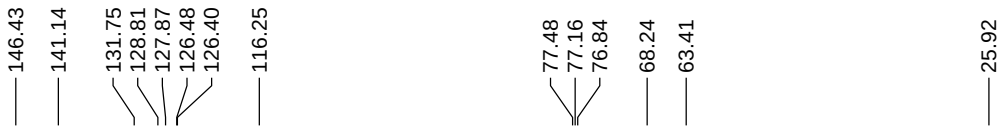
^{1}H NMR (400 MHz, $CDCl_3$)



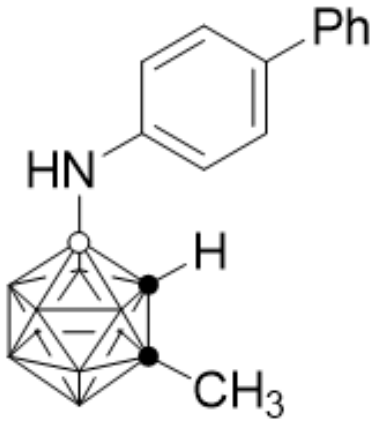
Current Data Parameters
NAME lhr-H-0526-phnhPH-CD
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161104
Time 12.40 h
INSTRUM spect
PROBHD Z108618_0257 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 9
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 57
DW 62.400 usec
DE 6.50 usec
TE 296.0 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 400.2300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



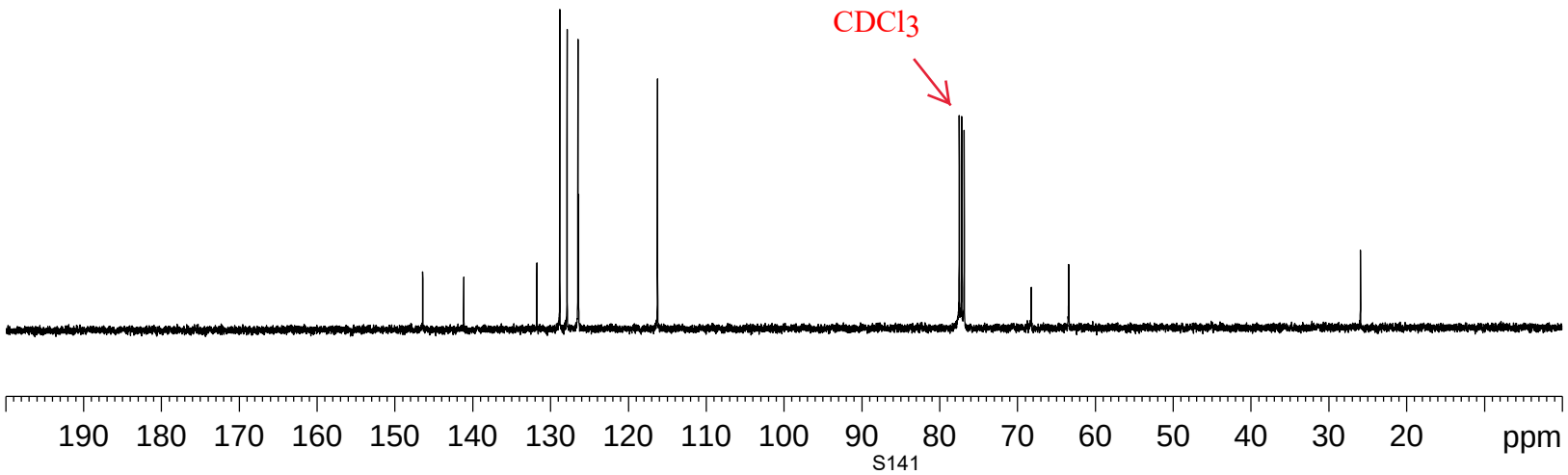
lhr-C-0526-phnhPH-(



10d

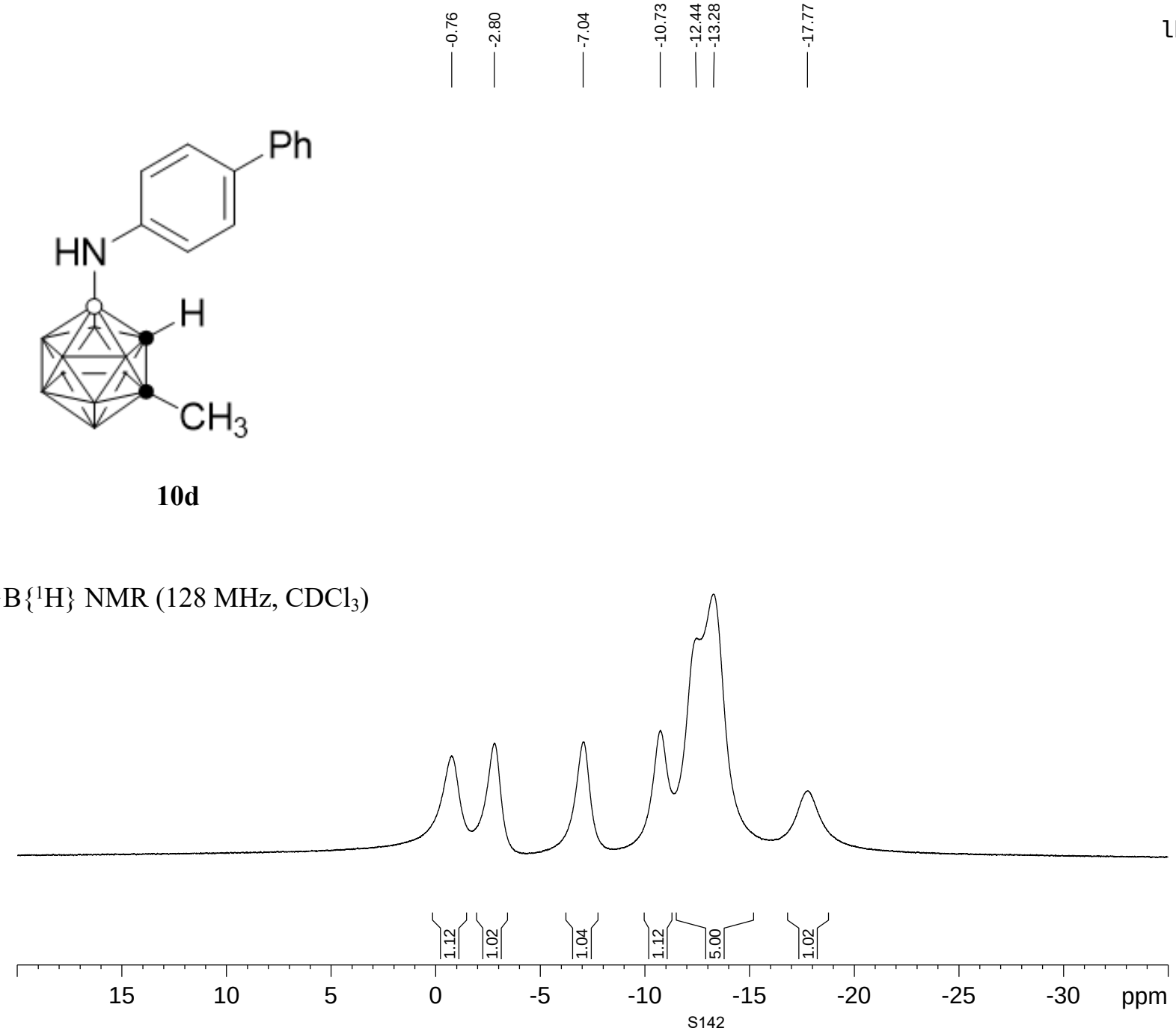
$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)

```
Current Data Parameters
NAME      lhr-C-0526-phnhPH-CDCl3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161104
Time              12.44 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  100
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG                  161
DW               16.800 usec
DE                6.50 usec
TE               296.6 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          100.6379063 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



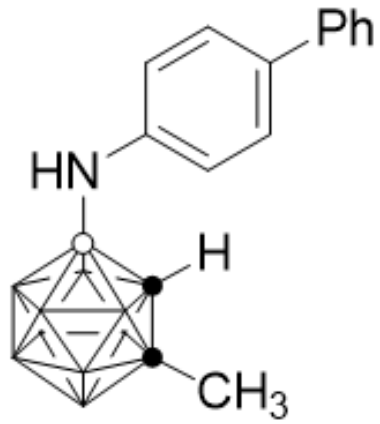
10d

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)



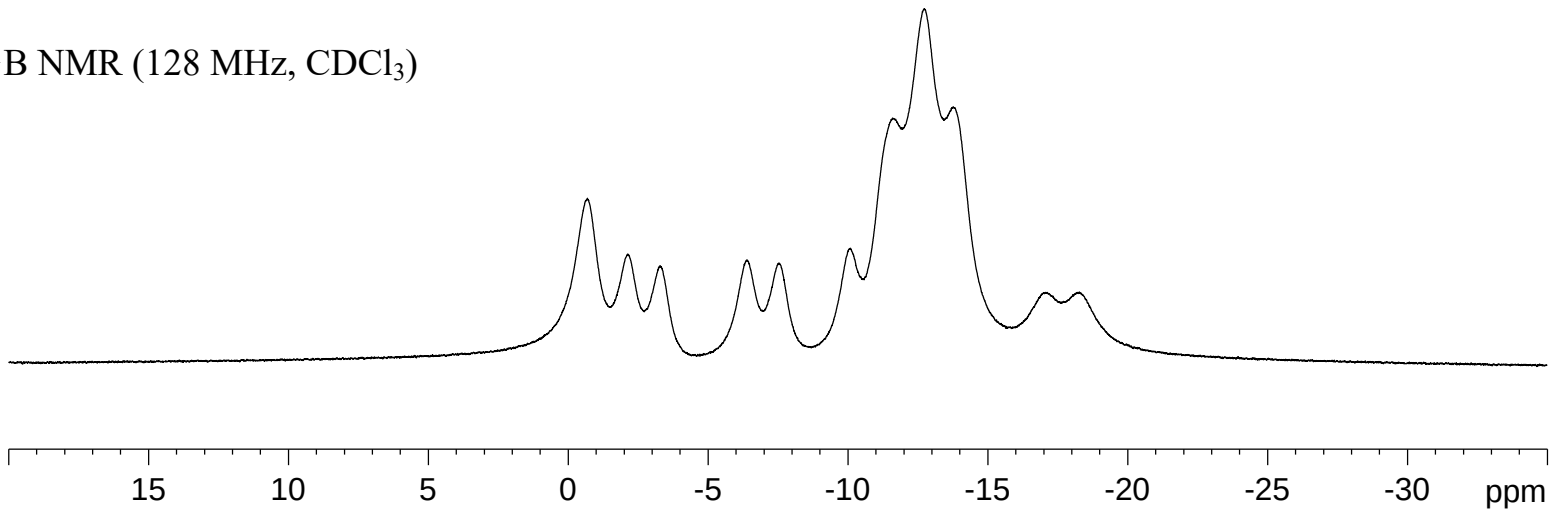
lhr-B-0526-phnhPH-CDCl3

```
Current Data Parameters
NAME      lhr-B-0526-phnhPH-CDCl3
EXPNO                          1
PROCNO                         1

F2 - Acquisition Parameters
Date_               20161104
Time                   12.49 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                       203
DW                    20.800 usec
DE                     6.50 usec
TE                    296.6 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
```



10d

^{11}B NMR (128 MHz, $CDCl_3$)



lhr-B-0526-phnhPH-CDCl3(C)

```
Current Data Parameters
NAME      lhr-B-0526-phnhPH-CDCl3(C)
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20161104
Time              12.51 h
INSTRUM           spect
PROBHD   Z108618_0257 (
PULPROG             zg
TD                65536
SOLVENT           CDCl3
NS                   18
DS                    2
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                  203
DW               20.800 usec
DE                6.50 usec
TE               296.3 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          128.4097504 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

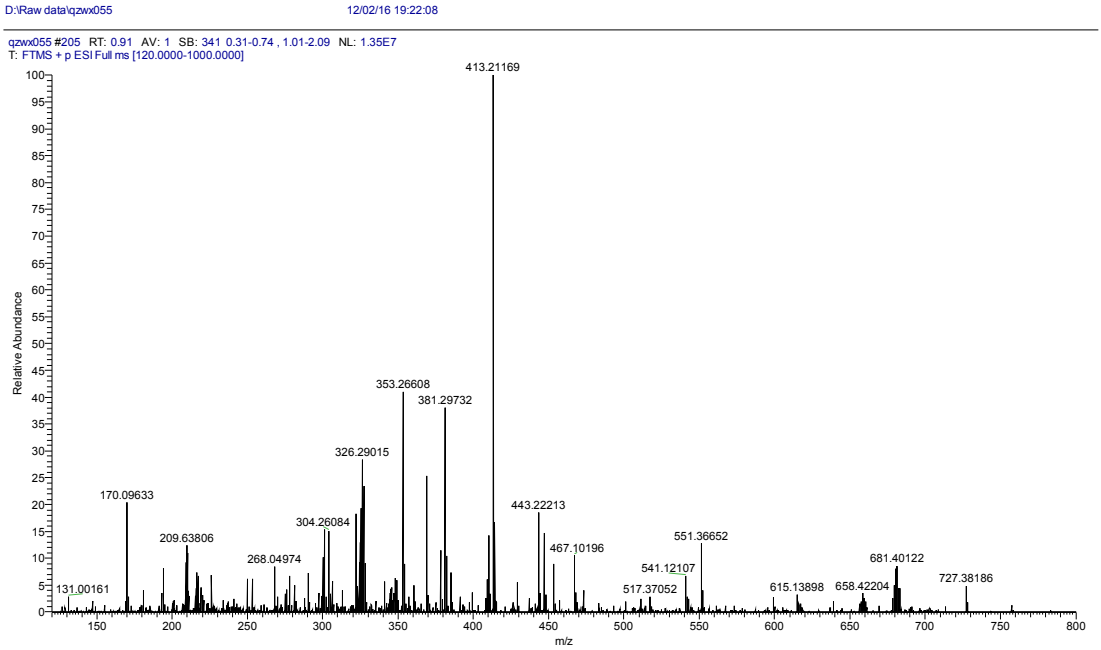
Thermo QEFMS Analysis Report

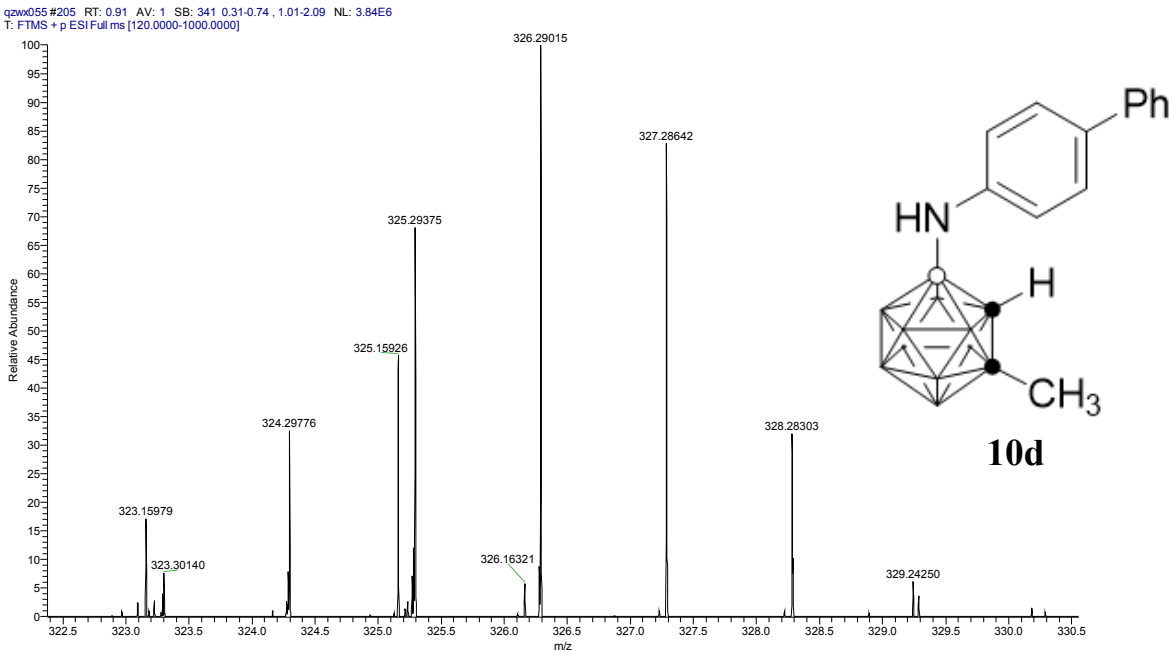
Analysis Info

Sample Name :	Lhr-526	Reference No.:	Qzwx055
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.5kV, by LC, with sheath gas		

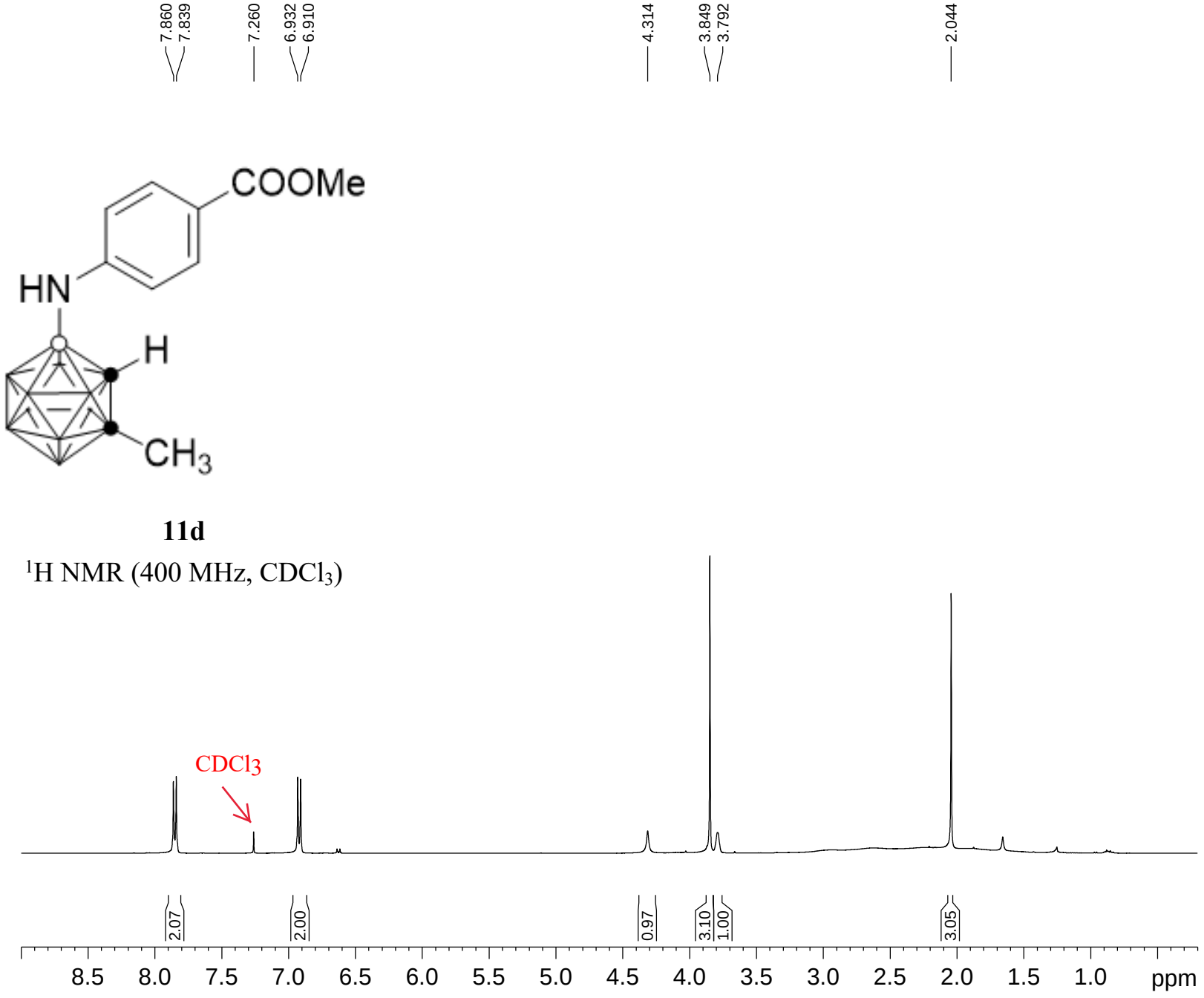
Accurate Mass Measurement

Molecular formula :	$C_{15}H_{23}B_{10}N$
Experimental Mass $[M+H]^+$:	326.29015
Theoretical Mass $[M+H]^+$:	326.29065
Error (ppm) :	1.5





10d



lhr-H-0519-phnhcoome

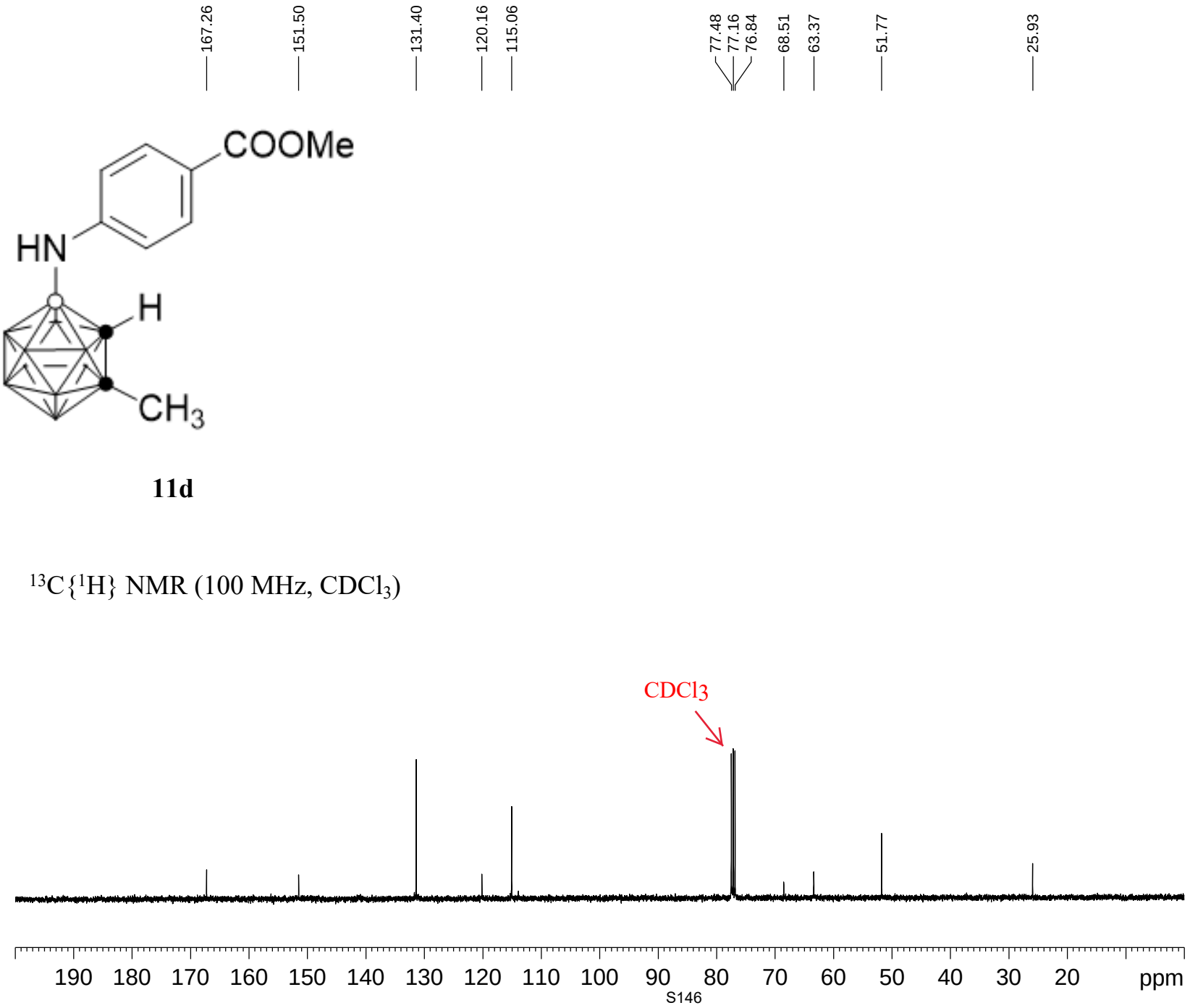
Current Data Parameters
NAME lhr-H-0519-phnhcoome-CDCl3
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161030
Time 14.13 h
INSTRUM spect
PROBHD Z108618_0257 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
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 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 400.2300108 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

11d

$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)



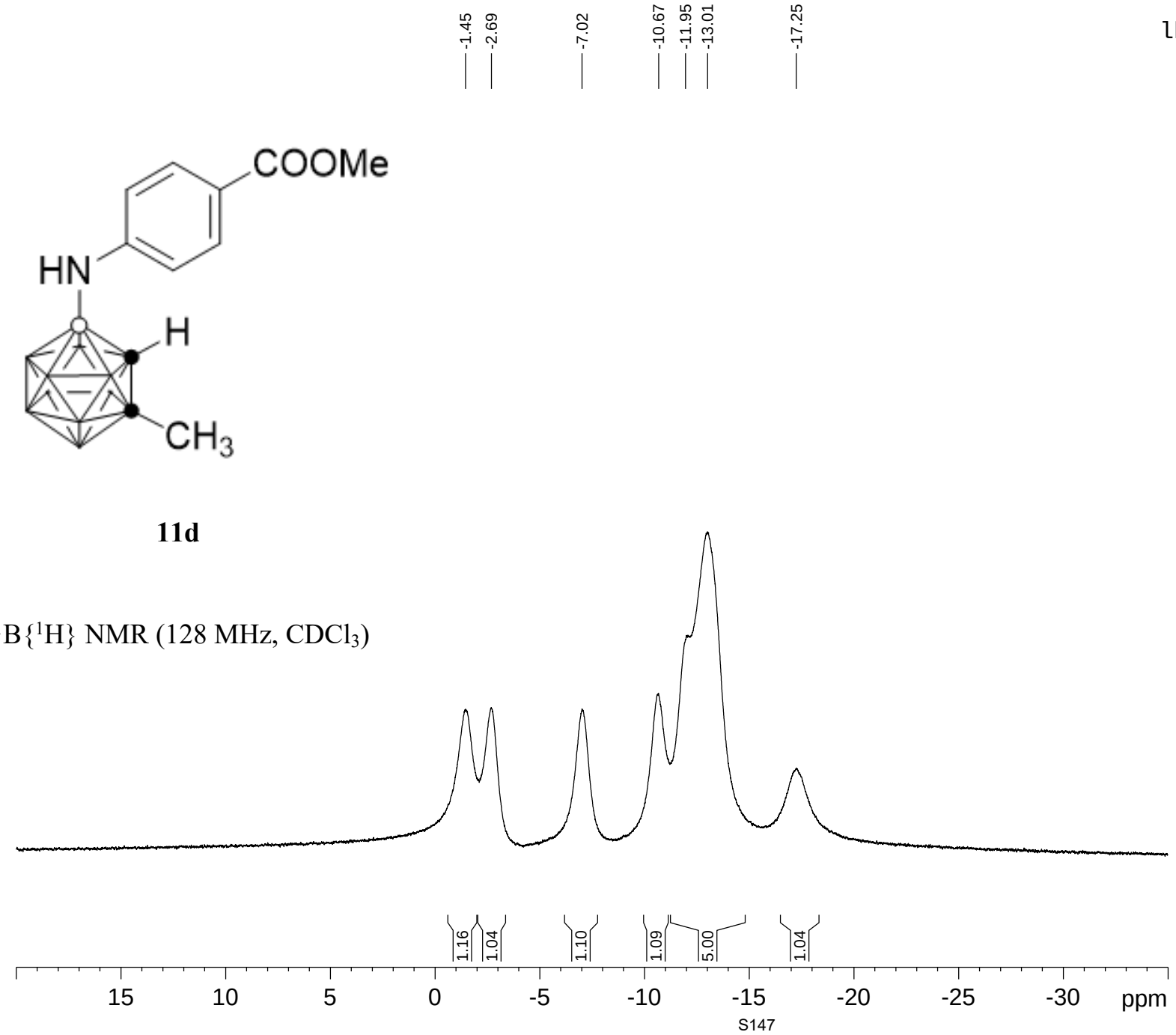
lhr-C-0519-phnhcoor

```
Current Data Parameters
NAME      lhr-C-0519-phnhcoome-CDCl3
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20161030
Time              19.38 h
INSTRUM           spect
PROBHD   Z108618_0257 (
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                  52
DS                   4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG                 161
DW              16.800 usec
DE               6.50 usec
TE              295.2 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          100.6379038 MHz
WDW                EM
SSB      0
LB                1.00 Hz
GB       0
PC                1.40
```

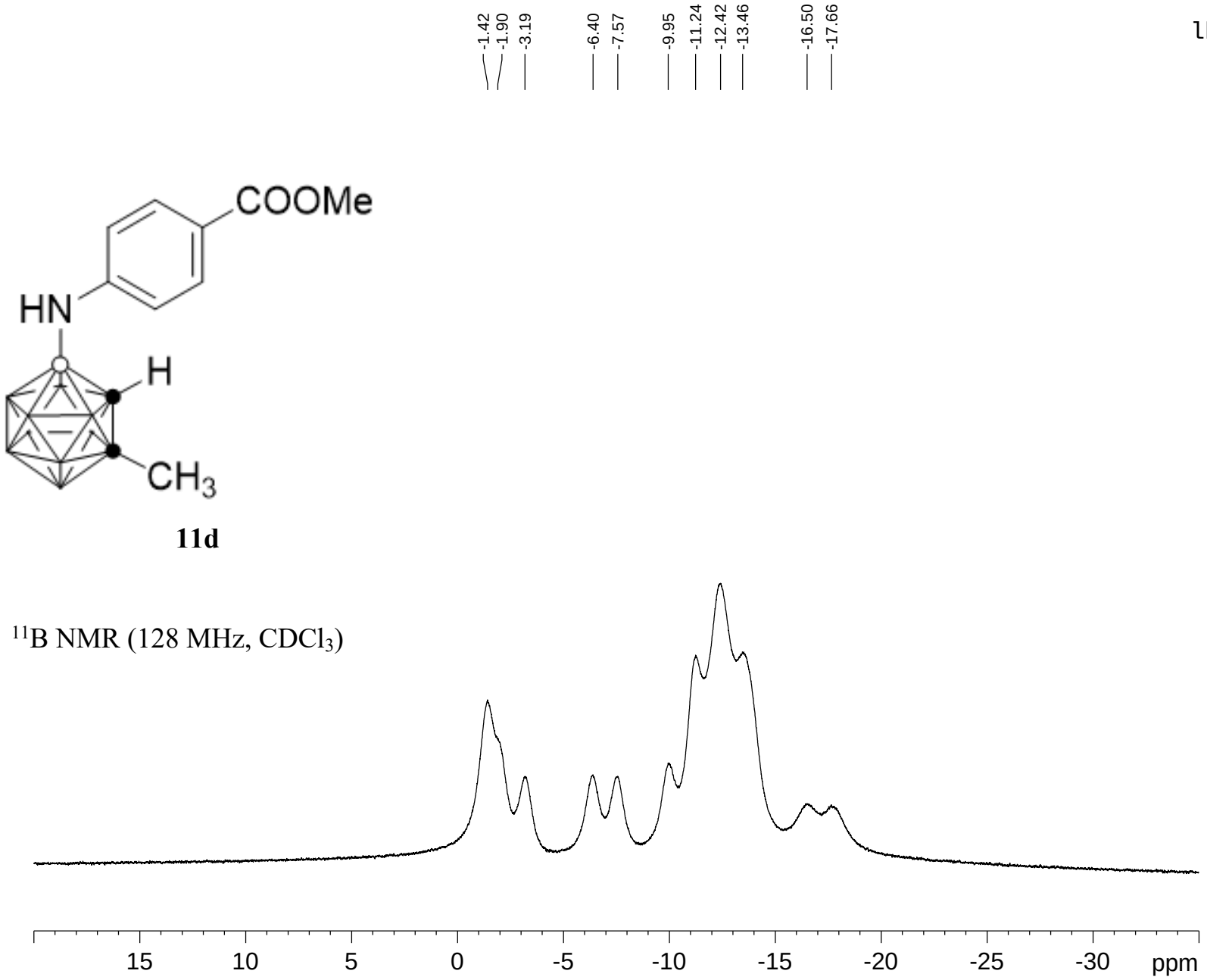
lhr-B-0519-phnhcoome-CDCl3

11d

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      lhr-B-0519-phnhcoome-CDCl3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161031
Time                9.49 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG             zgdc
TD                 65536
SOLVENT             C6D6
NS                     9
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                   287
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
```



11d

^{11}B NMR (128 MHz, $CDCl_3$)

lhr-B-0519-phnhcoome-CDCl3(C)

```
Current Data Parameters
NAME      lhr-B-0519-phnhcoome-CDCl3(C)
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20161031
Time                9.50 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG                zg
TD                  65536
SOLVENT             C6D6
NS                    12
DS                     2
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                   287
DW                20.800 usec
DE                 6.50 usec
TE                295.6 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           128.4097504 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

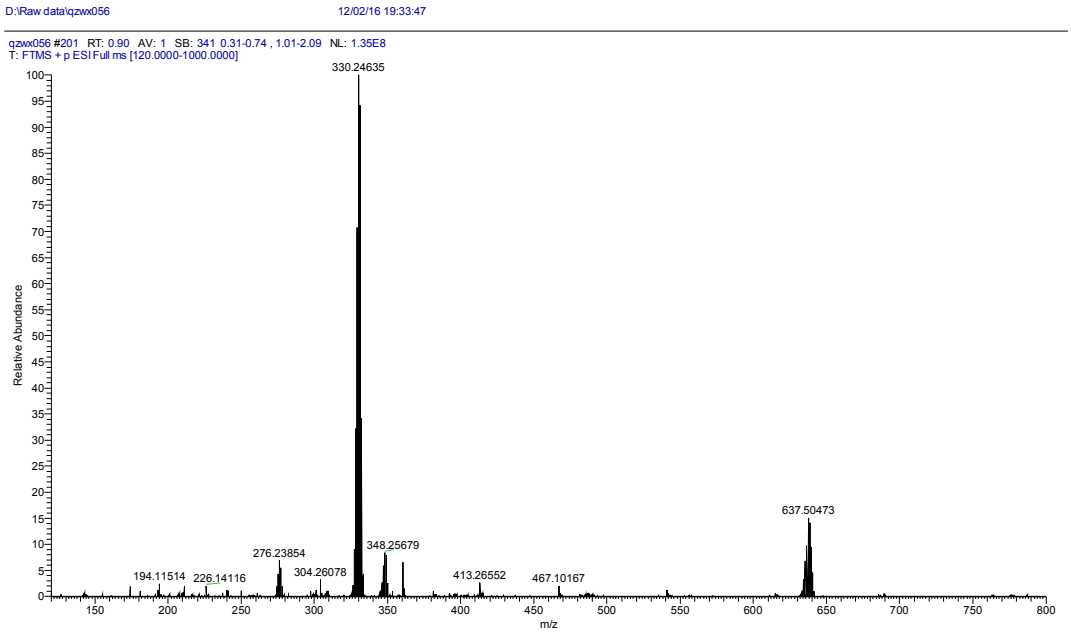
Thermo QEFMS Analysis Report

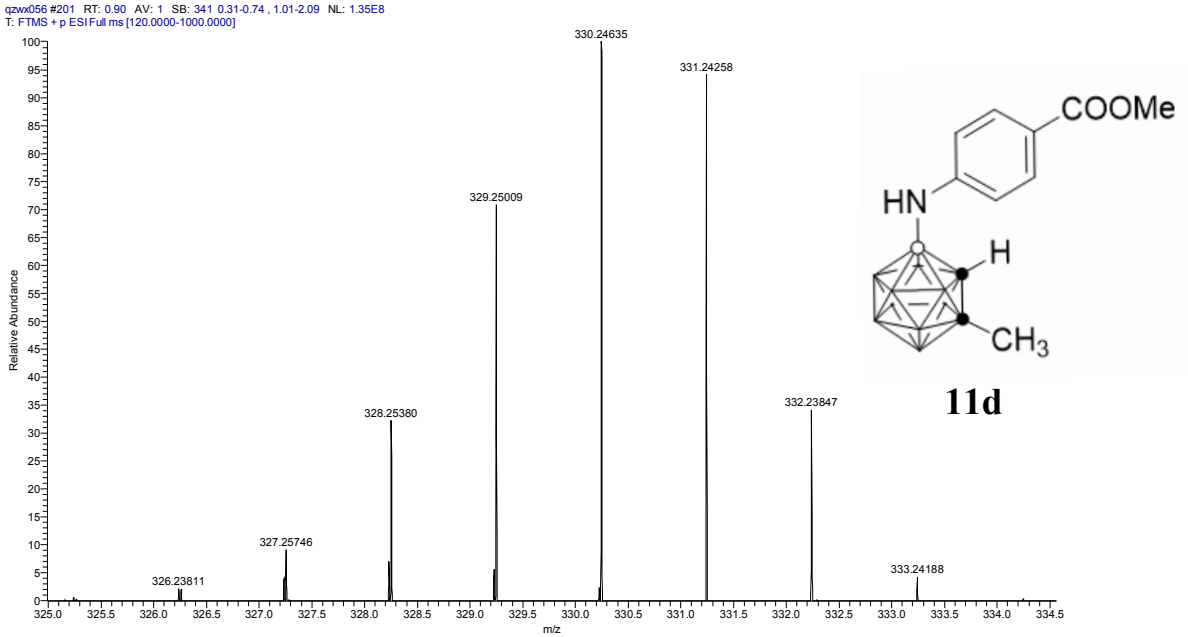
Analysis Info

Sample Name :	Lhr-519	Reference No.:	Qzwx056
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.5kV, by LC, with sheath gas		

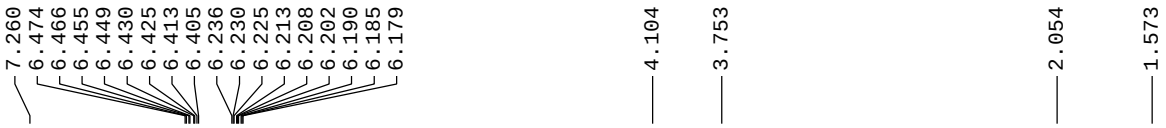
Accurate Mass Measurement

Molecular formula :	$C_{11}H_{21}B_{10}NO_2$
Experimental Mass $[M+Na]^+$:	330.24635
Theoretical Mass $[M+Na]^+$:	330.24677
Error (ppm) :	1.2



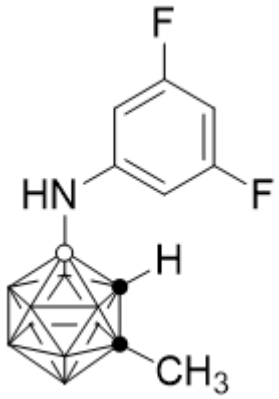


11d



lhr-H-0549-pn35f-CDCl3

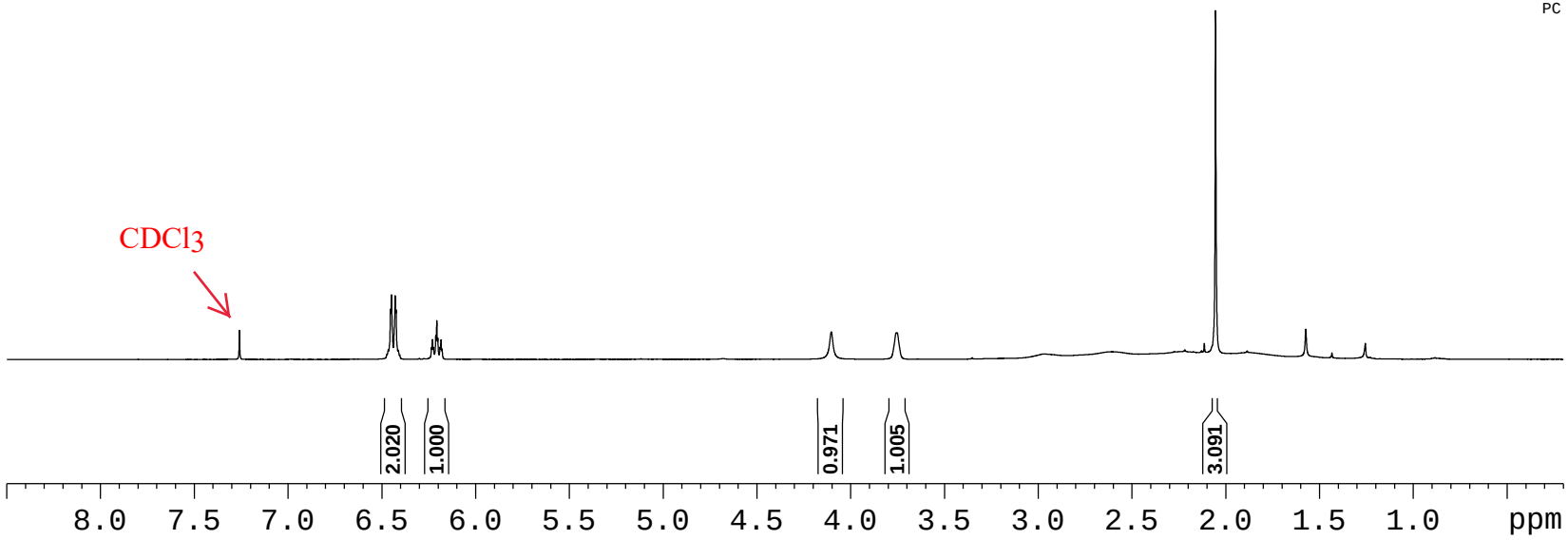
Bruker Advance III 400

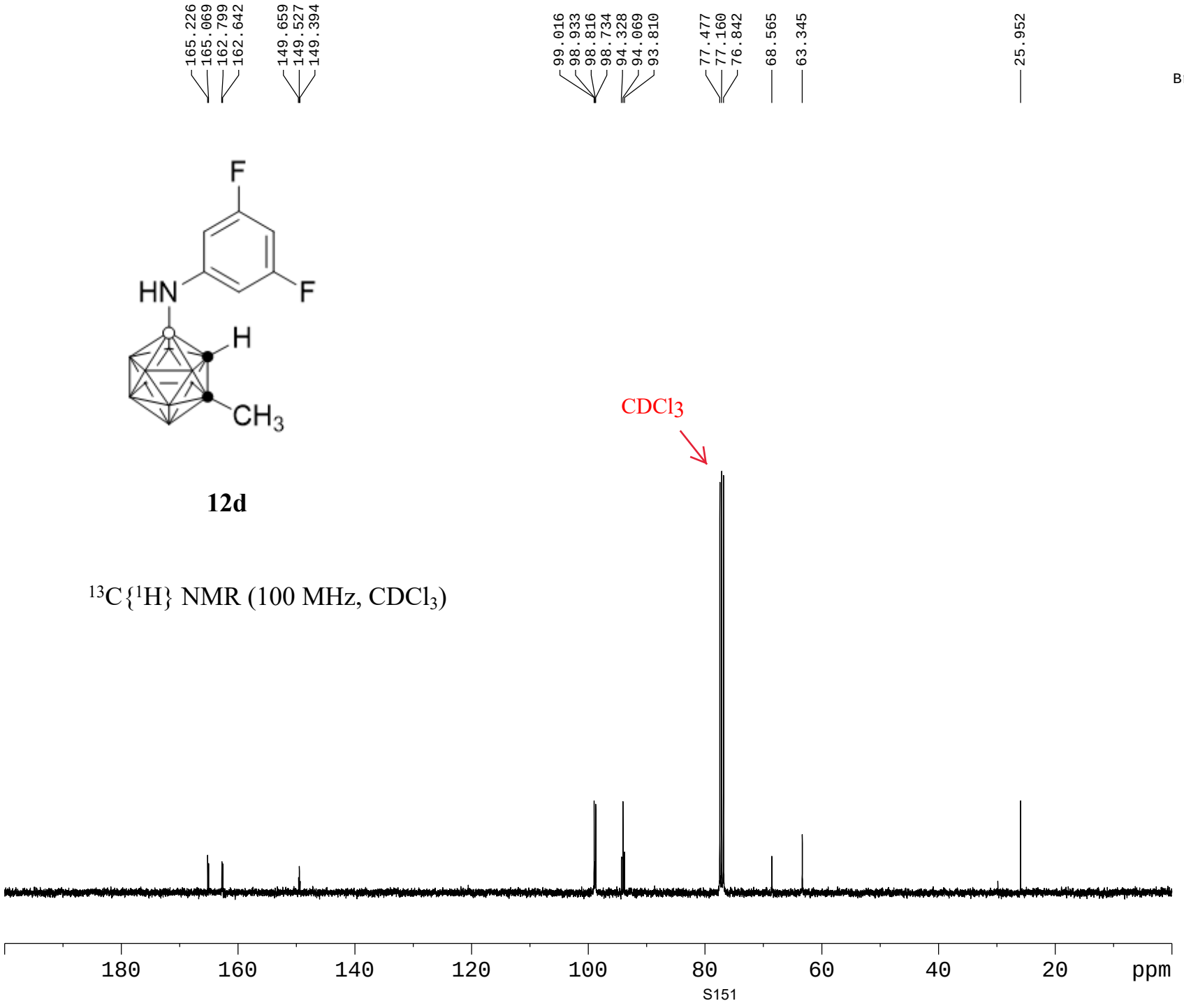


12d

^{1}H NMR (400 MHz, $CDCl_3$)

```
Current Data Parameters
NAME      lhr-H-0549-pn35f-CDCl3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161119
Time              21.10 h
INSTRUM             spect
PROBHD   Z824601_0021 (
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     13
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894465 sec
RG                    101
DW                 62.400 usec
DE                  6.50 usec
TE                 294.8 K
D1            1.00000000 sec
TD0                    1
SFO1         400.1324708 MHz
NUC1                  1H
P1                 15.00 usec
PLW1          8.31000042 W

F2 - Processing parameters
SI                  65536
SF           400.1300098 MHz
WDW                    EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```





12d

$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)

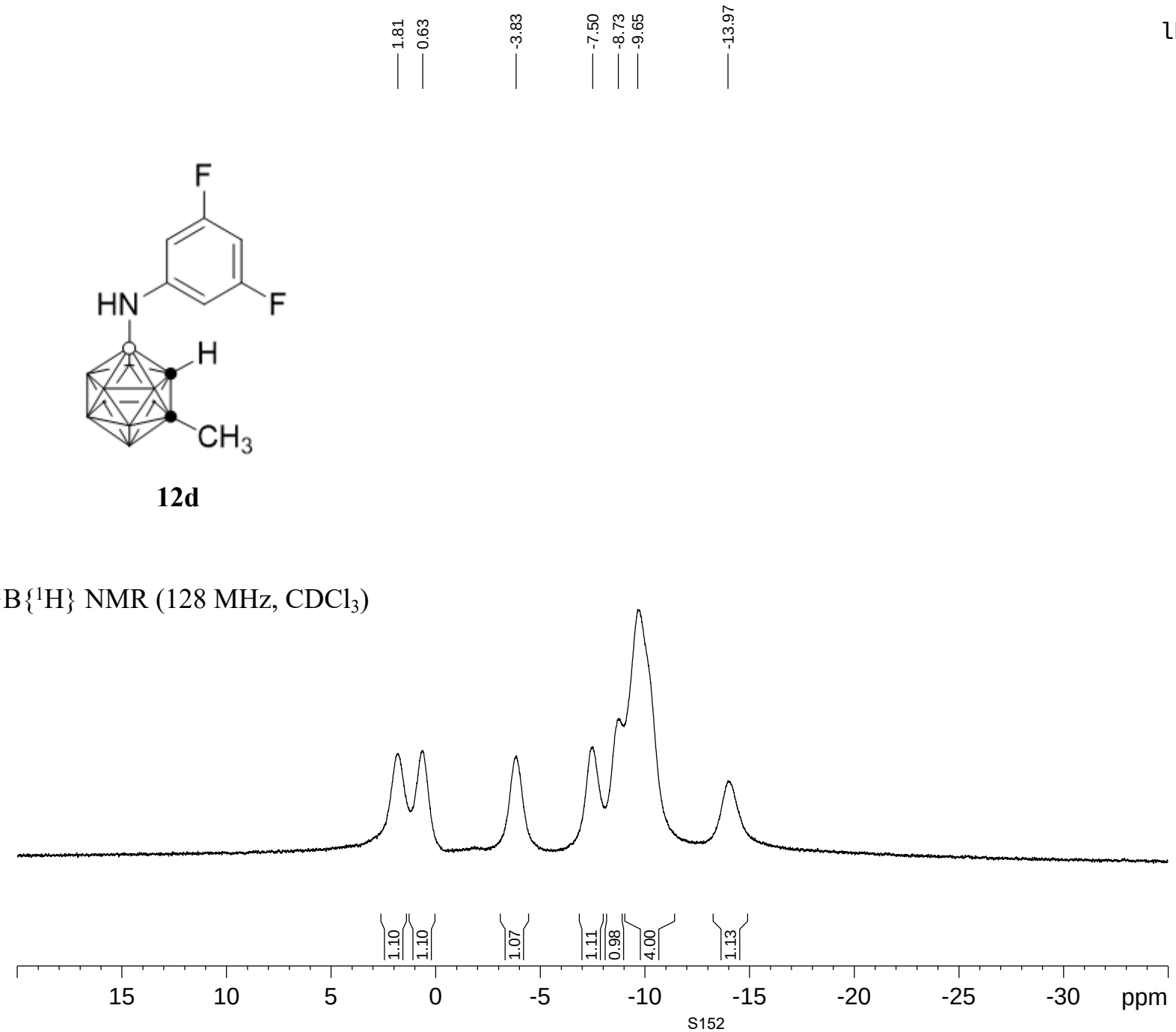
lhr-C-0549-pn35f-CDCl3

Bruker Advance III 400

