Supplementary Information for

B–H Functionalization of the monocarba-*closo*-dodecaborate anion by rhodium and iridium catalysis

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I General Information

Chemicals

If not otherwise specified, reagents and organic solvents were commercially available and used without further purification. Chloroform-*d,* acetone-d_6 and DMSO-d_6 were purchased from Cambridge Isotope Laboratories and filtered through Al_2O_3 prior to use. $[CB_{11}H_{12}]$- salts (starting material) and $[IrCp^*(OAc)_2]$ (for preparation of the B-H activation intermediate) were prepared according to the literature [1-3]. Anhydrous solvents were prepared by passage through activated Al_2O_3 and stored over 3 Å molecular sieves.

Reaction Conditions

Glassware for air-sensitive reations was dried at 150 °C for at least 12 h and allowed to cool in a vacuum.

The Iridium B-H activation intermediate was prepared in a glovebox under a nitrogen atmosphere with O_2, H_2O <1 ppm.

Characterization

Thin-layer chromatography (TLC) was carried out using silica gel 60, F254 with a thickness of 0.25 mm. Column chromatography was performed on silica gel 60 (200-30 mesh).

NMR spectra were recorded on a Bruker AVANCE III 500 spectrometer (^{1}H NMR 500.13 MHz, ^{13}C NMR 125.77 MHz, ^{11}B NMR 160.46 MHz) or a Bruker AVANCE III 400 spectrometer (^{1}H NMR 400.13 MHz, ^{13}C NMR 100.62 MHz, ^{11}B NMR 128.38 MHz) at the temperature indicated. Data are reported as follows: Chemical shift in ppm, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublets, etc.), coupling constant *J* in Hz, integration, and (where applicable) interpretation. Signals were referenced against solvent peaks (^{1}H:

residual $CHCl_3$ = 7.26 ppm, residual $CHD_2C(O)CD_3$ = 2.05 ppm, residual CHD_2CN = 1.94 ppm, residual $CHD_2S(O)CD_3$ = 2.50 ppm, $^{13}C\{^1H\}$: $CDCl_3$ = 77.00 ppm $CD_3C(O)CD_3$ = 29.84 ppm, CD_3CN = 1.32 ppm, $CD_3S(O)CD_3$ = 39.52 ppm). ^{11}B and $^{11}B\{^1H\}$ NMR spectra were calibrated against external BF_3*Et_2O = 0 ppm (BF_3*Et_2O capillary in C_6D_6). ^{19}F NMR spectra were referenced against internal fluorobenzene = -113.15 ppm.

General notes:

a) In certain ^1H and $^1H\{^{11}B\}$ NMR spectra measured in acetone-d_6, double water peaks were observed. This is a result of different resonances from H_2O and HOD and has been described in the literature.[4]

b) Products carrying the pyrrolidine amide moiety showed broad ^1H and $^{13}C\{^1H\}$ pyrrolidine resonances attributed to slow rotation about the C(O)–N bond. To obtain sharper/fewer resonances, some spectra were measured at 80 °C in DMSO-d_6. Specifically, this was done for **1a**, **1b**, **3d**, **3e**, **3h**, **3i** and **3k** (pages NMR13/16, 17/20, 40, 41/44, 55/58, 62 and 70).

c) ^{11}B-^{11}B COSY NMR spectra were recorded at 160 MHz (500 MHz for ^1H) under ^1H decoupling. For compounds with a low degree of substitution and having no B–C/B–N bonds, $^1J_{B\text{-}B}$ correlation signals were observed, such as for **1a**–**c**. Products with B–C/B–N bonds exhibited weak or even non-observable correlation signals. This is specifically displayed in the ^{11}B-^{11}B COSY NMR spectra of **3a**, **3c**, **3f**, **3g**, **3h**, **3i** and **3j**; on the other hand, much more information about the connectivity could be obtained for products **3k**–**m** (Section **V**, pages NMR83–96).

A detailed study by Grimes addressed the phenomenon of varying strength of correlation signals,[5] and an explanatory summary is given in the following. The detection of cross peaks requires that several criteria be fulfilled: (a) Sufficient electron density must exist directly between the respective boron atoms; (b) the relaxation times T_1 and T_2 relaxation times must be long enough and (c) the individual ^{11}B resonances in the 1D spectrum are fully or at partially resolved. All of these conditions influence scalar coupling and heavily depend on the number, nature and

position of the cage substituents. They cannot be changed by the measurement parameters, except for (c) where recording data at a higher field is beneficial.

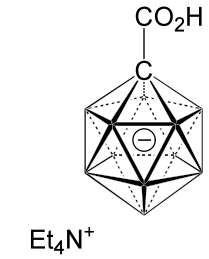
Low-resolution ESI-MS data were recorded on Advion Expression CMS instrument. High-resolution MS data were recorded using IT-TOF detection (Shimadzu, Japan) equipped with an electrospray ionization source (ESI). Accurate mass determination was corrected by calibration using sodium trifluoroacetate clusters as a reference.

Elemental analysis was measured in the Analytical Facilities of the Department of Chemistry of Zhejiang University.

Single-crystal X-ray diffraction studies were performed on an Oxford Diffraction Gemini A Ultra diffractometer equipped with an 135mm Atlas CCD detector and using Mo K-α radiation.

II Experimental Section

II. a) Synthesis of carborane acids and amides



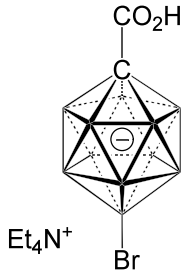
Carborane carboxylic acid $[Et_4N][1\text{-}COOH\text{-}CB_{11}H_{11}]$ was synthesized by a modified literature procedure.[6] A dry 50 mL round bottom flask equipped with magnetic stir bar was charged with $[Me_3NH][CB_{11}H_{12}]$ (250 mg, 1.23 mmol) and capped with a rubber septum. Anhydrous THF (15 mL) was then added to the flask and the resulting solution cooled to 0 °C in an ice bath. A solution of 1.6 M n-BuLi in hexane (2.3 mL, 3.69 mmol) was slowly added to the reaction flask. After 1 h of stirring at 0 °C, a slightly turbid, yellowish solution was obtained. Dry CO_2 gas was then bubbled through the mixture at 0 °C (2-3 bubbles/s) for 5 h. Water (2 mL) was slowly added and the solution was concentrated on a rotary evaporator, the residue was dissolved in water, washed with diethyl ether (under these basic conditions the dianionic product was in the water layer), the aqueous phase was acidified with 1 M HCl (pH=2) and extracted with diethyl ether (3 x 20 mL). The combined organic extracts were evaporated under reduced pressure, the crude product was dissolved in water (10 mL) and filtered through a glass frit. $[Et_4N]^+ Br^-$ (400 mg, 1.9 mmol) was added to the filtrate, and the resulting white precipitate was collected in a glass frit and dried in a vacuum to give $[Et_4N][1\text{-}COOH\text{-}CB_{11}H_{11}]$ as a colorless solid (320 mg, 82% yield).

$^1H\{^{11}B\}$ NMR (500 MHz, acetone-d_6, 23 °C): δ 3.46 (q, J = 7.3 Hz, 8H, CH_2 of cation), 2.30-1.10 (broad m, 11H, BH), 1.38 (tt, J = 7.3Hz, 1.9 Hz, 12H, CH_3 of cation).

^{11}B NMR (160 MHz, acetone-d_6, 23 °C): δ -6.49 (d, J = 133.8 Hz, 1B), -13.25 (d, J = 135.2 Hz, 5B), -14.10 (d, J = 148.2 Hz, 5B).

$^{11}B\{^1H\}$ NMR (160 MHz, acetone-d_6, 23 °C): δ -6.49(1B), -13.25(5B), -14.10 (5B).

$^{13}C\{^1H\}$ NMR (125 MHz, acetone-d_6, 23 °C): δ 168.17 (CO), 68.52 (cage C), 52.96 (CH_2 of cation), 7.62 (CH_3 of cation).



To a stirred solution of $[Et_4N][1\text{-}COOH\text{-}CB_{11}H_{11}]$ (300 mg, 0.95 mmol) in CH_3CN (10 mL) was added *N*-bromosuccinimide (196 mg, 1.1 mmol). The reaction mixture was then stirred for 20 h at 25 °C. Na_2SO_3 (13 mg, 0.1 mmol) was added to the mixture, followed by 1M HCl (20 ml). Acetonitrile was removed under reduced pressure. The aqueous solution was extracted with Et_2O (3 x 20 mL). Water (15 ml) was added to the combined extracts, and Et_2O was removed under reduced pressure. The aqueous solution was filtered, and $[Et_4N]^+ Br^-$ (230 mg, 1.09 mmol) was added to the filtrate. The precipitate that formed was collected by vacuum filtration, washed with water and dried in a vacuum to give $[Et_4N][1\text{-}COOH\text{-}12\text{-}Br\text{-}CB_{11}H_{10}]$ as a colorless solid (300 mg, 80% yield).

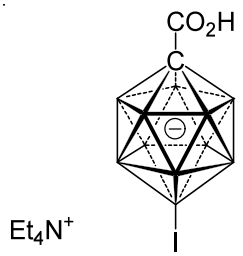
$^1H\{^{11}B\}$ NMR (500 MHz, acetone-d_6, 23 °C): δ 3.46 (q, J = 7.3 Hz, 8H, CH_2 of cation), 2.10-1.10 (broad m, 10H, BH), 1.38 (tt, J = 7.3Hz, 1.9 Hz, 12H, CH_3 of cation).

^{11}B NMR (160 MHz, acetone-d_6, 23 °C): δ -1.58 (s, 1B), -12.45 (d, J = 141.7 Hz, 5B), -14.74 (d, J = 156.5 Hz, 5B).

$^{11}B\{^1H\}$ NMR (160 MHz, acetone-d_6, 23 °C): δ -1.58 (1B), -12.45 (5B), -14.74 (5B).

$^{13}C\{^1H\}$ NMR (125 MHz, acetone-d_6, 23 °C): δ 168.42 (CO), 62.68 (cage C), 52.96 (CH_2 of cation), 7.65 (CH_3 of cation).

HRMS (ESI): *m/z* Calcd. for $[C_2H_{11}B_{11}BrO_2]^-$, 266.1034; found, 266.1036.



To a stirred solution of $[Et_4N][1\text{-}COOH\text{-}CB_{11}H_{11}]$ (400 mg, 1.26 mmol) in CH_3OH (10 mL) was added *N*-iodosuccinimide (312 mg, 1.39 mmol). The reaction mixture was then stirred for 5 h at 25 °C. Na_2SO_3 (16 mg, 0.13 mmol) was added to the mixture, followed by 1M HCl (25 ml). Methanol was removed under reduced pressure. The aqueous solution was extracted with Et_2O (3 x 25 mL). Water (20 ml) was added to the combined extracts, and Et_2O was removed under reduced pressure. The aqueous solution was filtered, and $[Et_4N]^+ Br^-$ (290 mg, 1.38 mmol) was added to the filtrate. The precipitate that formed was collected by vacuum filtration, washed with water and dried in a vacuum to give $[Et_4N][1\text{-}COOH\text{-}12\text{-}I\text{-}CB_{11}H_{10}]$ as a colorless solid (455 mg, 81% yield).

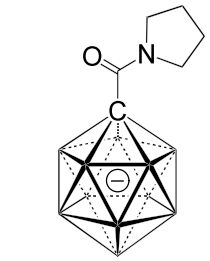
$^1H\{^{11}B\}$ NMR (500 MHz, acetonitrile-d_3, 23 °C): δ 3.15 (q, J = 7.3 Hz, 8H, CH_2 of cation), 2.30-1.30 (broad m, 10H, BH), 1.21 (tt, J = 7.3 Hz, 1.8 Hz, 12H, CH_3 of cation).

^{11}B NMR (160 MHz, acetonitrile-d_3, 23 °C): δ -11.98 (d, J = 143 Hz, 5B), -13.82 (d, J = 157 Hz, 5B), -17.19 (s, 1B).

$^{11}B\{^1H\}$ NMR (160 MHz, acetonitrile-d_3, 23 °C): δ -11.98 (5B), -13.82 (5B). -17.19 (s, 1B).

$^{13}C\{^1H\}$ NMR (125 MHz, acetonitrile-d_3, 23 °C): δ 169.06 (CO), 69.50 (cage C), 53.05 (CH_2 of cation), 7.68 (CH_3 of cation).

HRMS (ESI): *m/z* Calcd. for $[C_2H_{11}B_{11}IO_2]^-$, 313.0900; found, 313.0917.



1a: To a stirred suspension of carboxylic acid $[Et_4N][1\text{-}COOH\text{-}CB_{11}H_{11}]$ (200 mg, 0.63 mmol) in dry CH_2Cl_2 (5 mL) were added dimethylformamide (ca. 5 drops) and oxalyl chloride (0.056 mL, 0.65 mmol). The reaction mixture was allowed to stir for 30 min at room temperature. The volatiles were removed carefully under vacuum with nitrogen-cooled solvent traps. The acid chloride (dissolved in a minimum amount of dry CH_2Cl_2) was added dropwise to a solution of pyrrolidine (0.13 mL, 1.6 mmol) in CH_2Cl_2 (10 mL). The resulting mixture was stirred for 2 h. All volatile components were then removed completely in a vacuum, and the residue was purified by column chromatography on silica gel with CH_2Cl_2/CH_3CN (4:1 *v/v*) as the eluent to afford a white solid (205 mg, 88% yield).

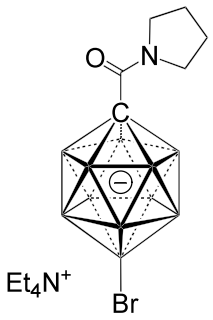
$^1H\{^{11}B\}$ NMR (500 MHz, DMSO-d_6, 80 °C): δ 3.54-3.45 (broad m, 4H, NCH_2CH_2), 3.24 (q, J = 7.3 Hz, 8H, CH_2 of cation), 2.20-1.30 (overlapping m, 15H, NCH_2CH_2 and BH), 1.22 (tt, J = 7.3 Hz, 1.9Hz, 12H, CH_3 of cation).

^{11}BNMR (160 MHz, DMSO-d_6, 80 °C): δ -5.59 (d, J = 133.8 Hz, 1B, B-12), -13.33 (overlapping d, J = 133.6 Hz, 10B).

$^{11}B\{^1H\}$ NMR (160 MHz, DMSO-d_6, 80 °C): δ -5.59 (1B, B-12), -13.33 (overlapping signals, 10B).

$^{13}C\{^1H\}$ NMR (125 MHz, DMSO-d_6, 80°C): δ 161.98 (CO), 71.33 (cage C), 51.57 (CH_2 of cation), 48.24 (NCH_2CH_2), 24.24 (NCH_2CH_2), 6.68 (CH_3 of cation).

HRMS (ESI): *m/z* Calcd. for $[C_6H_{19}B_{11}NO]^-$, 240.2572; found, 240.2582.



1b: Following a similar procedure as for the preparation of **1a,** using carboxylic acid $[Et_4N][1\text{-}COOH\text{-}12\text{-}Br\text{-}CB_{11}H_{10}]$ (200 mg, 0.51 mmol), **1b** (175 mg, 76%) was obtained as a white solid.

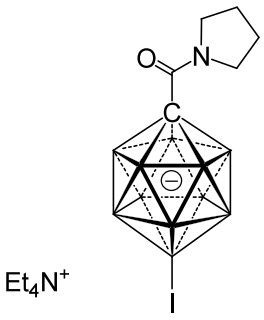
$^1H\{^{11}B\}$ NMR (500 MHz, DMSO-d_6, 80 °C): δ 3.53-3.41(m, 4H, NCH_2CH_2), 3.24(q, J = 7.3Hz, 8H, CH_2 of cation), 2.30-1.10 (overlapping m, 14H, NCH_2CH_2 and BH), 1.21 (tt, J = 7.3 Hz, 1.9 Hz, 12H, CH_3 of cation).

^{11}BNMR (160 MHz, MHz, acetone-d_6, 19 °C): δ -0.83 (s, 1B), -12.55 (d, J = 141.6 Hz, 5B), -14.38 (d, J = 154.7 Hz, 5B).

$^{11}B\{^1H\}$ NMR (160 MHz, acetone-d_6, 19 °C): δ -0.83 (1B), -12.55 (5B), -14.38 (5B).

$^{13}C\{^1H\}$ NMR (125 MHz, DMSO-d_6, 80°C): δ 161.93 (CO), 65.12 (cage C), 51.57 (CH_2 of cation), 48.36 (NCH_2CH_2), 24.22 (NCH_2CH_2), 6.69 (CH_3 of cation).

HRMS (ESI): *m/z* Calcd. for $[C_6H_{18}B_{11}BrNO]^-$, 319.1666; found, 319.1665.



1c: Following a similar procedure as for the preparation of **1a,** using carboxylic acid $[Et_4N][1\text{-}COOH\text{-}12\text{-}I\text{-}CB_{11}H_{10}]$ (300 mg, 0.68 mmol), **1c** (294 mg, 87%) was obtained as a white solid.

$^1H\{^{11}B\}$ NMR (500 MHz, acetonitrile-d_3, 23 °C): δ 3.85-3.53 (m, 2H, NCH_2CH_2), 3.46-3.20 (m, 2H, NCH_2CH_2), 3.17 (q, J = 7.3 Hz, 8H, CH_2 of cation), 2.10-1.50

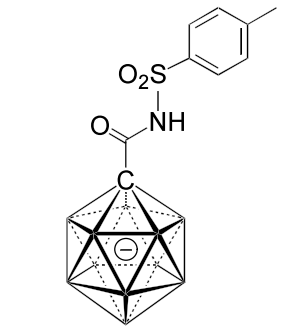
(overlapping m, 14H, NCH_2CH_2 and BH), 1.21 (tt, J = 7.3 Hz, 1.9 Hz, 12H, CH_3 of cation).

^{11}B NMR (160 MHz, acetonitrile-d_3, 23 °C): δ -11.82 (d, J = 144 Hz, 5B), -13.43 (d, J = 158 Hz, 5B), -15.96 (s, 1B).

$^{11}B\{^1H\}$ NMR (160 MHz, acetonitrile-d_3, 23 °C): δ -11.82 (5B), -13.43 (5B). -15.97 (s, 1B).

$^{13}C\{^1H\}$ NMR (125 MHz, acetonitrile-d_3, 23 °C): δ 163.86 (CO), 70.47 (cage C), 53.05 (CH_2 of cation), 50.14 (NCH_2CH_2, overlapping signals of the two carbon atoms), 27.91 (NCH_2CH_2), 23.82 (NCH_2CH_2), 7.66 (CH_3 of cation).

HRMS (ESI): *m/z* Calcd. for $[C_6H_{18}B_{11}INO]^-$, 366.1529; found, 366.1555



1d: This product was synthesized according to the literature procedure.[7] To a stirred suspension of salt $[Et_4N][1\text{-}COOH\text{-}CB_{11}H_{11}]$ (300 mg, 0.95 mmol) in dry CH_2Cl_2 (7 mL) were added dimethylformamide (ca. 5 drops) and oxalyl chloride (0.084 mL, 0.98 mmol). The reaction mixture was allowed to stir for 30 min at room temperature. All volatile components were then removed in *vacuo* and the residue was dissolved in dry THF (12 mL), *p*-toluene sulfonamide (163 mg, 0.95 mmol), triethylamine (0.277 mL, 2 mmol), and DMAP (1.5 mg, 0.012 mmol) were added and the resulting mixture was stirred at room temperature for 24 h. After evaporation of the solvent under reduced pressure, the crude product was purified by column chromatography over silica gel with CH_2Cl_2/CH_3CN (5:1 *v/v*) as the eluent to afford a white solid (322 mg, 72% yield).

$^1H\{^{11}B\}$ NMR (500 MHz, DMSO-d_6, 19 °C): δ 7.73 (d, J = 8.5 Hz, 2H, ArH), 7.40 (d,

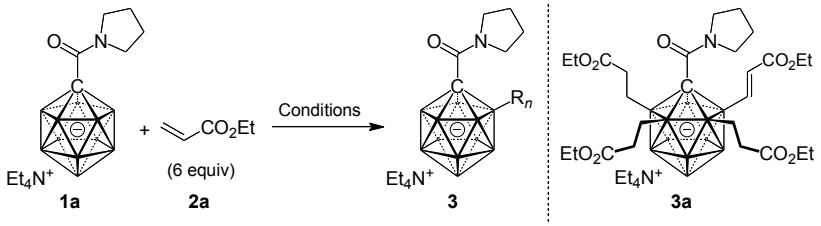
J = 8.5 Hz, 2H, ArH), 3.19 (q, *J* = 7.3 Hz, 8H, CH_2 of cation), 2.39 (s, 3H, $ArCH_3$), 2.20-1.20 (overlapping m, 11H, BH), 1.15 (tt, *J* = 7.3 Hz, 1.9 Hz, 12H, CH_3 of cation).

^{11}B NMR (160 MHz, acetone-d_6, 19 °C): δ -5.99 (d, *J* = 135 Hz, 1B), -12.85 (d, *J* = 140 Hz, 5B), -14.55 (d, *J* = 155 Hz , 5B).

$^{11}B\{^1H\}$NMR (160 MHz, acetone-d_6, 19 °C): δ -5.99 (1B), -12.85 (5B), -14.55 (5B).

$^{13}C\{^1H\}$ NMR (125 MHz, DMSO-d_6, 19 °C): δ 163.64 (CO), 144.19, 136.11, 129.44, 127.58, 69.35 (cage C), 51.38 (CH_2 of cation), 21.09 ($ArCH_3$), 7.08 (CH_3 of cation).

II. b) Optimization of reaction conditions for the reaction 1a → 3a



Scheme S1. Reaction of **1a** with ethyl acrylate to give substituted product **3**.

The model reaction between carborane amide **1a** and an excess of ethyl acrylate (**2a**) to give functionalized product(s) **3** with potential multiple substitution was studied (Scheme S1). The experiments were conducted under air in sealed vials, and the reaction mixtures were analyzed by ESI-MS and NMR spectroscopy (^{11}B, ^{11}B{^{1}H}). Specific conditions are given in Table S1.

Using 10 mol% $[RhCp^*Cl_2]_2$ in the polar aprotic solvent dimethylacetamide at 100 °C for 24 h resulted in no detectable conversion to substituted products (entry 1). 40 mol% $AgSbF_6$ as a chloride scavenger increased the catalyst reactivity substantially, leading to a mixture of tri- and tetra-substituted products (entry 2). In dimethylacetamide, the observed mass-spectrometric isotope patterns indicated that the first two substitutions afford C–C single bonds (reductive coupling), followed by introduction of an alkenyl ester (oxidative coupling), while in the last substitution the third C–C single bond is formed (reductive coupling). Addition of 1 equivalent of $Cu(OAc)_2 \cdot H_2O$ also provided tri- and tetra-substitution (entry 3). The combination of Ag(I) and Cu(II) was then investigated at different temperatures and found to give clean tetra-substituted **3a** at 100 °C or 120 °C (entries 4–6). The same result was obtained using 8 equiv of Cu(II), but reduced conversion was observed by adding KOAc instead of Cu(II) (entries 7 and 8). Carrying out the experiment under strictly oxygen-free conditions also afforded tetra-substituted **3a** (entry 9). While acetonitrile as the solvent provided a mixture, *N*-methylpyrrolidinone was comparable to dimethylacetamide (entries 10 and 11). $[IrCp^*Cl_2]_2$ as the catalyst also exhibited high activity, interestingly giving mainly di-substituted product in combination with Ag(I)

and Cu(II) (entries 12 and 13). In contrast, $[Ru(cymene)Cl_2]_2$ showed no catalytic activity (entry 14). Based on the screening experiments, conditions of entry 5 and 6 were chosen for the synthesis of tetrasubstituted products **3a–e** and **3g** on a preparative scale.

Table S1. Optimization of reaction conditions for **1a → 3a**.

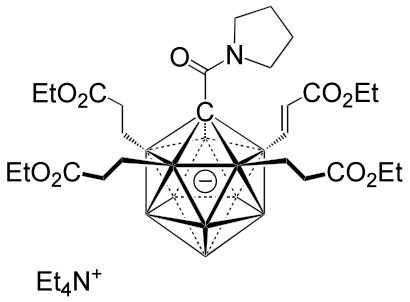
Entry	Catalyst[a]	Additive[b]	Solvent[c]	*T* [°C]	Result/substitution degree[d]
1	$[RhCp^*Cl_2]_2$	none	DMA	100	no reaction
2	$[RhCp^*Cl_2]_2$	Ag(I)	DMA	100	tri / tetra
3	$[RhCp^*Cl_2]_2$	Cu(II)	DMA	100	tri / tetra
4	$[RhCp^*Cl_2]_2$	Ag(I)/Cu(II)	DMA	60	tri / tetra
5	**$[RhCp^*Cl_2]_2$**	**Ag(I)/Cu(II)**	**DMA**	**100**	**tetra**
6	**$[RhCp^*Cl_2]_2$**	**Ag(I)/Cu(II)**	**DMA**	**120**	**tetra**
7	$[RhCp^*Cl_2]_2$	Ag(I)/Cu(II)[e]	DMA	100	tetra
8	$[RhCp^*Cl_2]_2$	Ag(I)/KOAc[f]	DMA	100	di / tri / tetra
9	$[RhCp^*Cl_2]_2$[g]	Ag(I)/Cu(II)	DMA	100	tetra
10	$[RhCp^*Cl_2]_2$	Ag(I)/Cu(II)	MeCN	100	di / tri / tetra
11	$[RhCp^*Cl_2]_2$	Ag(I)/Cu(II)	NMP	100	tetra
12	$[IrCp^*Cl_2]_2$	none	DMA	100	no reaction
13	$[IrCp^*Cl_2]_2$	Ag(I)/Cu(II)	DMA	100	di
14	$[Ru(cy)Cl_2]_2$	Ag(I)/Cu(II)	DMA	100	no reaction

[a]10 mol% of [TM] dimer, reaction run in a sealed vial under air; [b]Ag(I) = 40 mol% $AgSbF_6$, Cu(II) = 1 equiv $Cu(OAc)_2{\cdot}H_2O$; [c]DMA = dimethylacetamide, MeCN = acetonitrile, NMP = *N*-methyl-pyrrolidinone; [d]Assessed by ESI-MS and $^{11}B/^{11}B\{^1H\}$ NMR spectroscopy, di / tri / tetra = di-/tri-/tetra-substitution in positions B2–6, tetra = product **3a**; [e]Using 8 equiv $Cu(OAc)_2{\cdot}H_2O$; [f]2 equiv KOAc; [g]Reaction was set up in a glove box and run under N_2.

II. c) Synthesis of products 3

General procedure A for the Rh-catalyzed *ortho*-functionalization of the $[CB_{11}H_{12}]^-$ anion

To a mixture of pyrrolidine amide **1** (0.135 mmol), $[RhCp^*Cl_2]_2$ (0.0135 mmol), $AgSbF_6$ (0.054 mmol), and $Cu(OAc)_2 \cdot H_2O$ (0.135 mmol) was added DMA (2.5 mL) in a 10 mL screw capped vial equipped with a magnetic stir bar. To the stirring mixture was added the required alkene or alkyne (0.81 mmol) followed by heating using a thermostated oil bath (100 °C, 24 h for the preparation of **3a**, **3b, 3c**, and **3f** (MeCN solvent) and 120 °C, 48 h for **3d**, **3e** and **3g**). The mixture was filtered through celite and the filtrate evaporated to dryness under reduced pressure. The residue was taken up in dichloromethane and washed with 20% aqueous $[Et_4N]^+Br^-$ solution, dried ($MgSO_4$), and concentrated. Purification was done by chromatography on silica gel by eluting with a mixture of CH_2Cl_2/CH_3CN to afford the desired product.



3a： Prepared following the general procedure A, using **1a** and ethyl acrylate, **3a** (77 mg, 74%) was obtained as a colorless solid. Starting from 803 mg (2.17 mmol) of **1a**, the yield of **3a** was 70% (1.168 g, 1.52 mmol).

$^1H\{^{11}B\}$ NMR (500 MHz, acetonitrile-d_3, 23 °C): δ 6.94 (d, J = 17.6 Hz, 1H, alkenyl CH), 6.12 (d, J = 17.6 Hz, 1H, alkenyl CH), 4.15-4.05 (m, 2H, CH_3CH_2 of unsaturated ester), 4.09-4.00 (overlapping m, 6H, CH_3CH_2 of saturated ester), 3.60-3.42 (broad m, 2H, NCH_2CH_2), 3.41-3.31 (broad m, 2H, NCH_2CH_2), 3.16 (q, J =

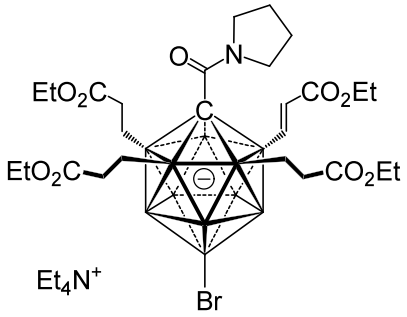
7.3 Hz, 8H, CH_2 of cation), 2.32-2.21 (m, 6H, BCH_2CH_2), 1.81-0.60 (overlapping signals, 41H, NCH_2CH_2, BCH_2, BH, cation CH_3, ester CH_3).

^{11}B NMR (160 MHz, DMSO-d_6, 80 °C): δ ca. 0 to -11 (overlapping signals, 5B), ca. -11 to 18 (overlapping signals, 6B).

$^{11}B\{^1H\}$ NMR (160 MHz, DMSO-d_6, 80 °C): δ ca. 0 to -11 (overlapping signals, 5B), ca. -11 to 18 (overlapping signals, 6B).

$^{13}C\{^1H\}$ NMR (125 MHz, acetone-d_6, 19 °C): δ 176.35 (ester CO), 176.04 (ester CO), 175.52 (ester CO), 166.67 (ester CO), 162.36 (amide CO), 151.69 (broad signal, B-*C*H), 130.12 (BCH*C*H), 73.93 (cage C), 60.15 (OCH_2), 60.08 (OCH_2), 9.92 (2 overlapping OCH_2), 52.99 (CH_2 of cation), 50.84 (2 overlapping NCH_2CH_2), 35.26 (BCH_2CH_2), 34.75 (BCH_2CH_2), 34.69 (BCH_2CH_2), 25.96 (br, 2 overlapping NCH_2CH_2), 14.62 (4 overlapping OCH_2CH_3), 11.76 (br, 3 overlapping BCH_2CH_2), 7.68 (CH_3 of cation).

HRMS (ESI): *m/z* Calcd. for $[C_{26}H_{49}B_{11}NO_9]^-$, 638.4524; found, 638.4534.



3b: Prepared following the general procedure A, using **1c** and ethyl acrylate, **3b** (74 mg, 65% yield) was obtained as a colorless solid.

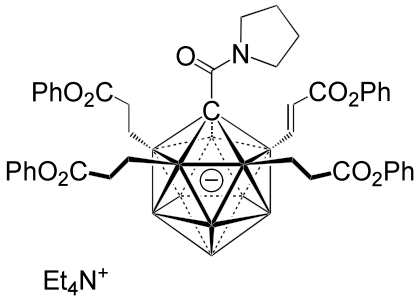
$^1H\{^{11}B\}$ NMR (500 MHz, acetone-d_6, 19 °C): δ 6.99 (d, J = 17.6 Hz, 1H, alkenyl CH), 6.24 (d, J = 17.6 Hz, 1H, alkenyl CH), 4.16-4.08 (m, 2H, CH_3CH_2 of unsaturated ester), 4.07-3.99 (overlapping m, 6H, CH_3CH_2 of saturated ester), 3.60-3.45 (overlapping signals, 10 H, NCH_2CH_2, CH_2 of cation), 3.38 (br, 2H, NCH_2CH_2), 2.45-2.26 (m, 6H, BCH_2CH_2), 2.03-0.77 (overlapping signals, 40H, NCH_2CH_2, BCH_2, BH, cation CH_3, ester CH_3).

^{11}B NMR (160 MHz, acetone-d_6, 19 °C): δ ca. 3 to -10 (overlapping signals, 5B), ca. -10 to 20 (overlapping signals, 6B).

^{11}B{^{1}H} NMR (160 MHz, acetone-d_6, 19 °C): δ ca. 3 to -10 (overlapping signals, 5B), ca. -10 to 20 (overlapping signals, 6B).

^{13}C{^{1}H} NMR (125 MHz, acetone-d_6, 19 °C): δ 176.03 (ester CO), 175.74 (ester CO), 175.27 (ester CO), 166.54 (ester CO), 162.61 (amide CO), 130.88 (BCH*C*H), 67.79 (cage C), 60.30 (OCH_2), 60.19 (OCH_2), 60.02 (2 overlapping OCH_2), 53.05 (CH_2 of cation), 50.91 (2 overlapping N*C*H_2CH$_2$), 35.17 (BCH$_2$*C*H$_2$), 34.56 (2 overlapping BCH$_2$*C*H$_2$), 26.05 (br, 2 overlapping NCH$_2$*C*H$_2$), 14.62 (4 overlapping B CH$_2$*C*H$_3$), 11.35 (br, 3 overlapping B*C*H$_2$CH$_2$), 7.70 (CH_3 of cation). The signal of B*C*HCH was not be detected unambiguously because of coupling to ^{10}B and ^{11}B.

HRMS (ESI): *m/z* Calcd. for [$C_{26}H_{48}B_{11}BrNO_9$]$^-$, 717.3572; found, 717.3646.



3c： Prepared following the general procedure A, using **1a** and phenyl acrylate, **3c** (67 mg, 52% yield) was obtained as a colorless solid.

^{1}H{^{11}B} NMR (500 MHz, acetonitrile-d_3, 19 °C): δ 7.44-7.35 (m, 8H, ArH), 7.29-7.20 (overlapping signals, 5H, ArH, alkenyl CH), 7.14-7.05 (m, 8H, ArH), 6.43 (d, J = 17.6 Hz, 1H, alkenyl CH), 3.70-3.52 (br m, 2H, NCH_2CH$_2$), 3.50-3.39 (m, 2H, NCH_2CH$_2$), 3.13 (q, J = 7.3 Hz, 8H, CH_2 of cation), 2.74-2.55 (m, 6H, BCH$_2$CH_2), 1.94-0.89 (overlapping signals, 29H, NCH$_2$CH_2, BCH_2, BH, cation CH_3).

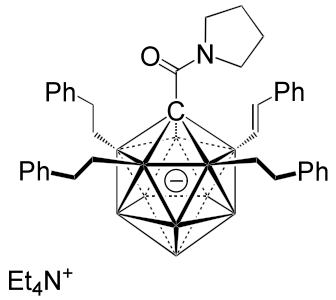
^{11}B NMR (160 MHz, acetonitrile-d_3, 19 °C): δ ca. 1 to -11 (overlapping signals, 5B), ca. -11 to 19 (overlapping signals, 6B).

^{11}B{^{1}H} NMR (160 MHz, acetonitrile-d_3, 19 °C): δ ca. 1 to -11 (overlapping signals,

5B), ca. -11 to 19 (overlapping signals, 6B).

$^{13}C\{^{1}H\}$ NMR (125 MHz, acetonitrile-d_3, 19 °C): δ 175.62 (ester CO), 175.32 (ester CO), 174.85 (ester CO), 165.66 (ester CO), 162.37 (amide CO), 154.10 (broad signal, B-CH), 152.41 (quaternary Ar-C), 152.34 (2 overlapping quaternary Ar-C), 152.16 (quaternary Ar-C), 130.34 (8 overlapping Ar CH), 129.55 (BCH*C*H), 126.44 (4 overlapping Ar CH), 122.94 (8 overlapping Ar CH), 74.52 (cage C), 53.01 (CH_2 of cation), 51.19 (2 overlapping N*C*H_2CH$_2$), 35.10 (BCH$_2$*C*H$_2$), 34.71 (BCH$_2$*C*H$_2$), 34.64 (BCH$_2$*C*H$_2$), 26.29 (broad signal, 2 overlapping NCH$_2$*C*H$_2$), 11.74 (br, 3 overlapping B*C*H$_2$CH$_2$), 7.66 (CH_3 of cation).

HRMS (ESI): *m/z* Calcd. for $[C_{42}H_{49}B_{11}NO_9]^-$, 830.4504; found, 830.4538.



3d: Prepared following the general procedure A, using **1a** and styrene, **3d** (65 mg, 61% yield) was obtained as a colorless crystalline solid. Starting from 815 mg (2.20 mmol) of **1a**, the yield of **3d** was 64% (1.110 g, 1.41 mmol).

$^{1}H\{^{11}B\}$ NMR (500 MHz, acetone-d_6, 19 °C): δ 7.40-7.01 (overlapping m, 20H, ArH), 6.93 (d, J = 17.9 Hz, 1H, alkenyl H), 6.44 (d, J =17.9 Hz, 1H, alkenyl H), 3.80-3.55 (broad m, 2H, NC*H*$_2$CH$_2$), 3.55-3.42 (m, 8H, CH_2 of cation), 3.42-3.30 (m, 2H, NC*H*$_2$CH$_2$), 2.90-2.69 (overlapping m, 6H, ArCH$_2$), 1.96-0.89 (overlapping m, 17H, NCH$_2$C*H*$_2$, BCH$_2$ and BH), 1.38 (tt, J = 7.3 Hz, 1.9 Hz, 12H, CH_3 of cation).

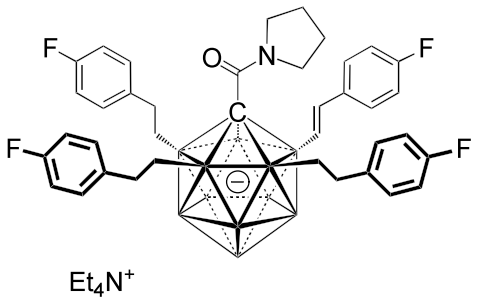
^{11}B NMR (160 MHz, acetone-d_6, 19 °C): δ ca. 0.0 to -11 (overlapping signals, 5B), ca. -11 to -20.7 (overlapping signals, 6B).

$^{11}B\{^{1}H\}$ NMR (160 MHz, acetone-d_6, 19 °C): δ ca. 0.0 to -11 (overlapping signals,

5B), ca. -11 to -20.7 (overlapping signals, 6B).

$^{13}C\{^{1}H\}$ NMR (125 MHz, DMSO-d_6, 80°C): δ 161.55 (CO), 146.84, 146.63, 145.75, 138.94, 137.59, 128.00-124.16 (overlapping signals), 72.97 (cage C), 51.52 (CH_2 of cation), 49.14 (NCH_2CH_2), 35.41 (BCH_2CH_2), 34.94 (2 overlapping BCH_2CH_2), 24.42 (NCH_2CH_2), 18.12 (br, 3 overlapping BCH_2CH_2), 6.65 (CH_3 of cation). The signal of B*C*HCH was not be detected unambiguously because of coupling to ^{10}B and ^{11}B.

HRMS (ESI): *m/z* Calcd. for $[C_{38}H_{49}B_{11}NO]^-$, 654.4936; found, 654.4886.



3e: Prepared following the general procedure A, using **1a** and 4-fluorostyrene, **3e** (66 mg, 57% yield) was obtained as a colorless crystalline solid.

$^{1}H\{^{11}B\}$ NMR (500 MHz, DMSO-d_6, 80 °C): δ 7.38-7.31 (m, 2H, ArH), 7.20-6.93 (overlapping m, 14H, ArH), 6.77 (d, J = 17.9 Hz, 1H, alkenyl CH), 6.21 (d, J = 17.9 Hz, 1H, alkenyl CH), 3.60-3.48 (broad m, 2H, NCH_2CH_2), 3.40-3.29 (m, 2H, NCH_2CH_2), 3.23 (q, J = 7.3 Hz, 8H, CH_2 of cation), 2.77-2.55 (overlapping m, 6H, $ArCH_2$), 1.90-0.80 (overlapping m, 17H, NCH_2CH_2, BCH_2 and BH), 1.20 (tt, J = 7.3 Hz, 1.9 Hz, 12H, CH_3 of cation).

^{11}B NMR (160 MHz, DMSO-d_6, 80 °C): δ ca 3 to -10 (overlapping signals, 5B), ca. -10 to -20 (overlapping signals, 6B).

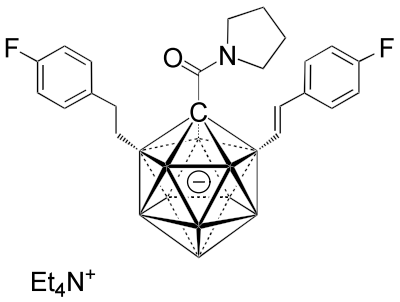
$^{11}B\{^{1}H\}$ NMR (160 MHz, DMSO-d_6, 80 °C): δ ca 3 to -10 (overlapping signals, 5B), ca. -10 to -20 (overlapping signals, 6B).

$^{13}C\{^{1}H\}$ NMR (125 MHz,DMSO-d_6, 80°C): Peaks that were detected between 170

and 110 ppm (some signals were not clearly detected because of coupling to ^{19}F, ^{10}B and ^{11}B): δ 161.50, 160.69, 158.79, 142.71, 142.51, 136.38, 135.48, 128.67, 126.62, 114.86, 114.69, 114.27, 114.10, 113.94. Region below 100 ppm: δ 72.96 (cage C), 51.54 (CH_2 of cation), 49.16 (NCH_2CH_2), 34.41 (BCH_2CH_2), 34.01 (BCH_2CH_2), 33.96 (BCH_2CH_2), 24.44 (NCH_2CH_2), 19.47 (broad, 3 overlapping BCH_2CH_2), 6.69 (CH_3 of cation).

^{19}F NMR (564 MHz, DMSO-d_6, 23 °C): δ -115.81 (m, 1F), -118.68 (m, 1F), -119.02 (m, 1F), -119.11 (m, 1F).

HRMS (ESI): *m/z* Calcd. for $[C_{38}H_{45}B_{11}F_4NO]^-$, 726.4553; found, 726.4517.



3f: Prepared following the general procedure A, using **1a** and 4-fluorostyrene as substrate and acetonitrile as solvent, **3f** (36 mg, 44% yield) was obtained as a colorless solid.

$^1H\{^{11}B\}$ NMR (500 MHz, acetonitrile-d_3, 19 °C): δ 7.42-7.34 (m, 2H, ArH), 7.19-7.12 (m, 2H, ArH), 7.05-6.89 (m, 4H, ArH), 6.72 (d, J = 18.2 Hz, 1H, alkenyl H), 6.60 (d, J = 18.2 Hz, 1H, alkenyl H), 3.82-3.23 (br overlapping m, 4H, NCH_2CH_2), 3.14 (q, J = 7.3 Hz, 8 H, CH_2 of cation), 2.72-2.64 (br m, 2H, $ArCH_2$), 2.10-0.98 (overlapping m, 15H, NCH_2CH_2, BCH_2 and BH), 1.20 (tt, J = 7.3 Hz, 1.9Hz, 12H, CH_3 of cation).

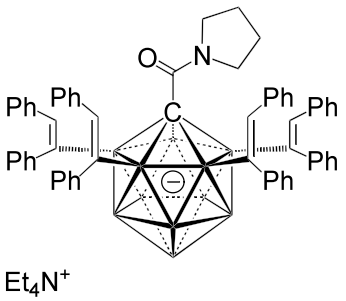
^{11}B NMR (160 MHz, acetonitrile-d_3, 19 °C): δ ca. 0 to -8 (overlapping signals, 3B), ca. -9 to -19 (overlapping signals, 8B).

$^{11}B\{^1H\}$ NMR (160 MHz, acetonitrile-d_3, 19 °C): δ ca. 0 to -8 (overlapping signals, 3B), ca. -9 to -19 (overlapping signals, 8B).

$^{13}C\{^{1}H\}$ NMR (125 MHz, acetonitrile-d_3, 23 °C): 164.00 (CO), 162.44 (d, J =242 Hz), 161.54.44 (d, J =240 Hz), 143.81, 137.40, 136.03, 130.22 (d, J =7.4 Hz), 128.02 (d, J =7.7 Hz), 115.88 (d, J =22 Hz), 115.32 (d, J =21 Hz), 73.03 (cage C), 50.36 (CH_2 of cation), 50.36 (two overlapping N*C*H_2CH$_2$), 35.57 (BCH$_2$*C*H$_2$), 27.39 (broad signal, NCH$_2$*C*H$_2$), 24.33 (broad signal, NCH$_2$*C*H$_2$), 20.90 (broad signal, B*C*H$_2$CH$_2$), 7.53 (CH_3 of cation). The B*C*HCH signal could not be detected clearly because of coupling to ^{10}B and ^{11}B).

^{19}F NMR (376 MHz, acetonitrile-d_3, 19 °C): δ -118.32 (m, 1F), -120.82 (m, 1F).

HRMS (ESI): *m/z* Calcd. for $[C_{22}H_{31}B_{11}F_2NO]^-$, 482.3470; found, 482.3477.



3g: Prepared following the general procedure A, using **1a** and diphenylacetylene, **3g** (73 mg, 50% yield) was obtained as a white solid.

NMR spectra were recorded in acetone-d_6 at 23 °C and in DMSO-d_6 at 80 °C. At 23 °C, rotational processes slow on the NMR time scale gave rise to distinct signals for the 4 CH_2 groups of the pyrrolidine moiety (broad for $^{1}H\{^{11}B\}$, better visible for $^{13}C\{^{1}H\}$). At 80 °C the signals were extremely broad, indicating that this is close to the coalescence temperature. We therefore provide the data recorded at 23 °C.

$^{1}H\{^{11}B\}$ NMR (500 MHz, acetone-d_6, 23 °C): δ 7.50-6.65 (overlapping m, 44H, ArH and alkenyl H), 3.42-3.32 (overlapping m, 10H, NC*H*$_2$CH$_2$ and CH_2 of cation), 3.17 (broad m, 2H, NC*H*$_2$CH$_2$), 1.90-1.10 (overlapping m, 23H, B-H, NCH$_2$C*H*$_2$ and CH_3 of cation).

^{11}B NMR (160 MHz, acetone-d_6, 23 °C): δ ca. 2 to -9 (overlapping signals, 5B), ca. -10 to - 20 (overlapping signals, 6B).

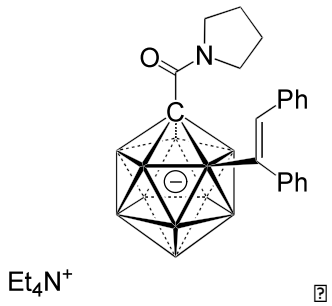
$^{11}B\{^{1}H\}$ NMR (160 MHz, acetone-d_6, 23 °C): δ ca. 2 to -9 (overlapping signals, 5B), ca. -10 to - 20 (overlapping signals, 6B).

$^{13}C\{^{1}H\}$ NMR (125 MHz, acetone-d_6, 23 °C): δ 161.59 (CO), 148.78, 146.62, 144.68, 141.63, 140.09, 137.11, 132 to 124 (multiple overlapping signals), 72.50 (cage C), 52.95 (CH_2 of cation), 51.39 (NCH_2CH_2), 50.79 (NCH_2CH_2), 27.77 (NCH_2CH_2), 22.95 (NCH_2CH_2), 7.65 (CH_3 of cation).

HRMS (ESI): *m/z* Calcd. for $[C_{62}H_{59}B_{11}NO]^-$, 953.5707; found, 953.5709.

General procedure B for the Ir-catalyzed *ortho*-functionalization of the $[CB_{11}H_{12}]^-$ anion

To a mixture of pyrrolidine amide **1a** or *N*-tosyl amide **1b** (0.107 mmol), $[IrCp^*Cl_2]_2$ (0.0107 mmol), and $AgSbF_6$ (0.0428 mmol) was added dichloroethane (2.5 ml) in a 10 mL screw capped vial equipped with a magnetic stir bar. To the stirring mixture was added sulfonyl azide or diphenylacetylene (0.535 mmol) followed by heating using a thermostated oil bath (80 °C , 8 h for the preparation of **3i** and **3j** and 100 °C, 24 h for **3h**). The solvent was removed in a vacuum, and the residue was purified on a silica gel column eluting with CH_2Cl_2/CH_3CN to afford the desired compound as a white solid.



3h: Prepared following the general procedure B, using pyrrolidine amide **1a** and diphenylacetylene, **3h** (32 mg, 55% yield) was obtained as a colorless solid. Starting from 807 mg (2.18 mmol) of **1a**, the yield of **3h** was 52% (625 mg, 1.14 mmol).

$^1H\{^{11}B\}$ NMR (500 MHz, DMSO-d_6, 80 °C): δ 7.23-7.16 (m, 2H), 7.13-7.08 (m, 1H), 7.03-6.95 (m, 3H), 6.94-6.89 (m, 2H), 6.88-6.84 (m, 1H), 6.78-6.72 (m, 2H), 3.32-3.18 (overlapping m, 12H, NCH_2CH_2 and CH_2 of cation), 2.15-1.30 (overlapping m, 14H, NCH_2CH_2 and BH), 1.21 (tt, J = 7.3 Hz, 1.9 Hz, 12H, CH_3 of cation).

^{11}B NMR (160 MHz, DMSO-d_6, 80 °C): δ ca. -1 to -7 (overlapping signals, 2B), ca. -10 to - 18 (overlapping signals, 9B).

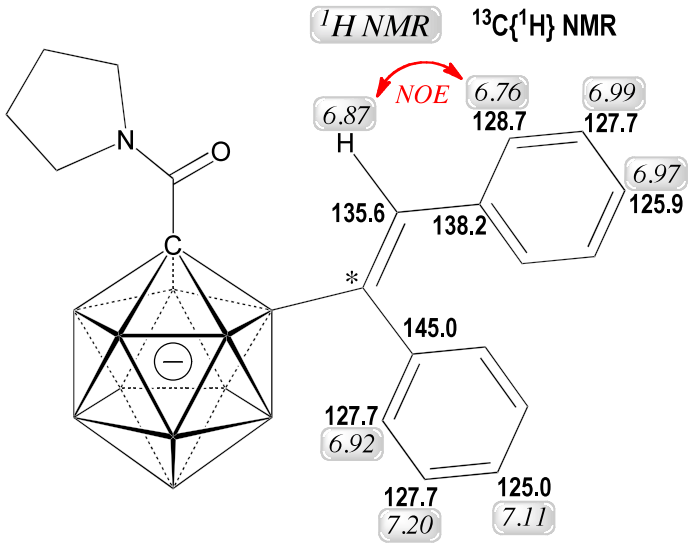
$^{11}B\{^1H\}$ NMR (160 MHz, DMSO-d_6, 80 °C): δ ca. -1 to -7 (overlapping signals, 2B), ca. -10 to - 18 (overlapping signals, 9B).

$^{13}C\{^1H\}$ NMR (125 MHz, DMSO-d_6, 80°C): δ 160.81 (CO), 144.88, 138.27, 134.96,

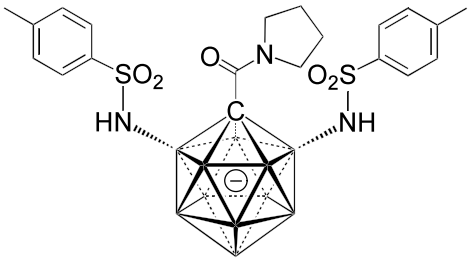
128.23, 127.58, 127.04, 126.91, 125.20, 124.31, 72.39 (cage C), 51.55 (CH_2 of cation), 47.98 (NCH_2CH_2), 24.12 (br, NCH_2CH_2), 6.69 (CH_3 of cation). The signal of B*C*HCH was not be detected unambiguously because of coupling to ^{10}B and ^{11}B.

HRMS (ESI): *m/z* Calcd. for $[C_{20}H_{29}B_{11}NOM]^-$, 418.3362; found, 418.3363.

Detailed assignment of ^1H and $^{13}C\{^1H\}$ NMR signals of **3h** based on COSY, HSQC, HMBC and NOESY NMR spectra in acetone-d_6 at 23 °C:



* ^{13}C signal could not be detected because of strong coupling to ^{10}B and ^{11}B.



Et_4N^+

3i: Prepared following the general procedure B, using pyrrolidine amide **1a** and sulfonyl azide TsN_3, **3i** (46 mg, 60% yield) was obtained as a colorless crystalline solid.

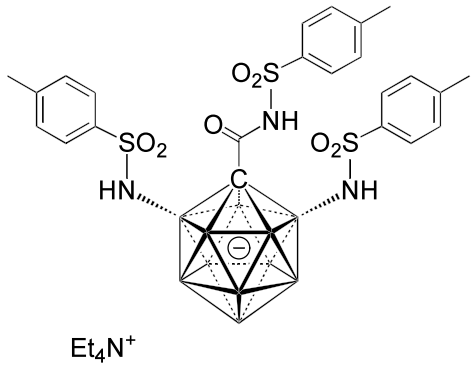
$^{1}H\{^{11}B\}$ NMR (500 MHz, acetone-d_6, 19 °C): δ 7.70 (d, J = 7.5 Hz, 2H, ArH), 7.25 (d, J = 7.5 Hz, 2H, ArH), 5.59 (broad s, 2H, NH), 3.51 (q, J = 7.3 Hz, 8H, CH_2 of cation), 3.41-3.24 (br m, 4H, overlapping NCH_2CH_2), 2.37 (s, 6H, CH_3), 2.10-1.18 (overlapping m, 13H, NCH_2CH_2 and BH), 1.40 (tt, J = 7.3 Hz, 1.9 Hz, 12H, CH_3 of cation).

^{11}B NMR (160 MHz, DMSO-d_6, 80 °C): δ ca. -2 to -9 (overlapping signals, 3B), ca. -10 to -22 (overlapping signals, 8B).

$^{11}B\{^{1}H\}$ NMR (160 MHz, DMSO-d_6, 80 °C): δ ca. -2 to -9 (overlapping signals, 3B), ca. -10 to -22 (overlapping signals, 8B).

