Supplementary Information for

Regioselective B2–6 penta-iodination of the $[CB_{11}H_{12}]^-$ monocarborane cluster by palladium catalysis

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1 General Information

Chemicals

$Pd(OAc)_2$ was purchased from Energy Chemicals. *N*-Iodosuccinimide (NIS) was purchased from Bidepharm. Na_2SO_3 and $[NEt_4]Br$ were purchased from Aladdin. *n*-BuLi was obtained from Energy Chemicals. HCl (aq.), THF, HOAc and 1,4-dioxane were received from Sinopharm. Anhydrous THF was prepared by passage though activated Al_2O_3 and stored over 3 Å molecular sieves. $[NEt_4][12\text{-Me-}CHB_{11}H_{10}]$, $[NEt_4][12\text{-Et-}CHB_{11}H_{10}]$, $[NEt_4][12\text{-Ph-}CHB_{11}H_{10}]$, $[NEt_4][1\text{-COOH-12-I-}CB_{11}H_{10}]$, and $[NEt_4][1\text{-COOH-12-Br-}CB_{11}H_{10}]$ were prepared according to the literature.[1,2,5]

Characterization

1) NMR spectra were recorded on a Bruker AVANCE III 400 spectrometer (^1H NMR 400.13 MHz, $^{13}C\{^1H\}$ NMR 100.62 MHz, ^{11}B NMR 128.38 MHz) or a Bruker AVANCE III 500 spectrometer (^1H NMR 500.13 MHz, ^{13}C NMR 125.77 MHz, ^{11}B NMR 160.46 MHz) at 296 K. Chemical shifts are given in ppm. $^1H\{^{11}B\}$ NMR and $^{13}C\{^1H\}$ NMR spectra were referenced using the solvent signals ($^1H\{^{11}B\}$: residual $CHD_2C(O)CD_3$ = 2.05 ppm, residual CHD_2CN = 1.94 ppm, $^{13}C\{^1H\}$: $CD_3C(O)CD_3$ = 29.84 ppm, CD_3CN = 1.32 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). 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.

Notes:

- In some spectra, the CH_3 group of the $[NEt_4]^+$ cation showed $^3J_{1H,14N}$ coupling to the central nitrogen atom, and therefore the signal appeared as a triplet of triplets ($^3J_{1H,1H}$ and $^3J_{1H,14N}$). Generally speaking, coupling to ^{14}N is sometimes observed in highly symmetrical nitrogen compounds; the coupling constant is not uniformly related to the distance to the nitrogen atom.
- In certain $^1H\{^{11}B\}$ NMR spectra measured in acetone-d_6, double water peaks were observed. This is a result of different resonances from H_2O and HOD.

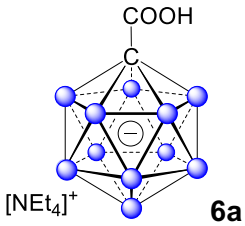
2) High-resolution MS data were recorded using an Agilent G6545 Q-TOF instrument equipped with an electrospray ionization source (ESI).

3) Single-crystal X-ray diffraction studies were performed on a Bruker D8 Venture diffractometer equipped with a 135 mm Atlas CCD detector and using a Mo X-ray source.

4) Infrared spectra were recorded on a Thermo NICOLET AVATAR 330FT-IR spectrometer.

2 Experimental Section

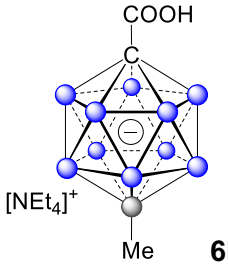
Synthesis of $[NEt_4][1\text{-}COOH\text{-}12\text{-}X\text{-}CB_{11}H_{10}]$ (X = H, Me, Et, Ph)



6a

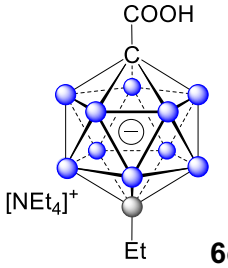
A dry 50 mL single-neck pear-shaped flask equipped with magnetic stir bar was charged with $[Cs][CHB_{11}H_{11}]$ (500 mg, 1.81 mmol) and capped with a rubber septum. Anhydrous THF (15 mL) was then added to the flask in glove box. A solution of *n*-BuLi (1.6 M in hexane, 4.5 mL, 7.2 mmol) was cautiously added at 25 °C, dropwise. After stirring for 4 h, a slightly turbid, white suspension was obtained. The mixture reacted overnight under with CO_2 (1 atmosphere, 2-3 bubbles/s), and the solution turned pale yellow. The reaction was quenched with water (5 mL), and then THF was removed with a rotary evaporator. $[NEt_4]NBr$ (1.142 g, 5.43 mmol) was added, and the resulting white precipitate was separated using a glass frit funnel (F porosity) and washed with water (3 x 2mL). HCl (1 M aq., 1 mL, 12 mmol) was added dropwise to the yellowish filtrate, the resulting white precipitate was collected in a glass frit and dried in a vacuum to give the salt $[NEt_4][1\text{-}COOH\text{-}CB_{11}H_{11}]$ as a white solid (522 mg, 1.647 mmol, 91%).

The NMR data were in accordance with the literature.[1]



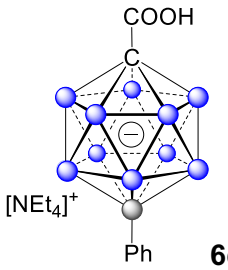
6b

Following a similar procedure as for the preparation of [NEt_4][1-COOH-$CB_{11}H_{11}$], using [NEt_4][12-Me-$CB_{11}H_{11}$] (100 mg, 0.349 mmol) as starting material, [NEt4][1-COOH-12-Me-$CB_{11}H_{10}$] was obtained as a colorless solid (79.8 mg, 69%).
The NMR data were in accordance with the literature.[4]



6c

Following a similar procedure as for the preparation of [NEt_4][1-COOH-$CB_{11}H_{11}$], using [NEt_4][12-Et-$CB_{11}H_{11}$] (100 mg, 0.333 mmol) as starting material, [NEt_4][1-COOH-12-Et-$CB_{11}H_{10}$] was obtained as a colorless solid (87 mg, 75%).
The NMR data were in accordance with the literature.[3]



6d

Following a similar procedure as for the preparation of [NEt_4][1-COOH-$CB_{11}H_{11}$], using [NEt_4][12-Ph-$CB_{11}H_{11}$] (178.7 mg, 0.513 mmol) as starting material, [NEt_4][1-COOH-12-Ph-$CB_{11}H_{10}$] was obtained as a colorless solid (155.6 mg, 77%).
The NMR data were in accordance with the literature.[3]

Optimization of reaction conditions

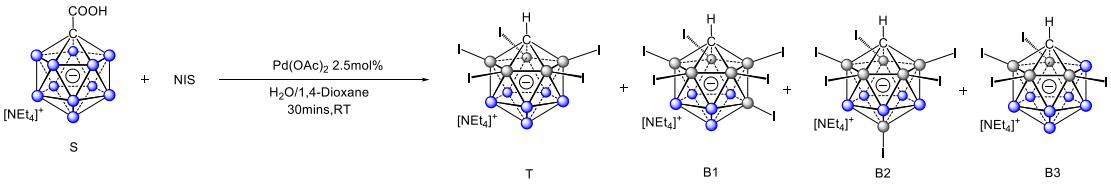


Figure S1. Reaction of the monocarborane carboxylic acid **S** leading to the desired product **T** and byproducts **B** (blue spheres = BH, gray spheres = B).

To a 2.5 mL glass vial equipped with a magnetic stir bar, **S** (5 mg, 1.0 equiv, 0.0158 mmol) and *N*-iodosuccinimide (NIS) (varying amounts, see below) were added (see Figure S1 for the definition of **S** and the products **T**/**B1–3**). Then a $Pd(OAc)_2$ solution (0.3 mL from a stock solution of 6.0 mg $Pd(OAc)_2$ in 20 mL anhydrous 1,4-dioxane; 2.5 mol%, 0.0004 mmol) and deionized H_2O (varying amounts, see below) were added. The reaction mixture was stirred for 30 mins at 25 °C. The reaction outcome was evaluated by $^{11}B\{^1H\}$ NMR spectroscopy.