```
Current Data Parameters
NAME      lhr-C-0549-pn35f-CDCl3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161119
Time              21.12 h
INSTRUM             spect
PROBHD   Z824601_0021 (
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    261
DS                      4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631488 sec
RG                    203
DW                 20.800 usec
DE                  6.50 usec
TE                 294.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1
SFO1         100.6228298 MHz
NUC1                 13C
P1                  9.50 usec
PLW1        41.25000000 W
SFO2        400.1316005 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2         8.31000042 W
PLW12        0.23083000 W
PLW13        0.11611000 W

F2 - Processing parameters
SI                  32768
SF           100.6127567 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

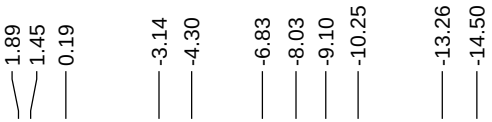
12d

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)



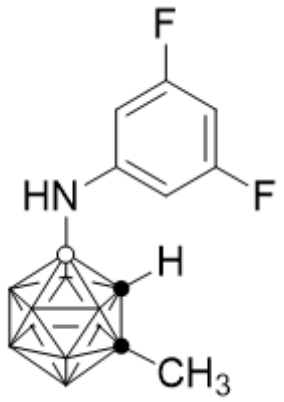
lhr-B-0549-pn35f-CDCl3

```
Current Data Parameters
NAME      lhr-B-0549-pn35f-CDCl3
EXPNO                          1
PROCNO                         1

F2 - Acquisition Parameters
Date_               20161119
Time                   21.31 h
INSTRUM                spect
PROBHD   Z108618_0257 (
PULPROG                 zgdc
TD                     65536
SOLVENT                 C6D6
NS                        12
DS                         4
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                 1.3631488 sec
RG                       512
DW                    20.800 usec
DE                     6.50 usec
TE                    296.6 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
```



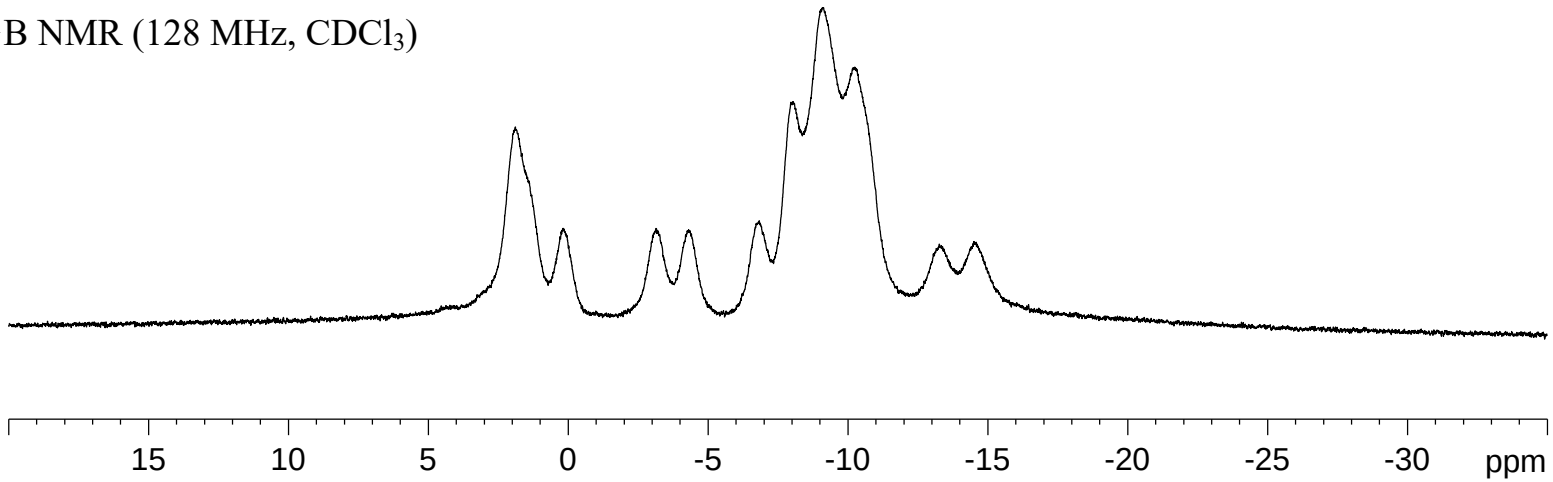
lhr-B-0549-pn35f-CDCl3(C)

```
Current Data Parameters
NAME      lhr-B-0549-pn35f-CDCl3(C)
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20161119
Time              21.33 h
INSTRUM           spect
PROBHD   Z108618_0257 (
PULPROG             zg
TD                65536
SOLVENT            C6D6
NS                   16
DS                    2
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                  512
DW                20.800 usec
DE                 6.50 usec
TE                296.3 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          128.4097504 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



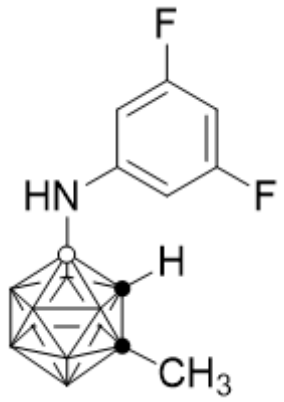
12d

^{11}B NMR (128 MHz, $CDCl_3$)



-106.83

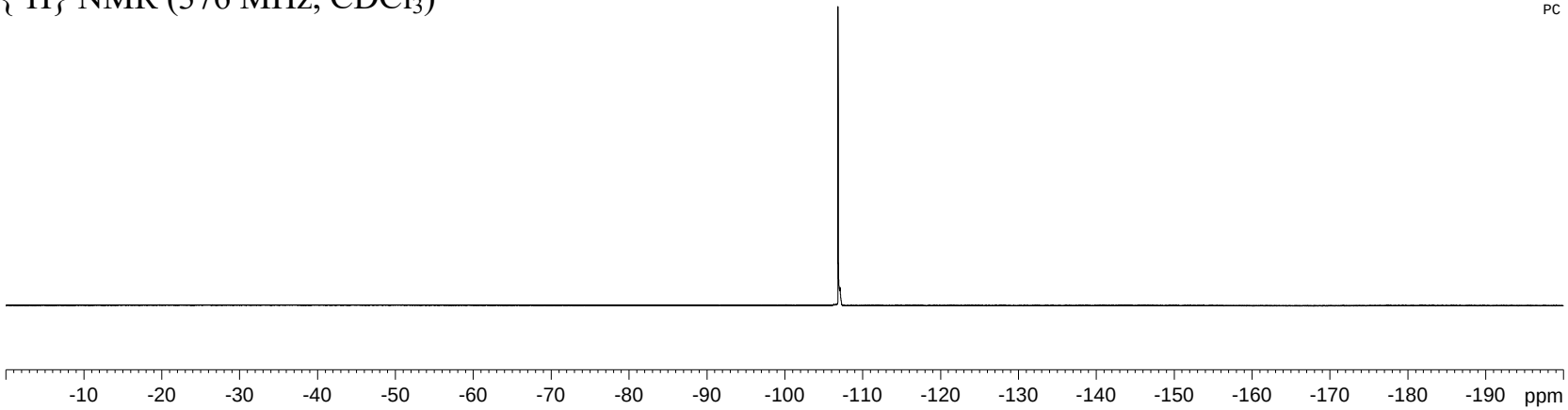
lhr-F-0549-pn35f-CDC



12d

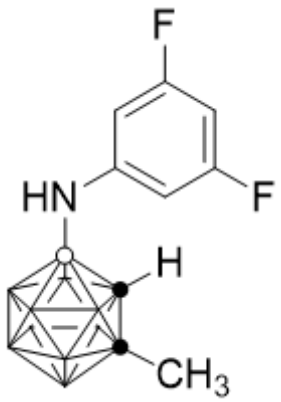
$^{19}F\{^{1}H\}$ NMR (376 MHz, $CDCl_3$)

```
Current Data Parameters
NAME      lhr-F-0549-pn35f-CDCl3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161119
Time              21.35 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG       zgfhigqn.2
TD                131072
SOLVENT             C6D6
NS                     8
DS                     4
SWH            89285.711 Hz
FIDRES          0.681196 Hz
AQ             0.7340032 sec
RG                   645
DW                 5.600 usec
DE                 6.50 usec
TE                296.2 K
D1           1.00000000 sec
D11          0.03000000 sec
D12          0.00002000 sec
TD0                    1
SFO1         376.5548010 MHz
NUC1                 19F
P1                14.70 usec
PLW1         18.36000061 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                 65536
SF           376.5924602 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



-106.82

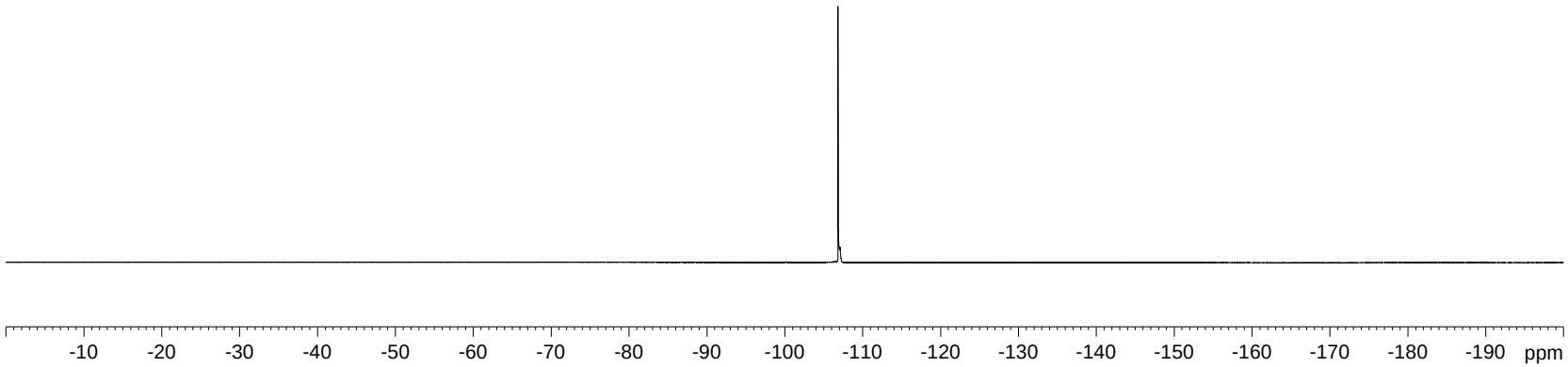
lhr-F-0549-pn35f-CDC



12d

^{19}F NMR (376 MHz, $CDCl_3$)

```
Current Data Parameters
NAME      lhr-F-0549-pn35f-CDCl3(C)
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20161119
Time              21.35 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG       zgfhigqn.2
TD                131072
SOLVENT             C6D6
NS                    16
DS                     4
SWH            89285.711 Hz
FIDRES          0.681196 Hz
AQ              0.7340032 sec
RG                   645
DW                 5.600 usec
DE                 6.50 usec
TE                296.3 K
D1           1.00000000 sec
D11          0.03000000 sec
D12          0.00002000 sec
TD0                    1
SFO1         376.5548010 MHz
NUC1                 19F
P1                14.70 usec
PLW1         18.36000061 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                 65536
SF           376.5924602 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



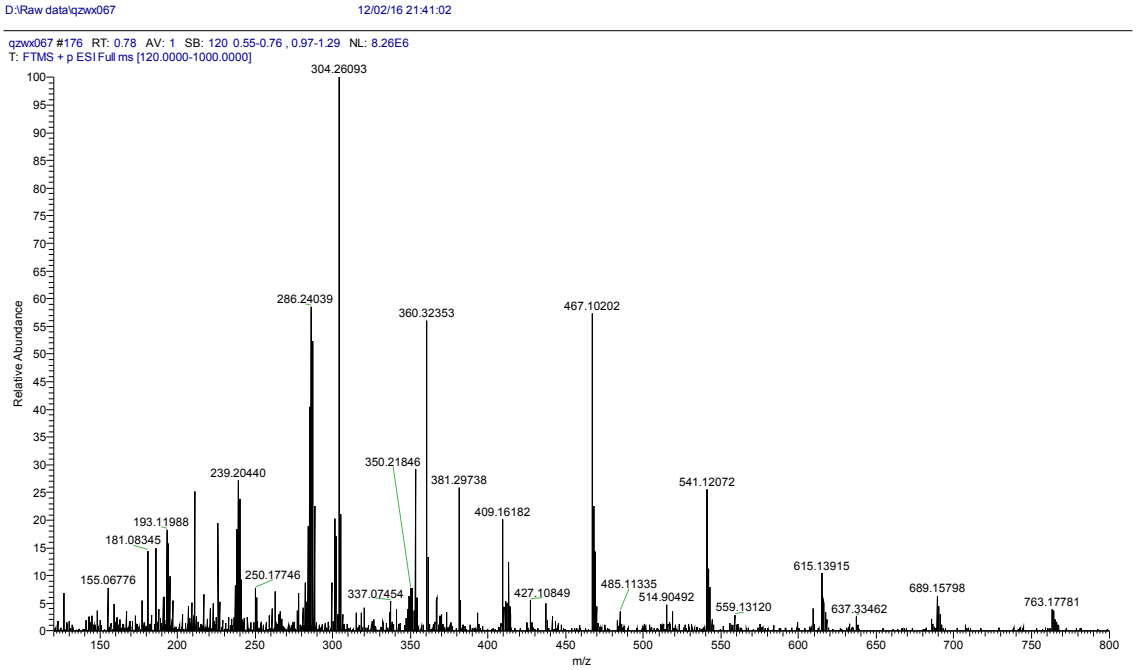
Thermo QEFMS Analysis Report

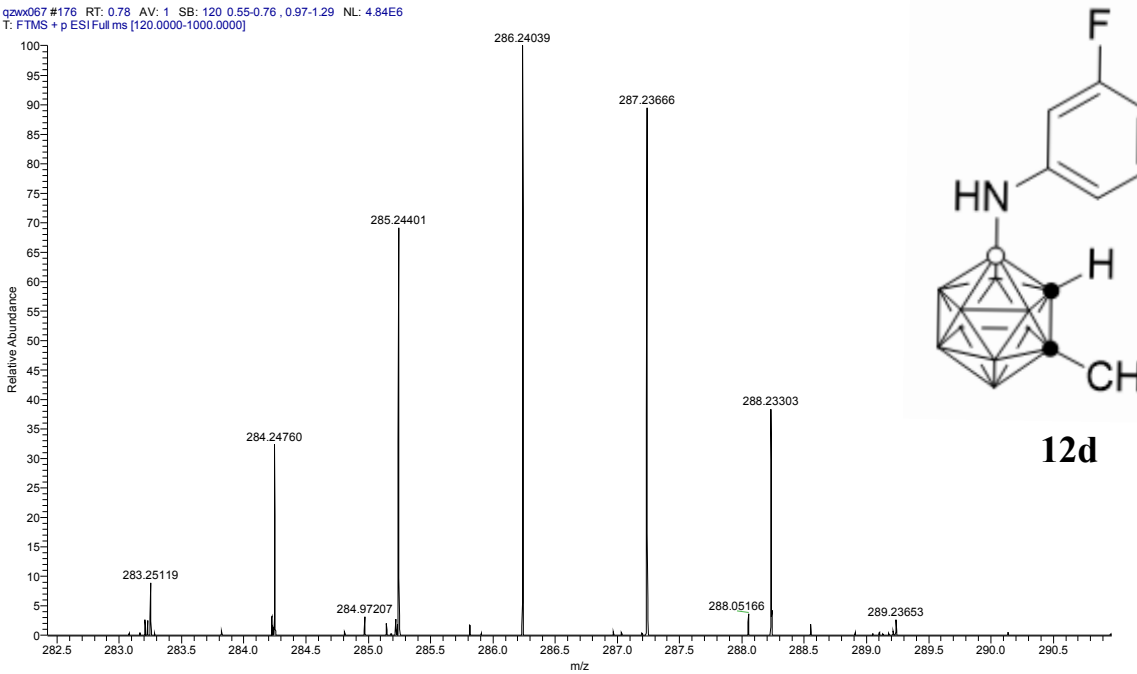
Analysis Info

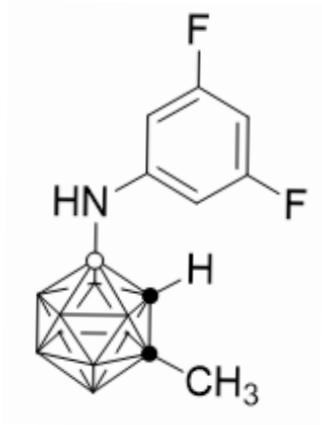
Sample Name :	Lhr-549	Reference No.:	Qzwx067
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.5kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_9H_{17}B_{10}F_2N$
Experimental Mass $[M+H]^+$:	286.24039
Theoretical Mass $[M+H]^+$:	286.24050
Error (ppm) :	0.3





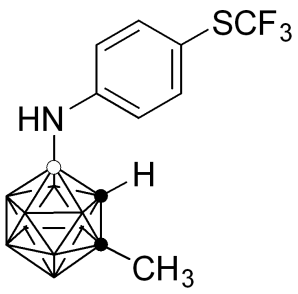


12d

7.440
7.418
7.260
6.960
6.938
4.189
3.760
2.051

lhr-H-0548-pnscf3-CDCl3

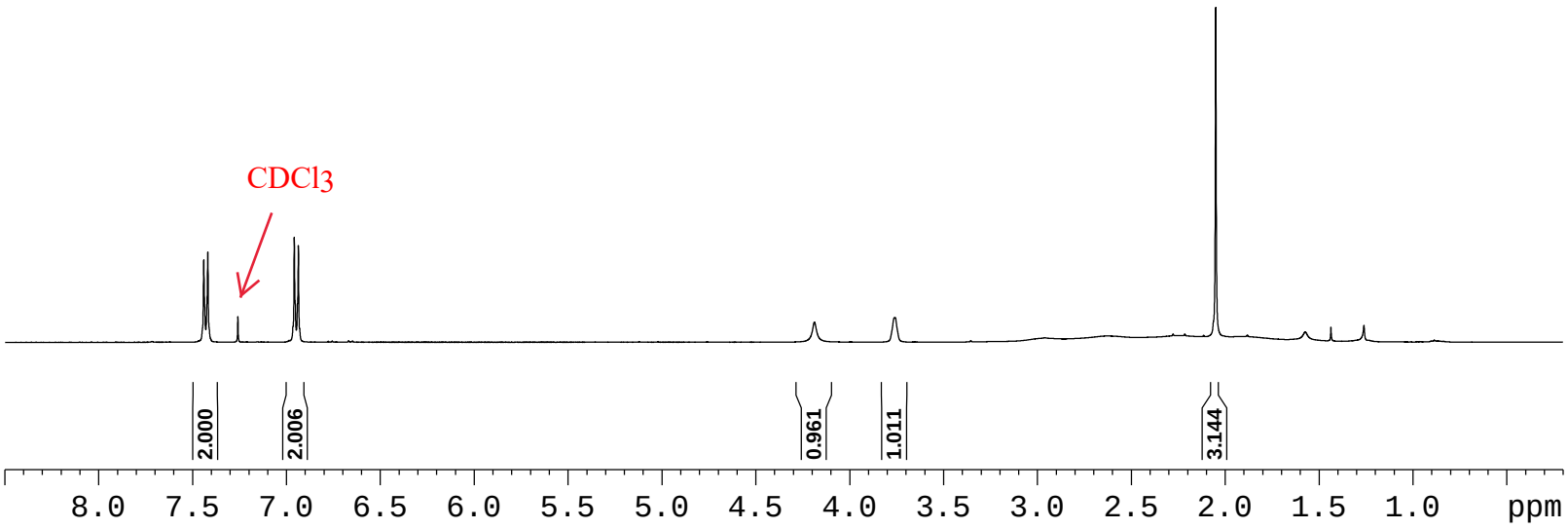
Bruker Advance III 400



13d

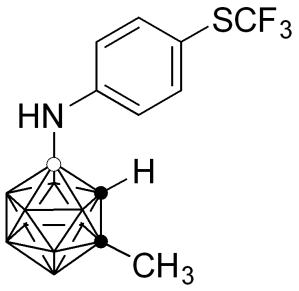
^{1}H NMR (400 MHz, $CDCl_3$)

```
Current Data Parameters
NAME      lhr-H-0548-pnscf3-CDCl3
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20161118
Time              19.11 h
INSTRUM           spect
PROBHD   Z824601_0021 (
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  10
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               295.0 K
D1            1.00000000 sec
TD0                  1
SFO1         400.1324708 MHz
NUC1                1H
P1               15.00 usec
PLW1          8.31000042 W

F2 - Processing parameters
SI               65536
SF           400.1300097 MHz
WDW                 EM
SSB      0
LB               0.30 Hz
GB       0
PC               1.00
```



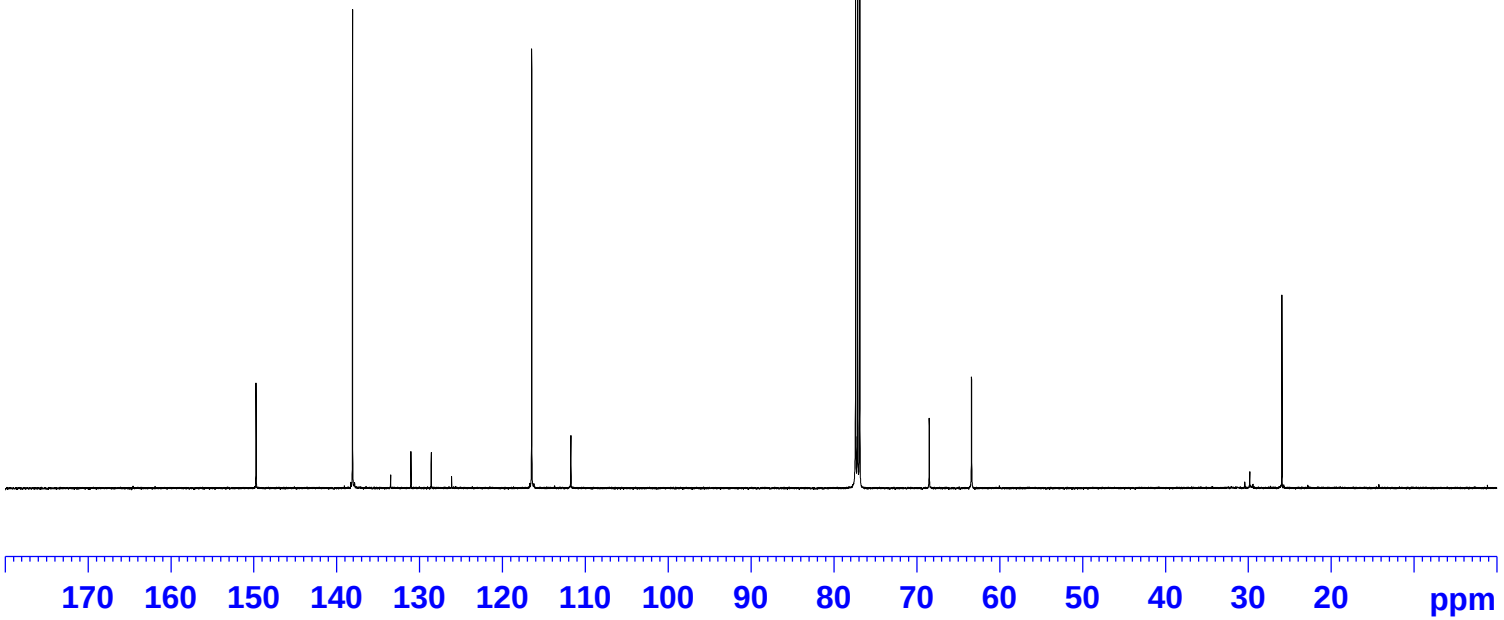
lhr-H-0548-re-cdcl3

149.73
138.13
133.53
131.08
128.62
126.17
116.50
111.79
111.77
77.41
77.16
76.91
68.52
63.42
25.96



13d

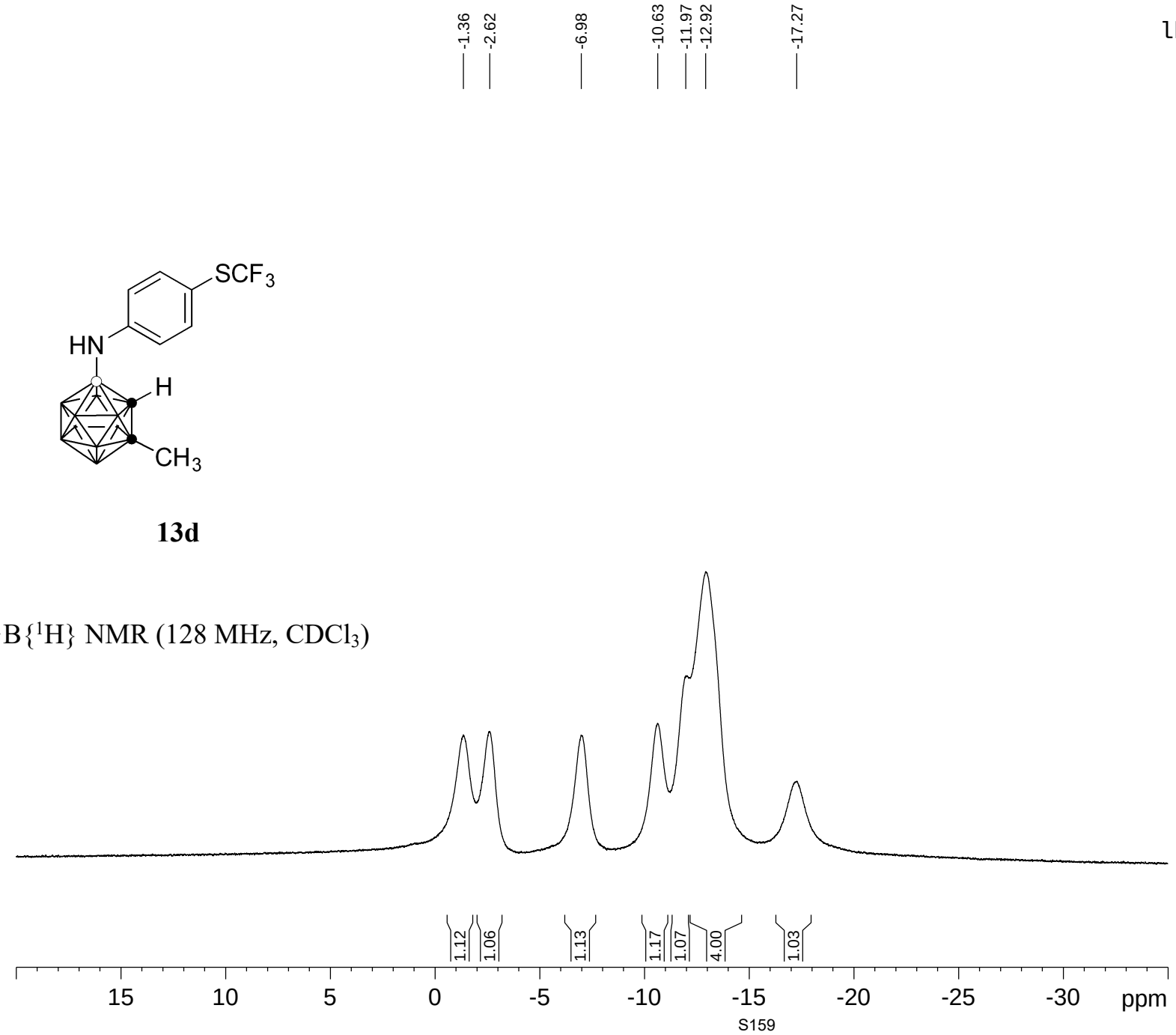
$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      lhr-H-0548-re-cdcl3
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20190403
Time                 11.01 h
INSTRUM                spect
PROBHD    Z149001_0010 (
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                   18.00 usec
TE                   298.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                       1
SFO1           125.7703643 MHz
NUC1                    13C
P1                   10.00 usec
PLW1           61.00000000 W
SFO2           500.1320005 MHz
NUC2                    1H
CPDPRG[2             waltz16
PCPD2                80.00 usec
PLW2           15.00000000 W
PLW12           0.29663000 W
PLW13           0.14920001 W

F2 - Processing parameters
SI                    32768
SF             125.7577735 MHz
WDW                      EM
SSB         0
LB                    1.00 Hz
GB          0
PC                    1.40
```

13d

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)



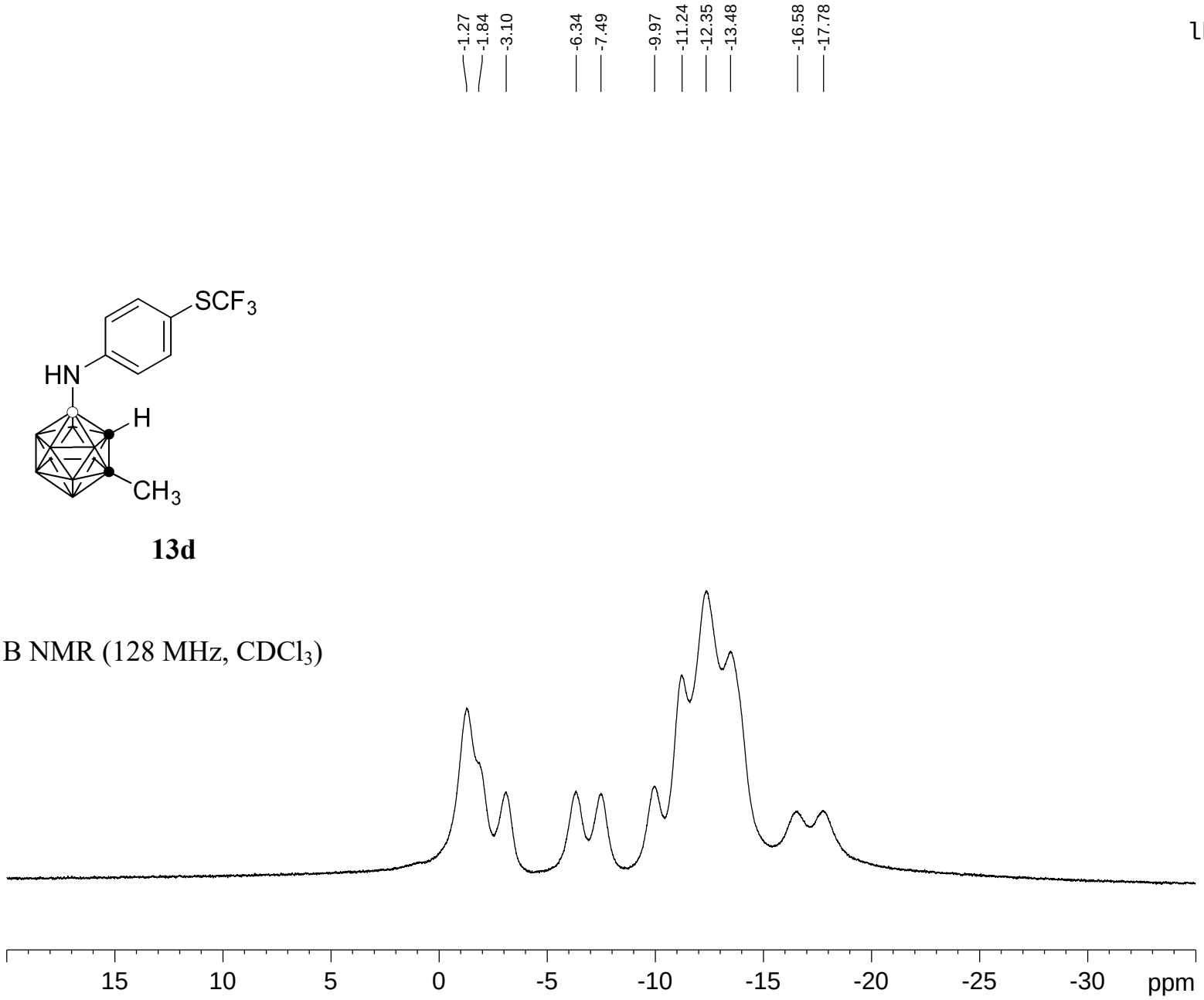
lhr-B-0548-pnscf3-CDCl3

```
Current Data Parameters
NAME      lhr-B-0548-pnscf3-CDCl3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161119
Time                9.40 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                    14
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                   287
DW                20.800 usec
DE                 6.50 usec
TE                296.5 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
```

lhr-B-0548-pnscf3-CDCl3(C)

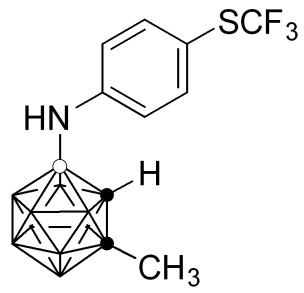
13d

^{11}B NMR (128 MHz, $CDCl_3$)



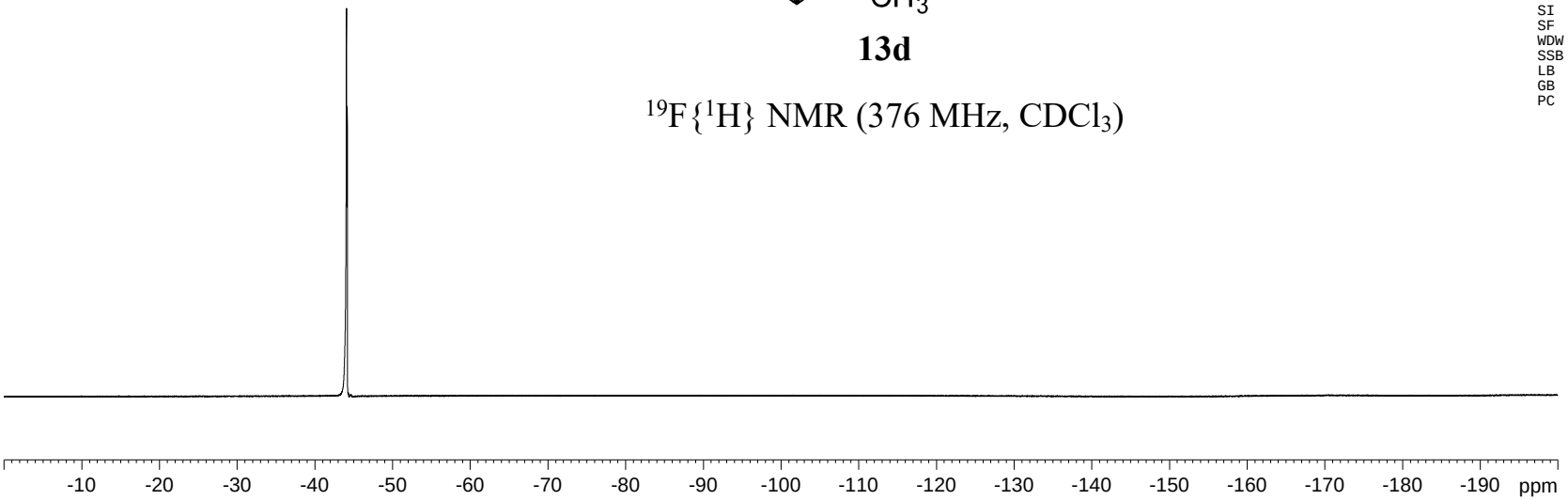

```
Current Data Parameters
NAME      lhr-B-0548-pnscf3-CDCl3(C)
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20161119
Time               9.41 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG              zg
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                  203
DW                20.800 usec
DE                 6.50 usec
TE                296.1 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          128.4097504 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

-44.09



13d

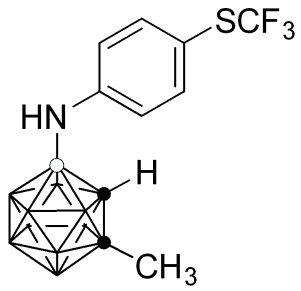
$^{19}F\{^{1}H\}$ NMR (376 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      lhr-F-0548-pnscf3-CDCl3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161119
Time                9.44 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG       zgfhigqn.2
TD                131072
SOLVENT             CDCl3
NS                     25
DS                      4
SWH             89285.711 Hz
FIDRES           0.681196 Hz
AQ               0.7340032 sec
RG                    645
DW                  5.600 usec
DE                  6.50 usec
TE                  296.1 K
D1            1.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec
TD0                    1
SFO1         376.5548010 MHz
NUC1                 19F
P1                 14.70 usec
PLW1         18.36000061 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                 65536
SF           376.5924602 MHz
WDW                  EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

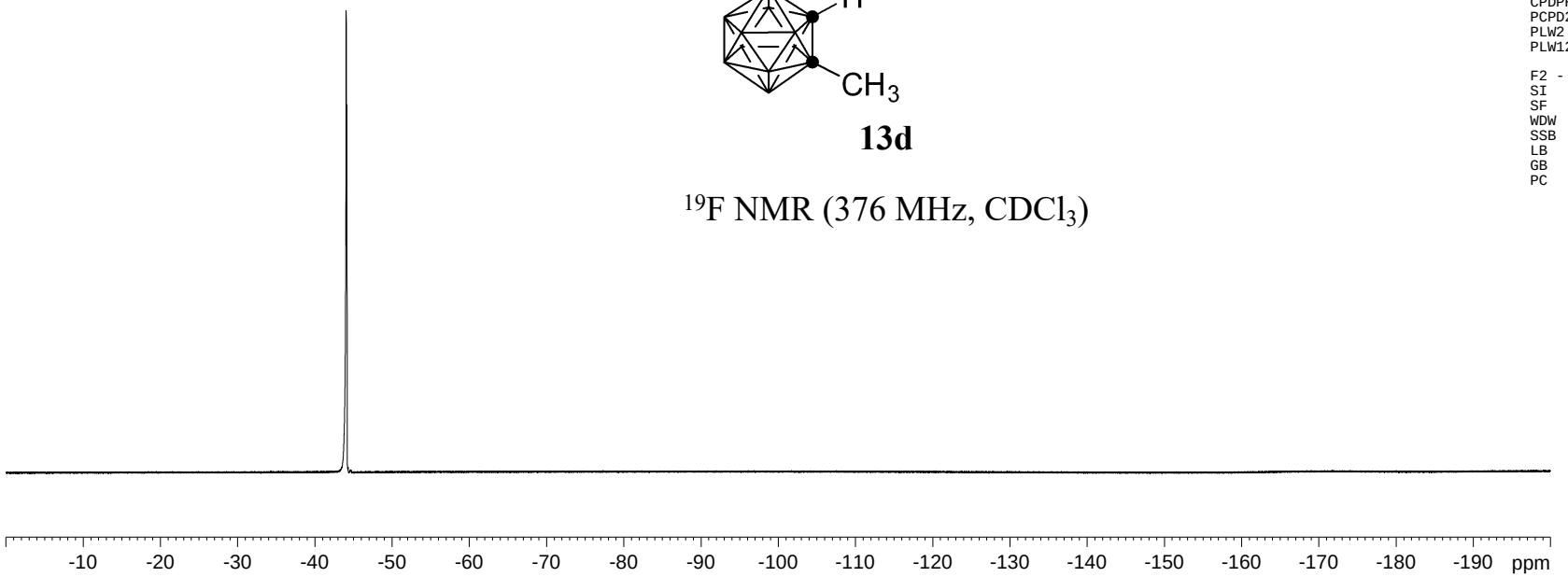
-44.08

```
Current Data Parameters
NAME      lhr-F-0548-pnscf3-CDCl3(C)
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161119
Time                9.45 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG       zgfhigqn.2
TD                131072
SOLVENT            CDCl3
NS                    16
DS                     4
SWH            89285.711 Hz
FIDRES          0.681196 Hz
AQ              0.7340032 sec
RG                   645
DW                 5.600 usec
DE                 6.50 usec
TE                296.2 K
D1           1.00000000 sec
D11          0.03000000 sec
D12          0.00002000 sec
TD0                    1
SFO1         376.5548010 MHz
NUC1                 19F
P1                 14.70 usec
PLW1         18.36000061 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                 65536
SF           376.5924602 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



13d

^{19}F NMR (376 MHz, $CDCl_3$)