$^{13}C\{^{1}H\}$ NMR (125 MHz, DMSO-d_6, 80°C): δ 159.03 (CO), 140.93 (quaternary aryl C), 140.51 (quaternary aryl C), 128.15 (aryl CH), 125.98 (aryl CH), 72.16 (cage C), 51.56 (CH_2 of cation), 48.01 (NCH_2CH_2), 24.05 (NCH_2CH_2), 20.37 ($ArCH_3$), 6.71 (CH_3 of cation).

HRMS (ESI): *m/z* Calcd. for $[C_{20}H_{33}B_{11}N_3O_5S_2]^-$, 578.2970; found, 578.2938.



3j: Prepared following the general procedure B, using *N*-tosyl amide **1b** and sulfonyl azide TsN_3, **3j** (37 mg, 43% yield) was obtained as a colorless crystalline solid.

$^{1}H\{^{11}B\}$ NMR (500 MHz, acetone-d_6, 19°C): δ 8.02 (d, J = 8.3 Hz, 2H, Ar-H), 7.76 (d, J = 8.3 Hz, 4H, Ar-H), 7.38 (d, J = 8.0 Hz, 2H, Ar-H), 7.29 (d, J = 8.0 Hz, 4H, Ar-H), 6.32 (broad s, 2H, NH), 3.49 (q, J = 7.3 Hz, 8H, CH_2 of cation), 2.44 (s, 3H, Ar-CH_3), 2.39 (s, 6H, Ar-CH_3), 1.88-1.10 (broad overlapping m, 9H, B-H), 1.39 (tt, J = 7.3 Hz, 1.9 Hz, 12H, CH_3 of cation).

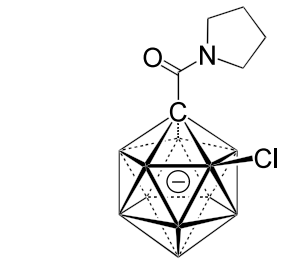
^{11}B NMR (160 MHz, acetone-d_6, 19 °C): δ ca. -2 to -9 (overlapping signals, 3B), ca.

-12 to -22 (overlapping signals, 8B).

$^{11}B\{^{1}H\}$ NMR (160 MHz, acetone-d_6, 19 °C): δ ca. -2 to -9 (overlapping signals, 3B), ca. -12 to -22 (overlapping signals, 8B).

^{13}C NMR (125 MHz, acetone-d_6, 19 °C): δ 142.58 (overlapping signals, 4 quaternary aryl C), 141.70 (2 quaternary aryl C), 130.11 (2 aryl CH), 129.68 (overlapping signals, 6 aryl CH), 127.98 (4 aryl CH), 52.97 (CH_2 of cation), 21.57 (1 $ArCH_3$), 21.40 (2 $ArCH_3$), 7.67 (CH_3 of cation). The assignment for the tolyl moieties was made based on a comparison to spectra of **3i**. The signals of CO and cage C could not be detected clearly.

HRMS (ESI): Calcd. for $[C_{23}H_{33}B_{11}N_3O_7S_3]^-$, 678.2591; found, 678.2549.



3k: A 10 mL glass vial equipped with a magnetic stir bar was charged with **1a** (50.0 mg, 0.135 mmol), $[RhCp^*Cl_2]_2$ (2 mg, 2.5 mol%), $AgBF_4$ (2.6 mg, 10 mol%), $Cu(OAc)_2 \cdot H_2O$ (27 mg, 0.135 mmol), *N*-chlorosuccinimide (54 mg, 0.405 mmol), and dichloroethane (3 mL). The glass vial was securely sealed and immersed in an oil bath and heated at 80 °C for 2 h. The mixture was filtered through celite and the filtrate evaporated to dryness in a vacuum. The residue was taken up in dichloromethane and washed with water, brine, dried ($MgSO_4$), and concentrated. Purification on a silica gel column with CH_2Cl_2/CH_3CN (4:1 *v:v*) as the eluent provided **3k** (45 mg, 82% yield) as a colorless solid.

$^{1}H\{^{11}B\}$ NMR (500 MHz, acetonitrile-d_3, 23 °C): δ 3.94-3.54 (broad m, 2H, N-CH_2), 3.54-3.22 (broad m, 2H, N-CH_2), 3.16 (q, J = 7.3 Hz, 8H, CH_2 of cation), 2.47-1.26 (overlapping m, 14H, N-CH_2-CH_2 and B-H), 1.21 (tt, J = 7.3 Hz, 1.9Hz, 12H, CH_3 of

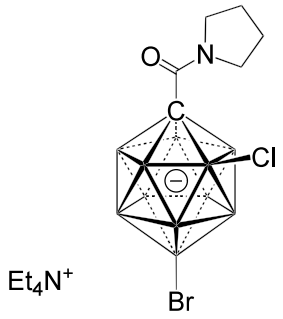
cation).

^{11}B NMR (160 MHz, DMSO-d_6, 80 °C): δ -3.75 (s, 1B, B-Cl), -5.56 (d, J = 137.1 H, 1B, B-12), ca. -10 to -16 (overlapping signals, 8B), -17.44 (d, J = 139.7 Hz, 1B).

^{11}B{^{1}H} NMR (160 MHz, DMSO-d_6, 80 °C): δ -3.74 (s, 1B, B-Cl), -5.56 (s, 1B, B-12), ca. -10 to -16 (overlapping signals, 8B), -17.44 (1B).

^{13}C{^{1}H} NMR (125 MHz, acetonitrile-d_3, 23 °C): δ 161.40 (CO), 74.45 (cage C), 53.04 (CH_2 of cation), 50.19 (overlapping signals, N*C*H$_2$CH$_2$), 27.93 (broad signal, NCH$_2$*C*H$_2$), 24.00 (broad signal, NCH$_2$*C*H$_2$), 7.62 (CH_3 of cation).

HRMS (ESI): *m/z* Calcd. for $[C_6H_{18}B_{11}ClNO]^-$, 274.2181; found, 274.2183.



3l: Following a similar procedure as for the preparation of **3k**, using **1c** (50 mg, 0.11 mmol) and *N*-chlorosuccinimide (44 mg, 0.33 mmol), **3l** (38 mg, 71% yield) was obtained as a colorless solid.

^{1}H{^{11}B} NMR (500 MHz, acetonitrile-d_3, 23 °C): δ 3.91-3.55 (broad m, 2H, NC*H*$_2$CH$_2$), 3.55-3.23 (broad m, 2H, NC*H*$_2$CH$_2$), 3.16 (q, J = 7.3 Hz, 8H, CH_2 of cation), 2.45-1.26 (overlapping m, 13H, NCH$_2$C*H*$_2$ and BH), 1.21 (tt, J = 7.3 Hz, 1.9 Hz, 12H, CH_3 of cation).

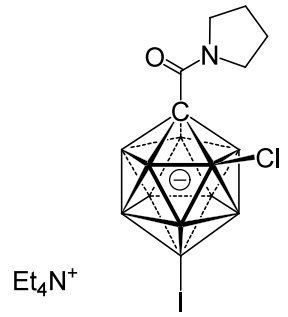
^{11}B NMR (160 MHz, acetone-d_6, 19 °C): δ -1.16 (s, 1B, B-Br), -4.51 (s, 1B, B-Cl), ca. -9 to -19 (overlapping signals, 9B).

^{11}B{^{1}H} NMR (160 MHz, acetone-d_6, 19 °C): δ -1.24 (s, 1B, B-Br), -4.56 (s, 1B, B-Cl), ca. -9 to -19 (overlapping signals, 9B).

^{13}C{^{1}H} NMR (125 MHz, acetonitrile-d_3, 23°C): δ 161.41 (CO), 68.09 (cage C), 53.04 (CH_2 of cation), 50.28 (2 overlapping signals, N*C*H$_2$CH$_2$), 27.82 (broad signal,

NCH_2CH_2), 24.02 (broad signal, NCH_2CH_2), 7.63 (CH_3 of cation).

HRMS (ESI): *m/z* Calcd. for $[C_6H_{17}B_{11}BrClNO]^-$, 353.1279; found, 353.1285.



3m: Following a similar procedure as for the preparation of **3k**, using **1d** (50 mg, 0.10 mmol) and *N*-chlorosuccinimide (26.7 mg, 0.20 mmol), **3m** (36 mg, 68% yield) was obtained as a colorless solid.

$^1H\{^{11}B\}$ NMR (500 MHz, acetonitrile-d_3, 23 °C): δ 3.90-3.53 (broad m, 2H, NCH_2CH_2), 3.52-3.25 (broad m, 2H, NCH_2CH_2), 3.17 (q, J = 7.3 Hz, 8H, CH_2 of cation), 2.50-1.50 (overlapping m, 13H, NCH_2CH_2 and BH), 1.21 (tt, J = 7.3 Hz, 1.9 Hz, 12H, CH_3 of cation).

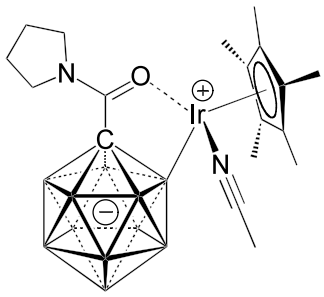
^{11}B NMR (160 MHz, acetonitrile-d_3, 23 °C): δ -3.80 (s, 1B, B-Cl), ca. -9.5 to -18 (overlapping signals, 10B).

$^{11}B\{^1H\}$ NMR (160 MHz, acetonitrile-d_3, 23 °C): δ -3.80 (s, 1B, B-Cl), ca. -9.5 to -18 (overlapping signals, 10B).

$^{13}C\{^1H\}$ NMR (125 MHz, acetonitrile-d_3, 23 °C): δ 161.59 (CO), 71.89 (cage C), 53.09 (CH_2 of cation), 50.28 (2 overlapping signals, NCH_2CH_2), 27.83 (broad signal, NCH_2CH_2), 23.83 (broad signal, NCH_2CH_2), 7.69 (CH_3 of cation).

HRMS (ESI): *m/z* Calcd. for $[C_6H_{17}B_{11}ClINO]^-$, 401.1103; found, 401.1134

c) Synthesis of iridium complex 4



[1-(CONC$_4$H$_8$)-CB$_{11}$H$_{10}$-IrCp*(CH$_3$CN)] (4): To a 2 mL glass vial equipped with a magnetic stir bar in a glovebox, IrCp*(OAc)$_2$ (35.6 mg, 0.08 mmol), **1a** (30 mg, 0.08 mmol) and acetonitrile (0.2 mL) were combined. The resulting mixture was stirred at for 3 h at 25 °C. The yellow precipitate that formed was collected by vacuum filtration through a fine glass frit, washed with hexane and dried in a vacuum. The filtrate was concentrated slowly at 25 °C (removal of ca. 50% of the solvent) to yield yellow crystals, which were collected by filtration and combined with the first fraction (40 mg, combined yield 82%).

^{1}H{^{11}B} NMR (500 MHz, acetonitrile-d_3, 23 °C): δ 3.91 (triplet-like m, 2H, NCH_2CH$_2$), 3.42 (triplet-like m, 2H, NCH_2CH$_2$), 2.51 (s, 3H, CH_3CN), 2.10-1.60 (overlapping m, 14H, NCH$_2$CH_2 and BH), 1.63 (s, 15H, Cp*CH_3).

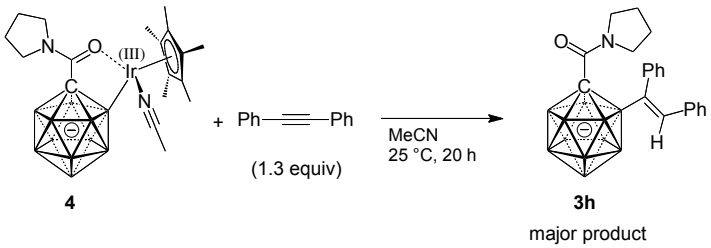
^{11}B NMR (160 MHz, acetonitrile-d_3, 23 °C): δ -4.41 (d, J = 136 Hz, 1B), -7.31 (s, 1B), ca. -10 to -17 (overlapping signals, 9B).

^{11}B{^{1}H} NMR (160 MHz, acetonitrile-d_3, 23 °C): δ -4.41 (1B), -7.31 (1B), ca. -10 to -17 (overlapping signals, 9B).

^{13}C{^{1}H} NMR (125 MHz, acetonitrile-d_3, 23 °C): δ 178.21 (CO), 89.89 (ring Cp*), 74.75 (cage C), 50.87 (NCH$_2$CH$_2$), 49.52 (NCH$_2$CH$_2$), 27.14 (NCH$_2$$CH_2$), 23.82 (NCH$_2$$CH_2$), 9.15 (Cp*$CH_3$).

Analysis calculated for C$_{18}$H$_{36}$B$_{11}$IrN$_2$O: C, 35.58; H, 5.97; N, 4.61; found: C, 35.12; H, 5.79; N, 4.51.

Reaction of 4 with diphenylacetylene



Scheme S2. Reaction of isolated **4** with diphenylacetylene.

In a glovebox, isolated complex **4** (10 mg) was dissolved in MeCN (0.8 mL) in a 4 mL glass vial. Diphenylacetylene (1.3 equiv) was added, and the solution was stirred at 25 °C. The reaction mixture was monitored by ESI-MS over 20 h, which showed gradual increase of the formation of **3h** as the major product. A small amount of di-substituted product was also observed (*ca.* 5%).

III X-ray Crystallography [8]

Crystal structure of 1a

Compound **1a** (10 mg, 0.027 mmol) was dissolved in acetone (0.5 mL) in a 1 mL glass vial. The resulting colorless solution was filtered into a 18 cm long NMR tube and layered with hexanes (1 mL). Colorless crystals of the composition $[Et_4N][C_6H_{19}B_{11}NO]$ suitable for X-ray diffraction grew within 3 d at room temperature.

```
Bond precision:  C-C = 0.0033 A          Wavelength=0.71073

Cell:            a=12.6563(8)     b=17.5897(10)     c=20.3134(12)
                 alpha=90         beta=90           gamma=90
Temperature:     172 K

                 Calculated                Reported
Volume           4522.2(5)                 4522.2(5)
Space group      P b c a                   P b c a
Hall group       -P 2ac 2ab                -P 2ac 2ab
Moiety formula   C6 H19 B11 N O, C8 H20 N  C6 H19 B11 N O, C8 H20 N
Sum formula      C14 H39 B11 N2 O          C14 H39 B11 N2 O
Mr               370.38                    370.38
Dx,g cm-3        1.088                     1.088
Z                8                         8
Mu (mm-1)        0.058                     0.058
F000             1600.0                    1600.0
F000'            1600.35
h,k,lmax         15,21,24                  15,21,24
Nref             4136                      4129
Tmin,Tmax        0.981,0.988               0.879,1.000
Tmin'            0.974

Correction method= MULTI-SCAN

Data completeness= 0.998          Theta(max)= 25.350

R(reflections)= 0.0581( 2919)     wR2(reflections)= 0.1609( 4129)

S = 1.041                 Npar= 257
```

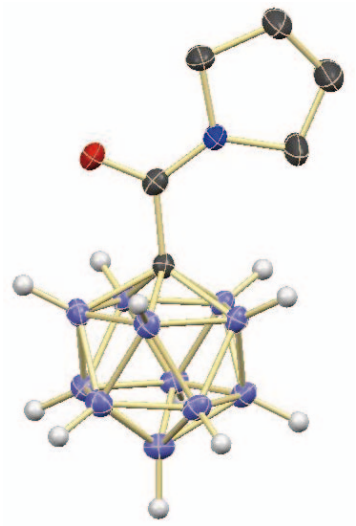


Figure S1. ORTEP representation of **1a**. Cation and hydrogen atoms except for B-H are omitted for clarity; 25% displacement ellipsoids.

Crystal structure of 1b

Compound **1b** (15 mg, 0.04 mmol) was dissolved in acetone (0.5 mL) in a 1 mL glass vial. The resulting colorless solution was filtered into a 18 cm long NMR tube and layered with hexanes (1 mL). Colorless crystals of the composition $[Et_4N][C_6H_{18}B_{11}BrNO]$ suitable for X-ray diffraction grew within 14 d at room temperature.

```
Bond precision:  C-C = 0.0063 A          Wavelength=0.71073

Cell:            a=12.7414(7)     b=18.7111(14)     c=20.5954(13)
                 alpha=90         beta=90           gamma=90
Temperature:     293 K

                 Calculated                  Reported
Volume           4910.1(6)                   4910.1(6)
Space group      P b c a                     P b c a
Hall group       -P 2ac 2ab                  -P 2ac 2ab
Moiety formula   C6 H18 B11 Br N O, C8 H20 N C6 H18 B11 Br N O, C8 H20 N
Sum formula      C14 H38 B11 Br N2 O         C14 H38 B11 Br N2 O
Mr               449.27                      449.27
Dx,g cm-3        1.215                       1.216
Z                8                           8
Mu (mm-1)        1.683                       1.683
F000             1872.0                      1872.0
F000'            1870.08
h,k,lmax         15,22,24                    15,22,24
Nref             4503                        4496
Tmin,Tmax        0.490,0.624                 0.662,1.000
Tmin'            0.480

Correction method= # Reported T Limits: Tmin=0.662 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.998          Theta(max)= 25.350

R(reflections)= 0.0557( 2982)     wR2(reflections)= 0.1632( 4496)

S = 1.027                 Npar= 266
```

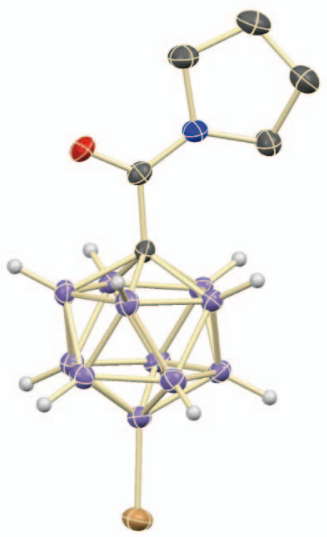


Figure S2. ORTEP representation of **1c**. Cation and hydrogen atoms except for B-H are omitted for clarity; 25% displacement ellipsoids.

Crystal structure of 3a

Compound **3a** (10 mg, 0.013 mmol) was dissolved in a mixture of water and ethanol (1 mL/0.5 mL) in a 4 mL glass vial. Slow evaporation of ethanol over 2 weeks at room temperature afforded colorless crystals of the composition $[Et_4N][C_{26}H_{49}B_{11}NO_9]$ suitable for X-ray diffraction.

Bond precision:	C-C = 0.0094 A	Wavelength=0.71073	
Cell:	a=15.7074(16)	b=17.9799(16)	c=16.5251(16)
	alpha=90	beta=98.384(9)	gamma=90
Temperature:	293 K		
	Calculated	Reported	
Volume	4617.1(8)	4617.1(8)	
Space group	P 21/n	P 21/n	
Hall group	-P 2yn	-P 2yn	
Moiety formula	C26 H49 B11 N O9, C8 H18.07 N	?	
Sum formula	C34 H67.07 B11 N2 O9	C34 H69 B11 N2 O9	
Mr	766.88	768.82	
Dx,g cm-3	1.103	1.106	
Z	4	4	
Mu (mm-1)	0.073	0.073	
F000	1648.3	1656.0	
F000'	1648.98		
h,k,lmax	18,21,19	18,21,19	
Nref	8462	8419	
Tmin,Tmax	0.974,0.981	0.540,1.000	
Tmin'	0.966		

Correction method= # Reported T Limits: Tmin=0.540 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.995 Theta(max)= 25.346

R(reflections)= 0.1015(3182) wR2(reflections)= 0.3875(8419)

S = 1.043 Npar= 559

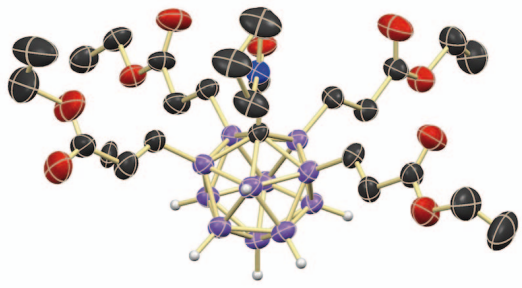
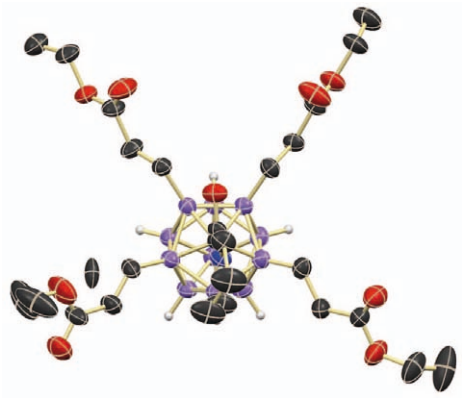
a)  b) 

Figure S3. ORTEP representation of **3a** (side view (a) and top view (b)). Cation and hydrogen atoms except for B-H are omitted for clarity; 25% displacement ellipsoids.

The R factor for this structure is relatively poor (10%). This crystal was measured at room temperature and diffracted quite weakly. There is some disorder concerning one of the ester groups (which is a terminal, floppy group of the anion), one CH_2 group of the pyrrolidine ring and the CH_2 groups of the cation. The latter disorder had to be treated with appropriate distance restraints.

Distinction of the alkenyl substituent *vs* the alkyl groups does not seem to be an issue. The corresponding bond lengths are:

C7–8 1.307(9)

C12–C13 1.426(6)

C17–C18 1.484(6)

C22–C23 1.488(6)

That is, the bond lengths ±3 times the standard uncertainty show a significant difference between the shortest distance and the next longer one, strongly suggesting one double bond (C7–8) and three single bonds (C12–13/17–18/22–23). Furthermore, this finding is consistent with the other crystal structures of this study.

Crystal structure of 3b

Compound **3b** (10 mg, 0.012 mmol) was dissolved in a mixture of water and ethanol (1 mL/0.5 mL) in a 4 mL glass vial. Slow evaporation of ethanol over 2 weeks at room temperature afforded colorless crystals of the composition $[Et_4N][C_{26}H_{48}B_{11}BrNO_9]$ suitable for X-ray diffraction.

Bond precision: C-C = 0.0168 A Wavelength=0.71073

Cell: a=15.7123(11) b=18.5073(11) c=21.4306(19)
alpha=90 beta=129.407(5) gamma=90
Temperature: 293 K

	Calculated	Reported
Volume	4815.1(7)	4815.1(6)
Space group	P 21/c	P 1 21/c 1
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C26 H48 B11 Br N O9, C8 H20 N	C26 H48 B11 Br N O9, C8 H20 N
Sum formula	C34 H68 B11 Br N2 O9	C34 H68 B11 Br N2 O9
Mr	847.71	847.72
Dx,g cm-3	1.169	1.169
Z	4	4
Mu (mm-1)	0.900	0.900
F000	1792.0	1792.0
F000'	1791.53	
h,k,lmax	18,22,25	18,22,25
Nref	8809	8676
Tmin,Tmax	0.656,0.698	0.901,1.000
Tmin'	0.643	

Correction method= # Reported T Limits: Tmin=0.901 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.985 Theta(max)= 25.350

R(reflections)= 0.0834(4243) wR2(reflections)= 0.2746(8676)

S = 1.028 Npar= 522

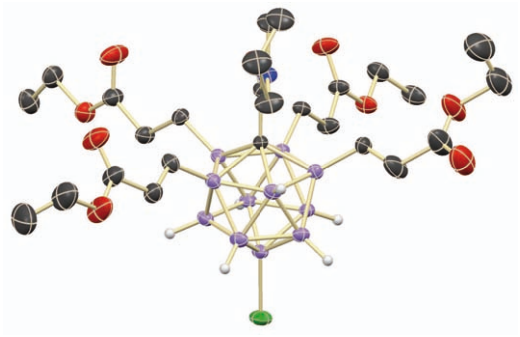
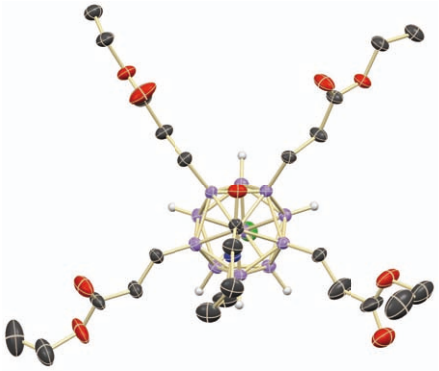
a)  b) 

Figure S4. ORTEP representation of **3b** (side view (a) and top view (b)). Cation and hydrogen atoms except for B-H are omitted for clarity; 25% displacement ellipsoids.

Crystal structure of 3d

Compound **3d** (15 mg, 0.019 mmol) was dissolved in acetone (0.5 mL) a 1 mL glass vial. The resulting colorless solution was filtered into a 18 cm long NMR tube and layered with hexanes (1 mL). Colorless crystals of the composition $[Et_4N][C_{38}H_{49}B_{11}NO]$ suitable for X-ray diffraction grew within 3 d at room temperature.

Bond precision:	C-C = 0.0090 A	Wavelength=0.71073	
Cell:	a=18.6641(7)	b=12.5049(5)	c=20.3898(11)
	alpha=90	beta=90	gamma=90
Temperature:	293 K		
	Calculated	Reported	
Volume	4758.8(4)	4758.8(4)	
Space group	P n a 21	P n a 21	
Hall group	P 2c -2n	P 2c -2n	
Moiety formula	C38 H49 B11 N O, C8 H20 N	C38 H49 B11 N O, C8 H20 N	
Sum formula	C46 H69 B11 N2 O	C46 H69 B11 N2 O	
Mr	784.94	784.94	
Dx,g cm-3	1.096	1.096	
Z	4	4	
Mu (mm-1)	0.060	0.060	
F000	1688.0	1688.0	
F000'	1688.49		
h,k,lmax	22,15,24	22,15,24	
Nref	8713[4490]	4482	
Tmin,Tmax	0.981,0.986	0.713,1.000	
Tmin'	0.981		

Correction method= MULTI-SCAN

Data completeness= 1.00/0.51 Theta(max)= 25.350

R(reflections)= 0.0604(3198) wR2(reflections)= 0.1824(4482)

S = 1.018 Npar= 545

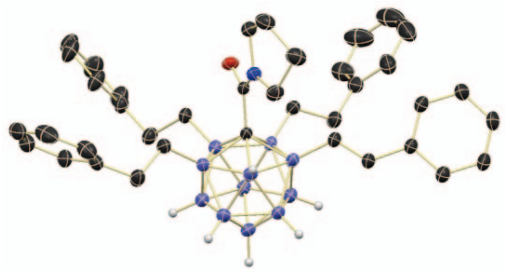
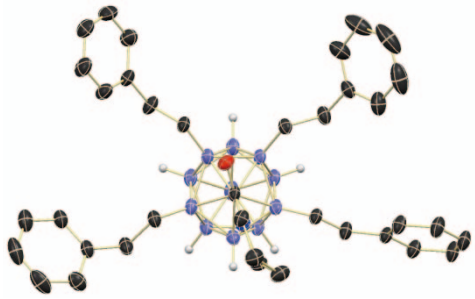
a)  b) 

Figure S5. ORTEP representation of **3d** (side view (a) and top view (b)). Cation and hydrogen atoms except for B-H are omitted for clarity; 25% displacement ellipsoids.

Crystal structure of 3e

Compound **3e** (12 mg, 0.014 mmol) was dissolved in acetone (0.5 mL) a 1 mL glass vial. The resulting colorless solution was filtered into a 18 cm long NMR tube and layered with hexanes (1 mL). Colorless crystals of the composition $[Et_4N][C_{38}H_{45}B_{11}F_4NO]$ suitable for X-ray diffraction grew within 3 d at room temperature.

```
Bond precision:  C-C = 0.0064 A          Wavelength=0.71073

Cell:            a=10.3433(7)    b=25.3537(15)     c=18.9697(13)
                 alpha=90        beta=99.089(6)    gamma=90
Temperature:     293 K

                 Calculated                        Reported
Volume           4912.2(6)                         4912.2(6)
Space group      P 21/n                            P 1 21/n 1
Hall group       -P 2yn                            -P 2yn
Moiety formula   C38 H45 B11 F4 N O, C8 H20 N      C38 H45 B11 F4 N O, C8 H20 N
Sum formula      C46 H65 B11 F4 N2 O               C46 H65 B11 F4 N2 O
Mr               856.91                            856.91
Dx,g cm-3        1.159                             1.159
Z                4                                 4
Mu (mm-1)        0.075                             0.075
F000             1816.0                            1816.0
F000'            1816.75
h,k,lmax         12,30,22                          12,30,22
Nref             9001                              8940
Tmin,Tmax        0.977,0.985                       0.945,1.000
Tmin'            0.969

Correction method= # Reported T Limits: Tmin=0.945 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.993          Theta(max)= 25.350

R(reflections)= 0.0744( 4259)     wR2(reflections)= 0.2466( 8940)

S = 1.033                 Npar= 581
```

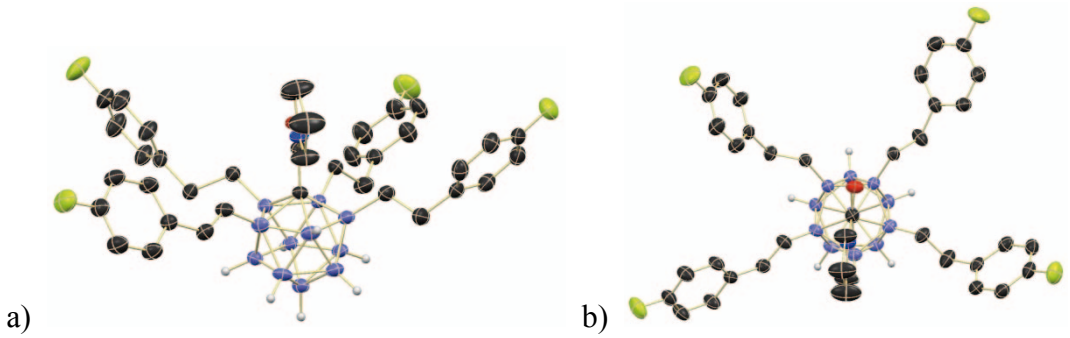


Figure S6. ORTEP representation of **3e** (side view (a) and top view (b)). Cation and hydrogen atoms except for B-H are omitted for clarity; 25% displacement ellipsoids.

Crystal structure of 3h

Compound **3h** (10 mg, 0.018 mmol) was converted to its $[Ph_4P]^+$ salt by cation exchange, which was accomplished by formation of the $[H_3O]^+$ salt (1 M aq. HCl/Et_2O extraction) and subsequent concentration/precipitation with 1 equiv aq. $[Ph_4P][Br]$. The phosphonium salt was dissolved in acetone (0.5 mL) a 1 mL glass vial. The resulting colorless solution was filtered into a 18 cm long NMR tube and layered with hexanes (1 mL). Colorless crystals of the composition $[Ph_4P][C_{20}H_{29}B_{11}NO]$ suitable for X-ray diffraction grew within 3 d at room temperature.

```
Bond precision:  C-C = 0.0057 A          Wavelength=0.71073

Cell:            a=9.3495(6)    b=18.1075(9)       c=25.4205(11)
                 alpha=90       beta=94.671(5)     gamma=90
Temperature:     293 K

                 Calculated                 Reported
Volume           4289.3(4)                  4289.3(4)
Space group      C c                        C 1 c 1
Hall group       C -2yc                     C -2yc
Moiety formula   C20 H29 B11 N O, C24 H20 P C20 H29 B11 N O, C24 H20 P
Sum formula      C44 H49 B11 N O P          C44 H49 B11 N O P
Mr               757.72                     757.72
Dx,g cm-3        1.173                      1.173
Z                4                          4
Mu (mm-1)        0.100                      0.100
F000             1592.0                     1592.0
F000'            1592.88
h,k,lmax         11,21,30                   11,21,30
Nref             7860[ 3935]                3927
Tmin,Tmax        0.952,0.963                0.958,1.000
Tmin'            0.952

Correction method= # Reported T Limits: Tmin=0.958 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 1.00/0.50      Theta(max)= 25.350

R(reflections)= 0.0419( 3426)     wR2(reflections)= 0.0978( 3927)

S = 1.018                 Npar= 523
```

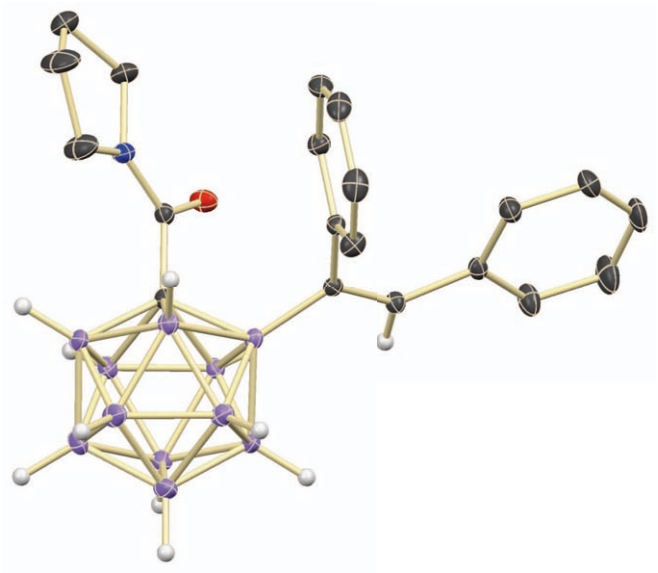


Figure S7. ORTEP representation of **3h**. Cation and hydrogen atoms except for B-H and the alkenyl C–H are omitted for clarity; 25% displacement ellipsoids.

Crystal structure of 3i

Compound **3i** (10 mg, 0.014 mmol) was dissolved in acetone (0.5 mL) a 1 mL glass vial. The resulting colorless solution was filtered into a 18 cm long NMR tube and layered with hexanes (1 mL). Colorless crystals of the composition $[Et_4N][C_{20}H_{33}B_{11}N_3O_5S_2]\cdot$acetone suitable for X-ray diffraction grew within 2 d at room temperature.

```
Bond precision:  C-C = 0.0067 A          Wavelength=0.71073

Cell:          a=13.0426(7)       b=13.4923(7)     c=13.9657(9)
               alpha=118.275(6)   beta=95.886(5)   gamma=96.271(4)
Temperature:   293 K
```

	Calculated	Reported
Volume	2117.8(2)	2117.7(2)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C20 H33 B11 N3 O5 S2, C8 H20 N, C3 H6 O	C20 H33 B11 N3 O5 S2, C8 H20 N, C3 H6 O
Sum formula	C31 H59 B11 N4 O6 S2	C31 H59 B11 N4 O6 S2
Mr	766.85	766.85
Dx,g cm-3	1.203	1.203
Z	2	2
Mu (mm-1)	0.170	0.170
F000	816.0	816.0
F000'	816.77	
h,k,lmax	15,16,16	15,16,16
Nref	7752	7725
Tmin,Tmax	0.937,0.952	0.770,1.000
Tmin'	0.937	

```
Correction method= # Reported T Limits: Tmin=0.770 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.997          Theta(max)= 25.350

R(reflections)= 0.0610( 5170)     wR2(reflections)= 0.1767( 7725)

S = 1.038                 Npar= 495
```

---

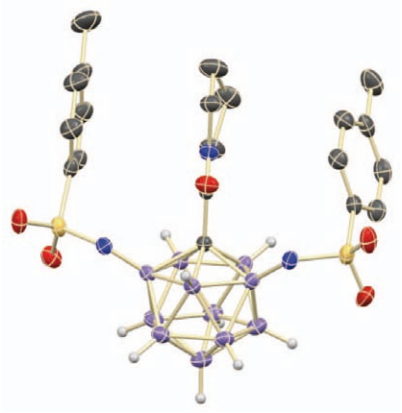
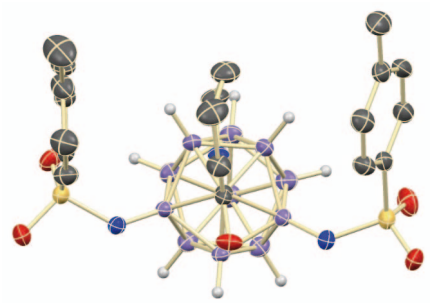
a)  b) 

Figure S8. ORTEP representation of **3i** (side view (a) and top view (b)). Cation, hydrogen atoms except for B-H and solvent molecule are omitted for clarity; 25% displacement ellipsoids.

Crystal structure of 3j

Compound **3j** (10 mg, 0.012 mmol) was dissolved in acetone (0.5 mL) a 1 mL glass vial. The resulting colorless solution was filtered into a 18 cm long NMR tube and layered with hexanes (1 mL). Colorless crystals of the composition $[Et_4N][C_{23}H_{33}B_{11}N_3O_7S_3]$ suitable for X-ray diffraction grew within 2 d at room temperature.

Bond precision: C-C = 0.0126 A Wavelength=0.71073

Cell: a=11.7951(8) b=18.427(1) c=20.4333(16)
alpha=90 beta=99.150(7) gamma=90

Temperature: 293 K

	Calculated	Reported
Volume	4384.6(5)	4384.6(5)
Space group	P 21/n	P 1 21/n 1
Hall group	-P 2yn	-P 2yn
Moiety formula	C23 H33 B11 N3 O7 S3, C7 H17 N, C H3	C23 H33 B11 N3 O7 S3, C8 H20 N
Sum formula	C31 H53 B11 N4 O7 S3	C31 H53 B11 N4 O7 S3
Mr	808.86	808.86
Dx,g cm-3	1.225	1.225
Z	4	4
Mu (mm-1)	0.216	0.216
F000	1704.0	1704.0
F000'	1706.08	
h,k,lmax	14,22,24	14,22,24
Nref	8026	8005
Tmin,Tmax	0.937,0.952	0.831,1.000
Tmin'	0.937	

Correction method= MULTI-SCAN

Data completeness= 0.997 Theta(max)= 25.350

R(reflections)= 0.1074(5099) wR2(reflections)= 0.3609(8005)

S = 1.300 Npar= 512

---

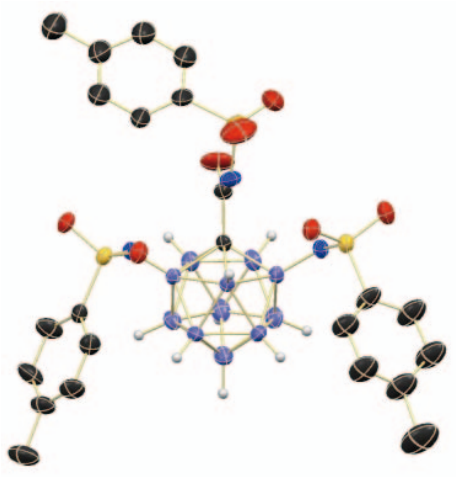
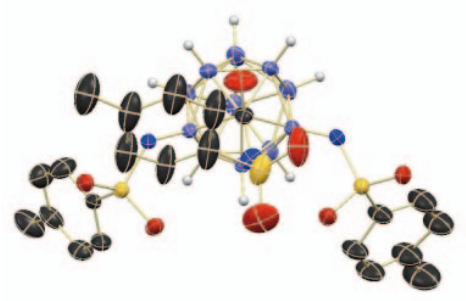
a)  b) 

Figure S9. ORTEP representation of **3j** (side view (a) and top view (b)). Cation and hydrogen atoms except for B-H are omitted for clarity; 25% displacement ellipsoids.

Crystal structure of 3k

Compound **3k** (10 mg, 0.025 mmol) was dissolved in dichloromethane (0.5 mL) a 1 mL glass vial. The resulting colorless solution was filtered into a 18 cm long NMR tube and layered with ethers (1 mL). Colorless crystals of the composition $[Et_4N][C_6H_{18}B_{11}ClNO]$ suitable for X-ray diffraction grew within 3 d at room temperature.

Bond precision:	**C-C = 0.0104 A**	**Wavelength=0.71073**
Cell:	**a=10.5179(11)**	**b=17.071(2)** **c=27.284(3)**
	alpha=90	**beta=90** **gamma=90**
Temperature:	**293 K**	
	Calculated	**Reported**
Volume	**4898.9(9)**	**4898.9(10)**
Space group	**P b c a**	**P b c a**
Hall group	**-P 2ac 2ab**	**-P 2ac 2ab**
Moiety formula	**C6 H18 B11 Cl N O, C8 H20 N**	**C6 H18 B11 Cl N O, C8 H20 N**
Sum formula	**C14 H38 B11 Cl N2 O**	**C14 H38 B11 Cl N2 O**
Mr	**404.82**	**404.82**
Dx,g cm-3	**1.098**	**1.098**
Z	**8**	**8**
Mu (mm-1)	**0.164**	**0.164**
F000	**1728.0**	**1728.0**
F000'	**1729.54**	
h,k,lmax	**12,20,32**	**12,20,32**
Nref	**4496**	**4479**
Tmin,Tmax	**0.923,0.940**	**0.870,1.000**
Tmin'	**0.923**	

Correction method= MULTI-SCAN

Data completeness= 0.996 **Theta(max)= 25.350**

R(reflections)= 0.1370(2219) **wR2(reflections)= 0.4423(4479)**

S = 1.399 **Npar= 276**

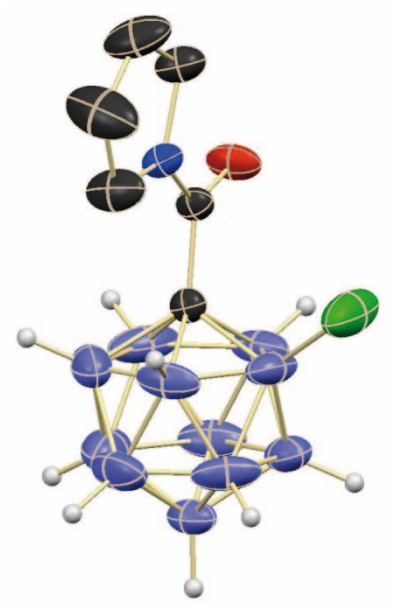


Figure S10. ORTEP representation of **3k**. Cation and hydrogen atoms except for B-H are omitted for clarity; 25% displacement ellipsoids.

Crystal structure of 4

Compound **4** (10 mg, 0.016 mmol) was dissolved in acetonitrile (0.5 mL) a 2 mL glass vial. The resulting yellow solution was left to evaporate slowly at room temperature. Yellow crystals of the composition $C_{18}H_{36}B_{11}IrN_2O$ suitable for X-ray diffraction grew within 2 d.

Bond precision:	**C-C = 0.0085 A**		**Wavelength=0.71073**
Cell:	**a=15.4017(7)**	**b=14.6479(6)**	**c=11.4845(5)**
	alpha=90	**beta=102.683(5)**	**gamma=90**
Temperature:	**293 K**		
	Calculated		**Reported**
Volume	**2527.7(2)**		**2527.72(19)**
Space group	**P 21/c**		**P 1 21/c 1**
Hall group	**-P 2ybc**		**-P 2ybc**
Moiety formula	**C18 H36 B11 Ir N2 O**		**C18 H36 B11 Ir N2 O**
Sum formula	**C18 H36 B11 Ir N2 O**		**C18 H36 B11 Ir N2 O**
Mr	**607.62**		**607.60**
Dx,g cm-3	**1.597**		**1.597**
Z	**4**		**4**
Mu (mm-1)	**5.297**		**5.297**
F000	**1192.0**		**1192.0**
F000'	**1186.68**		
h,k,lmax	**18,17,13**		**18,17,13**
Nref	**4633**		**4619**
Tmin,Tmax	**0.241,0.452**		**0.582,1.000**
Tmin'	**0.218**		

Correction method= # Reported T Limits: Tmin=0.582 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.997 **Theta(max)= 25.350**

R(reflections)= 0.0290(3824) **wR2(reflections)= 0.0664(4619)**

S = 1.049 **Npar= 304**

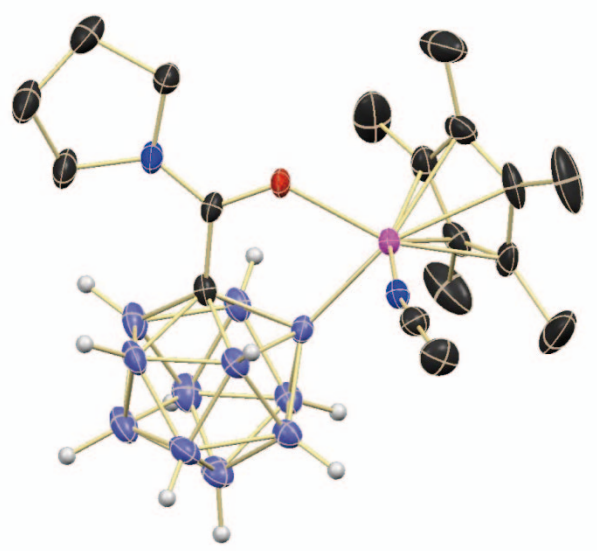


Figure S11. ORTEP representation of **4**. Hydrogen atoms except for B-H are omitted for clarity; 30% displacement ellipsoids.

IV References

1. Plešek, J.; Jelínek, T.; Drdákova, E.; Hermánek, S.; Štibr, B.; *Collect. Czech. Chem. Commun.*, **1984**, *49*, 1559–1562.
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8. CCDC1433744 (**1a**), CCDC1433745 (**1b**), CCDC1512465 (**3a**), CCDC1512466 (**3b**), CCDC1433746 (**3d**), CCDC1433747 (**3e**), CCDC1512467 (**3h**), CCDC1433748 (**3i**), CCDC1433749 (**3j**), CCDC1433750 (**3k**) and CCDC1433751 (**4**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

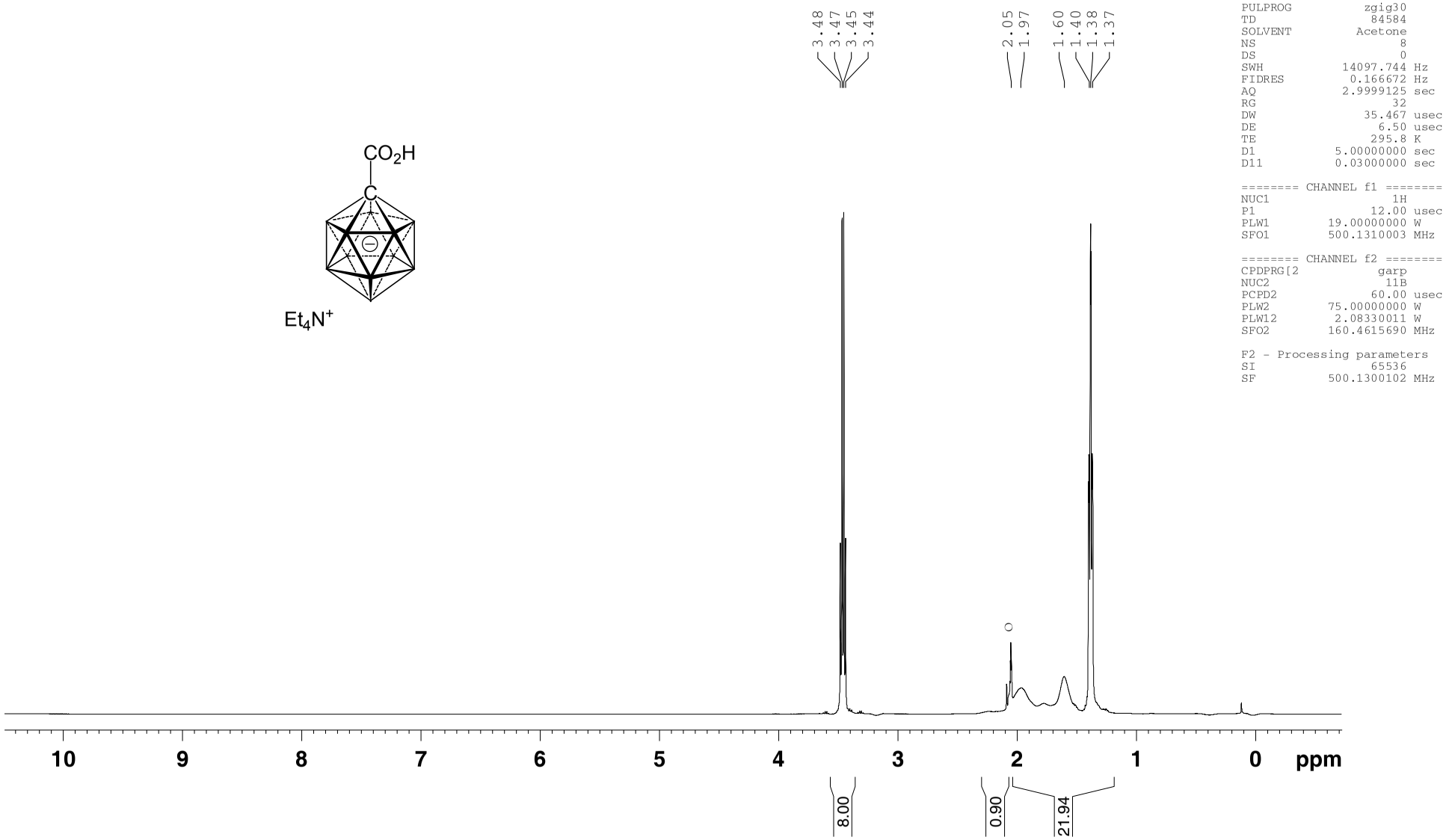
V NMR Spectra

Following on p. NMR1–NMR96

VI Mass Spectra

Following on p. MS1–MS18

1H{11B} NMR, 30 mg in 0.6 mL acetone-d6 T = 23 C
Solvent residual peak= 2.05 ppm○



NMR1

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EXPNO                    1
PROCNO                   1

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PULPROG           zgig30
TD                 84584
SOLVENT          Acetone
NS                     8
DS                     0
SWH            14097.744 Hz
FIDRES          0.166672 Hz
AQ             2.9999125 sec
RG                    32
DW                35.467 usec
DE                 6.50 usec
TE                295.8 K
D1           5.00000000 sec
D11          0.03000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.00 usec
PLW1         19.00000000 W
SFO1        500.1310003 MHz

======== CHANNEL f2 ========
CPDPRG[2              garp
NUC2                 11B
PCPD2              60.00 usec
PLW2         75.00000000 W
PLW12         2.08330011 W
SFO2        160.4615690 MHz

F2 - Processing parameters
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SF          500.1300102 MHz
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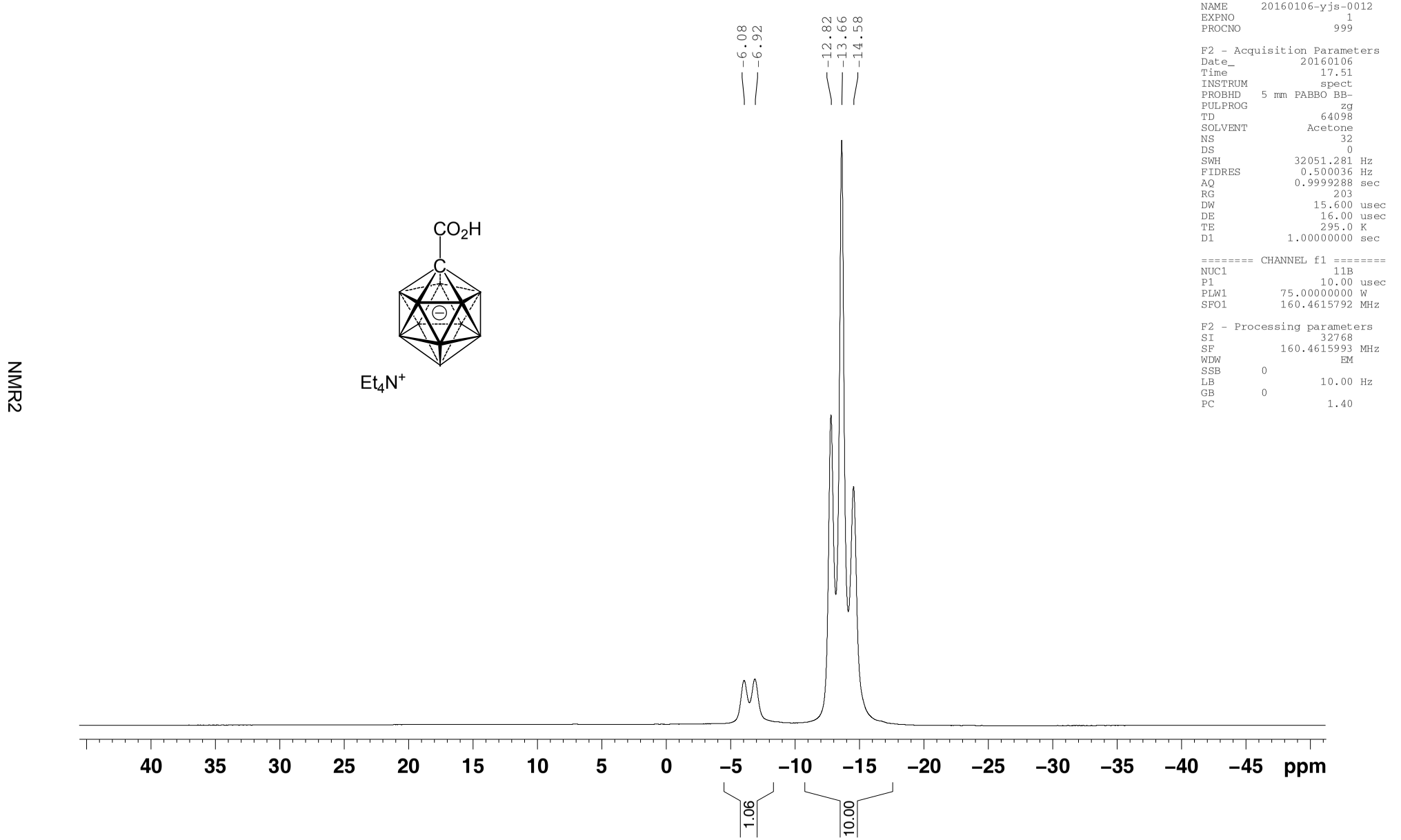
11B NMR, 30 mg in 0.6 mL acetone–d6 T = 23 C