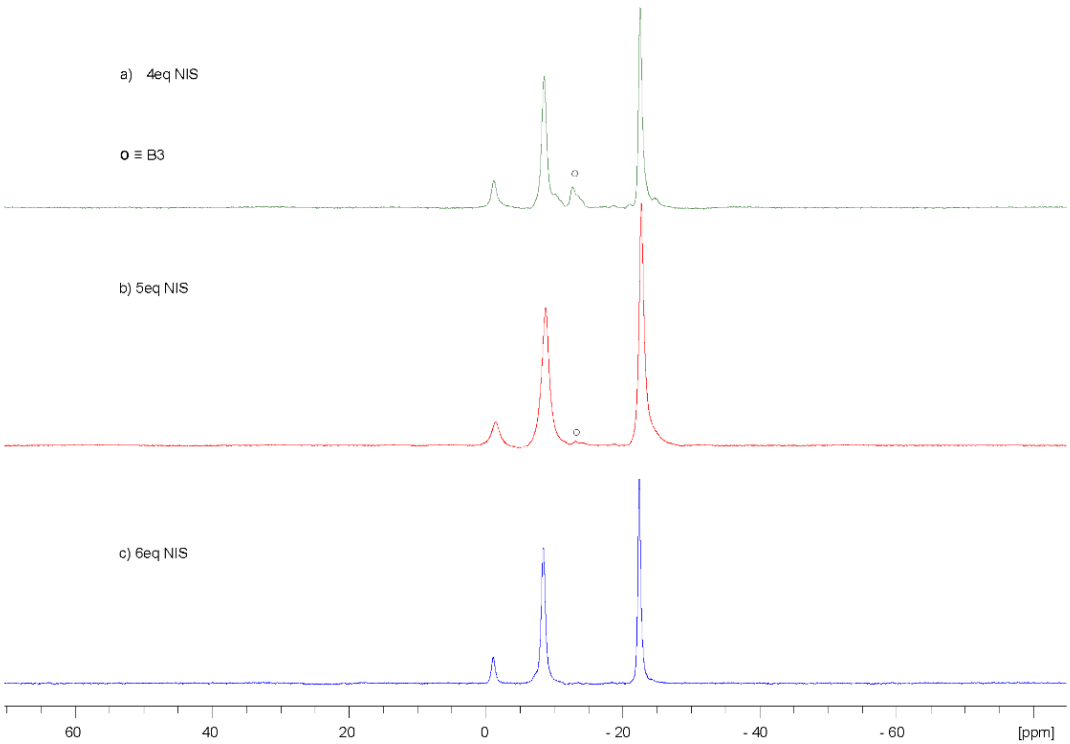


Figure S2. All reactions were performed with 6% H_2O; a) with 4 equiv of NIS; b) with 5 equiv of NIS; c) with 6 equiv of NIS.

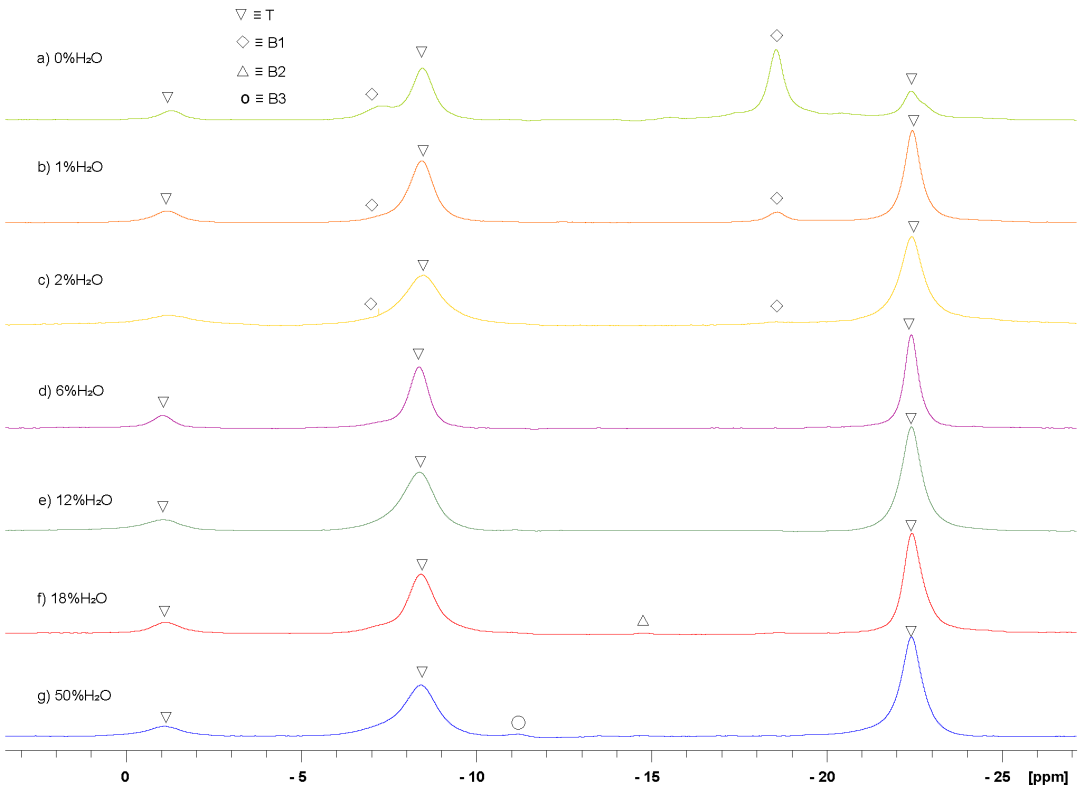


Figure S3. All reactions were performed with 6 equiv of NIS; a) 0% H_2O; b) 1% H_2O; c) 2% H_2O; d) 6% H_2O; e) 12% H_2O; f) 18% H_2O; g) 50% H_2O.

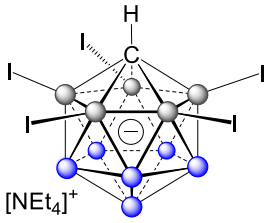
Remarks: A ratio of H_2O equal to 6% (marked as "6% H_2O" in the spectra) means that the solvent was a mixture of 18 µL H_2O and 0.3 mL 1,4-dioxane. Other percentages having a similar meaning.

Figure S2 shows the effect of the amount of NIS, while H_2O was kept at 6%. The use of 6 equiv of NIS gave the desired product **T** as the major product, as indicated by the spectrum of the reaction mixture.

Figure S3 shows the effect of the amount of H_2O, while NIS was kept at 6 equivalents. The use of 6% to 18% H_2O gave the best results.

In summary, the screening of conditions indicated that a combination of 6 equivalents of NIS and 6–18% H_2O allowed for the formation of the desired penta-iodinated product under convenient conditions, namely, 30 min at 25 °C.

Synthesis of [NEt_4][2,3,4,5,6-I_5-12-X-$CHB_{11}H_6$] (X = H, Me, Et, Ph, I, Br)



8a

To a 10 mL glass vial equipped with a magnetic stir bar, [NEt_4][1-COOH-$CB_{11}H_{11}$] (50 mg, 1.0 equiv, 0.158 mmol) and NIS (213 mg, 6.0 equiv, 0.947 mmol) were added. Then $Pd(OAc)_2$ solution (3.0 mL from a stock solution of 6.0 mg $Pd(OAc)_2$ in 20 mL anhydrous 1,4-dioxane; 2.5 mol%, 0.004 mmol) and deionized H_2O (0.36 mL) were added. The reaction mixture was stirred for 30 min at 25 °C. Aqueous Na_2SO_3 solution (29.8 mg Na_2SO_3 in 5 mL deionized H_2O; 1.5 equiv, 0.237 mmol) was added to the mixture dropwise. NEt_4Br (99.4 mg, 3.0 equiv, 0.473 mmol) was added to precipitate the product under sonification. 1,4-dioxane was then removed under reduced pressure. The resulting mixture was placed in a glass frit funnel (F porosity), the aqueous layer was removed, and the remaining solid was washed three times with H_2O and dried in a vacuum to give salt [NEt_4][2,3,4,5,6-I_5-$CHB_{11}H_6$] as a white powder (129 mg, 0.143 mmol, 91% yield).

Reaction scale-up: To a 100 mL round bottom flask equipped with a magnetic stir bar, [NEt_4][1-COOH-$CB_{11}H_{11}$] (500 mg, 1.0 equiv, 1.58 mmol) and NIS (2.13 g, 6.0 equiv, 9.467 mmol) were added. Then $Pd(OAc)_2$ solution (9 mg $Pd(OAc)_2$ in 30 mL anhydrous 1,4-dioxane; 2.5 mol%, 0.04 mmol) and deionized H_2O (3.6 mL) were added. The reaction mixture was stirred for 30 min at 25 °C. Aqueous Na_2SO_3 solution (298 mg in 30 mL deionized H_2O; 1.5 equiv, 2.37 mmol) was added to the mixture dropwise. NEt_4Br (994 mg, 3 equiv, 4.73 mmol) was added to precipitate the product under sonification. 1,4-dioxane was then removed under reduced pressure. The resulting mixture was placed in a glass frit funnel (F porosity), the aqueous layer was removed, and the remaining solid was washed three times with H_2O and dried in a vacuum to give salt [NEt_4][2,3,4,5,6-I_5-$CHB_{11}H_6$] as a white powder (1.31 g, 1.45 mmol, 92% yield).

$^{1}H\{^{11}B\}$ NMR (500 MHz, acetonitrile-d_3, 296 K): δ 3.15 (q, J = 7.3 Hz, 8H, cation CH_2), 3.08 (s, 1H, cage CH), 2.67 (s, 5H, BH), 2.09 (broad signal overlapping with H_2O signal, 1H, BH), 1.21 (tt, J = 7.3Hz, 1.9 Hz, 12H, cation CH_3).