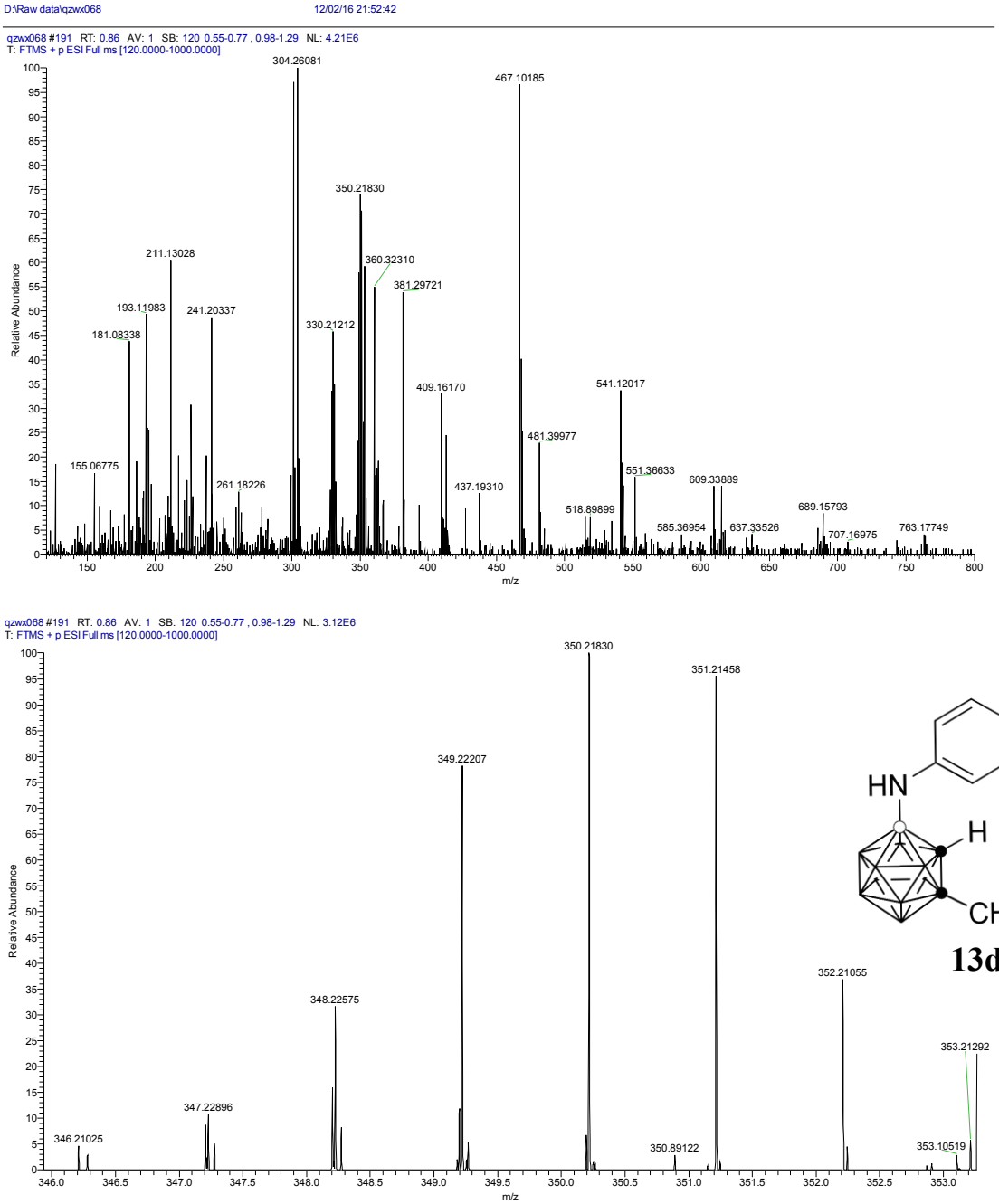
Thermo QEFMS Analysis Report

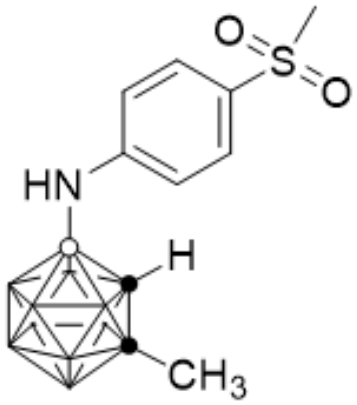
Analysis Info

Sample Name :	Lhr-548	Reference No.:	Qzwx068
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.5kV, by LC, with sheath gas		

Accurate Mass Measurement

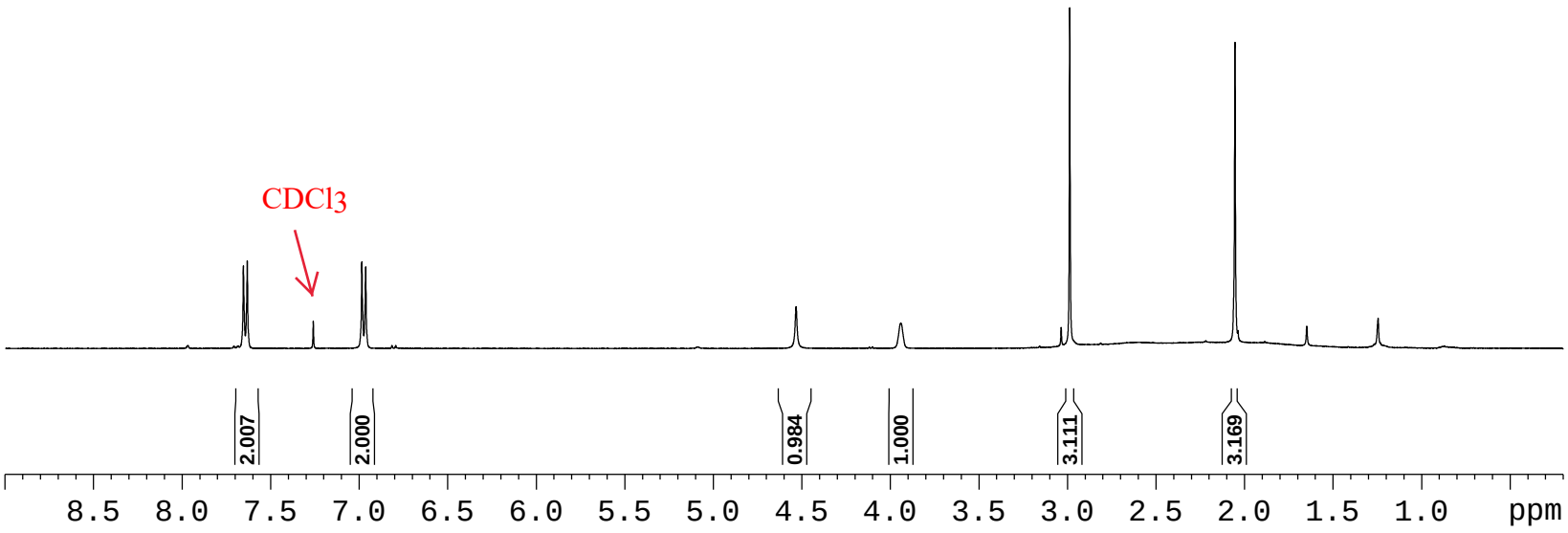
Molecular formula :	$C_{10}H_{18}B_{10}F_3NS$
Experimental Mass $[M+H]^+$:	350.21830
Theoretical Mass $[M+H]^+$:	350.21886
Error (ppm) :	1.6





14d

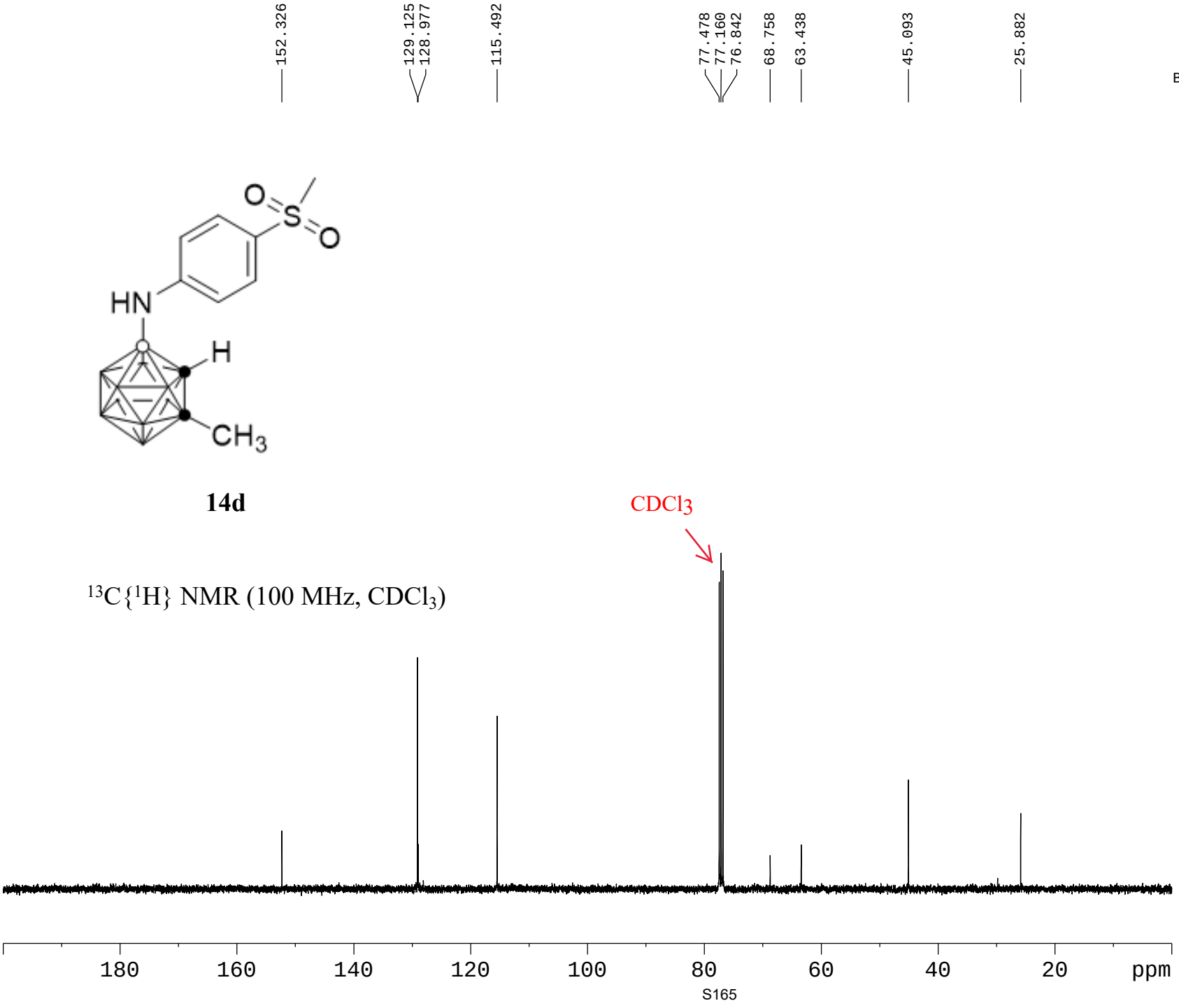
^{1}H NMR (400 MHz, $CDCl_3$)



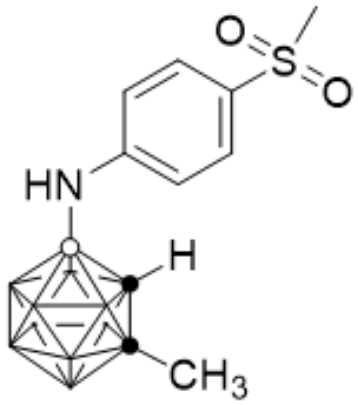

```
lhr-H-0550-pnsooc-CDCl3

Bruker Advance III 400

Current Data Parameters
NAME      lhr-H-0550-pnsooc-CDCl3
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161123
Time              16.25 h
INSTRUM            spect
PROBHD   Z824601_0021 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    12
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                     1
DW                62.400 usec
DE                 6.50 usec
TE                295.2 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1324708 MHz
NUC1                  1H
P1                15.00 usec
PLW1           8.31000042 W

F2 - Processing parameters
SI                 65536
SF            400.1300098 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

14d

$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)

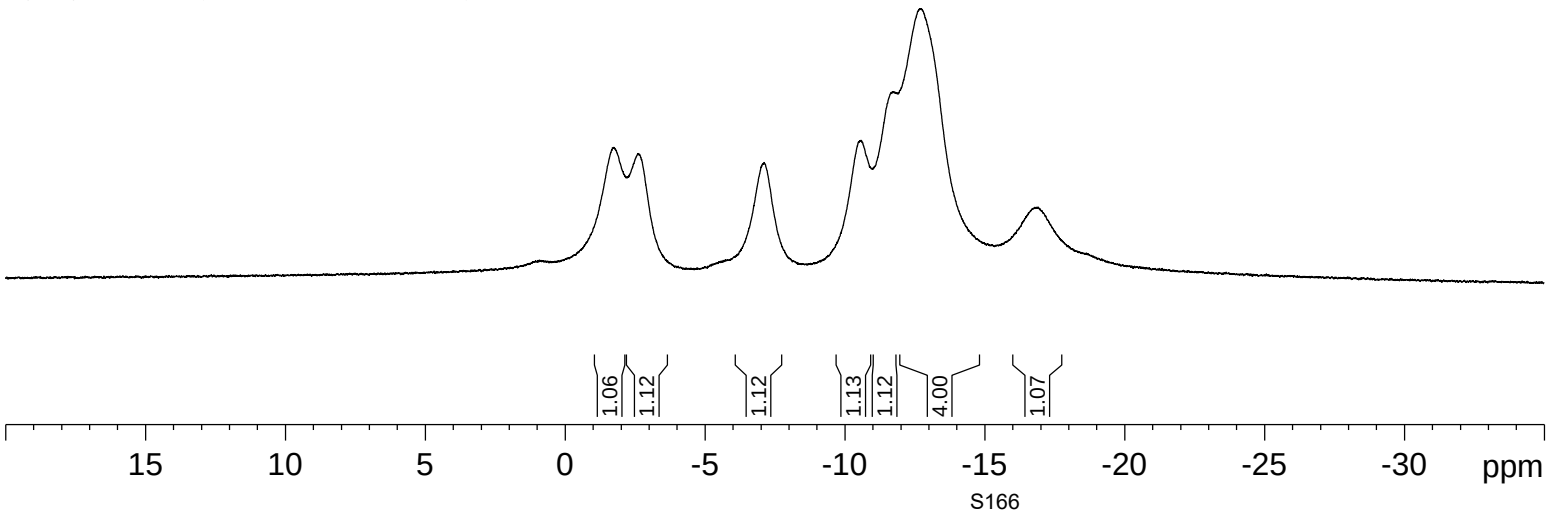



```
lhr-C-0550-pnsooc-CDCl3

Bruker Advance III 400

Current Data Parameters
NAME      lhr-C-0550-pnsooc-CDCl3
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161123
Time              16.28 h
INSTRUM             spect
PROBHD   Z824601_0021 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                  167
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                  203
DW               20.800 usec
DE                6.50 usec
TE               295.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P1                 9.50 usec
PLW1        41.25000000 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         8.31000042 W
PLW12        0.23083000 W
PLW13        0.11611000 W

F2 - Processing parameters
SI                32768
SF          100.6127582 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



14d

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)

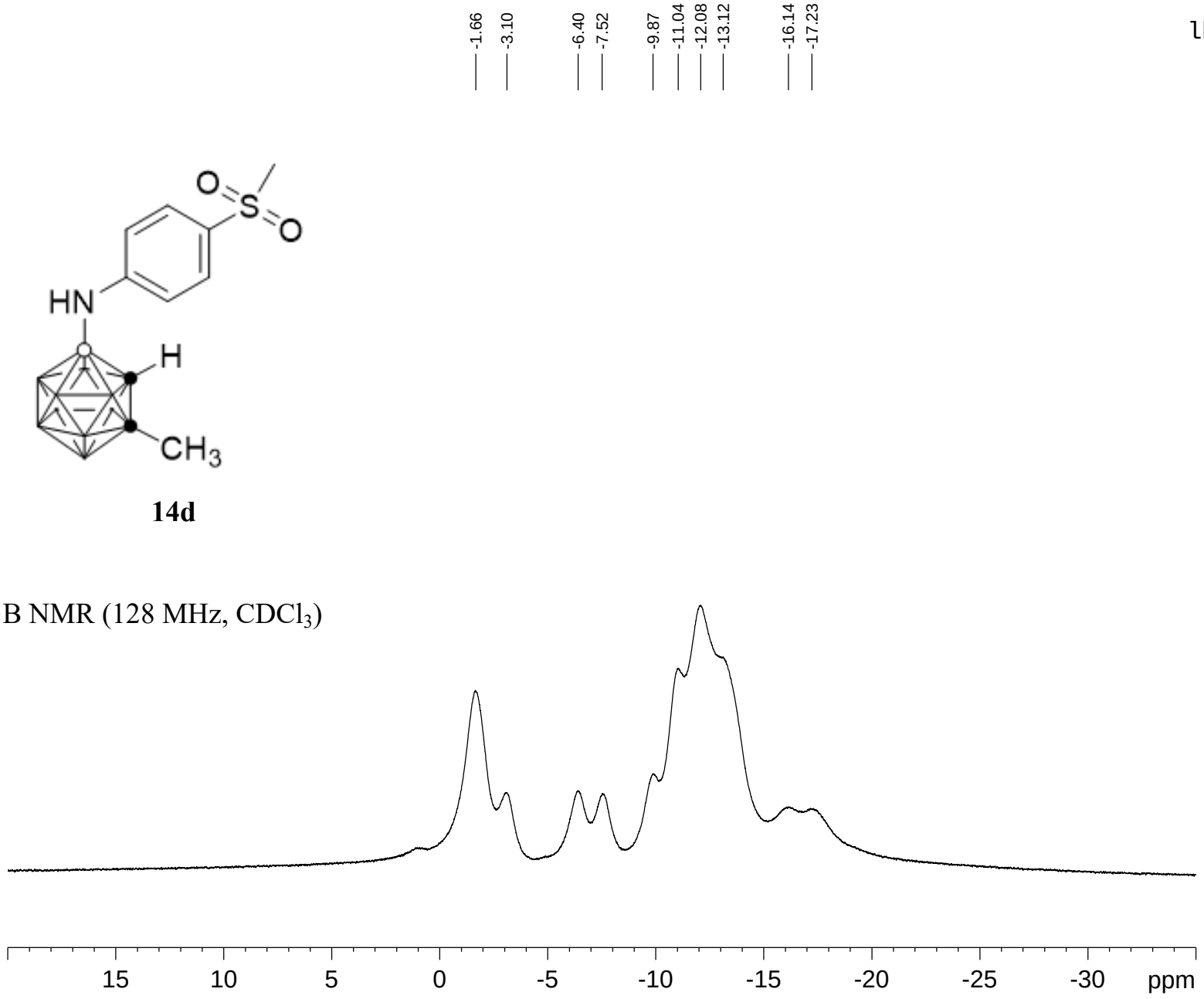


lhr-B-0550-pnsooc-CDCl3

```
Current Data Parameters
NAME      lhr-B-0550-pnsooc-CDCl3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161123
Time               16.41 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG              zgdc
TD                  65536
SOLVENT             CDCl3
NS                     21
DS                      4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                    287
DW                 20.800 usec
DE                  6.50 usec
TE                 296.4 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
```

14d

^{11}B NMR (128 MHz, $CDCl_3$)



lhr-B-0550-pnsooc-CDCl3(

```
Current Data Parameters
NAME      lhr-B-0550-pnsooc-CDCl3(C)
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161123
Time              16.44 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG               zg
TD                 65536
SOLVENT            CDCl3
NS                    30
DS                     2
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                   287
DW                20.800 usec
DE                 6.50 usec
TE                296.0 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           128.4097504 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

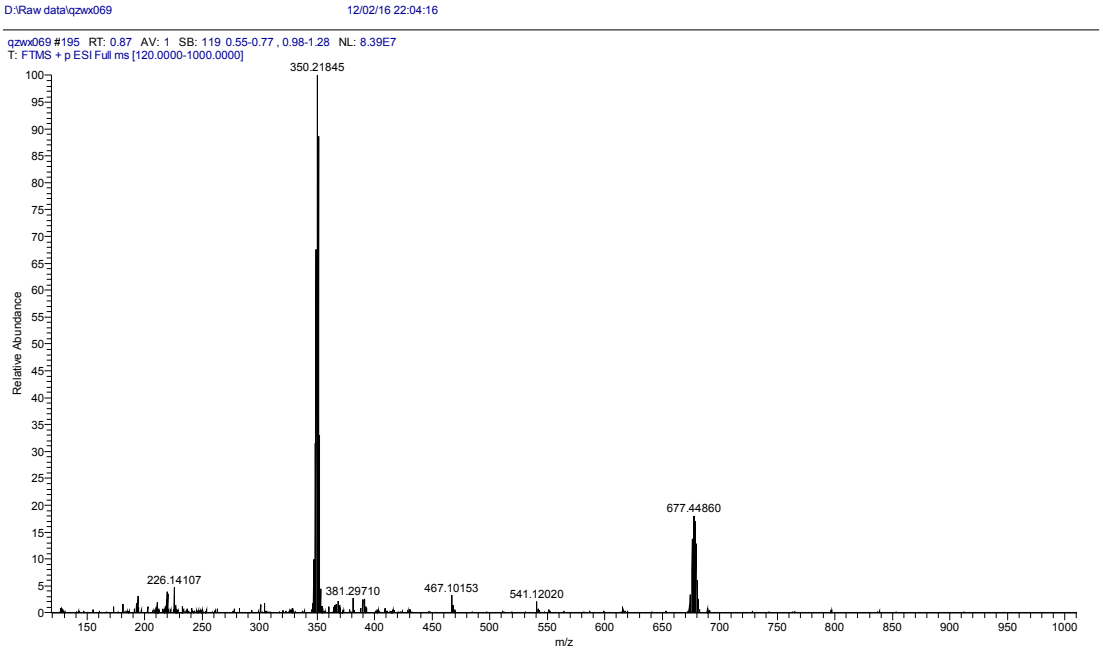
Thermo QEFMS Analysis Report

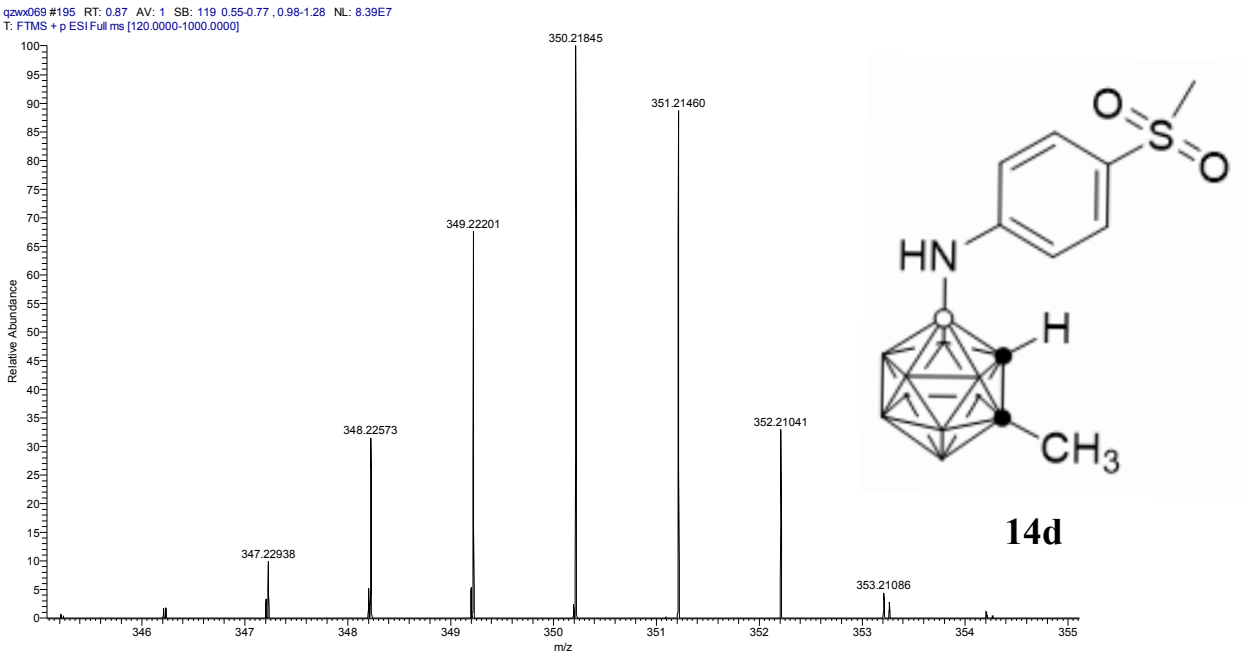
Analysis Info

Sample Name :	Lhr-550	Reference No.:	Qzwx069
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.5kV, by LC, with sheath gas		

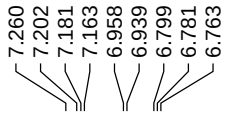
Accurate Mass Measurement

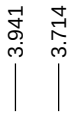
Molecular formula :	$C_{10}H_{21}B_{10}NO_2S$
Experimental Mass $[M+Na]^+$:	350.21845
Theoretical Mass $[M+Na]^+$:	350.21890
Error (ppm) :	1.2

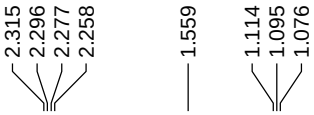




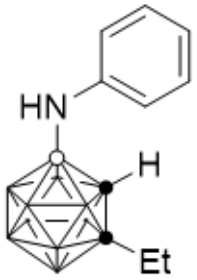
14d







lhr-H-0522-Etphnh-C



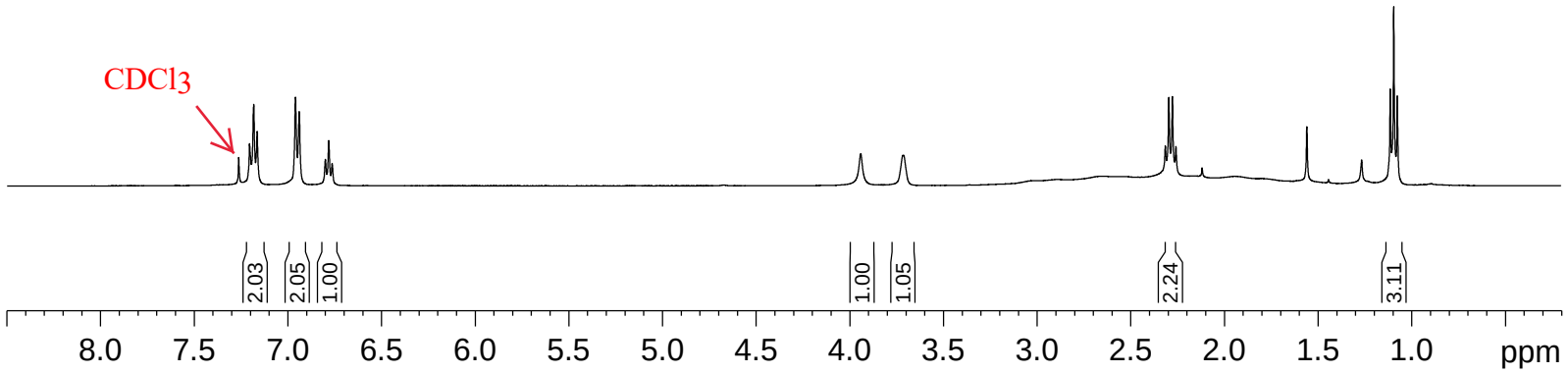
15d

^{1}H NMR (400 MHz, $CDCl_3$)

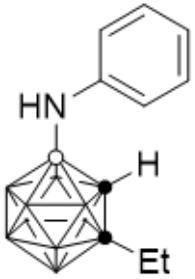
Current Data Parameters
NAME lhr-H-0522-Etphnh-CD
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161102
Time 9.53 h
INSTRUM spect
PROBHD Z108618_0257 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 10
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 90.5
DW 62.400 usec
DE 6.50 usec
TE 295.9 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 400.2300110 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

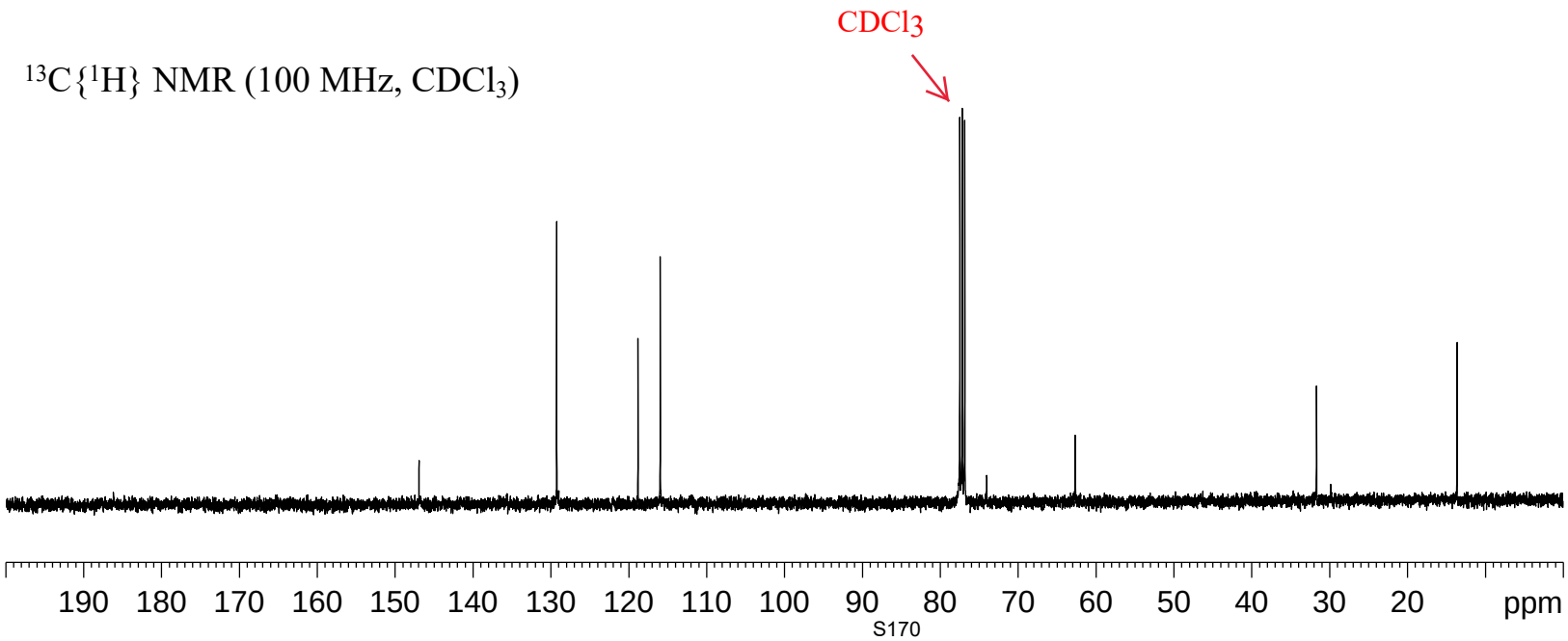


lhr-C-0522-Etphnh-(

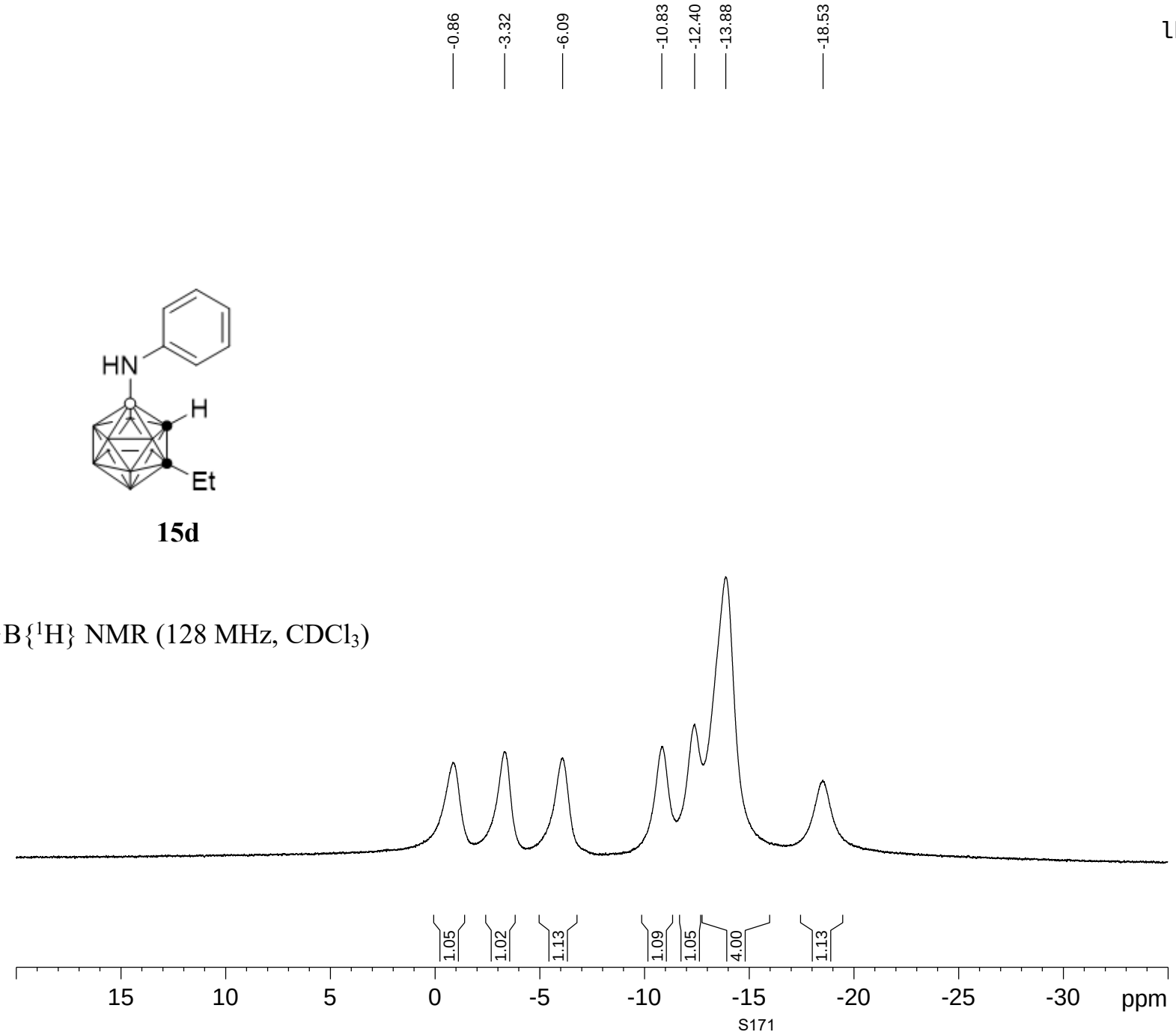


15d

$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      lhr-C-0522-Etphnh-CDCl
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161102
Time               9.56 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  120
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG                  161
DW               16.800 usec
DE                6.50 usec
TE                296.2 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          100.6379029 MHz
WDW                EM
SSB       0
LB               1.00 Hz
GB        0
PC               1.40
```

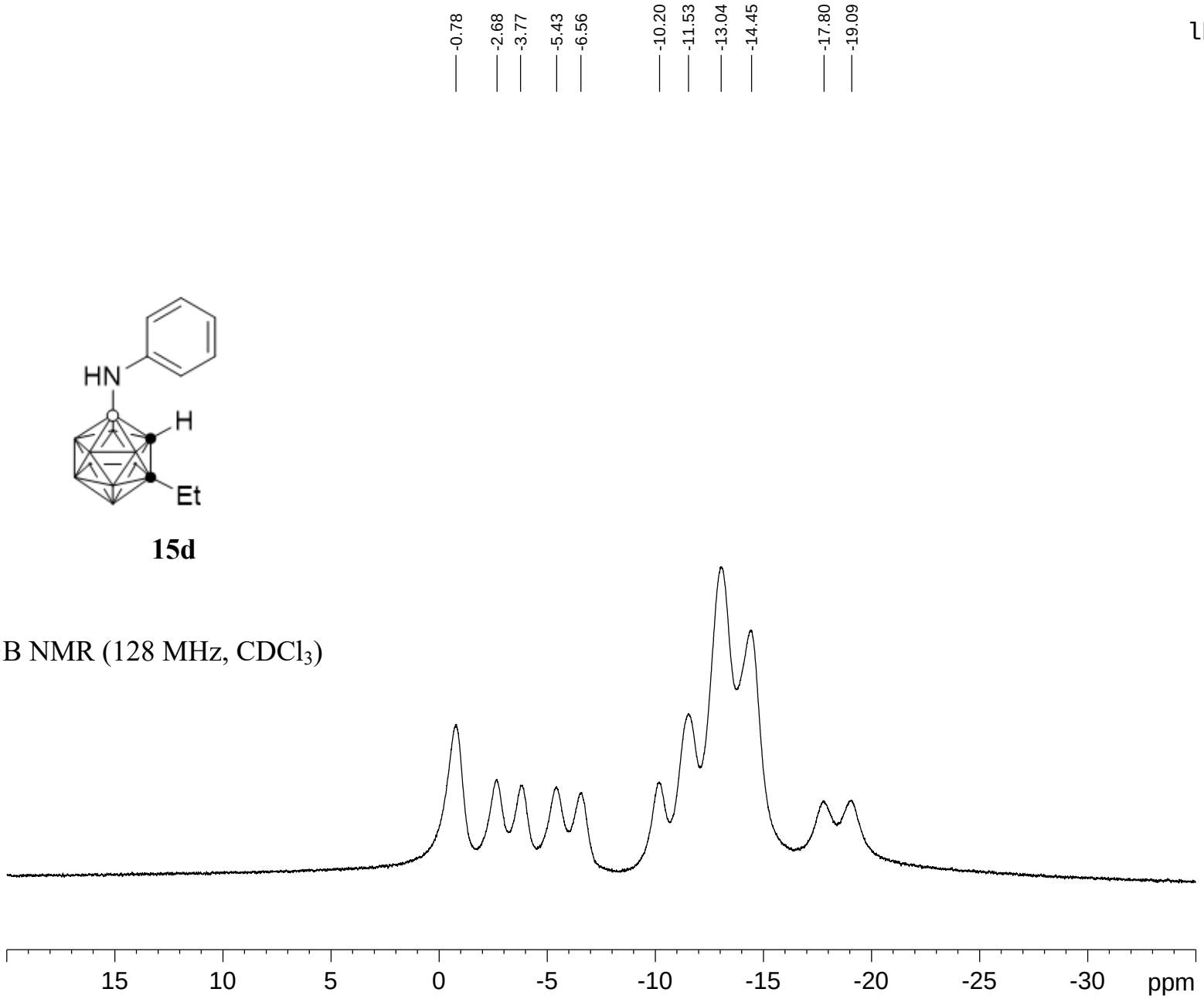


lhr-B-0522-Etphnh-CDCl3

```
Current Data Parameters
NAME      lhr-B-0522-Etphnh-CDCl3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161102
Time                9.43 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                    12
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                   287
DW                20.800 usec
DE                 6.50 usec
TE                296.4 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
```