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EXPNO                     1
PROCNO                  999

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PROBHD   5 mm PABBO BB-
PULPROG                zg
TD                  64098
SOLVENT           Acetone
NS                     32
DS                      0
SWH             32051.281 Hz
FIDRES           0.500036 Hz
AQ              0.9999288 sec
RG                    203
DW                 15.600 usec
DE                 16.00 usec
TE                 295.0 K
D1            1.00000000 sec

======== CHANNEL f1 ========
NUC1                  11B
P1                 10.00 usec
PLW1         75.00000000 W
SFO1         160.4615792 MHz

F2 - Processing parameters
SI                    32768
SF           160.4615993 MHz
WDW                     EM
SSB       0
LB                 10.00 Hz
GB        0
PC                  1.40
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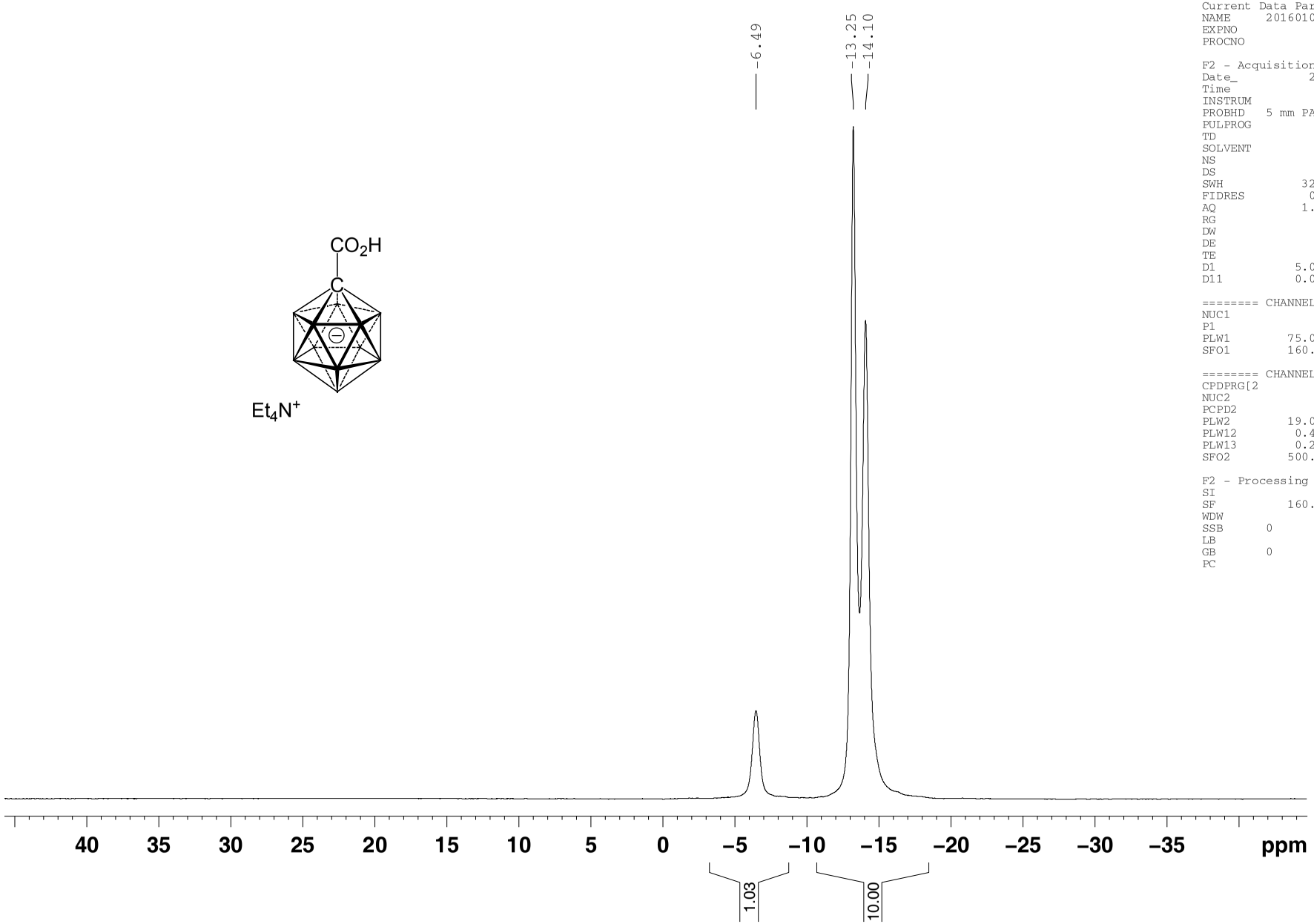
11B{1H} NMR, 30 mg in 0.6 mL acetone-d6 T = 23 C



NMR3

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EXPNO                     2
PROCNO                  999

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INSTRUM               spect
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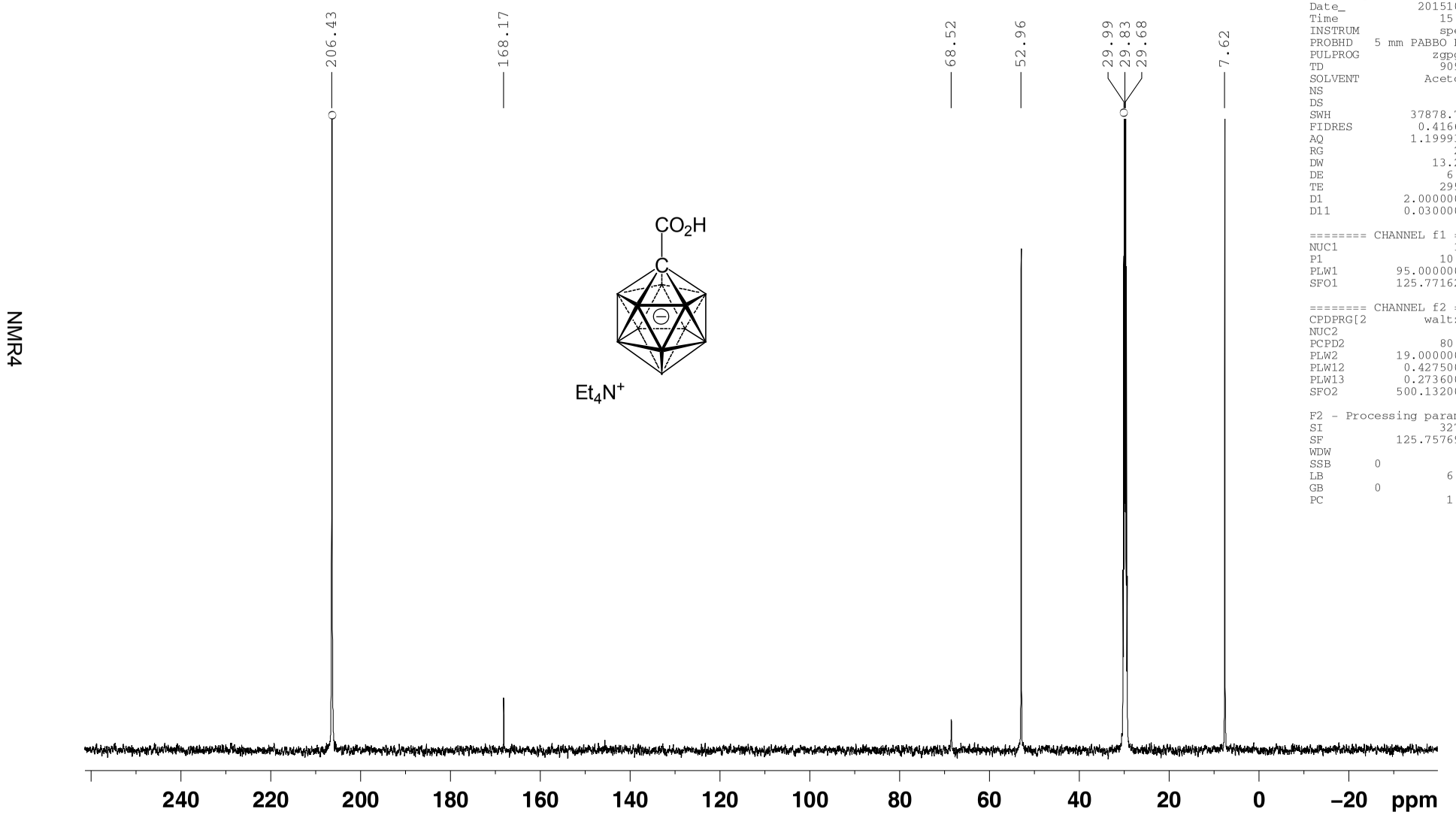
PULPROG             zgpg30
TD                   65536
SOLVENT            Acetone
NS                        32
DS                         0
SWH              32051.281 Hz
FIDRES            0.489064 Hz
AQ               1.0223616 sec
RG                      203
DW                 15.600 usec
DE                  6.50 usec
TE                  295.4 K
D1              5.00000000 sec
D11             0.03000000 sec

======== CHANNEL f1 ========
NUC1                    11B
P1                  10.00 usec
PLW1          75.00000000 W
SFO1         160.4615792 MHz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                    1H
PCPD2               80.00 usec
PLW2          19.00000000 W
PLW12          0.42750001 W
PLW13          0.27360001 W
SFO2         500.1330885 MHz

F2 - Processing parameters
SI                      32768
SF           160.4615993 MHz
WDW                        EM
SSB       0
LB                  10.00 Hz
GB        0
PC                    1.40
```

13C{1H} NMR, 30 mg in 0.6 mL acetone-d6 T = 23 C
Solvent peak ○



NMR4

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EXPNO                    4
PROCNO                   1

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Time              15.40
INSTRUM           spect
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PULPROG          zgpg30
TD                90904
SOLVENT         Acetone
NS                   72
DS                    0
SWH           37878.789 Hz
FIDRES         0.416690 Hz
AQ             1.1999328 sec
RG                  203
DW               13.200 usec
DE                6.50 usec
TE                295.5 K
D1           2.00000000 sec
D11          0.03000000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                10.00 usec
PLW1        95.00000000 W
SFO1        125.7716224 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2             80.00 usec
PLW2        19.00000000 W
PLW12        0.42750001 W
PLW13        0.27360001 W
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                32768
SF          125.7576917 MHz
WDW                EM
SSB       0
LB                6.00 Hz
GB        0
PC                1.40
```
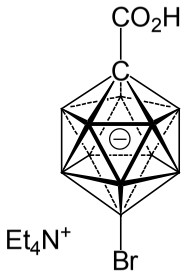

1H{11B} NMR, 20 mg in 0.6 mL acetone-d6 T = 23 C
Solvent residual peak= 2.05 ppm



NMR5


```
Current Data Parameters
NAME      20151014-yjs-0024-1H-13C
EXPNO                 11
PROCNO                 1

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Date_           20151014
Time               16.00
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgig30
TD                84584
SOLVENT         Acetone
NS                    8
DS                    0
SWH           14097.744 Hz
FIDRES         0.166672 Hz
AQ             2.9999125 sec
RG                 20.2
DW               35.467 usec
DE                6.50 usec
TE                296.0 K
D1           5.00000000 sec
d11          0.03000000 sec
TD0                   1
SFO1        500.1310003 MHz
NUC1                1H
P1                12.00 usec
PLW1        19.00000000 W
SFO2        160.4615690 MHz
NUC2               11B
CPDPRG[2          garp
PCPD2            60.00 usec
PLW2        75.00000000 W
PLW12        2.08330011 W

F2 - Processing parameters
SI                65536
SF          500.1300102 MHz
WDW                  no
SSB       0
LB        0 Hz
GB        0
PC                1.00
```
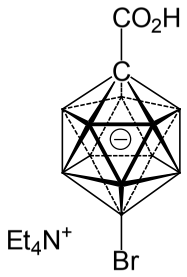
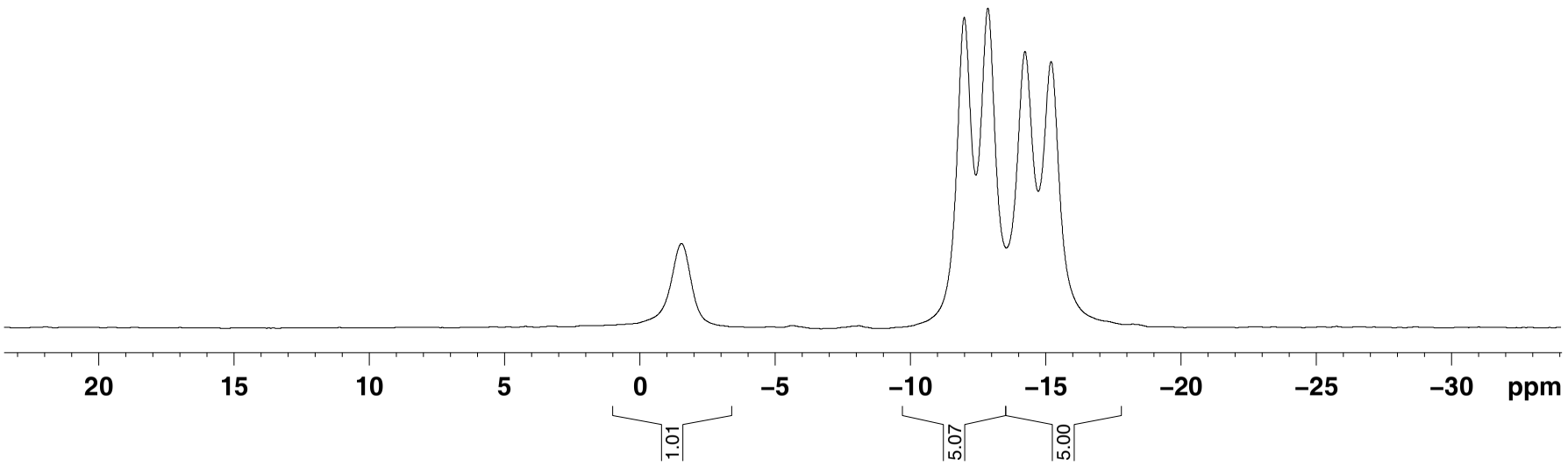

11B NMR, 12-Br ,1-COOH- CB11, 15 mg in 0.6 mL acetone-d6,19 C

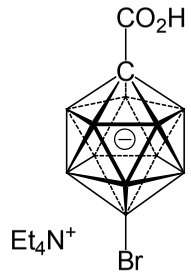




NMR6

```
Current Data Parameters
NAME      20151201-yjs-0024
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20151201
Time              17.06
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG             zg
TD                64098
SOLVENT         Acetone
NS                   32
DS                    0
SWH           32051.281 Hz
FIDRES         0.500036 Hz
AQ            0.9999288 sec
RG                  203
DW               15.600 usec
DE               16.00 usec
TE               295.9 K
D1            1.00000000 sec

======== CHANNEL f1 ========
NUC1                11B
P1               10.00 usec
PLW1        75.00000000 W
SFO1        160.4615792 MHz

F2 - Processing parameters
SI                32768
SF          160.4615993 MHz
WDW                EM
SSB       0
LB               40.00 Hz
GB        0
PC                1.40
```

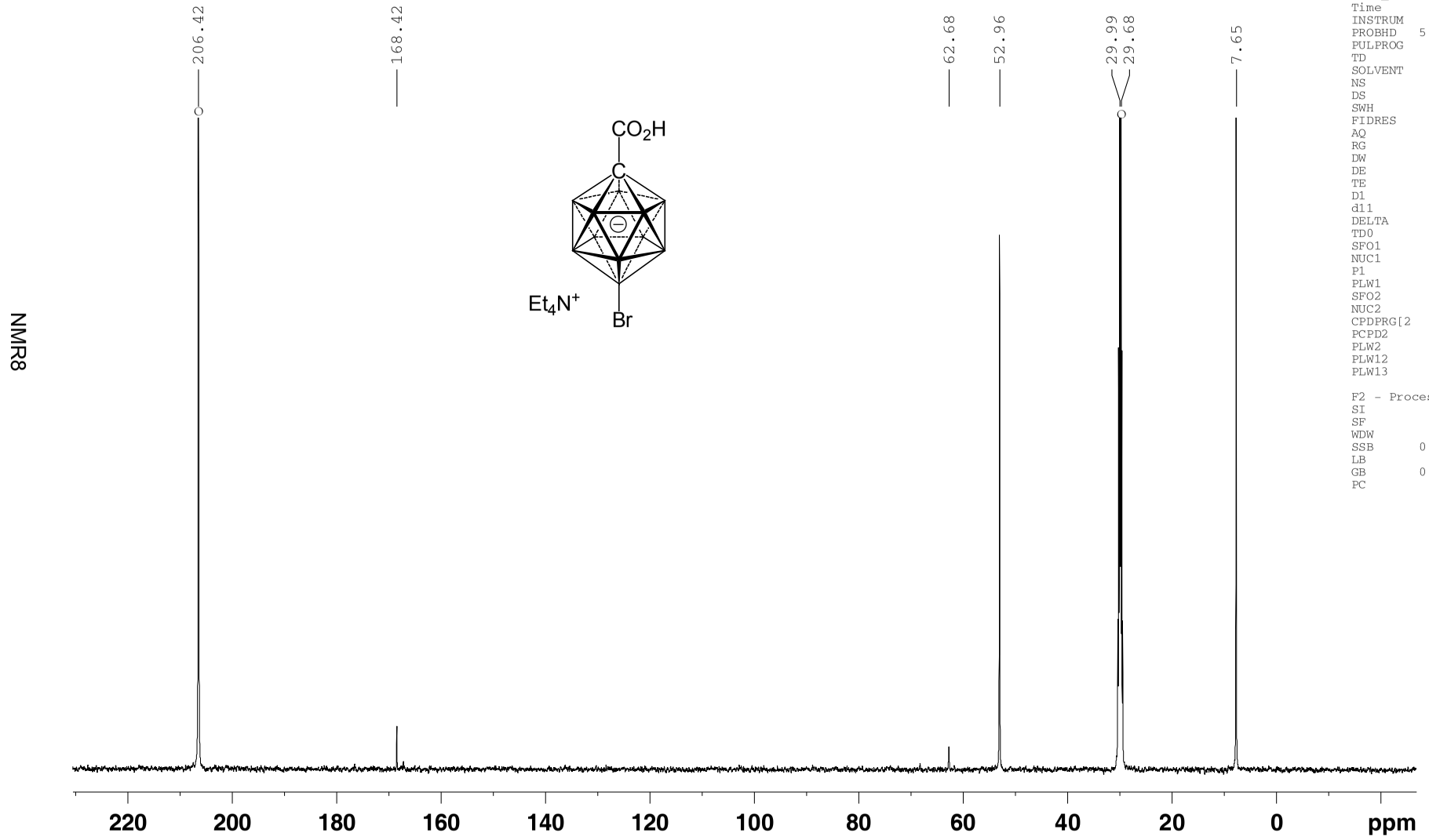
11B{1H} NMR, 12-Br ,1-COOH- CB11, 15 mg in 0.6 mL acetone-d6,19 C



NMR7

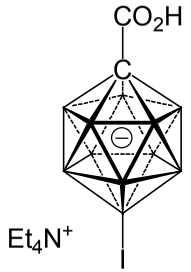

```
Current Data Parameters
NAME      20151201-yjs-0024
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_           20151201
Time               17.09
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                   32
DS                    0
SWH           32051.281 Hz
FIDRES         0.489064 Hz
AQ            1.0223616 sec
RG                  203
DW               15.600 usec
DE                6.50 usec
TE               296.0 K
D1           2.00000000 sec
D11          0.03000000 sec

======== CHANNEL f1 ========
NUC1                11B
P1                10.00 usec
PLW1        75.00000000 W
SFO1       160.4615792 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2             80.00 usec
PLW2        19.00000000 W
PLW12        0.42750001 W
PLW13        0.27360001 W
SFO2       500.1330885 MHz

F2 - Processing parameters
SI                 32768
SF          160.4615993 MHz
WDW                   EM
SSB       0
LB                40.00 Hz
GB        0
PC                 1.40
```

13C{1H} NMR, 20 mg in 0.6 mL acetone-d6 T = 23 C
Solvent peak ○




```
Current Data Parameters
NAME      20151014-yjs-0024-1H-13C
EXPNO                            4
PROCNO                           1

F2 - Acquisition Parameters
Date_                     20151014
Time                         15.45
INSTRUM                      spect
PROBHD    5 mm PABBO BB-
PULPROG                   zgpg30
TD                           90904
SOLVENT                    Acetone
NS                             204
DS                               0
SWH                      37878.789 Hz
FIDRES                    0.416690 Hz
AQ                        1.1999328 sec
RG                             203
DW                          13.200 usec
DE                           6.50 usec
TE                          296.1 K
D1                     2.00000000 sec
d11                    0.03000000 sec
DELTA                  1.89999998 sec
TD0                              1
SFO1                 125.7716224 MHz
NUC1                          13C
P1                          10.00 usec
PLW1                  95.00000000 W
SFO2                 500.1320005 MHz
NUC2                          1H
CPDPRG[2                  waltz16
PCPD2                       80.00 usec
PLW2                  19.00000000 W
PLW12                  0.42750001 W
PLW13                  0.27360001 W

F2 - Processing parameters
SI                           32768
SF                   125.7576949 MHz
WDW                             EM
SSB                 0
LB                           6.00 Hz
GB                  0
PC                           1.40
```

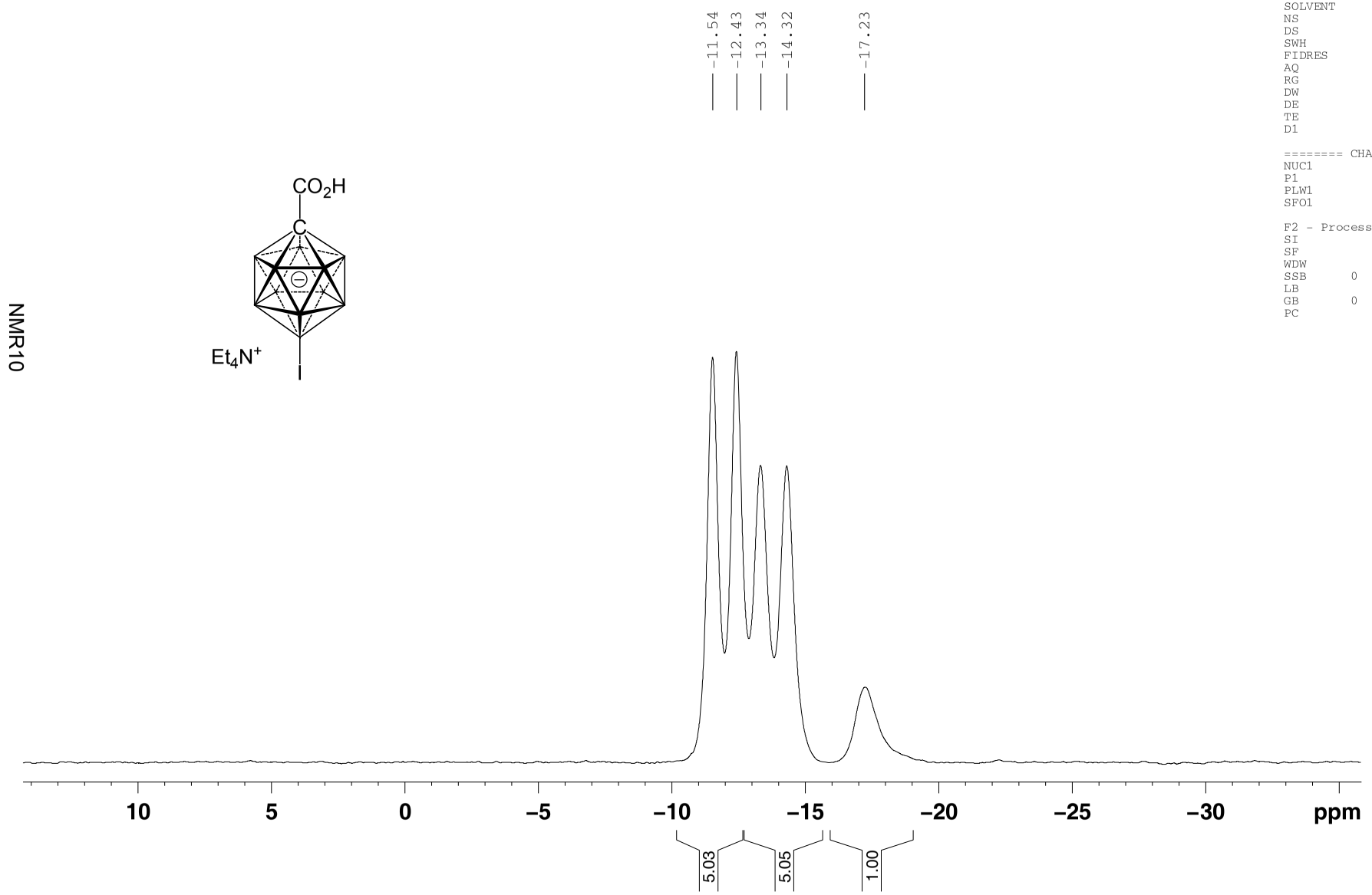
1H{11B} NMR, 1–carboxylic acid,12–I CB11, 15 mg in 0.6 mL acetonitrile–d3, T = 23 C
Solvent residual peak ○



NMR9

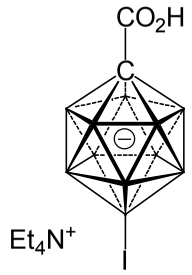

```
Current Data Parameters
NAME      20160726-yjs-0156
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20160726
Time                 20.58
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgig30
TD                   65536
SOLVENT              CD3CN
NS                      16
DS                       0
SWH           12500.000 Hz
FIDRES            0.190735 Hz
AQ                2.6214399 sec
RG                     114
DW                  40.000 usec
DE                   6.50 usec
TE                  298.5 K
D1             5.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                   11.60 usec
PLW1          19.00000000 W
SFO1         500.1335009 MHz

======== CHANNEL f2 ========
CPDPRG[2               garp
NUC2                   11B
PCPD2              100.00 usec
PLW2          95.00000000 W
PLW12          1.63030005 W
SFO2         160.4615690 MHz

F2 - Processing parameters
SI                   65536
SF           500.1300180 MHz
WDW                     no
SSB       0
LB        0 Hz
GB        0
PC                    1.00
```

11B NMR, 1–carboxylic acid,12–I CB11, 15 mg in 0.6 mL acetonitrile–d3, T = 23 C




```
Current Data Parameters
NAME      20160729-yjs-0156
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_           20160729
Time                2.44
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 64098
SOLVENT            CD3CN
NS                    64
DS                     0
SWH            32051.281 Hz
FIDRES          0.500036 Hz
AQ             0.9999288 sec
RG                   203
DW                15.600 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                 11B
P1                 13.10 usec
PLW1         95.00000000 W
SFO1         160.4615792 MHz

F2 - Processing parameters
SI                 32768
SF          160.4615790 MHz
WDW                   EM
SSB       0
LB                 20.00 Hz
GB        0
PC                  1.40
```

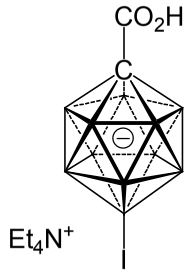
11B{1H} NMR, 1–carboxylic acid,12–I CB11, 15 mg in 0.6 mL acetonitrile–d3, T = 23 C



NMR11


```
Current Data Parameters
NAME      20160729-yjs-0156
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_             20160728
Time                 23.51
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CD3CN
NS                     64
DS                      0
SWH            32051.281 Hz
FIDRES          0.489064 Hz
AQ             1.0223616 sec
RG                    203
DW                15.600 usec
DE                 6.50 usec
TE                298.9 K
D1           1.00000000 sec
D11          0.03000000 sec

======== CHANNEL f1 ========
NUC1                 11B
P1                 13.10 usec
PLW1         95.00000000 W
SFO1        160.4615790 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PLW2         19.00000000 W
PLW12         0.39947000 W
PLW13         0.25566000 W
SFO2        500.1325007 MHz

F2 - Processing parameters
SI                   32768
SF          160.4615790 MHz
WDW                     EM
SSB       0
LB                 20.00 Hz
GB        0
PC                  1.40
```

13C{1H} NMR, 1–carboxylic acid,12–I CB11, 15 mg in 0.6 mL acetonitrile–d3, T = 23 C
Solvent peak ○

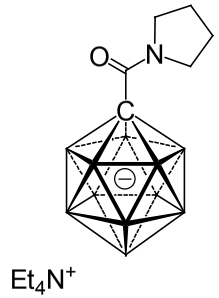


NMR12

```
Current Data Parameters
NAME      20160729-yjs-0156
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_              20160728
Time                 23.55
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CD3CN
NS                   4096
DS                      4
SWH           37878.789 Hz
FIDRES         0.577984 Hz
AQ             0.8650752 sec
RG                    203
DW                13.200 usec
DE                 6.50 usec
TE                299.0 K
D1           1.50000000 sec
D11          0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                10.50 usec
PLW1         95.00000000 W
SFO1        125.7716224 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PLW2         19.00000000 W
PLW12         0.39947000 W
PLW13         0.25566000 W
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                   32768
SF          125.7576704 MHz
WDW                     EM
SSB       0
LB                10.00 Hz
GB        0
PC                 1.40
```

1H{11B} NMR, pyrrolidine amide, 19 mg dissolved in 0.6 mL DMSO-d6, quartz NMR tube,T = 80 C
Solvent residual peak= 2.5 ppm ○

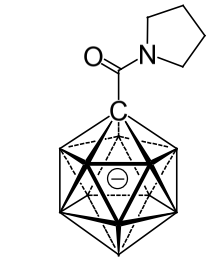
NMR13



Et_4N^+


```
Current Data Parameters
NAME      20141211-yjs-0045
EXPNO                  201
PROCNO                   1

F2 - Acquisition Parameters
Date_              20141211
Time                 16.54
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG             zgig30
TD                   84584
SOLVENT               DMSO
NS                       8
DS                       0
SWH              14097.744 Hz
FIDRES           0.166672 Hz
AQ               2.9999125 sec
RG                      57
DW                 35.467 usec
DE                  6.50 usec
TE                 352.2 K
D1              5.00000000 sec
d11             0.03000000 sec
TD0                      1
SFO1           500.1310003 MHz
NUC1                    1H
P1                   12.00 usec
PLW1           19.00000000 W
SFO2           160.4615690 MHz
NUC2                   11B
CPDPRG[2              garp
PCPD2                60.00 usec
PLW2           75.00000000 W
PLW12           2.08330011 W

F2 - Processing parameters
SI                    65536
SF             500.1300056 MHz
WDW                      no
SSB         0
LB          0 Hz
GB          0
PC                    1.00
```

11B NMR, pyrrolidine amide, 19 mg dissolved in 0.6 mL DMSO–d6, quartz NMR tube,T = 80 C

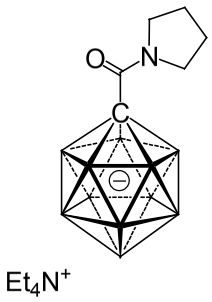


Et_4N^+

NMR14


```
Current Data Parameters
NAME      20141211-yjs-0045
EXPNO                  202
PROCNO                   1

F2 - Acquisition Parameters
Date_             20141211
Time                 16.56
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG                 zg
TD                   64098
SOLVENT               DMSO
NS                      32
DS                       0
SWH              32051.281 Hz
FIDRES            0.500036 Hz
AQ                0.9999288 sec
RG                     203
DW                  15.600 usec
DE                  16.00 usec
TE                  352.0 K
D1             1.00000000 sec
TD0                      1
SFO1           160.4615792 MHz
NUC1                   11B
P1                  10.00 usec
PLW1           75.00000000 W

F2 - Processing parameters
SI                   32768
SF           160.4615790 MHz
WDW                     EM
SSB       0
LB                  20.00 Hz
GB        0
PC                   1.40
```

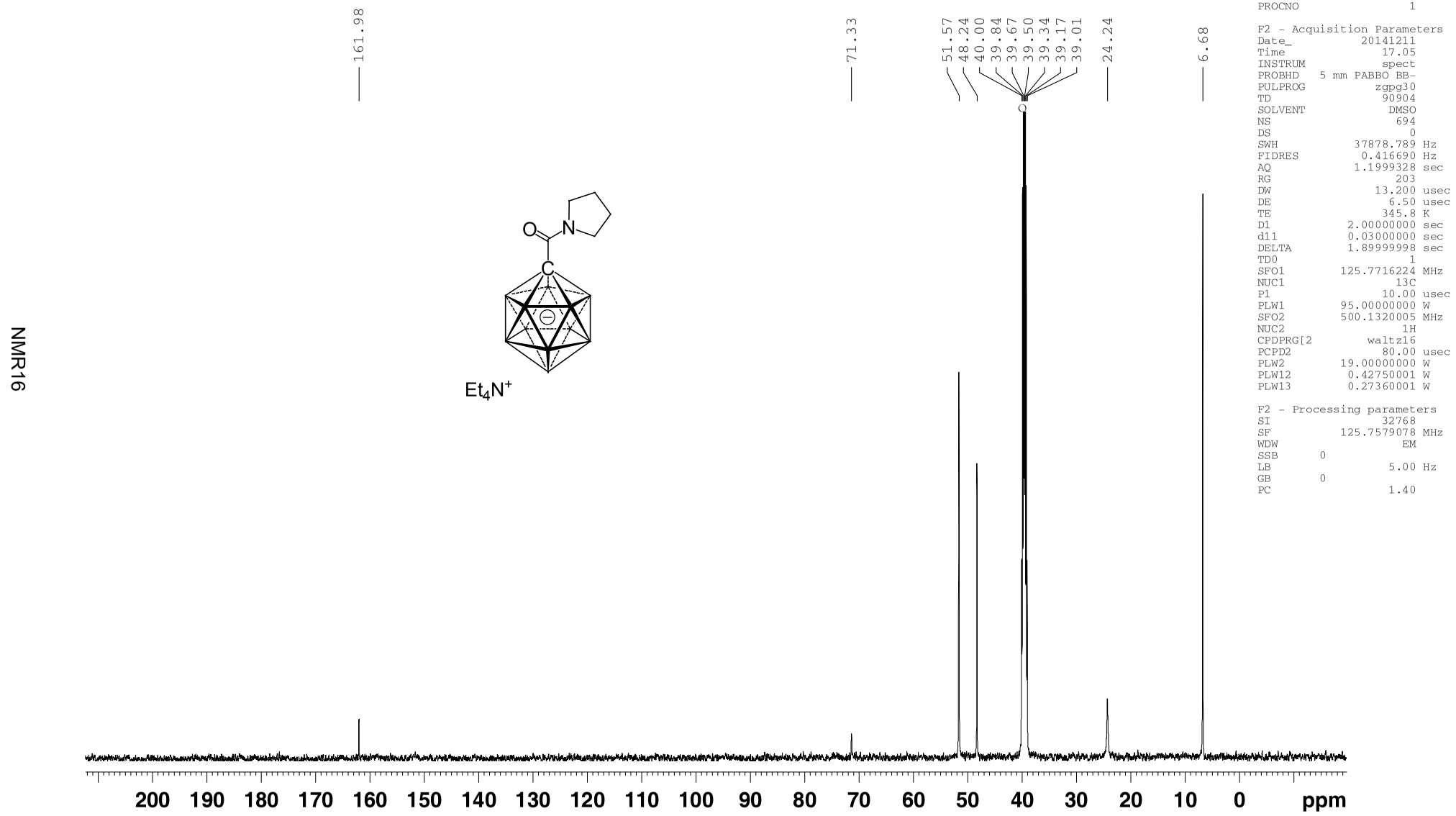
11B{1H} NMR, pyrrolidine amide, 19 mg dissolved in 0.6 mL DMSO–d6, quartz NMR tube,T = 80 C



NMR15

```
Current Data Parameters
NAME      20141211-yjs-0045
EXPNO                  203
PROCNO                   1

F2 - Acquisition Parameters
Date_             20141211
Time                 16.57
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                  192304
SOLVENT                DMSO
NS                       18
DS                        0
SWH               32051.281 Hz
FIDRES             0.166670 Hz
AQ                 2.9999423 sec
RG                       203
DW                    15.600 usec
DE                     6.50 usec
TE                    352.2 K
D1              5.00000000 sec
d11             0.03000000 sec
DELTA           4.90000010 sec
TD0                      1
SFO1            160.4615792 MHz
NUC1                    11B
P1                    10.00 usec
PLW1           75.00000000 W
SFO2           500.1330885 MHz
NUC2                    1H
CPDPRG[2            waltz16
PCPD2                 80.00 usec
PLW2           19.00000000 W
PLW12           0.42750001 W
PLW13           0.27360001 W

F2 - Processing parameters
SI                    32768
SF              160.4615790 MHz
WDW                      EM
SSB          0
LB                    20.00 Hz
GB           0
PC                     1.40
```

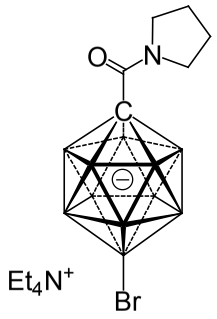
13C{1H} NMR, pyrrolidine amide, 19 mg dissolved in 0.6 mL DMSO-d6, quartz NMR tube,T = 80 C
Solvent peak



NMR16

```
Current Data Parameters
NAME      20141211-yjs-0045
EXPNO                  204
PROCNO                   1

F2 - Acquisition Parameters
Date_           20141211
Time               17.05
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                90904
SOLVENT            DMSO
NS                  694
DS                    0
SWH           37878.789 Hz
FIDRES        0.416690 Hz
AQ            1.1999328 sec
RG                  203
DW               13.200 usec
DE                6.50 usec
TE                345.8 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1
SFO1        125.7716224 MHz
NUC1                13C
P1               10.00 usec
PLW1        95.00000000 W
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2            80.00 usec
PLW2        19.00000000 W
PLW12        0.42750001 W
PLW13        0.27360001 W

F2 - Processing parameters
SI                32768
SF          125.7579078 MHz
WDW                EM
SSB       0
LB               5.00 Hz
GB        0
PC               1.40
```

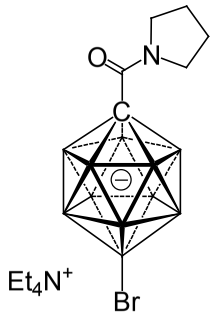
1H{11B} NMR, 12–Br prollidine amide 15 mg in 0.6 mL DMSO–d6 T = 80 C
Solvent residual peak ○



NMR17

```
Current Data Parameters
NAME      20150929-yjs-0117
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20150929
Time                  19.34
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG              zgig30
TD                    65536
SOLVENT                DMSO
NS                       16
DS                        0
SWH              10000.000 Hz
FIDRES           0.152588 Hz
AQ               3.2767999 sec
RG                     40.3
DW                  50.000 usec
DE                   6.50 usec
TE                  352.0 K
D1            10.00000000 sec
d11            0.03000000 sec
TD0                       1
SFO1          500.1325200 MHz
NUC1                    1H
P1                  12.00 usec
PLW1          19.00000000 W
SFO2          160.4615690 MHz
NUC2                   11B
CPDPRG[2               garp
PCPD2               60.00 usec
PLW2          75.00000000 W
PLW12          2.08330011 W

F2 - Processing parameters
SI                    65536
SF            500.1300051 MHz
WDW                      no
SSB       0
LB        0 Hz
GB        0
PC                    1.00
```

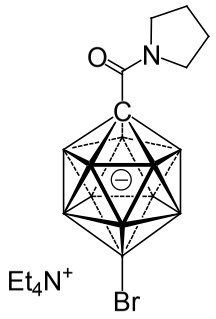
11B NMR, 12–Br prollidine amide 13 mg in 0.6 mL acetone–d6 T = 19 C




```
Current Data Parameters
NAME      20151130-yjs-0117
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_            20151130
Time                16.57
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG                zg
TD                  64098
SOLVENT           Acetone
NS                     32
DS                      0
SWH             32051.281 Hz
FIDRES          0.500036 Hz
AQ              0.9999288 sec
RG                    203
DW                15.600 usec
DE                16.00 usec
TE                294.4 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                  11B
P1                 10.00 usec
PLW1         75.00000000 W
SFO1         160.4615792 MHz

F2 - Processing parameters
SI                    32768
SF           160.4615993 MHz
WDW                      EM
SSB       0
LB                 30.00 Hz
GB        0
PC                   1.40
```

NMR18

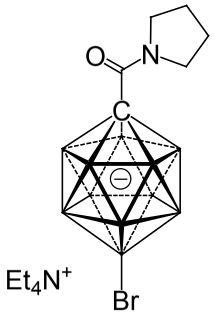
11B{1H} NMR, 12–Br prollidine amide 13 mg in 0.6 mL acetone–d6 T = 19 C



NMR19

```
Current Data Parameters
NAME      20151130-yjs-0117
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_            20151130
Time                17.02
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT           Acetone
NS                     17
DS                      0
SWH             32051.281 Hz
FIDRES          0.489064 Hz
AQ              1.0223616 sec
RG                    203
DW                15.600 usec
DE                 6.50 usec
TE                294.9 K
D1           5.00000000 sec
D11          0.03000000 sec

======== CHANNEL f1 ========
NUC1                  11B
P1                 10.00 usec
PLW1         75.00000000 W
SFO1        160.4615792 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PLW2         19.00000000 W
PLW12         0.42750001 W
PLW13         0.27360001 W
SFO2        500.1330885 MHz

F2 - Processing parameters
SI                    32768
SF          160.4615993 MHz
WDW                      EM
SSB       0
LB                 40.00 Hz
GB        0
PC                  1.40
```

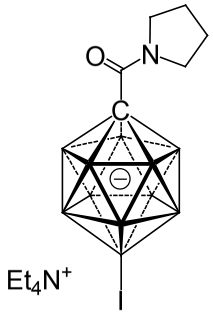
13C{1H} NMR, 12–Br prollidine amide 15 mg in 0.6 mL DMSO–d6 T = 80 C
Solvent peak ○



NMR20

```
Current Data Parameters
NAME      20150929-yjs-0117
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150929
Time              19.45
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD                65536
SOLVENT             DMSO
NS                  886
DS                    4
SWH           37878.789 Hz
FIDRES         0.577984 Hz
AQ            0.8650752 sec
RG                  203
DW               13.200 usec
DE                6.50 usec
TE                352.1 K
D1           1.50000000 sec
d11          0.03000000 sec
DELTA        1.39999998 sec
TD0                   1
SFO1        125.7716224 MHz
NUC1               13C
P1               10.00 usec
PLW1        95.00000000 W
SFO2        500.1320005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            80.00 usec
PLW2        19.00000000 W
PLW12        0.39262000 W
PLW13        0.25127000 W

F2 - Processing parameters
SI                32768
SF          125.7579052 MHz
WDW                EM
SSB       0
LB               7.00 Hz
GB        0
PC               1.40
```

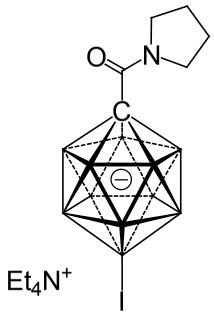
1H{11B} NMR, 12–I ,Pyrrolidine amdie,CB11, 20 mg in 0.6 mL acetonitrile–d3, T = 23 C
Solvent residual peak ○

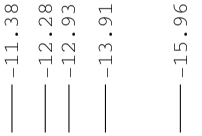


NMR21

```
Current Data Parameters
NAME      20160724-yjs-0157
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20160726
Time                  9.06
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG              zgig30
TD                    65536
SOLVENT               CD3CN
NS                       16
DS                        0
SWH               12500.000 Hz
FIDRES                0.190735 Hz
AQ                2.6214399 sec
RG                      114
DW                   40.000 usec
DE                    6.50 usec
TE                   298.5 K
D1             5.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                    11.60 usec
PLW1          19.00000000 W
SFO1          500.1335009 MHz

======== CHANNEL f2 ========
CPDPRG[2                garp
NUC2                     11B
PCPD2                100.00 usec
PLW2          95.00000000 W
PLW12          1.63030005 W
SFO2          160.4615690 MHz

F2 - Processing parameters
SI                    65536
SF            500.1300158 MHz
WDW                      no
SSB       0
LB        0 Hz
GB        0
PC                    1.00
```

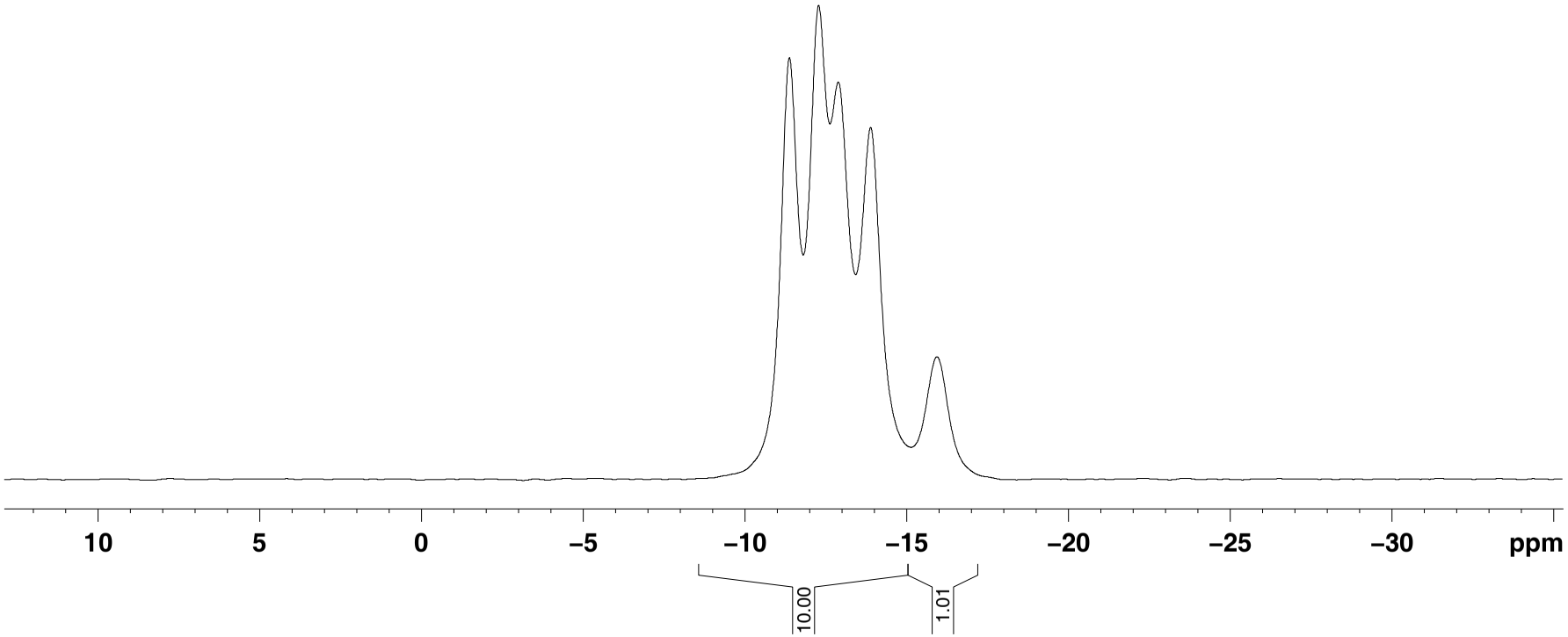
11B NMR, 12–I ,Pyrrolidine amdie,CB11, 20 mg in 0.6 mL acetonitrile–d3, T = 23 C



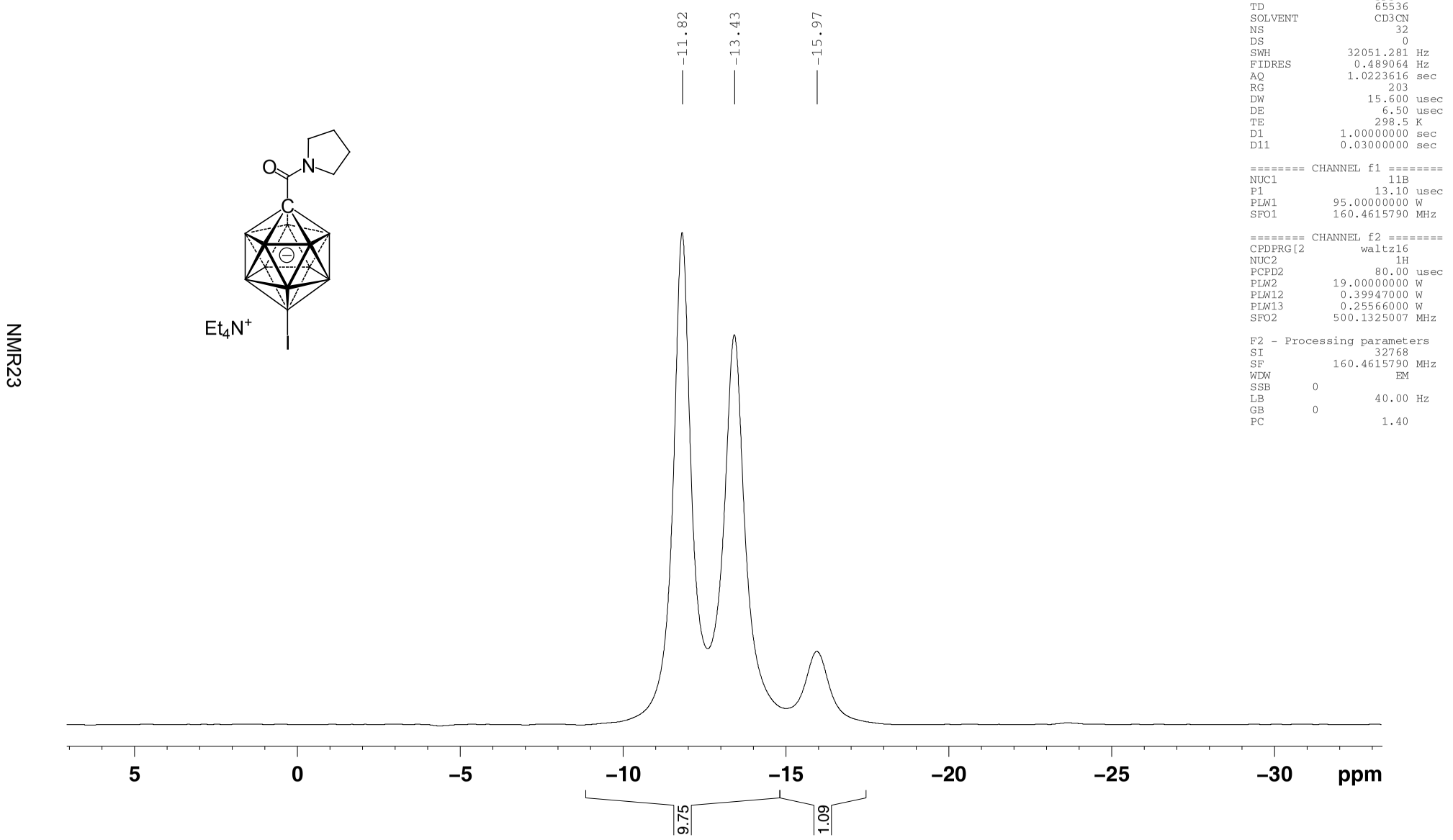



```
Current Data Parameters
NAME      20160724-yjs-0157
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20160726
Time                  9.08
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG               zg30
TD                   64098
SOLVENT              CD3CN
NS                      32
DS                       0
SWH              32051.281 Hz
FIDRES            0.500036 Hz
AQ                0.9999288 sec
RG                     203
DW                  15.600 usec
DE                   6.50 usec
TE                  298.2 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                   11B
P1                   13.10 usec
PLW1          95.00000000 W
SFO1         160.4615792 MHz

F2 - Processing parameters
SI                   32768
SF           160.4615790 MHz
WDW                     EM
SSB       0
LB                   40.00 Hz
GB        0
PC                    1.40
```