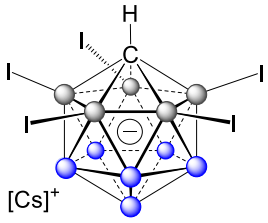
$^{11}B\{^{1}H\}$ NMR (128 MHz, acetone-d_6, 296 K): δ -1.66 (1B), -9.08 (5B), -23.17 (5B).

$^{13}C\{^{1}H\}$ NMR (101 MHz, acetone-d_6, 296 K): δ 64.87 (cage C),52.01 (cation CH2), 6.70 (cation CH_3).

HRMS (ESI): *m*/*z* Calcd for $[CB_{11}I_5H_7]^-$, 772.6867; found, 772.6877.

IR (KBr): ν 3551, 3483, 3416, 3032, 2560, 1618, 1482, 1102, 912, 841, 782, 617.

Ion Exchange:



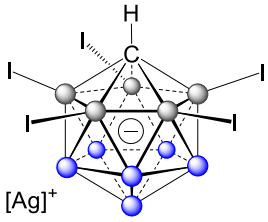
[Cs][2,3,4,5,6-I_5-$CHB_{11}H_6$]

To a 100 mL glass bottle equipped with a magnetic stir bar, [NEt_4][2,3,4,5,6-I_5-$CHB_{11}H_6$] (300mg, 1.0 equiv, 0.3330 mmol), deionized H_2O (10 mL), HCl (1M aq.) (0.3 mL, 10.8 equiv, 3.6 mmol) and ether (50mL) were added. The mixture was stirred at room temperature until the solution became clear. The aqueous phase was extracted with ether (3 x 10mL), then then the combined organic phases were treated with deionized H_2O (10 mL) and Cs_2CO_3 (108.6 mg, 1.0 equiv, 0.3330 mmol). All volatiles were removed in a vacuum, then the remaining solids were dissolved in acetone and filtered. The filtrate contained the desired product and was concentrated on a rotary evaporator to give a solid. It was further dried in a vacuum to give salt [Cs][2,3,4,5,6-I_5-$CHB_{11}H_6$] as a brownish-gray solid (284 mg, 0.3135 mmol, 94% yield).

$^{1}H\{^{11}B\}$ NMR (400 MHz, acetone-d_6, 296 K): 2.78 (s, 1H, cage CH), 2.74 (s, 5H, BH), 2.14 (s, 1H, BH).

$^{11}B\{^{1}H\}$ NMR (128 MHz, acetone-d_6, 296 K): δ -1.60 (1B), -9.05 (5B), -23.17 (5B).

$^{13}C\{^{1}H\}$ NMR (101 MHz, acetone-d_6, 296 K): δ 65.88 (cage C).



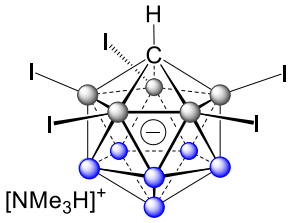
[Ag][2,3,4,5,6-I_5-$CHB_{11}H_6$]

To a 10 mL glass vial equipped with a magnetic stir bar, [Cs][2,3,4,5,6-I_5-$CHB_{11}H_6$] (100 mg,1.0 equiv, 0.1104 mmol) was added. Deionized H_2O (1.0 mL) was added, and the mixture was heated to a boil, upon which the solution became clear. $AgNO_3$ (20.73 mg, 1.1 equiv, 0.1219 mmol) was dissolved in deionized H_2O (0.4 mL) and then slowly added dropwise. A white precipitate was obtained, which was filtered and washed with cold water and dried in a vacuum to give salt [Ag][2,3,4,5,6-I_5-$CHB_{11}H_6$] as an off-white solid (87.2 mg, 0.0989 mmol, 90% yield).

$^{1}H\{^{11}B\}$ NMR (400 MHz, acetonitrile-d_3, 296 K): δ 3.10 (s, 1H, cage CH), 2.67 (s, 5H, BH), 2.08 (s, 1H, BH).

$^{11}B\{^{1}H\}$ NMR (128 MHz, acetonitrile-d_3, 296 K): δ -1.65 (1B), -9.02 (5B), -23.28 (5B).

$^{13}C\{^{1}H\}$ NMR (101 MHz, acetonitrile-d_3, 296 K): δ 64.98 (cage C).



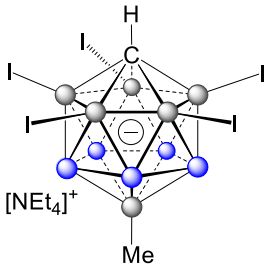
[Me_3NH][2,3,4,5,6-I_5-$CHB_{11}H_6$]

To a 10 mL glass vial equipped with a magnetic stir bar, [Cs][2,3,4,5,6-I_5-$CHB_{11}H_6$] (90 mg, 1.0 equiv, 0.0994 mmol) was added. Then deionized H_2O (1.0 mL) was added, and the mixture was heated to a boil, upon which the solution became clear. Me_3N·HCl (28.8 mg, 3.0 equiv, 0.2982 mmol) was dissolved in deionized H_2O (0.4 mL) and then slowly added dropwise. A white precipitate was obtained, which was filtered and washed with cold water and dried in a vacuum to give salt [Me_3NH][2,3,4,5,6-I_5-$CHB_{11}H_6$] as a white solid (72.9 mg, 0.0874mmol, 88% yield).

$^{1}H\{^{11}B\}$ NMR (400 MHz, acetonitrile-d_3, 296 K): 3.23 (s, 9H, cation CH_3), 2.90 (broad signal, 1H, cation NH), 2.78 (s, 1H, cage CH), 2.74 (s, 5H, BH), 2.14 (s, 1H, BH).

$^{11}B\{^{1}H\}$ NMR (128 MHz, acetonitrile-d_3, 296 K): δ -1.60 (1B), -9.06 (5B), -23.17 (5B).

$^{13}C\{^{1}H\}$ NMR (101 MHz, acetonitrile-d_3, 296 K): δ 65.90 (cage C), 46.27 (cation CH_3).



8b

Starting from (20.1 mg, 0.0604 mml) of $[NEt_4][1\text{-}COOH\text{-}12\text{-}Me\text{-}CB_{11}H_{10}]$ and following the procedure for the synthesis of $[NEt_4][2,3,4,5,6\text{-}I_5\text{-}CHB_{11}H_6]$, $[NEt_4][2,3,4,5,6\text{-}I_5\text{-}12\text{-}Me\text{-}CHB_{11}H_5]$ was obtained as a white solid (43.8 mg, 0.0478 mmol, 79% yield).

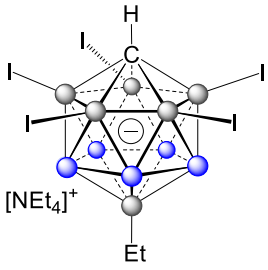
$^{1}H\{^{11}B\}$ NMR (500 MHz, acetonitrile-d_3, 296 K): δ 3.15 (q, J = 7.3 Hz, 8H, cation CH_2), 2.81 (s, 1H, cage CH), 2.67 (s, 5H, BH), 1.21 (tt, J = 7.3Hz, 1.9 Hz, 12H, cation CH_3), 0.00 (s, 3H, methyl CH_3).

$^{11}B\{^{1}H\}$ NMR (128 MHz, acetone-d_6, 296 K): δ 7.51 (1B), -7.96 (5B), -23.71 (5B).

$^{13}C\{^{1}H\}$ NMR (101 MHz, acetone-d_6, 296 K): δ 58.69(cage C),53.03 (cation CH_2), 7.72 (cation CH_3). The B-CH_3 signal could not be detected unambiguously but was detected indirectly using an HSQC spectrum at 4.1 ppm.

HRMS (ESI): *m/z* Calcd for $[C_2B_{11}I_5H_9]^-$, 786.7024; found, 786.7039.

IR(KBr): ν 3436, 2986, 2565.88, 1631, 1480, 1182, 1096, 912, 835, 697.



8c

Starting from (20.0 mg, 0.0580 mml) of $[NEt_4][1\text{-}COOH\text{-}12\text{-}Et\text{-}CB_{11}H_{10}]$ and following the procedure for the synthesis of $[NEt_4][2,3,4,5,6\text{-}I_5\text{-}CHB_{11}H_6]$, $[NEt_4][2,3,4,5,6\text{-}I_5\text{-}12\text{-}Et\text{-}CHB_{11}H_5]$ was obtained as a white solid (39.0 mg, 0.0419 mmol, 72% yield).