15d

^{11}B NMR (128 MHz, $CDCl_3$)



lhr-B-0522-Etphnh-CDCl3(C)

```
Current Data Parameters
NAME      lhr-B-0522-Etphnh-CDCl3(C)
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20161102
Time               9.45 h
INSTRUM           spect
PROBHD   Z108618_0257 (
PULPROG             zg
TD                65536
SOLVENT           CDCl3
NS                   14
DS                    2
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                  287
DW                20.800 usec
DE                 6.50 usec
TE                296.2 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          128.4097504 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

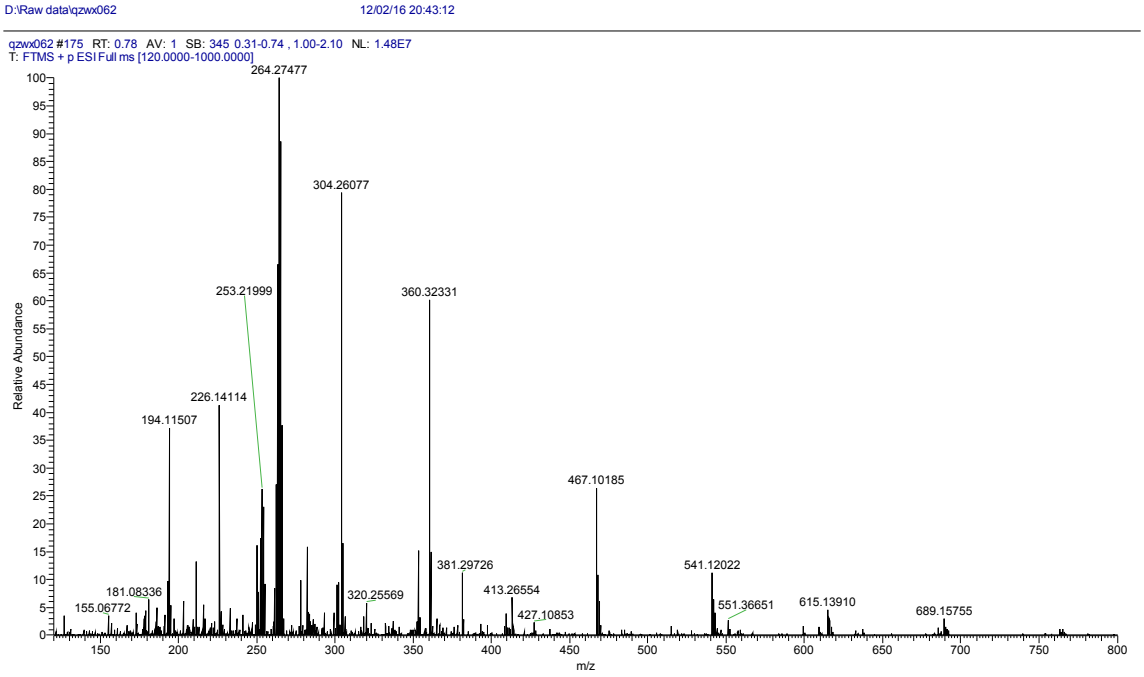
Thermo QEFMS Analysis Report

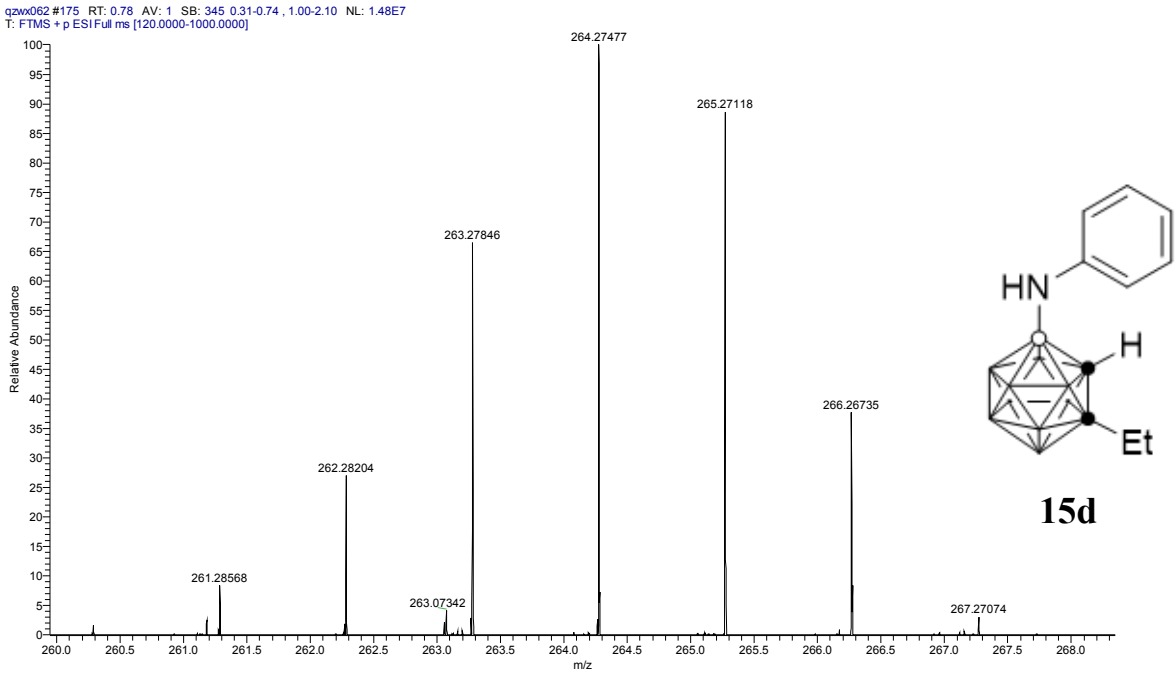
Analysis Info

Sample Name :	Lhr-522	Reference No.:	Qzwx062
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.5kV, by LC, with sheath gas		

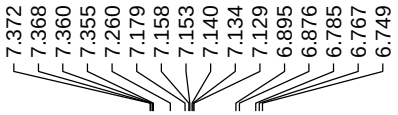
Accurate Mass Measurement

Molecular formula :	$C_{10}H_{21}B_{10}N$
Experimental Mass $[M+H]^+$:	264.27477
Theoretical Mass $[M+H]^+$:	264.27500
Error (ppm) :	0.8

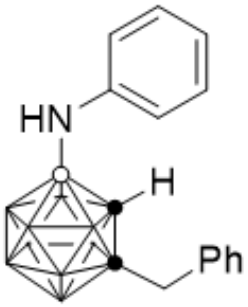




15d



lhr-H-0541-2-Bnphnh



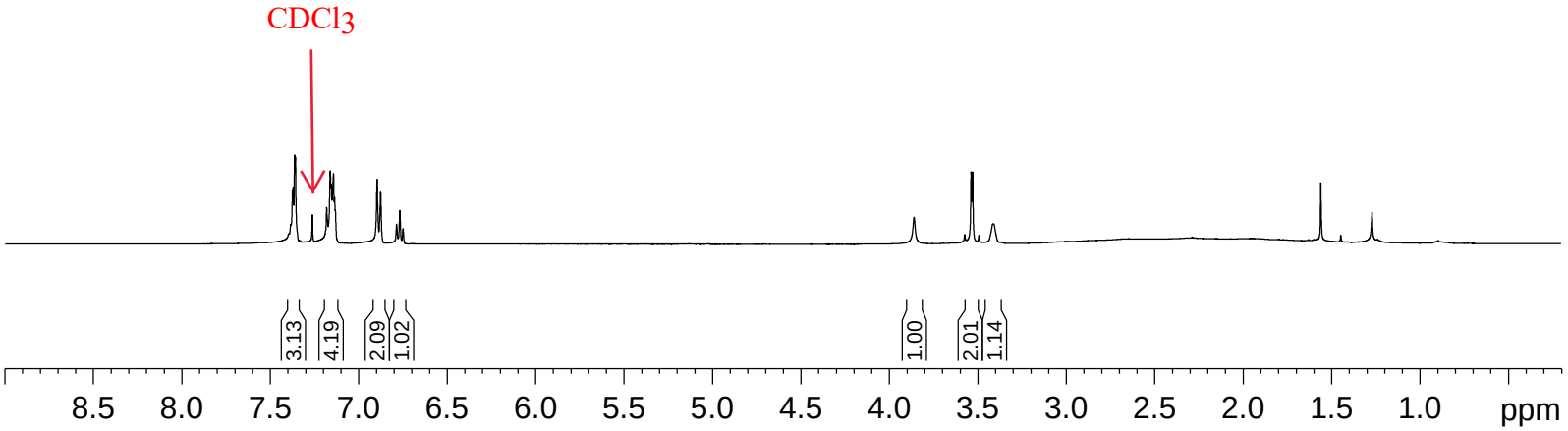
16d

1H NMR (400 MHz, $CDCl_3$)

Current Data Parameters
NAME lhr-H-0541-2-Bnphnh-CD
EXPNO 1
PROCNO 1

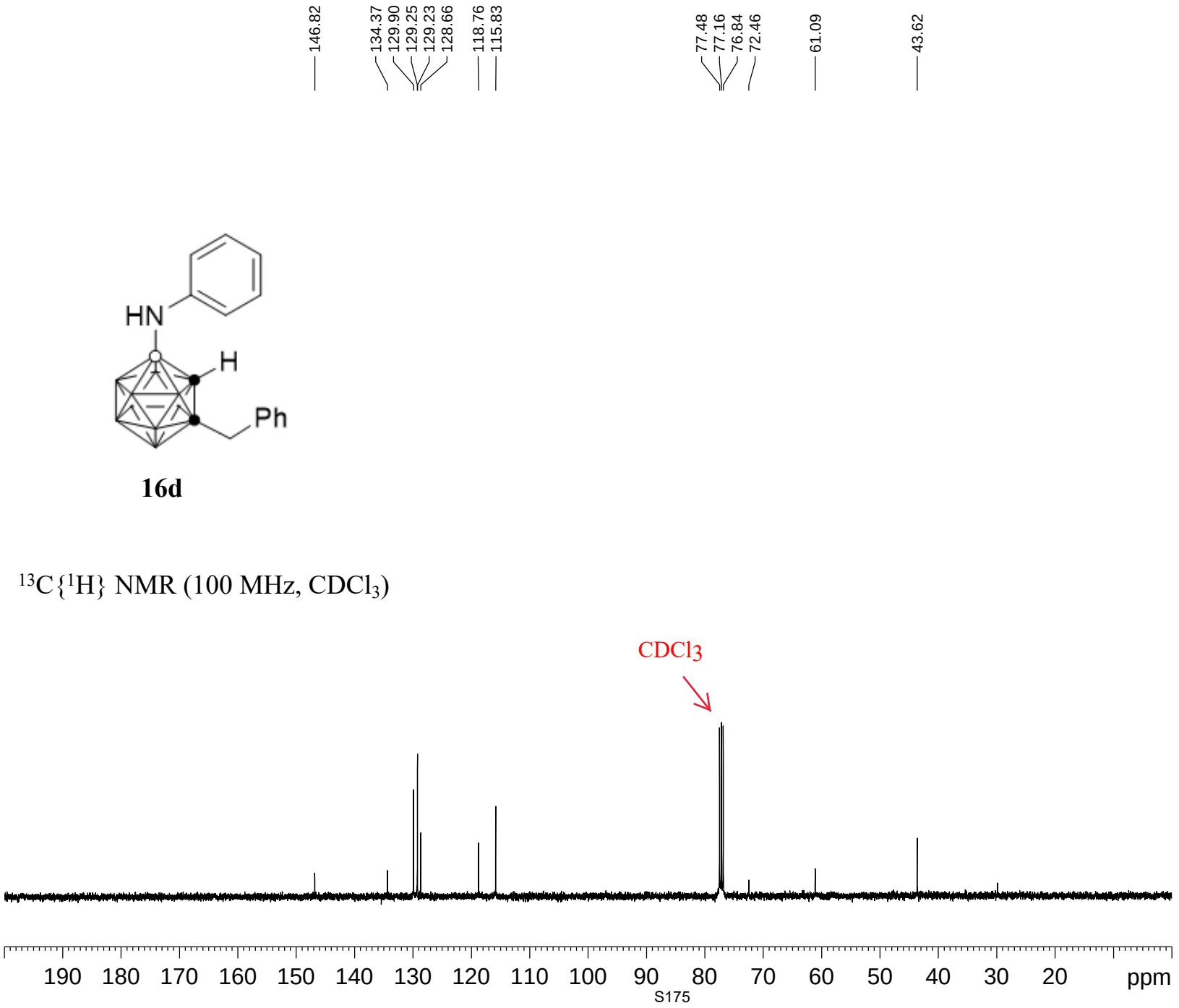
F2 - Acquisition Parameters
Date_ 20161113
Time 19.37 h
INSTRUM spect
PROBHD Z108618_0257 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 14
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 64
DW 62.400 usec
DE 6.50 usec
TE 296.1 K
D1 1.00000000 sec
TD0 1
SFO1 400.2324714 MHz
NUC1 1H
P1 12.80 usec
PLW1 13.56000042 W

F2 - Processing parameters
SI 65536
SF 400.2300102 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



16d

$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)

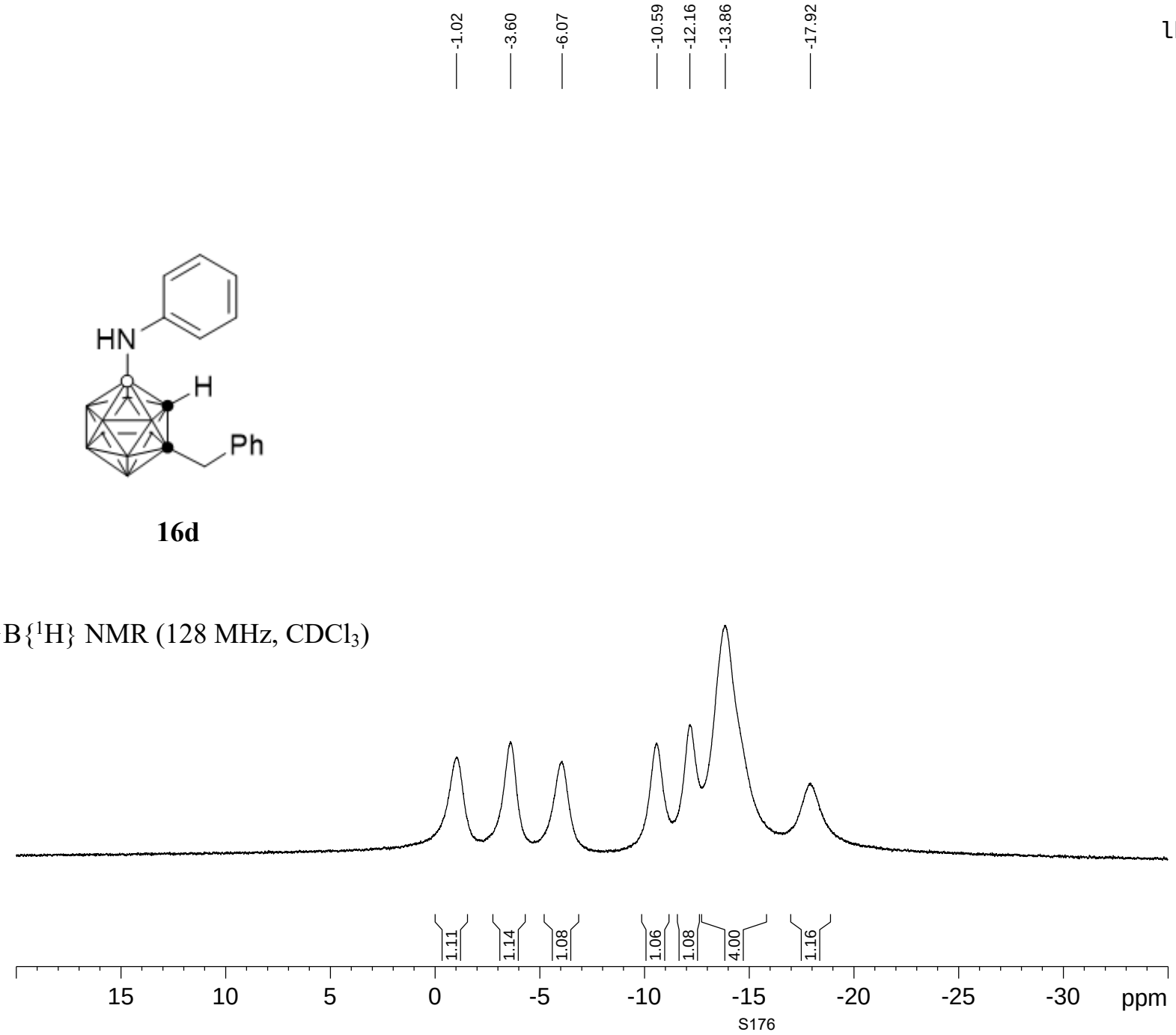


lhr-C-0541-2-Bnphnl

```
Current Data Parameters
NAME      lhr-C-0541-2-Bnphnh-CDCl
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161113
Time              19.42 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     68
DS                      4
SWH            29761.904 Hz
FIDRES          0.454131 Hz
AQ             1.1010048 sec
RG                    181
DW                16.800 usec
DE                 6.50 usec
TE                296.5 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           100.6379029 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

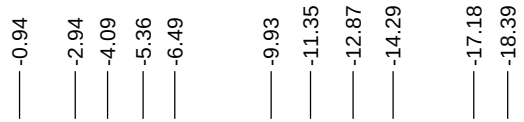
16d

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)



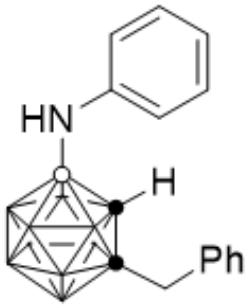
lhr-B-0541-2-Bnphnh-CDCl3

```
Current Data Parameters
NAME      lhr-B-0541-2-Bnphnh-CDCl3
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20161113
Time              19.46 h
INSTRUM           spect
PROBHD   Z108618_0257 (
PULPROG           zgdc
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                  203
DW                20.800 usec
DE                 6.50 usec
TE                296.6 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
```



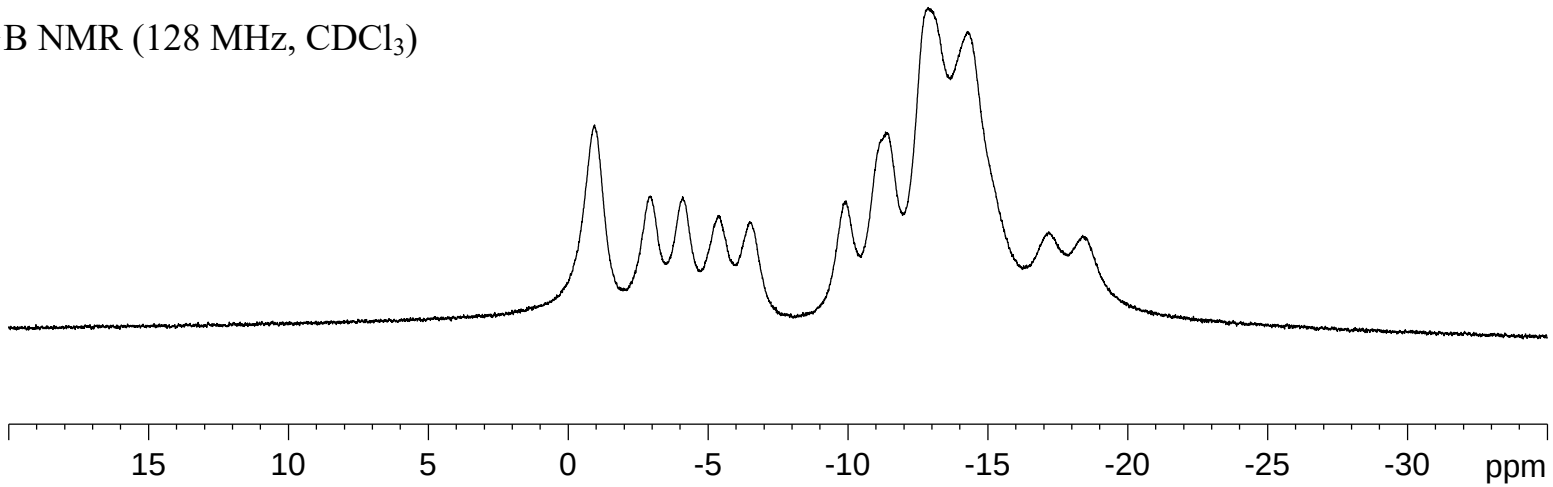
lhr-B-0541-2-Bnphnh-CDCl3(C)

```
Current Data Parameters
NAME     lhr-B-0541-2-Bnphnh-CDCl3(C)
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20161113
Time              19.48 h
INSTRUM           spect
PROBHD   Z108618_0257 (
PULPROG             zg
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                  203
DW               20.800 usec
DE                6.50 usec
TE               296.2 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          128.4097504 MHz
WDW                  EM
SSB      0
LB                1.00 Hz
GB       0
PC                1.40
```



16d

^{11}B NMR (128 MHz, $CDCl_3$)



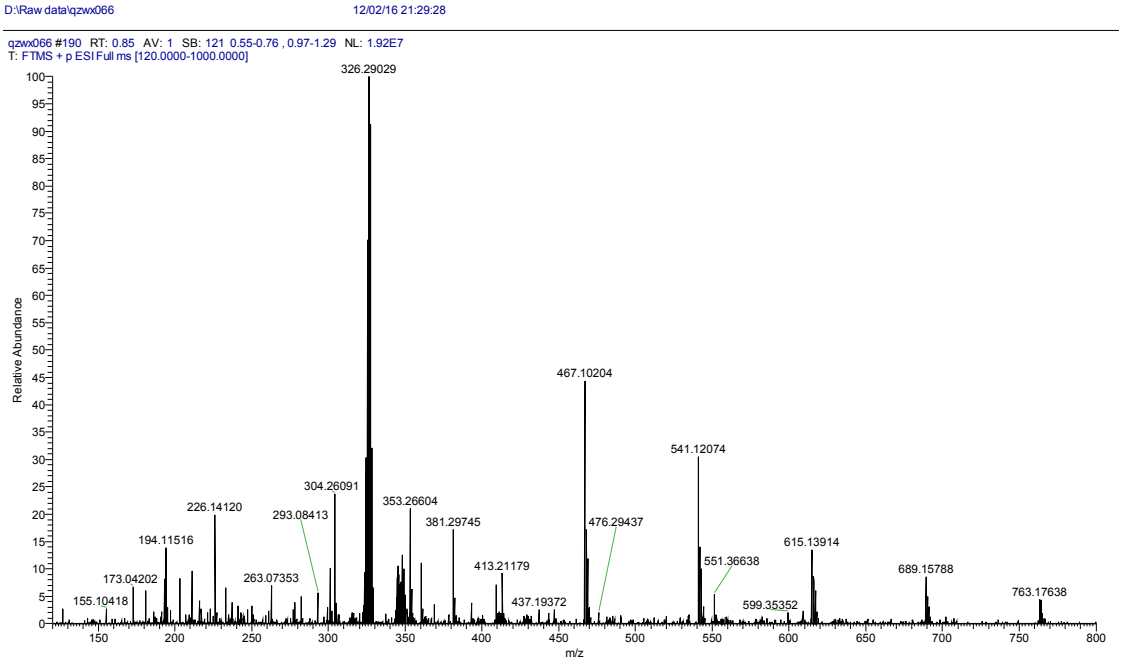
Thermo QEFMS Analysis Report

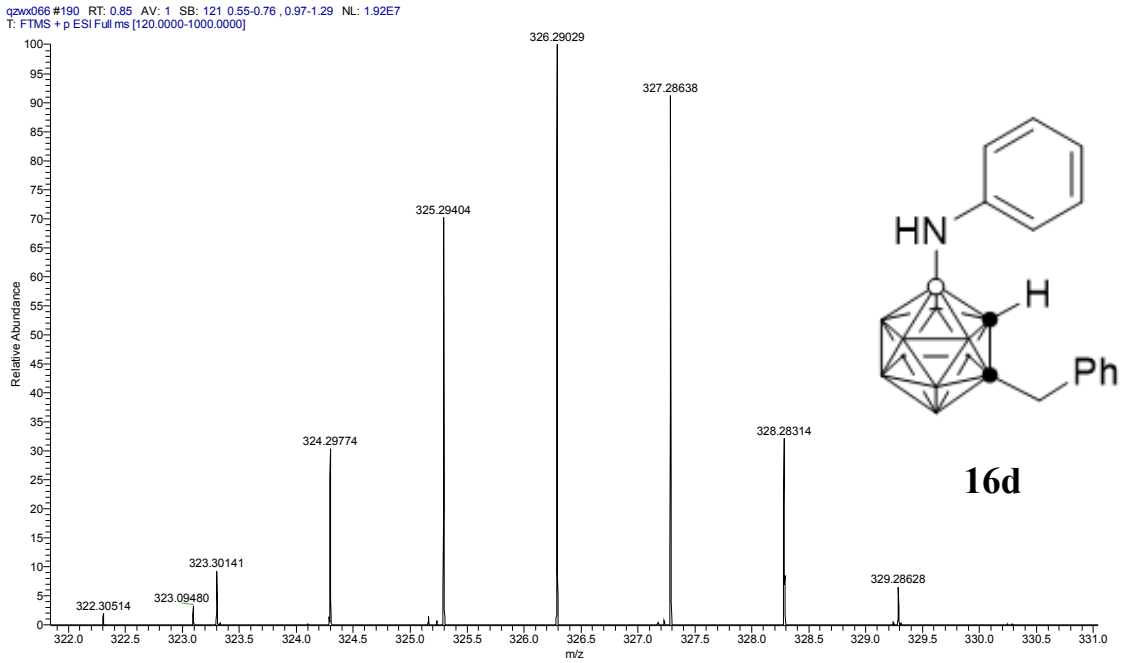
Analysis Info

Sample Name :	Lhr-541	Reference No.:	Qzwx066
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.5kV, by LC, with sheath gas		

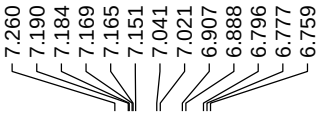
Accurate Mass Measurement

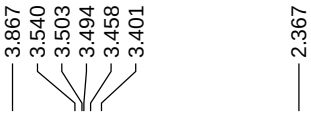
Molecular formula :	$C_{15}H_{23}B_{10}N$
Experimental Mass $[M+H]^+$:	326.29029
Theoretical Mass $[M+H]^+$:	326.29065
Error (ppm) :	1.1



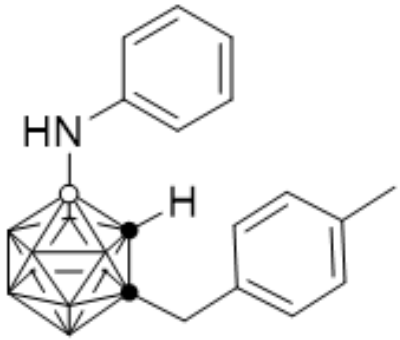


16d





lhr-H-0527-Bnch3phnh



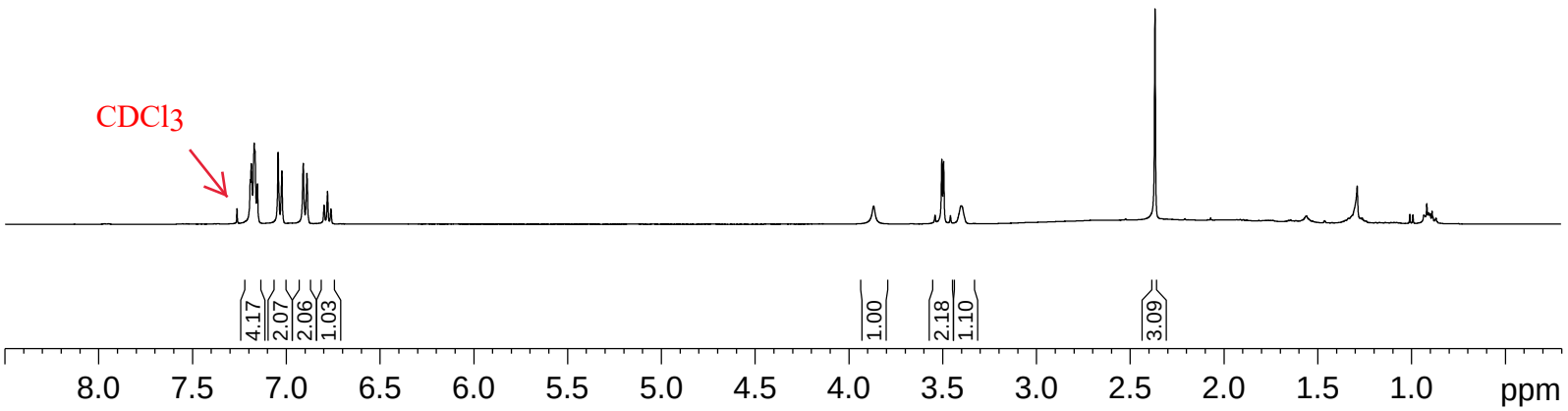
17d

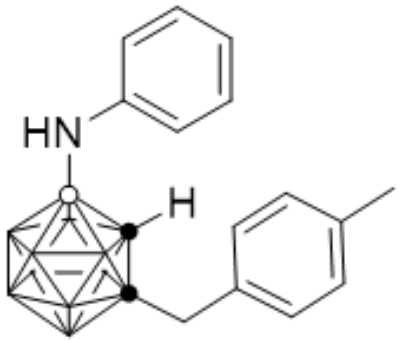
^{1}H NMR (400 MHz, $CDCl_3$)

Current Data Parameters
NAME lhr-H-0527-Bnch3phnh-Cl
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161103
Time 17.06 h
INSTRUM spect
PROBHD Z108618_0257 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 9
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 36
DW 62.400 usec
DE 6.50 usec
TE 295.8 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



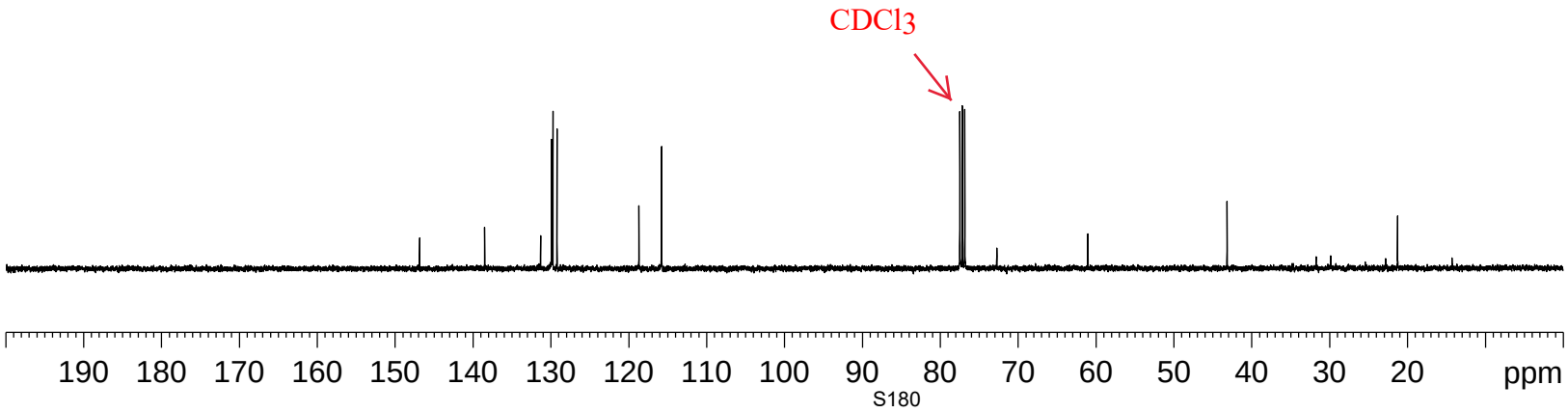


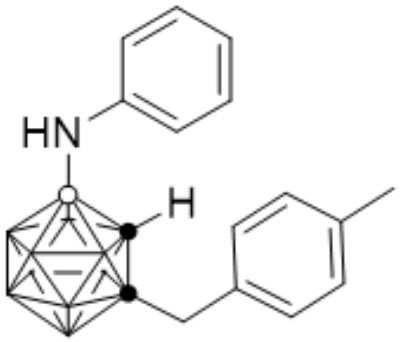
17d

$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)

lhr-C-0527-Bnch3phı

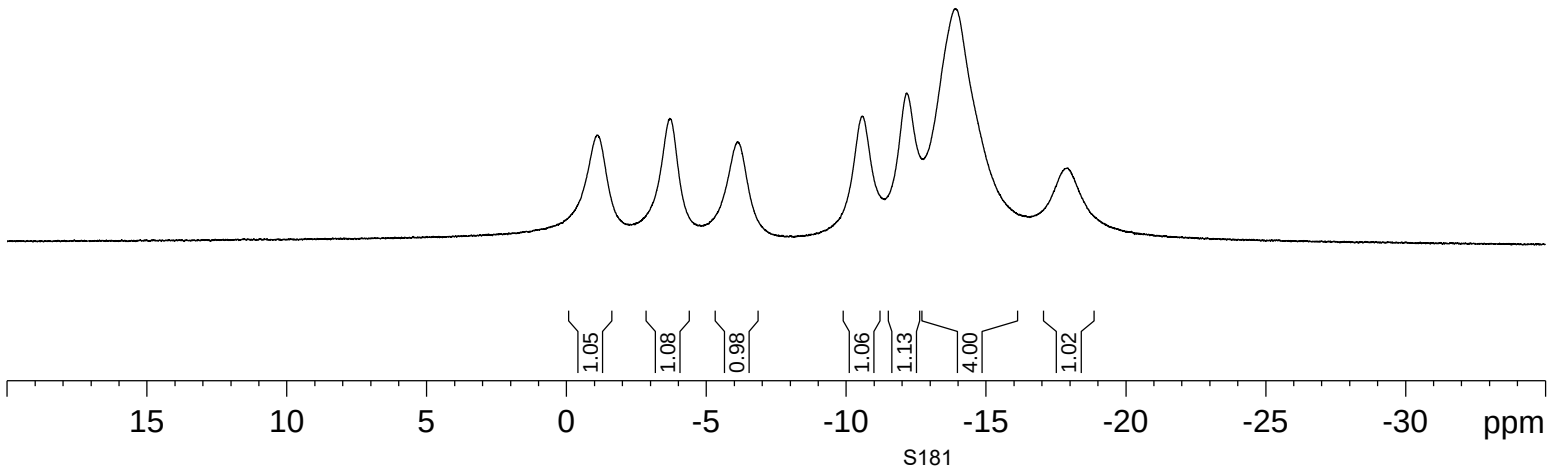
```
Current Data Parameters
NAME      lhr-C-0527-Bnch3phnh-CDCl3
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20161103
Time              17.10 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                   36
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG                  161
DW               16.800 usec
DE                6.50 usec
TE                296.2 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          100.6379056 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```





17d

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)



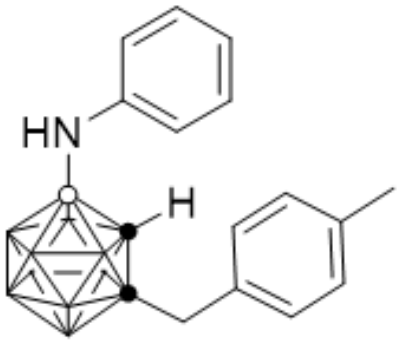
lhr-B-0527-Bnch3phnh-CDC

```
Current Data Parameters
NAME      lhr-B-0527-Bnch3phnh-CDCl3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161103
Time              17.13 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                    12
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                   203
DW                20.800 usec
DE                 6.50 usec
TE                296.3 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
```

-0.99 -3.01 -4.16 -5.42 -6.60 -9.89 -11.36 -12.83 -14.32 -17.12 -18.33

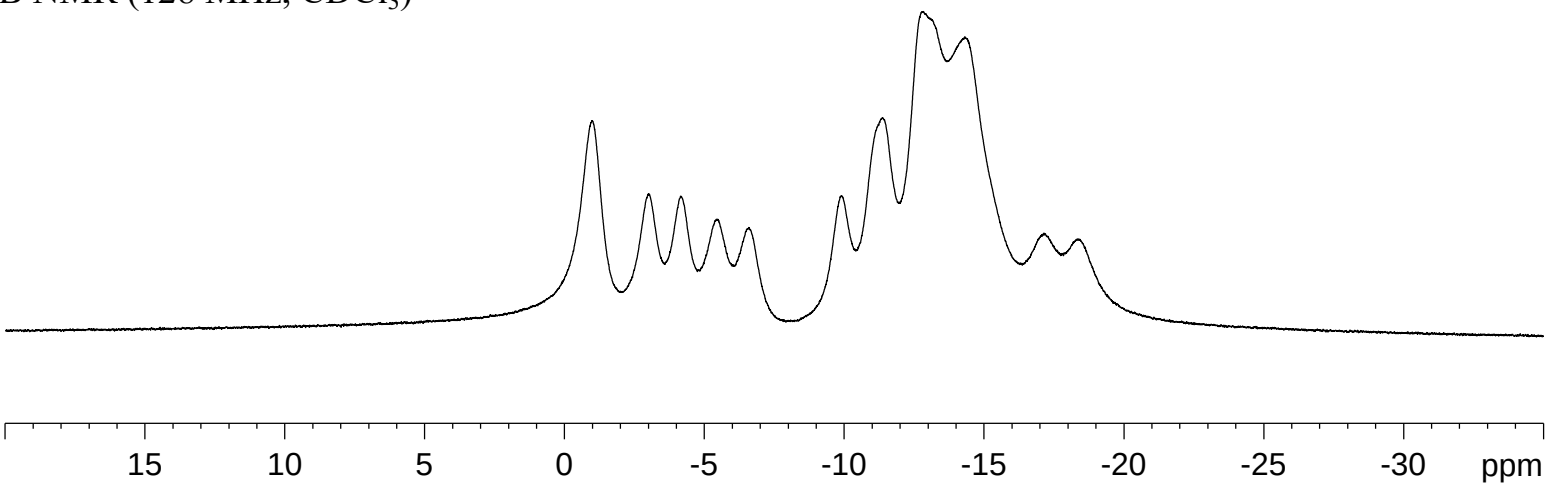
lhr-B-0527-Bnch3phnh-CDC

```
Current Data Parameters
NAME      lhr-B-0527-Bnch3phnh-CDCl3(C)
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161103
Time              17.15 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG               zg
TD                 65536
SOLVENT            CDCl3
NS                    20
DS                     2
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                   203
DW                20.800 usec
DE                 6.50 usec
TE                296.0 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           128.4097504 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



17d

^{11}B NMR (128 MHz, $CDCl_3$)



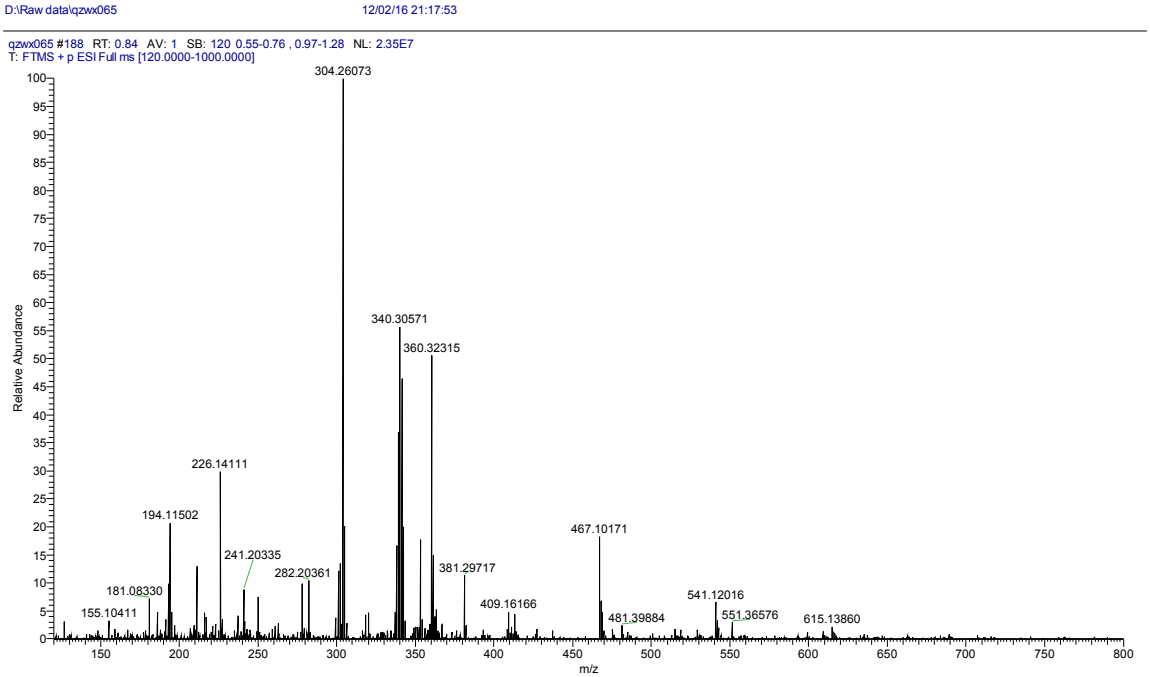
Thermo QEFMS Analysis Report

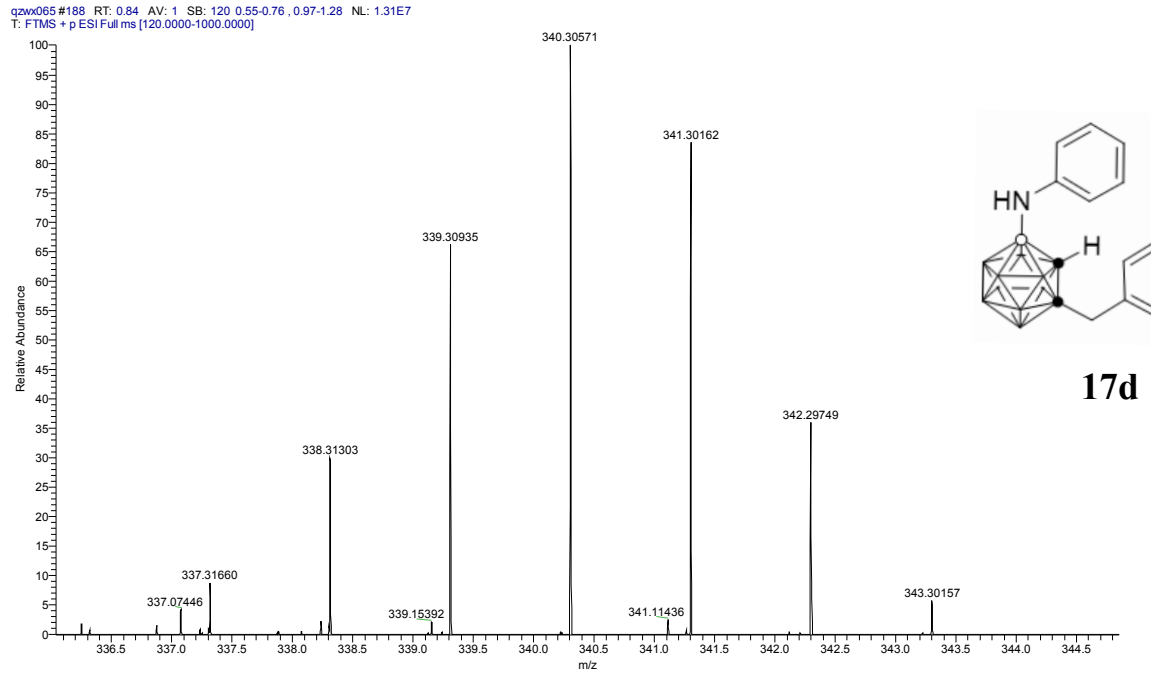
Analysis Info

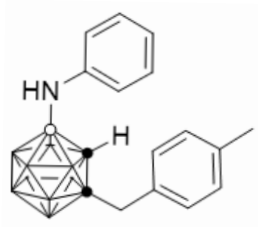
Sample Name :	Lhr-527	Reference No.:	Qzwx065
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.5kV, by LC, with sheath gas		

Accurate Mass Measurement

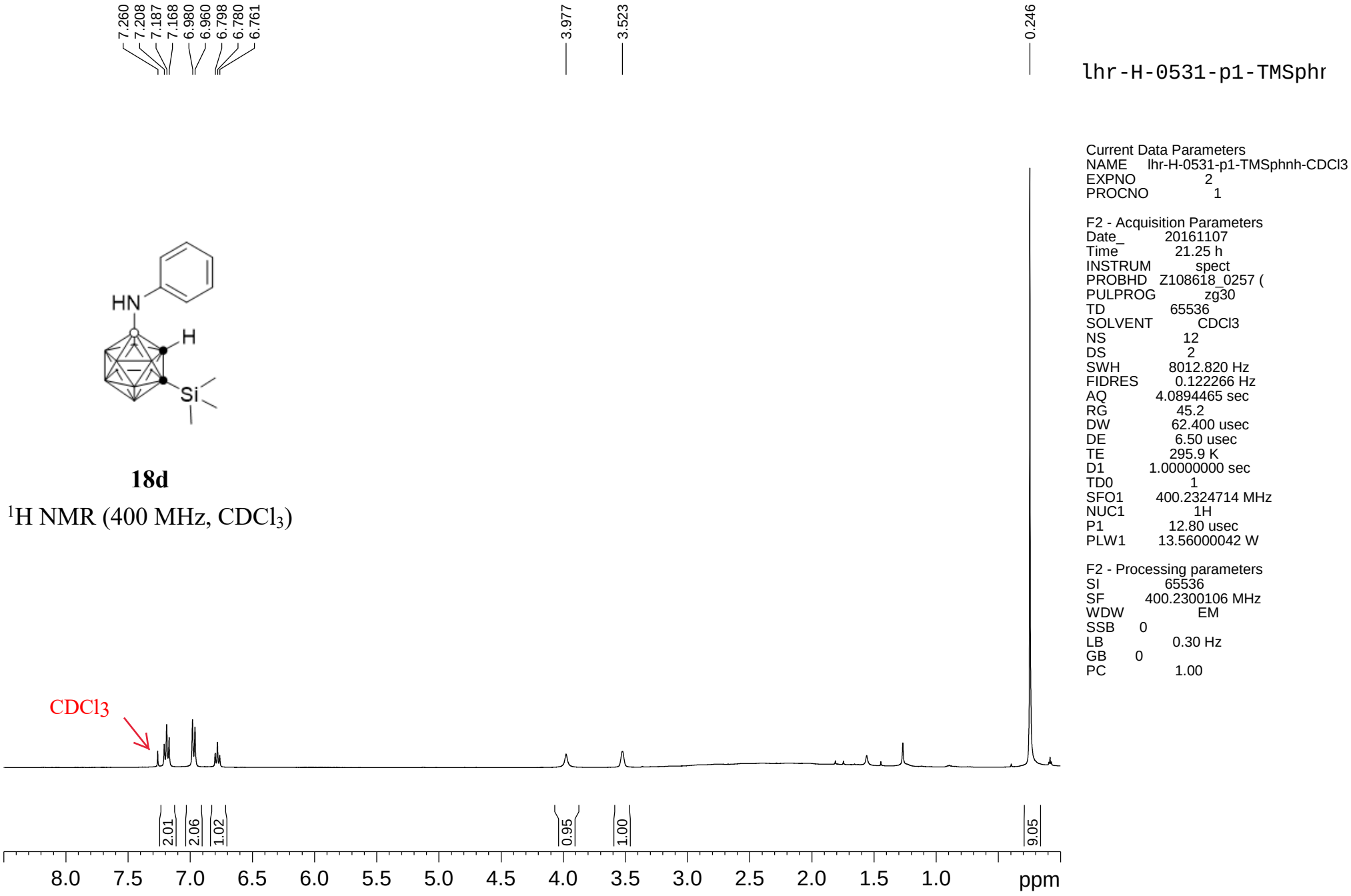
Molecular formula :	$C_{16}H_{25}B_{10}N$
Experimental Mass $[M+H]^+$:	340.30571
Theoretical Mass $[M+H]^+$:	340.30630
Error (ppm) :	1.7





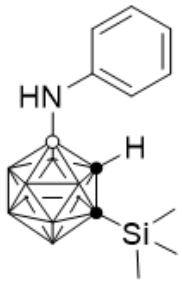


17d


7.260
7.208
7.187
7.168
6.980
6.960
6.798
6.780
6.761
3.977
3.523
0.246
lhr-H-0531-p1-TMSphr
Current Data Parameters
NAME lhr-H-0531-p1-TMSphnh-CDCl3
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161107
Time 21.25 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 45.2
DW 62.400 usec
DE 6.50 usec
TE 295.9 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 400.2300106 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
HN
H
Si
18d
1H NMR (400 MHz, CDCl3)
CDCl3
2.01
2.06
1.02
0.95
1.00
9.05
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

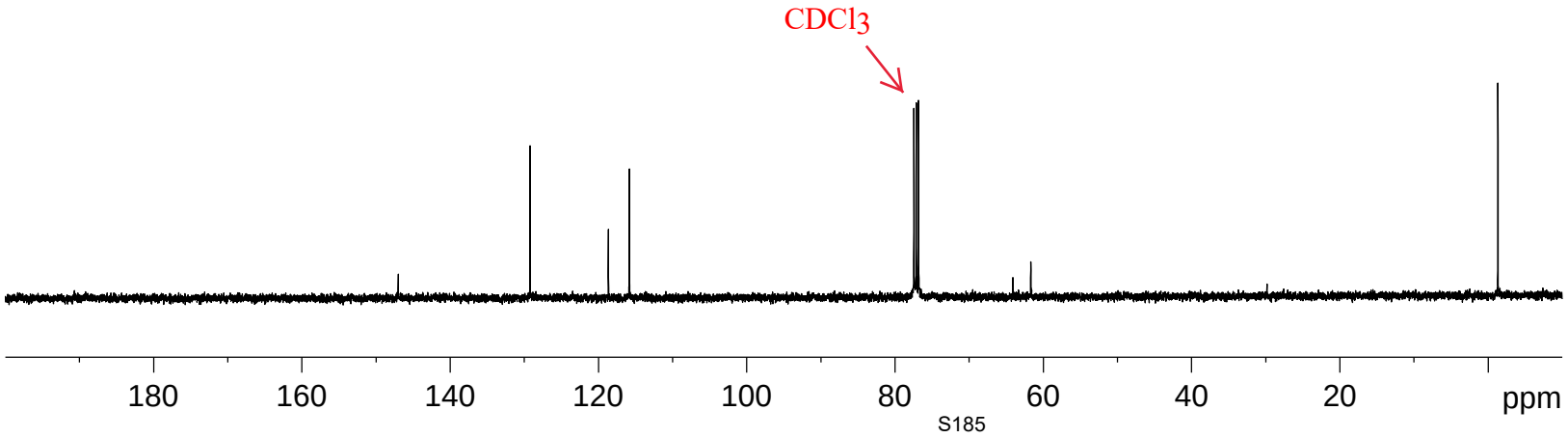
147.02
129.25
118.68
115.86
77.48
77.16
76.84
64.11
61.70
-1.30

lhr-C-0531-p1-TMSpl

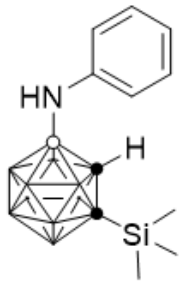


18d

$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)