NMR22



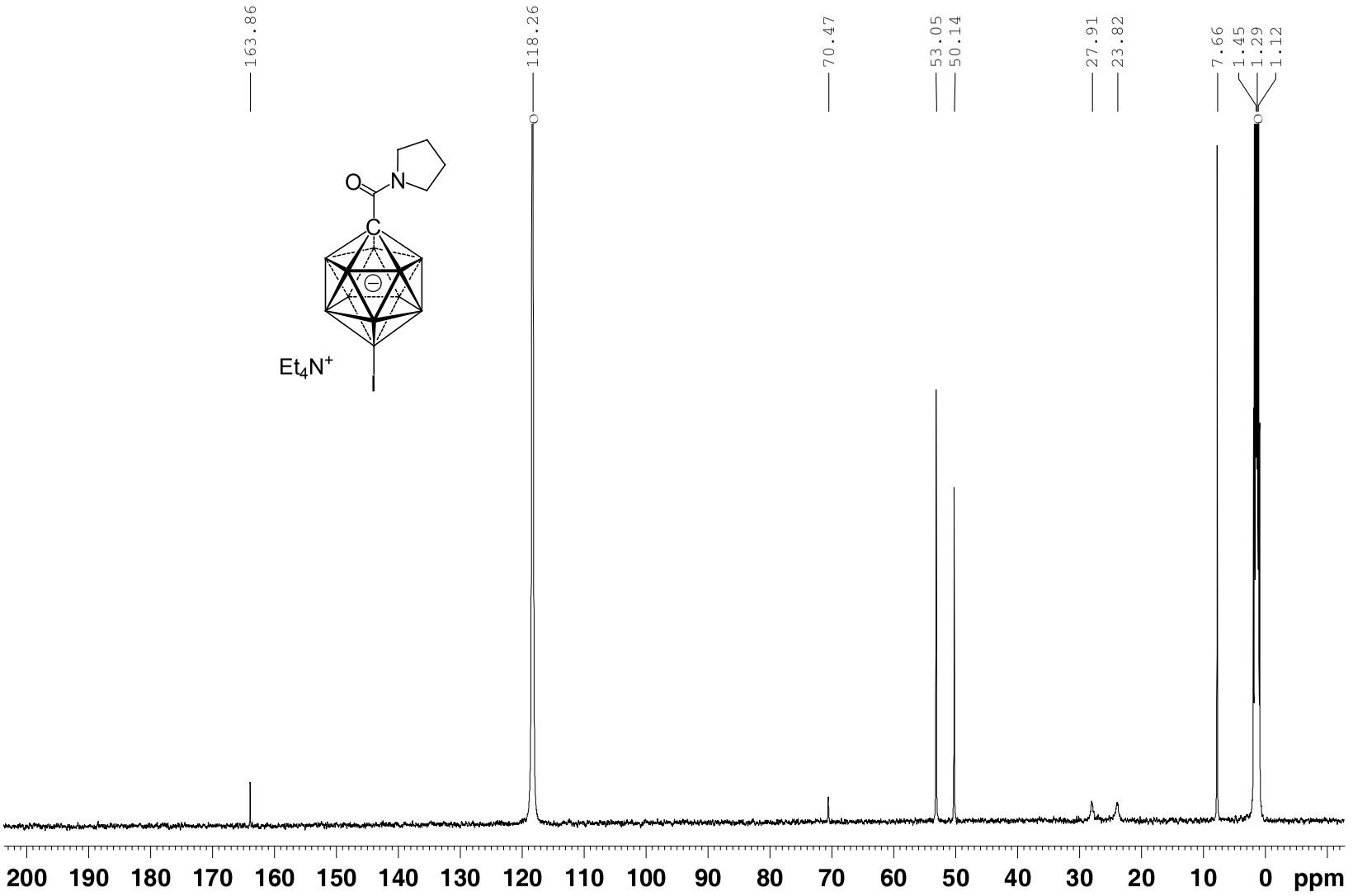
11B{1H} NMR, 12–I ,Pyrrolidine amdie,CB11, 20 mg in 0.6 mL acetonitrile–d3, T = 23 C



NMR23

```
Current Data Parameters
NAME      20160724-yjs-0157
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_              20160726
Time                   9.10
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT               CD3CN
NS                       32
DS                        0
SWH               32051.281 Hz
FIDRES             0.489064 Hz
AQ                1.0223616 sec
RG                      203
DW                   15.600 usec
DE                    6.50 usec
TE                   298.5 K
D1              1.00000000 sec
D11             0.03000000 sec

======== CHANNEL f1 ========
NUC1                    11B
P1                    13.10 usec
PLW1           95.00000000 W
SFO1          160.4615790 MHz

======== CHANNEL f2 ========
CPDPRG[2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PLW2           19.00000000 W
PLW12           0.39947000 W
PLW13           0.25566000 W
SFO2          500.1325007 MHz

F2 - Processing parameters
SI                      32768
SF            160.4615790 MHz
WDW                        EM
SSB       0
LB                    40.00 Hz
GB        0
PC                     1.40
```

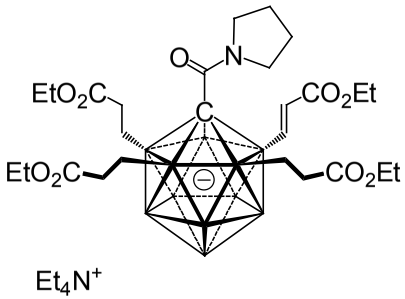
13C{1H} NMR, 12–I ,Pyrrolidine amdie,CB11, 20 mg in 0.6 mL acetonitrile–d3, T = 23 C
Solvent peak ○



NMR24

```
Current Data Parameters
NAME      20160724-yjs-0157
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_           20160726
Time              11.16
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CD3CN
NS                 3072
DS                    4
SWH           37878.789 Hz
FIDRES         0.577984 Hz
AQ             0.8650752 sec
RG                  203
DW               13.200 usec
DE                6.50 usec
TE                298.5 K
D1           1.50000000 sec
D11          0.03000000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                10.50 usec
PLW1         95.00000000 W
SFO1        125.7716224 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2             80.00 usec
PLW2         19.00000000 W
PLW12         0.39947000 W
PLW13         0.25566000 W
SFO2         500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF          125.7576708 MHz
WDW                   EM
SSB       0
LB                 5.00 Hz
GB        0
PC                 1.40
```

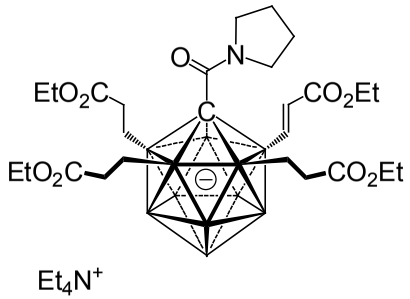
1H{11B} NMR, Ethyl ester CB11, 15 mg in 0.6 ml acetone-d6, T= 23 C
Solvent residual peak ○




```
Current Data Parameters
NAME      20161116-yjs-0103
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20161116
Time                 21.43
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgig30
TD                   65536
SOLVENT              CD3CN
NS                      16
DS                       0
SWH              12500.000 Hz
FIDRES               0.190735 Hz
AQ                2.6214399 sec
RG                     114
DW                  40.000 usec
DE                   6.50 usec
TE                  296.5 K
D1             5.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                   11.60 usec
PLW1          19.00000000 W
SFO1         500.1335009 MHz

======== CHANNEL f2 ========
CPDPRG[2                garp
NUC2                    11B
PCPD2                100.00 usec
PLW2          95.00000000 W
PLW12          1.63030005 W
SFO2         160.4615690 MHz

F2 - Processing parameters
SI                    65536
SF           500.1300158 MHz
WDW                      no
SSB       0
LB        0 Hz
GB        0
PC                    1.00
```

NMR25

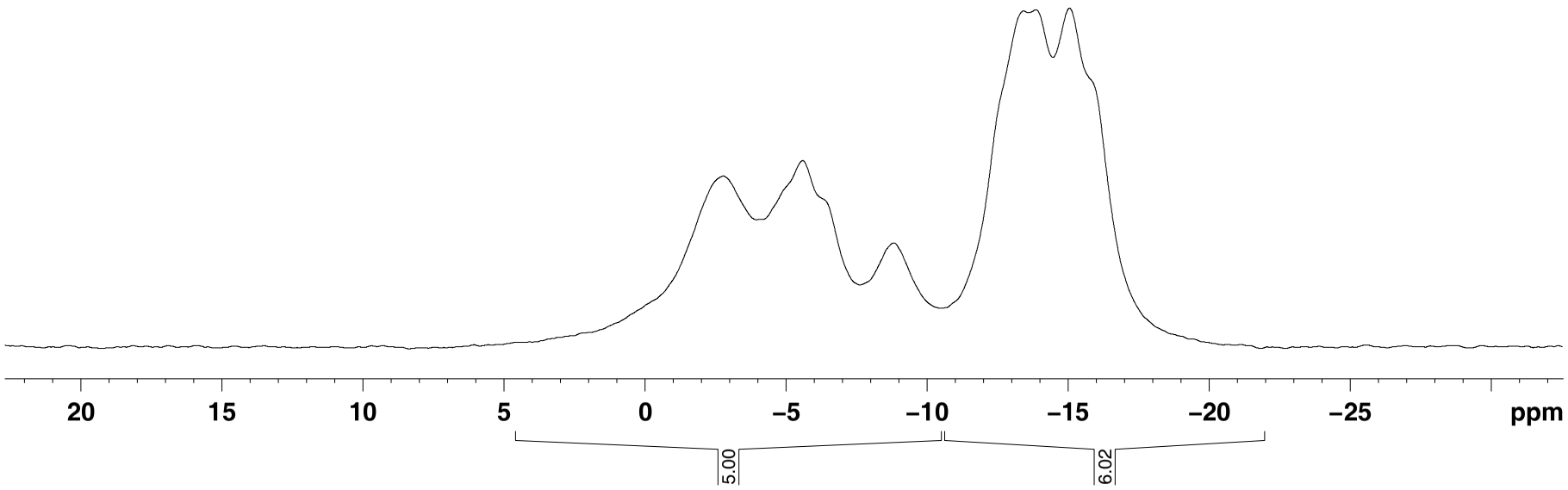
11B NMR,Ethyl ester CB11, 20 mg in 0.6 mL DMSO–d6, T = 80 C



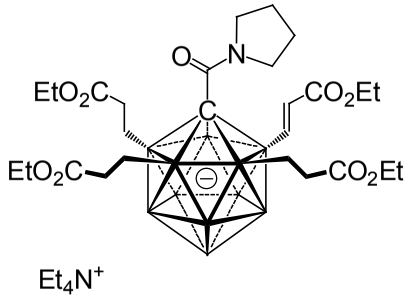
-2.67
-5.63
-8.74
-13.45
-15.00

```
Current Data Parameters
NAME      20150203-yjs-0103
EXPNO                   23
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150203
Time               22.17
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG              zg
TD                64098
SOLVENT            DMSO
NS                    32
DS                     0
SWH           32051.281 Hz
FIDRES         0.500036 Hz
AQ            0.9999288 sec
RG                   203
DW                15.600 usec
DE                16.00 usec
TE                352.0 K
D1           1.00000000 sec
TD0                    1
SFO1        160.4615792 MHz
NUC1                11B
P1                10.00 usec
PLW1        75.00000000 W

F2 - Processing parameters
SI                 32768
SF          160.4615993 MHz
WDW                   EM
SSB       0
LB                30.00 Hz
GB        0
PC                 1.40
```

NMR26



11B{1H} NMR,Ethyl ester CB11, 20 mg in 0.6 mL DMSO–d6, T = 80 C

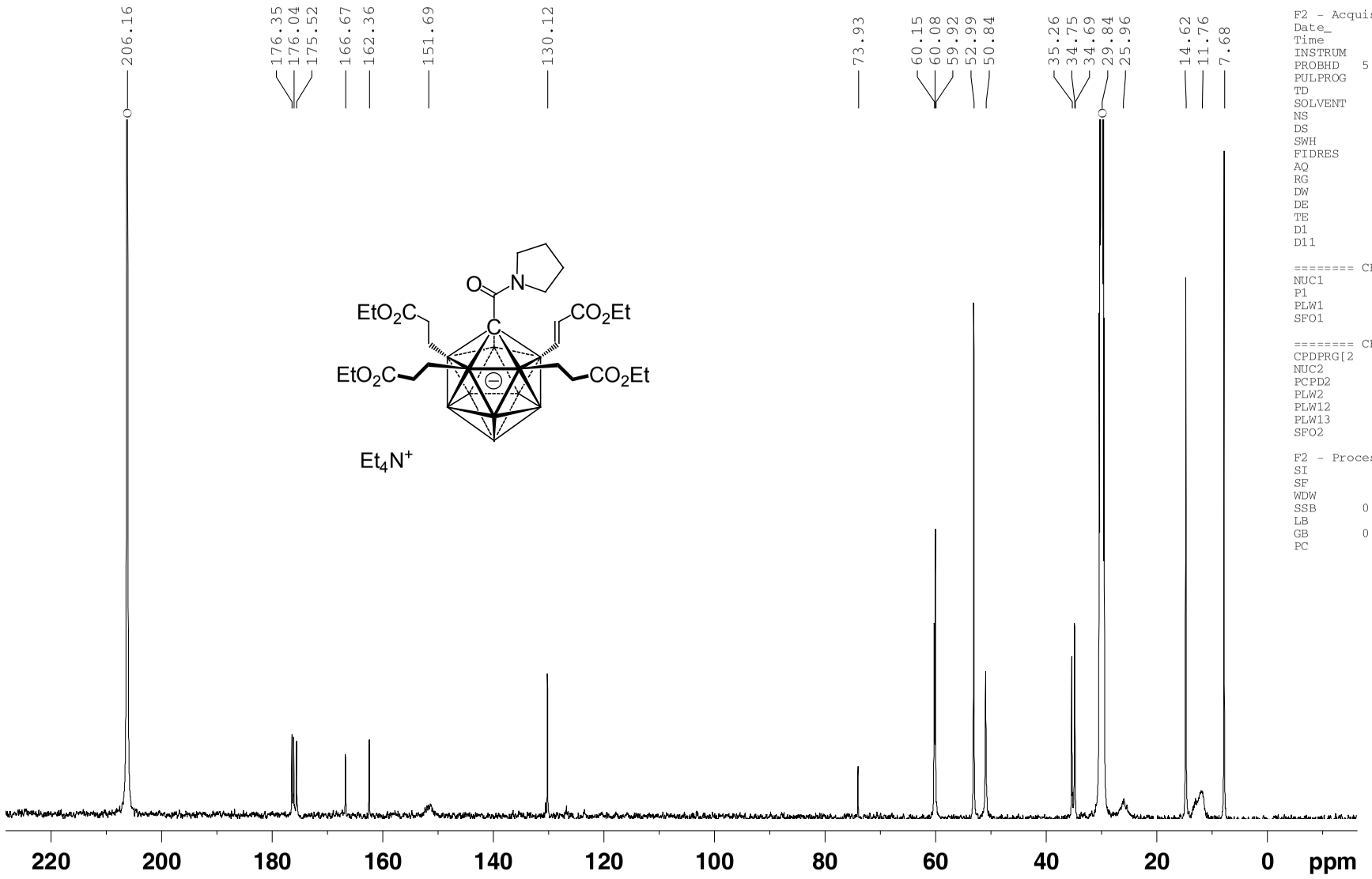


NMR27

```
Current Data Parameters
NAME      20150203-yjs-0103
EXPNO                   24
PROCNO                   1

F2 - Acquisition Parameters
Date_              20150203
Time                 22.21
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT                DMSO
NS                       32
DS                        0
SWH              32051.281 Hz
FIDRES            0.489064 Hz
AQ               1.0223616 sec
RG                     203
DW                15.600 usec
DE                 6.50 usec
TE                352.0 K
D1             5.00000000 sec
d11            0.03000000 sec
DELTA          4.90000010 sec
TD0                      1
SFO1         160.4615792 MHz
NUC1                  11B
P1                 10.00 usec
PLW1          75.00000000 W
SFO2         500.1330885 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2          19.00000000 W
PLW12          0.42750001 W
PLW13          0.27360001 W

F2 - Processing parameters
SI                   32768
SF           160.4615993 MHz
WDW                     EM
SSB       0
LB                 40.00 Hz
GB        0
PC                  1.40
```

13C{1H} NMR,Ethyl ester CB11, 30 mg in 0.6 mL acetone-d6 T = 19 C
Solvent peak ○

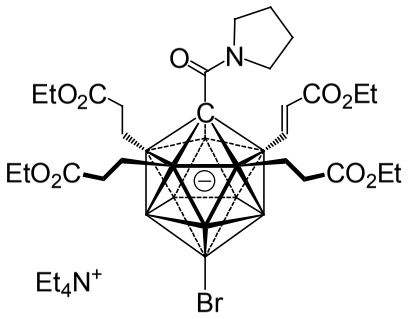
NMR28




```
Current Data Parameters
NAME      20151229-yjs-4Et-13C
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_              20151231
Time                   3.34
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT             Acetone
NS                     2048
DS                        4
SWH                37878.789 Hz
FIDRES             0.577984 Hz
AQ                 0.8650752 sec
RG                       203
DW                    13.200 usec
DE                     6.50 usec
TE                    296.1 K
D1              1.50000000 sec
D11             0.03000000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                   10.00 usec
PLW1           95.00000000 W
SFO1          125.7716224 MHz

======== CHANNEL f2 ========
CPDPRG[2              waltz16
NUC2                     1H
PCPD2                80.00 usec
PLW2           19.00000000 W
PLW12           0.39262000 W
PLW13           0.25127000 W
SFO2          500.1320005 MHz

F2 - Processing parameters
SI                       32768
SF            125.7576812 MHz
WDW                         EM
SSB          0
LB                     7.00 Hz
GB           0
PC                     1.40
```

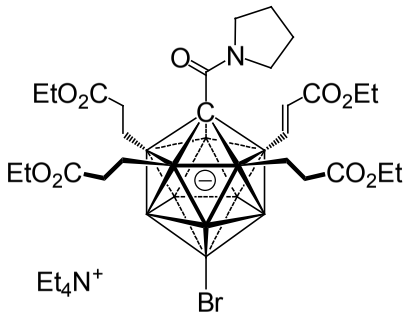
1H{11B} NMR, 12-Br,Pyrrolidine amide,tetraester CB11, 15 mg in 0.6 mLacetone-d6, T = 19 C
Solvent residual peak ○

NMR29




```
Current Data Parameters
NAME      20160414-yjs-0143
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20160414
Time                   3.22
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG              zgig30
TD                    65536
SOLVENT             Acetone
NS                       16
DS                        0
SWH               12500.000 Hz
FIDRES             0.190735 Hz
AQ                2.6214399 sec
RG                      114
DW                   40.000 usec
DE                    6.50 usec
TE                   298.8 K
D1              5.00000000 sec
D11             0.03000000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                    11.60 usec
PLW1           19.00000000 W
SFO1          500.1335009 MHz

======== CHANNEL f2 ========
CPDPRG[2                garp
NUC2                    11B
PCPD2                100.00 usec
PLW2           95.00000000 W
PLW12           1.63030005 W
SFO2          160.4615690 MHz

F2 - Processing parameters
SI                      65536
SF            500.1300100 MHz
WDW                        EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.00
```

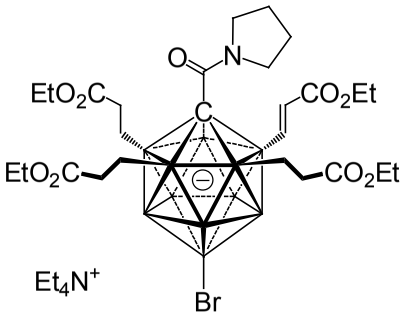
11B NMR, 12-Br,Pyrrolidine amide,tetraester, 15 mg in 0.6 mLacetone-d6, T = 19 C



NMR30

```
Current Data Parameters
NAME      20160414-yjs-0143
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_           20160414
Time                3.24
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 64098
SOLVENT          Acetone
NS                    32
DS                     0
SWH            32051.281 Hz
FIDRES          0.500036 Hz
AQ             0.9999288 sec
RG                   203
DW                15.600 usec
DE                 6.50 usec
TE                298.5 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                 11B
P1                 13.10 usec
PLW1         95.00000000 W
SFO1         160.4615792 MHz

F2 - Processing parameters
SI                 32768
SF          160.4615790 MHz
WDW                   EM
SSB       0
LB                150.00 Hz
GB        0
PC                  1.40
```

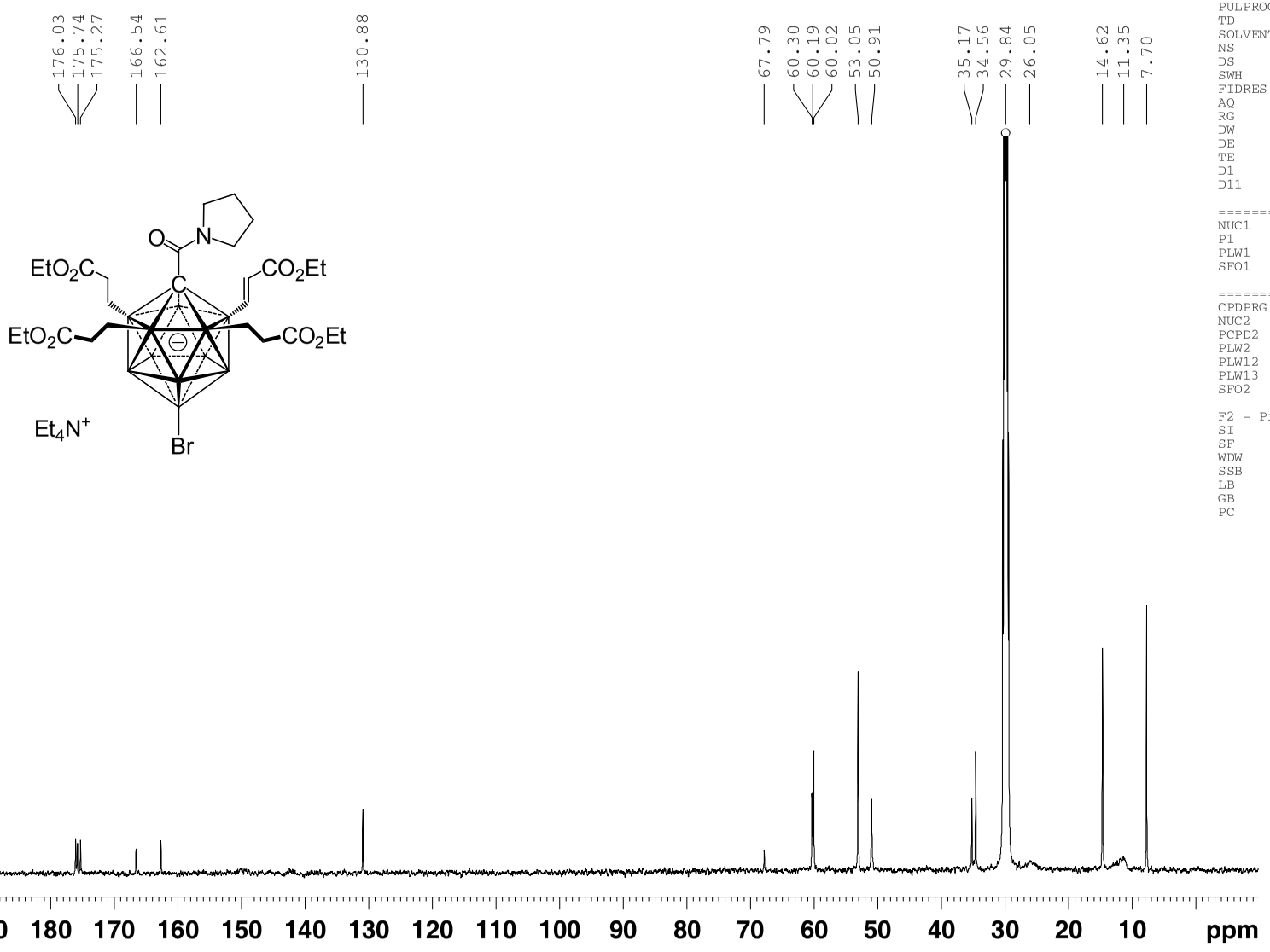
11B{1H} NMR, 12-Br,Pyrrolidine amide,tetraester, 15 mg in 0.6 mLacetone-d6, T = 19 C



NMR31


```
Current Data Parameters
NAME      20160414-yjs-0143
EXPNO                     3
PROCNO                    1

F2 - Acquisition Parameters
Date_              20160414
Time                   3.26
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT             Acetone
NS                       16
DS                        0
SWH              32051.281 Hz
FIDRES            0.489064 Hz
AQ               1.0223616 sec
RG                      203
DW                 15.600 usec
DE                  6.50 usec
TE                  298.6 K
D1              1.00000000 sec
D11             0.03000000 sec

======== CHANNEL f1 ========
NUC1                    11B
P1                  13.10 usec
PLW1           95.00000000 W
SFO1          160.4615790 MHz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                    1H
PCPD2               80.00 usec
PLW2           19.00000000 W
PLW12           0.39947000 W
PLW13           0.25566000 W
SFO2          500.1325007 MHz

F2 - Processing parameters
SI                   32768
SF            160.4615790 MHz
WDW                     EM
SSB       0
LB                150.00 Hz
GB        0
PC                   1.40
```

13C{1H} NMR, 12-Br,Pyrrolidine amide,tetraester, 15 mg in 0.6 mLacetone-d6, T = 19 C
Solvent peak

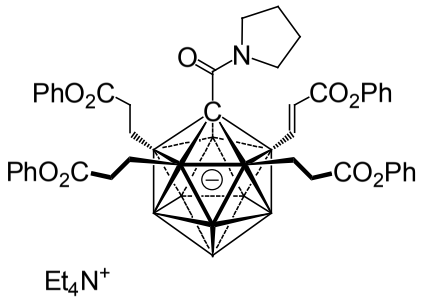


NMR32

```
Current Data Parameters
NAME      20160414-yjs-0143
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_              20160414
Time                   5.31
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT             Acetone
NS                     3072
DS                        4
SWH               37878.789 Hz
FIDRES             0.577984 Hz
AQ                 0.8650752 sec
RG                      203
DW                   13.200 usec
DE                    6.50 usec
TE                   299.2 K
D1              1.50000000 sec
D11             0.03000000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                   10.50 usec
PLW1           95.00000000 W
SFO1          125.7716224 MHz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                    1H
PCPD2                80.00 usec
PLW2           19.00000000 W
PLW12           0.39947000 W
PLW13           0.25566000 W
SFO2          500.1320005 MHz

F2 - Processing parameters
SI                      32768
SF            125.7576770 MHz
WDW                        EM
SSB       0
LB                    7.00 Hz
GB        0
PC                    1.40
```

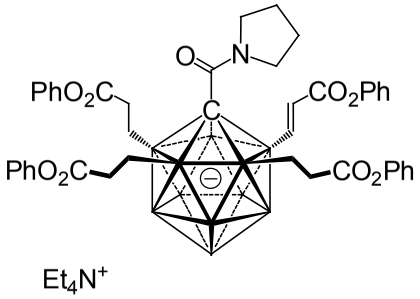
1H{11B} NMR, Phenyl ester CB11, 18 mg in 0.6 mL acetonitrile-d3, T = 19 C
Solvent residual peak ○

NMR33




```
Current Data Parameters
NAME      20151222-yjs-0150
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_            20151222
Time                18.08
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgig30
TD                 65536
SOLVENT            CD3CN
NS                    16
DS                     0
SWH           10000.000 Hz
FIDRES            0.152588 Hz
AQ             3.2767999 sec
RG                    64
DW                50.000 usec
DE                 6.50 usec
TE                296.4 K
D1           10.00000000 sec
D11           0.03000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                12.00 usec
PLW1         19.00000000 W
SFO1        500.1325200 MHz

======== CHANNEL f2 ========
CPDPRG[2             garp
NUC2                 11B
PCPD2             60.00 usec
PLW2         75.00000000 W
PLW12         2.08330011 W
SFO2        160.4615690 MHz

F2 - Processing parameters
SI                 65536
SF          500.1300156 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                  1.00
```

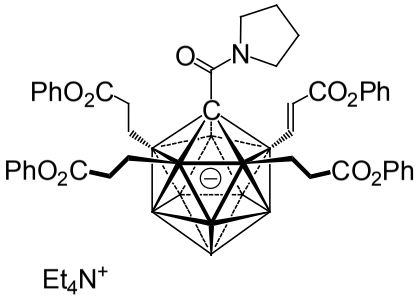
11B NMR, Phenyl ester CB11, 18 mg in 0.6 mL acetonitrile–d3, T = 19 C




```
Current Data Parameters
NAME      20151225-yjs-0150
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20151225
Time              20.31
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               64098
SOLVENT          CD3CN
NS                 256
DS                   0
SWH          32051.281 Hz
FIDRES        0.500036 Hz
AQ           0.9999288 sec
RG                 203
DW              15.600 usec
DE               6.50 usec
TE               295.9 K
D1          1.00000000 sec

======== CHANNEL f1 ========
NUC1               11B
P1              13.10 usec
PLW1        95.00000000 W
SFO1        160.4615792 MHz

F2 - Processing parameters
SI               32768
SF          160.4615790 MHz
WDW                EM
SSB       0
LB              40.00 Hz
GB        0
PC               1.40
```

NMR34

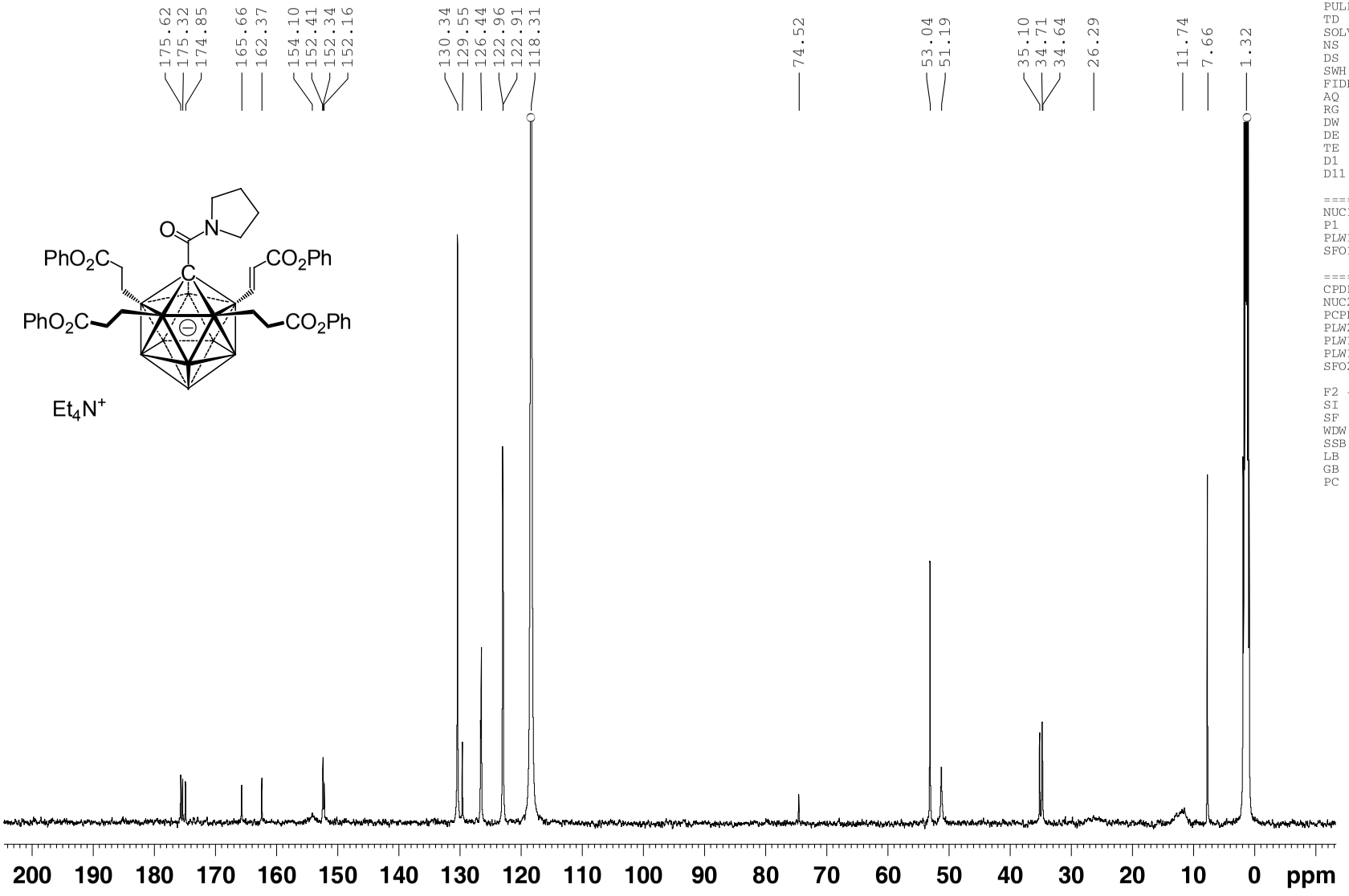
11B{1H} NMR, Phenyl ester CB11, 18 mg in 0.6 mL acetonitrile-d3, T = 19 C



NMR35

```
Current Data Parameters
NAME      20151225-yjs-0150
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_           20151225
Time              20.41
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CD3CN
NS                  256
DS                    0
SWH           32051.281 Hz
FIDRES         0.489064 Hz
AQ            1.0223616 sec
RG                  203
DW               15.600 usec
DE                6.50 usec
TE               295.9 K
D1            1.00000000 sec
D11           0.03000000 sec

======== CHANNEL f1 ========
NUC1                11B
P1                13.10 usec
PLW1         95.00000000 W
SFO1        160.4615790 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2            80.00 usec
PLW2         19.00000000 W
PLW12         0.39262000 W
PLW13         0.25127000 W
SFO2        500.1325007 MHz

F2 - Processing parameters
SI                32768
SF          160.4615790 MHz
WDW                EM
SSB       0
LB               40.00 Hz
GB        0
PC                1.40
```

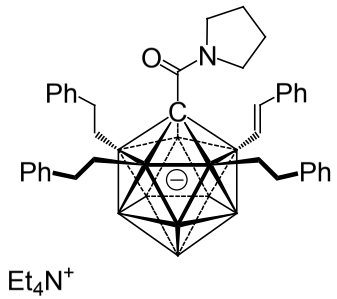
13C{1H} NMR, Phenyl ester CB11, 18 mg in 0.6 mL acetonitrile–d3, T = 19 C
Solvent peak ○



NMR36

```
Current Data Parameters
NAME      20151224-yjs-0150
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20151224
Time                   2.51
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT               CD3CN
NS                     2048
DS                        4
SWH               37878.789 Hz
FIDRES            0.577984 Hz
AQ                0.8650752 sec
RG                      203
DW                 13.200 usec
DE                  6.50 usec
TE                  297.1 K
D1             1.50000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                  10.00 usec
PLW1          95.00000000 W
SFO1         125.7716224 MHz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                   1H
PCPD2               80.00 usec
PLW2          19.00000000 W
PLW12          0.39262000 W
PLW13          0.25127000 W
SFO2         500.1320005 MHz

F2 - Processing parameters
SI                  4194304
SF           125.7576670 MHz
WDW                     EM
SSB       0
LB                  7.00 Hz
GB        0
PC                  1.40
```

1H{11B} NMR, Styrene, 1–Pyrrolidine amide–CB11, 5 mg in 0.6 mL acetone–d6, 19 C
Solvent residual peak ○

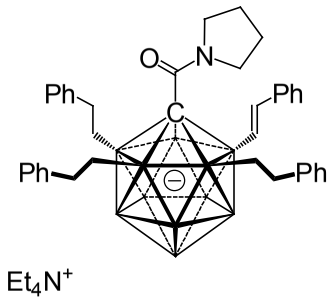


Et_4N^+

NMR37


```
Current Data Parameters
NAME      20151130-yjs-0061-1H
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20151203
Time                 16.42
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG             zgig30
TD                   65536
SOLVENT            Acetone
NS                      16
DS                       0
SWH              10000.000 Hz
FIDRES               0.152588 Hz
AQ                3.2767999 sec
RG                     181
DW                  50.000 usec
DE                   6.50 usec
TE                  295.9 K
D1           10.00000000 sec
D11           0.03000000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                   12.00 usec
PLW1          19.00000000 W
SFO1         500.1325200 MHz

======== CHANNEL f2 ========
CPDPRG[2                garp
NUC2                     11B
PCPD2                60.00 usec
PLW2          75.00000000 W
PLW12          2.08330011 W
SFO2         160.4615690 MHz

F2 - Processing parameters
SI                       65536
SF           500.1300101 MHz
WDW                        no
SSB       0
LB        0 Hz
GB        0
PC                     1.00
```

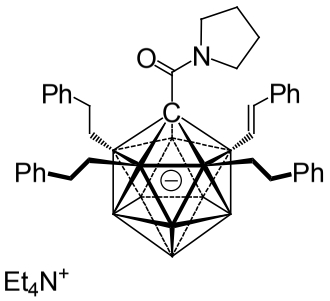
11B NMR, Styrene, 1-Pyrrolidine amide-CB11, 15 mg in 0.6 mL acetone-d6, 19 C



NMR38

```
Current Data Parameters
NAME      20160108-yjs-0061
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_           20160108
Time               16.48
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG              zg
TD                64098
SOLVENT         Acetone
NS                  256
DS                    0
SWH           32051.281 Hz
FIDRES         0.500036 Hz
AQ            0.9999288 sec
RG                  203
DW               15.600 usec
DE                6.50 usec
TE               292.6 K
D1            1.00000000 sec

======== CHANNEL f1 ========
NUC1                11B
P1                10.00 usec
PLW1         75.00000000 W
SFO1         160.4615792 MHz

F2 - Processing parameters
SI                 32768
SF          160.4615993 MHz
WDW                   EM
SSB       0
LB                20.00 Hz
GB        0
PC                 1.40
```

11B{1H} NMR, Styrene, 1–Pyrrolidine amide–CB11, 15 mg in 0.6 mL acetone–d6, 19 C

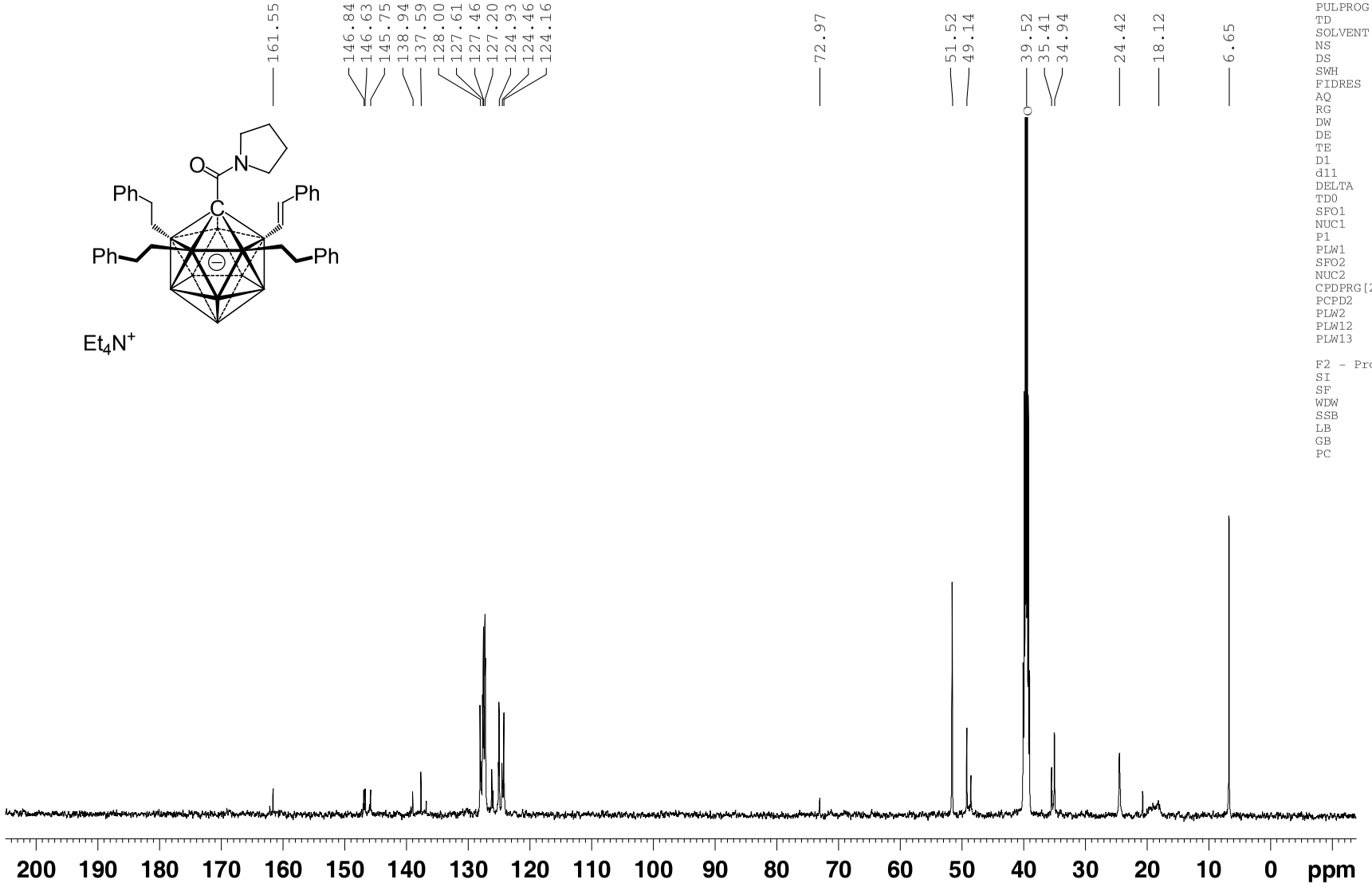


NMR39


```
Current Data Parameters
NAME      20160108-yjs-0061
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_           20160108
Time               16.55
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  256
DS                    0
SWH           32051.281 Hz
FIDRES         0.489064 Hz
AQ            1.0223616 sec
RG                  203
DW               15.600 usec
DE                6.50 usec
TE               293.0 K
D1           2.00000000 sec
D11          0.03000000 sec

======== CHANNEL f1 ========
NUC1                11B
P1                10.00 usec
PLW1         75.00000000 W
SFO1        160.4615792 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2             80.00 usec
PLW2         19.00000000 W
PLW12         0.42750001 W
PLW13         0.27360001 W
SFO2        500.1330885 MHz

F2 - Processing parameters
SI                 32768
SF          160.4615993 MHz
WDW                   EM
SSB       0
LB                40.00 Hz
GB        0
PC                 1.40
```

13C{1H} NMR, Styrene, 1–Pyrrolidine amide–CB11, 15 mg in 0.6 mL DMSO–d6 T = 80C
Solvent peak ○

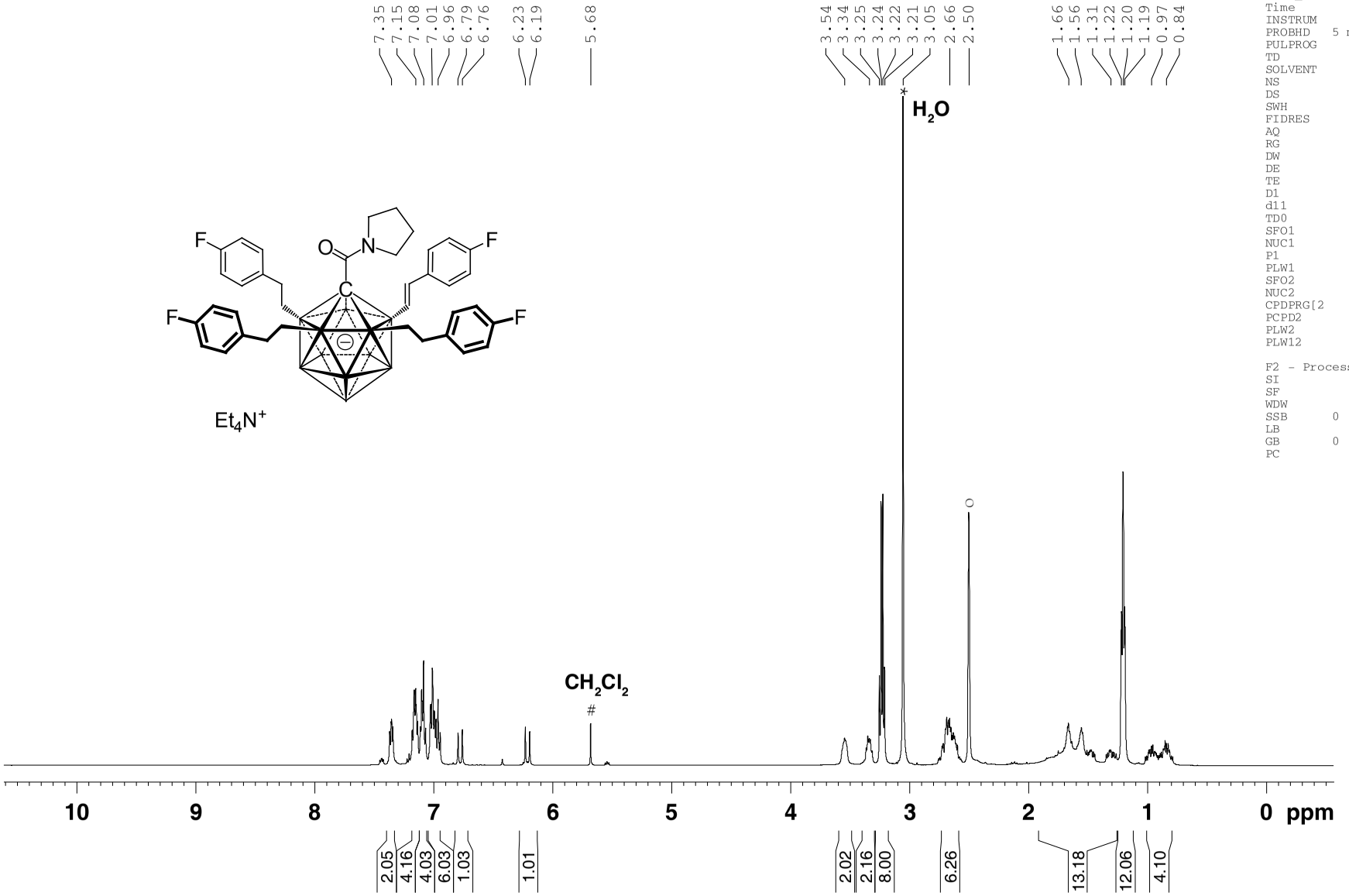
NMR40




```
Current Data Parameters
NAME      20150929-yjs-0061
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150930
Time                3.10
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT             DMSO
NS                    490
DS                      4
SWH             37878.789 Hz
FIDRES          0.577984 Hz
AQ              0.8650752 sec
RG                    203
DW                 13.200 usec
DE                  6.50 usec
TE                 352.0 K
D1            1.50000000 sec
d11           0.03000000 sec
DELTA         1.39999998 sec
TD0                     1
SFO1          125.7716224 MHz
NUC1                  13C
P1                 10.00 usec
PLW1          95.00000000 W
SFO2          500.1320005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2          19.00000000 W
PLW12          0.39262000 W
PLW13          0.25127000 W

F2 - Processing parameters
SI                  32768
SF            125.7579045 MHz
WDW                    EM
SSB       0
LB                 6.00 Hz
GB        0
PC                 1.40
```

1H{11B} NMR, [Et4N][pyrrolidine amide (fluorostyrene)4], 15 mg in 0.6 mL DMSO–d6 T = 80 C
Solvent residual peak ○

NMR41

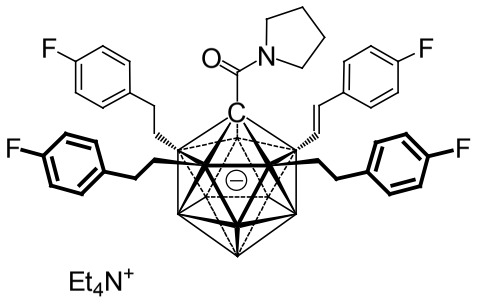


Current Data Parameters
NAME 20150203-yjs-0108
EXPNO 22
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150204
Time 4.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgig30
TD 65536
SOLVENT DMSO
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 57
DW 50.000 usec
DE 6.50 usec
TE 352.0 K
D1 10.00000000 sec
d11 0.03000000 sec
TD0 1
SFO1 500.1325200 MHz
NUC1 1H
P1 12.00 usec
PLW1 19.00000000 W
SFO2 160.4615690 MHz
NUC2 11B
CPDPRG[2 garp
PCPD2 60.00 usec
PLW2 75.00000000 W
PLW12 2.08330011 W

F2 - Processing parameters
SI 65536
SF 500.1300058 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

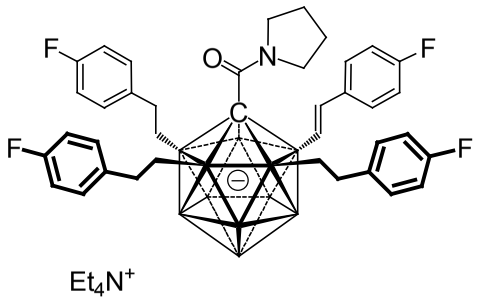
11B NMR, [Et4N][pyrrolidine amide (fluorostyrene)4], 15 mg in 0.6 mL DMSO–d6 T = 80 C




```
Current Data Parameters
NAME      20150203-yjs-0108
EXPNO                   23
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150204
Time                4.43
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG               zg
TD                 64098
SOLVENT             DMSO
NS                    32
DS                     0
SWH            32051.281 Hz
FIDRES          0.500036 Hz
AQ              0.9999288 sec
RG                   203
DW                15.600 usec
DE                16.00 usec
TE                352.0 K
D1           1.00000000 sec
TD0                    1
SFO1         160.4615792 MHz
NUC1                11B
P1                10.00 usec
PLW1         75.00000000 W

F2 - Processing parameters
SI                 32768
SF           160.4615993 MHz
WDW                   EM
SSB       0
LB                60.00 Hz
GB        0
PC                 1.40
```

NMR42

11B{1H} NMR, [Et4N][pyrrolidine amide (fluorostyrene)4], 15 mg in 0.6 mL DMSO–d6 T = 80 C

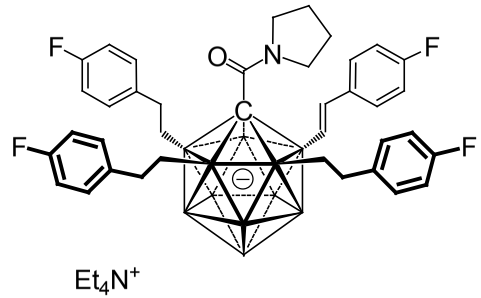


NMR43

```
Current Data Parameters
NAME      20150203-yjs-0108
EXPNO                   24
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150204
Time                4.46
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT             DMSO
NS                    32
DS                     0
SWH            32051.281 Hz
FIDRES          0.489064 Hz
AQ              1.0223616 sec
RG                   203
DW                15.600 usec
DE                 6.50 usec
TE                352.0 K
D1             5.00000000 sec
d11            0.03000000 sec
DELTA          4.90000010 sec
TD0                    1
SFO1         160.4615792 MHz
NUC1                11B
P1                 10.00 usec
PLW1          75.00000000 W
SFO2         500.1330885 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2              80.00 usec
PLW2          19.00000000 W
PLW12          0.42750001 W
PLW13          0.27360001 W

F2 - Processing parameters
SI                 32768
SF           160.4615993 MHz
WDW                   EM
SSB       0
LB                 80.00 Hz
GB        0
PC                  1.40
```

13C{1H} NMR, [Et4N][pyrrolidine amide (fluorostyrene)4], 15 mg in 0.6 mL DMSO-d6 T = 80 C
Solvent peak ○

NMR44

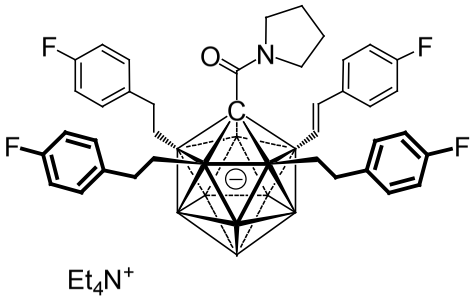


Et_4N^+


```
Current Data Parameters
NAME      20150203-yjs-0108
EXPNO                   25
PROCNO                   1

F2 - Acquisition Parameters
Date_              20150204
Time                   5.08
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT                DMSO
NS                       854
DS                         0
SWH                37878.789 Hz
FIDRES              0.577984 Hz
AQ                  0.8650752 sec
RG                       203
DW                    13.200 usec
DE                     6.50 usec
TE                     352.0 K
D1                1.50000000 sec
d11               0.03000000 sec
DELTA             1.39999998 sec
TD0                        1
SFO1             125.7716224 MHz
NUC1                     13C
P1                    10.00 usec
PLW1             95.00000000 W
SFO2             500.1320005 MHz
NUC2                     1H
CPDPRG[2              waltz16
PCPD2                 80.00 usec
PLW2             19.00000000 W
PLW12             0.42750001 W
PLW13             0.27360001 W

F2 - Processing parameters
SI                       32768
SF               125.7579028 MHz
WDW                         EM
SSB          0
LB                     6.00 Hz
GB           0
PC                     1.40
```

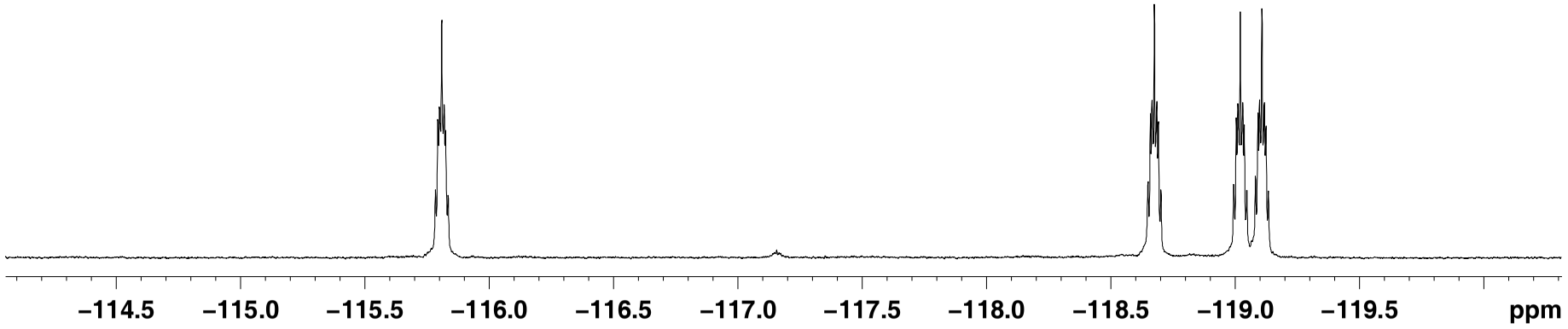
19F NMR, [Et4N][pyrrolidine amide (fluorostyrene)4] product, 15 mg in 0.6 mL DMSO–d6 T = 23 C