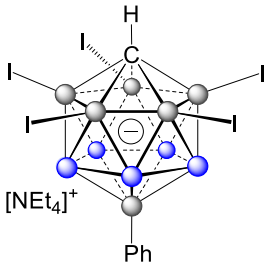
$^{1}H\{^{11}B\}$ NMR (500 MHz, acetonitrile-d_3, 296 K): δ 3.15 (q, J = 7.3 Hz, 8H, cation CH_2), 2.86 (s, 1H, cage CH), 2.65 (s, 5H, BH), 1.21 (tt, J = 7.3Hz, 1.9 Hz, 12H, cation CH_3), 0.75 (t, 3H, ethyl CH_3), 0.49 (t, 2H, ethyl CH_2).

$^{11}B\{^{1}H\}$ NMR (128 MHz, acetone-d_6, 296 K): δ 9.64 (1B), -8.56 (5B), -23.81 (5B).

$^{13}C\{^{1}H\}$ NMR (101 MHz, acetone-d_6, 296 K): δ 59.38 (cage C), 53.02 (cation CH_2), 13.79 (ethyl CH_3), 7.71 (cation CH_3).The B-CH_2 signal could not be detected unambiguously but was detected indirectly using HSQC and HMBC spectra at 12.5 ppm.

HRMS (ESI): *m/z* Calcd for $[C_3B_{11}I_5H_{11}]^-$, 800.7180; found, 800.7188.

IR(KBr): ν 439, 2946, 2560, 1630, 1479, 1170, 1095, 913, 837.



8d

Starting from (50 mg, 0.1272 mml) of $[NEt_4][1\text{-}COOH\text{-}12\text{-}Ph\text{-}CB_{11}H_{10}]$ and following the procedure for the synthesis of $[NEt_4][2,3,4,5,6\text{-}I_5\text{-}CHB_{11}H_6]$, $[NEt_4][2,3,4,5,6\text{-}I_5\text{-}12\text{-}Ph\text{-}CHB_{11}H_5]$ was obtained as a white solid (103.5 mg, 0.1058 mmol, 83% yield).

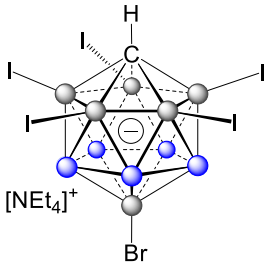
$^{1}H\{^{11}B\}$ NMR (400 MHz, acetone-d_6, 296 K): δ 7.40-7.20 (m, 2H, ArH, ortho), 7.15-7.00 (m, 3H, ArH, meta and para), 3.48(q, J = 7.3 Hz, 8H, cation CH_2), 2.71(s, 1H, cage CH), 2.98 (s, 5H, BH), 1.39 (tt, J = 7.3Hz, 1.9 Hz, 12H, cation CH_3).

$^{11}B\{^{1}H\}$ NMR (128 MHz, acetone-d_6, 296 K): δ 8.16 (1B), -8.52 (5B), -23.67 (5B).

$^{13}C\{^{1}H\}$ NMR (101 MHz, acetone-d_6, 296 K): δ 132.95 (ortho C), 127.78 (meta C), 127.02 (para C), 60.52 (cage C),53.06(cation CH_2), 7.79(cation CH_3). The B-C(ipso) signal could not be detected unambiguously but was detected indirectly using HSQC and HMBC spectra at 143 ppm.

HRMS (ESI): *m/z* Calcd for $[C_7B_{11}I_5H_{11}]^-$, 848.7180.; found, 848.7206.

IR(KBr): ν 3439, 3254, 2924, 2537, 1586, 1464, 1306, 1154, 1033, 690, 551.



8e

Starting from (20.3 mg, 0.0507 mml) of $[NEt_4][1\text{-}COOH\text{-}12\text{-}Br\text{-}CB_{11}H_{10}]$ and following the procedure for the synthesis of $[NEt_4][2,3,4,5,6\text{-}I_5\text{-}CHB_{11}H_6]$, $[NEt_4][2,3,4,5,6\text{-}I_5\text{-}12\text{-}Br\text{-}CHB_{11}H_5]$ was obtained as a white solid after recrystallization from hot methanol (0.2 mL) (38.5 mg, 0.0393 mmol, 78% yield). In this case the co-solvent was 24% HOAc instead of 12% H_2O.

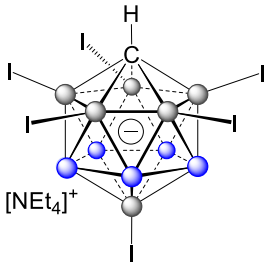
$^{1}H\{^{11}B\}$ NMR (400 MHz, acetonitrile-d_3, 296 K): δ 3.18 (q, J = 7.3 Hz, 8H, cation CH2), 3.10 (s, 1H, cage CH), 3.01(s, 1H, BH), 1.24 (tt, J = 7.3Hz, 1.9 Hz, 12H, cation CH3).

$^{11}B\{^{1}H\}$ NMR (160 MHz, acetone-d_6, 296 K): δ 1.20 (1B), -8.45 (5B), -24.42 (5B).

$^{13}C\{^{1}H\}$ NMR (101 MHz, acetone-d_6, 296 K): δ 58.06 (cage C), 53.03 (cation CH2), 7.71 (cation CH3).

HRMS (ESI): *m/z* Calcd for $[CB_{11}I_5BrH_6]^-$, 850.5973; found, 850.6001

IR(KBr): ν 3450, 2977, 2566, 1795, 1630, 1481, 1170, 1023, 998, 890, 837, 786, 742.



8f

Starting from (50.1 mg, 0.113 mml) of $[NEt_4][1\text{-}COOH\text{-}12\text{-}I\text{-}CB_{11}H_{10}]$ and following the procedure for the synthesis of $[NEt_4][2,3,4,5,6\text{-}I_5\text{-}CHB_{11}H_6]$, $[NEt_4][2,3,4,5,6,12\text{-}I_6\text{-}CHB_{11}H_5]$ was obtained as a white solid after recrystallization from hot methanol (0.5 mL) (85.1 mg, 0.0828 mmol, 73% yield). In this case the co-solvent was 36% HOAc instead of 12% H_2O.

$^{1}H\{^{11}B\}$ NMR (400 MHz, acetone-d_6, 296 K): δ 3.51 (q, J = 7.3 Hz, 8H, cation CH_2), 3.18 (s, 5H, BH), 3.06 (s, 1H, cage CH), 1.41 (tt, J = 7.3Hz, 1.9 Hz, 12H, cation CH_3).

$^{11}B\{^{1}H\}$ NMR (128 MHz, acetone-d_6, 296 K): δ -7.87 (5B), -15.29 (1B), -23.56 (5B).

$^{13}C\{^{1}H\}$ NMR (101 MHz, acetone-d_6, 296 K): δ 62.20 (cage C), 53.03 (cation CH_2), 7.71(cation CH_3).

HRMS (ESI): *m/z* Calcd for $[CB_{11}I_6H_6]^-$, 898.5834; found, 898.5856.

IR(KBr): ν 3431, 2924, 2567, 1657, 1480, 1391, 1103, 913, 836, 782.

Proposed mechanism for the penta-iodination

A plausible mechanism for the penta-iodination is displayed in Figure S4. Binding of the carboxylate group of $[NEt_4][1\text{-}COOH\text{-}12\text{-}X\text{-}CB_{11}H_{10}]$ (X = H, Me, Et, Ph, I, Br) to Pd(II) affords the initial intermediate **IM-a**. Cyclometalation–deprotonation then gives palladacycle **IM-Pd** with a direct B–Pd bond. Intermediate **IM-b** is formed by NIS insertion, which is followed by elimination to furnish complex **IM-a'.** Then succinimide leaves the cycle, and the Pd center activates a geminal or adjacent B-H position. Subsequent iodination steps follow in a similar manner. Finally, the carboxylate group is eliminated as CO_2, and Pd(II) combines with another $[NEt_4][1\text{-}COOH\text{-}12\text{-}X\text{-}CB_{11}H_{10}]$ to enter the next cycle.