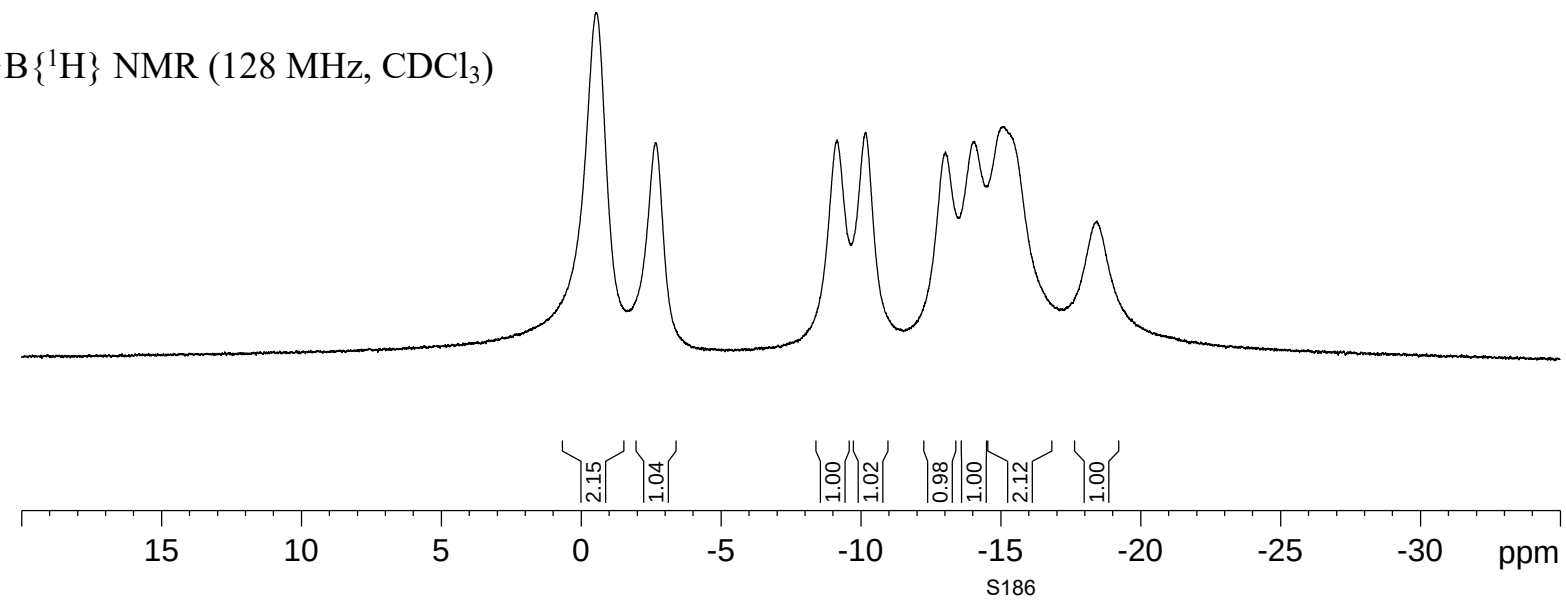



```
Current Data Parameters
NAME      lhr-C-0531-p1-TMSphnh-CDCl3
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20161107
Time              21.06 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   100
DS                     4
SWH            29761.904 Hz
FIDRES          0.454131 Hz
AQ              1.1010048 sec
RG                   203
DW                16.800 usec
DE                 6.50 usec
TE                296.3 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           100.6379032 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



18d

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)



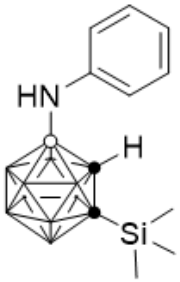
lhr-B-0531-p1-TMSphnh-CDCl3

```
Current Data Parameters
NAME      lhr-B-0531-p1-TMSphnh-CDCl3
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20161107
Time                21.18 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG              zgdc
TD                  65536
SOLVENT             CDCl3
NS                     21
DS                      4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631488 sec
RG                    203
DW                 20.800 usec
DE                  6.50 usec
TE                 296.6 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
```

-0.29 -1.13 -2.01 -3.21 -8.47 -9.57 -10.68 -12.35 -13.51 -14.53 -15.56 -17.70 -18.96

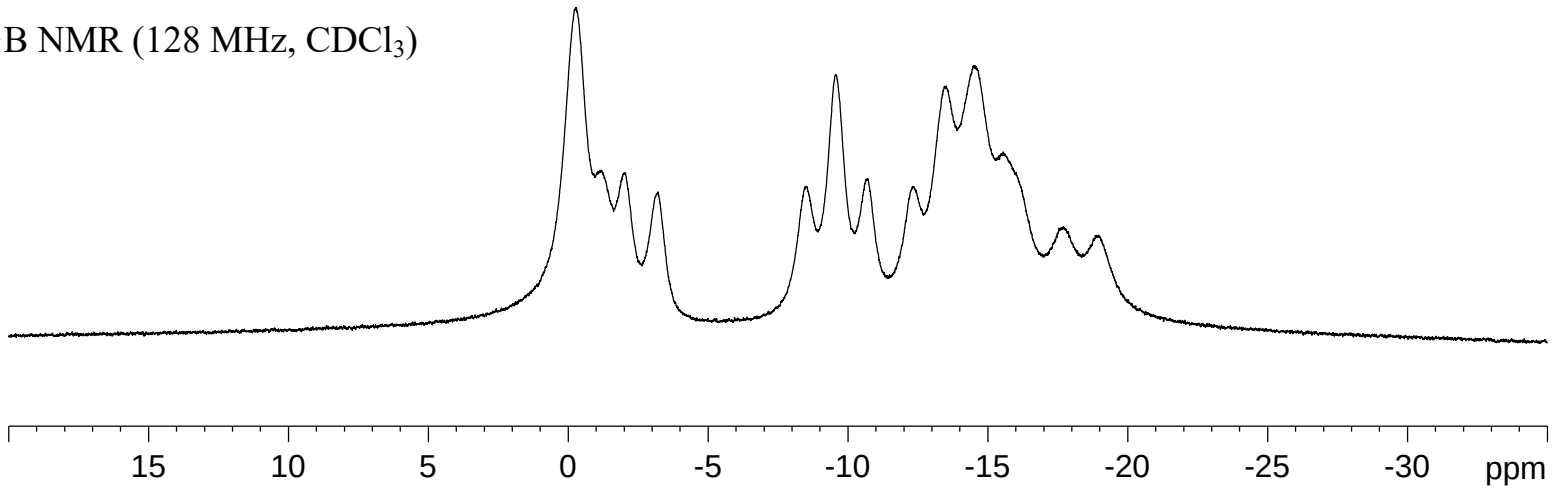
lhr-B-0531-p1-TMSphnh-CDCl3(C)

```
Current Data Parameters
NAME      lhr-B-0531-p1-TMSphnh-CDCl3(C)
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20161107
Time               21.16 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG                zg
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                    203
DW                20.800 usec
DE                 6.50 usec
TE                296.1 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           128.4097504 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



18d

^{11}B NMR (128 MHz, $CDCl_3$)



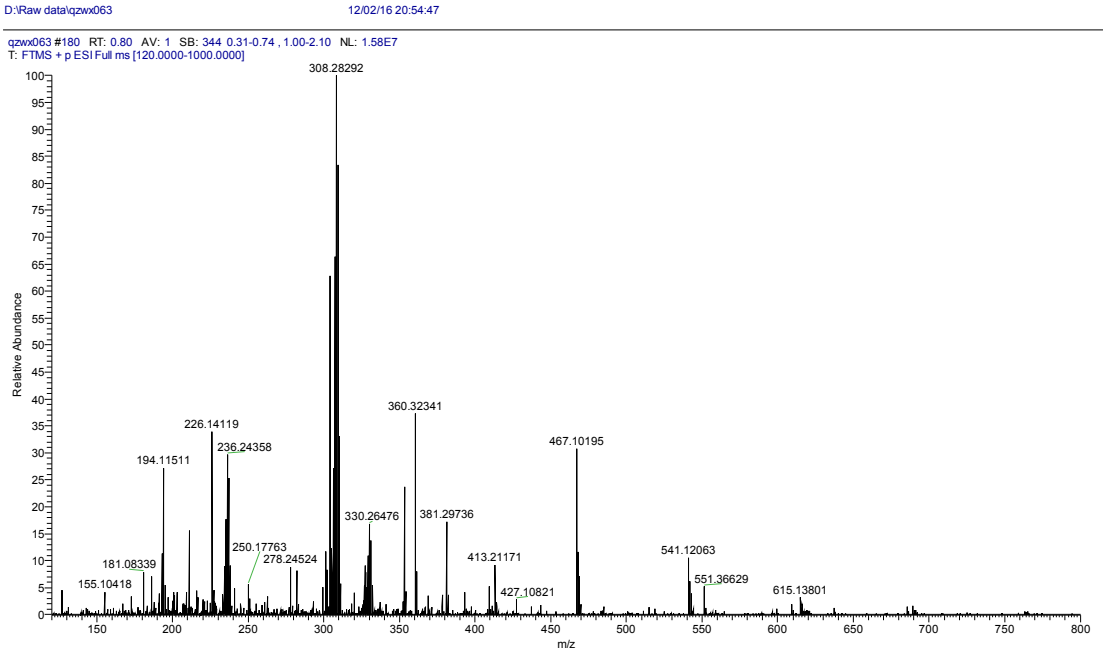
Thermo QEFMS Analysis Report

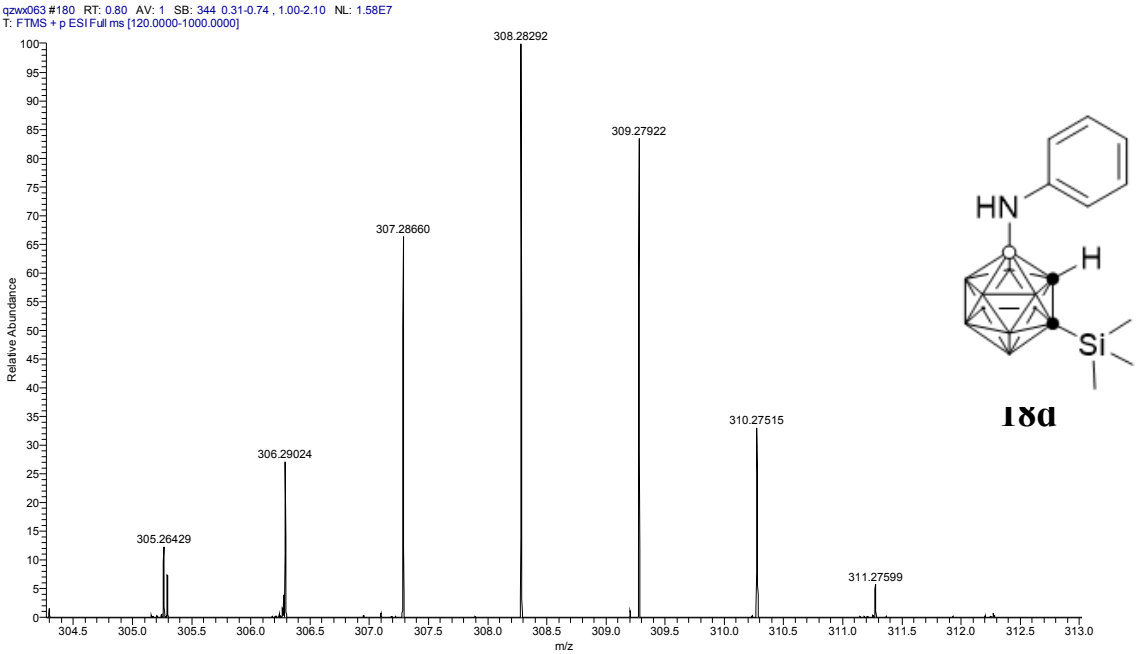
Analysis Info

Sample Name :	Lhr-531-P1	Reference No.:	Qzwx063
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.5kV, by LC, with sheath gas		

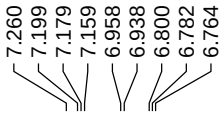
Accurate Mass Measurement

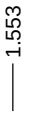
Molecular formula :	$C_{11}H_{25}B_{10}NSi$
Experimental Mass $[M+H]^+$:	308.28292
Theoretical Mass $[M+H]^+$:	308.28334
Error (ppm) :	1.3



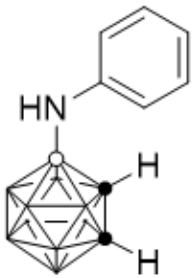


18d



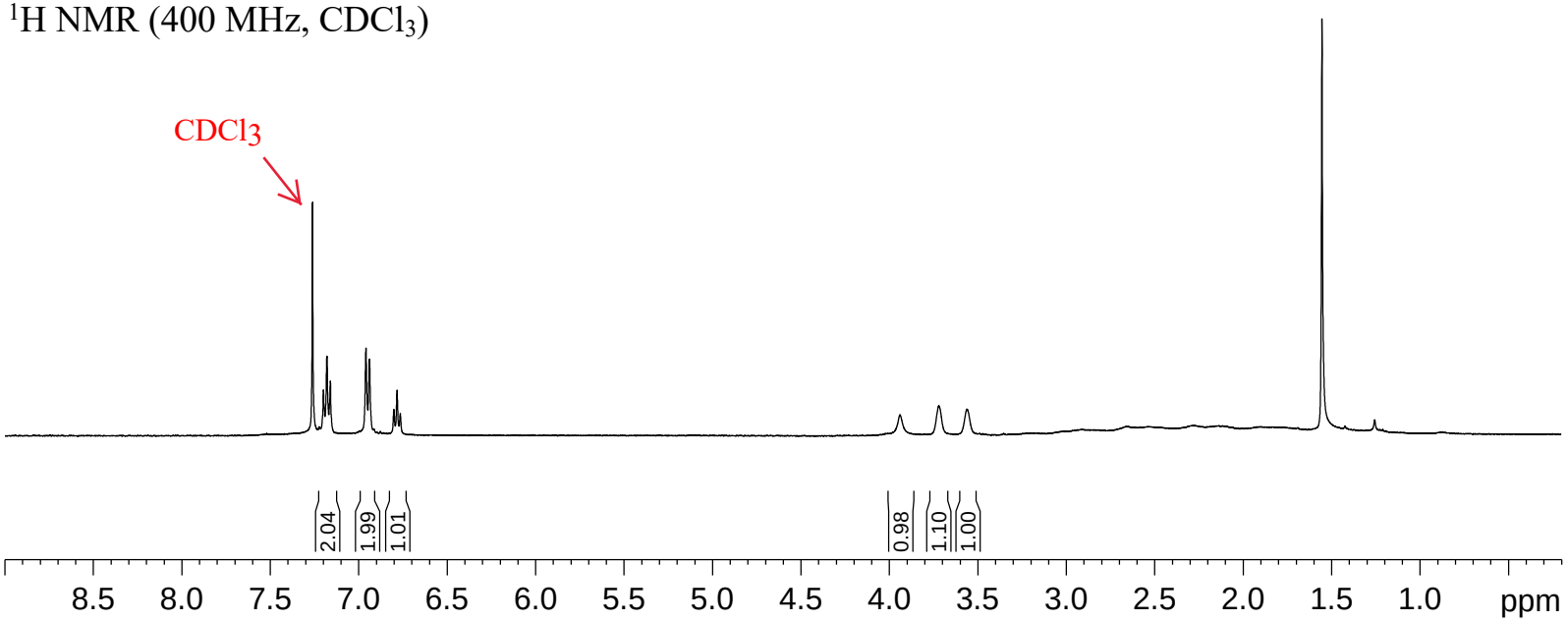


lhr-H-0531-p2-Hphnh



19d

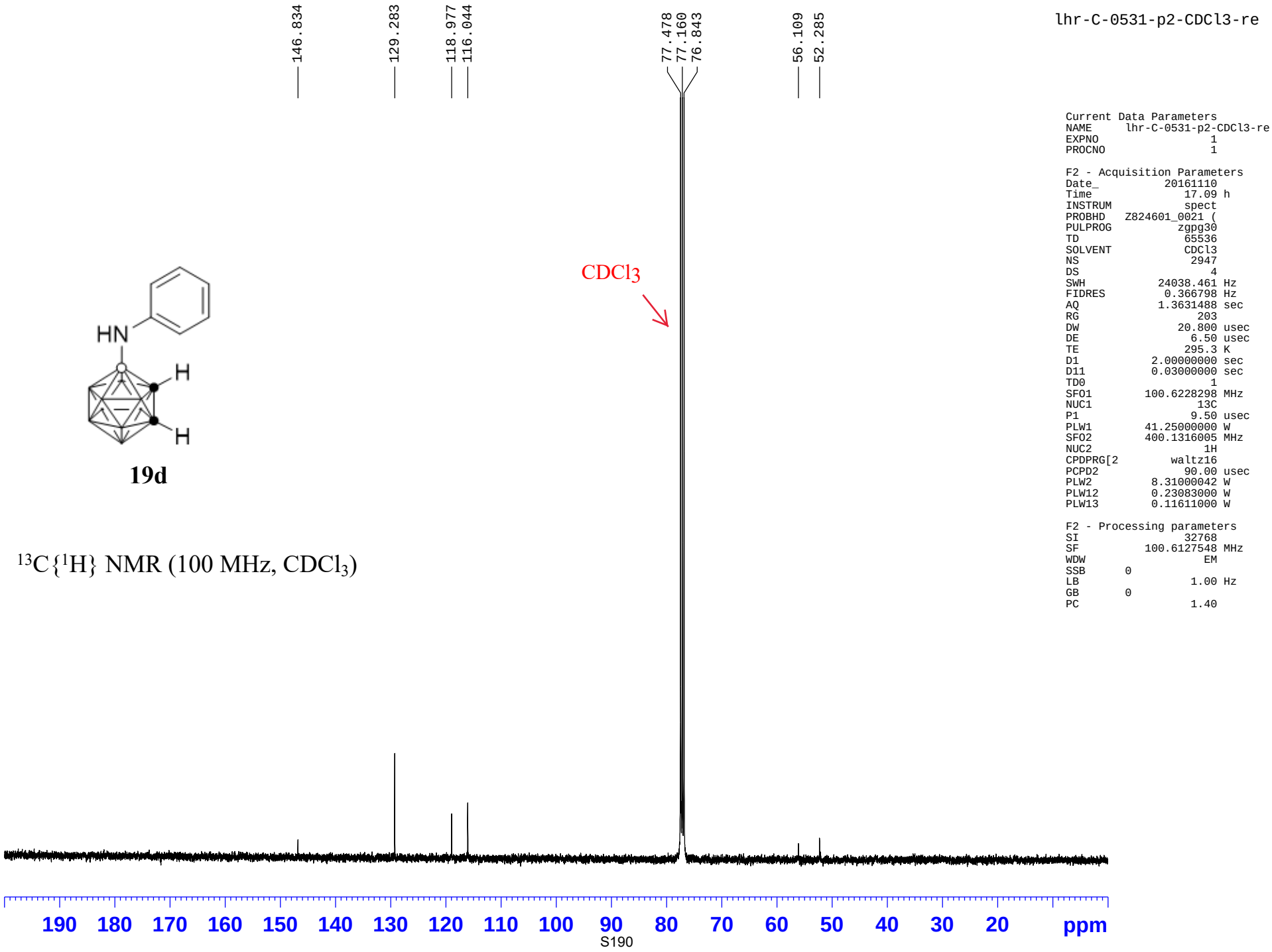
^{1}H NMR (400 MHz, $CDCl_3$)



Current Data Parameters
NAME lhr-H-0531-p2-Hphnh-CD
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161110
Time 10.12 h
INSTRUM spect
PROBHD Z108618_0257 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 228
DW 62.400 usec
DE 6.50 usec
TE 297.4 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 400.2300105 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

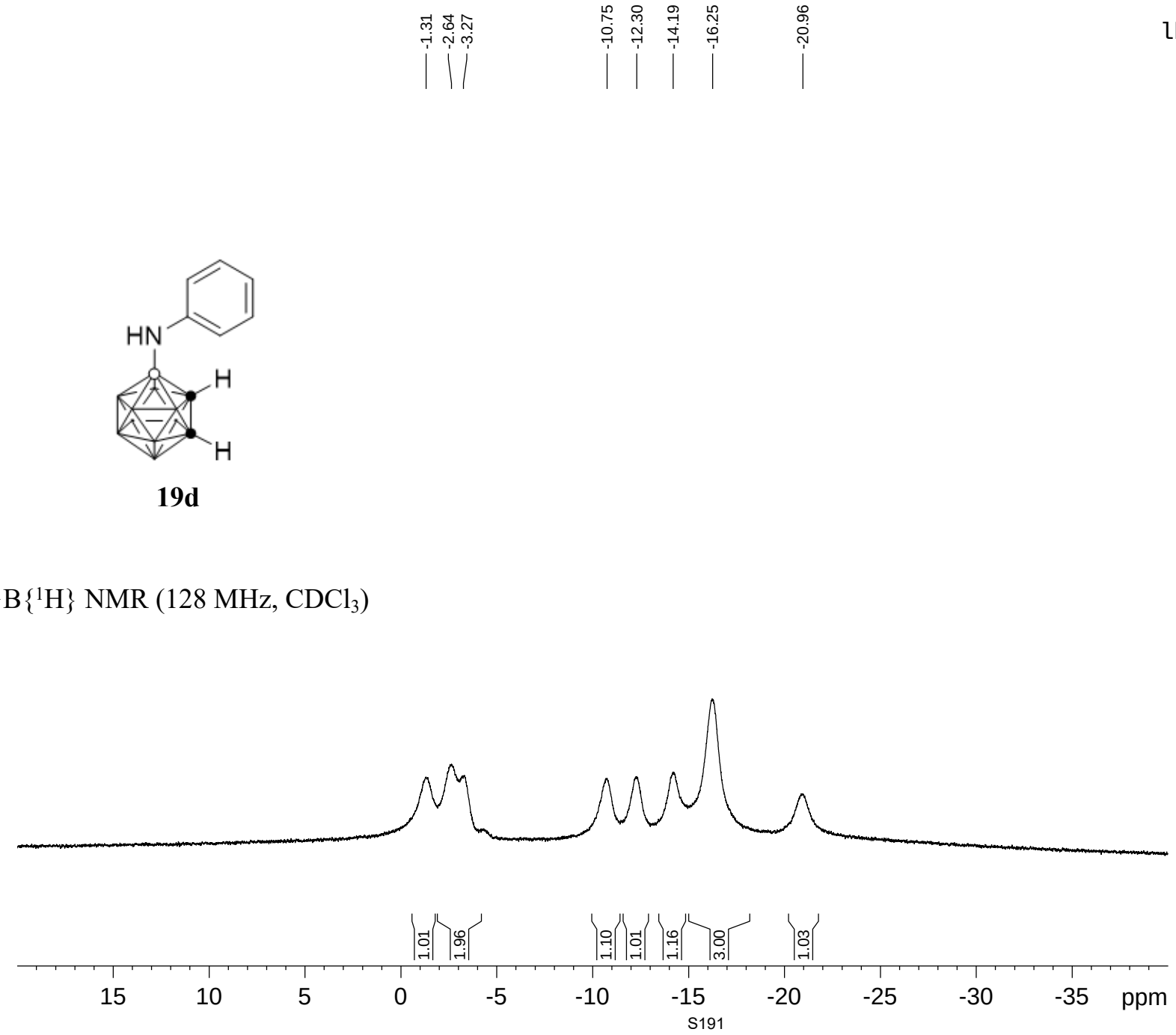


lhr-C-0531-p2-CDCl3-re

```
Current Data Parameters
NAME      lhr-C-0531-p2-CDCl3-re
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161110
Time              17.09 h
INSTRUM             spect
PROBHD   Z824601_0021 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  2947
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                   203
DW                20.800 usec
DE                 6.50 usec
TE                295.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1
SFO1         100.6228298 MHz
NUC1                13C
P1                  9.50 usec
PLW1         41.25000000 W
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2          8.31000042 W
PLW12         0.23083000 W
PLW13         0.11611000 W

F2 - Processing parameters
SI                 32768
SF           100.6127548 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

19d

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)

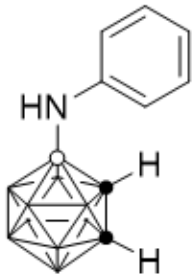


lhr-B-0531-p2-Hphnh-CDCl

```
Current Data Parameters
NAME      lhr-B-0531-p2-Hphnh-CDCl3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161108
Time              10.33 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                    20
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631488 sec
RG                   456
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
```

-1.29
-1.82
-2.71
-10.01
-11.31
-12.77
-13.46
-14.75
-15.44
-16.82
-20.14
-21.48

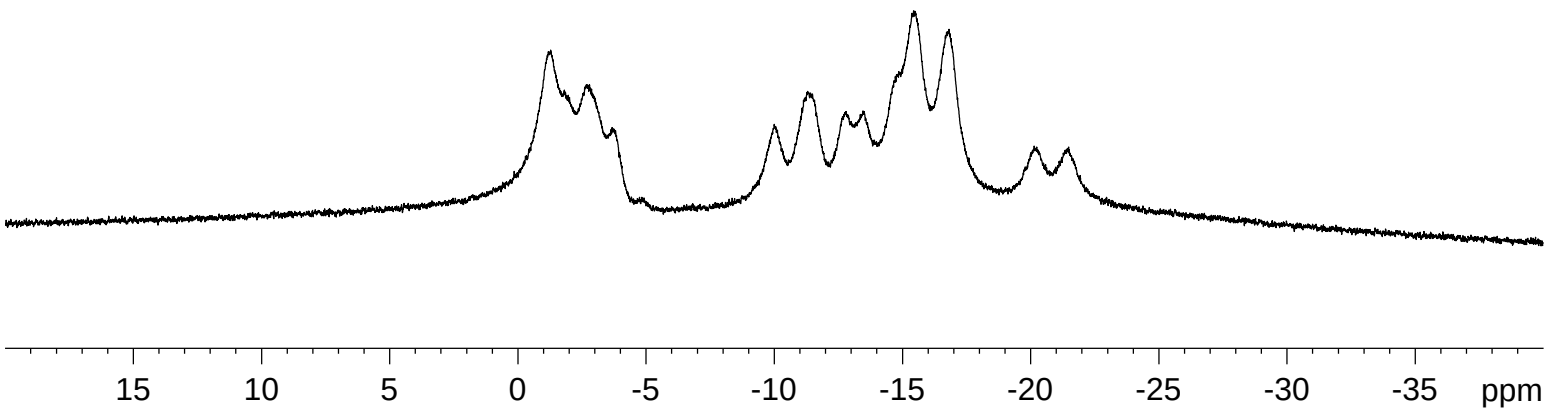
lhr-B-0531-p2-Hphnh-CDCl3



19d

```
Current Data Parameters
NAME      lhr-B-0531-p2-Hphnh-CDCl3(C)
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20161108
Time              10.36 h
INSTRUM           spect
PROBHD   Z108618_0257 (
PULPROG             zg
TD                65536
SOLVENT           CDCl3
NS                   28
DS                    2
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                  456
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          128.4097504 MHz
WDW                EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{11}B NMR (128 MHz, $CDCl_3$)



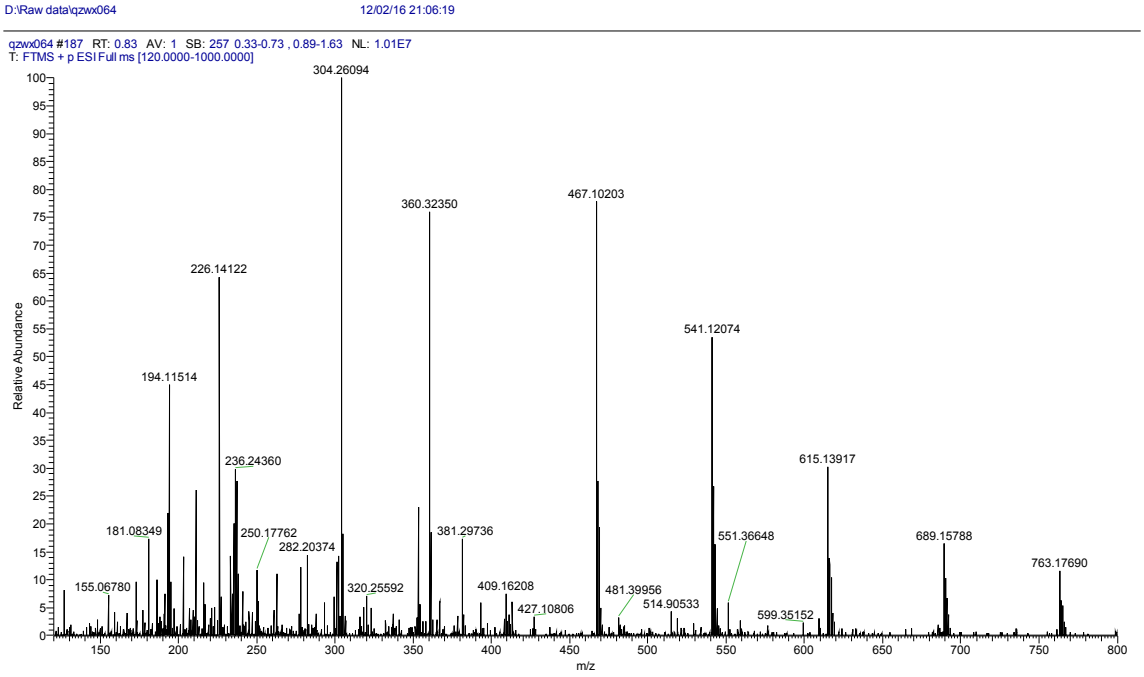
Thermo QEFMS Analysis Report

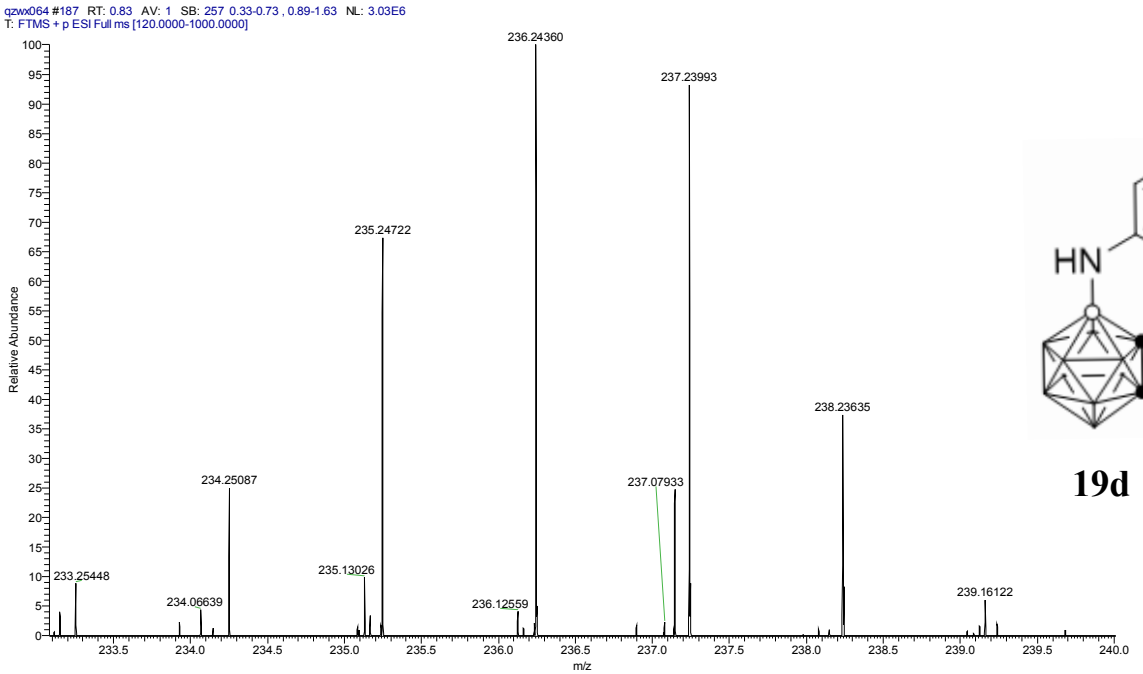
Analysis Info

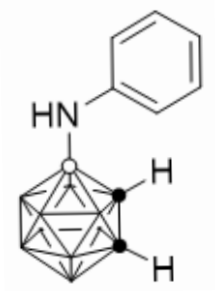
Sample Name :	Lhr-531-P2	Reference No.:	Qzwx064
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.5kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_8H_{17}B_{10}N$
Experimental Mass $[M+H]^+$:	236.24360
Theoretical Mass $[M+H]^+$:	236.24370
Error (ppm) :	0.4







19d

7.260

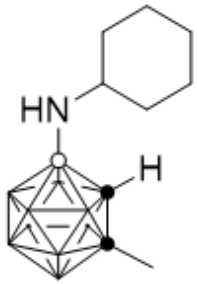
3.529

2.705
2.680
2.655

2.014
1.902
1.875
1.686
1.656
1.564
1.543
1.299
1.252
1.239
1.207
1.139
1.109
1.078
0.991
0.964

lhr-H-0556-12-cc-CDCl3

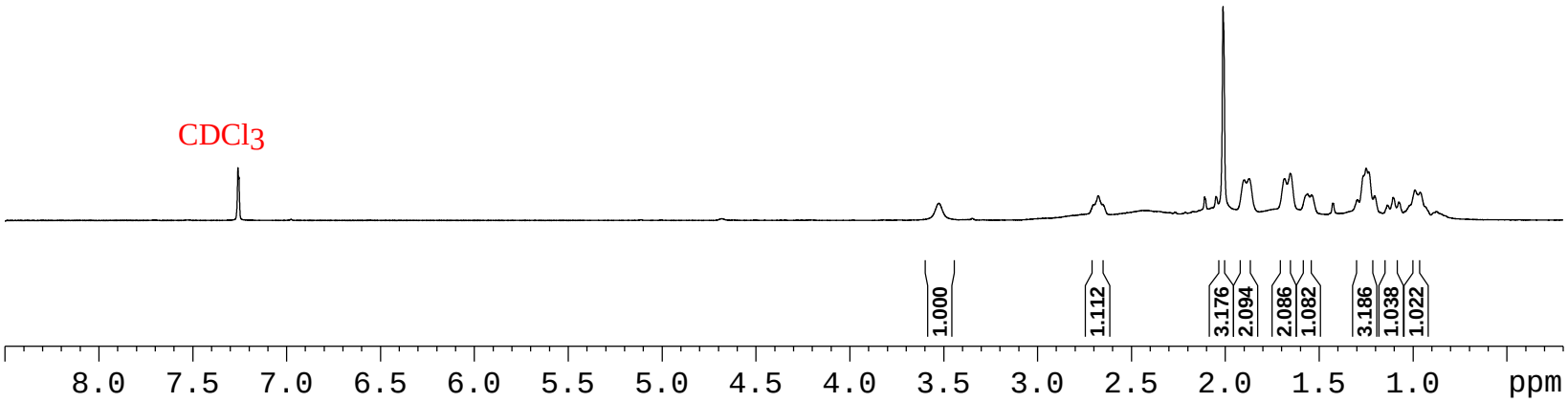
Bruker Advance III 400



20d

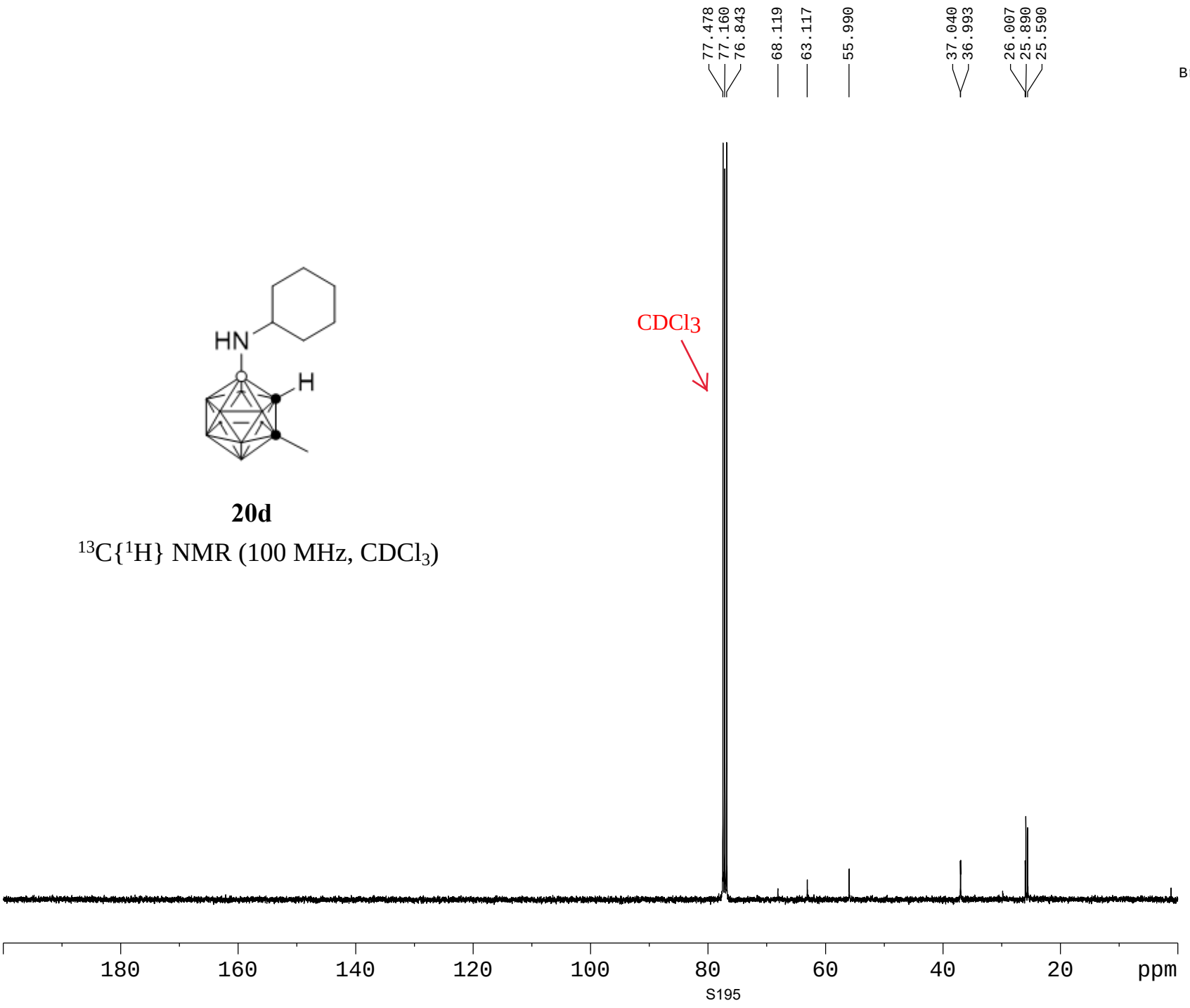
^{1}H NMR (400 MHz, $CDCl_3$)

```
Current Data Parameters
NAME      lhr-H-0556-12-cc-CDCl3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161216
Time                9.35 h
INSTRUM            spect
PROBHD   Z824601_0021 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    12
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                296.8 K
D1           1.00000000 sec
TD0                    1
SFO1         400.1324708 MHz
NUC1                  1H
P1                15.00 usec
PLW1          8.31000042 W

F2 - Processing parameters
SI                 65536
SF           400.1300104 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

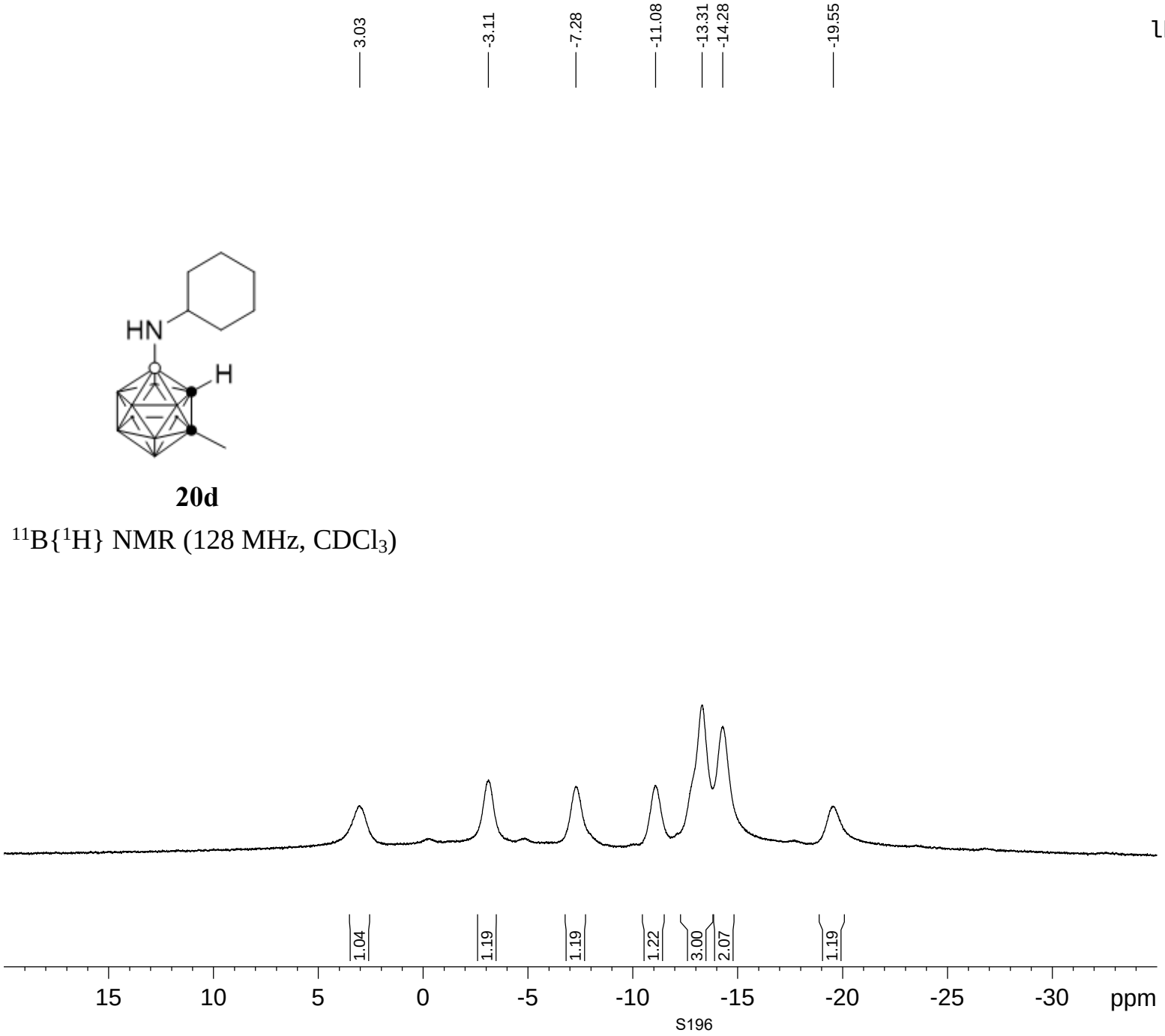


20d

$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)




```
lhr-C-0556-12-cc-CDCl3

Bruker Advance III 400

Current Data Parameters
NAME      lhr-C-0556-12-cc-CDCl3
EXPNO                          1
PROCNO                         1

F2 - Acquisition Parameters
Date_           20161216
Time              14.12 h
INSTRUM             spect
PROBHD   Z824601_0021 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1351
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                  203
DW                20.800 usec
DE                 6.50 usec
TE                295.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1               13C
P1                 9.50 usec
PLW1        41.25000000 W
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         8.31000042 W
PLW12        0.23083000 W
PLW13        0.11611000 W

F2 - Processing parameters
SI                32768
SF          100.6127549 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



20d

$^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$)

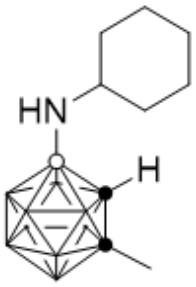
lhr-B-0556-12-cc-CDCl3

```
Current Data Parameters
NAME      lhr-B-0556-12-cc-CDCl3
EXPNO                          1
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20161216
Time                       10.37 h
INSTRUM                    spect
PROBHD   Z108618_0257 (
PULPROG                     zgdc
TD                         65536
SOLVENT                     C6D6
NS                            22
DS                             4
SWH                    24038.461 Hz
FIDRES                  0.366798 Hz
AQ                     1.3631488 sec
RG                           287
DW                        20.800 usec
DE                         6.50 usec
TE                        296.6 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
```