NMR45

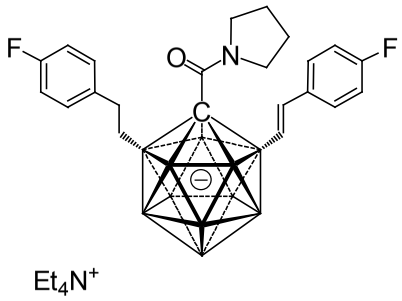


-115.81
-118.68
-119.02
-119.11

```
Current Data Parameters
NAME            1345.fid
EXPNO                  1
PROCNO                 1

F2 - Processing parameters
SI                262144
SF          564.3555613 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.00
```



1H{11B} NMR, Flurostyrene CB11, 12 mg in 0.6 ml acetonitrile-d3, T= 23 C
Solvent residual peak ○

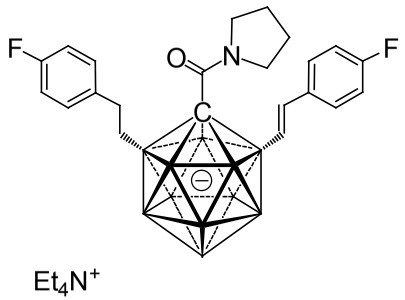


Et_4N^+

NMR46


```
Current Data Parameters
NAME      20161116-yjs-0122
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20161116
Time                 21.50
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG           zgig30
TD                 65536
SOLVENT            CD3CN
NS                    16
DS                     0
SWH           12500.000 Hz
FIDRES            0.190735 Hz
AQ             2.6214399 sec
RG                   114
DW                40.000 usec
DE                 6.50 usec
TE                296.5 K
D1             5.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 11.60 usec
PLW1          19.00000000 W
SFO1         500.1335009 MHz

======== CHANNEL f2 ========
CPDPRG[2             garp
NUC2                  11B
PCPD2              100.00 usec
PLW2          95.00000000 W
PLW12          1.63030005 W
SFO2         160.4615690 MHz

F2 - Processing parameters
SI                   65536
SF           500.1300157 MHz
WDW                    EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.00
```

11B NMR, Flurostyrene CB11, 12 mg in 0.6 mL acetonitrile–d3, T = 19 C

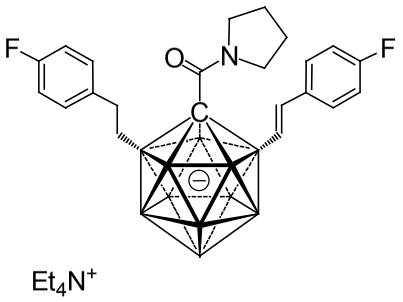


Et_4N^+

NMR47


```
Current Data Parameters
NAME      20151225-yjs-0122
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20151225
Time                  20.54
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    64098
SOLVENT               CD3CN
NS                      256
DS                        0
SWH               32051.281 Hz
FIDRES             0.500036 Hz
AQ                0.9999288 sec
RG                      203
DW                   15.600 usec
DE                    6.50 usec
TE                   295.9 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                   11B
P1                   13.10 usec
PLW1          95.00000000 W
SFO1          160.4615792 MHz

F2 - Processing parameters
SI                     32768
SF            160.4615790 MHz
WDW                    EM
SSB       0
LB                   30.00 Hz
GB        0
PC                    1.40
```

11B{1H} NMR, Flurostyrene CB11, 12 mg in 0.6 mL acetonitrile-d3, T = 19 C

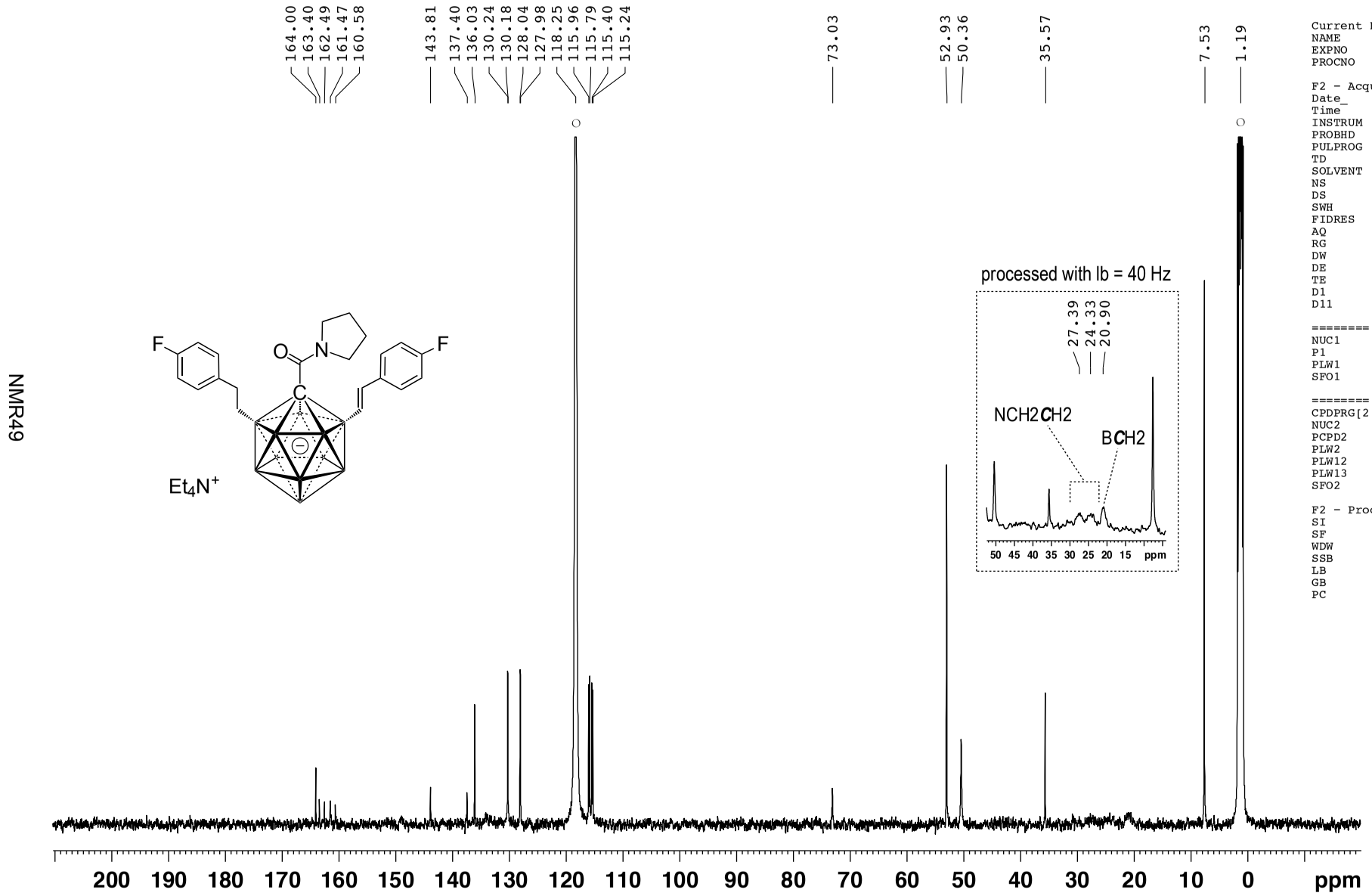


Et_4N^+

NMR48


```
Current Data Parameters
NAME      20151225-yjs-0122
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_           20151225
Time               21.04
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CD3CN
NS                  256
DS                    0
SWH           32051.281 Hz
FIDRES         0.489064 Hz
AQ            1.0223616 sec
RG                  203
DW               15.600 usec
DE                6.50 usec
TE               295.9 K
D1            1.00000000 sec
D11           0.03000000 sec

======== CHANNEL f1 ========
NUC1                11B
P1                13.10 usec
PLW1         95.00000000 W
SFO1        160.4615790 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2            80.00 usec
PLW2         19.00000000 W
PLW12         0.39262000 W
PLW13         0.25127000 W
SFO2        500.1325007 MHz

F2 - Processing parameters
SI                 32768
SF          160.4615790 MHz
WDW                   EM
SSB       0
LB                30.00 Hz
GB        0
PC                 1.40
```

13C{1H} NMR, Fluorostyrene CB11, 15 mg in 0.6 mL acetonitrile–d3, T = 23 C
Solvent peak

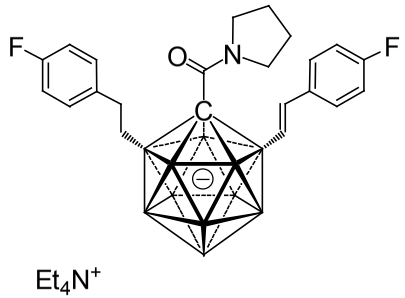


NMR49

```
Current Data Parameters
NAME      YJS_pub02_20160929_yjs-012
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20160929
Time               4.34
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD               65536
SOLVENT          CD3CN
NS                4096
DS                   4
SWH          37878.789 Hz
FIDRES        0.577984 Hz
AQ            0.8650752 sec
RG                 203
DW              13.200 usec
DE               6.50 usec
TE               297.9 K
D1          1.50000000 sec
D11         0.03000000 sec

======== CHANNEL f1 ========
NUC1               13C
P1               10.50 usec
PLW1        95.00000000 W
SFO1       125.7716224 MHz

======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2               1H
PCPD2            80.00 usec
PLW2        19.00000000 W
PLW12        0.39947000 W
PLW13        0.25566000 W
SFO2       500.1320005 MHz

F2 - Processing parameters
SI                32768
SF         125.7576799 MHz
WDW                EM
SSB       0
LB               5.00 Hz
GB        0
PC               1.40
```

19F NMR, Flurostyrene CB11, 12 mg in 0.6 mL acetonitrile-d3, T = 19 C

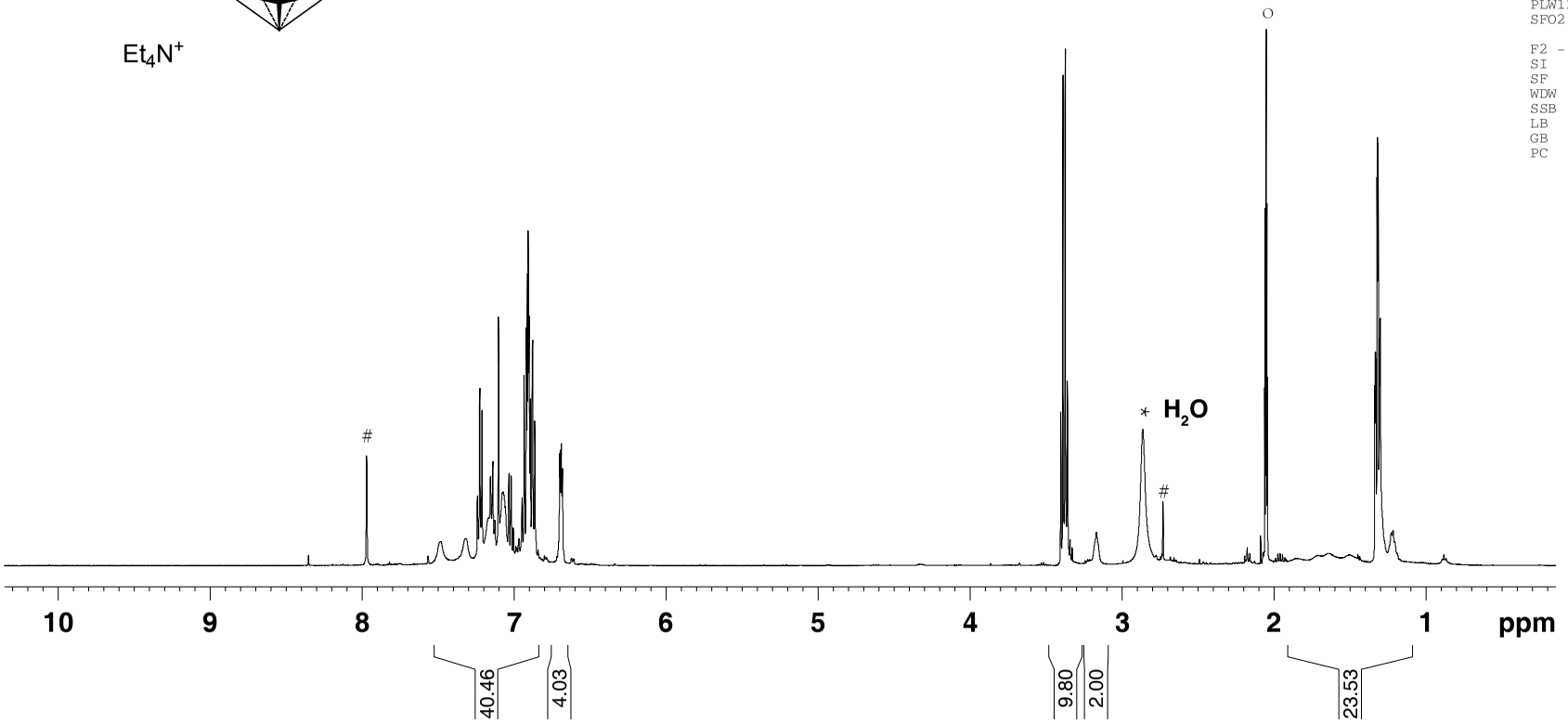
NMR50




```
Current Data Parameters
NAME      20151224-yjs-0122
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20151225
Time                   6.34
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG              zgflqn
TD                   131072
SOLVENT               CD3CN
NS                       16
DS                        4
SWH              89285.711 Hz
FIDRES            0.681196 Hz
AQ               0.7340032 sec
RG                  193.34
DW                   5.600 usec
DE                   6.50 usec
TE                  294.0 K
D1             5.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    19F
P1                   15.00 usec
PLW1          20.00000000 W
SFO1          376.4607164 MHz

F2 - Processing parameters
SI                    524288
SF            376.4983660 MHz
WDW                     EM
SSB       0
LB                    0.80 Hz
GB        0
PC                    1.00
```

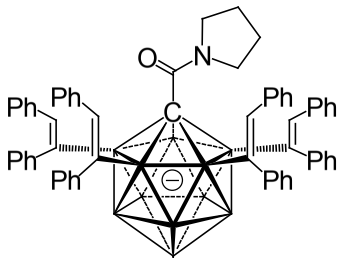
1H{11B} NMR, [Et4N][pyrrolidine amide (diphenylacetylene)4] , 15 mg dissolved in 0.6 mL acetone-d6, T = 23 C
Solvent residual peak ○ Dimethylformamide #

NMR51




```
Current Data Parameters
NAME      20151028-yjs-0104
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20151028
Time                 23.07
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG             zgig30
TD                   65536
SOLVENT            Acetone
NS                      16
DS                       0
SWH               9920.635 Hz
FIDRES            0.151377 Hz
AQ                3.3030145 sec
RG                      64
DW                  50.400 usec
DE                   6.50 usec
TE                  295.5 K
D1             3.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                   12.00 usec
PLW1          19.00000000 W
SFO1         500.1330269 MHz

======== CHANNEL f2 ========
CPDPRG[2                garp
NUC2                    11B
PCPD2                60.00 usec
PLW2          75.00000000 W
PLW12          2.08330011 W
SFO2         160.4615690 MHz

F2 - Processing parameters
SI                      65536
SF           500.1300100 MHz
WDW                        no
SSB       0
LB        0 Hz
GB        0
PC                     1.00
```

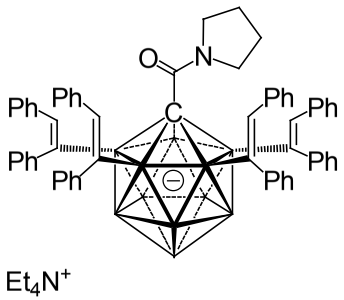
11B NMR, [Et4N][pyrrolidine amide (diphenylacetylene)4] , 15 mg dissolved in 0.6 mL acetone-d6, T = 23 C



NMR52

```
Current Data Parameters
NAME      20151229-yjs-0104
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_           20151229
Time               18.18
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG              zg
TD                64098
SOLVENT         Acetone
NS                  256
DS                    0
SWH           32051.281 Hz
FIDRES         0.500036 Hz
AQ            0.9999288 sec
RG                  203
DW               15.600 usec
DE                6.50 usec
TE               293.0 K
D1            1.00000000 sec

======== CHANNEL f1 ========
NUC1                11B
P1                10.00 usec
PLW1         75.00000000 W
SFO1         160.4615792 MHz

F2 - Processing parameters
SI                 32768
SF          160.4615993 MHz
WDW                  EM
SSB       0
LB                40.00 Hz
GB        0
PC                 1.40
```

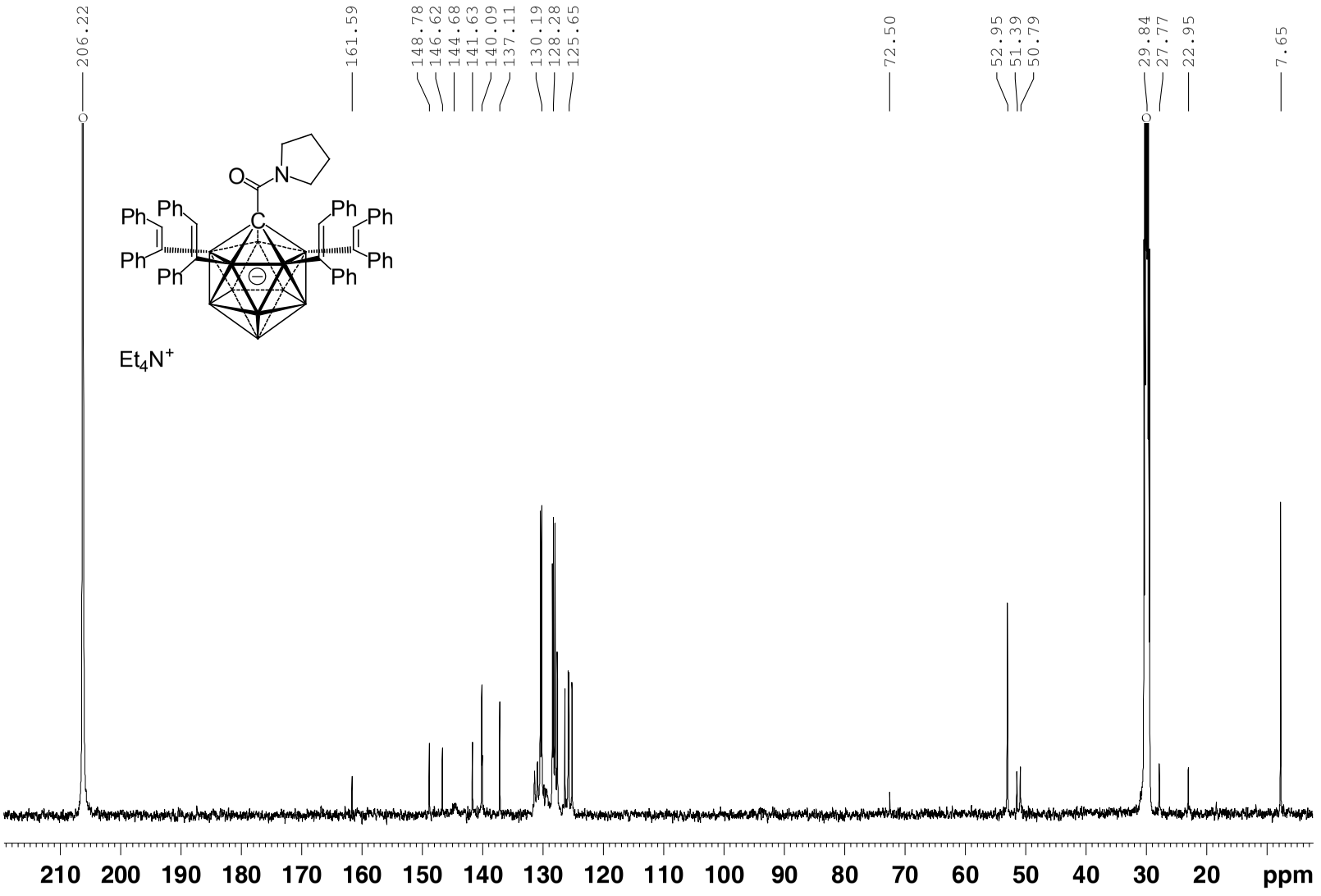
11B{1H} NMR, [Et4N][pyrrolidine amide (diphenylacetylene)4] , 15 mg dissolved in 0.6 mL acetone-d6, T = 23 C



NMR53


```
Current Data Parameters
NAME      20151229-yjs-0104
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_              20151229
Time                  18.34
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT            Acetone
NS                      256
DS                        0
SWH               32051.281 Hz
FIDRES             0.489064 Hz
AQ                1.0223616 sec
RG                      203
DW                   15.600 usec
DE                    6.50 usec
TE                   293.9 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                    11B
P1                    10.00 usec
PLW1          75.00000000 W
SFO1         160.4615792 MHz

======== CHANNEL f2 ========
CPDPRG[2             waltz16
NUC2                    1H
PCPD2                 80.00 usec
PLW2          19.00000000 W
PLW12          0.42750001 W
PLW13          0.27360001 W
SFO2         500.1330885 MHz

F2 - Processing parameters
SI                      32768
SF           160.4615993 MHz
WDW                        EM
SSB       0
LB                    40.00 Hz
GB        0
PC                     1.40
```

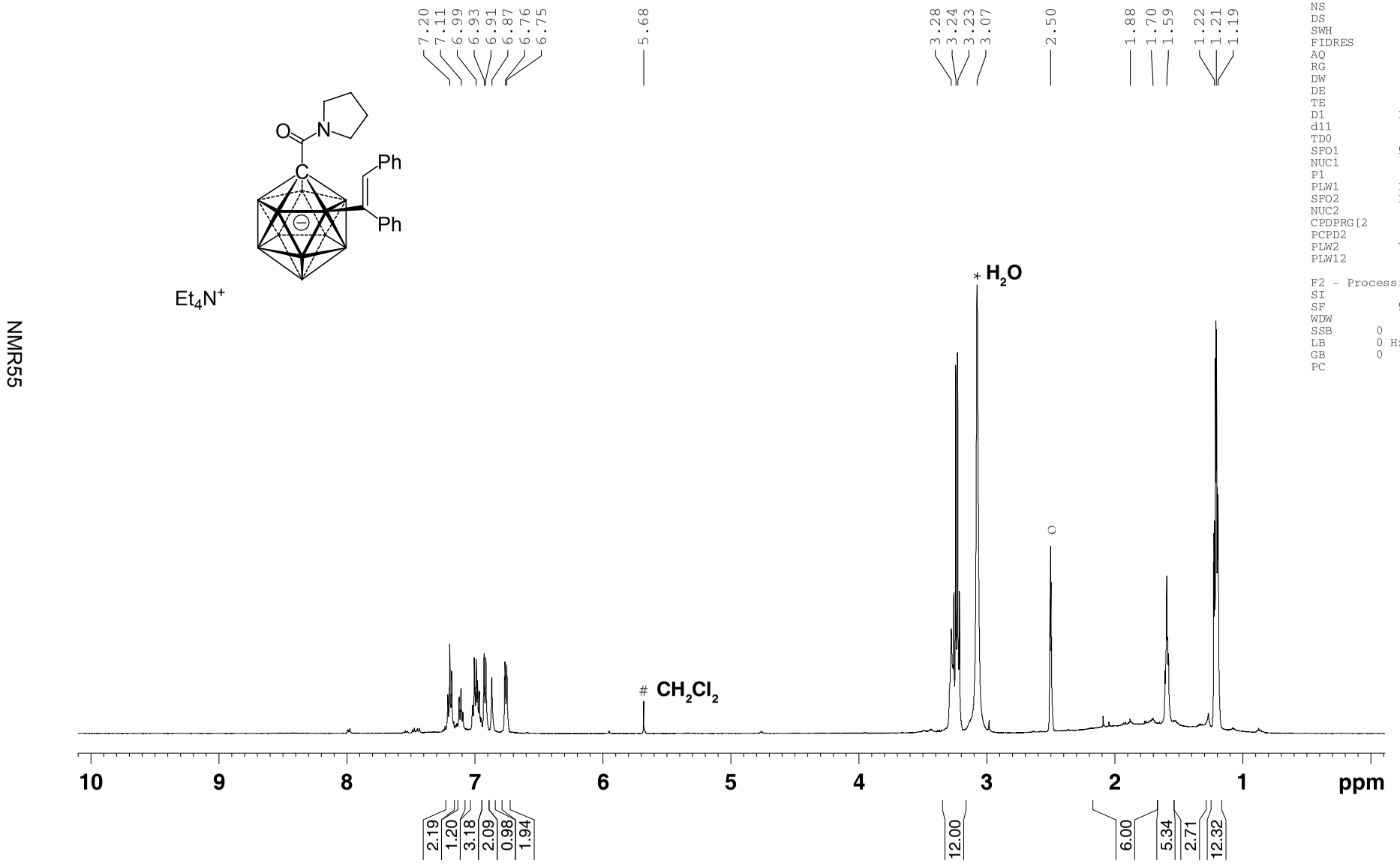
13C{1H} NMR, [Et4N][pyrrolidine amide (diphenylacetylene)4] , 15 mg dissolved in 0.6 mL acetone–d6, T = 23 C
Solvent peak



NMR54

```
Current Data Parameters
NAME      20151028-yjs-0104
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_           20151028
Time              23.23
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                65536
SOLVENT           Acetone
NS                1024
DS                4
SWH           37878.789 Hz
FIDRES         0.577984 Hz
AQ            0.8650752 sec
RG                203
DW              13.200 usec
DE               6.50 usec
TE              295.5 K
D1           1.50000000 sec
D11          0.03000000 sec

======== CHANNEL f1 ========
NUC1              13C
P1              10.00 usec
PLW1        95.00000000 W
SFO1       125.7716224 MHz

======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2              1H
PCPD2           80.00 usec
PLW2        19.00000000 W
PLW12        0.39262000 W
PLW13        0.25127000 W
SFO2       500.1320005 MHz

F2 - Processing parameters
SI               32768
SF          125.7576813 MHz
WDW                 EM
SSB      0
LB                5.00 Hz
GB       0
PC                1.40
```

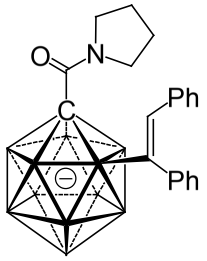
1H{11B} NMR, Mono-substituted CB11,15 mg in 0.6 mL DMSO-d6 T = 80 C
Solvent residual peak ○



NMR55

```
Current Data Parameters
NAME      20150929-yjs-0125
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20150929
Time                 21.45
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG             zgig30
TD                   65536
SOLVENT                DMSO
NS                      16
DS                       0
SWH             10000.000 Hz
FIDRES              0.152588 Hz
AQ                3.2767999 sec
RG                     114
DW                  50.000 usec
DE                   6.50 usec
TE                  352.0 K
D1             10.00000000 sec
d11             0.03000000 sec
TD0                      1
SFO1           500.1325200 MHz
NUC1                    1H
P1                   12.00 usec
PLW1           19.00000000 W
SFO2           160.4615690 MHz
NUC2                   11B
CPDPRG[2               garp
PCPD2               60.00 usec
PLW2           75.00000000 W
PLW12           2.08330011 W

F2 - Processing parameters
SI                     65536
SF           500.1300048 MHz
WDW                      no
SSB          0
LB           0 Hz
GB           0
PC                     1.00
```

11B NMR, Mono-substituted CB11,15 mg in 0.6 mL DMSO-d6 T = 80 C

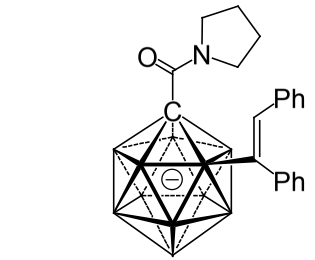


Et_4N^+

NMR56


```
Current Data Parameters
NAME      20150929-yjs-0125
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150929
Time              21.52
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG             zg
TD                64098
SOLVENT            DMSO
NS                   32
DS                    0
SWH           32051.281 Hz
FIDRES         0.500036 Hz
AQ             0.9999288 sec
RG                  203
DW               15.600 usec
DE                6.50 usec
TE               352.0 K
D1            1.00000000 sec
TD0                   1
SFO1         160.4615792 MHz
NUC1               11B
P1               10.00 usec
PLW1         75.00000000 W

F2 - Processing parameters
SI                32768
SF           160.4615993 MHz
WDW                EM
SSB       0
LB               40.00 Hz
GB        0
PC                1.40
```

11B{1H} NMR, Mono-substituted CB11,15 mg in 0.6 mL DMSO-d6 T = 80 C

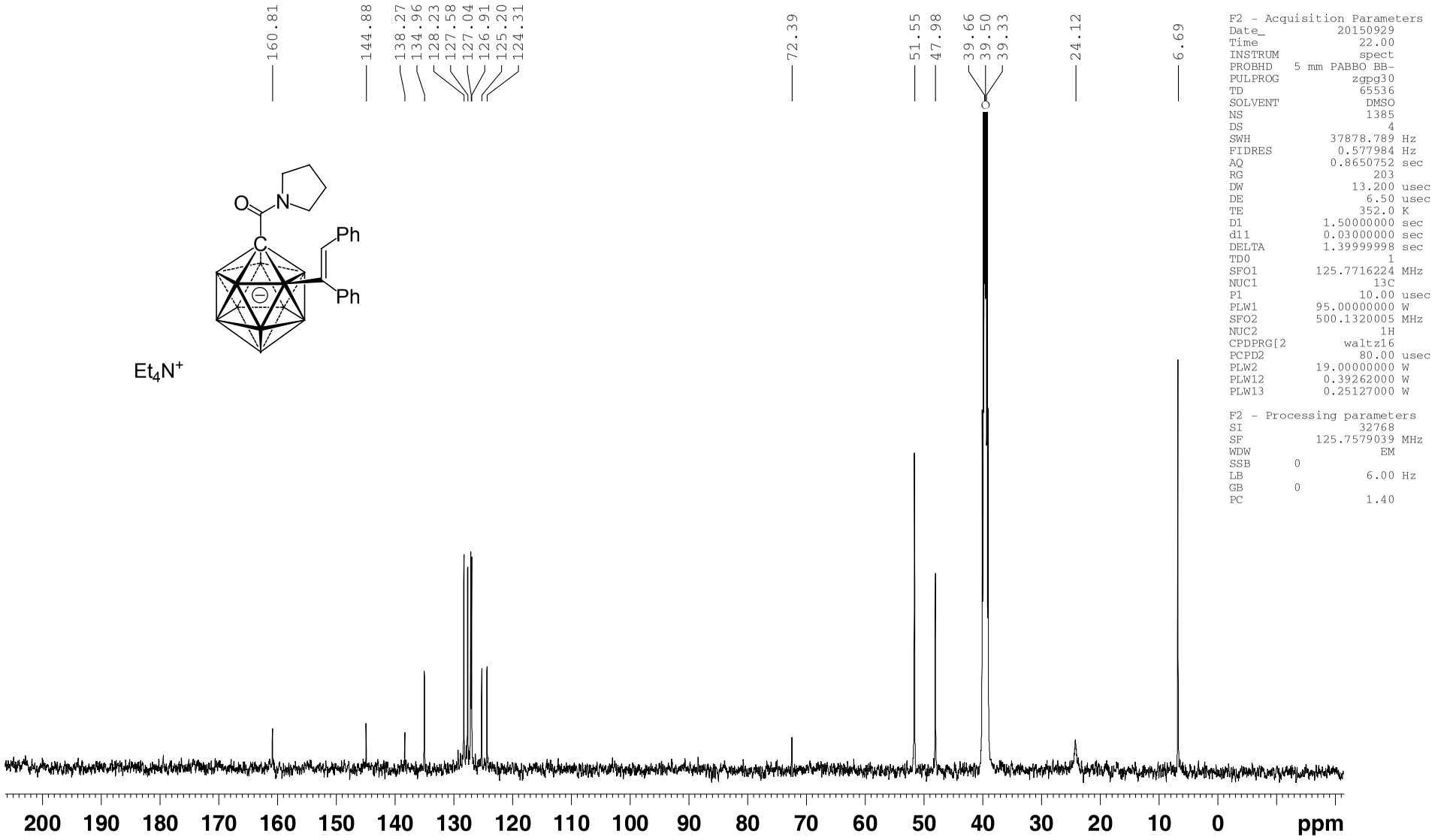


Et_4N^+

NMR57


```
Current Data Parameters
NAME      20150929-yjs-0125
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150929
Time              21.55
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD                65536
SOLVENT             DMSO
NS                    32
DS                     0
SWH           32051.281 Hz
FIDRES         0.489064 Hz
AQ             1.0223616 sec
RG                   203
DW                15.600 usec
DE                 6.50 usec
TE                352.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1
SFO1        160.4615792 MHz
NUC1                11B
P1                10.00 usec
PLW1        75.00000000 W
SFO2        500.1330885 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        19.00000000 W
PLW12        0.42750001 W
PLW13        0.27360001 W

F2 - Processing parameters
SI                32768
SF          160.4615993 MHz
WDW                  EM
SSB       0
LB                60.00 Hz
GB        0
PC                 1.40
```

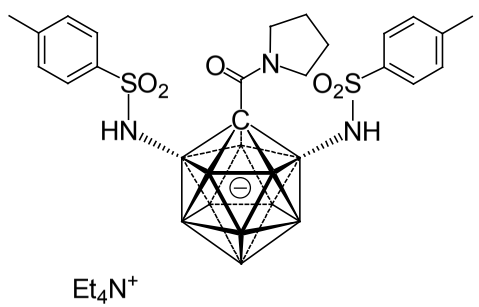
13C{1H} NMR, Mono-substituted CB11,15 mg in 0.6 mL DMSO-d6 T = 80 C
Solvent peak ○



NMR58

```
Current Data Parameters
NAME      20150929-yjs-0125
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_             20150929
Time                 22.00
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                65536
SOLVENT            DMSO
NS                 1385
DS                    4
SWH           37878.789 Hz
FIDRES         0.577984 Hz
AQ             0.8650752 sec
RG                  203
DW               13.200 usec
DE                6.50 usec
TE                352.0 K
D1           1.50000000 sec
d11          0.03000000 sec
DELTA        1.39999998 sec
TD0                   1
SFO1        125.7716224 MHz
NUC1                13C
P1               10.00 usec
PLW1        95.00000000 W
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2            80.00 usec
PLW2        19.00000000 W
PLW12        0.39262000 W
PLW13        0.25127000 W

F2 - Processing parameters
SI                32768
SF          125.7579039 MHz
WDW                  EM
SSB       0
LB                6.00 Hz
GB        0
PC                1.40
```

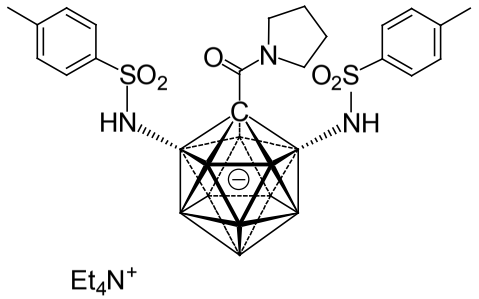
1H{11B} NMR, 2,4-NHTs, 1-Pyrrolidine amide-CB11, 5 mg in 0.6 mL acetone-d6, T= 19 C
Solvent residual peak

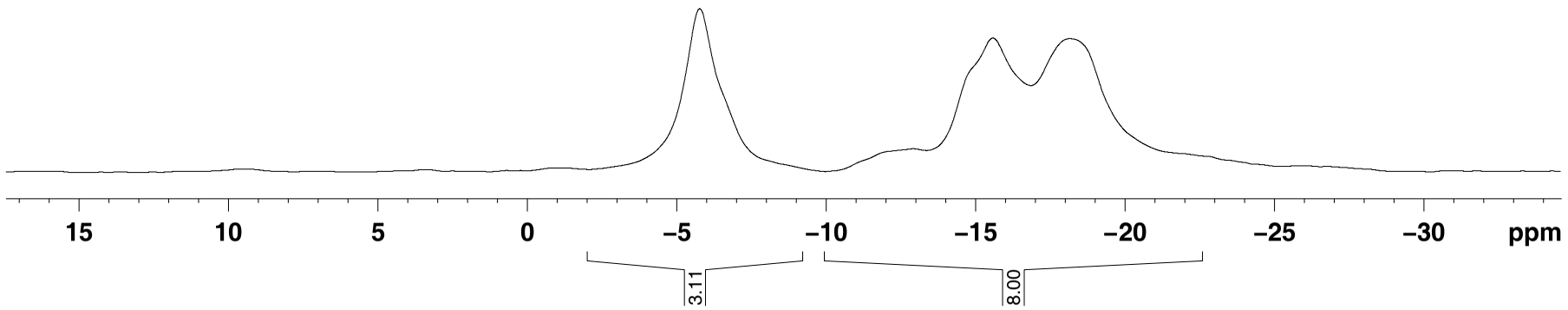


NMR59


```
Current Data Parameters
NAME      20151209-yjs-0105
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20151130
Time                 17.11
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgig30
TD                   65536
SOLVENT            Acetone
NS                      16
DS                       0
SWH              10000.000 Hz
FIDRES               0.152588 Hz
AQ                3.2767999 sec
RG                     181
DW                  50.000 usec
DE                   6.50 usec
TE                  295.0 K
D1           10.00000000 sec
D11           0.03000000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                   12.00 usec
PLW1         19.00000000 W
SFO1        500.1325200 MHz

======== CHANNEL f2 ========
CPDPRG[2                garp
NUC2                   11B
PCPD2                60.00 usec
PLW2         75.00000000 W
PLW12         2.08330011 W
SFO2        160.4615690 MHz

F2 - Processing parameters
SI                     65536
SF          500.1300102 MHz
WDW                       no
SSB       0
LB        0 Hz
GB        0
PC                     1.00
```

11B NMR, 2,4-NHTs, 1-Pyrrolidine amide-CB11, 15 mg in 0.6 mL DMSO-d6, T= 80 C

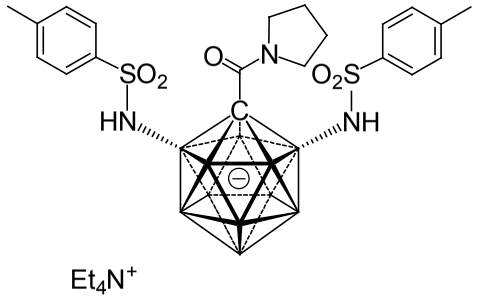




NMR60

```
Current Data Parameters
NAME      20150203-yjs-0105
EXPNO                   23
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150204
Time                2.40
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG                 zg
TD                  64098
SOLVENT              DMSO
NS                     32
DS                      0
SWH             32051.281 Hz
FIDRES          0.500036 Hz
AQ              0.9999288 sec
RG                    203
DW                15.600 usec
DE                16.00 usec
TE                352.0 K
D1           1.00000000 sec
TD0                     1
SFO1         160.4615792 MHz
NUC1                 11B
P1                10.00 usec
PLW1         75.00000000 W

F2 - Processing parameters
SI                  32768
SF           160.4615993 MHz
WDW                    EM
SSB       0
LB                60.00 Hz
GB        0
PC                 1.40
```

11B{1H} NMR, 2,4-NHTs, 1-Pyrrolidine amide-CB11, 15 mg in 0.6 mL DMSO-d6, T= 80 C

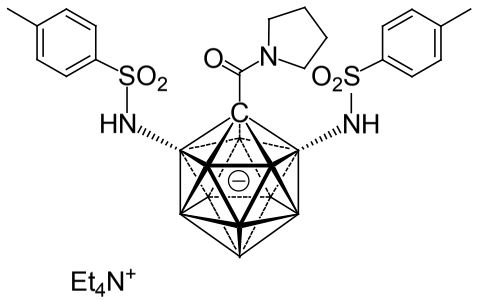


NMR61


```
Current Data Parameters
NAME      20150203-yjs-0105
EXPNO                   24
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150204
Time                2.43
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT              DMSO
NS                     32
DS                      0
SWH             32051.281 Hz
FIDRES          0.489064 Hz
AQ              1.0223616 sec
RG                    203
DW                15.600 usec
DE                 6.50 usec
TE                352.0 K
D1             5.00000000 sec
d11            0.03000000 sec
DELTA          4.90000010 sec
TD0                     1
SFO1         160.4615792 MHz
NUC1                  11B
P1                 10.00 usec
PLW1          75.00000000 W
SFO2         500.1330885 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2          19.00000000 W
PLW12          0.42750001 W
PLW13          0.27360001 W

F2 - Processing parameters
SI                  32768
SF           160.4615993 MHz
WDW                    EM
SSB       0
LB                 60.00 Hz
GB        0
PC                  1.40
```

13C{1H} NMR, 2,4–NHTs, 1–Pyrrolidine amide–CB11, 15 mg in 0.6 mL DMSO–d6, T= 80 C
Solvent peak ○

NMR62

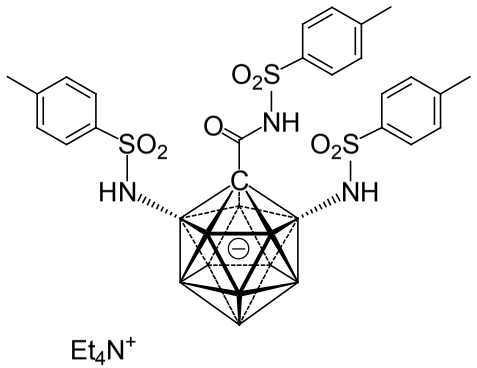


Et_4N^+


```
Current Data Parameters
NAME      20150203-yjs-0105
EXPNO                   25
PROCNO                   1

F2 - Acquisition Parameters
Date_              20150204
Time                   3.26
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT                DMSO
NS                     1460
DS                        0
SWH               37878.789 Hz
FIDRES             0.577984 Hz
AQ                 0.8650752 sec
RG                      203
DW                   13.200 usec
DE                    6.50 usec
TE                   352.2 K
D1              1.50000000 sec
d11             0.03000000 sec
DELTA           1.39999998 sec
TD0                       1
SFO1            125.7716224 MHz
NUC1                    13C
P1                   10.00 usec
PLW1            95.00000000 W
SFO2            500.1320005 MHz
NUC2                    1H
CPDPRG[2            waltz16
PCPD2                80.00 usec
PLW2            19.00000000 W
PLW12            0.42750001 W
PLW13            0.27360001 W

F2 - Processing parameters
SI                     32768
SF              125.7579020 MHz
WDW                       EM
SSB       0
LB                    6.00 Hz
GB        0
PC                    1.40
```

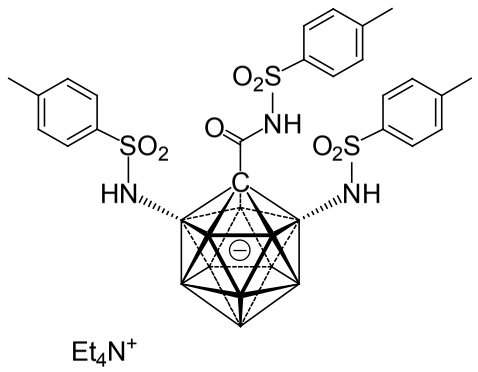
1H{11B} NMR, [Et4N][tosyl amide]+2NHTs, 10 mg in 0.6 mL acetone, T = 19 C
Solvent residue peak ○

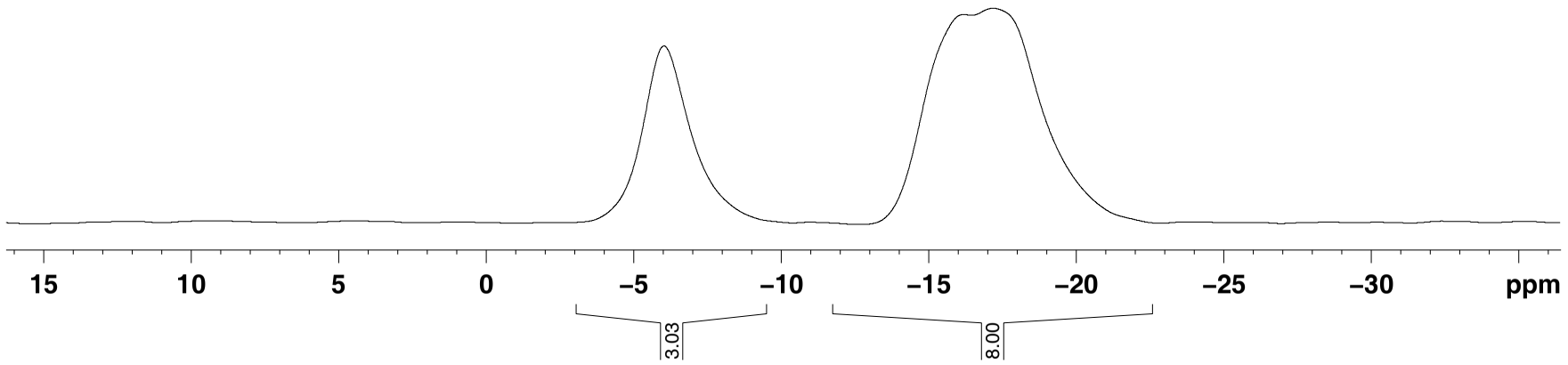
NMR63




```
Current Data Parameters
NAME      20150310-yjs-0057
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_              20150310
Time                 16.39
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG            zgig30
TD                  65536
SOLVENT           Acetone
NS                     16
DS                      0
SWH             14097.744 Hz
FIDRES           0.215115 Hz
AQ               2.3243434 sec
RG                     144
DW                35.467 usec
DE                 6.50 usec
TE                293.1 K
D1           10.00000000 sec
d11           0.03000000 sec
TD0                     1
SFO1         500.1310003 MHz
NUC1                   1H
P1                 12.00 usec
PLW1         19.00000000 W
SFO2         160.4615690 MHz
NUC2                  11B
CPDPRG[2             garp
PCPD2             60.00 usec
PLW2         75.00000000 W
PLW12         2.08330011 W

F2 - Processing parameters
SI                  65536
SF           500.1300102 MHz
WDW                    no
SSB       0
LB        0 Hz
GB        0
PC                   1.00
```

11B NMR, [Et4N][tosyl amide]+2NHTs, 10 mg in 0.6 mL acetone, T = 19 C



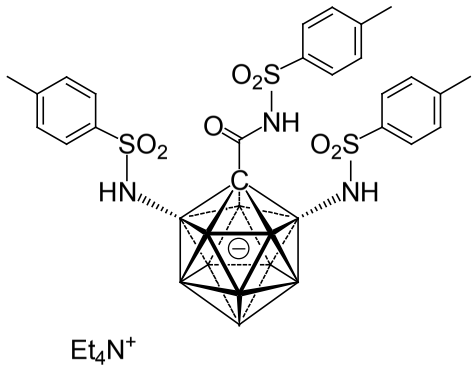


NMR64

```
Current Data Parameters
NAME      20150310-yjs-0057
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150310
Time              16.42
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG             zg
TD                64098
SOLVENT         Acetone
NS                   32
DS                    0
SWH           32051.281 Hz
FIDRES         0.500036 Hz
AQ             0.9999288 sec
RG                  203
DW               15.600 usec
DE               16.00 usec
TE               292.9 K
D1            1.00000000 sec
TD0                   1
SFO1         160.4615792 MHz
NUC1              11B
P1               10.00 usec
PLW1         75.00000000 W

F2 - Processing parameters
SI                32768
SF           160.4615993 MHz
WDW                  EM
SSB       0
LB             150.00 Hz
GB        0
PC                1.40
```

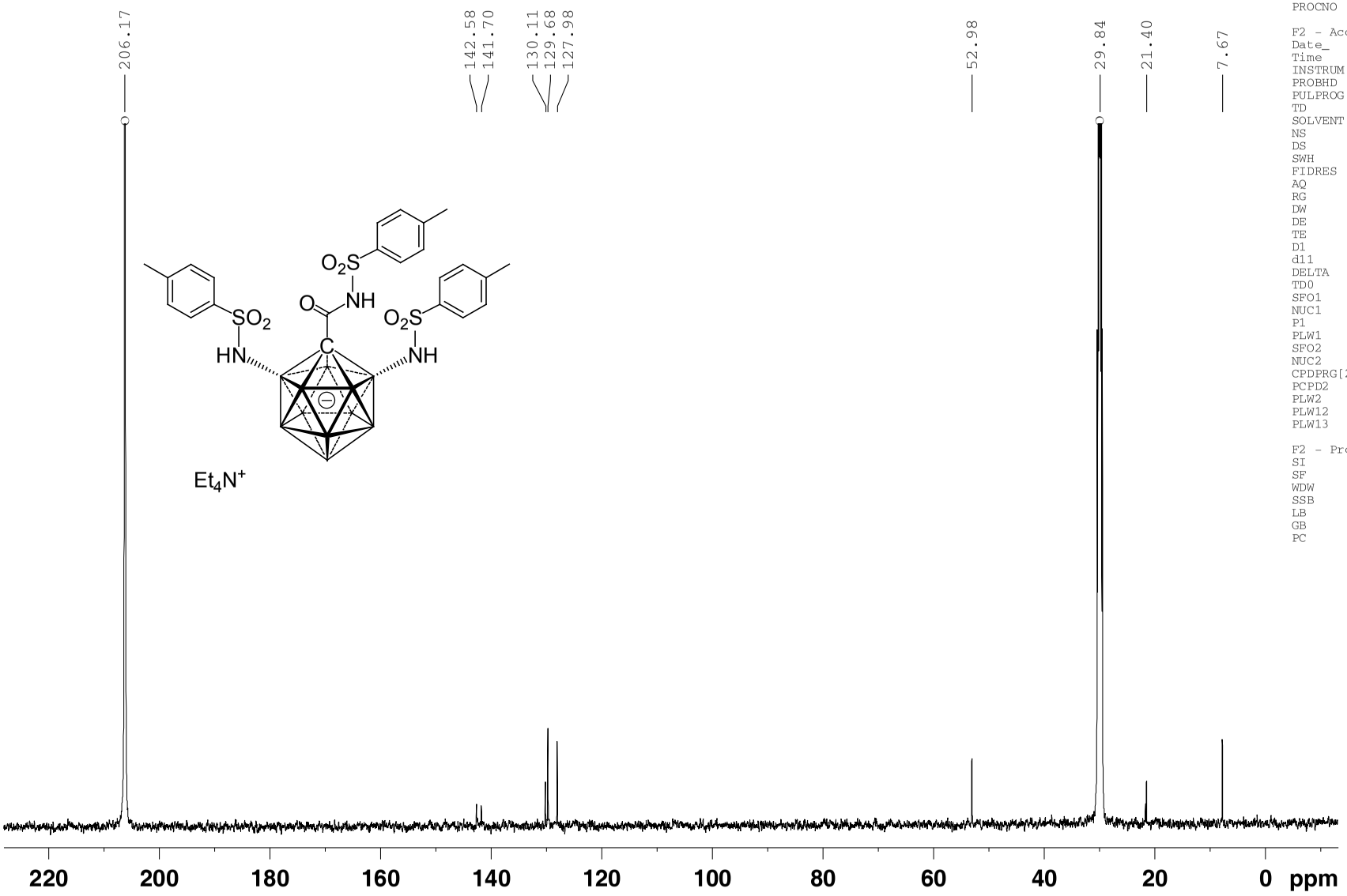
11B{1H} NMR, [Et4N][tosyl amide]+2NHTs, 10 mg in 0.6 mL acetone, T = 19 C

NMR65




```
Current Data Parameters
NAME      20150310-yjs-0057
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_            20150310
Time                16.49
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT           Acetone
NS                     32
DS                      0
SWH             32051.281 Hz
FIDRES          0.489064 Hz
AQ              1.0223616 sec
RG                    203
DW                 15.600 usec
DE                  6.50 usec
TE                 293.6 K
D1            5.00000000 sec
d11           0.03000000 sec
DELTA         4.90000010 sec
TD0                     1
SFO1          160.4615792 MHz
NUC1                  11B
P1                 10.00 usec
PLW1          75.00000000 W
SFO2          500.1330885 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2          19.00000000 W
PLW12          0.42750001 W
PLW13          0.27360001 W

F2 - Processing parameters
SI                  32768
SF            160.4615993 MHz
WDW                    EM
SSB       0
LB                150.00 Hz
GB        0
PC                  1.40
```

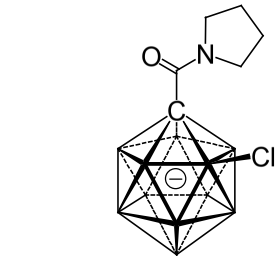
13C{1H} NMR, [Et4N][tosyl amide]+2NHTs, 10 mg in 0.6 mL acetone-d6, T = 19 C
Solvent peak ○



NMR66

```
Current Data Parameters
NAME      20150310-yjs-0057
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150310
Time              17.06
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD                65536
SOLVENT         Acetone
NS                 1016
DS                    0
SWH           37878.789 Hz
FIDRES         0.577984 Hz
AQ            0.8650752 sec
RG                  203
DW               13.200 usec
DE                6.50 usec
TE                294.0 K
D1           1.50000000 sec
d11          0.03000000 sec
DELTA        1.39999998 sec
TD0                   1
SFO1        125.7716224 MHz
NUC1                13C
P1               10.00 usec
PLW1        95.00000000 W
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2            80.00 usec
PLW2        19.00000000 W
PLW12        0.42750001 W
PLW13        0.27360001 W

F2 - Processing parameters
SI                32768
SF          125.7576790 MHz
WDW                EM
SSB       0
LB               6.00 Hz
GB        0
PC               1.40
```

1H{11B} NMR, 2–Cl CB11, 15 mg in 0.6 ml acetonitrile–d3, T= 23 C
Solvent residual peak ○