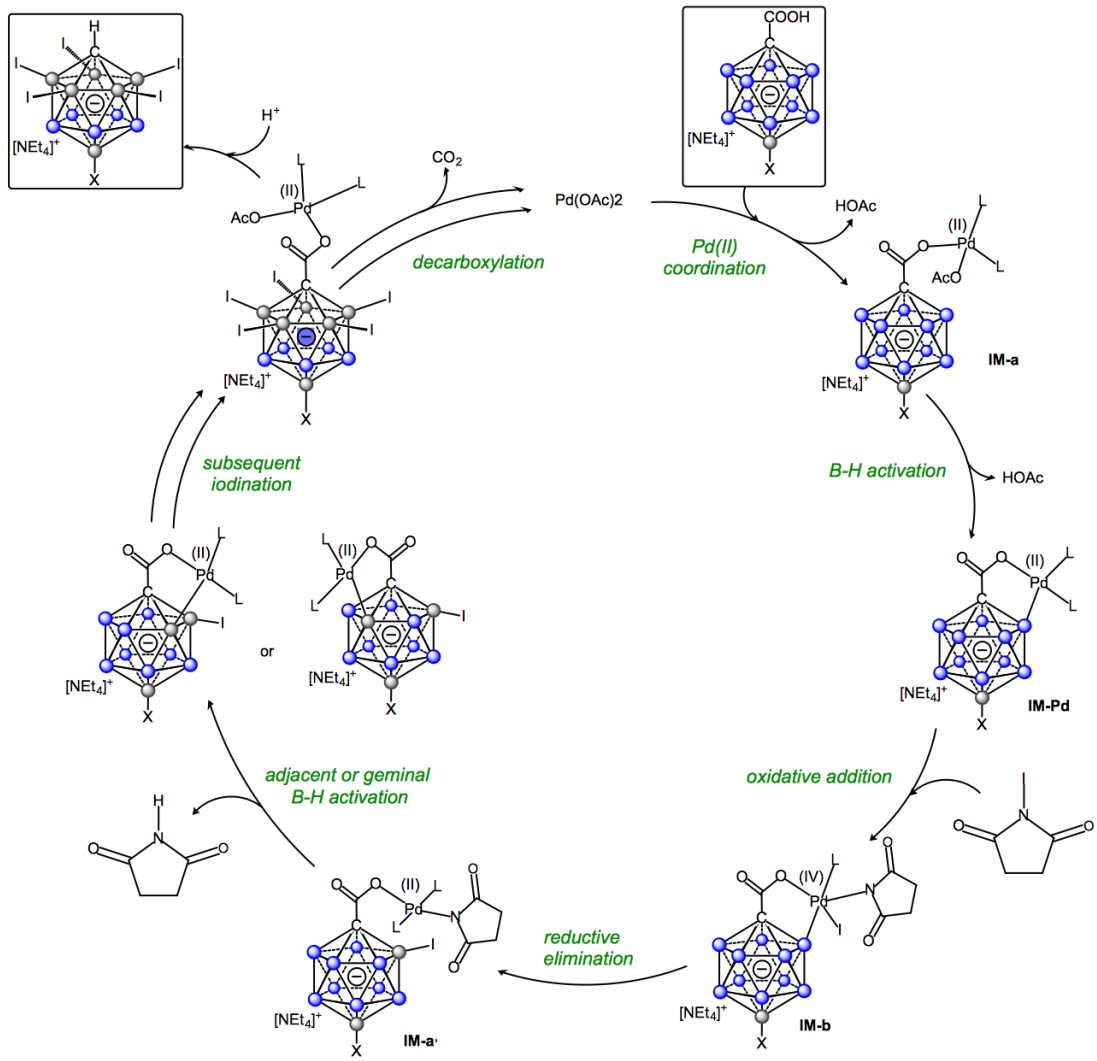


Figure S4. Proposed mechanism for the five-fold B-H activation/B-I coupling cascade; L=solvent molecule or AcO^- (blue spheres = BH, gray spheres = B).

3 X-ray Crystallography

Crystal structure of [NEt_4][2,3,4,5,6-I_5-$CHB_{11}H_6$] (CCDC 2247602)

[NEt_4][2,3,4,5,6-I_5-$CHB_{11}H_6$] (20 mg, 0.018 mmol) was dissolved in boiling methanol (0.2 mL) in a 2.5 mL glass vial. The resulting colorless solution was slowly cooled down to 25 °C and then placed in a refrigerator at 13 °C. Within 12 h, single crystals suitable for X-ray diffraction were obtained.

```
Bond precision:   C-C = 0.0077 A          Wavelength=0.71073

Cell:             a=9.3682(3)     b=11.1316(3)        c=25.1118(9)
                  alpha=90        beta=92.214(1)      gamma=90
Temperature:      170 K

                 Calculated                Reported
Volume           2616.78(14)               2616.78(14)
Space group      P 21/c                    P 1 21/c 1
Hall group       -P 2ybc                   -P 2ybc
Moiety formula   C H7 B11 I5, C8 H20 N     C H7 B11 I5, C8 H20 N
Sum formula      C9 H27 B11 I5 N           C9 H27 B11 I5 N
Mr               902.73                    902.72
Dx,g cm-3        2.291                     2.291
Z                4                         4
Mu (mm-1)        5.941                     5.941
F000             1632.0                    1632.0
F000'            1623.02
h,k,lmax         12,14,32                  12,14,32
Nref             5774                      5771
Tmin,Tmax        0.211,0.552               0.301,0.746
Tmin'            0.085

Correction method= # Reported T Limits: Tmin=0.301 Tmax=0.746
AbsCorr = MULTI-SCAN

Data completeness= 0.999          Theta(max)= 27.116

R(reflections)= 0.0289( 5446)                wR2(reflections)=
                                             0.0649( 5771)
S = 1.110                 Npar= 239
```

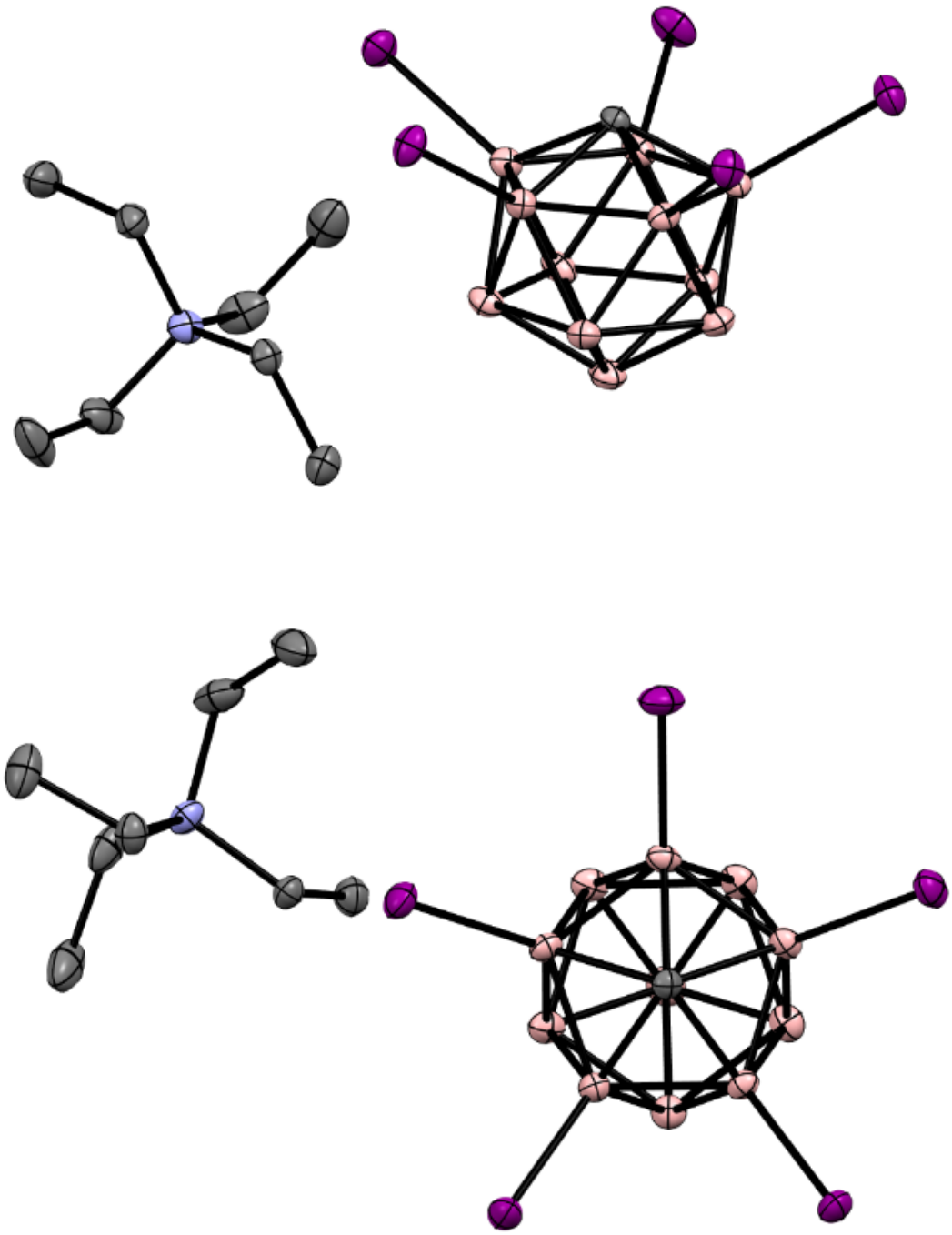


Figure S5. ORTEP representation of $[NEt_4][2,3,4,5,6\text{-}I_5\text{-}CHB_{11}H_6]$ (top: side view; bottom: view along C(cage)-B12 axis); hydrogen atoms are omitted for clarity; 30% displacement ellipsoids.