3.21 -2.35 -3.50 -6.55 -7.65 -10.33 -11.50 -12.51 -13.58 -14.72 -18.70 -19.99

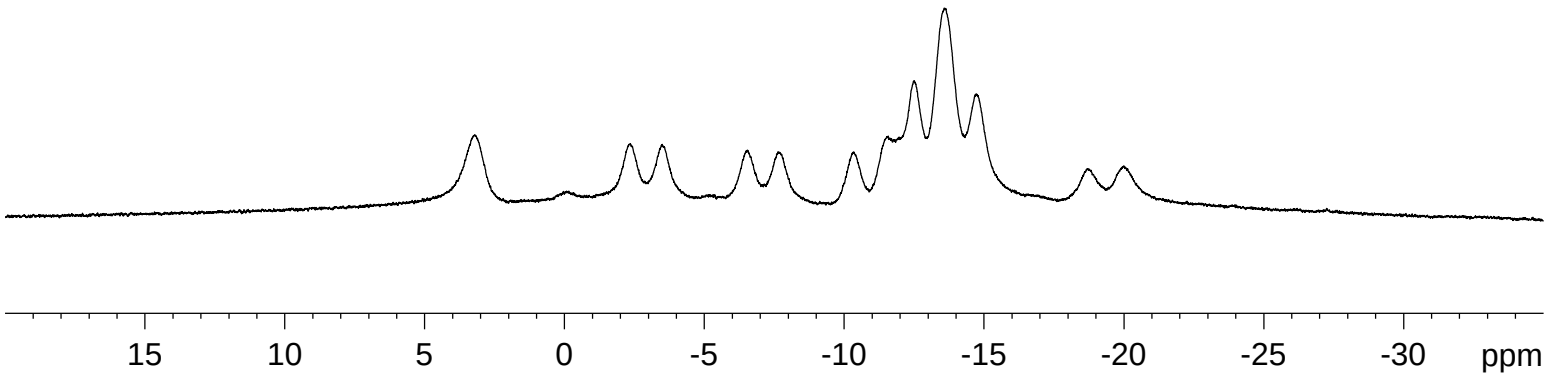
lhr-B-0556-12-cc-CDCl3(C

```
Current Data Parameters
NAME      lhr-B-0556-12-cc-CDCl3(C)
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161216
Time              10.39 h
INSTRUM            spect
PROBHD   Z108618_0257 (
PULPROG               zg
TD                 65536
SOLVENT             C6D6
NS                    31
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                296.1 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
```



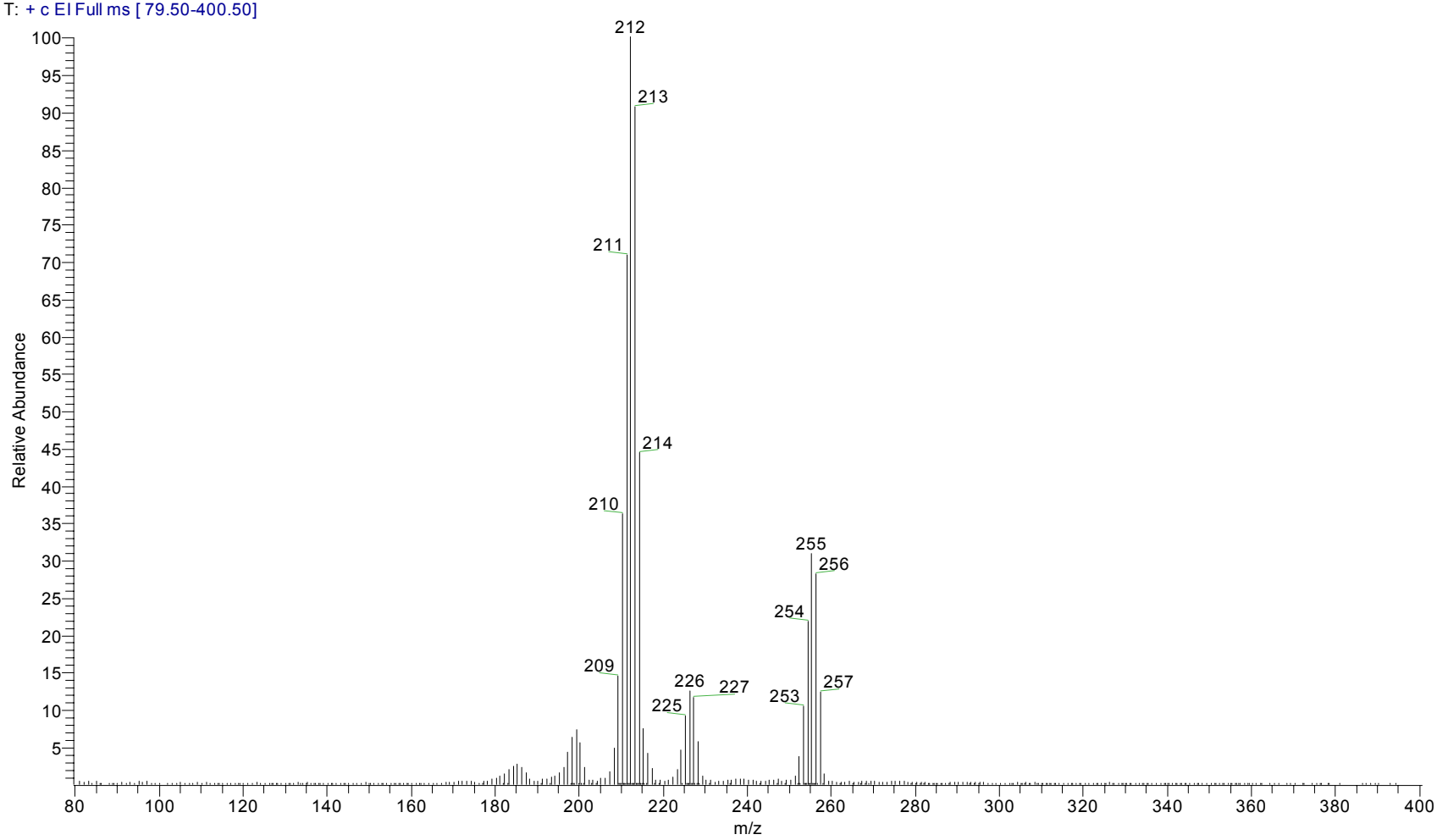
20d

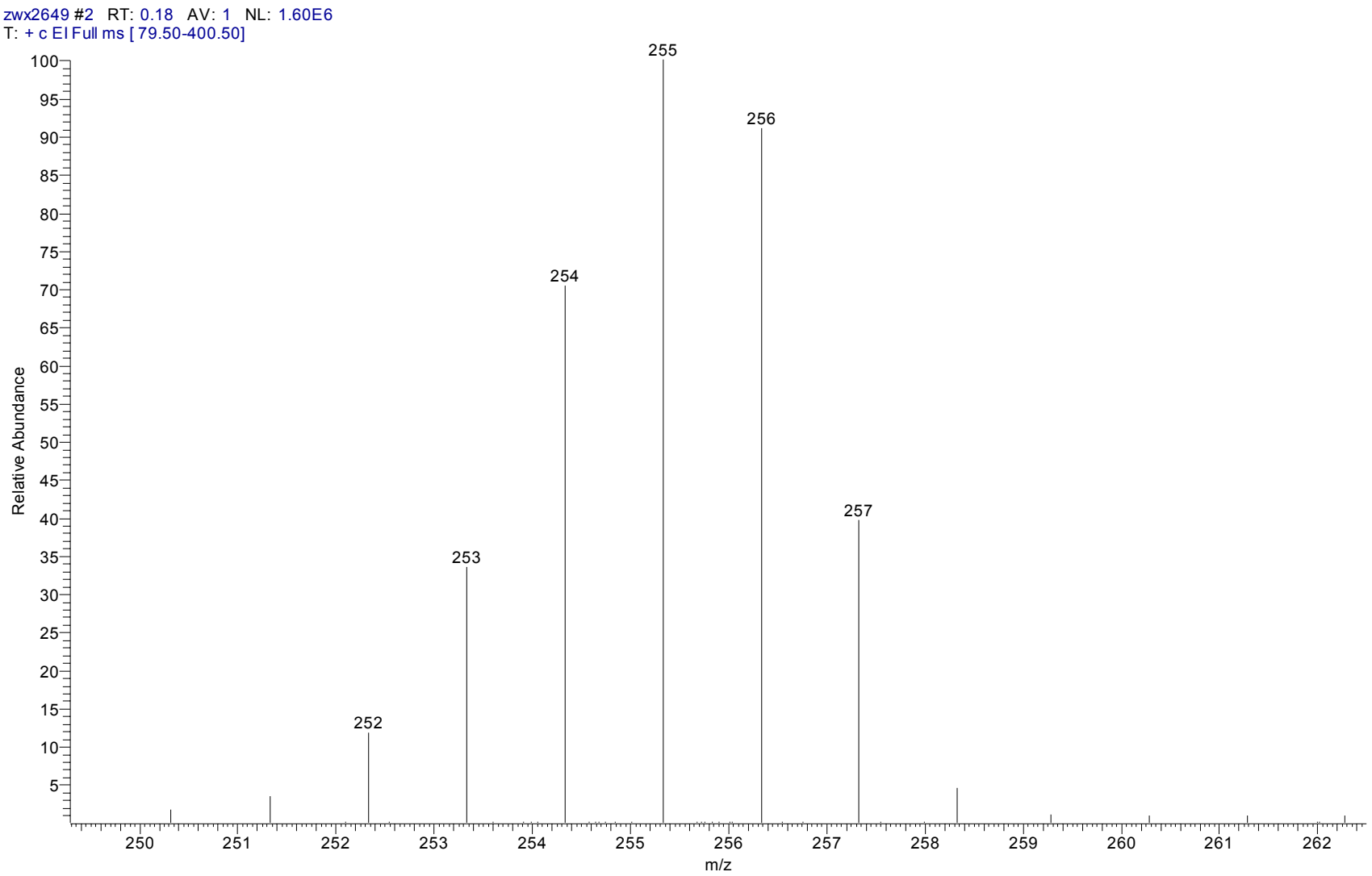
^{11}B NMR (128 MHz, $CDCl_3$)



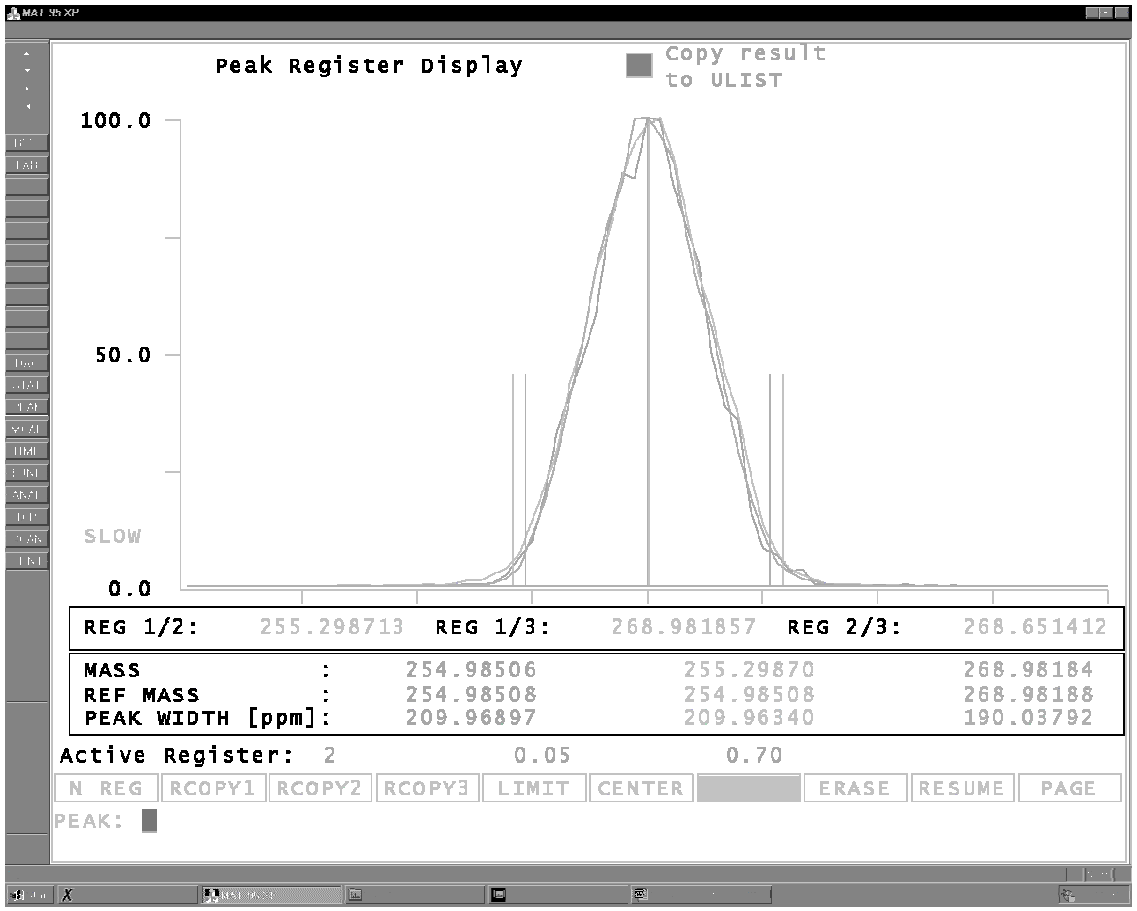
D:\MS_raw_data\zwx2649 03/14/17 02:23:21 PM lhr-0556
ei pos, 50eV, 0.7mA, unknown conc.

zwx2649 #2 RT: 0.18 AV: 1 NL: 5.17E6
T: + c EI Full ms [79.50-400.50]

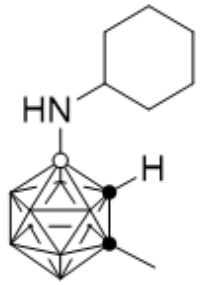



zwx2649 #2 RT: 0.18 AV: 1 NL: 1.60E6
T: + c EI Full ms [79.50-400.50]
Relative Abundance
m/z
255
256
254
253
257
252
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
250
251
252
253
254
255
256
257
258
259
260
261
262

Accurate Mass Measurement



Molecular formula
$C_9H_{25}B_{10}N$
$[M]^+$ (theoretical)
= 255.2985



20d

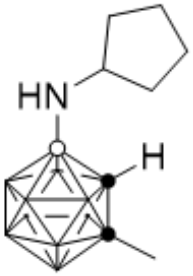
7.160

3.361 3.344 3.326 3.309 3.292

2.542 1.867 1.852 1.837 1.823 1.810 1.569 1.547 1.536 1.530 1.425 1.414 1.408 1.396 1.361 1.211 1.153 1.135 1.117 1.103 1.085 1.063

lhr-H-0578-ce-CDCl3

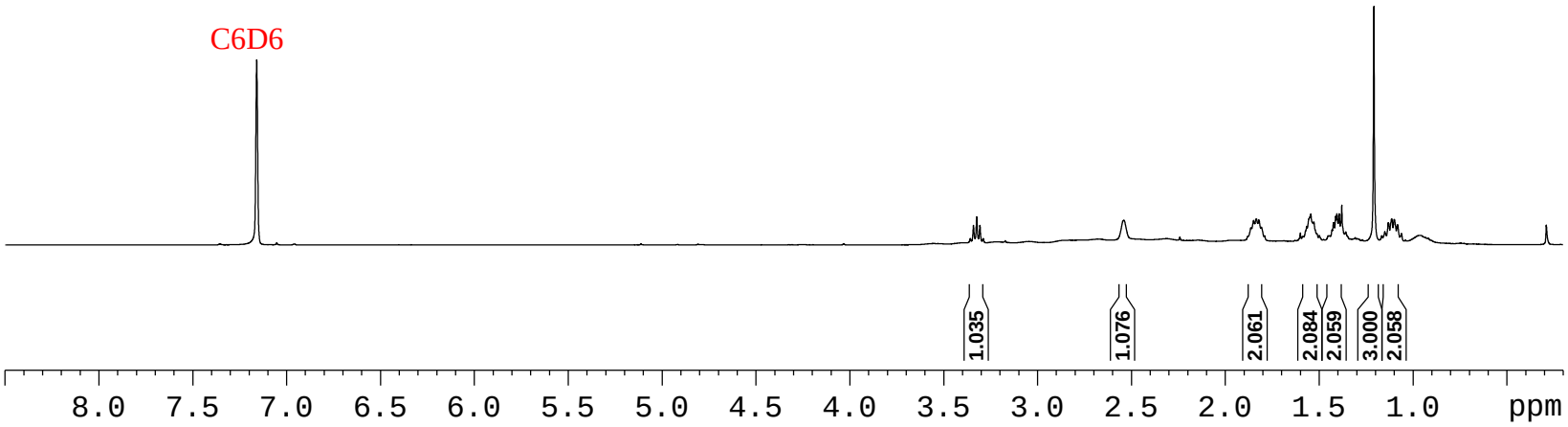
Bruker Advance III 400

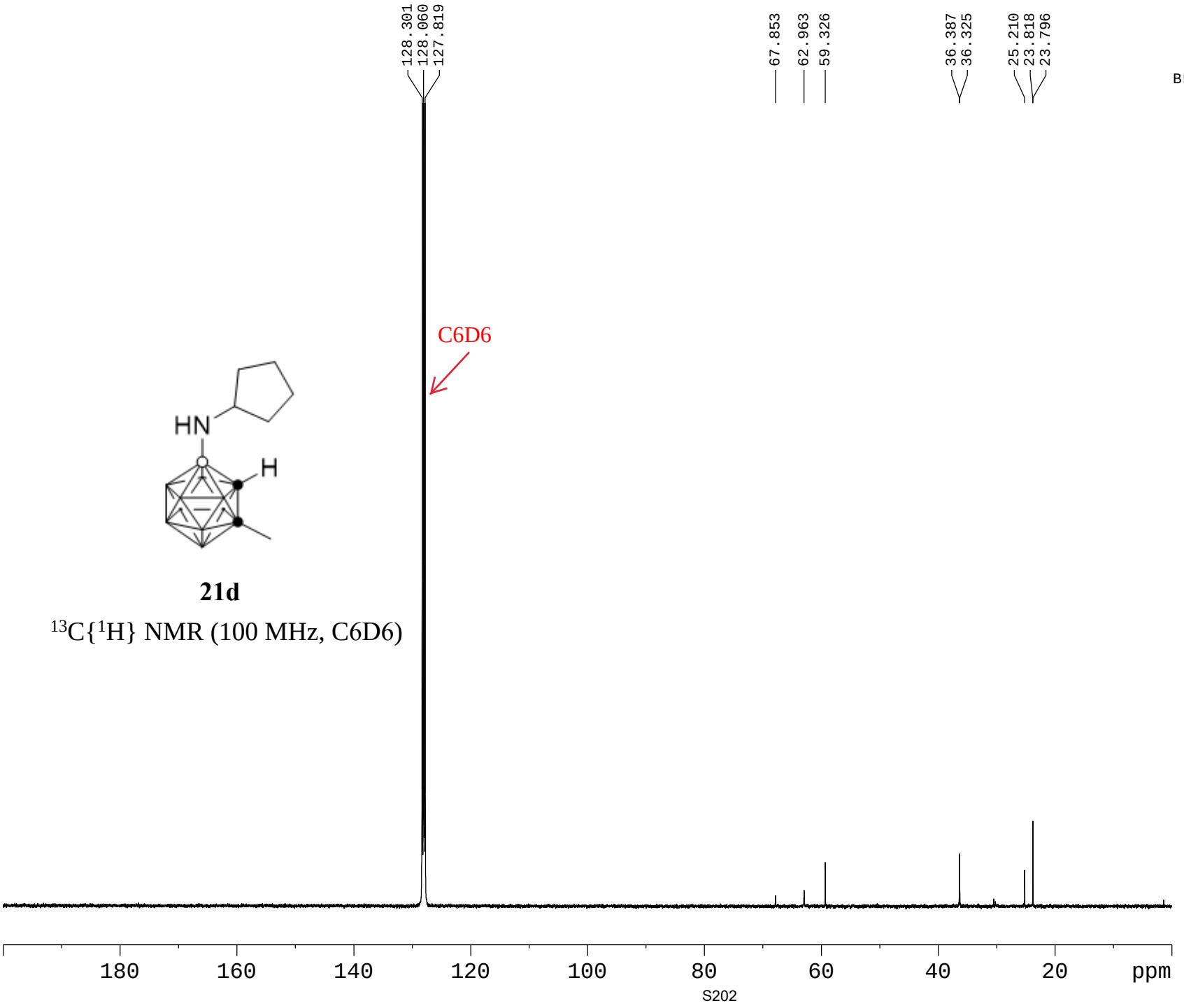


21d

^{1}H NMR (400 MHz, C6D6)

```
Current Data Parameters
NAME      lhr-H-0578-ce-CDCl3
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20161229
Time              17.25 h
INSTRUM             spect
PROBHD   Z824601_0021 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    15
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                295.1 K
D1             1.00000000 sec
TD0                    1
SFO1         400.1324708 MHz
NUC1                  1H
P1                15.00 usec
PLW1          8.31000042 W

F2 - Processing parameters
SI                 65536
SF           400.1300286 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```





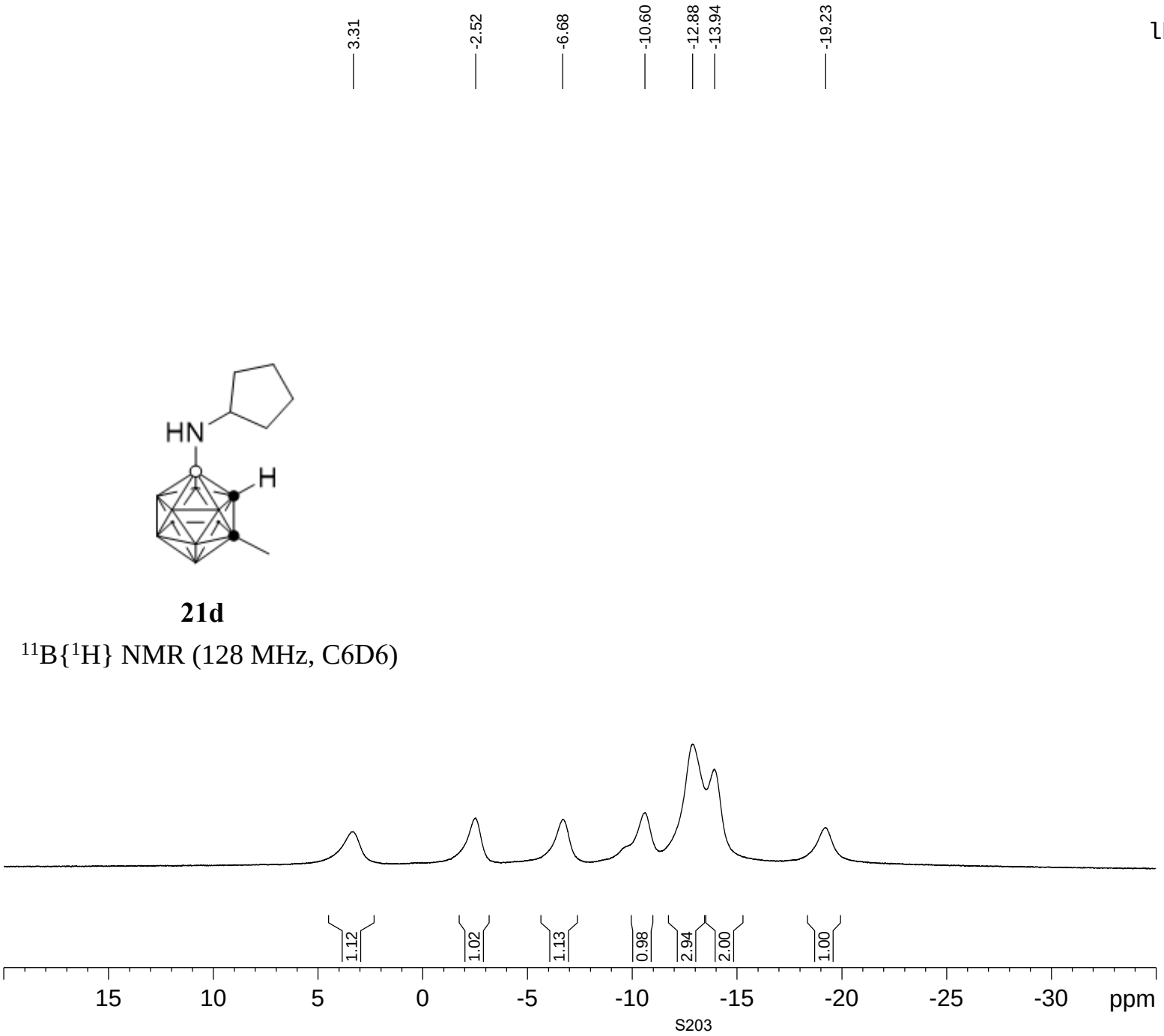
21d

$^{13}C\{^{1}H\}$ NMR (100 MHz, C6D6)

lhr-C-0578-ce-C6D6

Bruker Advance III 400

```
Current Data Parameters
NAME      lhr-C-0578-ce-C6D6
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_           20161229
Time              17.32 h
INSTRUM             spect
PROBHD    Z824601_0021 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1173
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                   203
DW                20.800 usec
DE                 6.50 usec
TE                295.7 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1
SFO1         100.6228298 MHz
NUC1                 13C
P1                 9.50 usec
PLW1         41.25000000 W
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2             90.00 usec
PLW2          8.31000042 W
PLW12         0.23083000 W
PLW13         0.11611000 W

F2 - Processing parameters
SI                 32768
SF           100.6127391 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

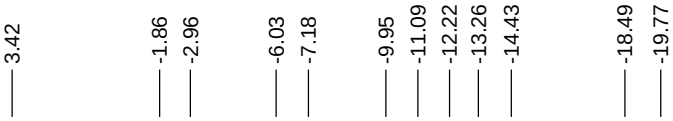


21d

$^{11}B\{^{1}H\}$ NMR (128 MHz, C6D6)

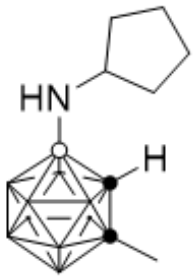
lhr-B-0578-ce-C6D6

```
Current Data Parameters
NAME      lhr-B-0578-ce-C6D6
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20161229
Time                  18.41 h
INSTRUM               spect
PROBHD   Z108618_0257 (
PULPROG                zgdc
TD                    65536
SOLVENT               CDCl3
NS                       21
DS                        4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                1.3631488 sec
RG                      287
DW                   20.800 usec
DE                    6.50 usec
TE                   294.6 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
```



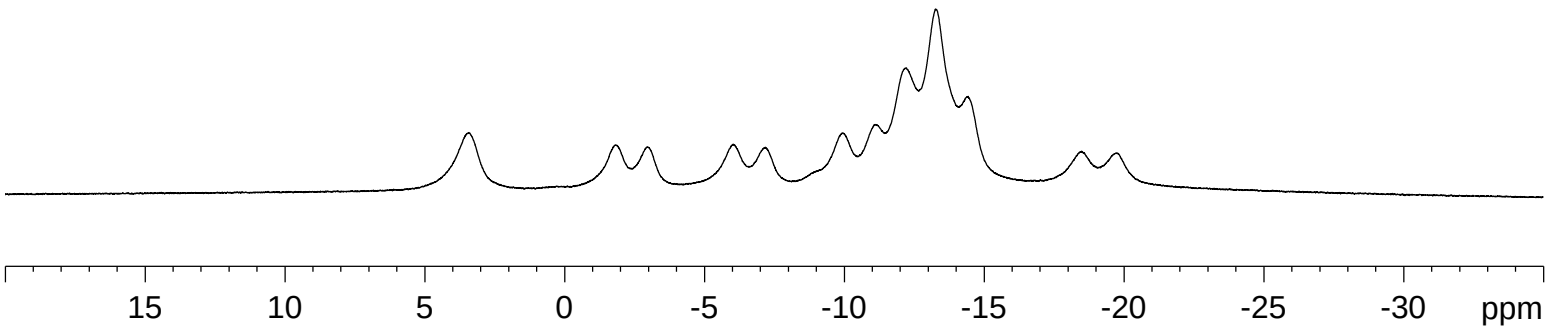
lhr-B-0578-ce-C6D6(C)

```
Current Data Parameters
NAME      lhr-B-0578-ce-C6D6(C)
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_              20161229
Time                  18.45 h
INSTRUM               spect
PROBHD   Z108618_0257 (
PULPROG                   zg
TD                     65536
SOLVENT                CDCl3
NS                        37
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                    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              128.4097504 MHz
WDW                       EM
SSB       0
LB                     1.00 Hz
GB        0
PC                     1.40
```



21d

^{11}B NMR (128 MHz, C6D6)



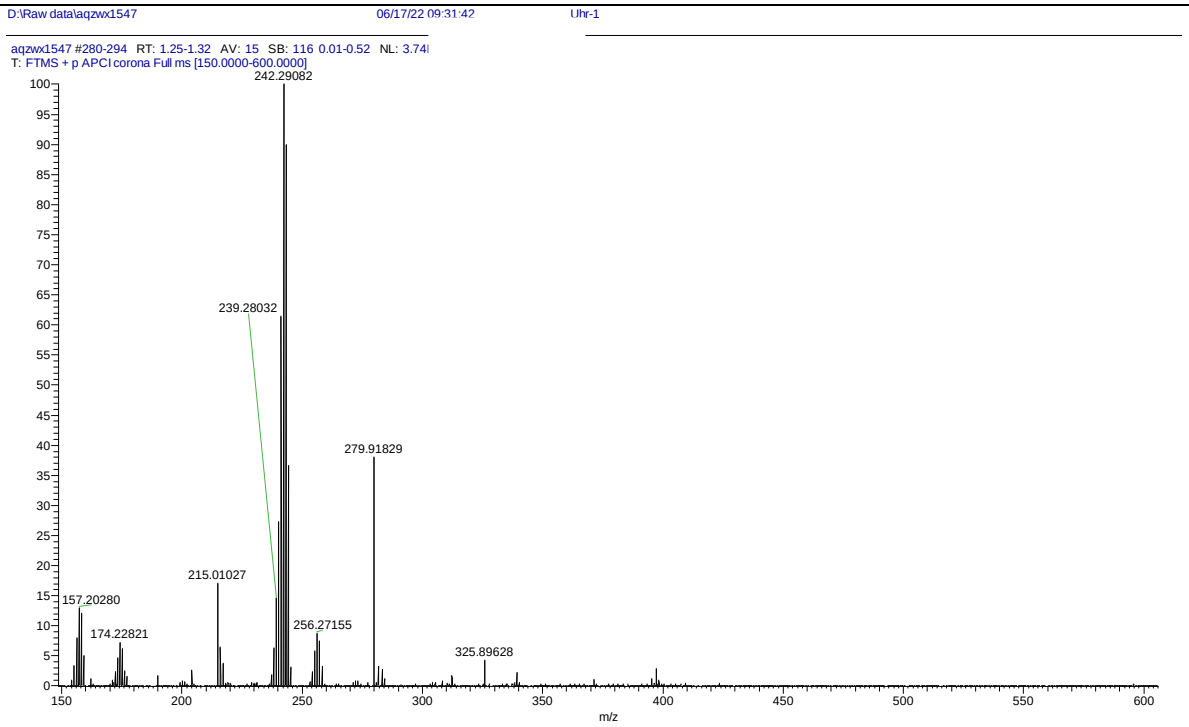
Thermo QEFMS Analysis Report

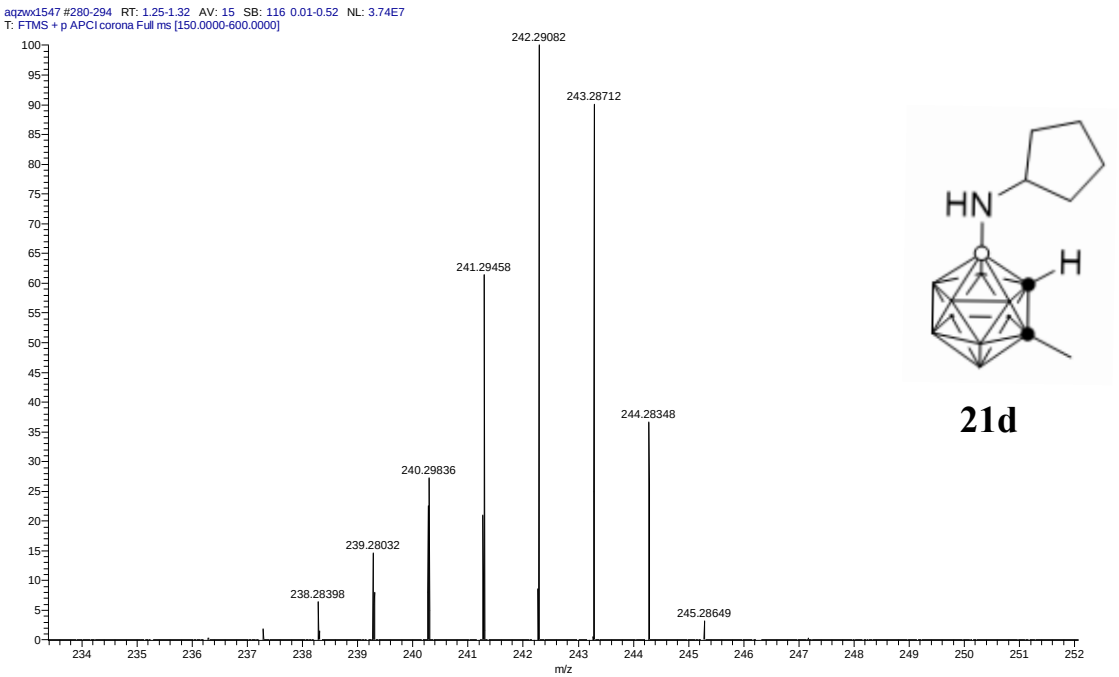
Analysis Info

Sample Name :	Uhr-1	Reference No.:	aqzwx1547
Instrument :	Q Exactive Focus Orbitrap		
Source :	APCI	Polarity :	Positive
Comment :	APCI, 4uA, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_8H_{23}B_{10}N$
Experimental Mass $[M+H]^+$:	242.29082
Theoretical Mass $[M+H]^+$:	242.29105
Error (ppm) :	-1.0

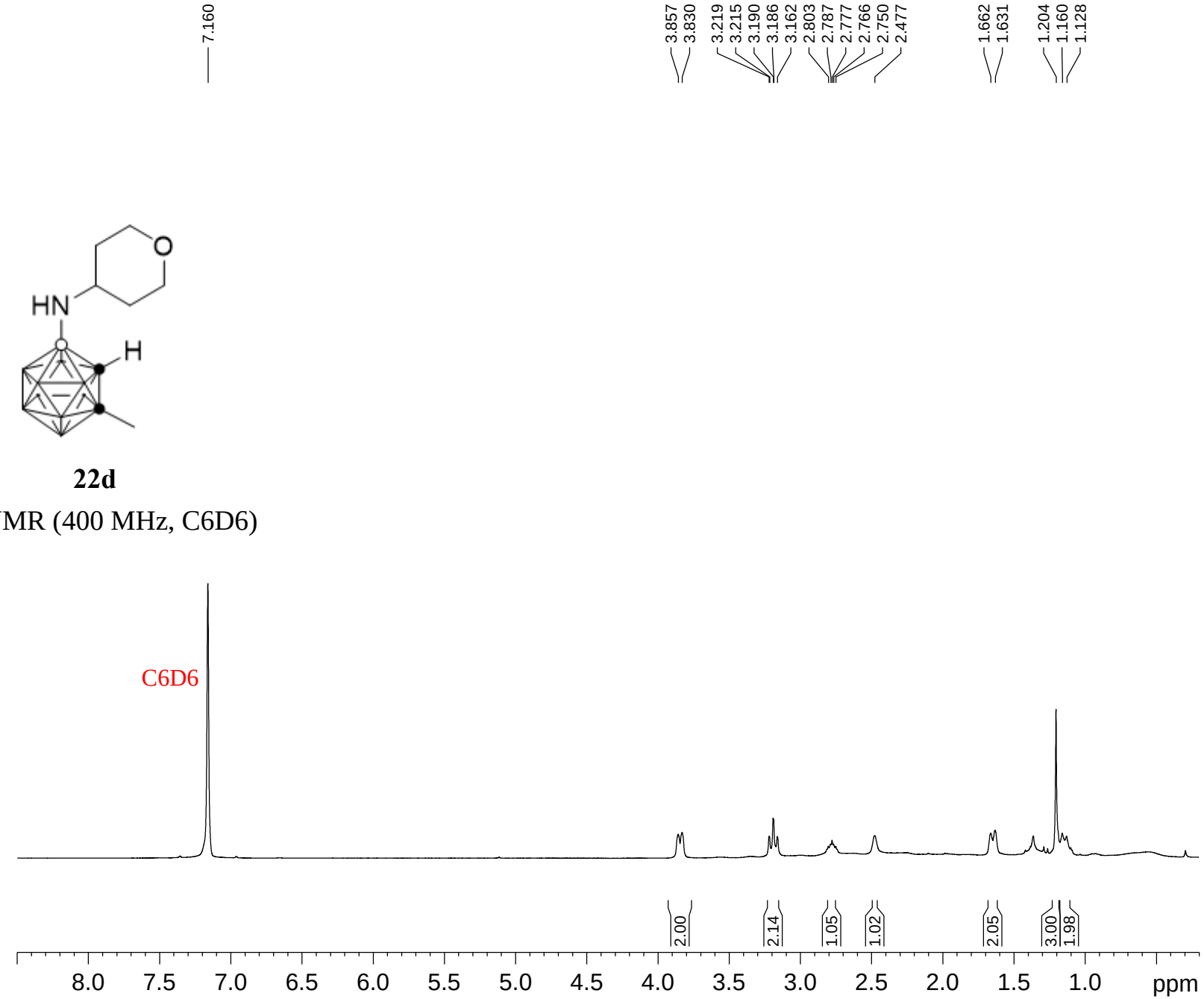




21d

22d

^{1}H NMR (400 MHz, C6D6)

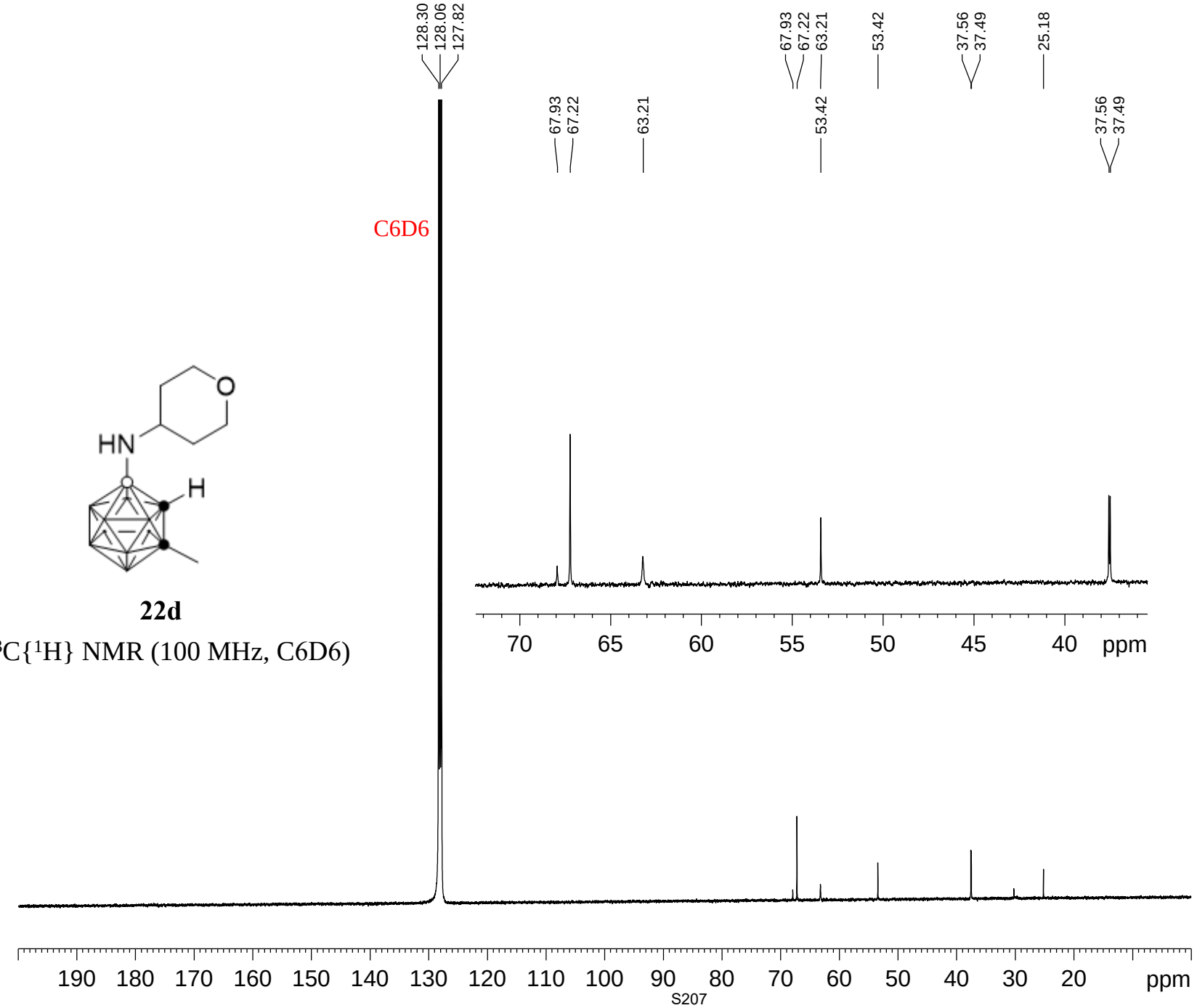


lhr-H-0581-1-cc-C6D

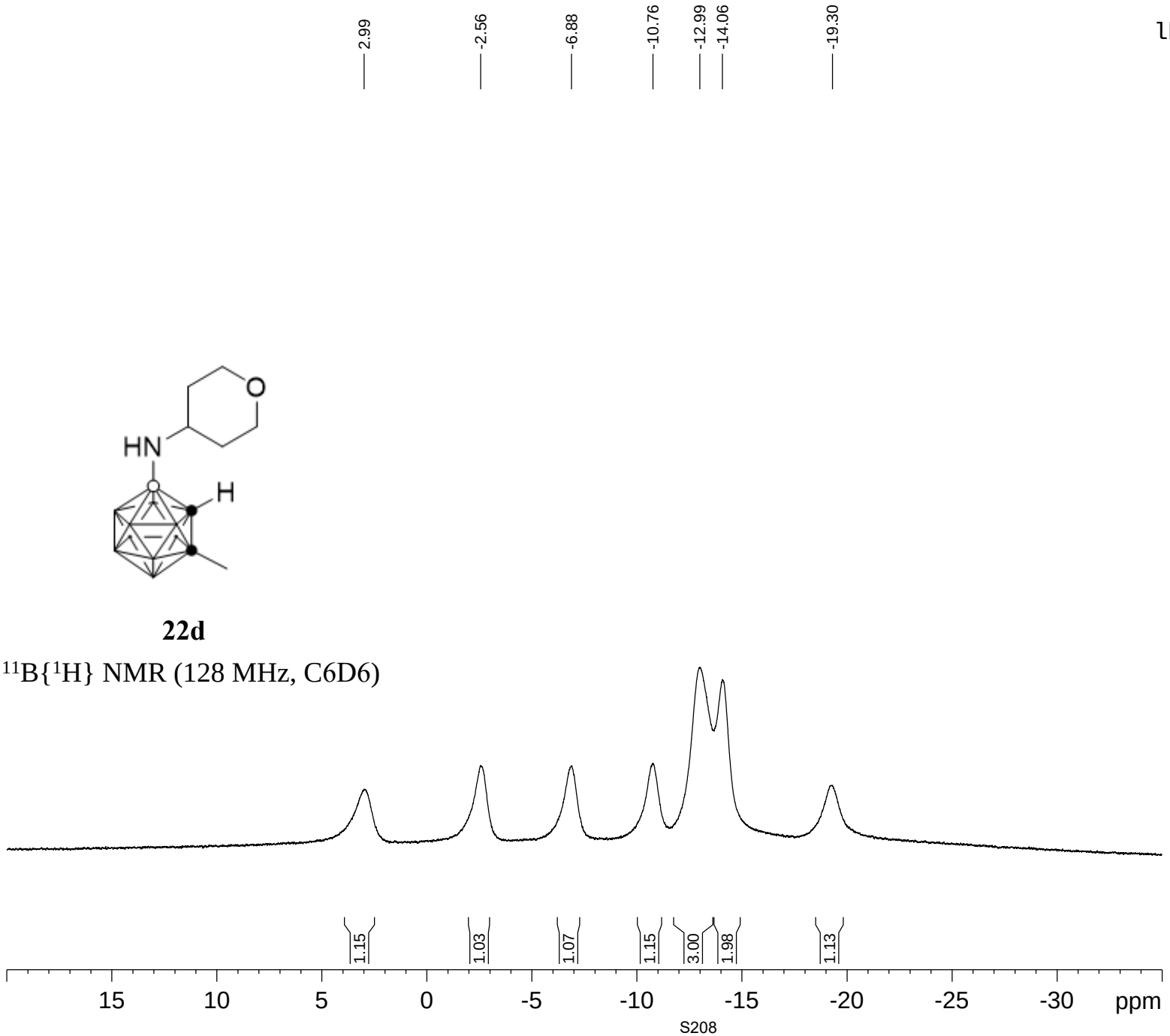
Current Data Parameters
NAME lhr-H-0581-1-cc-C6
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170101
Time 16.34 h
INSTRUM spect
PROBHD Z108618_0257 (
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 20
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 128
DW 62.400 usec
DE 6.50 usec
TE 294.1 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 400.2299971 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00




```
lhr-C-0581-1-cc-C6I

Current Data Parameters
NAME      lhr-C-0581-1-cc-C6D6
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_              20170101
Time                 16.41 h
INSTRUM                spect
PROBHD   Z108618_0257 (
PULPROG              zgpg30
TD                    65536
SOLVENT                C6D6
NS                     3769
DS                        4
SWH              29761.904 Hz
FIDRES            0.454131 Hz
AQ               1.1010048 sec
RG                      203
DW                 16.800 usec
DE                  6.50 usec
TE                 294.7 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             100.6378775 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

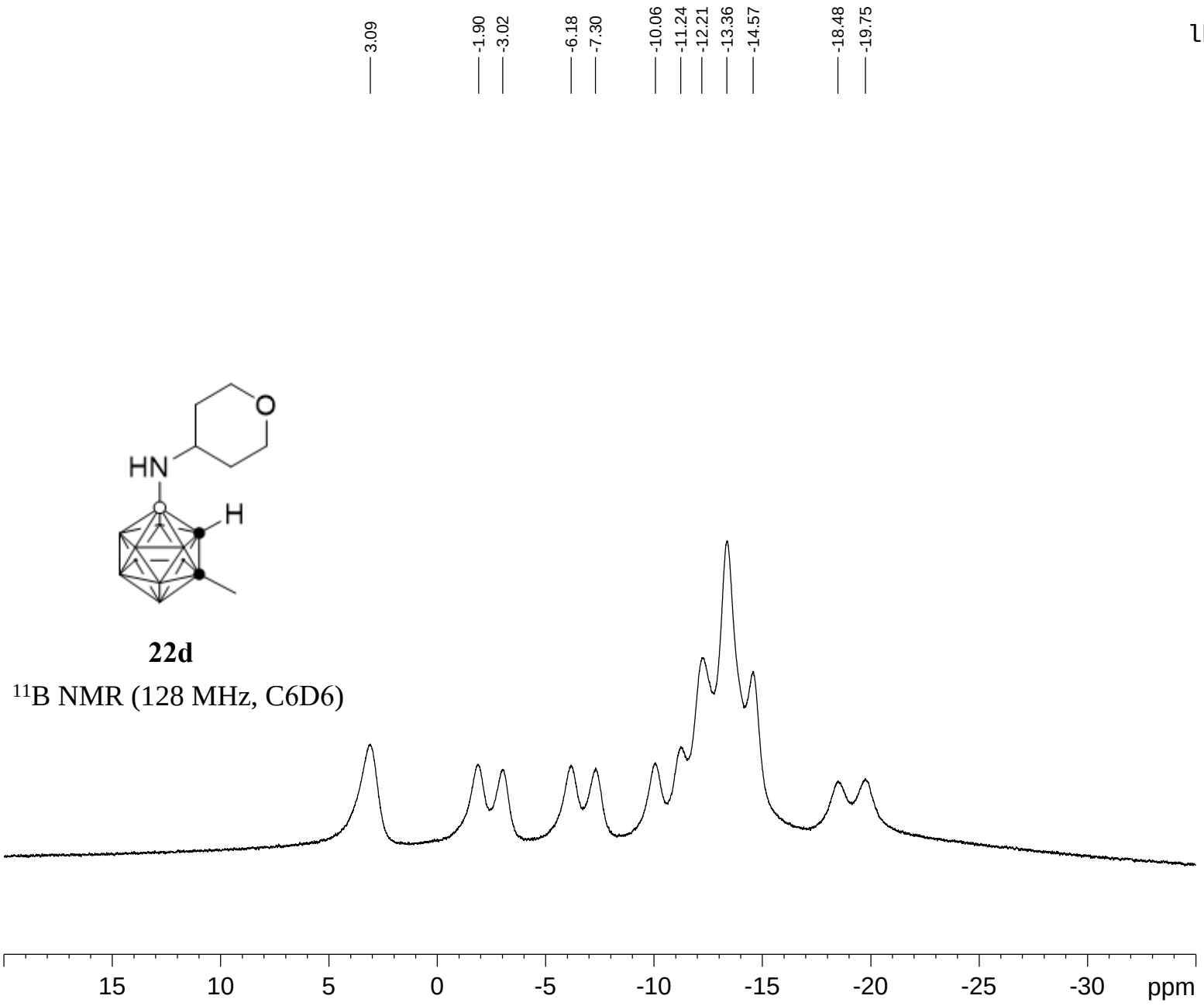


22d

$^{11}B\{^{1}H\}$ NMR (128 MHz, C6D6)

lhr-B-0581-1-cc-C6D6