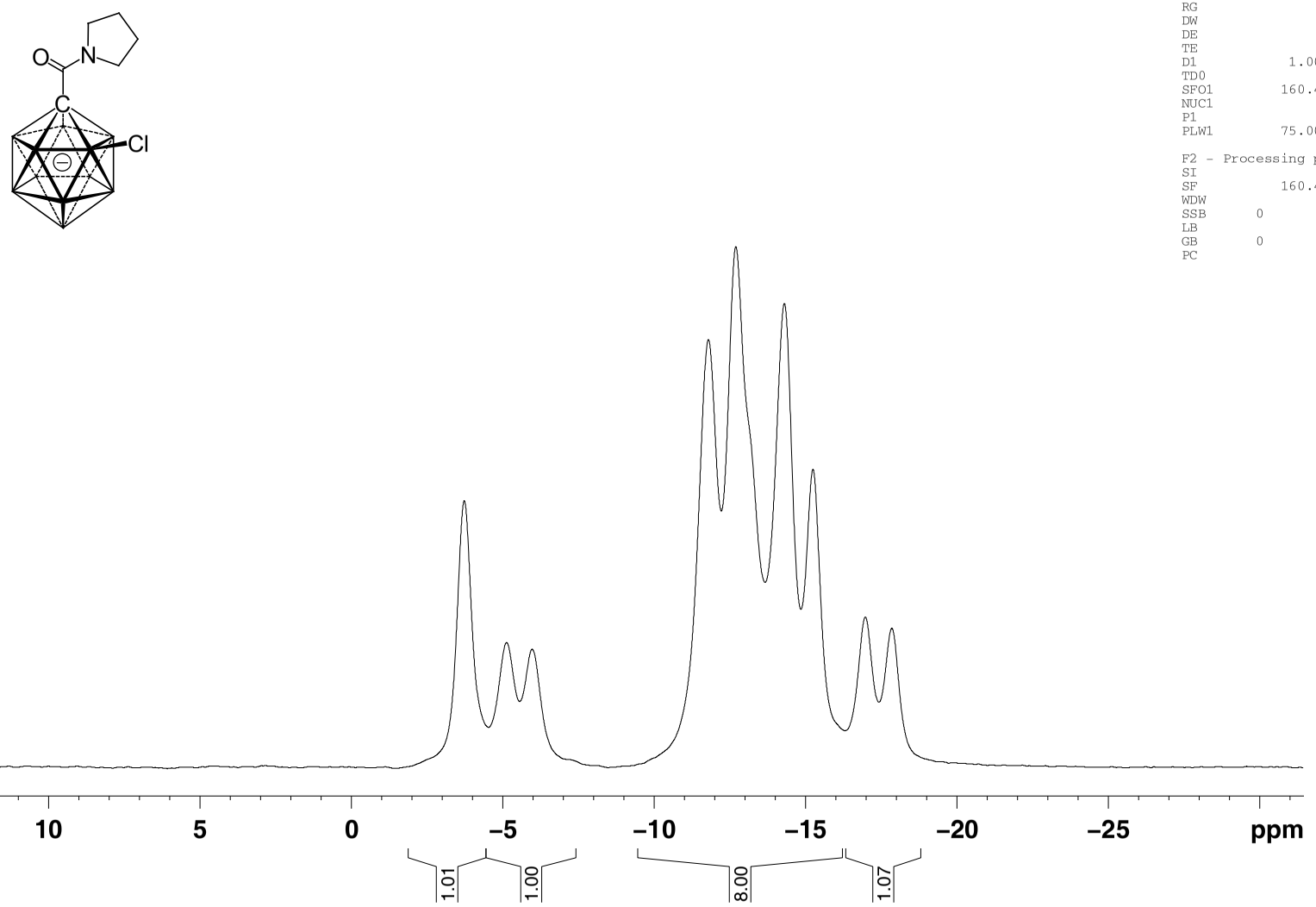


Et_4N^+

NMR67


```
Current Data Parameters
NAME      20161116-yjs-0078
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20161116
Time                 21.31
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgig30
TD                   65536
SOLVENT              CD3CN
NS                      16
DS                       0
SWH              12500.000 Hz
FIDRES               0.190735 Hz
AQ                2.6214399 sec
RG                     114
DW                  40.000 usec
DE                   6.50 usec
TE                  296.4 K
D1             5.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                   11.60 usec
PLW1          19.00000000 W
SFO1         500.1335009 MHz

======== CHANNEL f2 ========
CPDPRG[2                garp
NUC2                    11B
PCPD2                100.00 usec
PLW2          95.00000000 W
PLW12          1.63030005 W
SFO2         160.4615690 MHz

F2 - Processing parameters
SI                     65536
SF           500.1300157 MHz
WDW                     EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.00
```

11B NMR, 2–Cl CB11, 20 mg dissolved in 0.6 mL DMSO–d6, T = 80 C

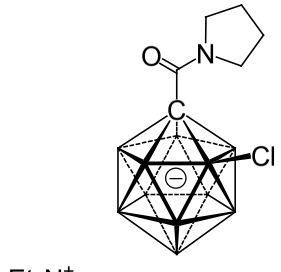


Et_4N^+

NMR68

```
Current Data Parameters
NAME      20141215-yjs-0078
EXPNO                  202
PROCNO                   1

F2 - Acquisition Parameters
Date_           20141215
Time               16.27
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG              zg
TD                64098
SOLVENT            DMSO
NS                   32
DS                    0
SWH           32051.281 Hz
FIDRES         0.500036 Hz
AQ            0.9999288 sec
RG                  203
DW               15.600 usec
DE               16.00 usec
TE               352.0 K
D1          1.00000000 sec
TD0                   1
SFO1        160.4615792 MHz
NUC1                11B
P1               10.00 usec
PLW1        75.00000000 W

F2 - Processing parameters
SI                32768
SF          160.4615790 MHz
WDW                  EM
SSB       0
LB               20.00 Hz
GB        0
PC                1.40
```

11B{1H} NMR, 2-Cl CB11, 20 mg dissolved in 0.6 mL DMSO-d6, quartz NMR tube, T = 80 C

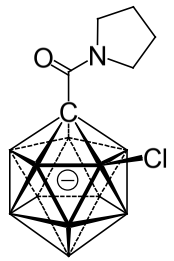


Et_4N^+

NMR69

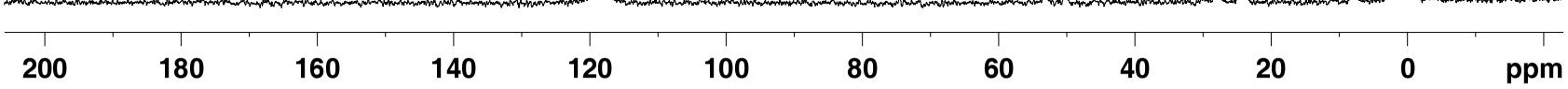

```
Current Data Parameters
NAME      20141215-yjs-0078
EXPNO                  203
PROCNO                   1

F2 - Acquisition Parameters
Date_           20141215
Time               16.30
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD               192304
SOLVENT             DMSO
NS                    32
DS                     0
SWH            32051.281 Hz
FIDRES          0.166670 Hz
AQ              2.9999423 sec
RG                   203
DW                15.600 usec
DE                 6.50 usec
TE                352.0 K
D1           5.00000000 sec
d11          0.03000000 sec
DELTA        4.90000010 sec
TD0                    1
SFO1         160.4615792 MHz
NUC1                11B
P1                10.00 usec
PLW1         75.00000000 W
SFO2         500.1330885 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2         19.00000000 W
PLW12         0.42750001 W
PLW13         0.27360001 W

F2 - Processing parameters
SI                 32768
SF           160.4615790 MHz
WDW                   EM
SSB      0
LB                20.00 Hz
GB       0
PC                 1.40
```

13C{1H} NMR, 2-Cl CB11, 12 mg dissolved in 0.6 mL acetonitrile-d3, T = 23 C
Solvent peak ○



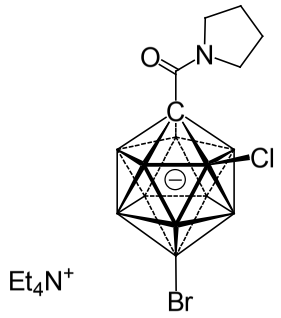
Et_4N^+

NMR70




```
Current Data Parameters
NAME      20160729-yjs-0078
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_             20160729
Time                  6.45
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CD3CN
NS                    4096
DS                       4
SWH           37878.789 Hz
FIDRES         0.577984 Hz
AQ             0.8650752 sec
RG                     203
DW                13.200 usec
DE                 6.50 usec
TE                298.4 K
D1           1.50000000 sec
D11          0.03000000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.50 usec
PLW1         95.00000000 W
SFO1        125.7716224 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PLW2         19.00000000 W
PLW12         0.39947000 W
PLW13         0.25566000 W
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                   32768
SF          125.7576708 MHz
WDW                     EM
SSB       0
LB                 10.00 Hz
GB        0
PC                  1.40
```

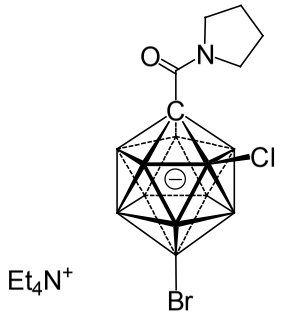
1H{11B} NMR, 12-Br,2-Cl CB11, 15 mg in 0.6 ml acetonitrile-d3, T= 23 C
Solvent residual peak ○



NMR71

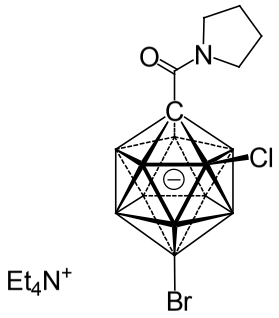

```
Current Data Parameters
NAME      20161116-yjs-0118
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20161116
Time                 21.36
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgig30
TD                   65536
SOLVENT              CD3CN
NS                      16
DS                       0
SWH           12500.000 Hz
FIDRES            0.190735 Hz
AQ                2.6214399 sec
RG                     114
DW                  40.000 usec
DE                   6.50 usec
TE                  296.4 K
D1             5.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                   11.60 usec
PLW1          19.00000000 W
SFO1         500.1335009 MHz

======== CHANNEL f2 ========
CPDPRG[2                garp
NUC2                   11B
PCPD2               100.00 usec
PLW2          95.00000000 W
PLW12          1.63030005 W
SFO2         160.4615690 MHz

F2 - Processing parameters
SI                   65536
SF           500.1300157 MHz
WDW                     EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.00
```

11B NMR, 12–Br, 2–Cl CB11, 15 mg in 0.6 mL acetone–d6 T = 19 C




```
Current Data Parameters
NAME      20151209-yjs-0118
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20151209
Time                 17.19
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG                 zg
TD                   64098
SOLVENT            Acetone
NS                       256
DS                         0
SWH              32051.281 Hz
FIDRES            0.500036 Hz
AQ               0.9999288 sec
RG                       203
DW                  15.600 usec
DE                   6.50 usec
TE                  295.0 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                    11B
P1                   10.00 usec
PLW1          75.00000000 W
SFO1          160.4615792 MHz

F2 - Processing parameters
SI                      32768
SF            160.4615993 MHz
WDW                     EM
SSB       0
LB                   30.00 Hz
GB        0
PC                    1.40
```

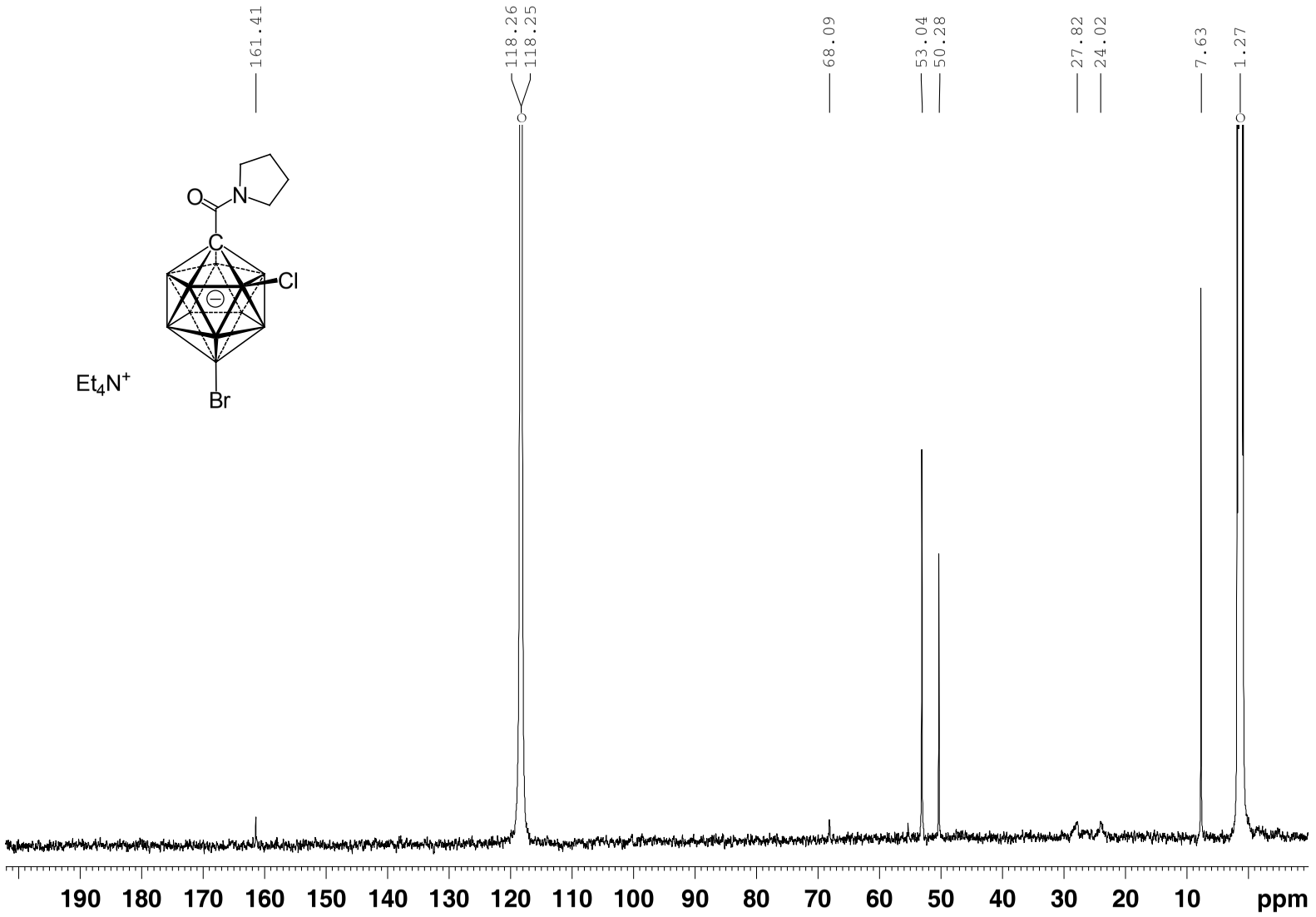
11B{1H} NMR, 12-Br, 2-Cl CB11, 15 mg in 0.6 mL acetone-d6 T = 19 C



NMR73


```
Current Data Parameters
NAME      20151209-yjs-0118
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_           20151209
Time               17.28
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  256
DS                    0
SWH           32051.281 Hz
FIDRES         0.489064 Hz
AQ            1.0223616 sec
RG                  203
DW               15.600 usec
DE                6.50 usec
TE               295.8 K
D1            2.00000000 sec
D11           0.03000000 sec

======== CHANNEL f1 ========
NUC1                11B
P1                10.00 usec
PLW1         75.00000000 W
SFO1        160.4615792 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2             80.00 usec
PLW2         19.00000000 W
PLW12         0.42750001 W
PLW13         0.27360001 W
SFO2        500.1330885 MHz

F2 - Processing parameters
SI                 32768
SF          160.4615993 MHz
WDW                   EM
SSB       0
LB                30.00 Hz
GB        0
PC                 1.40
```

13C{1H} NMR, 12–Br, 2–Cl CB11, 15 mg in 0.6 mL acetonitrile–d3 T = 23 C
Solvent peak ○

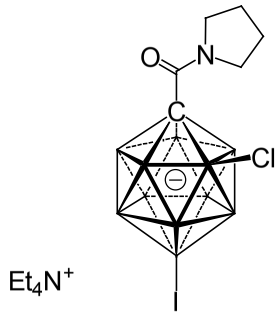


NMR74

```
Current Data Parameters
NAME      20160727-yjs-0118
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_           20160727
Time              22.55
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CD3CN
NS                 4096
DS                    4
SWH           37878.789 Hz
FIDRES         0.577984 Hz
AQ             0.8650752 sec
RG                  203
DW               13.200 usec
DE                6.50 usec
TE                298.8 K
D1           1.50000000 sec
D11          0.03000000 sec

======== CHANNEL f1 ========
NUC1               13C
P1               10.50 usec
PLW1        95.00000000 W
SFO1       125.7716224 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2            80.00 usec
PLW2        19.00000000 W
PLW12        0.39947000 W
PLW13        0.25566000 W
SFO2       500.1320005 MHz

F2 - Processing parameters
SI                32768
SF          125.7576701 MHz
WDW                EM
SSB       0
LB               6.00 Hz
GB        0
PC               1.40
```

1H{11B} NMR, Monochloro– Pyrrolidine amdie,12–I CB11, 12 mg in 0.6 mL acetonitrile–d3, T = 23 C
Solvent residual peak ○

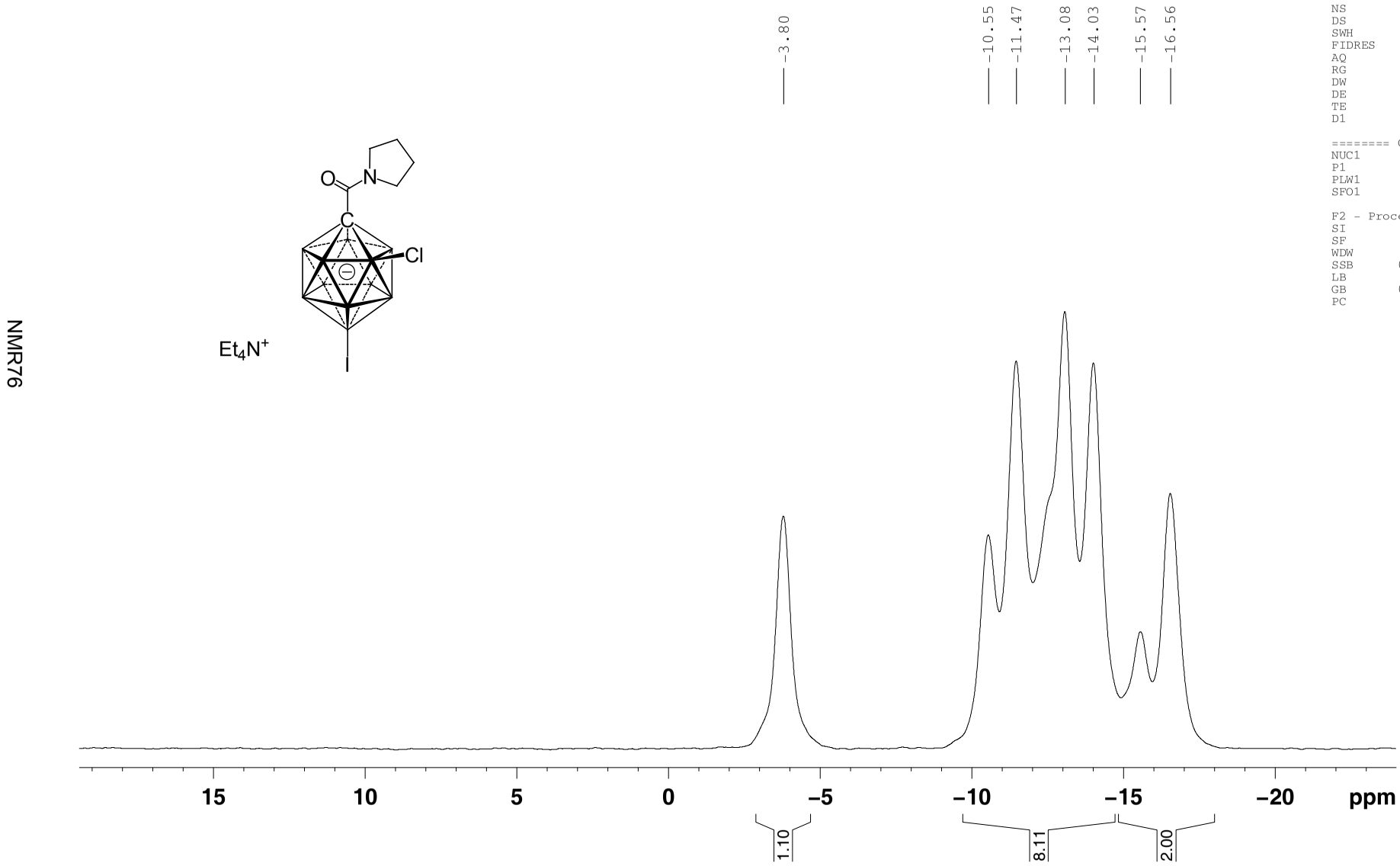
NMR75



Et_4N^+


```
Current Data Parameters
NAME      20160721-yjs-0159
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20160721
Time              22.27
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgig30
TD                65536
SOLVENT           CD3CN
NS                   16
DS                    0
SWH           12500.000 Hz
FIDRES        0.190735 Hz
AQ            2.6214399 sec
RG                  114
DW               40.000 usec
DE                6.50 usec
TE               299.4 K
D1           5.00000000 sec
D11          0.03000000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                11.60 usec
PLW1        19.00000000 W
SFO1        500.1335009 MHz

======== CHANNEL f2 ========
CPDPRG[2          garp
NUC2               11B
PCPD2           100.00 usec
PLW2        95.00000000 W
PLW12        1.63030005 W
SFO2        160.4615690 MHz

F2 - Processing parameters
SI                65536
SF          500.1300155 MHz
WDW                 EM
SSB      0
LB                1.00 Hz
GB       0
PC                1.00
```

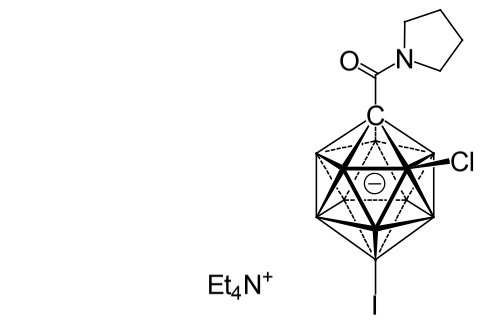
11B NMR, Monochloro– Pyrrolidine amdie,12–I CB11, 12 mg in 0.6 mL acetonitrile–d3, T = 23 C



NMR76

```
Current Data Parameters
NAME      20160721-yjs-0159
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_           20160721
Time              22.53
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               64098
SOLVENT          CD3CN
NS                  64
DS                   0
SWH          32051.281 Hz
FIDRES        0.500036 Hz
AQ            0.9999288 sec
RG                 203
DW              15.600 usec
DE               6.50 usec
TE               299.2 K
D1          1.00000000 sec

======== CHANNEL f1 ========
NUC1               11B
P1               13.10 usec
PLW1       95.00000000 W
SFO1       160.4615792 MHz

F2 - Processing parameters
SI               32768
SF          160.4615790 MHz
WDW                EM
SSB       0
LB              20.00 Hz
GB        0
PC               1.40
```

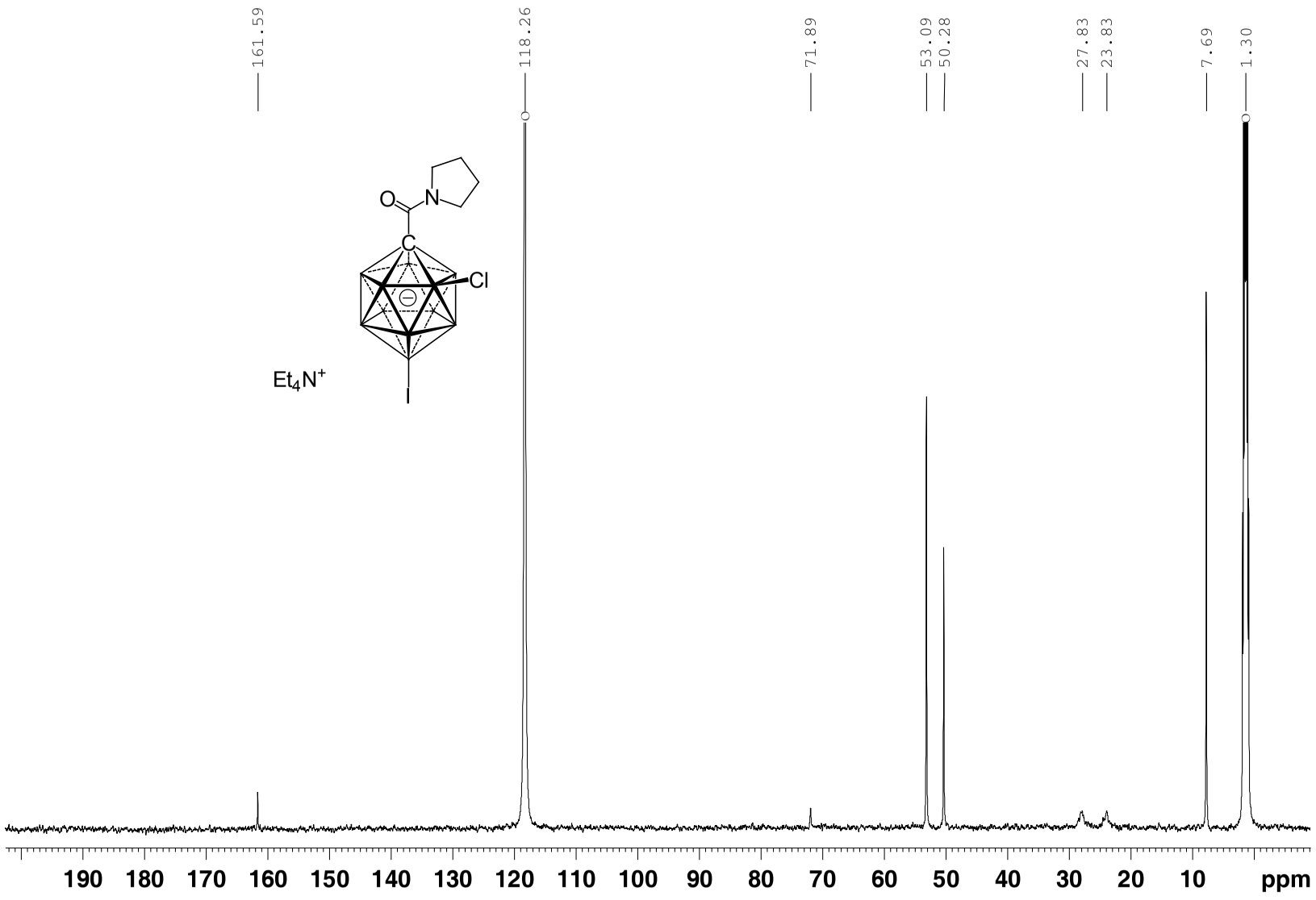
11B{1H} NMR, Monochloro– Pyrrolidine amdie,12–I CB11, 12 mg in 0.6 mL acetonitrile–d3, T = 23 C



NMR77

```
Current Data Parameters
NAME      20160721-yjs-0159
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_           20160721
Time              22.56
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD                65536
SOLVENT           CD3CN
NS                   64
DS                    0
SWH           32051.281 Hz
FIDRES         0.489064 Hz
AQ             1.0223616 sec
RG                  203
DW               15.600 usec
DE                6.50 usec
TE                299.6 K
D1           1.00000000 sec
D11          0.03000000 sec

======== CHANNEL f1 ========
NUC1                11B
P1                13.10 usec
PLW1        95.00000000 W
SFO1       160.4615790 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2            80.00 usec
PLW2        19.00000000 W
PLW12        0.39947000 W
PLW13        0.25566000 W
SFO2       500.1325007 MHz

F2 - Processing parameters
SI                32768
SF         160.4615790 MHz
WDW                EM
SSB       0
LB               20.00 Hz
GB        0
PC                1.40
```

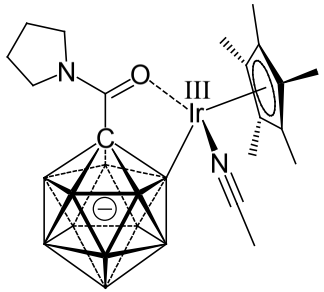
13C{1H} NMR, Monochloro– Pyrrolidine amdie,12–I CB11, 12 mg in 0.6 mL acetonitrile–d3, T = 23 C
Solvent peak



NMR78

```
Current Data Parameters
NAME      20160721-yjs-0159
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_              20160721
Time                 23.11
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CD3CN
NS                    2048
DS                       4
SWH             37878.789 Hz
FIDRES           0.577984 Hz
AQ              0.8650752 sec
RG                     203
DW                13.200 usec
DE                 6.50 usec
TE                 299.8 K
D1            1.50000000 sec
D11           0.03000000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                  10.50 usec
PLW1          95.00000000 W
SFO1         125.7716224 MHz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                   1H
PCPD2               80.00 usec
PLW2          19.00000000 W
PLW12          0.39947000 W
PLW13          0.25566000 W
SFO2         500.1320005 MHz

F2 - Processing parameters
SI                   32768
SF           125.7576687 MHz
WDW                     EM
SSB       0
LB                   8.00 Hz
GB        0
PC                   1.40
```

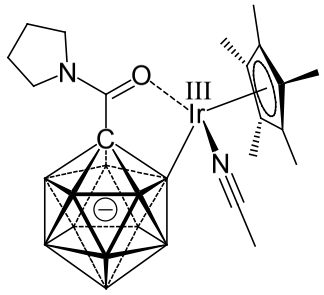
1H{11B} NMR Ir intermediate, Pyrrolidine amide CB11, 12 mg in 0.6 ml acetonitrile-d3, T=23 C
Solvent residual peak ○



NMR79


```
Current Data Parameters
NAME      20161019-yjs-0090
EXPNO                     6
PROCNO                    1

F2 - Acquisition Parameters
Date_              20161019
Time                   2.11
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CD3CN
NS                      16
DS                       2
SWH             15000.000 Hz
FIDRES              0.228882 Hz
AQ                2.1845334 sec
RG                      161
DW                 33.333 usec
DE                  6.50 usec
TE                  297.3 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                   11.60 usec
PLW1          19.00000000 W
SFO1          500.1340010 MHz

F2 - Processing parameters
SI                    65536
SF            500.1300155 MHz
WDW                      EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```

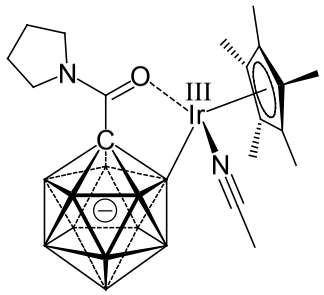
11B NMR Ir intermediate, Pyrrolidine amide CB11, 12 mg in 0.6 ml acetonitrile–d3, T=23 C



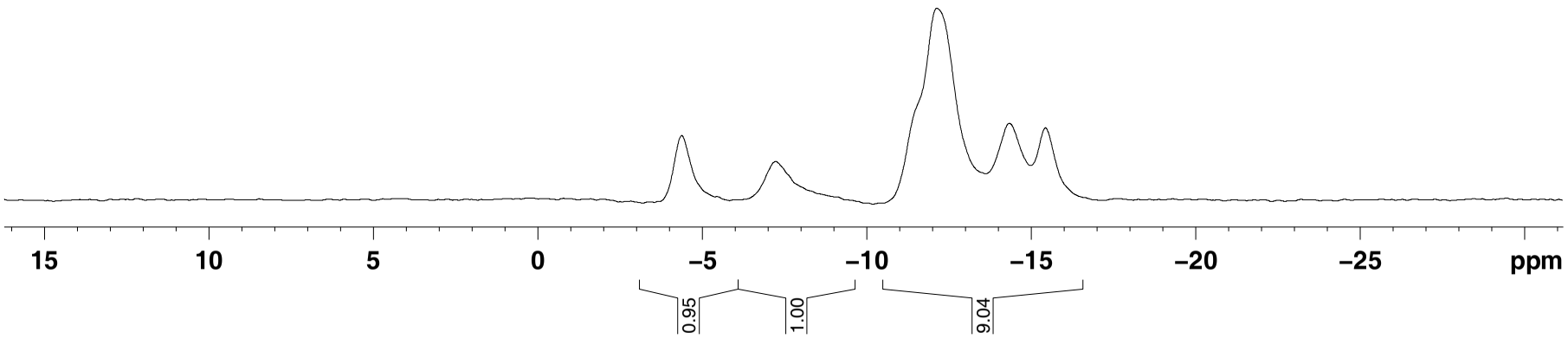

```
Current Data Parameters
NAME      20161019-yjs-0090
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_           20161019
Time                2.06
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 64098
SOLVENT            CD3CN
NS                    64
DS                     0
SWH            32051.281 Hz
FIDRES          0.500036 Hz
AQ             0.9999288 sec
RG                   203
DW                15.600 usec
DE                 6.50 usec
TE                297.4 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                11B
P1                13.10 usec
PLW1        95.00000000 W
SFO1        160.4615792 MHz

F2 - Processing parameters
SI                 32768
SF          160.4615790 MHz
WDW                   EM
SSB       0
LB                20.00 Hz
GB        0
PC                 1.40
```

NMR80

11B{1H} NMR Ir intermediate, Pyrrolidine amide CB11, 12 mg in 0.6 ml acetonitrile-d3, T=23 C



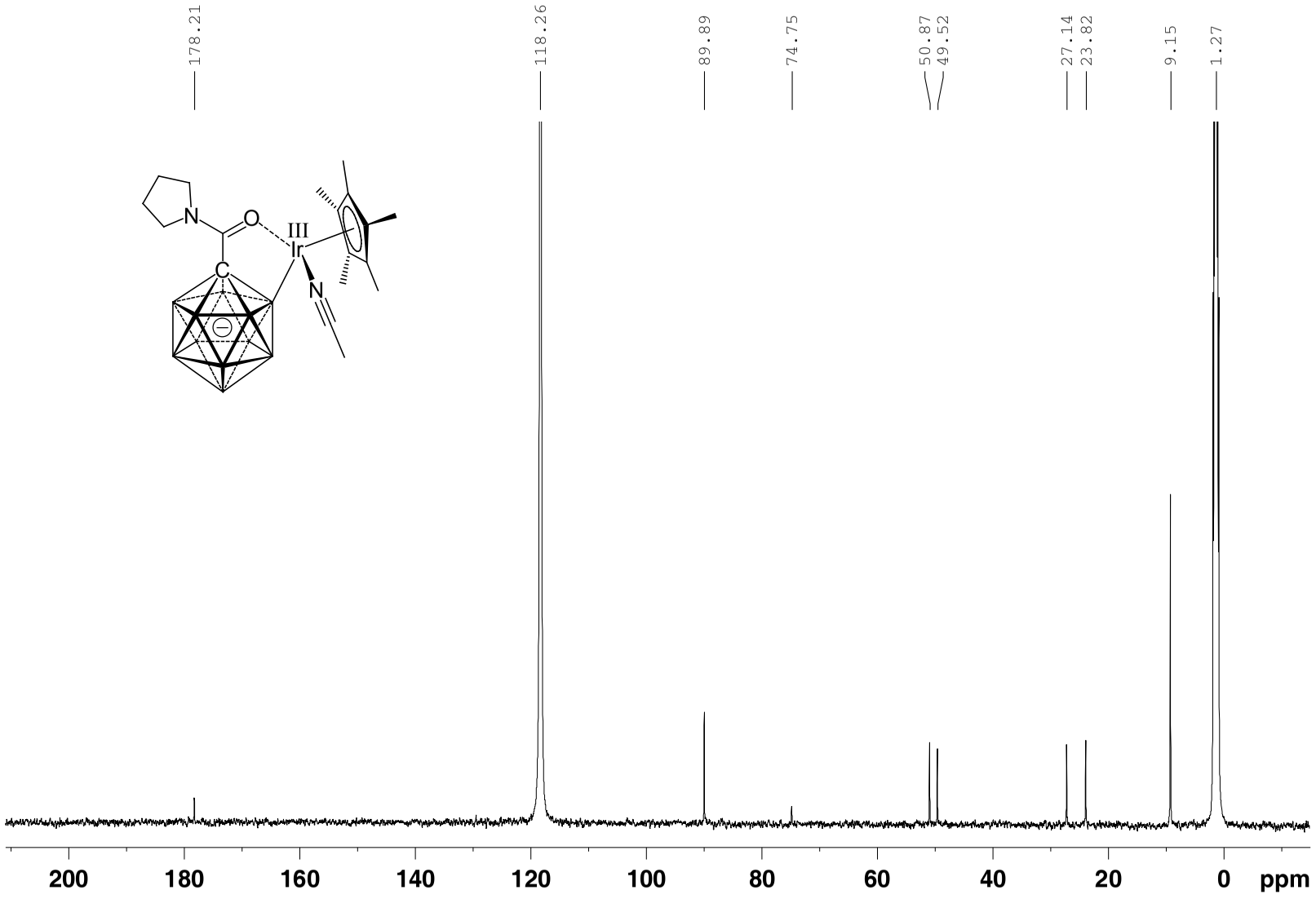
-4.31
-7.26
-12.05
-14.32
-15.51



NMR81

```
Current Data Parameters
NAME      20161019-yjs-0090
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_           20161019
Time                2.09
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CD3CN
NS                    64
DS                     0
SWH            32051.281 Hz
FIDRES          0.489064 Hz
AQ             1.0223616 sec
RG                   203
DW                15.600 usec
DE                 6.50 usec
TE                297.8 K
D1           1.00000000 sec
D11          0.03000000 sec

======== CHANNEL f1 ========
NUC1                  11B
P1                  13.10 usec
PLW1         95.00000000 W
SFO1        160.4615790 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PLW2         19.00000000 W
PLW12         0.39947000 W
PLW13         0.25566000 W
SFO2        500.1325007 MHz

F2 - Processing parameters
SI                 32768
SF          160.4615790 MHz
WDW                   EM
SSB       0
LB                 20.00 Hz
GB        0
PC                  1.40
```

13C{1H} NMR Ir intermediate, Pyrrolidine amide CB11, 12 mg in 0.6 ml acetonitrile-d3, T=23 C

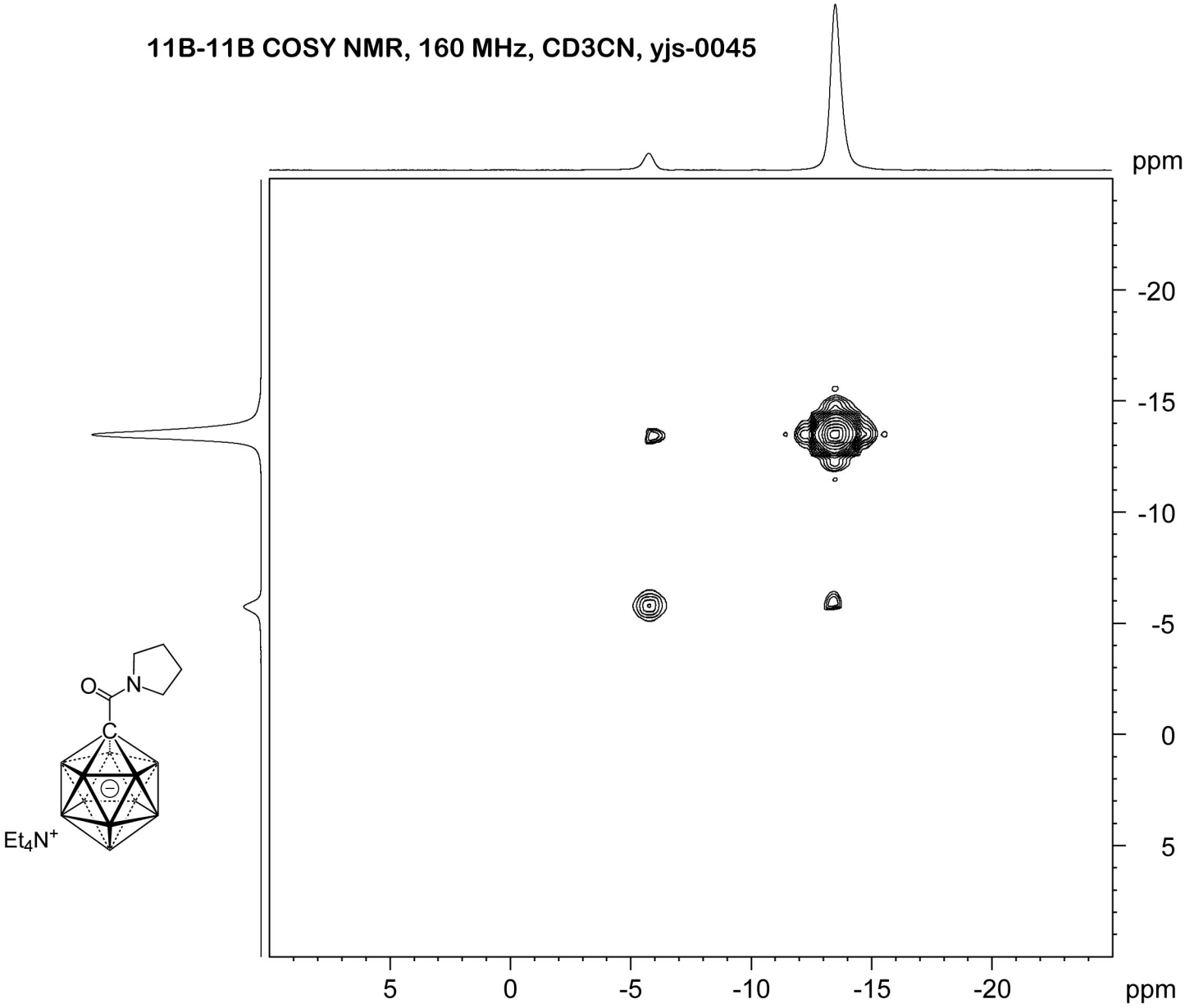


NMR82

```
Current Data Parameters
NAME      20161019-yjs-0090
EXPNO                    5
PROCNO                   1

F2 - Acquisition Parameters
Date_             20161019
Time                  1.58
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CD3CN
NS                    4096
DS                       4
SWH              37878.789 Hz
FIDRES           0.577984 Hz
AQ               0.8650752 sec
RG                     203
DW                13.200 usec
DE                 6.50 usec
TE                 297.7 K
D1             1.50000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.50 usec
PLW1         95.00000000 W
SFO1        125.7716224 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PLW2         19.00000000 W
PLW12         0.39947000 W
PLW13         0.25566000 W
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                   32768
SF          125.7576698 MHz
WDW                     EM
SSB       0
LB                 7.00 Hz
GB        0
PC                 1.40
```

NMR83

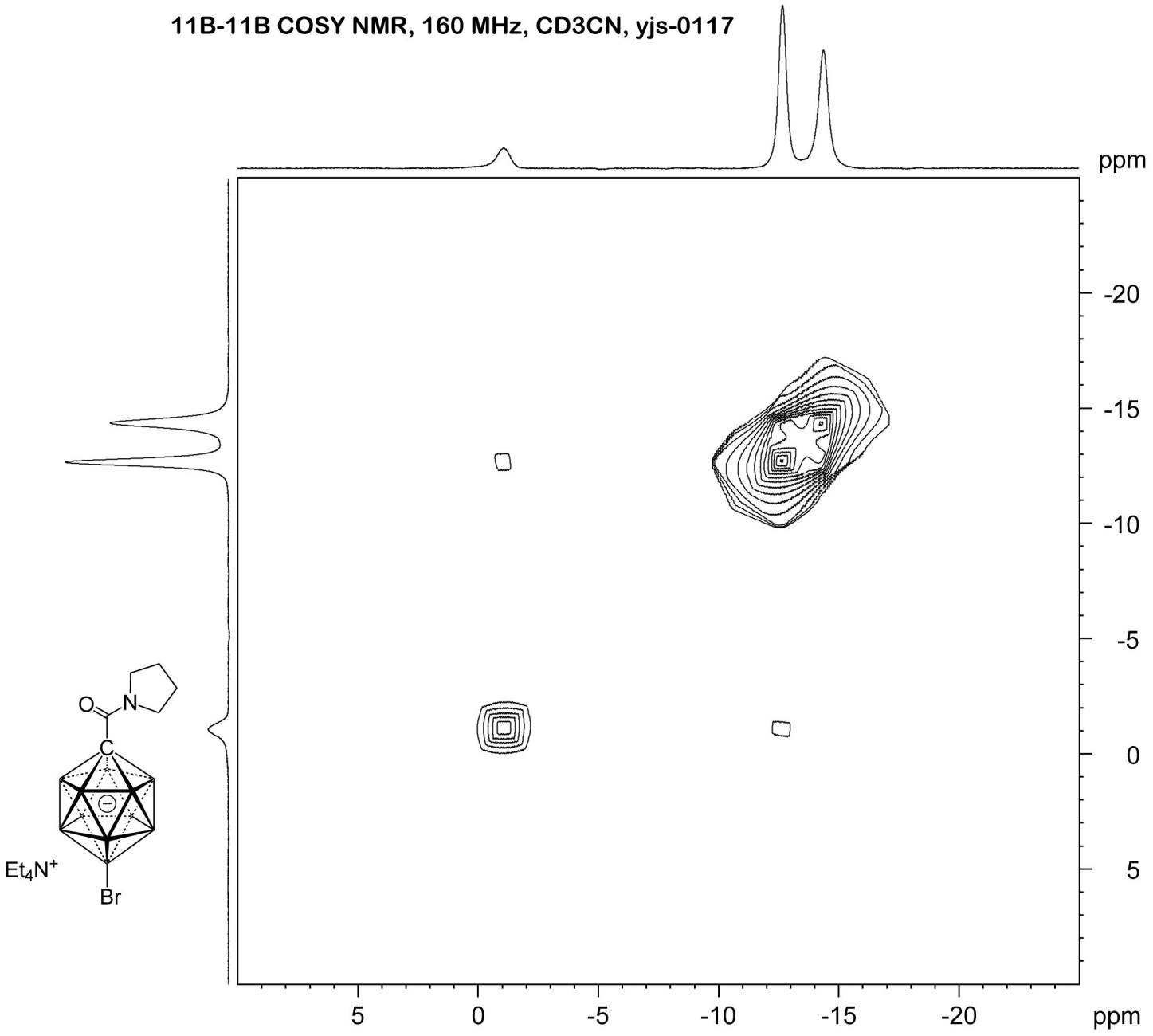
11B-11B COSY NMR, 160 MHz, CD3CN, yjs-0045




```
Current Data Parameters
NAME      YJS_pub01_20160728-2D-yjs-py
EXPNO                 100
PROCNO                  1

F2 - Acquisition Parameters
Date_              20160728
Time                  22.29
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG      cosygpqfdec
TD                    1922
SOLVENT              CD3CN
NS                      32
DS                      16
SWH                7211.539 Hz
FIDRES             3.752101 Hz
AQ                 0.1332587 sec
RG                      203
DW                    69.333 usec
DE                     6.50 usec
TE                    298.9 K
D0                 0.00000300 sec
D1                 0.30000001 sec
D11                0.03000000 sec
D13                0.00000400 sec
D16                0.00020000 sec
IN0                0.00013850 sec

======== CHANNEL f1 ========
NUC1                    11B
P0                    13.10 usec
P1                    13.10 usec
PLW1           95.00000000 W
SFO1          160.4603790 MHz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                    1H
PCPD2                 80.00 usec
PLW2           19.00000000 W
PLW12           0.39947000 W
SFO2          500.1312000 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]       SMSQ10.100
GPZ1                10.00 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                      64
SFO1             160.4604 MHz
FIDRES          112.823563 Hz
SW                   45.000 ppm
FnMODE                  QF

F2 - Processing parameters
SI                    4096
SF            160.4616024 MHz
WDW                     EM
SSB          0
LB                   20.00 Hz
GB           0
PC                    1.40

F1 - Processing parameters
SI                    1024
MC2                     QF
SF            160.4615959 MHz
WDW                   SINE
SSB          0
LB           0 Hz
GB           0
```

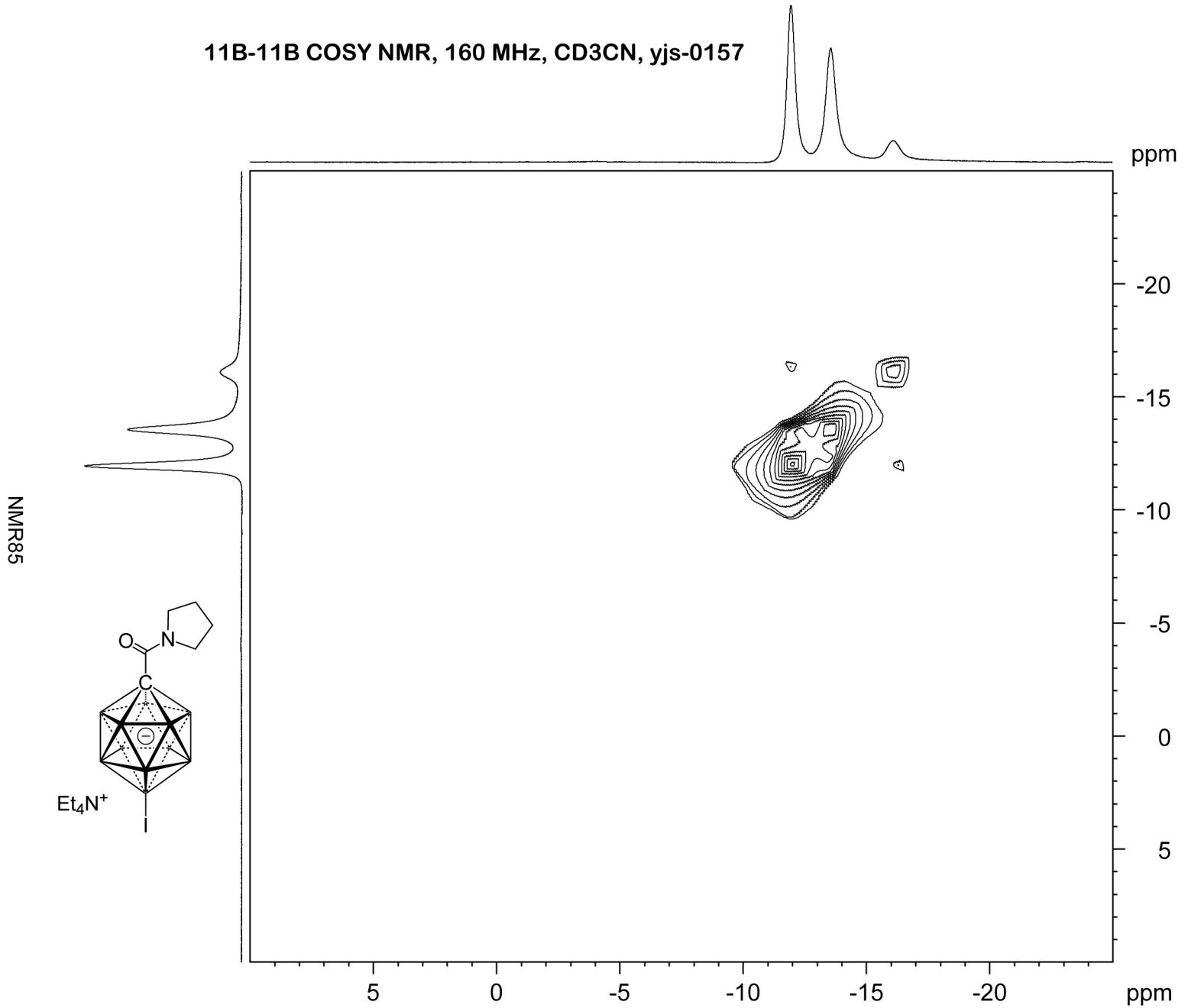
NMR84

11B-11B COSY NMR, 160 MHz, CD3CN, yjs-0117




```
Current Data Parameters
NAME      YJS_pub02_20161117-yjs-pybr--2D
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161117
Time              20.24
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG     cosygpqfdec
TD                2566
SOLVENT           CD3CN
NS                  64
DS                  16
SWH           12820.513 Hz
FIDRES         4.996303 Hz
AQ            0.1000740 sec
RG                  203
DW               39.000 usec
DE                6.50 usec
TE                297.1 K
D0           0.00000300 sec
D1           0.30000001 sec
D11          0.03000000 sec
D13          0.00000400 sec
D16          0.00020000 sec
IN0          0.00007790 sec

======== CHANNEL f1 ========
NUC1                11B
P0                13.10 usec
P1                13.10 usec
PLW1         95.00000000 W
SFO1        160.4610976 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2             80.00 usec
PLW2         19.00000000 W
PLW12         0.42750001 W
SFO2        500.1312000 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]     SMSQ10.100
GPZ1              10.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                  39
SFO1           160.4611 MHz
FIDRES        658.299316 Hz
SW                80.000 ppm
FnMODE              QF

F2 - Processing parameters
SI                4096
SF           160.4615932 MHz
WDW                EM
SSB       0
LB                20.00 Hz
GB        0
PC                 1.40