Crystal structure of [NEt_4][2,3,4,5,6-I_5-12-Ph-$CHB_{11}H_5$] (CCDC 2247603)

[NEt_4][2,3,4,5,6-I_5-12-Ph-$CHB_{11}H_5$] (20 mg, 0.018 mmol) was dissolved in boilng methanol (0.2 mL) in a 2.5 mL glass vial. The resulting colorless solution was slowly cooled down to 25 °C and then placed in a refrigerator at 13 °C. Within 12 h, single crystals suitable for X-ray diffraction were obtained.

```
Bond precision:   C-C = 0.0081 A          Wavelength=0.71073

Cell:          a=9.8044(5)        b=11.8241(6)      c=13.6777(7)
               alpha=100.782(2)   beta=98.898(2)    gamma=94.691(2)
Temperature:   296 K

                Calculated                Reported
Volume          1528.85(14)               1528.85(14)
Space group     P -1                      P -1
Hall group      -P 1                      -P 1
Moiety formula  C7 H11 B11 I5, C8 H20 N   C7 H11 B11 I5, C8 H20 N
Sum formula     C15 H31 B11 I5 N          C15 H31 B11 I5 N
Mr              978.82                    978.82
Dx,g cm-3       2.126                     2.126
Z               2                         2
Mu (mm-1)       5.093                     5.093
F000            896.0                     896.0
F000'           891.52
h,k,lmax        12,15,17                  12,15,17
Nref            6768                      6760
Tmin,Tmax       0.135,0.516               0.498,0.746
Tmin'           0.102

Correction method= # Reported T Limits: Tmin=0.498 Tmax=0.746
AbsCorr = MULTI-SCAN

Data completeness= 0.999          Theta(max)= 27.124

R(reflections)= 0.0329( 6176)                wR2(reflections)=
                                             0.0742( 6760)
S = 1.026                 Npar= 293
```

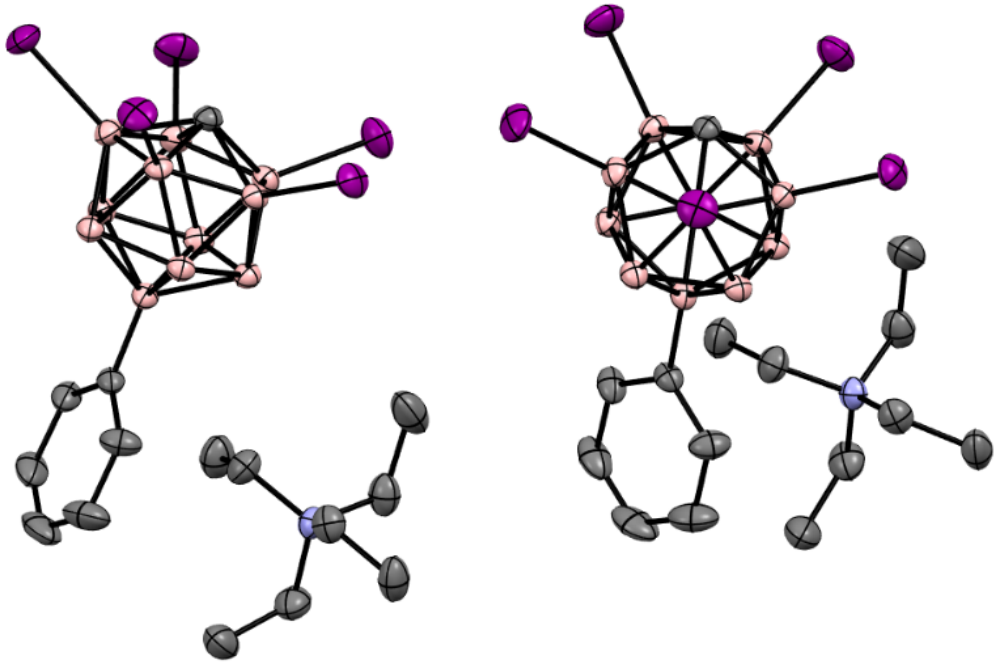


Figure S6. ORTEP representation of $[NEt_4][2,3,4,5,6\text{-}I_5\text{-}12\text{-}Ph\text{-}CHB_{11}H_5]$ (left: side view; right: view along B_2-B_9 axis); hydrogen atoms are omitted for clarity; 30% displacement ellipsoids.

Crystal structure of [NEt_4][2,3,4,5,6,12-I_6-$CHB_{11}H_5$] (CCDC 2247604)

[NEt_4][2,3,4,5,6-I_5-12-I-$CHB_{11}H_5$] (20 mg, 0.018 mmol) was dissolved in boiling methanol (1.0 mL) in a 2.5 mL glass vial. The resulting colorless solution was slowly cool down to RT. The resulting colorless solution was slowly cooled down to 25 °C and then placed in a refrigerator at 13 °C. Within 12 h, single crystals suitable for X-ray diffraction were obtained.

```
Bond precision:   B- B = 0.0129 A          Wavelength=0.71073

Cell:             a=9.4164(6)     b=16.9039(12)       c=17.2551(12)
                  alpha=90        beta=90.216(2)      gamma=90
Temperature:      170 K

                 Calculated                  Reported
Volume           2746.5(3)                   2746.5(3)
Space group      P 21/c                      P 1 21/c 1
Hall group       -P 2ybc                     -P 2ybc
Moiety formula   C H6 B11 I6, C8 H20 N       C H6 B11 I6, C8 H20 N
Sum formula      C9 H26 B11 I6 N             C9 H26 B11 I6 N
Mr               1028.62                     1028.62
Dx,g cm-3        2.488                       2.488
Z                4                           4
Mu (mm-1)        6.786                       6.786
F000             1840.0                      1840.0
F000'            1829.24
h,k,lmax         13,24,24                    13,24,24
Nref             8426                        8376
Tmin,Tmax        0.672,0.816                 0.629,0.746
Tmin'            0.538

Correction method= # Reported T Limits: Tmin=0.629 Tmax=0.746
AbsCorr = MULTI-SCAN

Data completeness= 0.994           Theta(max)= 30.548

R(reflections)= 0.0524( 7006)                    wR2(reflections)=
                                                 0.1219( 8376)
S = 1.093                Npar= 334
```

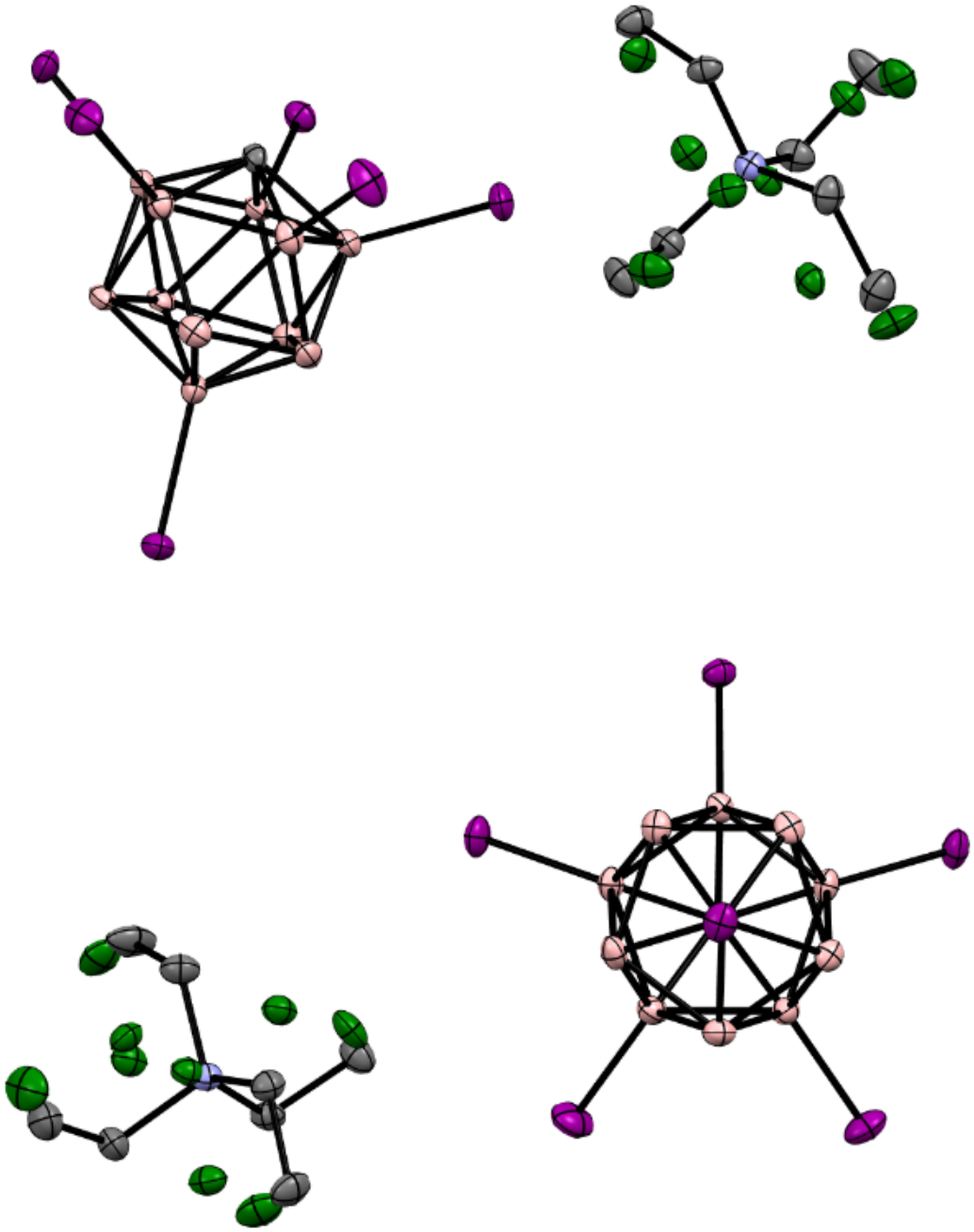


Figure S7. ORTEP representation of $[NEt_4][2,3,4,5,6,12\text{-}I_6\text{-}CHB_{11}H_5]$ (top: side view; bottom: view along B12-C(cage) axis); hydrogen atoms are omitted for clarity; 30% displacement ellipsoids. Minor component of disordered cation shown in green.