```
Current Data Parameters
NAME      lhr-B-0581-1-cc-C6D6
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_              20170101
Time                  20.33 h
INSTRUM               spect
PROBHD   Z108618_0257 (
PULPROG                zgdc
TD                    65536
SOLVENT               CDCl3
NS                       43
DS                        4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                1.3631488 sec
RG                      287
DW                   20.800 usec
DE                    6.50 usec
TE                   295.8 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
```



22d

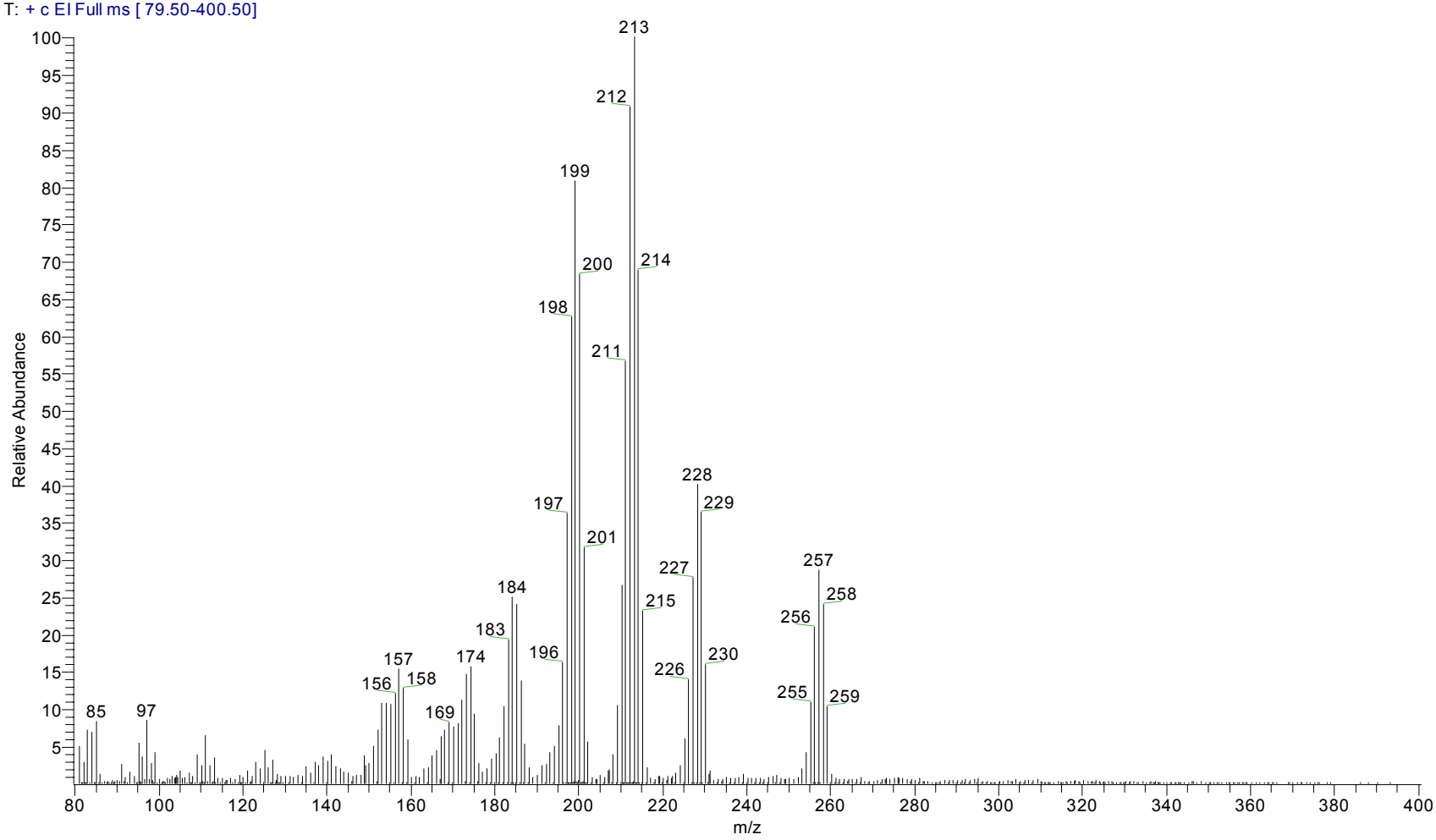
^{11}B NMR (128 MHz, C6D6)

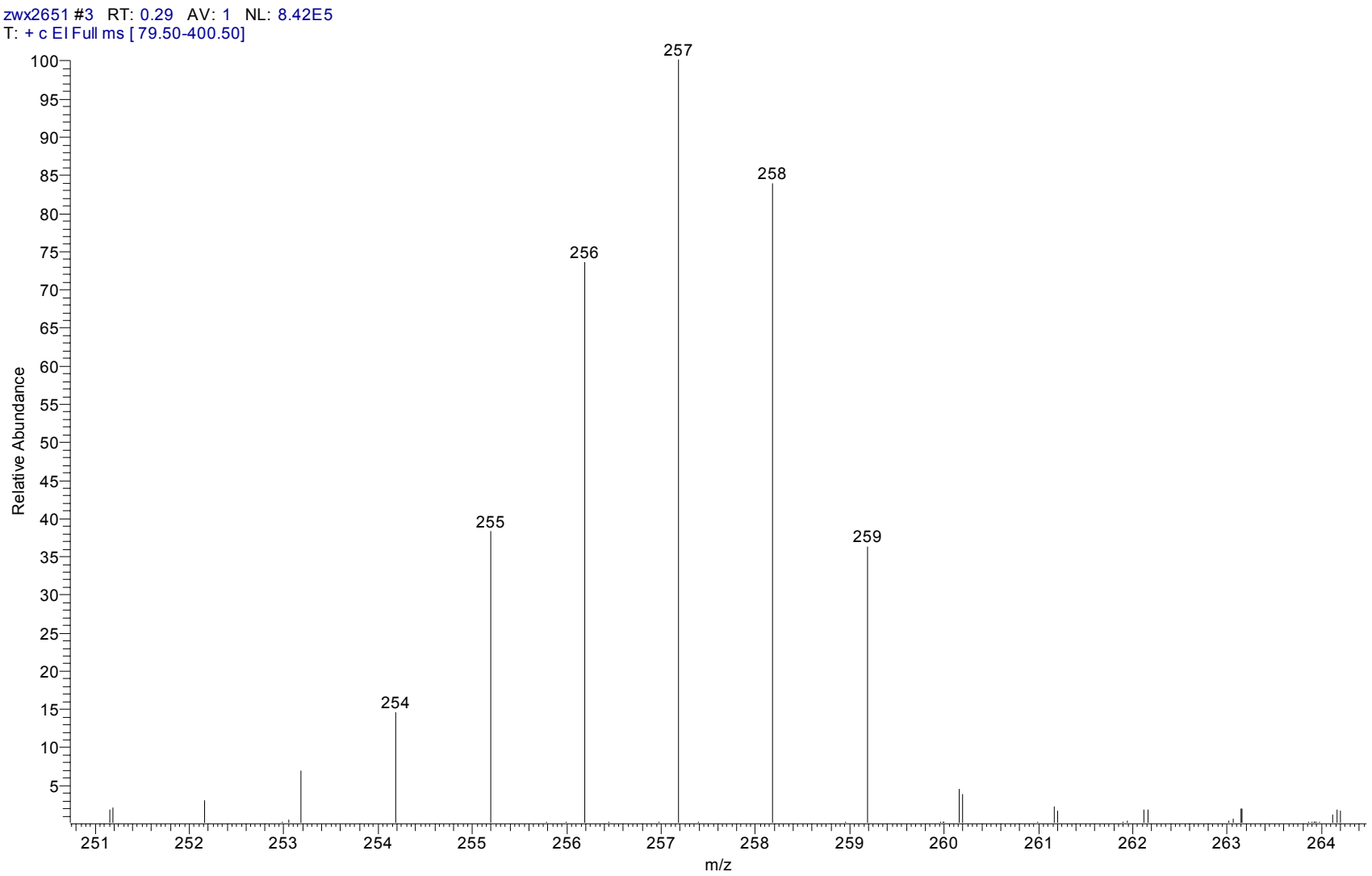
lhr-B-0581-1-cc-C6D6(C)

```
Current Data Parameters
NAME      lhr-B-0581-1-cc-C6D6(C)
EXPNO                          1
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20170101
Time                       20.35 h
INSTRUM                    spect
PROBHD    Z108618_0257 (
PULPROG                       zg
TD                         65536
SOLVENT                    CDCl3
NS                            95
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                        295.3 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                  128.4097504 MHz
WDW                           EM
SSB       0
LB                         1.00 Hz
GB        0
PC                         1.40
```

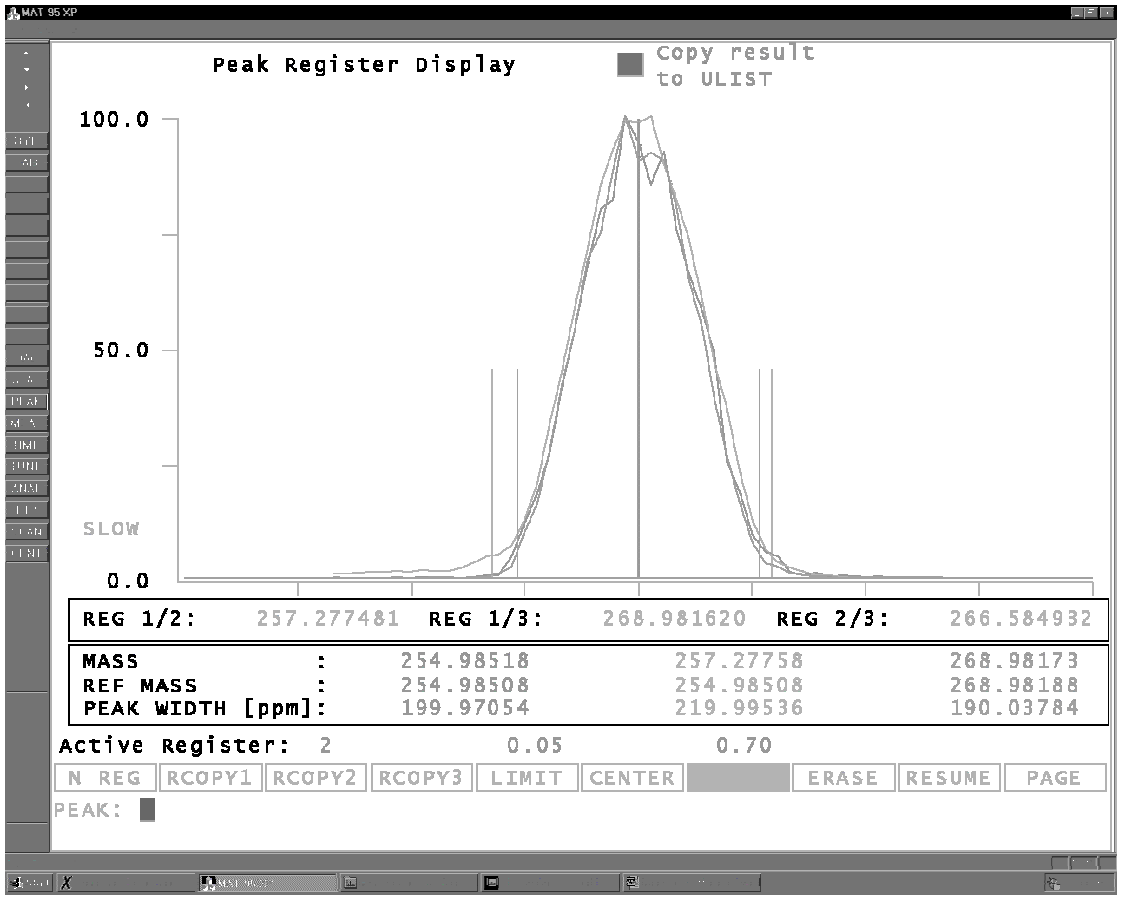
D:\MS_raw_data\zwx2651 03/14/17 02:54:56 PM lhr-0581-1
ei pos, 50eV, 0.7mA, unknown conc.

zwx2651 #3 RT: 0.29 AV: 1 NL: 2.95E6
T: + c EI Full ms [79.50-400.50]

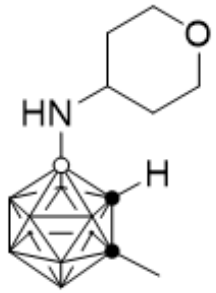



zwx2651 #3 RT: 0.29 AV: 1 NL: 8.42E5
T: + c EI Full ms [79.50-400.50]
Relative Abundance
m/z
257
258
256
255
259
254

Accurate Mass Measurement

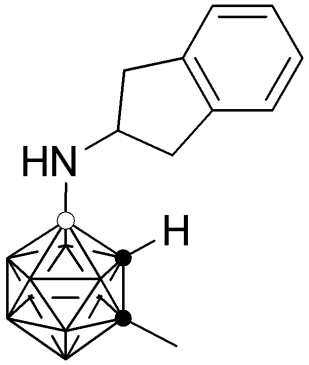


Molecular formula
$C_8H_{23}B_{10}NO$
[M]+ (theoretical)
= 257.2777



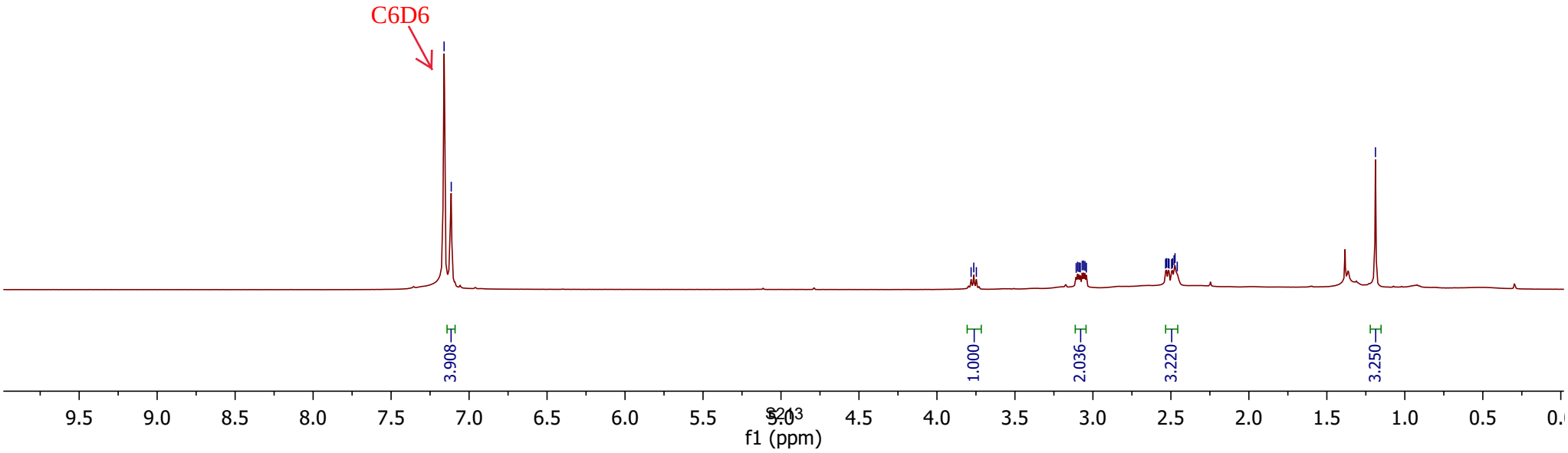
22d

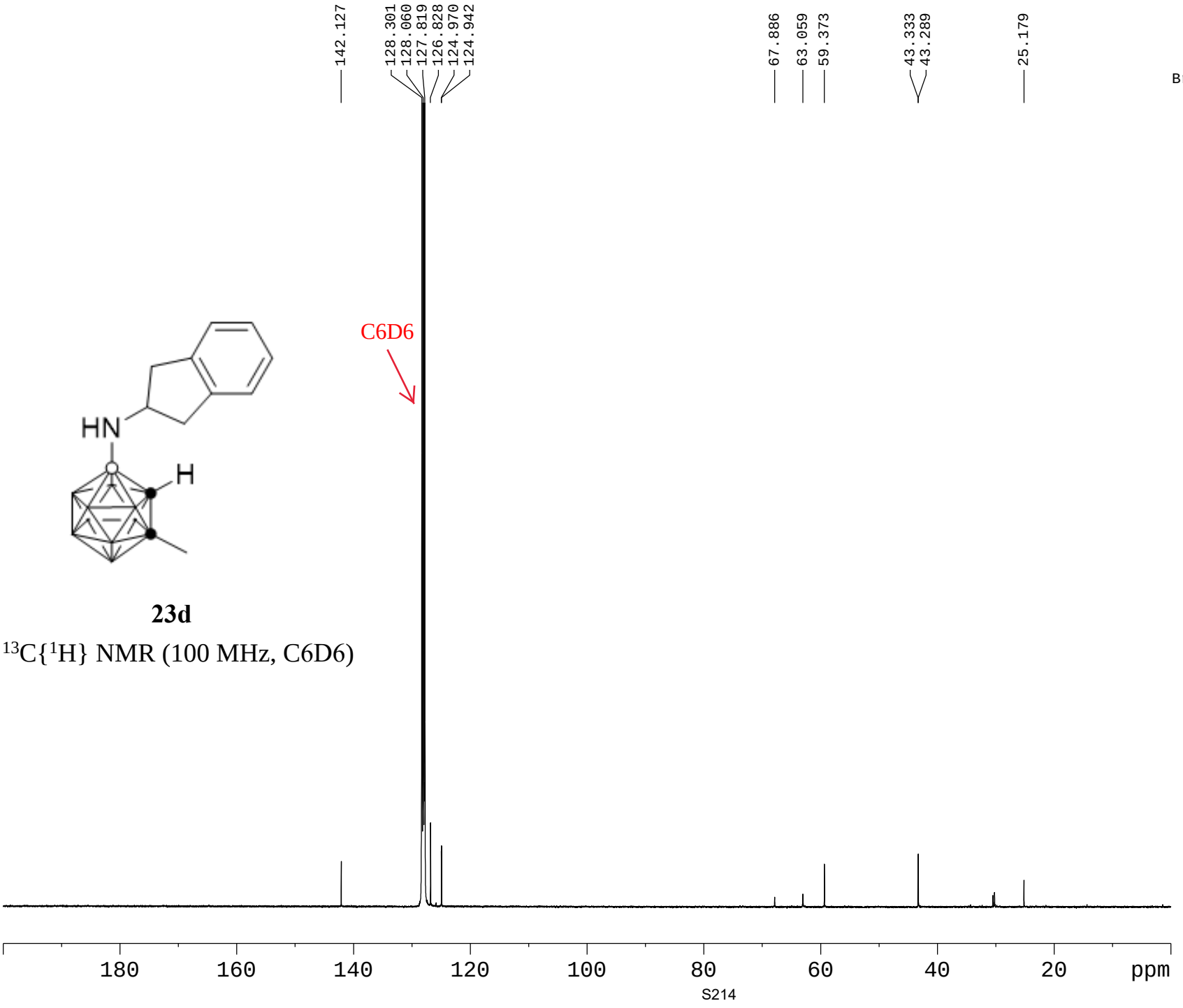
lhr-H-0580-cc-C6D6.1.1.1r
lhr-H-0580-cc-C6D6



23d

^{1}H NMR (400 MHz, C_6D_6)





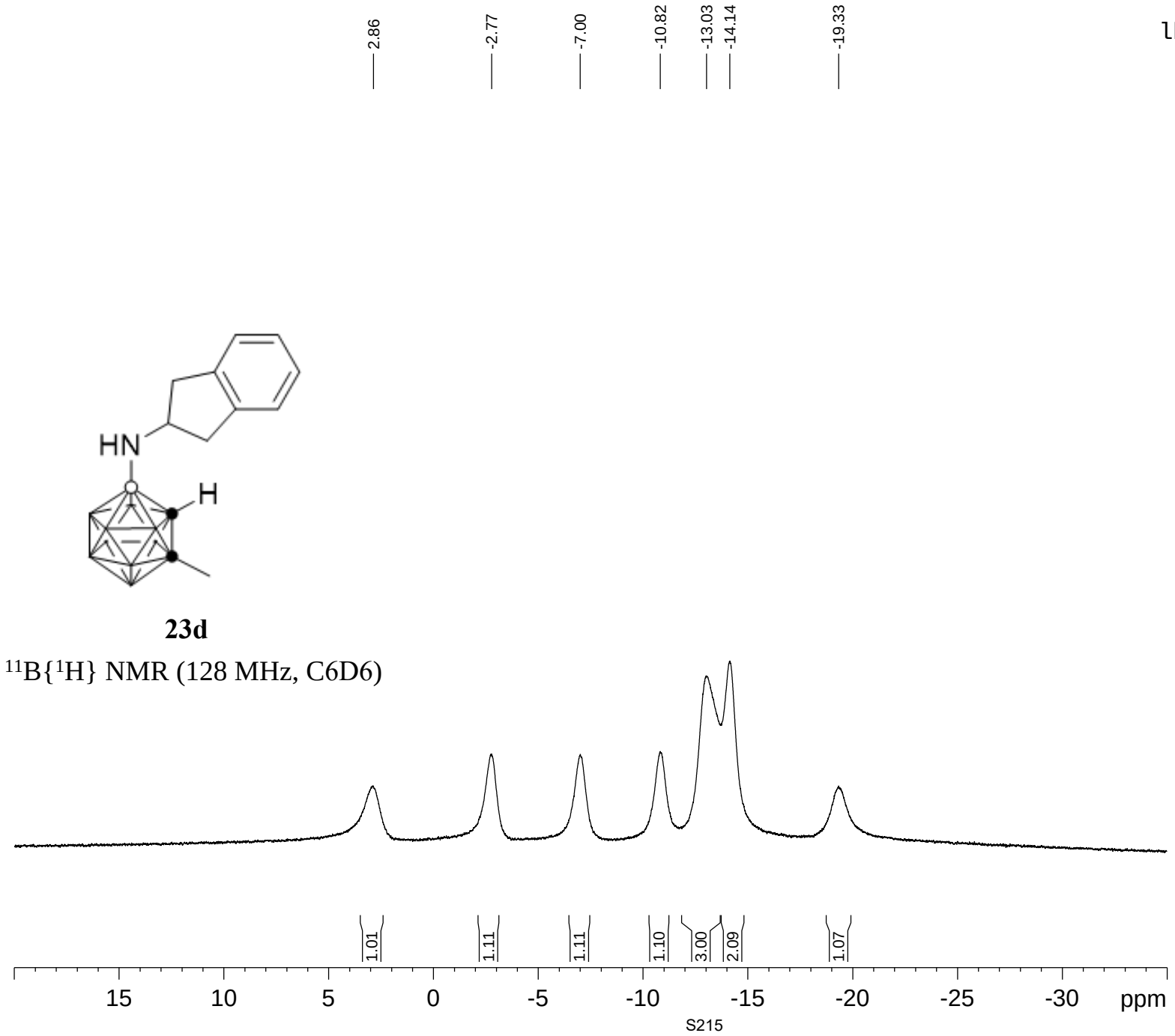
23d

$^{13}C\{^{1}H\}$ NMR (100 MHz, C6D6)

lhr-C-0580-cc-C6D6

Bruker Advance III 400

```
Current Data Parameters
NAME      lhr-C-0580-cc-C6D6
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_            20170103
Time                21.55 h
INSTRUM             spect
PROBHD    Z824601_0021 (
PULPROG           zgpg30
TD                 65536
SOLVENT             C6D6
NS                 12213
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                   203
DW                20.800 usec
DE                 6.50 usec
TE                295.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1
SFO1         100.6228298 MHz
NUC1                 13C
P1                 9.50 usec
PLW1         41.25000000 W
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2          8.31000042 W
PLW12         0.23083000 W
PLW13         0.11611000 W

F2 - Processing parameters
SI                 32768
SF           100.6127315 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

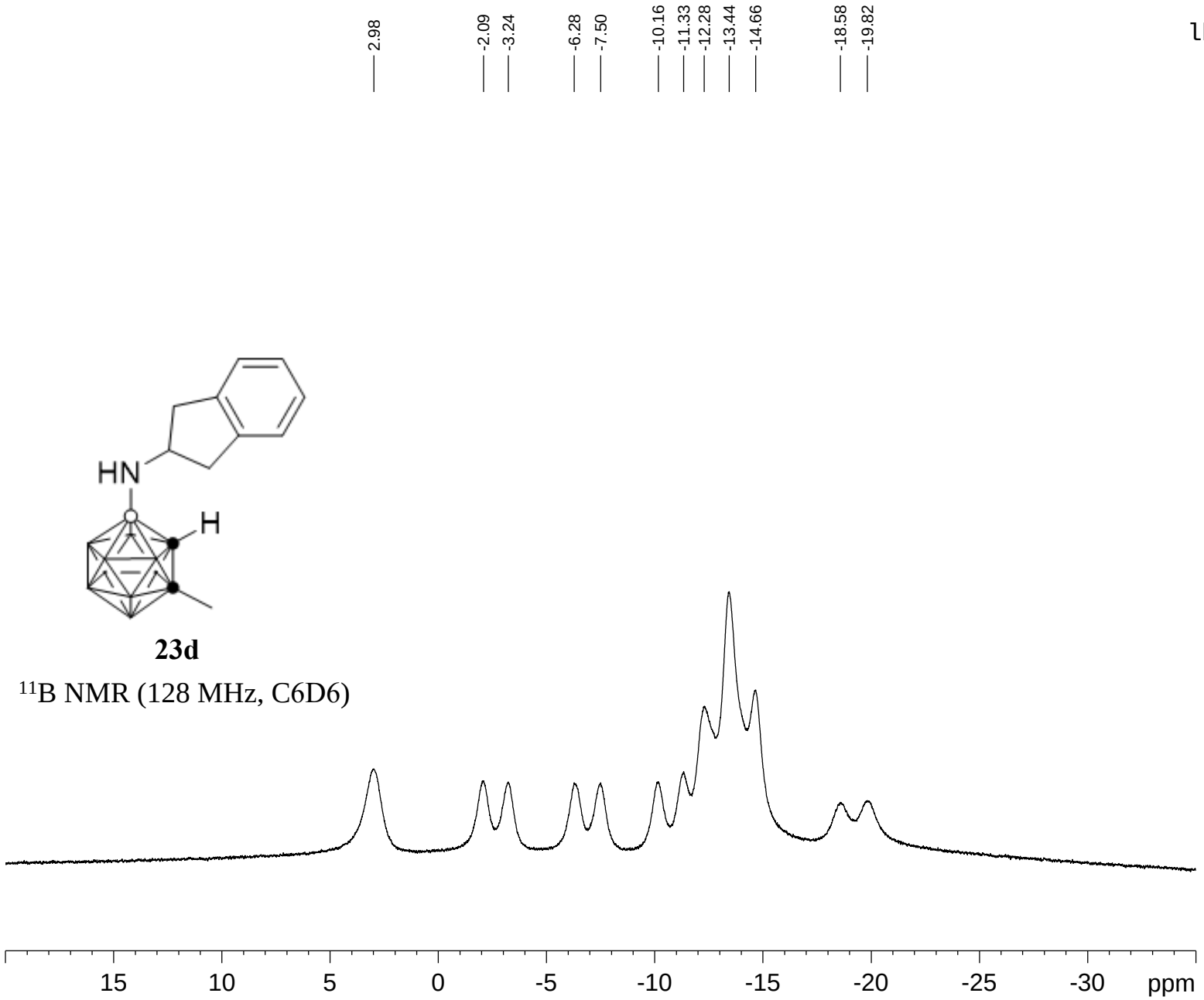


23d

$^{11}B\{^{1}H\}$ NMR (128 MHz, C6D6)

lhr-B-0580-cc-C6D6

```
Current Data Parameters
NAME      lhr-B-0580-cc-C6D6
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20170103
Time                  19.06 h
INSTRUM               spect
PROBHD   Z108618_0257 (
PULPROG                zgdc
TD                    65536
SOLVENT                C6D6
NS                       32
DS                        4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631488 sec
RG                      287
DW                   20.800 usec
DE                    6.50 usec
TE                   295.4 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
```



23d

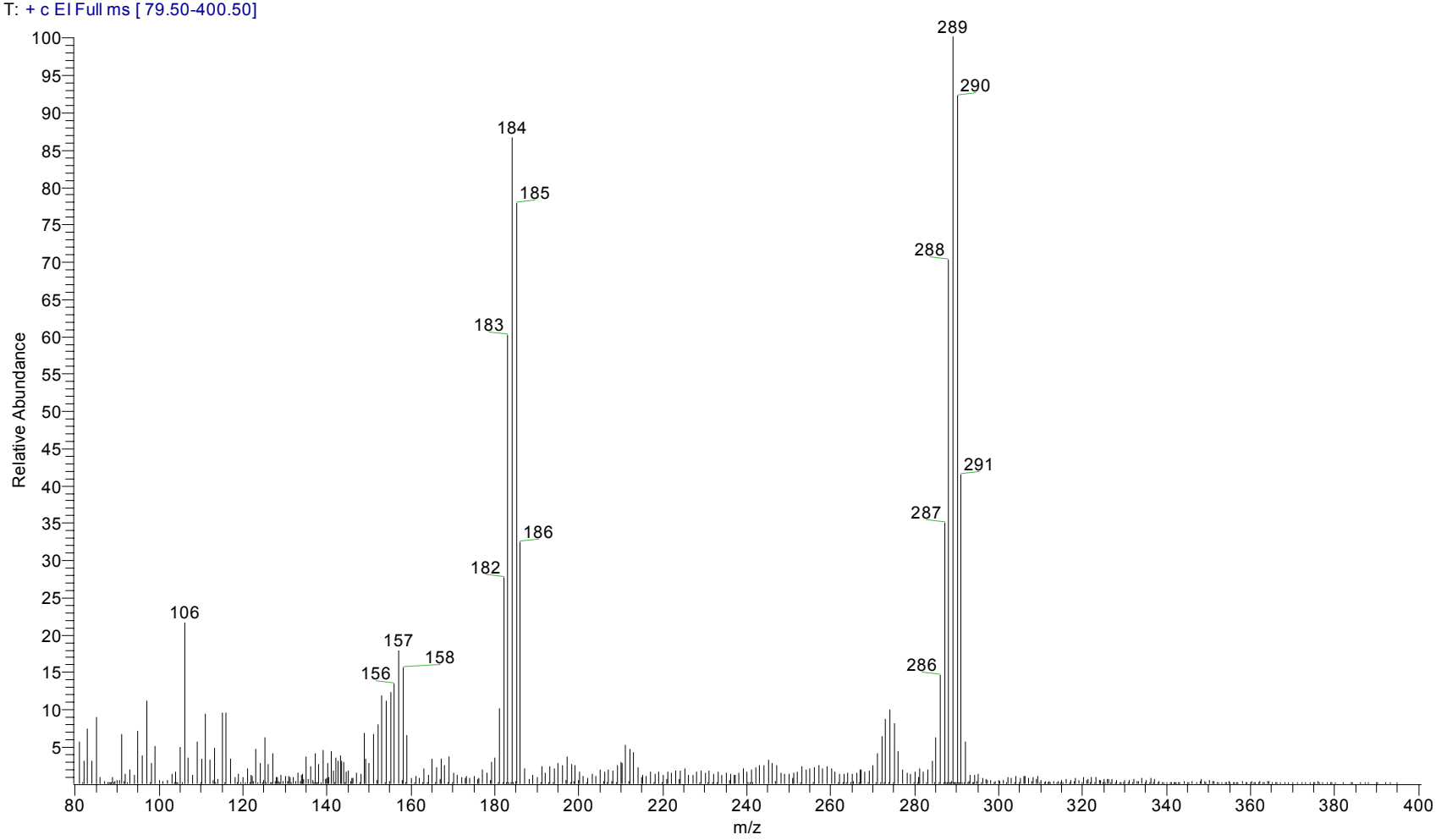
^{11}B NMR (128 MHz, C6D6)