F1 - Processing parameters
SI                1024
MC2                QF
SF           160.4615986 MHz
WDW               SINE
SSB       0
LB        0 Hz
GB        0
```

11B-11B COSY NMR, 160 MHz, CD3CN, yjs-0157

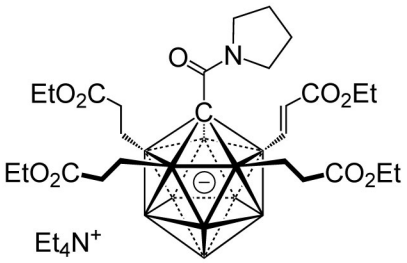


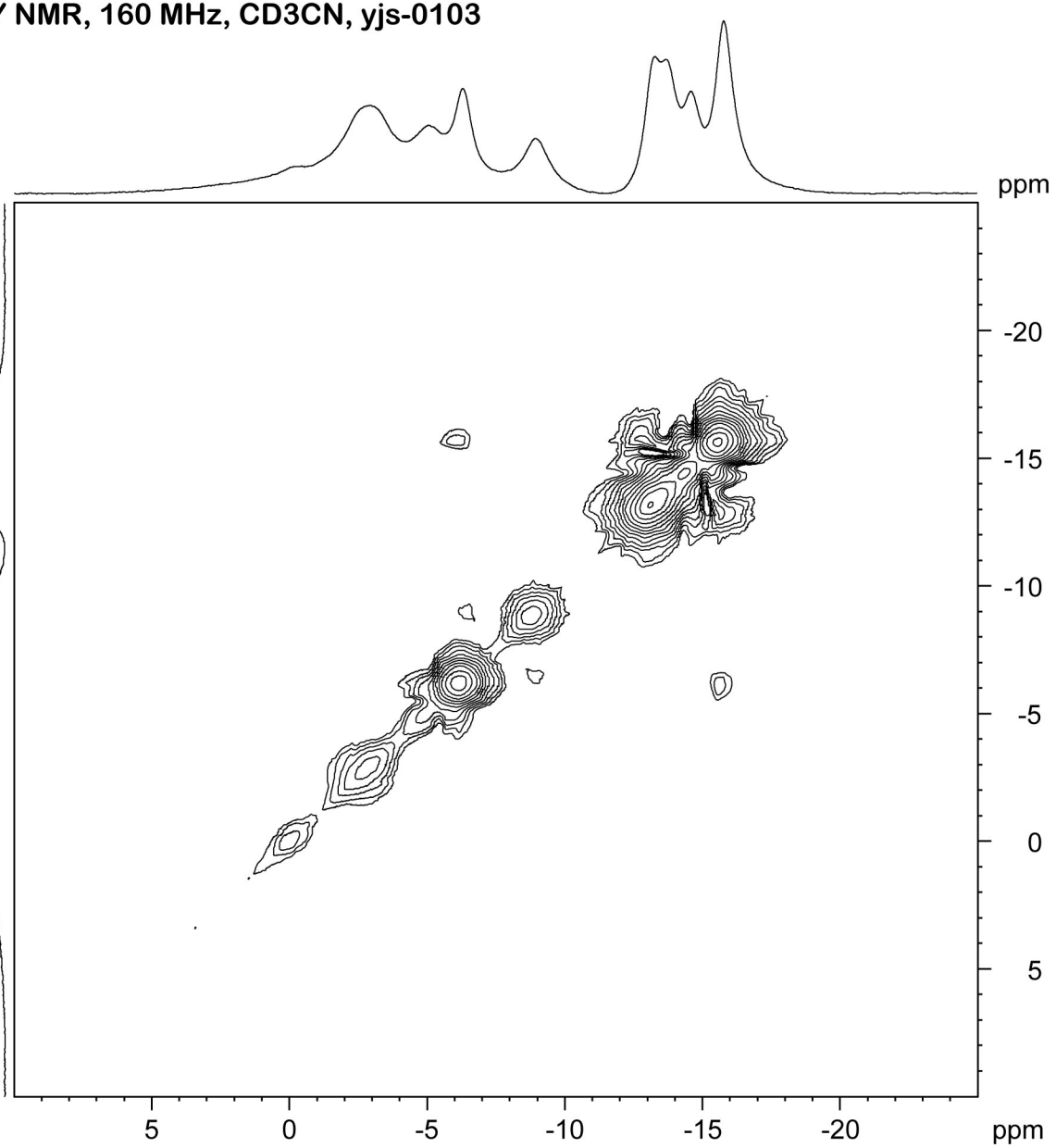
NMR85

```
Current Data Parameters
NAME      YJS_pub02_20161117-yjs-pyi--2D
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161117
Time              16.44
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG     cosygpqfdec
TD                2566
SOLVENT           CD3CN
NS                  64
DS                  16
SWH           12820.513 Hz
FIDRES         4.996303 Hz
AQ             0.1000740 sec
RG                  203
DW               39.000 usec
DE                6.50 usec
TE                296.8 K
D0            0.00000300 sec
D1            0.30000001 sec
D11           0.03000000 sec
D13           0.00000400 sec
D16           0.00020000 sec
IN0           0.00007790 sec

======== CHANNEL f1 ========
NUC1                11B
P0                13.10 usec
P1                13.10 usec
PLW1         95.00000000 W
SFO1        160.4610976 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2             80.00 usec
PLW2         19.00000000 W
PLW12         0.42750001 W
SFO2         500.1312000 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]     SMSQ10.100
GPZ1              10.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                  45
SFO1           160.4611 MHz
FIDRES        570.526062 Hz
SW               80.000 ppm
FnMODE              QF

F2 - Processing parameters
SI                4096
SF           160.4616085 MHz
WDW                EM
SSB       0
LB                20.00 Hz
GB        0
PC                 1.40

F1 - Processing parameters
SI                1024
MC2                QF
SF           160.4616130 MHz
WDW               SINE
SSB       0
LB        0 Hz
GB        0
```

11B-11B COSY NMR, 160 MHz, CD3CN, yjs-0103

NMR86

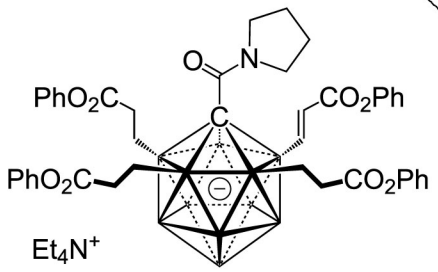


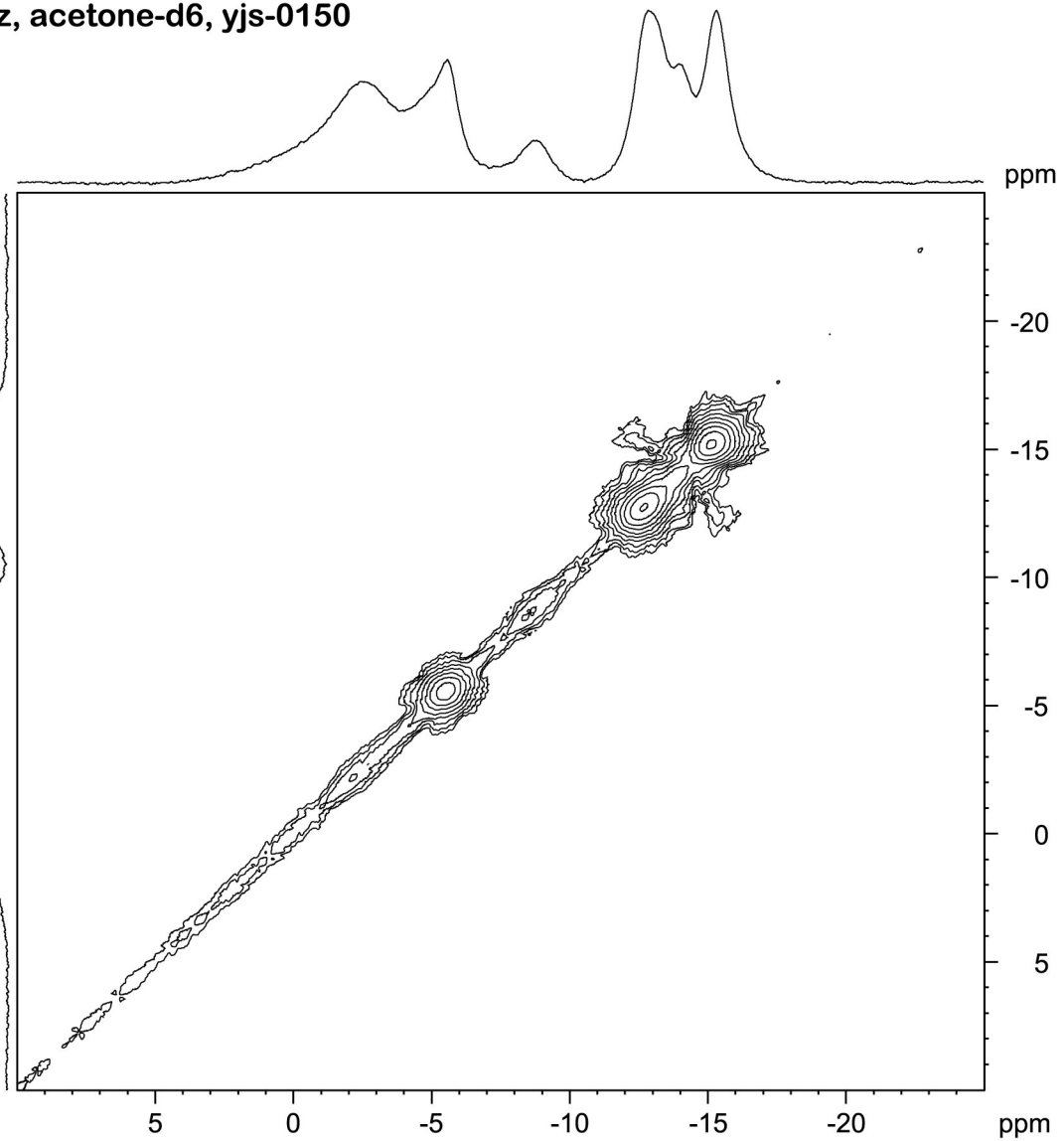



```
Current Data Parameters
NAME      YJS_pub01_20160728-2D-yjs-4Et
EXPNO                 100
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160728
Time              20.10
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG     cosygpqfdec
TD                1922
SOLVENT           CD3CN
NS                  32
DS                  16
SWH            7211.539 Hz
FIDRES         3.752101 Hz
AQ             0.1332587 sec
RG                203
DW               69.333 usec
DE                6.50 usec
TE                299.0 K
D0           0.00000300 sec
D1           0.30000001 sec
D11          0.03000000 sec
D13          0.00000400 sec
D16          0.00020000 sec
IN0          0.00013850 sec

======== CHANNEL f1 ========
NUC1                11B
P0                13.10 usec
P1                13.10 usec
PLW1        95.00000000 W
SFO1       160.4603790 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2             80.00 usec
PLW2        19.00000000 W
PLW12        0.39947000 W
SFO2       500.1312000 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPZ1              10.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                  64
SFO1           160.4604 MHz
FIDRES        225.647125 Hz
SW                45.000 ppm
FnMODE              QF

F2 - Processing parameters
SI                4096
SF           160.4615731 MHz
WDW                 EM
SSB       0
LB                20.00 Hz
GB        0
PC                 1.40

F1 - Processing parameters
SI                1024
MC2                 QF
SF           160.4615735 MHz
WDW               SINE
SSB       0
LB        0 Hz
GB        0
```

11B-11B COSY NMR, 160 MHz, acetone-d6, yjs-0150

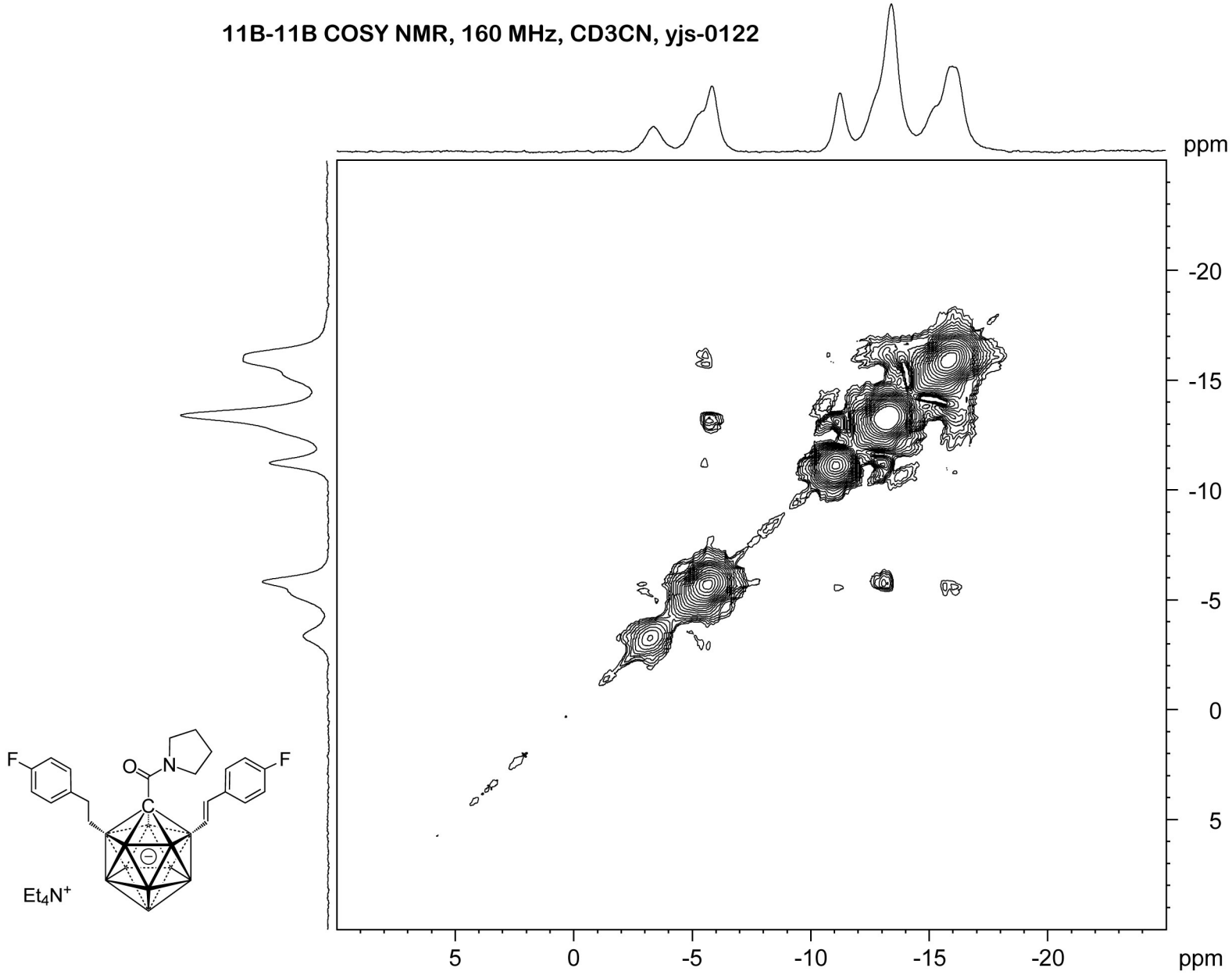
NMR87






```
Current Data Parameters
NAME      YJS_pub01_20160728-2D-yjs-4COOPh
EXPNO                  100
PROCNO                   1

F2 - Acquisition Parameters
Date_            20160728
Time                20.43
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG     cosygpqfdec
TD                  1922
SOLVENT          Acetone
NS                    32
DS                    16
SWH             7211.539 Hz
FIDRES          3.752101 Hz
AQ              0.1332587 sec
RG                  203
DW                69.333 usec
DE                 6.50 usec
TE                298.8 K
D0            0.00000300 sec
D1            0.30000001 sec
D11           0.03000000 sec
D13           0.00000400 sec
D16           0.00020000 sec
IN0           0.00013850 sec

======== CHANNEL f1 ========
NUC1                 11B
P0                 13.10 usec
P1                 13.10 usec
PLW1         95.00000000 W
SFO1        160.4603790 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PLW2         19.00000000 W
PLW12         0.39947000 W
SFO2        500.1312000 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPZ1               10.00 %
P16              1000.00 usec

F1 - Acquisition parameters
TD                    64
SFO1            160.4604 MHz
FIDRES        225.647125 Hz
SW                45.000 ppm
FnMODE                QF

F2 - Processing parameters
SI                  4096
SF           160.4615731 MHz
WDW                   EM
SSB       0
LB                20.00 Hz
GB        0
PC                 1.40

F1 - Processing parameters
SI                  1024
MC2                   QF
SF           160.4615735 MHz
WDW                 SINE
SSB       0
LB        0 Hz
GB        0
```

11B-11B COSY NMR, 160 MHz, CD3CN, yjs-0122

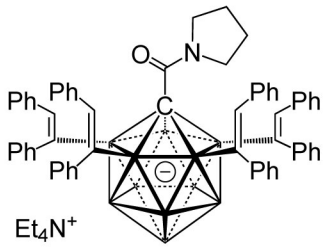


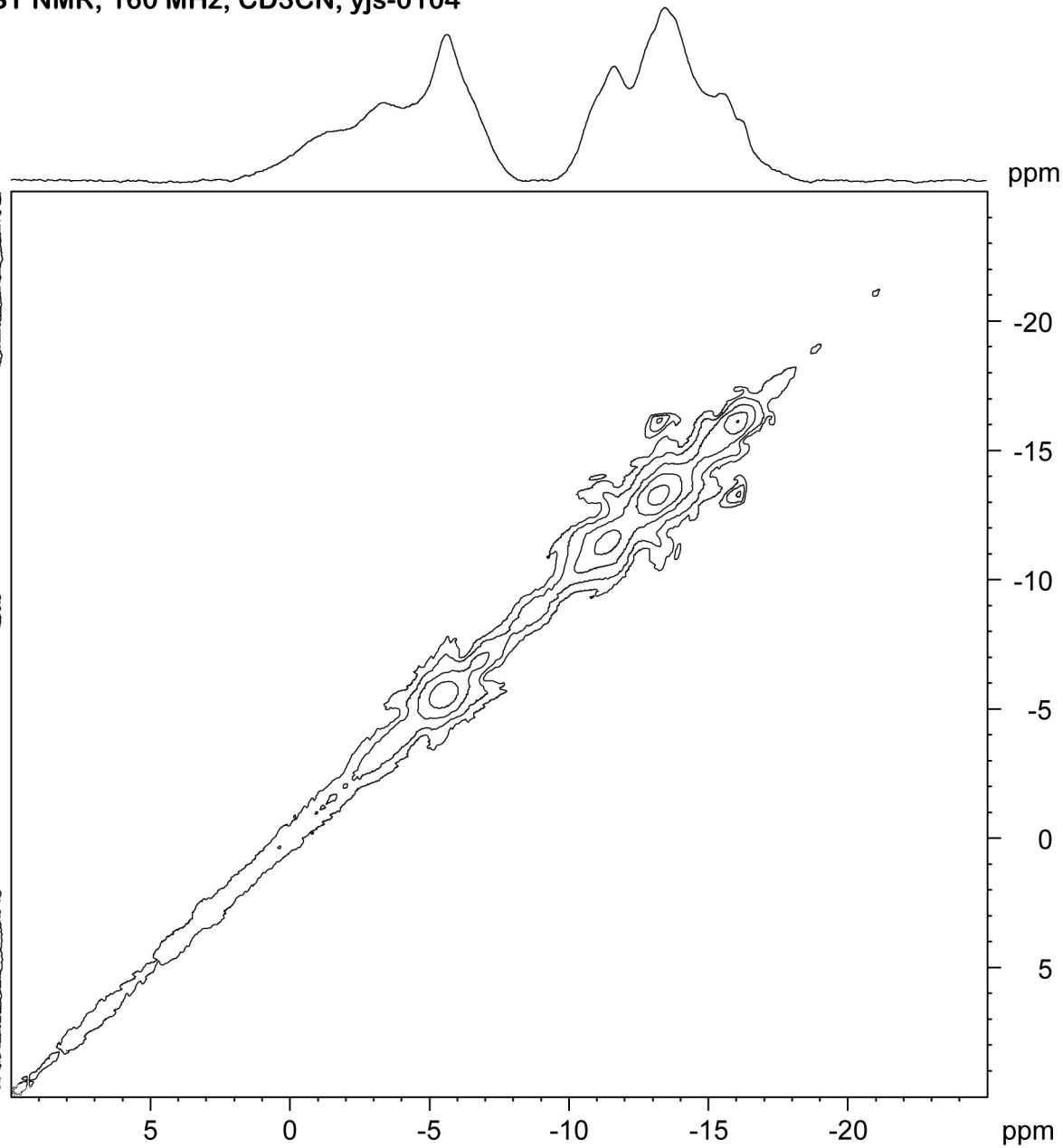
NMR88

```
Current Data Parameters
NAME      YJS_pub01_20160728-2D-yjs-2F
EXPNO                 100
PROCNO                  1

F2 - Acquisition Parameters
Date_              20160728
Time                 16.28
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG      cosygpqfdec
TD                    1922
SOLVENT              CD3CN
NS                      32
DS                      16
SWH               7211.539 Hz
FIDRES            3.752101 Hz
AQ                0.1332587 sec
RG                      203
DW                   69.333 usec
DE                    6.50 usec
TE                   299.0 K
D0              0.00000300 sec
D1              0.30000001 sec
D11             0.03000000 sec
D13             0.00000400 sec
D16             0.00020000 sec
IN0             0.00013850 sec

======== CHANNEL f1 ========
NUC1                   11B
P0                   13.10 usec
P1                   13.10 usec
PLW1          95.00000000 W
SFO1         160.4603790 MHz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                   1H
PCPD2                80.00 usec
PLW2          19.00000000 W
PLW12          0.39947000 W
SFO2         500.1312000 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPZ1                 10.00 %
P16                1000.00 usec

F1 - Acquisition parameters
TD                     64
SFO1             160.4604 MHz
FIDRES         225.647125 Hz
SW                  45.000 ppm
FnMODE                 QF

F2 - Processing parameters
SI                   4096
SF             160.4615731 MHz
WDW                    EM
SSB       0
LB                   20.00 Hz
GB        0
PC                    1.40

F1 - Processing parameters
SI                   1024
MC2                    QF
SF             160.4615735 MHz
WDW                  SINE
SSB       0
LB        0 Hz
GB        0
```

11B-11B COSY NMR, 160 MHz, CD3CN, yjs-0104

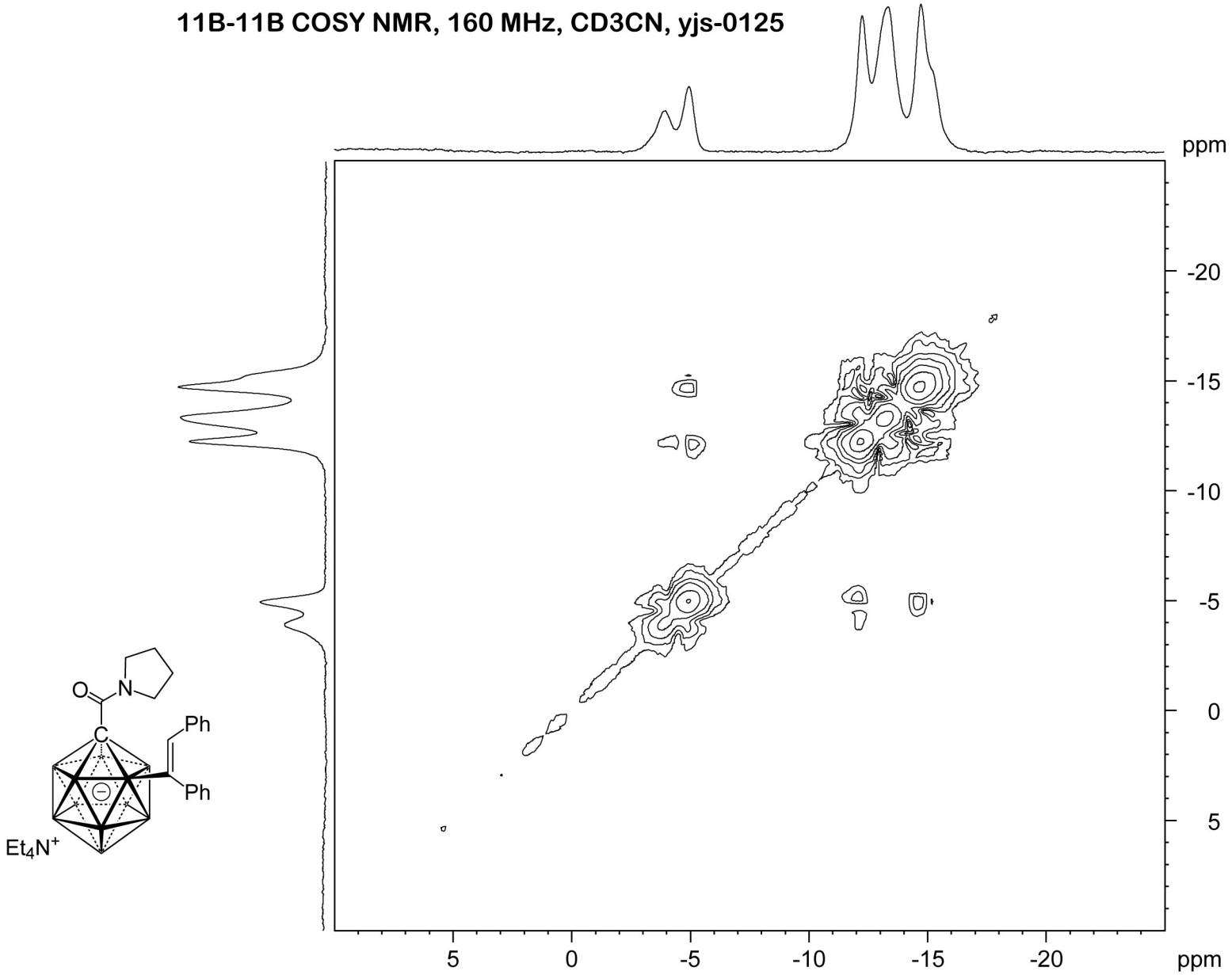
NMR89






```
Current Data Parameters
NAME      YJS_pub01_20160728-2D-yjs-4que
EXPNO                  100
PROCNO                   1

F2 - Acquisition Parameters
Date_           20160728
Time               17.22
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG     cosygpqfdec
TD                 1922
SOLVENT            CD3CN
NS                   32
DS                   16
SWH             7211.539 Hz
FIDRES          3.752101 Hz
AQ              0.1332587 sec
RG                   203
DW                69.333 usec
DE                 6.50 usec
TE                298.9 K
D0             0.00000300 sec
D1             0.30000001 sec
D11            0.03000000 sec
D13            0.00000400 sec
D16            0.00020000 sec
IN0            0.00013850 sec

======== CHANNEL f1 ========
NUC1                 11B
P0                 13.10 usec
P1                 13.10 usec
PLW1         95.00000000 W
SFO1        160.4603790 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2              80.00 usec
PLW2         19.00000000 W
PLW12         0.39947000 W
SFO2        500.1312000 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]     SMSQ10.100
GPZ1               10.00 %
P16              1000.00 usec

F1 - Acquisition parameters
TD                   64
SFO1            160.4604 MHz
FIDRES        225.647125 Hz
SW                45.000 ppm
FnMODE               QF

F2 - Processing parameters
SI                 4096
SF            160.4615731 MHz
WDW                  EM
SSB       0
LB                20.00 Hz
GB        0
PC                 1.40

F1 - Processing parameters
SI                 1024
MC2                  QF
SF            160.4615735 MHz
WDW                SINE
SSB       0
LB        0 Hz
GB        0
```

11B-11B COSY NMR, 160 MHz, CD3CN, yjs-0125

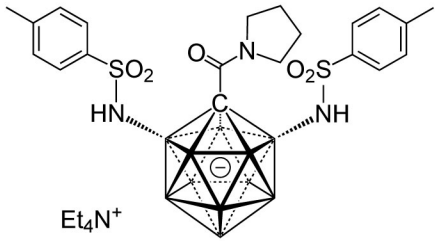


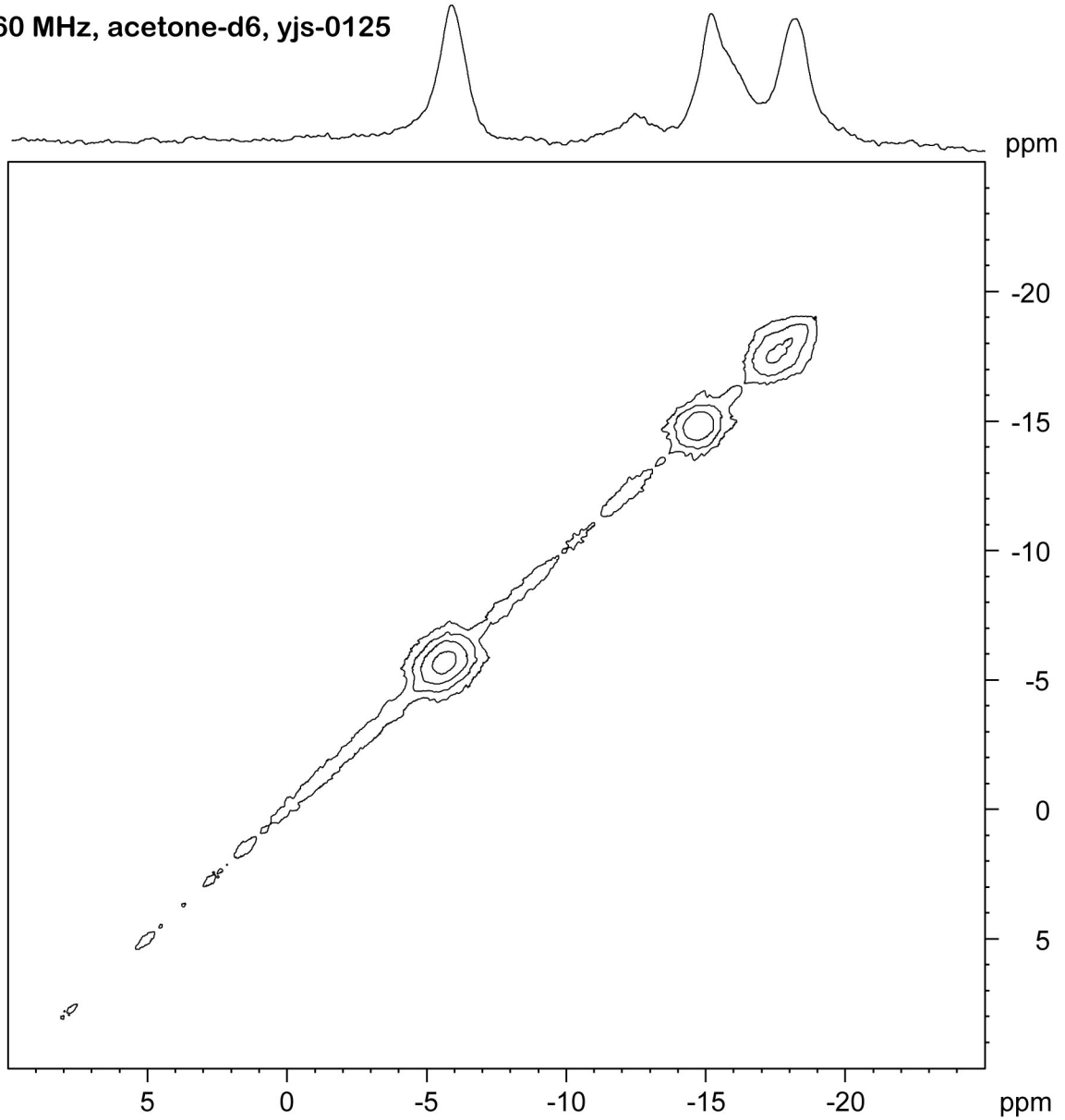
NMR90

```
Current Data Parameters
NAME      YJS_pub01_20160728-2D-yjs-dan
EXPNO                 100
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160728
Time              16.54
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG     cosygpqfdec
TD                1922
SOLVENT           CD3CN
NS                  32
DS                  16
SWH            7211.539 Hz
FIDRES         3.752101 Hz
AQ            0.1332587 sec
RG                  203
DW               69.333 usec
DE                6.50 usec
TE               298.9 K
D0            0.00000300 sec
D1            0.30000001 sec
D11           0.03000000 sec
D13           0.00000400 sec
D16           0.00020000 sec
IN0           0.00013850 sec

======== CHANNEL f1 ========
NUC1                11B
P0                13.10 usec
P1                13.10 usec
PLW1        95.00000000 W
SFO1       160.4603790 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2             80.00 usec
PLW2        19.00000000 W
PLW12        0.39947000 W
SFO2       500.1312000 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPZ1              10.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                  64
SFO1           160.4604 MHz
FIDRES        225.647125 Hz
SW                45.000 ppm
FnMODE              QF

F2 - Processing parameters
SI                4096
SF           160.4615894 MHz
WDW                 EM
SSB       0
LB               20.00 Hz
GB        0
PC                1.40

F1 - Processing parameters
SI                1024
MC2                 QF
SF           160.4615927 MHz
WDW               SINE
SSB       0
LB        0 Hz
GB        0
```

11B-11B COSY NMR, 160 MHz, acetone-d6, yjs-0125

NMR91

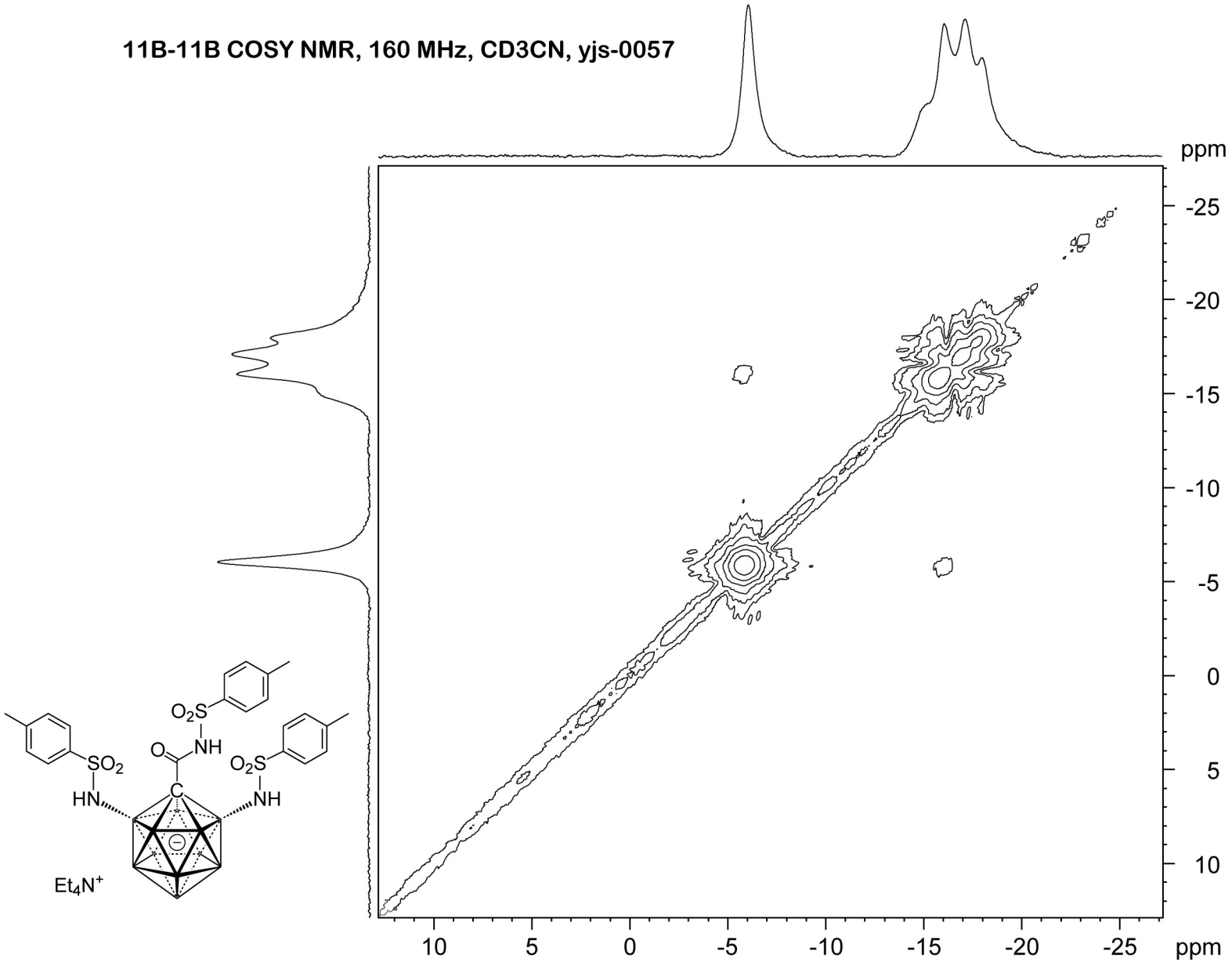





```
Current Data Parameters
NAME      YJS_pub01_20160728-2D-yjs-pyts
EXPNO                 100
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160728
Time              19.36
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG     cosygpqfdec
TD                1922
SOLVENT        Acetone
NS                  32
DS                  16
SWH           7211.539 Hz
FIDRES        3.752101 Hz
AQ           0.1332587 sec
RG                 203
DW              69.333 usec
DE                6.50 usec
TE               298.7 K
D0           0.00000300 sec
D1           0.30000001 sec
D11          0.03000000 sec
D13          0.00000400 sec
D16          0.00020000 sec
IN0          0.00013850 sec

======== CHANNEL f1 ========
NUC1               11B
P0               13.10 usec
P1               13.10 usec
PLW1        95.00000000 W
SFO1       160.4603790 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2               1H
PCPD2            80.00 usec
PLW2        19.00000000 W
PLW12        0.39947000 W
SFO2       500.1312000 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]     SMSQ10.100
GPZ1             10.00 %
P16            1000.00 usec

F1 - Acquisition parameters
TD                  64
SFO1          160.4604 MHz
FIDRES       225.647125 Hz
SW            45.000 ppm
FnMODE              QF

F2 - Processing parameters
SI                4096
SF          160.4615731 MHz
WDW               EM
SSB       0
LB              20.00 Hz
GB        0
PC                1.40

F1 - Processing parameters
SI                1024
MC2                QF
SF          160.4615735 MHz
WDW              SINE
SSB       0
LB        0 Hz
GB        0
```

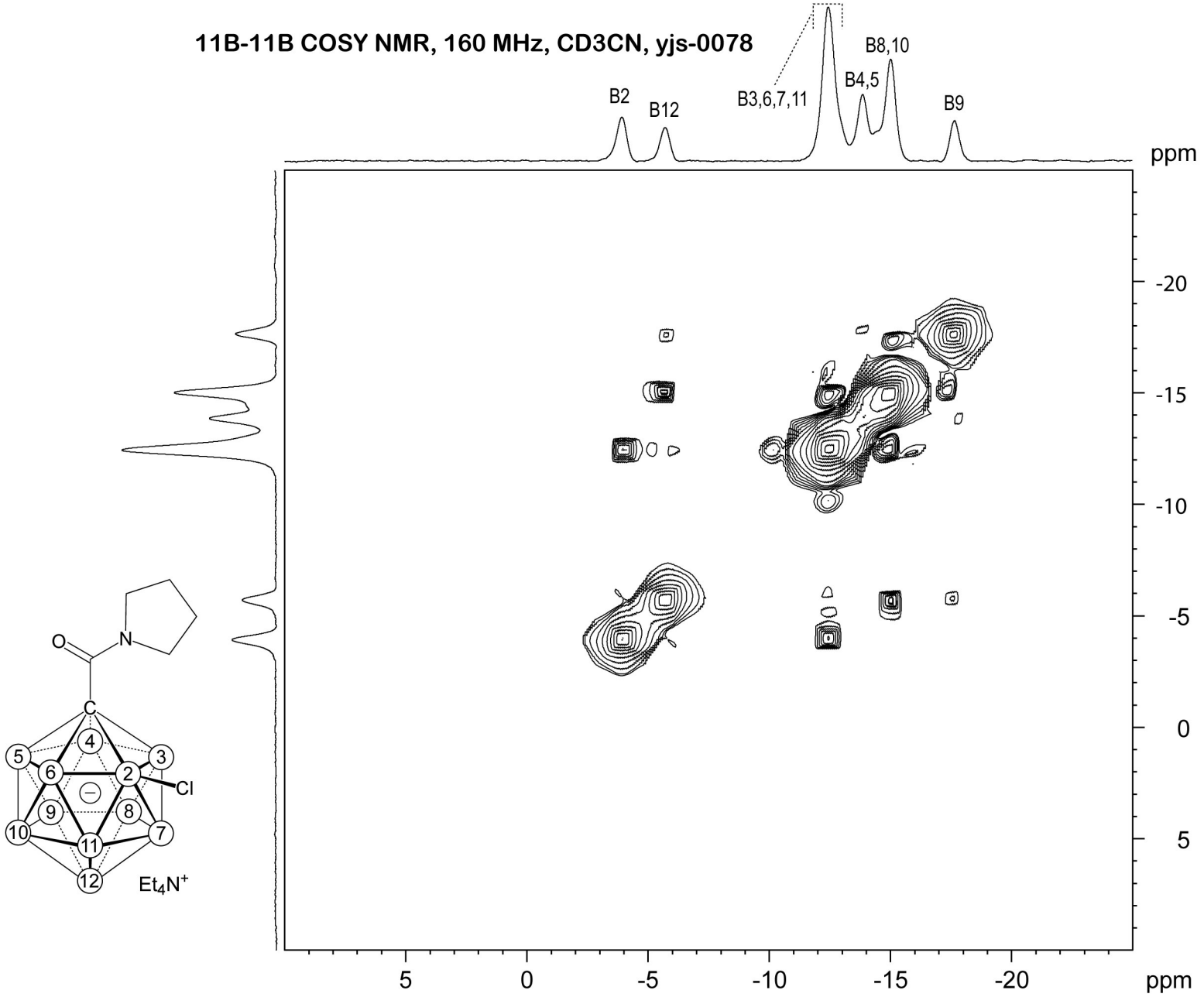
11B-11B COSY NMR, 160 MHz, CD3CN, yjs-0057

NMR92




```
Current Data Parameters
NAME      YJS_pub01_20160728-2D-yjs-tststs
EXPNO                 100
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160728
Time              17.51
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG     cosygpqfdec
TD                1922
SOLVENT           CD3CN
NS                  32
DS                  16
SWH           7211.539 Hz
FIDRES        3.752101 Hz
AQ            0.1332587 sec
RG                  203
DW               69.333 usec
DE                6.50 usec
TE                298.7 K
D0           0.00000300 sec
D1           0.30000001 sec
D11          0.03000000 sec
D13          0.00000400 sec
D16          0.00020000 sec
IN0          0.00013850 sec

======== CHANNEL f1 ========
NUC1                11B
P0                13.10 usec
P1                13.10 usec
PLW1         95.00000000 W
SFO1        160.4603790 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2             80.00 usec
PLW2         19.00000000 W
PLW12         0.39947000 W
SFO2        500.1312000 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPZ1              10.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                  64
SFO1           160.4604 MHz
FIDRES        225.647125 Hz
SW                45.000 ppm
FnMODE              QF

F2 - Processing parameters
SI                4096
SF           160.4615731 MHz
WDW                 EM
SSB       0
LB                20.00 Hz
GB        0
PC                1.40

F1 - Processing parameters
SI                1024
MC2                 QF
SF           160.4615735 MHz
WDW               SINE
SSB       0
LB        0 Hz
GB        0
```

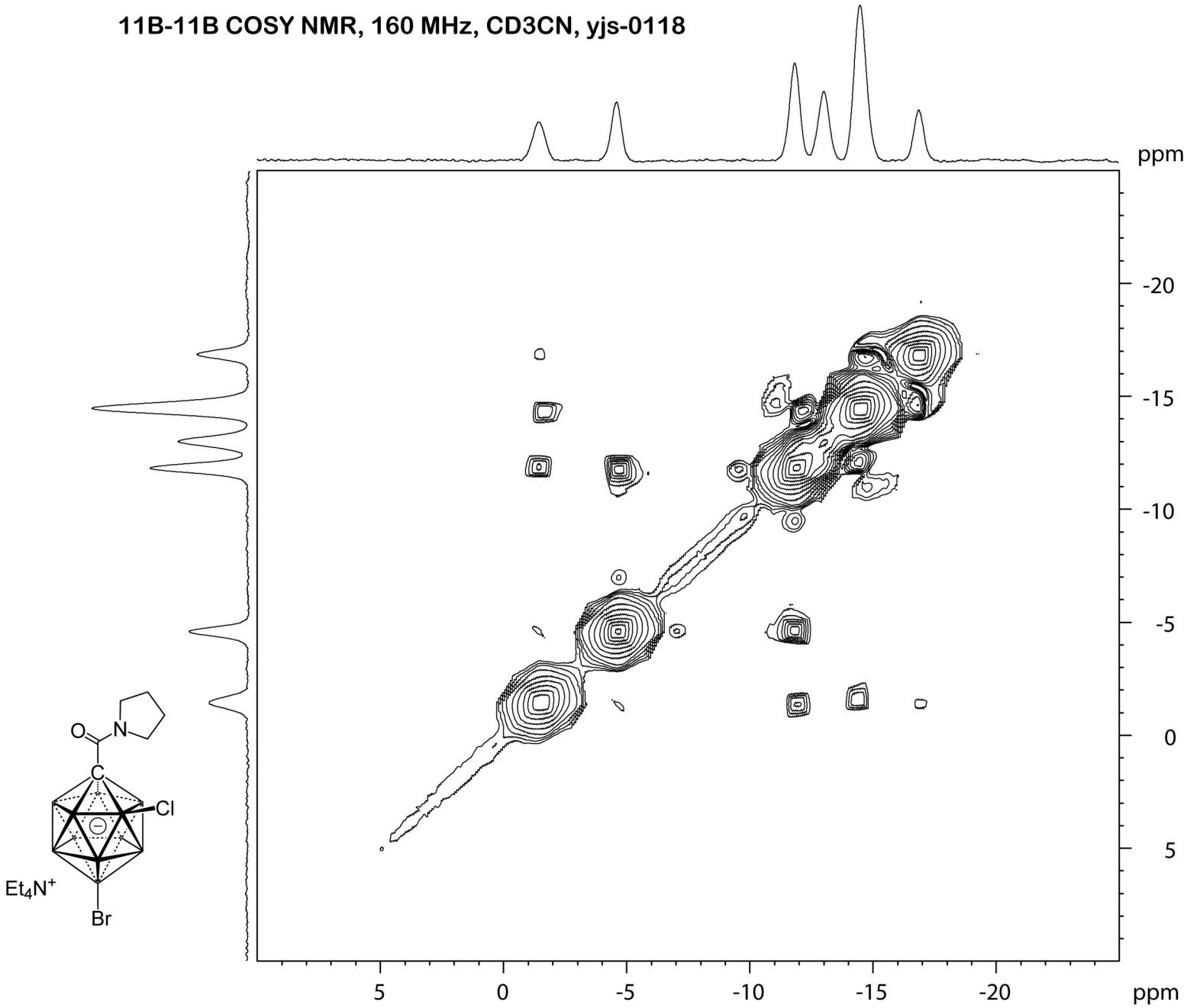
11B-11B COSY NMR, 160 MHz, CD3CN, yjs-0078



NMR93


```
Current Data Parameters
NAME      20160930-yjs-pycl-repeat-2D
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160930
Time              21.56
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG     cosygpqfdec
TD                2566
SOLVENT           CD3CN
NS                  64
DS                  16
SWH           12820.513 Hz
FIDRES         4.996303 Hz
AQ             0.1000740 sec
RG                 203
DW                39.000 usec
DE                 6.50 usec
TE                297.2 K
D0            0.00000300 sec
D1            0.30000001 sec
D11           0.03000000 sec
D13           0.00000400 sec
D16           0.00020000 sec
IN0           0.00007790 sec

======== CHANNEL f1 ========
NUC1                11B
P0                13.10 usec
P1                13.10 usec
PLW1         95.00000000 W
SFO1        160.4610976 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2             80.00 usec
PLW2         19.00000000 W
PLW12         0.42750001 W
SFO2        500.1312000 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPZ1              10.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                  64
SFO1           160.4611 MHz
FIDRES        401.151154 Hz
SW                80.000 ppm
FnMODE              QF

F2 - Processing parameters
SI                4096
SF          160.4615976 MHz
WDW                EM
SSB       0
LB                20.00 Hz
GB        0
PC                 1.40

F1 - Processing parameters
SI                1024
MC2                QF
SF          160.4615967 MHz
WDW               SINE
SSB       0
LB        0 Hz
GB        0
```

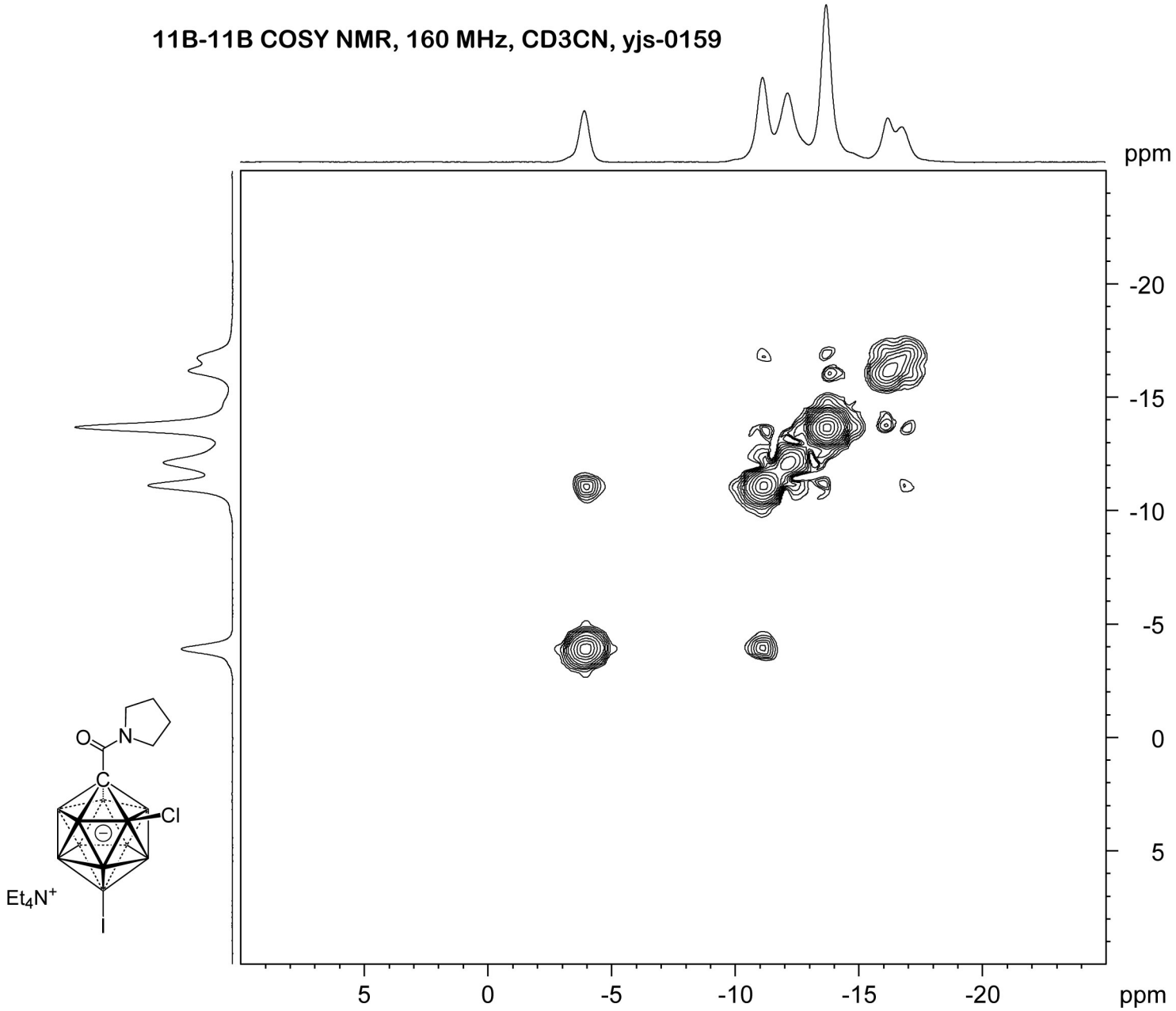
11B-11B COSY NMR, 160 MHz, CD3CN, yjs-0118



NMR94

```
Current Data Parameters
NAME      20160929-yjs-brcl-repeat-2D
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160929
Time              22.12
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG     cosygpqfdec
TD                2566
SOLVENT           CD3CN
NS                  64
DS                  16
SWH          12820.513 Hz
FIDRES        4.996303 Hz
AQ            0.1000740 sec
RG                 203
DW              39.000 usec
DE               6.50 usec
TE              298.0 K
D0           0.00000300 sec
D1           0.30000001 sec
D11          0.03000000 sec
D13          0.00000400 sec
D16          0.00020000 sec
IN0          0.00007790 sec

======== CHANNEL f1 ========
NUC1                11B
P0                13.10 usec
P1                13.10 usec
PLW1         95.00000000 W
SFO1        160.4610976 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2             80.00 usec
PLW2         19.00000000 W
PLW12         0.42750001 W
SFO2        500.1312000 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPZ1              10.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                  64
SFO1           160.4611 MHz
FIDRES        401.151154 Hz
SW              80.000 ppm
FnMODE              QF

F2 - Processing parameters
SI                4096
SF          160.4616085 MHz
WDW                EM
SSB       0
LB              20.00 Hz
GB        0
PC                1.40