4 References

[1] Shen, Y. J.; Pan, Y. N.; Zhang, K.; Liang, X. W.; Liu, J.Y.; Spingler B.; Duttwyler, S. *Dalton Trans*. **2017**, *46*, 3135.

[2] Himmelspach, A.; Reiss, G. J.; Finze, M. *Inorg. Chem*. **2012**, *51*, 2679–2688.

[3] Lin, F. R.; Shen, Y. J.; Zhang, Y. B.; Sun, Y. J.; Liu, J. Y.; Duttwyler, S. *Chem. Eur. J*. **2018**, *24*, 551–555.

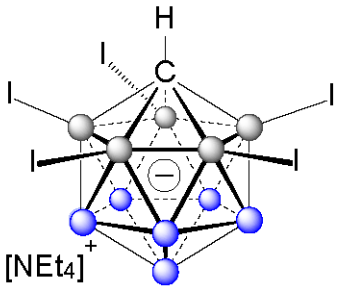
[4] Shen, Y. J.; Zhang, K.; Liang, X. W.; Dontha, R.; Duttwyler, S. *Chem. Sci.* **2019**, *10*, 4177.

[5] Grüner, B.; Janoušek, Z.; King, B. T.; Woodford, J. N.; Wang, C. H.; Všetečka V.; Michl J. *J. Am. Chem. Soc.* **1999**, *121*, 3122–3126.

5 NMR Spectra, Mass Spectra and IR spectra

Displayed on p. S24–77.

[NEt4][2,3,4,5,6-5I-CHB11H6]
500MHz,1H{11B},6mg,0.6mL Acetonitrile-d3*,296K

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Current Data Parameters
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EXPNO                  1
PROCNO                 1

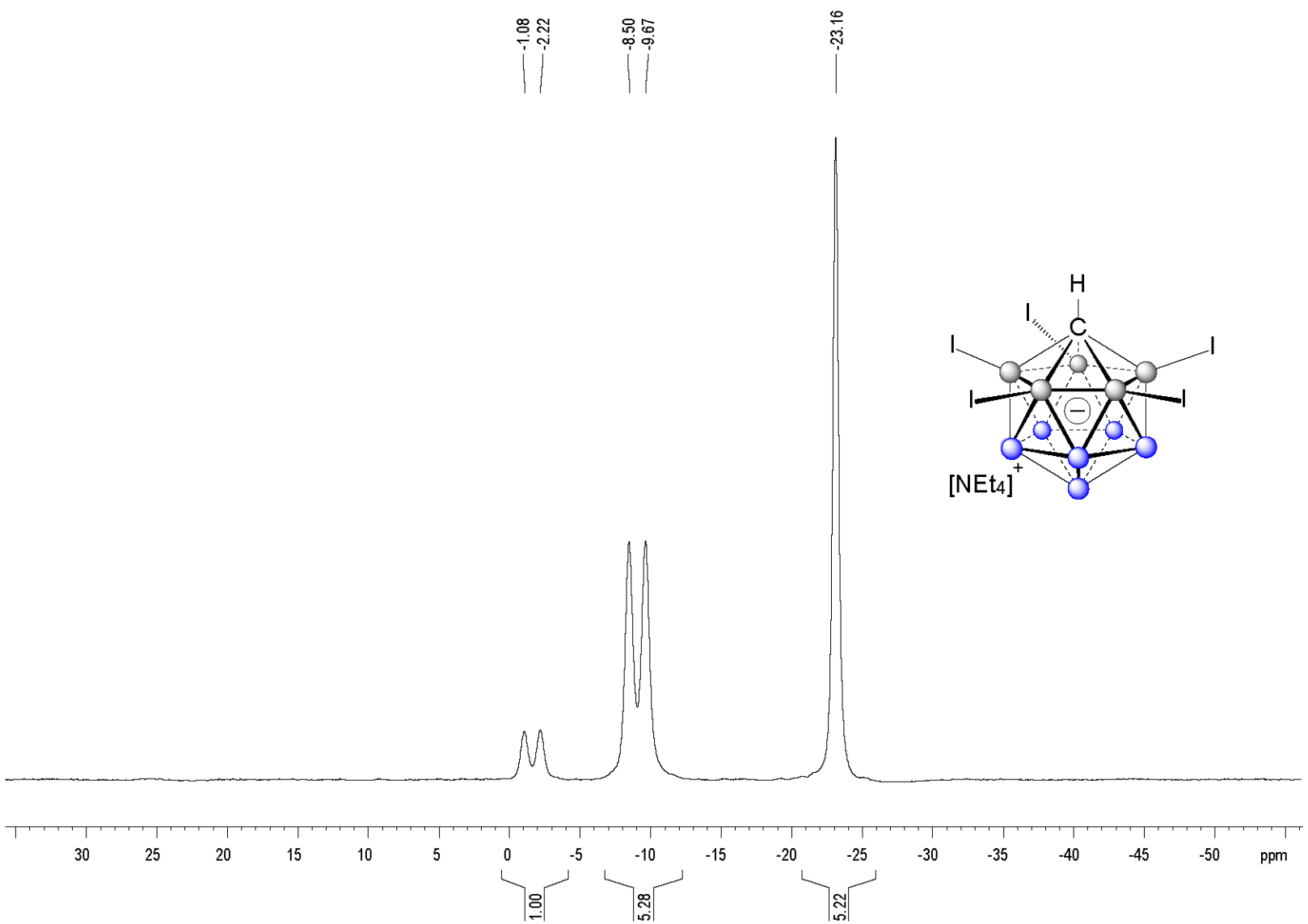
F2 - Acquisition Parameters
Date_           20230206
Time_             19.05
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           zgig30
TD                65536
SOLVENT           CD3CN
NS                   16
DS                    0
SWH           12500.000 Hz
FIDRES         0.190735 Hz
AQ            2.6214399 sec
RG                  181
DW               40.000 usec
DE                6.50 usec
TE               295.5 K
D1            5.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        500.1735009 MHz
NUC1                1H
P1                12.00 usec
PLW1        19.00000000 W

======== CHANNEL f2 ========
SFO2        160.4744030 MHz
NUC2               11B
CPDPRG[2          garp
PCPD2           100.00 usec
PLW2        95.00000000 W
PLW12        1.63030005 W