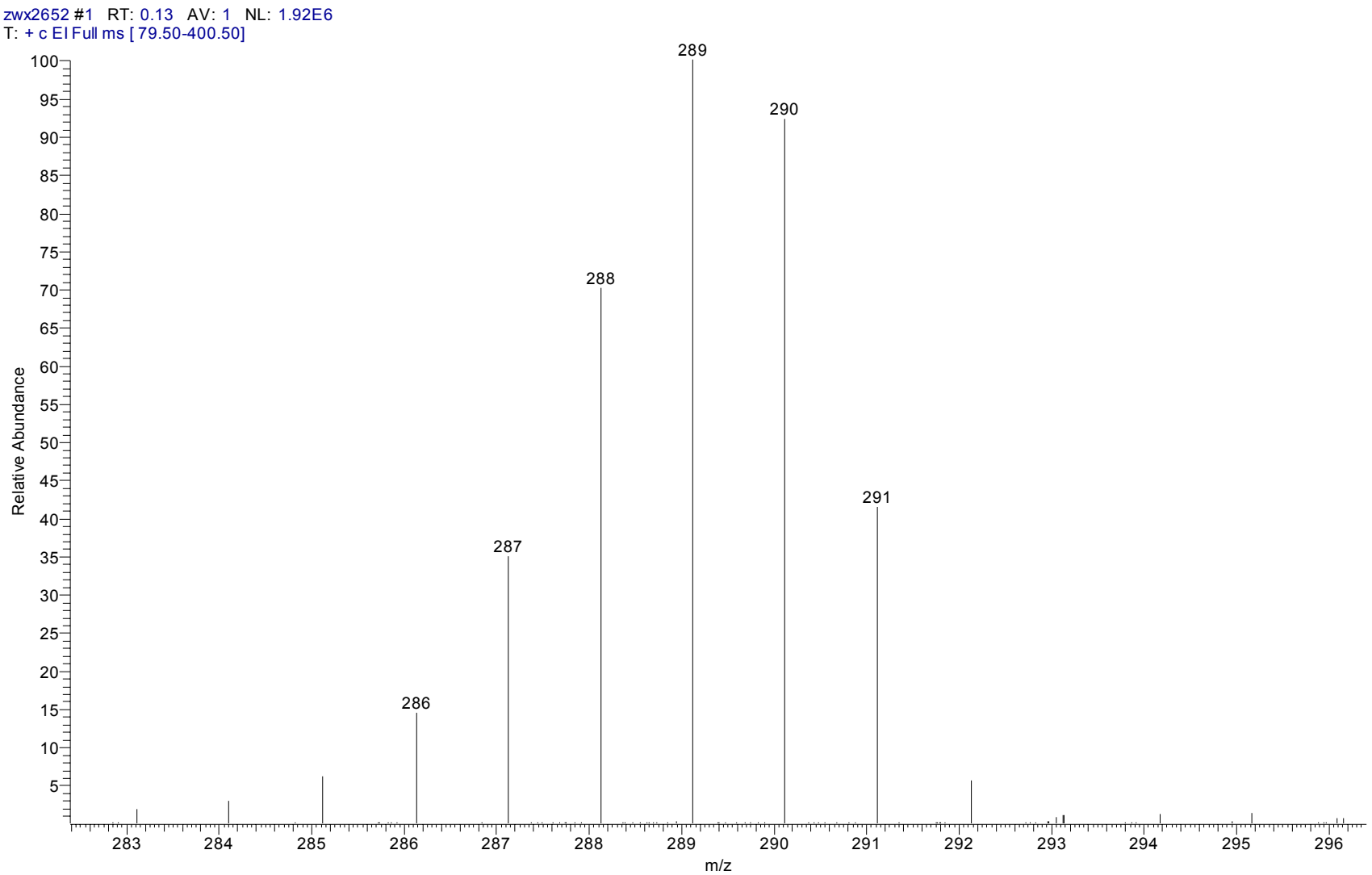
lhr-B-0580-cc-C6D6(C)

```
Current Data Parameters
NAME      lhr-B-0580-cc-C6D6(C)
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_              20170103
Time                  19.10 h
INSTRUM               spect
PROBHD   Z108618_0257 (
PULPROG                   zg
TD                     65536
SOLVENT                C6D6
NS                       58
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

F2 - Processing parameters
SI                     32768
SF             128.4097504 MHz
WDW                       EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

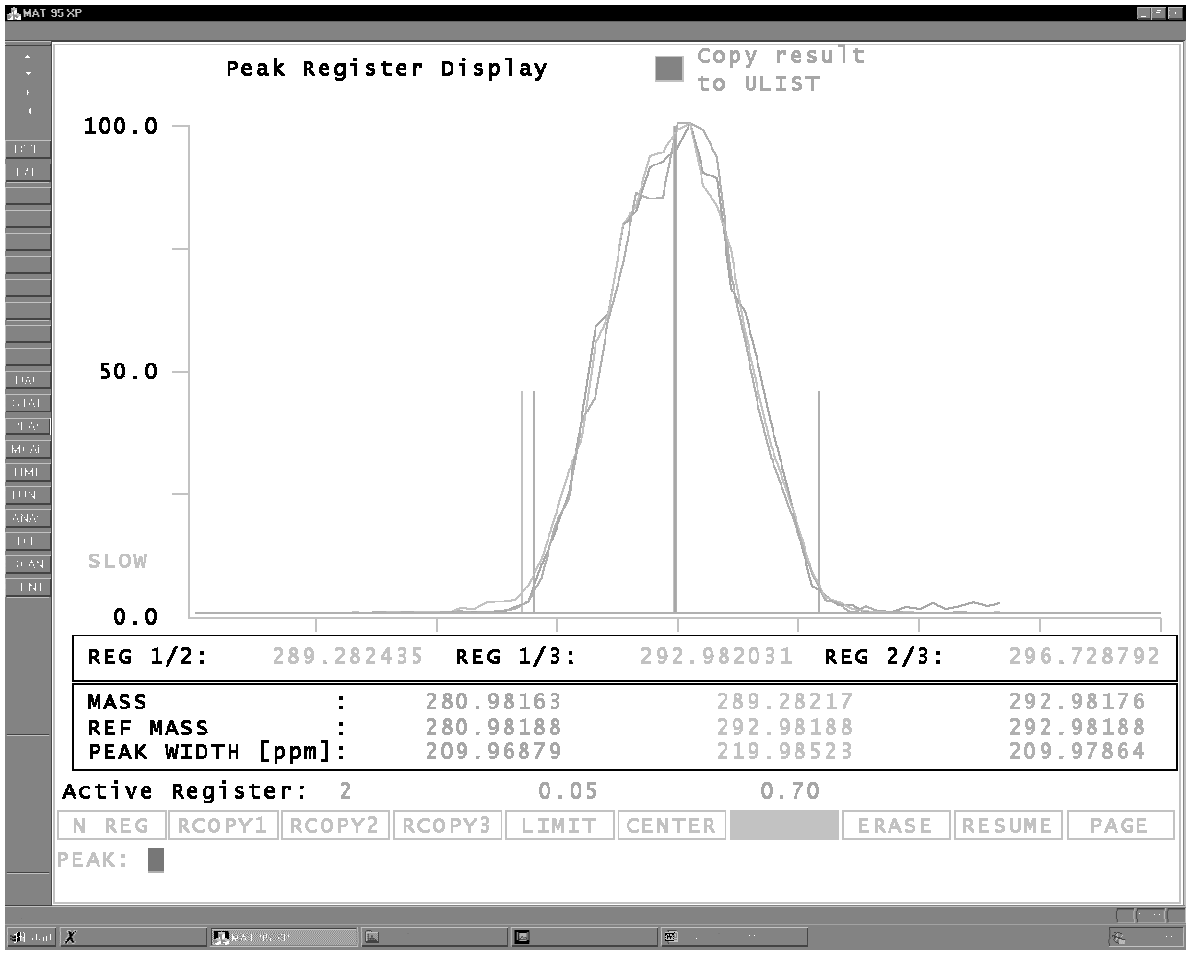
D:\MS_raw_data\zwx2652 03/14/17 03:09:18 PM lhr-0580-1
ei pos, 50eV, 0.7mA, unknown conc.

zwx2652 #1 RT: 0.13 AV: 1 NL: 1.92E6
T: + c EI Full ms [79.50-400.50]

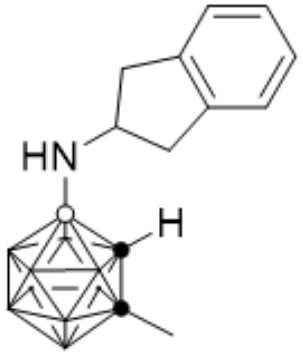



zwx2652 #1 RT: 0.13 AV: 1 NL: 1.92E6
T: + c EI Full ms [79.50-400.50]
Relative Abundance
m/z
289
290
288
291
287
286
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
283
284
285
286
287
288
289
290
291
292
293
294
295
296

Accurate Mass Measurement



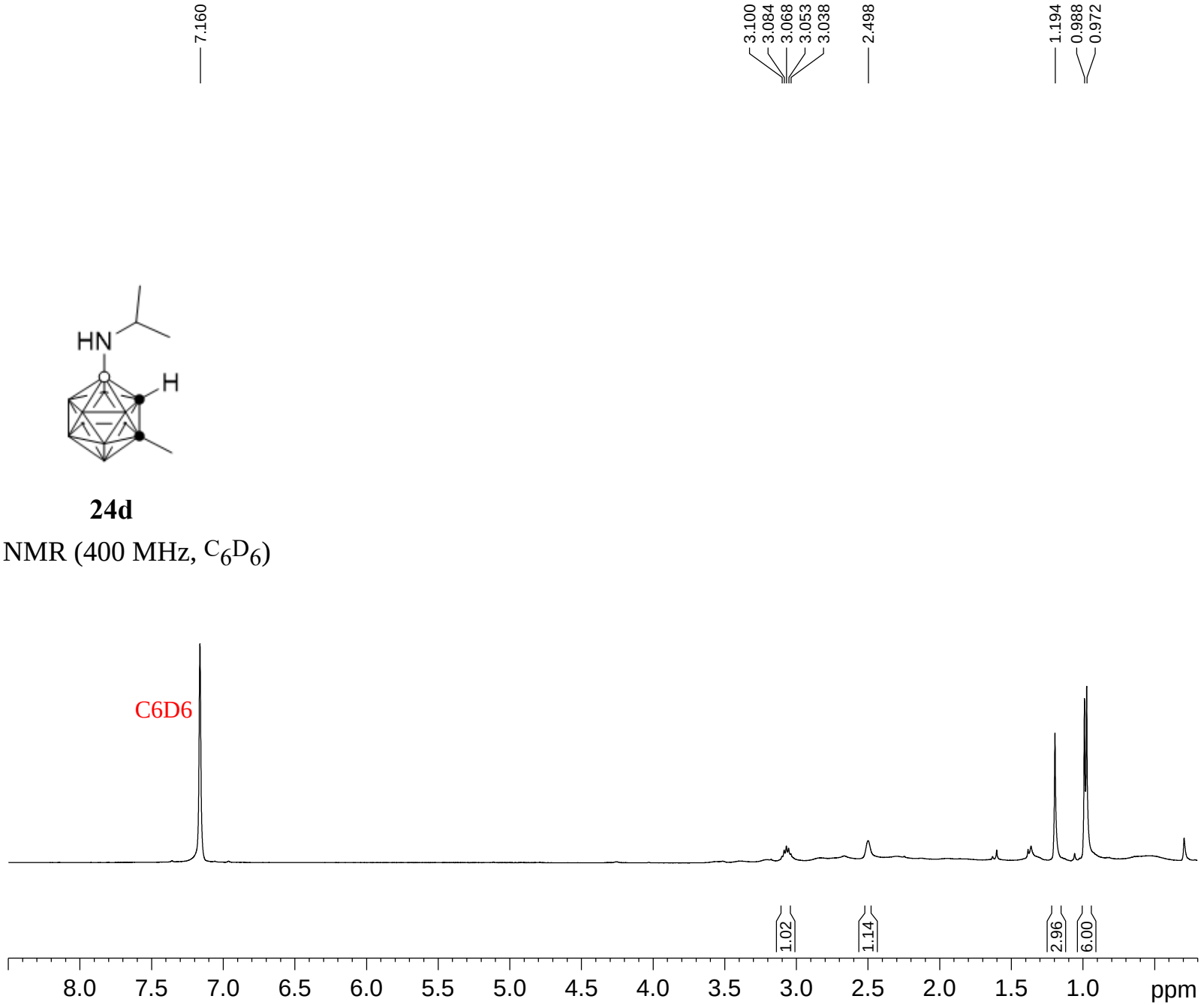
Molecular formula
$C_{12}H_{23}B_{10}N$
$[M]^+$ (theoretical)
= 289.2828



23d

24d

^{1}H NMR (400 MHz, C_6D_6)

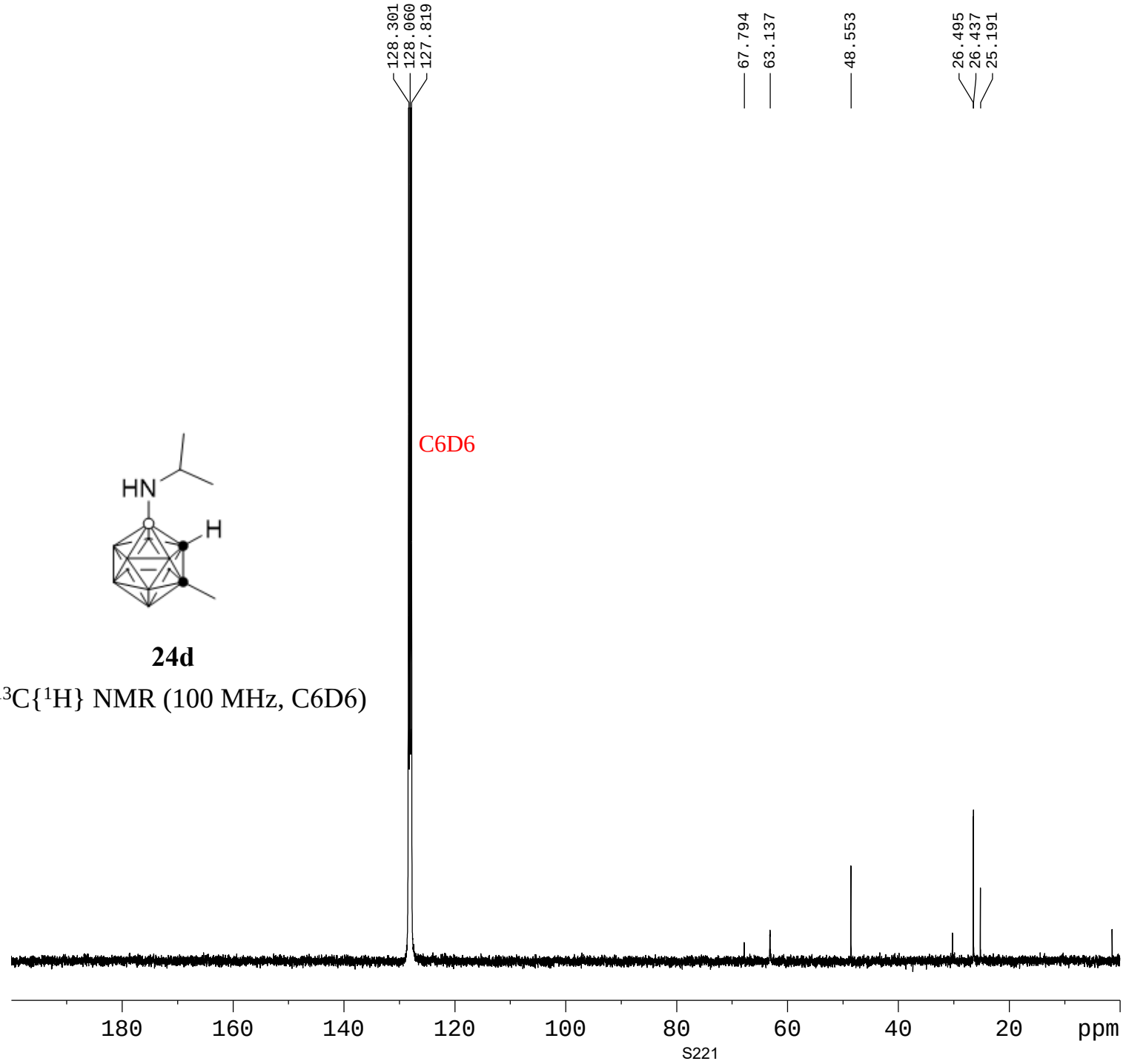


lhr-H-0596-cc-C6D6

Current Data Parameters
NAME lhr-H-0596-cc-C6D
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170119
Time 17.16 h
INSTRUM spect
PROBHD Z108618_0257 (
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 12
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 101
DW 62.400 usec
DE 6.50 usec
TE 294.8 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 400.2299981 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



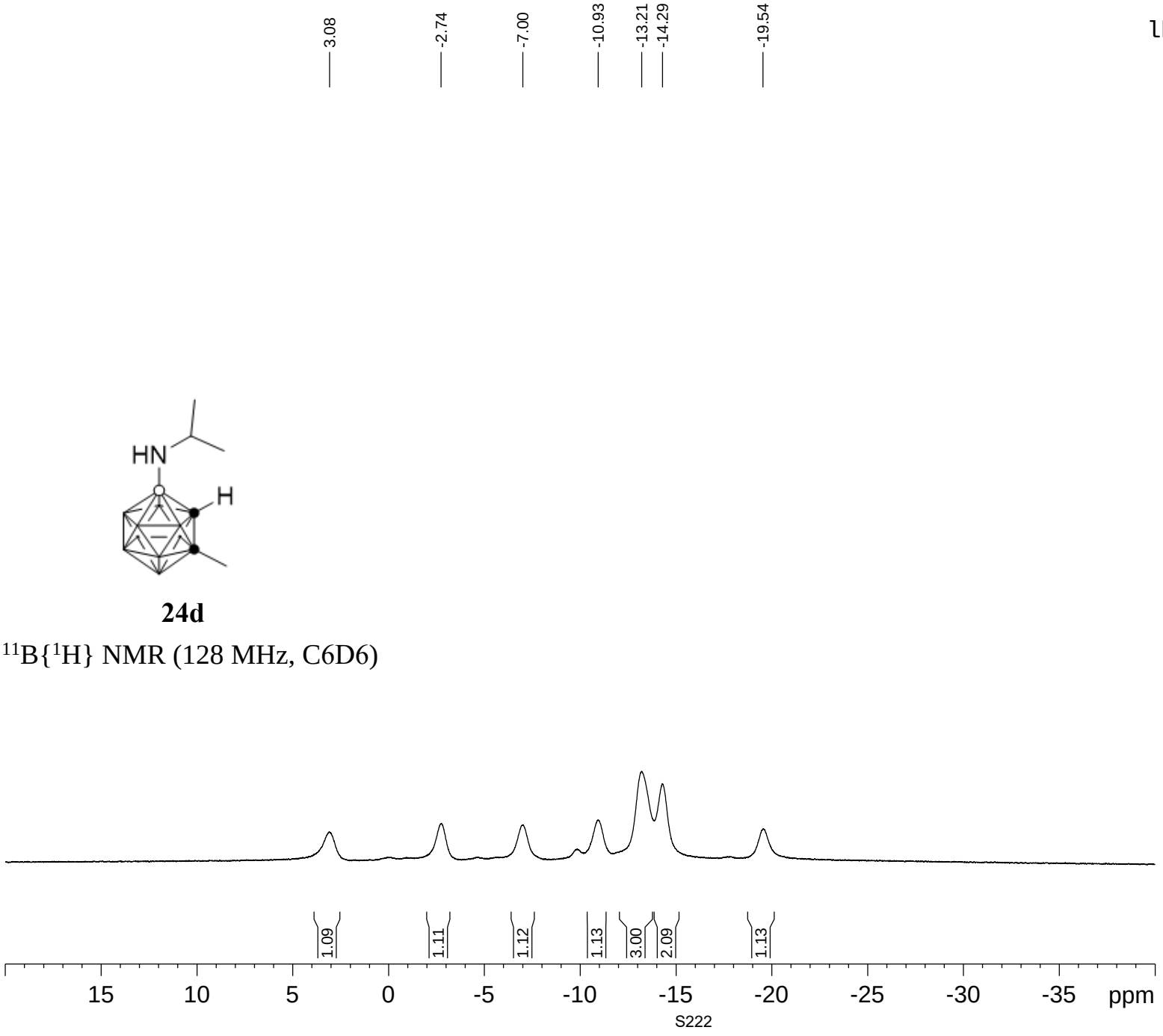
24d

$^{13}C\{^{1}H\}$ NMR (100 MHz, C6D6)

lhr-C-0596-cc-C6D6

Bruker Advance III 400

```
Current Data Parameters
NAME      lhr-C-0596-cc-C6D6
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20170119
Time               17.27 h
INSTRUM             spect
PROBHD    Z824601_0021 (
PULPROG           zgpg30
TD                 65536
SOLVENT              C6D6
NS                   1411
DS                      4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                    203
DW                20.800 usec
DE                 6.50 usec
TE                295.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                     1
SFO1         100.6228298 MHz
NUC1                 13C
P1                  9.50 usec
PLW1         41.25000000 W
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2          8.31000042 W
PLW12         0.23083000 W
PLW13         0.11611000 W

F2 - Processing parameters
SI                  32768
SF           100.6127314 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

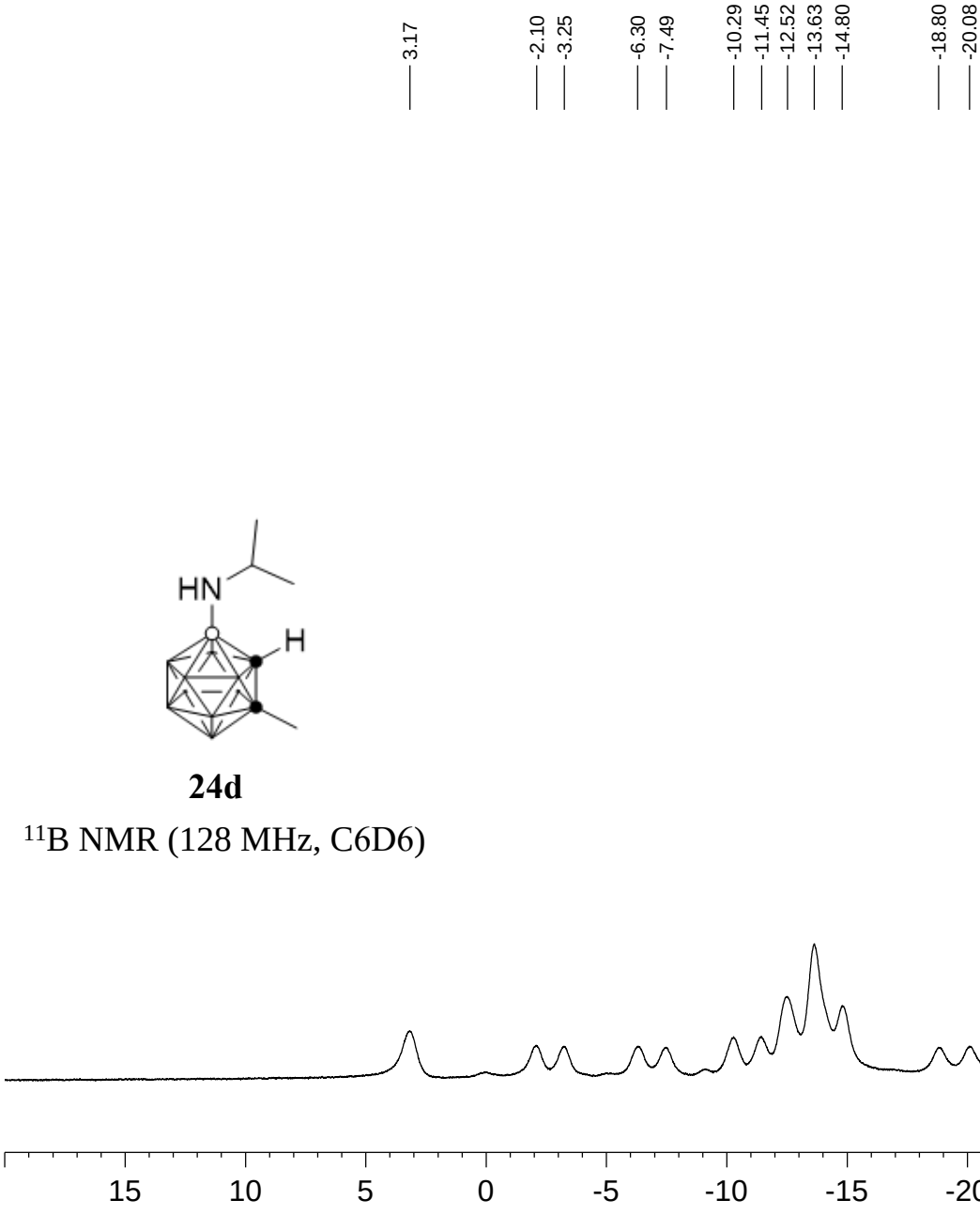


24d

$^{11}B\{^{1}H\}$ NMR (128 MHz, C6D6)

lhr-B-0596-cc-C6D6

```
Current Data Parameters
NAME      lhr-B-0596-cc-C6D6
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_            20170119
Time                17.10 h
INSTRUM              spect
PROBHD   Z108618_0257 (
PULPROG               zgdc
TD                   65536
SOLVENT               C6D6
NS                      16
DS                       4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631488 sec
RG                     287
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
```



lhr-B-0596-cc-C6D6(C)

```
Current Data Parameters
NAME      lhr-B-0596-cc-C6D6(C)
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_              20170119
Time                  17.12 h
INSTRUM               spect
PROBHD   Z108618_0257 (
PULPROG                   zg
TD                     65536
SOLVENT                C6D6
NS                       20
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                   294.9 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             128.4097504 MHz
WDW                       EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```


24d

^{11}B NMR (128 MHz, C6D6)

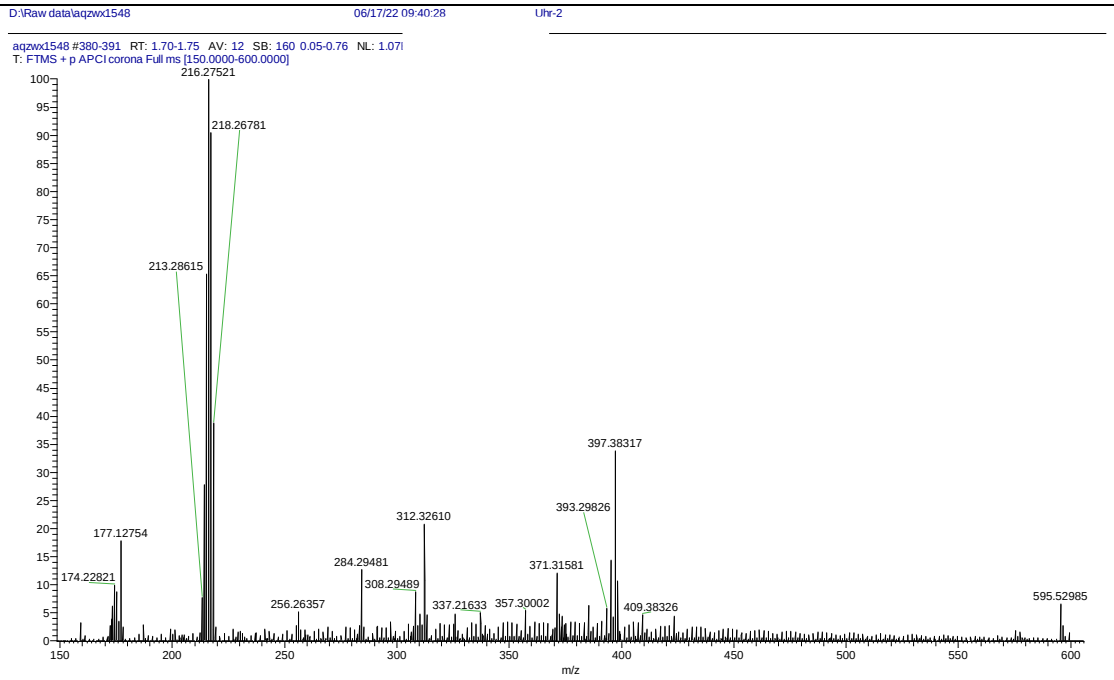
Thermo QEFMS Analysis Report

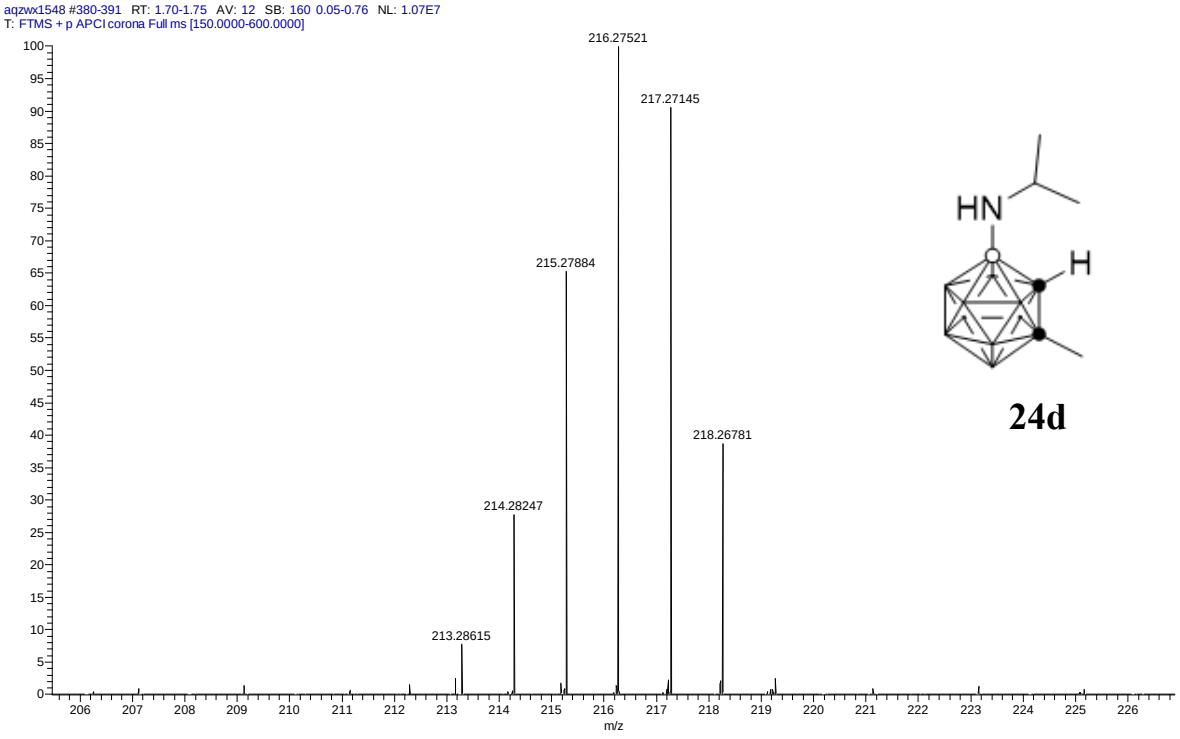
Analysis Info

Sample Name :	Uhr-2	Reference No.:	aqzwx1548
Instrument :	Q Exactive Focus Orbitrap		
Source :	APCI	Polarity :	Positive
Comment :	APCI, 4uA, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_6H_{21}B_{10}N$
Experimental Mass $[M+H]^+$:	216.27521
Theoretical Mass $[M+H]^+$:	216.27504
Error (ppm) :	0.8

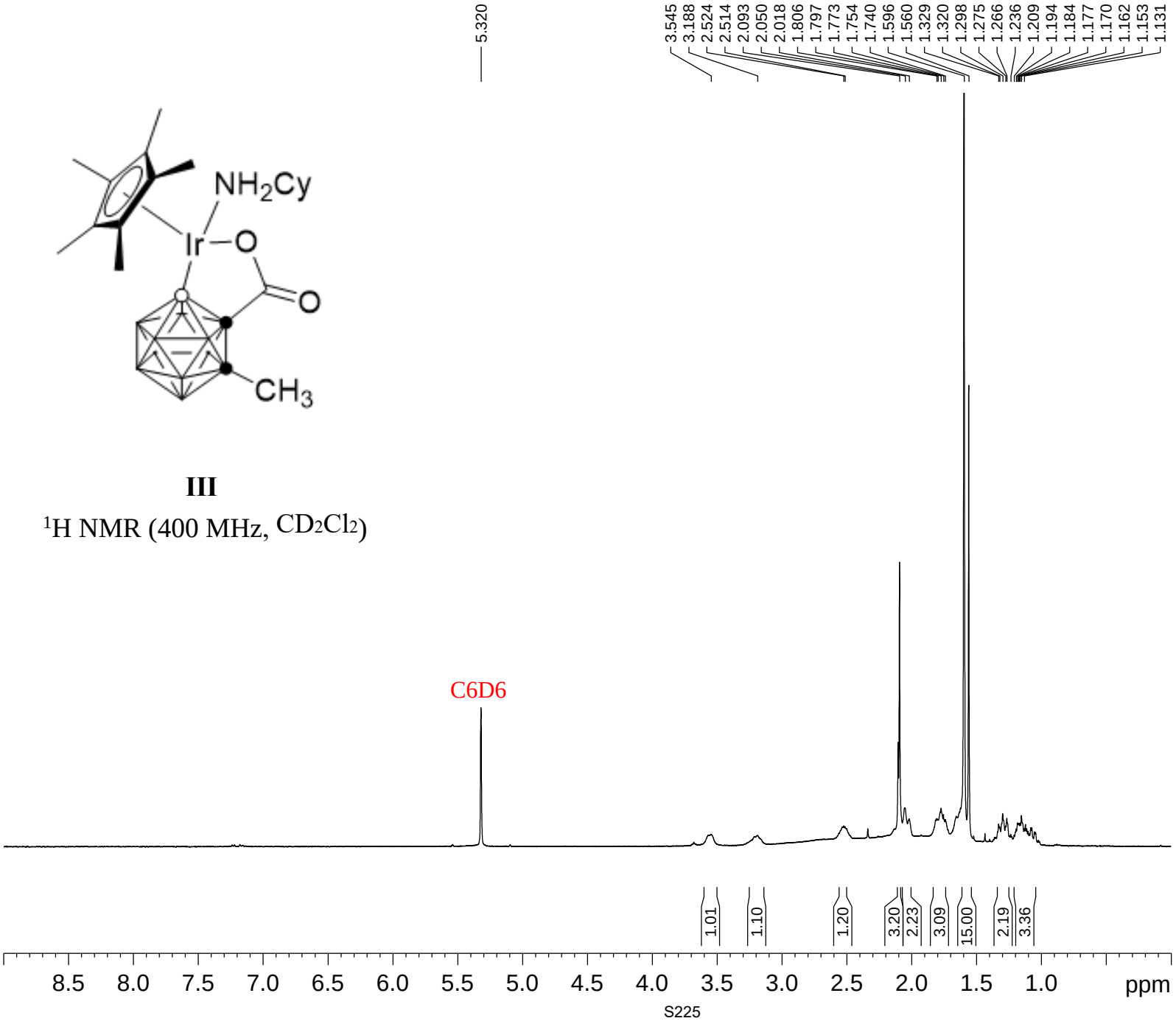




24d

III

^{1}H NMR (400 MHz, CD_2Cl_2)



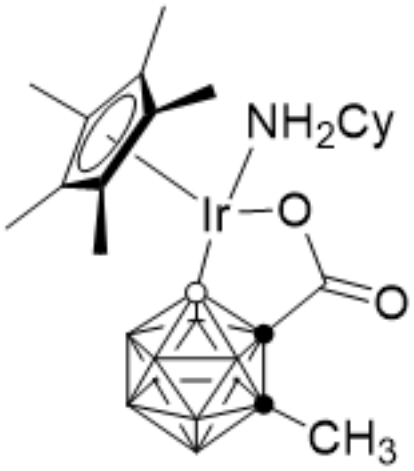
lhr-H-Irim-20180421

Current Data Parameters
NAME lhr-H-Irim-20180421-cd
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180421
Time 21.34 h
INSTRUM spect
PROBHD Z108618_0257 (
PULPROG zg30
TD 65536
SOLVENT CD2Cl2
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 128
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

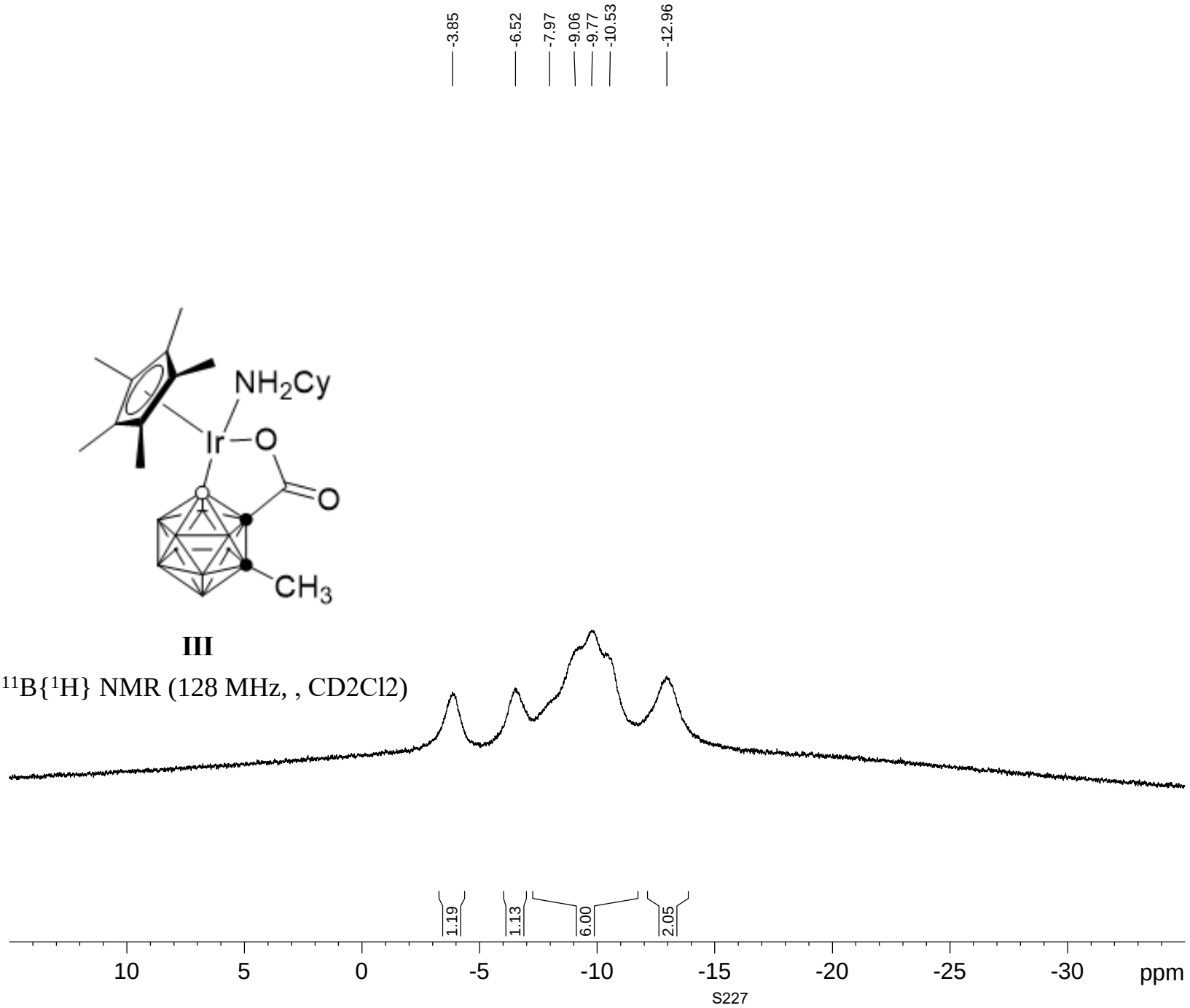
F2 - Processing parameters
SI 65536
SF 400.2300156 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

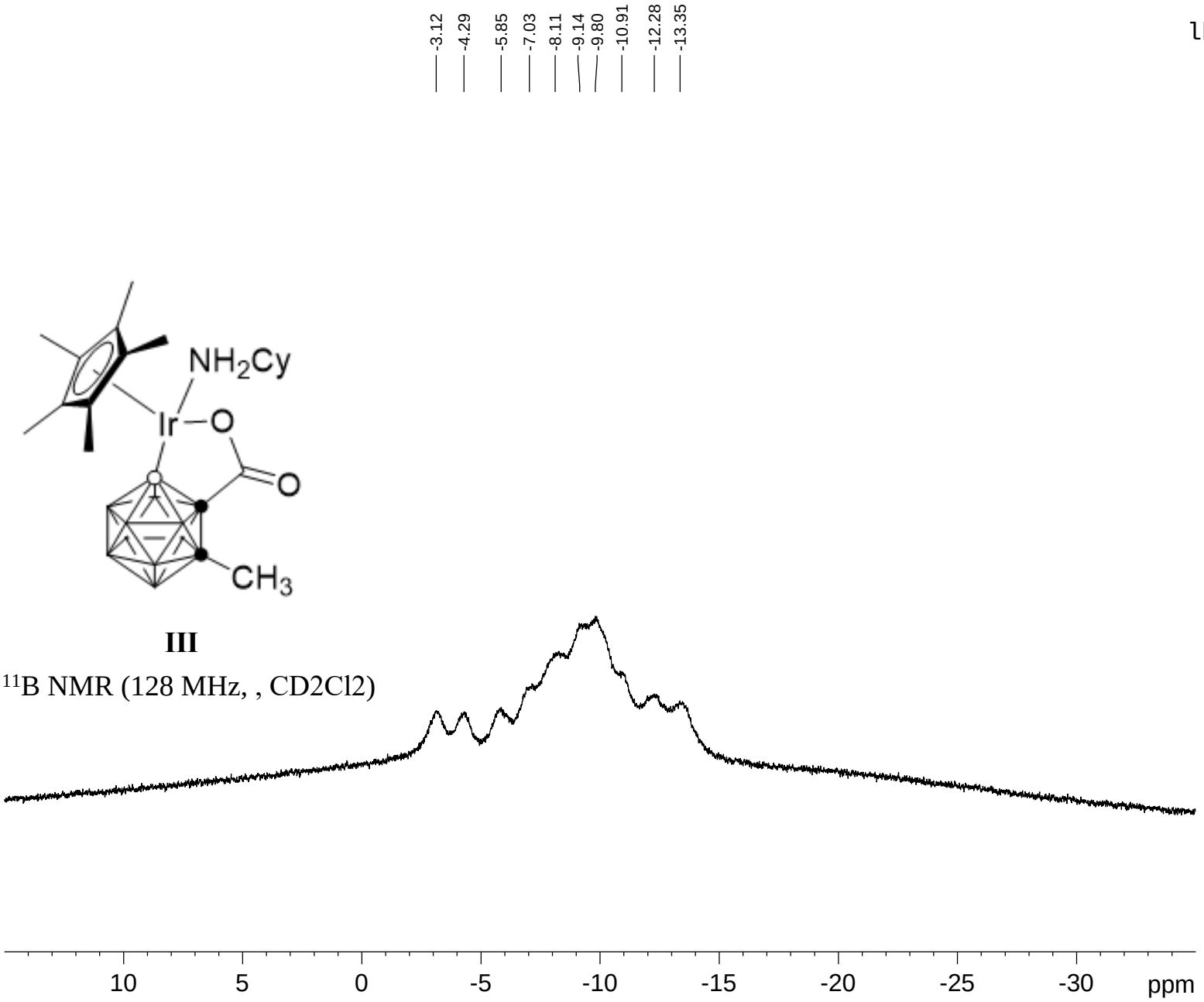
lhr-C-irim20180421-cd2cl2
lhr-C-irim20180421-cd2cl2



III

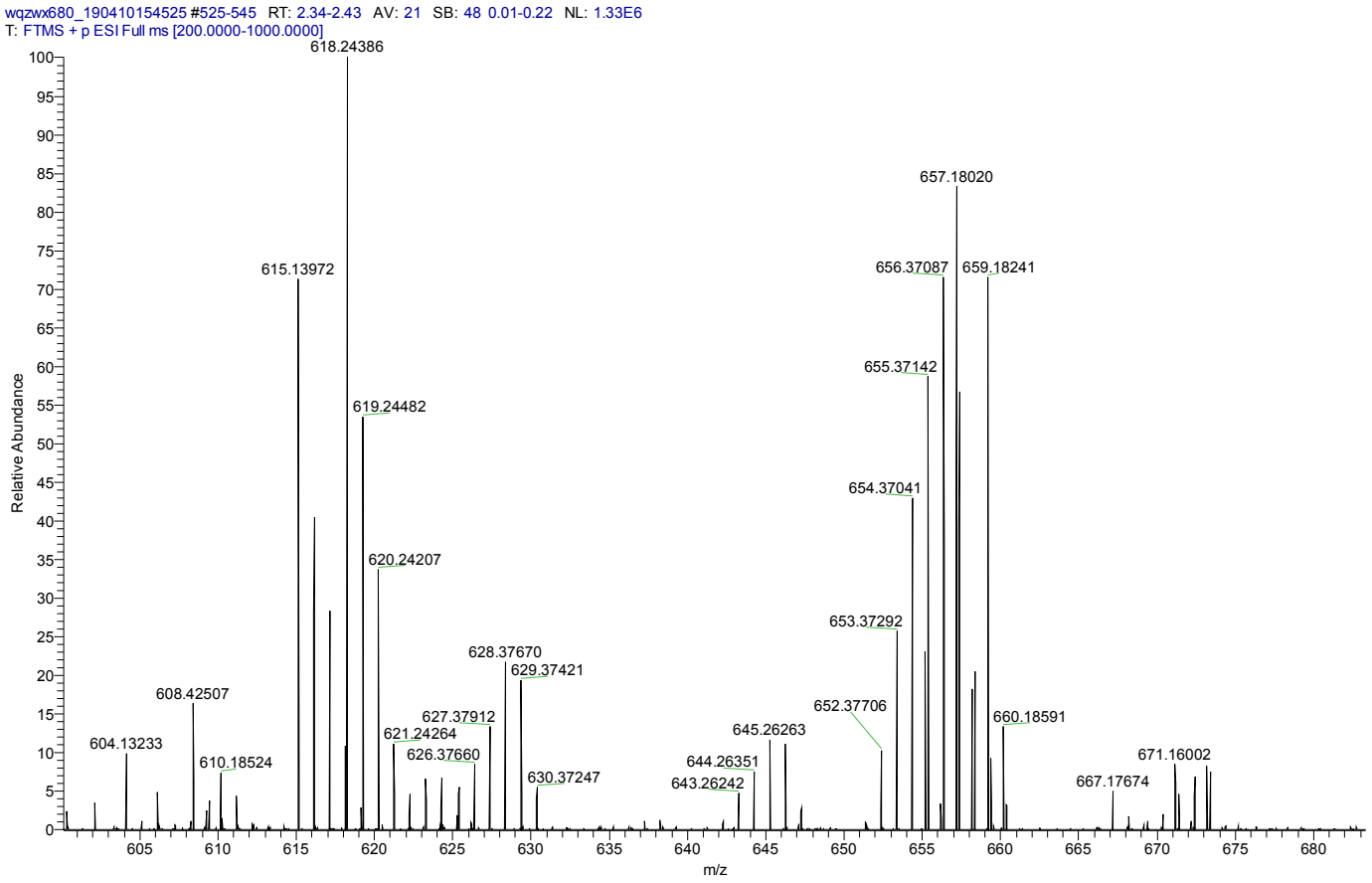
$^{13}C\{^{1}H\}$ NMR (100 MHz, CD2Cl2)

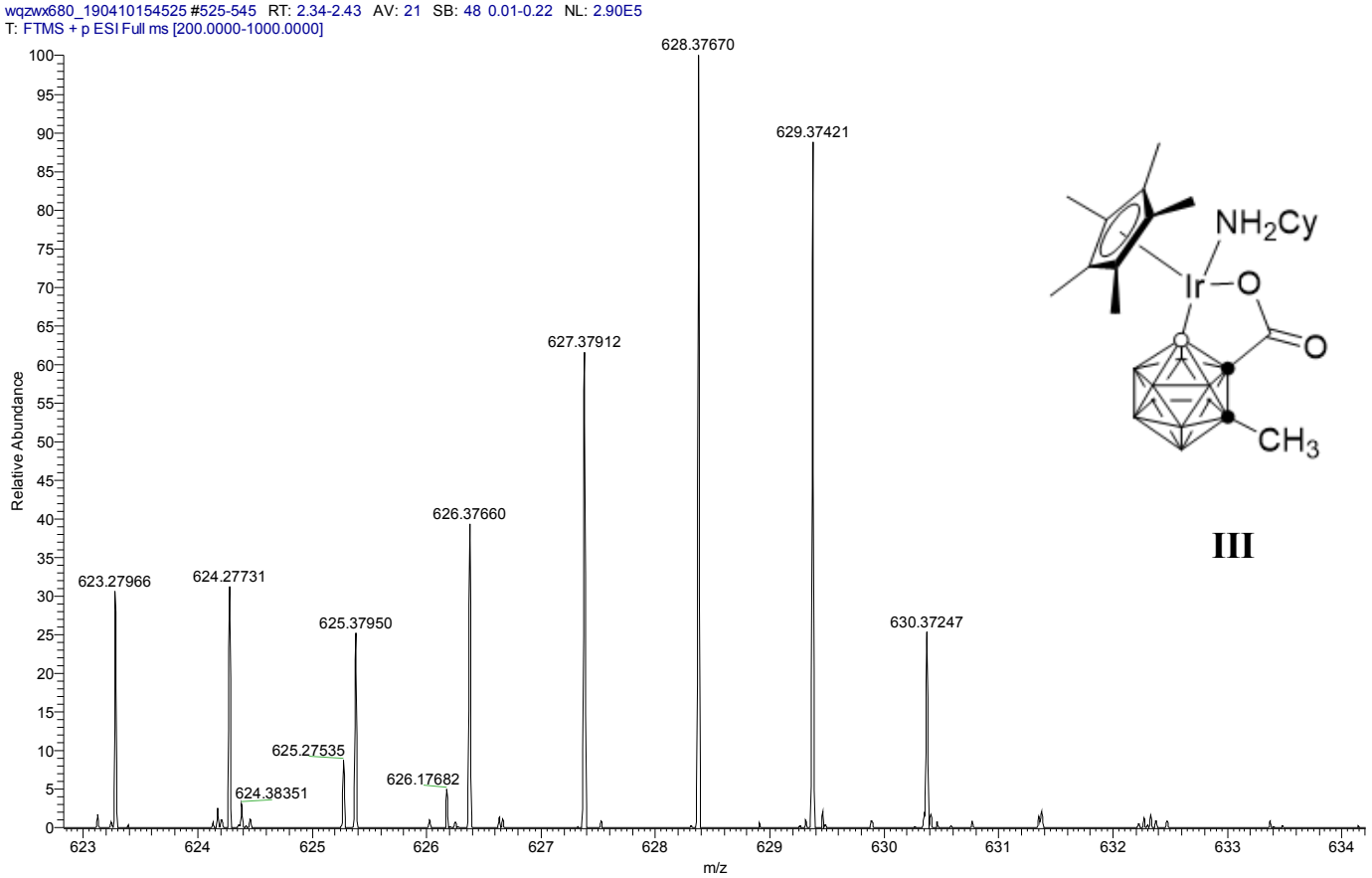

-3.85
-6.52
-7.97
-9.06
-9.77
-10.53
-12.96
lhr-B-Irim-20180421-cd2cl2
Current Data Parameters
NAME lhr-B-Irim-20180421-cd2cl2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180421
Time 21.39 h
INSTRUM spect
PROBHD Z108618_0257 (
PULPROG zgdc
TD 65536
SOLVENT CD2Cl2
NS 64
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 406
DW 20.800 usec
DE 6.50 usec
TE 295.7 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
NH2Cy
Ir
O
O
CH3
III
11B{1H} NMR (128 MHz, , CD2Cl2)
1.19
1.13
6.00
2.05
10
5
0
-5
-10
-15
-20
-25
-30
ppm



III

^{11}B NMR (128 MHz, , CD2Cl2)


wqzwx680_190410154525 #525-545 RT: 2.34-2.43 AV: 21 SB: 48 0.01-0.22 NL: 1.33E6
T: FTMS + p ESI Full ms [200.0000-1000.0000]
618.24386
657.18020
615.13972
656.37087
659.18241
655.37142
619.24482
654.37041
620.24207
653.37292
628.37670
629.37421
608.42507
652.37706
660.18591
627.37912
621.24264
645.26263
604.13233
626.37660
644.26351
671.16002
610.18524
630.37247
643.26242
667.17674
Relative Abundance
m/z


wqzwx680_190410154525 #525-545 RT: 2.34-2.43 AV: 21 SB: 48 0.01-0.22 NL: 2.90E5
T: FTMS + p ESI Full ms [200.0000-1000.0000]
628.37670
629.37421
627.37912
626.37660
623.27966
624.27731
625.37950
630.37247
625.27535
626.17682
624.38351
Relative Abundance
m/z

NH2Cy
Ir
O
O
CH3

III