F1 - Processing parameters
SI                1024
MC2                QF
SF          160.4615986 MHz
WDW              SINE
SSB       0
LB        0 Hz
GB        0
```

11B-11B COSY NMR, 160 MHz, CD3CN, yjs-0159

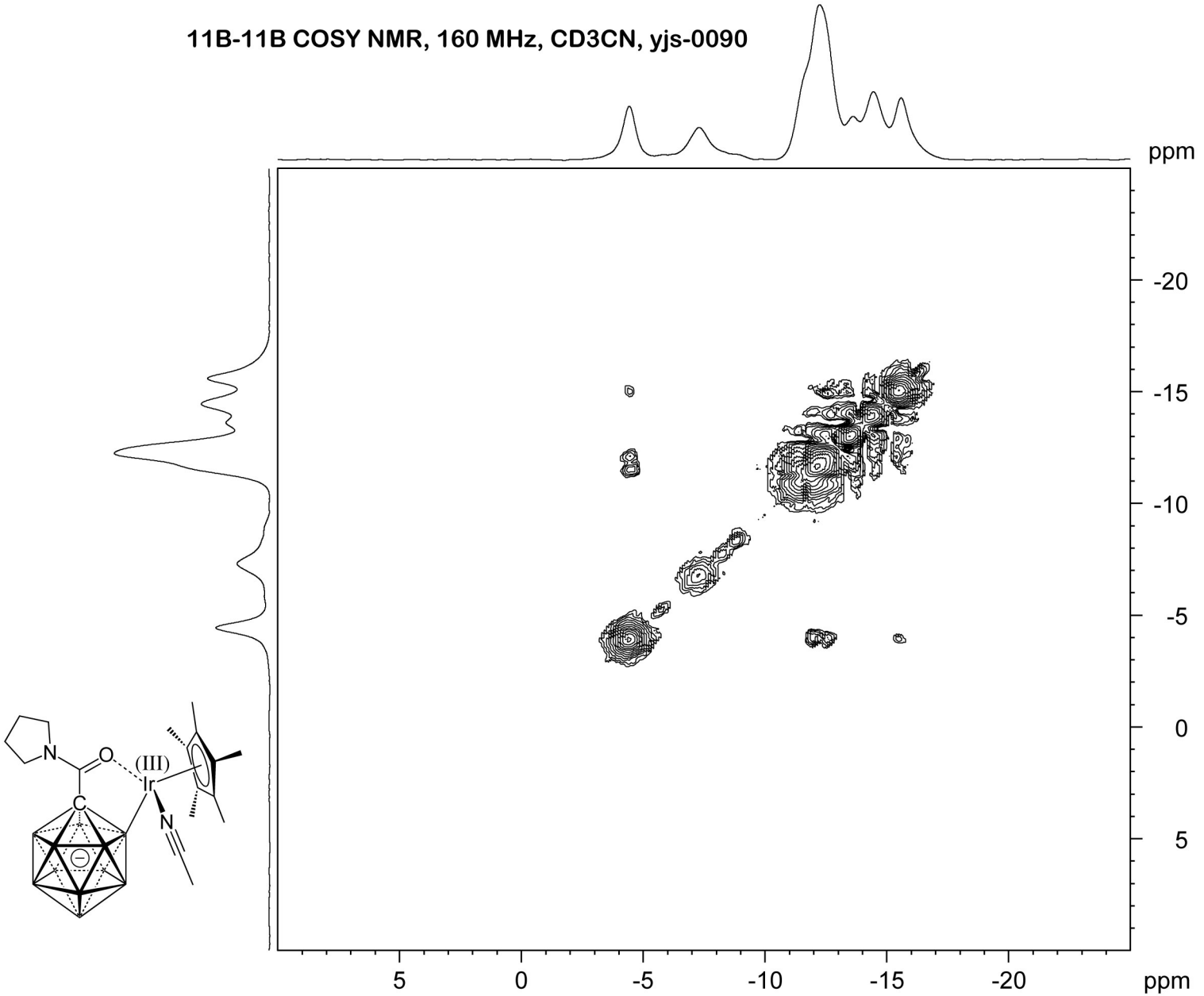


NMR95

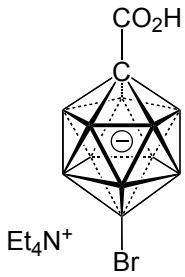
```
Current Data Parameters
NAME      YJS_pub01_20160722-2D-yjs-pyI-Cl
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160722
Time              16.46
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG     cosygpqfdec
TD                2566
SOLVENT           CD3CN
NS                  32
DS                  16
SWH            6421.233 Hz
FIDRES         2.502429 Hz
AQ             0.1998059 sec
RG                  203
DW               77.867 usec
DE                6.50 usec
TE                299.1 K
D0           0.00000300 sec
D1           0.30000001 sec
D11          0.03000000 sec
D13          0.00000400 sec
D16          0.00020000 sec
IN0          0.00015580 sec

======== CHANNEL f1 ========
NUC1               11B
P0               13.10 usec
P1               13.10 usec
PLW1        95.00000000 W
SFO1       160.4604558 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2             80.00 usec
PLW2        19.00000000 W
PLW12        0.42750001 W
SFO2       500.1312000 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPZ1              10.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                  64
SFO1           160.4605 MHz
FIDRES         200.574966 Hz
SW                40.000 ppm
FnMODE              QF

F2 - Processing parameters
SI                4096
SF           160.4616085 MHz
WDW                EM
SSB       0
LB               20.00 Hz
GB        0
PC                1.40

F1 - Processing parameters
SI                1024
MC2                 QF
SF           160.4615986 MHz
WDW               SINE
SSB       0
LB        0 Hz
GB        0
```

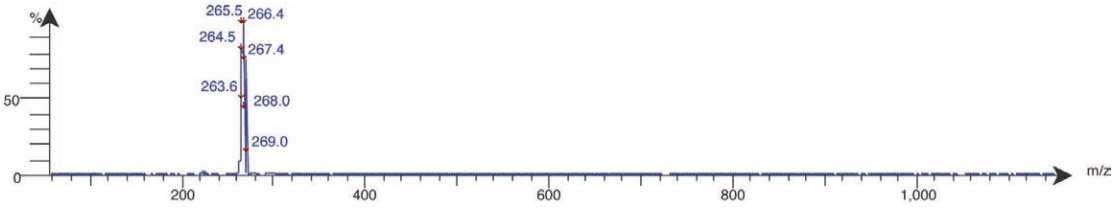
11B-11B COSY NMR, 160 MHz, CD3CN, yjs-0090

NMR96

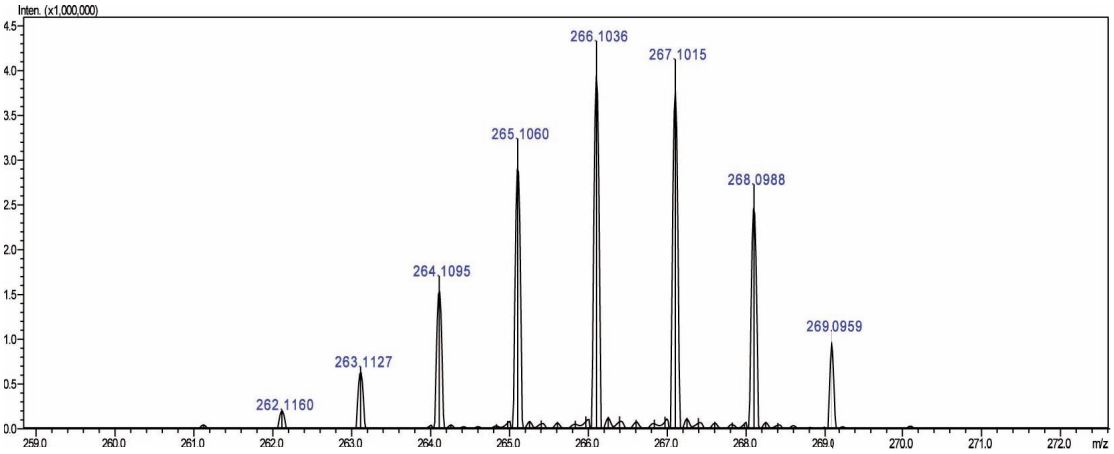



```
Current Data Parameters
NAME      YJS_pub01_20160727-2D-yjs-pyIr
EXPNO                 100
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160727
Time              15.44
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG     cosygpqfdec
TD                2048
SOLVENT           CD3CN
NS                   8
DS                  16
SWH           16025.641 Hz
FIDRES         7.825020 Hz
AQ             0.0638976 sec
RG                 203
DW               31.200 usec
DE                6.50 usec
TE               297.9 K
D0            0.00000300 sec
D1            0.50000000 sec
D11           0.03000000 sec
D13           0.00000400 sec
D16           0.00020000 sec
IN0           0.00006230 sec

======== CHANNEL f1 ========
NUC1                11B
P0                13.10 usec
P1                13.10 usec
PLW1         95.00000000 W
SFO1        160.4607767 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2             80.00 usec
PLW2         19.00000000 W
PLW12         0.42750001 W
SFO2        500.1325007 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPZ1              10.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                  303
SFO1           160.4608 MHz
FIDRES        105.913811 Hz
SW             99.999 ppm
FnMODE              QF

F2 - Processing parameters
SI                 4096
SF           160.4615903 MHz
WDW                EM
SSB       0
LB              20.00 Hz
GB        0
PC               1.40

F1 - Processing parameters
SI                 1024
MC2                QF
SF           160.4615089 MHz
WDW              SINE
SSB       0
LB        0 Hz
GB        0
```





(–)-ESI-MS Advion Expression CMS



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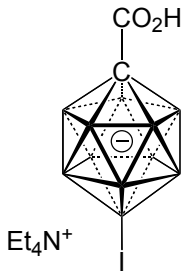
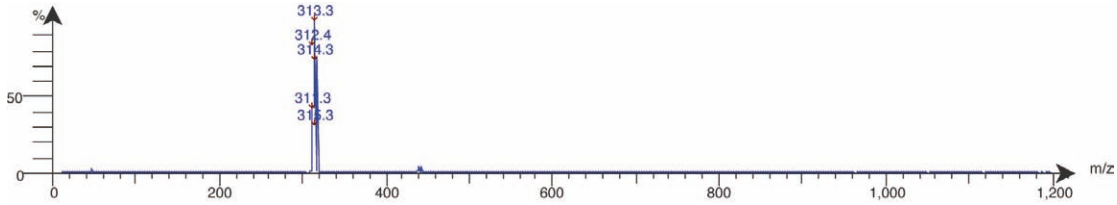
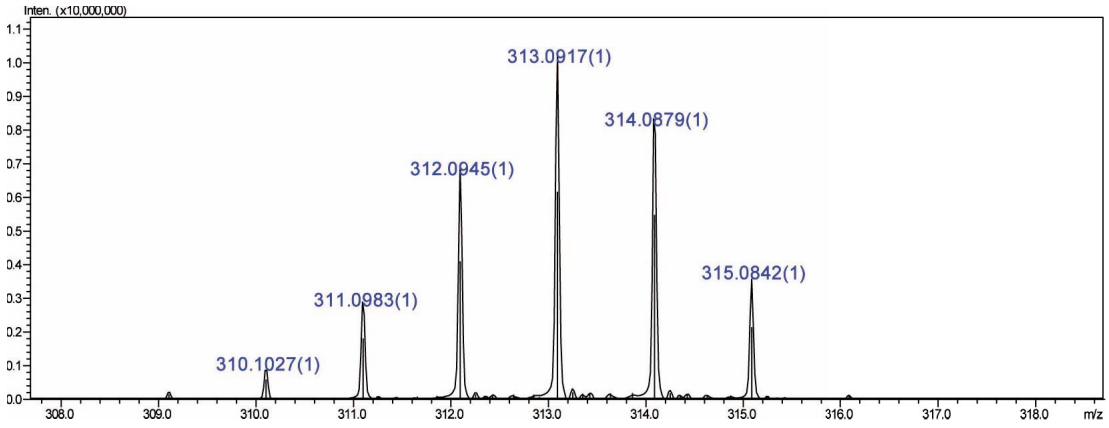
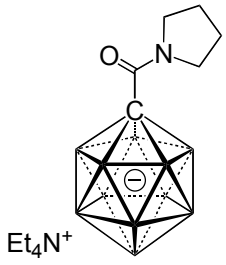


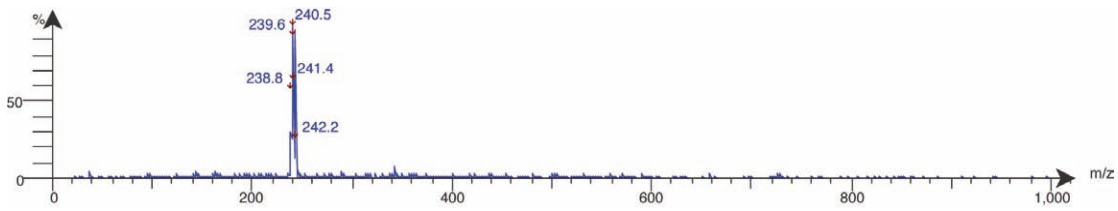


(–)-ESI-MS Advion Expression CMS

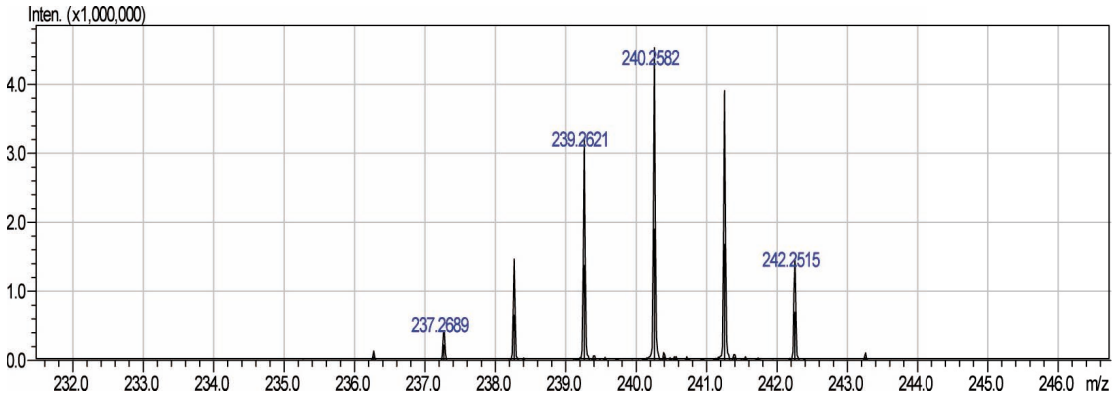


(–)-ESI-HRMS Shimadzu IT-TOF





(–)-ESI-MS Advion Expression CMS



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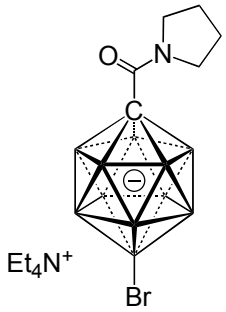

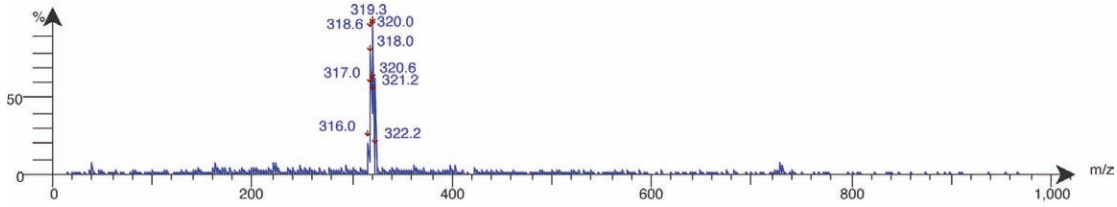

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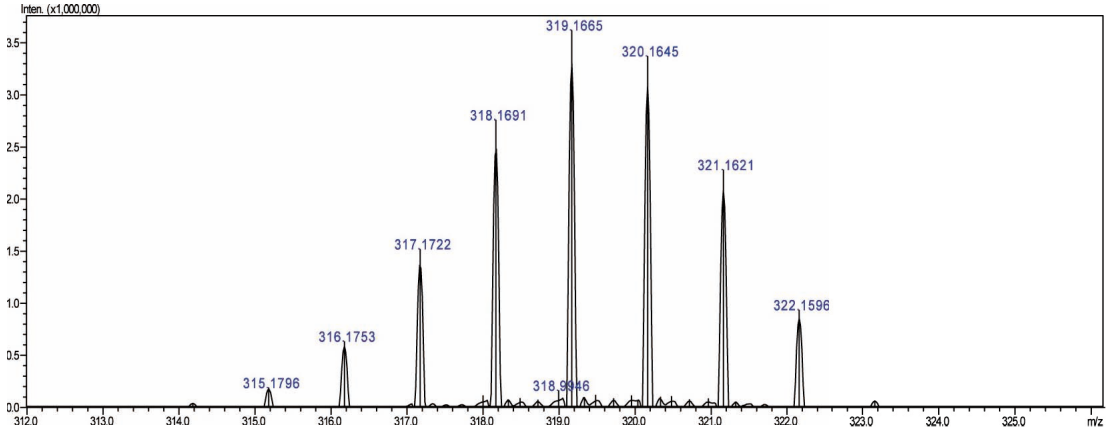

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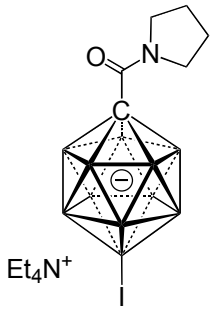

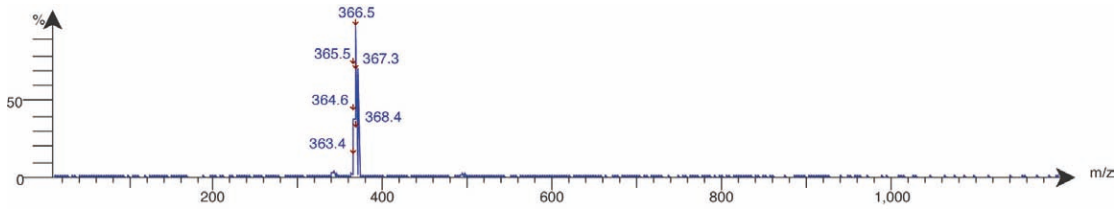

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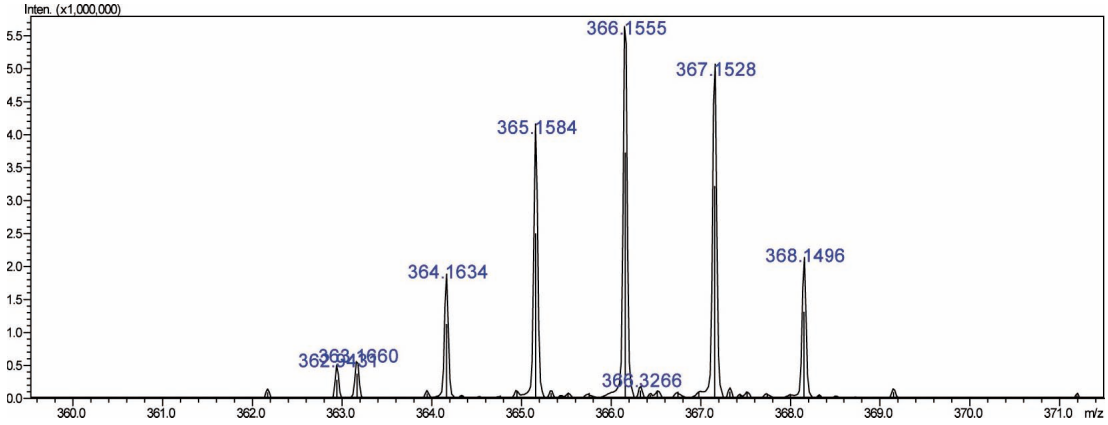

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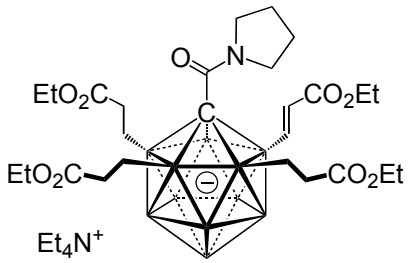
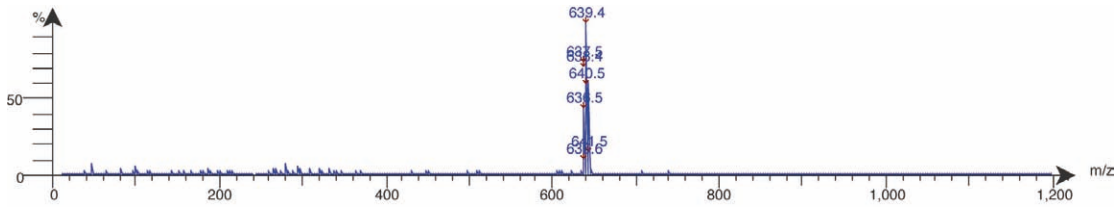
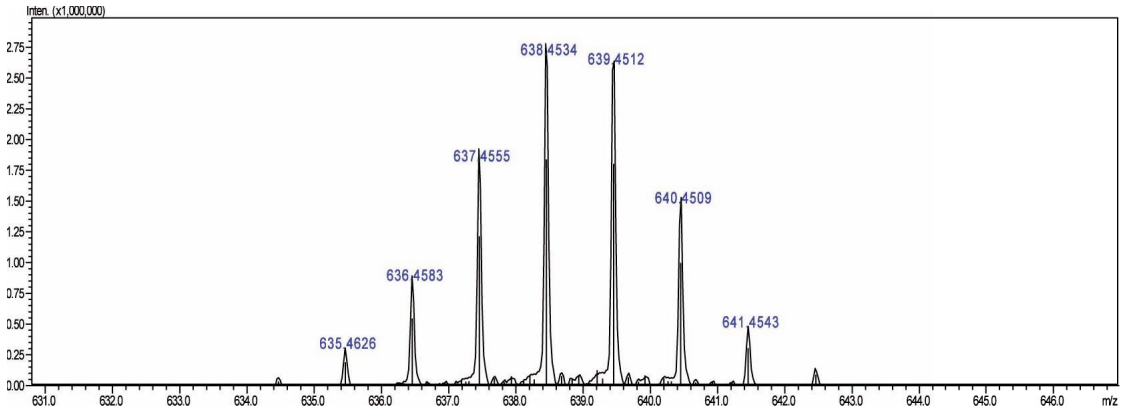
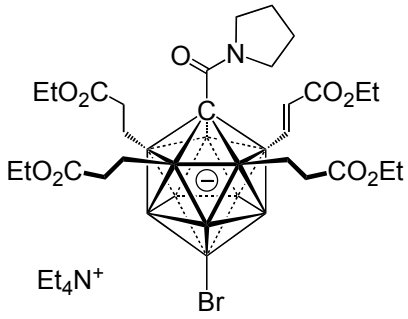


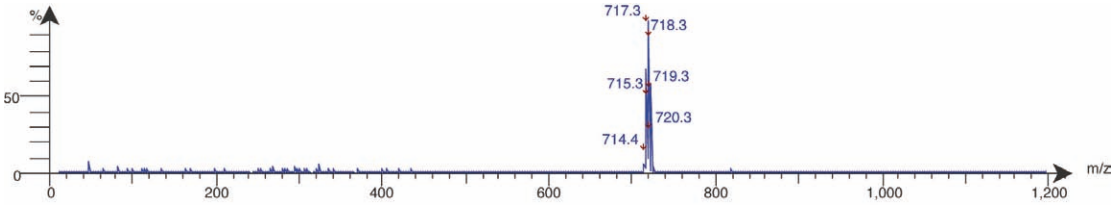


(–)-ESI-MS Advion Expression CMS



(–)-ESI-HRMS Shimadzu IT-TOF





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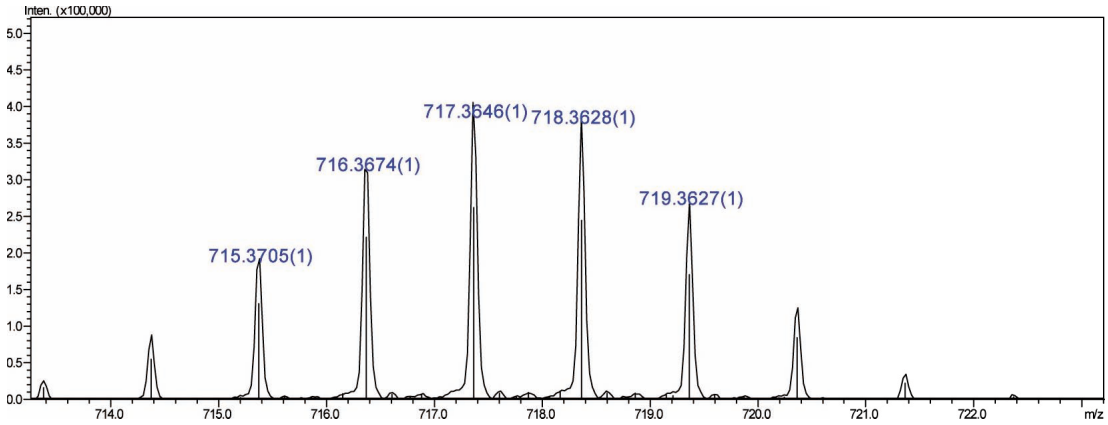
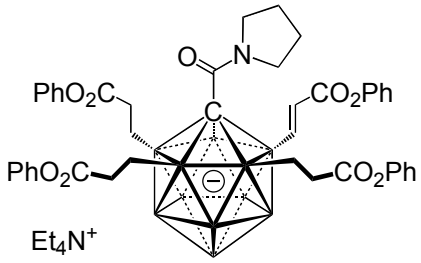
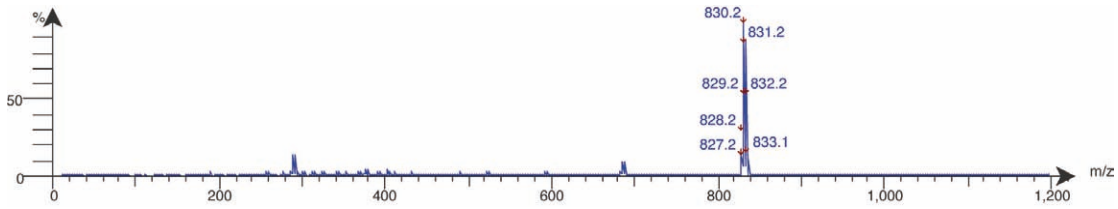


(–)-ESI-HRMS Shimadzu IT-TOF





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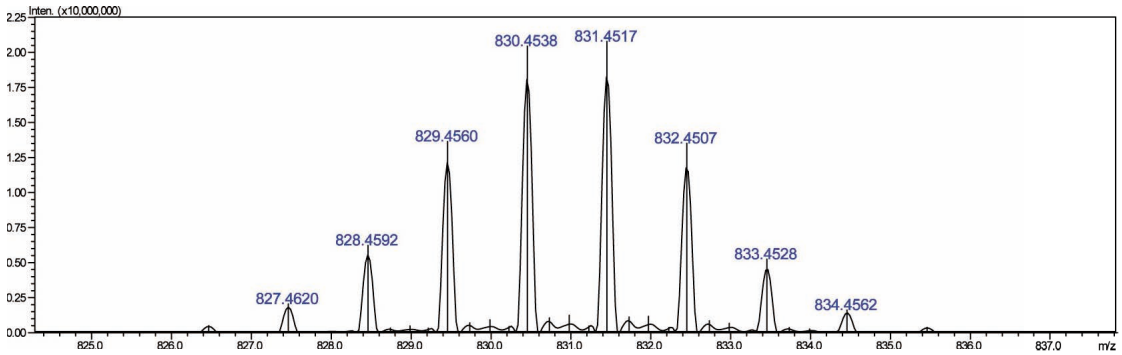
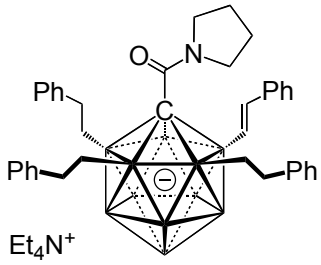
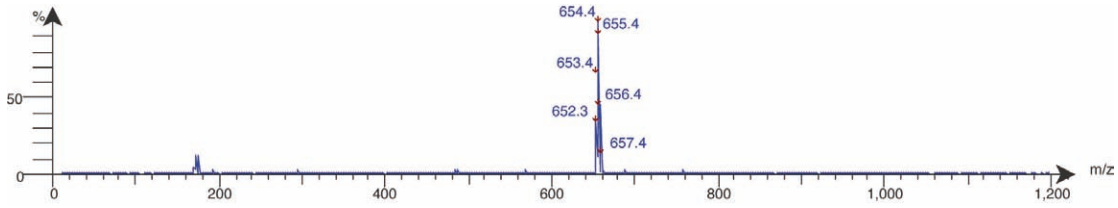
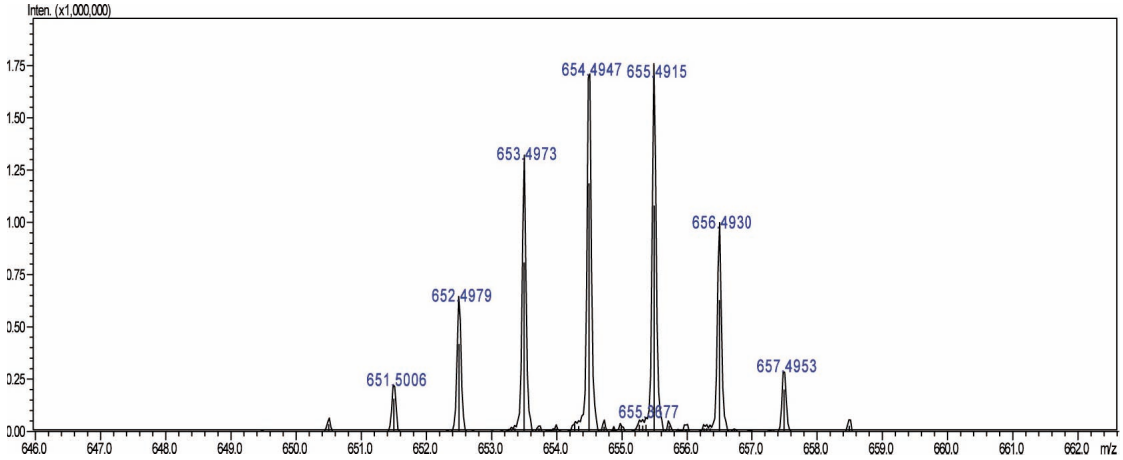


(–)-ESI-HRMS Shimadzu IT-TOF





(–)-ESI-MS Advion Expression CMS



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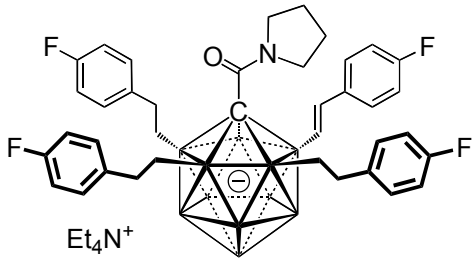
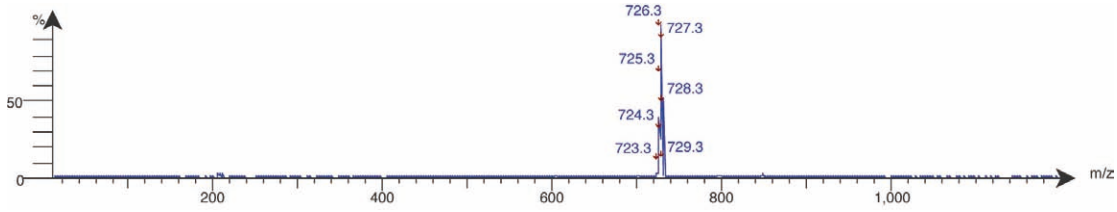
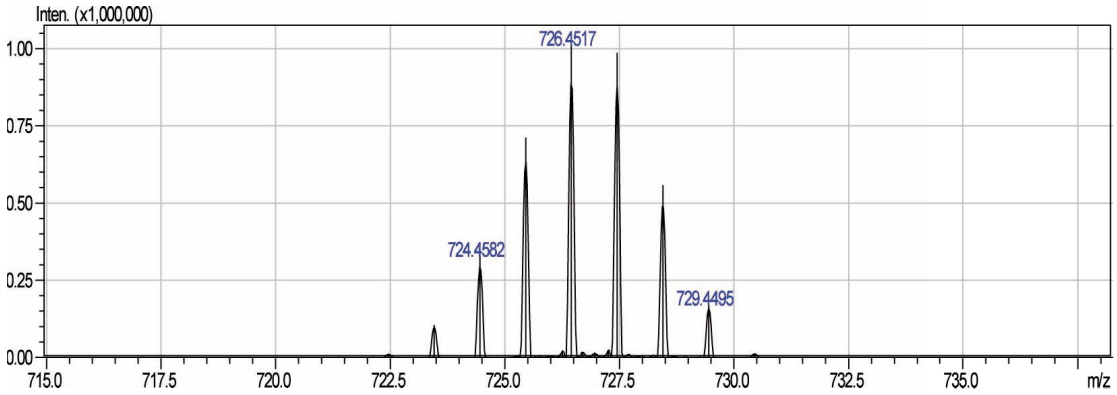
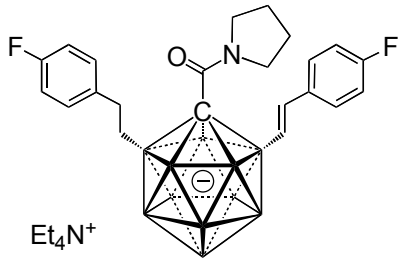


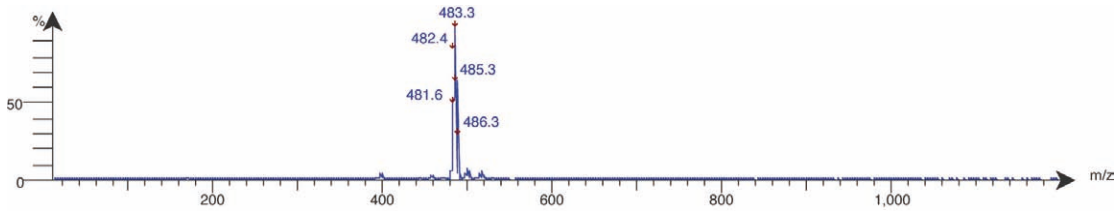


(–)-ESI-MS Advion Expression CMS



(–)-ESI-HRMS Shimadzu IT-TOF





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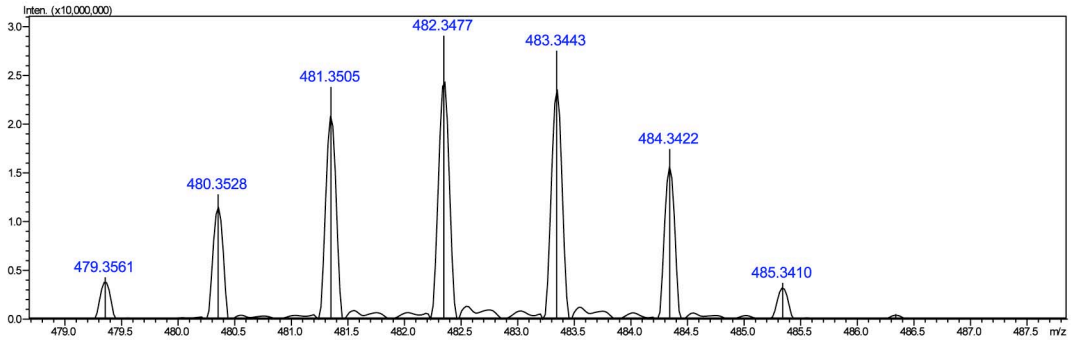

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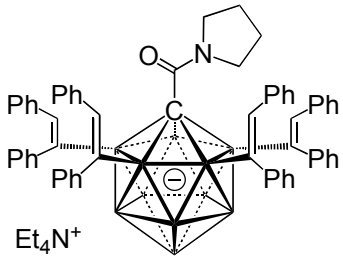

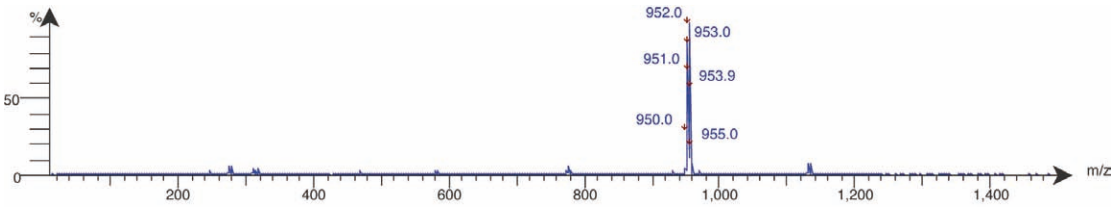

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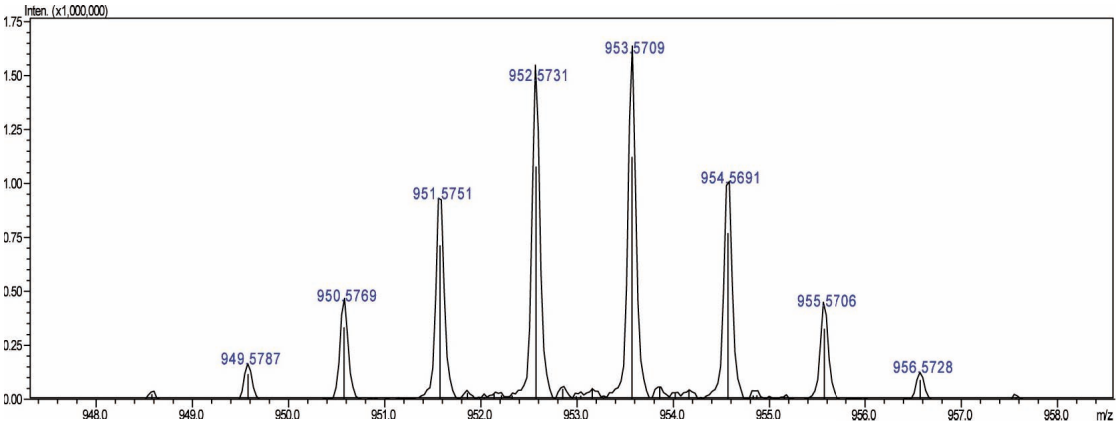

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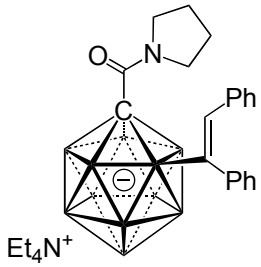

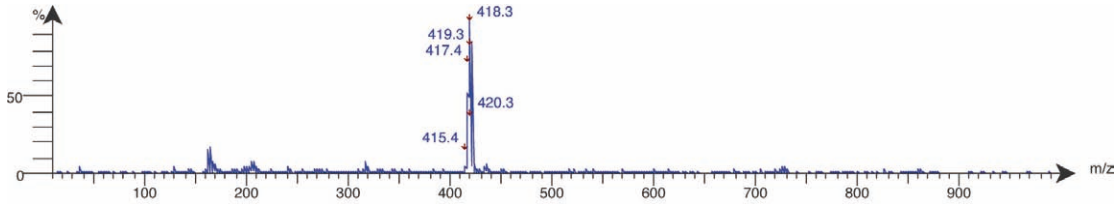

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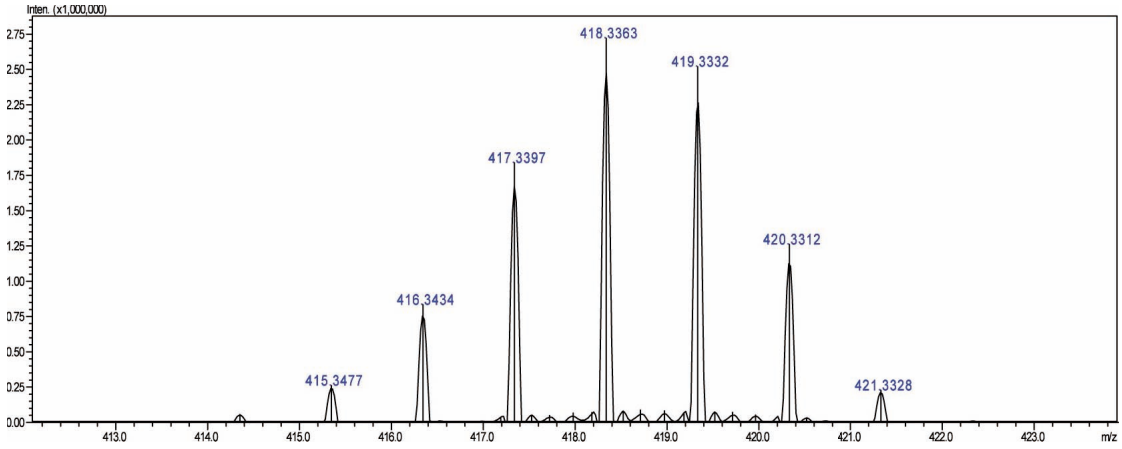

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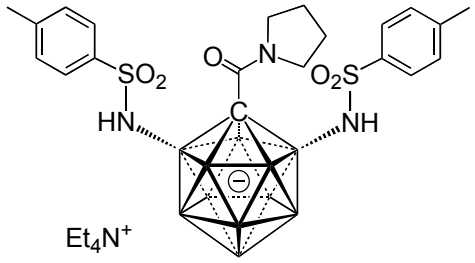

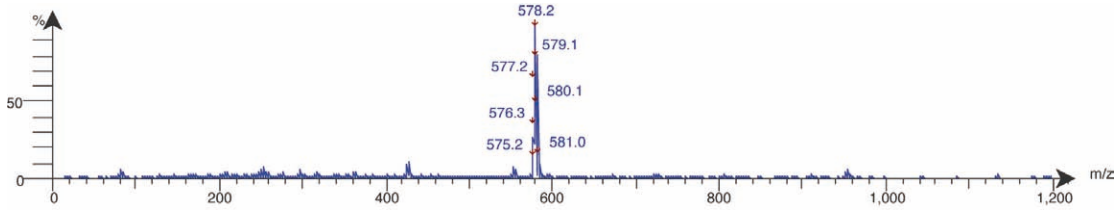

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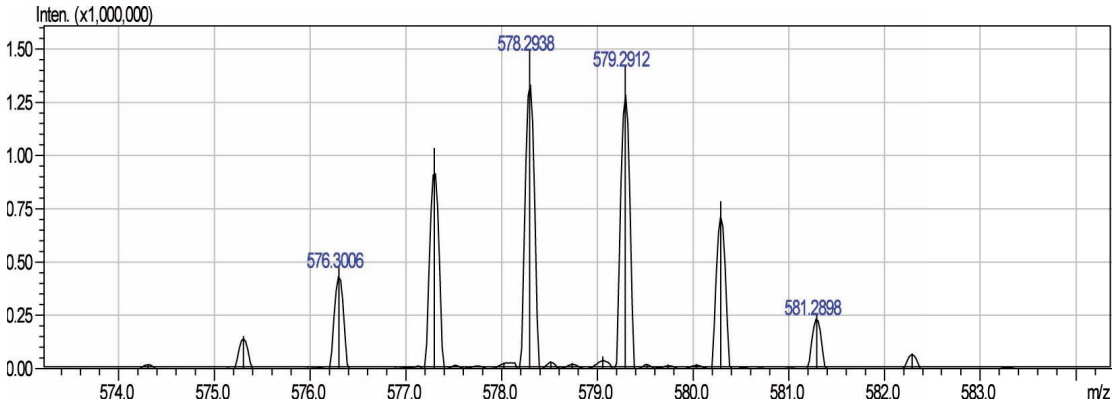

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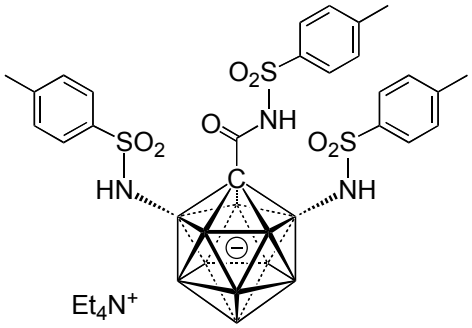

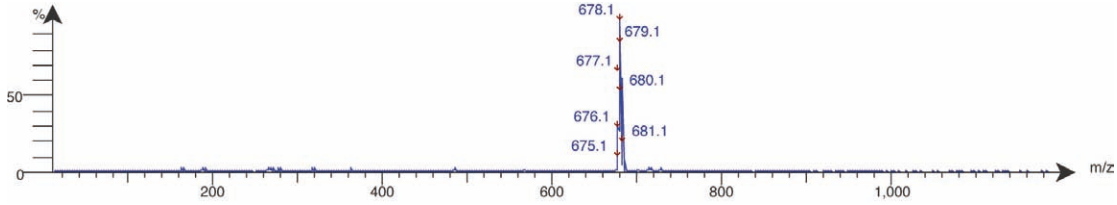

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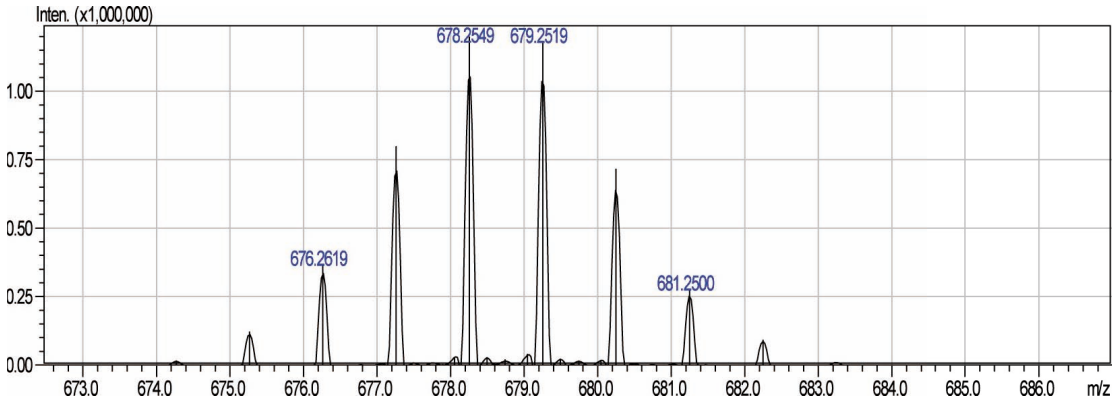

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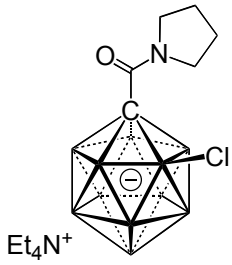

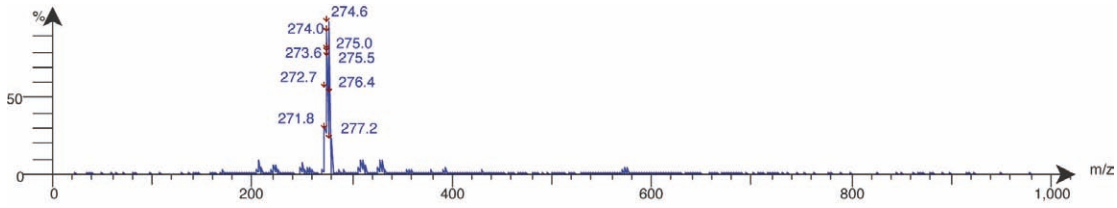

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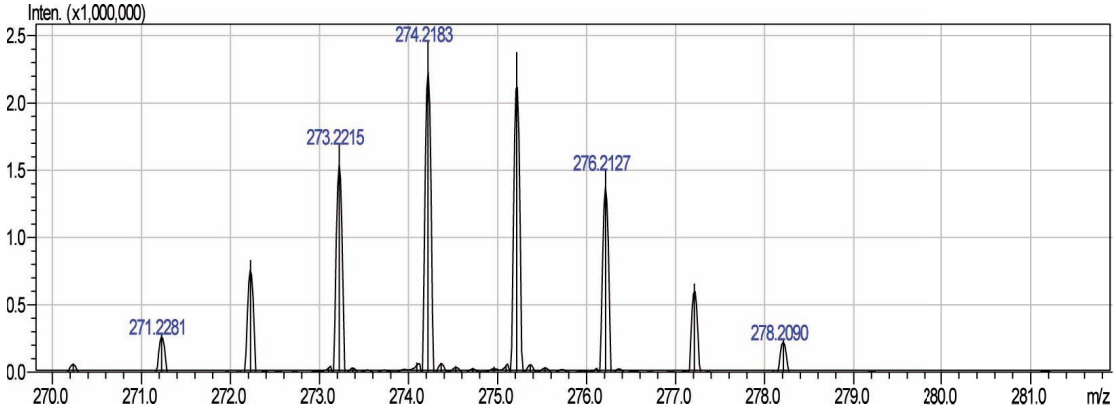

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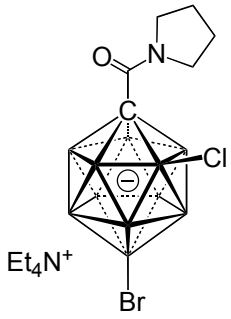

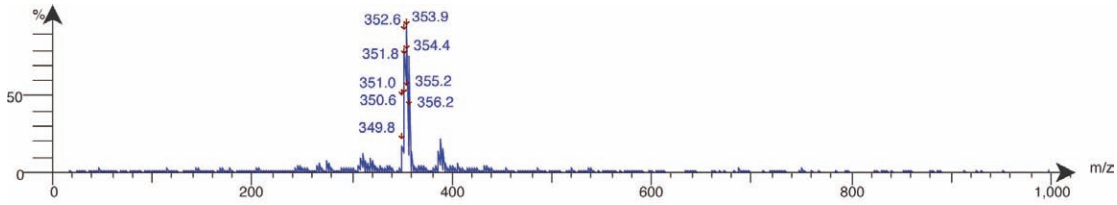

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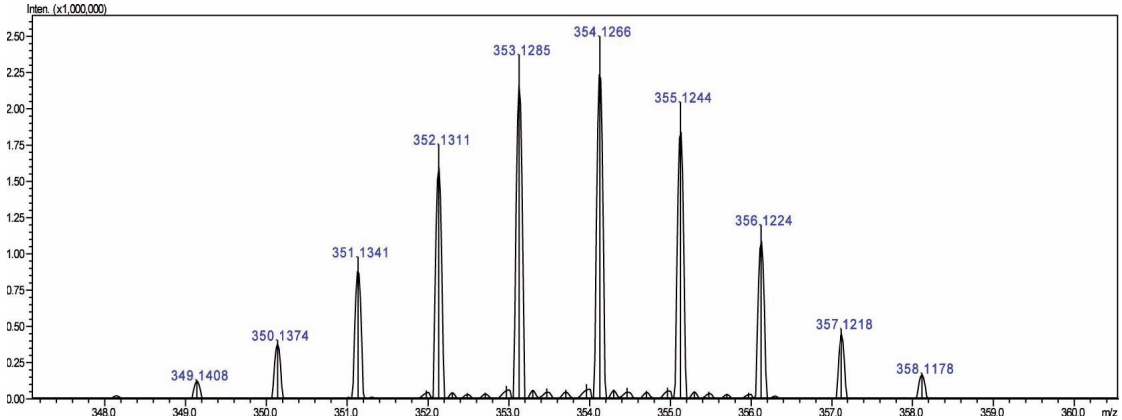

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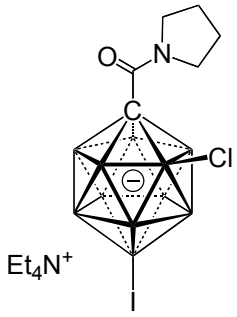

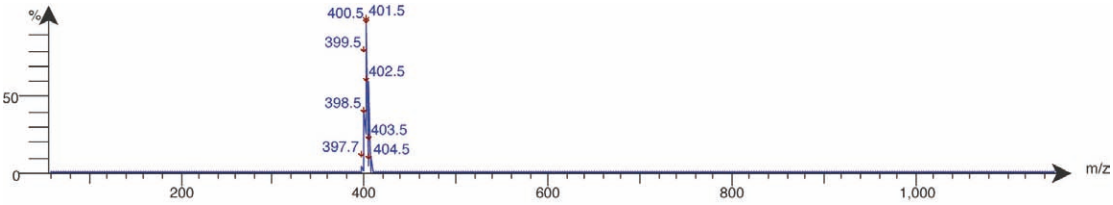

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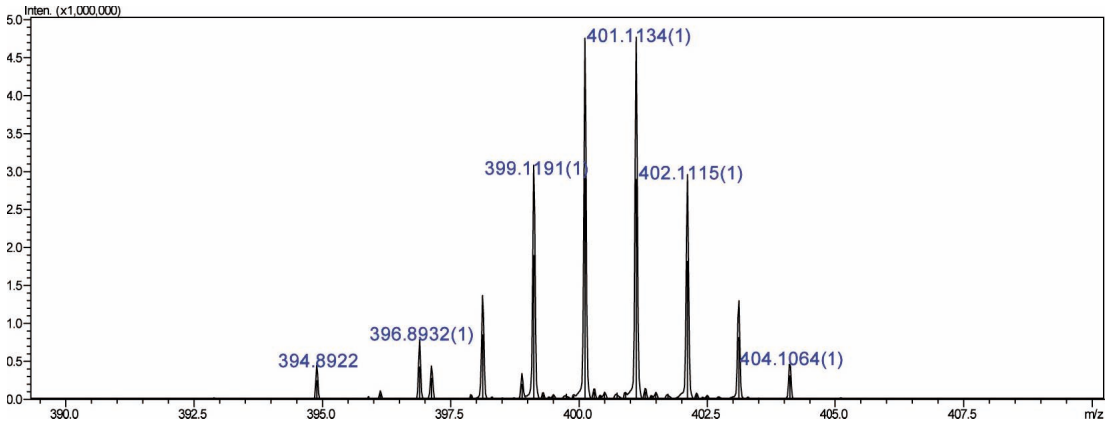

(–)-ESI-HRMS Shimadzu IT-TOF