F2 - Processing parameters
SI                65536
SF          500.1700143 MHz
WDW                EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.00
```

[NEt4][2,3,4,5,6-5I-CHB11H6]
128M,11B NMR,10mg,0.6mL Acetone-d6*,296k



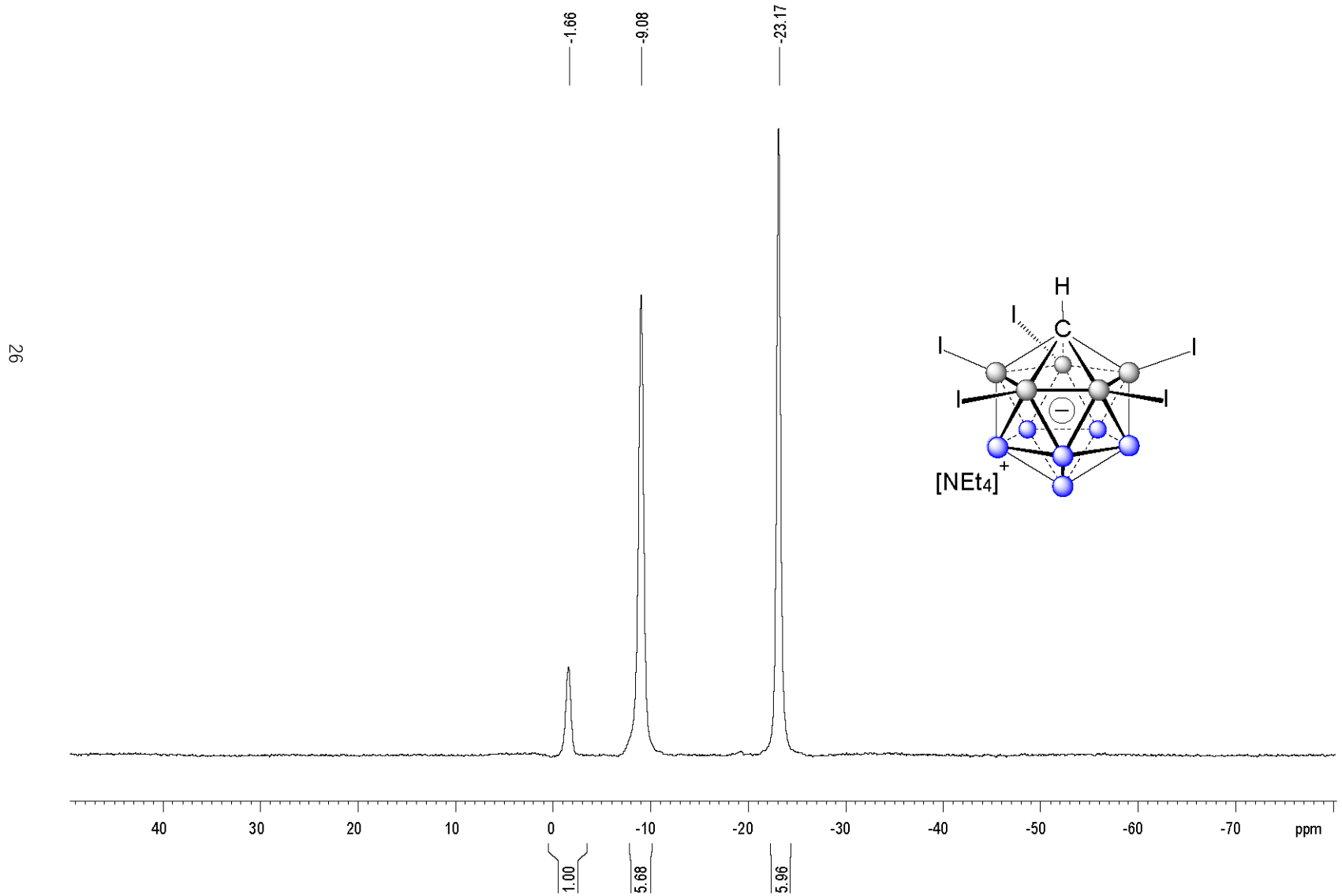

```
Current Data Parameters
NAME      11B NMR,400M,Aceton
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_           20221022
Time                18.20
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG                zg
TD                  65536
SOLVENT           Acetone
NS                    128
DS                      4
SWH             25510.203 Hz
FIDRES          0.389255 Hz
AQ              1.2845056 sec
RG                 193.34
DW                19.600 usec
DE                 6.50 usec
TE                295.2 K
D1           1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   11B
P1                    9.93 usec
PLW1         52.96599960 W
SFO1         128.3776052 MHz

F2 - Processing parameters
SI                  32768
SF           128.3776050 MHz
WDW                    EM
SSB                     0
LB                  10.00 Hz
GB                      0
PC                   1.40
```

[NEt4][2,3,4,5,6-5I-CHB11H6]
128MHz,11B{1H},10mg,0.6mL Acetone-d6*,296K

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Current Data Parameters
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EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
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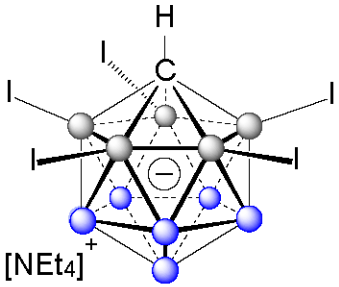
Time_             18.14
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  128
DS                    4
SWH           25510.203 Hz
FIDRES         0.389255 Hz
AQ             1.2845056 sec
RG                193.34
DW                19.600 usec
DE                 6.50 usec
TE                296.5 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 11B
P1                  9.93 usec
PLW1         52.96599960 W
SFO1        128.3776050 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PLW2         12.50000000 W
PLW12         0.43945000 W
PLW13         0.28125000 W
SFO2        400.1320007 MHz

F2 - Processing parameters
SI                32768
SF          128.3776050 MHz
WDW                  EM
SSB                   0
LB                10.00 Hz
GB                    0
PC                 1.40
```

[NEt4][2,3,4,5,6-5I-CHB11H6]
101MHz,13C{1H} NMR,20mg, 0.6mL Acetone-d6*,296k



27

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Current Data Parameters
NAME      13C{1H} NMR,400M,Aceton
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PROCNO               1

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Time               1.09
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT        Acetone
NS                1024
DS                   4
SWH          29761.904 Hz
FIDRES         0.454131 Hz
AQ           1.1010048 sec
RG              193.34
DW              16.800 usec
DE               6.50 usec
TE              296.6 K
D1          1.50000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1               10.00 usec
PLW1        53.00000000 W
SFO1       100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2                1H
PCPD2            80.00 usec
PLW2        12.50000000 W
PLW12        0.43945000 W
PLW13        0.28125000 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                32768
SF         100.6127842 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
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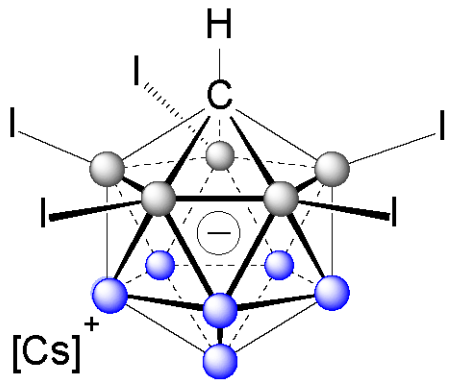
[Cs][2,3,4,5,6-5I-CHB11H6]
400M,1H{11B} NMR,20mg,Acetone-d6*,296K




```
Current Data Parameters
NAME      400M-[Cs][2,3,4,5,6-5I
EXPNO                          2
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20221030
Time                       14.15
INSTRUM                    spect
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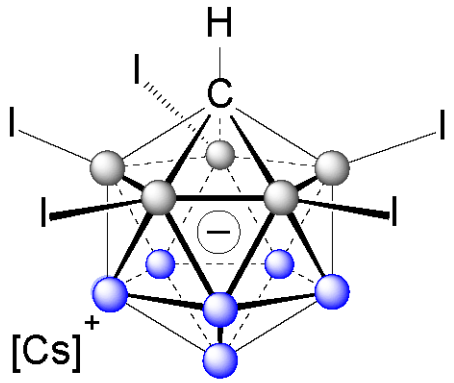
PULPROG                   zgig30
TD                         16384
SOLVENT                  Acetone
NS                            16
DS                             4
SWH                     8012.820 Hz
FIDRES                  0.489064 Hz
AQ                    1.0223616 sec
RG                        193.34
DW                        62.400 usec
DE                         6.50 usec
TE                        296.3 K
D1                   1.00000000 sec
D11                  0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                          1H
P1                        15.00 usec
PLW1                12.50000000 W
SFO1                400.1320007 MHz

======== CHANNEL f2 ========
CPDPRG[2                  garp4
NUC2                         11B
PCPD2                     90.00 usec
PLW2                52.96599960 W
PLW12                0.64477998 W
SFO2                128.3776050 MHz

F2 - Processing parameters
SI                         32768
SF                  400.1300071 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40
```

[Cs][2,3,4,5,6-5I-CHB11H6]
128M,11B NMR,20mg,Acetone-d6*,296K

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Current Data Parameters
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EXPNO                4
PROCNO               1

F2 - Acquisition Parameters
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Time              14.28
INSTRUM           spect
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PULPROG             zg
TD                65536
SOLVENT         Acetone
NS                  128
DS                    4
SWH           25510.203 Hz
FIDRES         0.389255 Hz
AQ            1.2845056 sec
RG               193.34
DW               19.600 usec
DE                6.50 usec
TE               296.1 K
D1            1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                11B
P1                 9.93 usec
PLW1        52.96599960 W
SFO1       128.3776052 MHz

F2 - Processing parameters
SI                32768
SF         128.3776050 MHz
WDW                EM
SSB                 0
LB                1.00 Hz
GB                  0
PC                1.40
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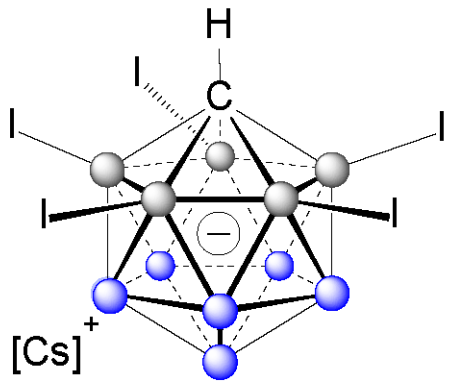
[Cs][2,3,4,5,6-5I-CHB11H6]
128M,11B{1H} NMR,20mg,Acetone-d6*,296K



30

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Current Data Parameters
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EXPNO                        3
PROCNO                       1

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Time                  14.22
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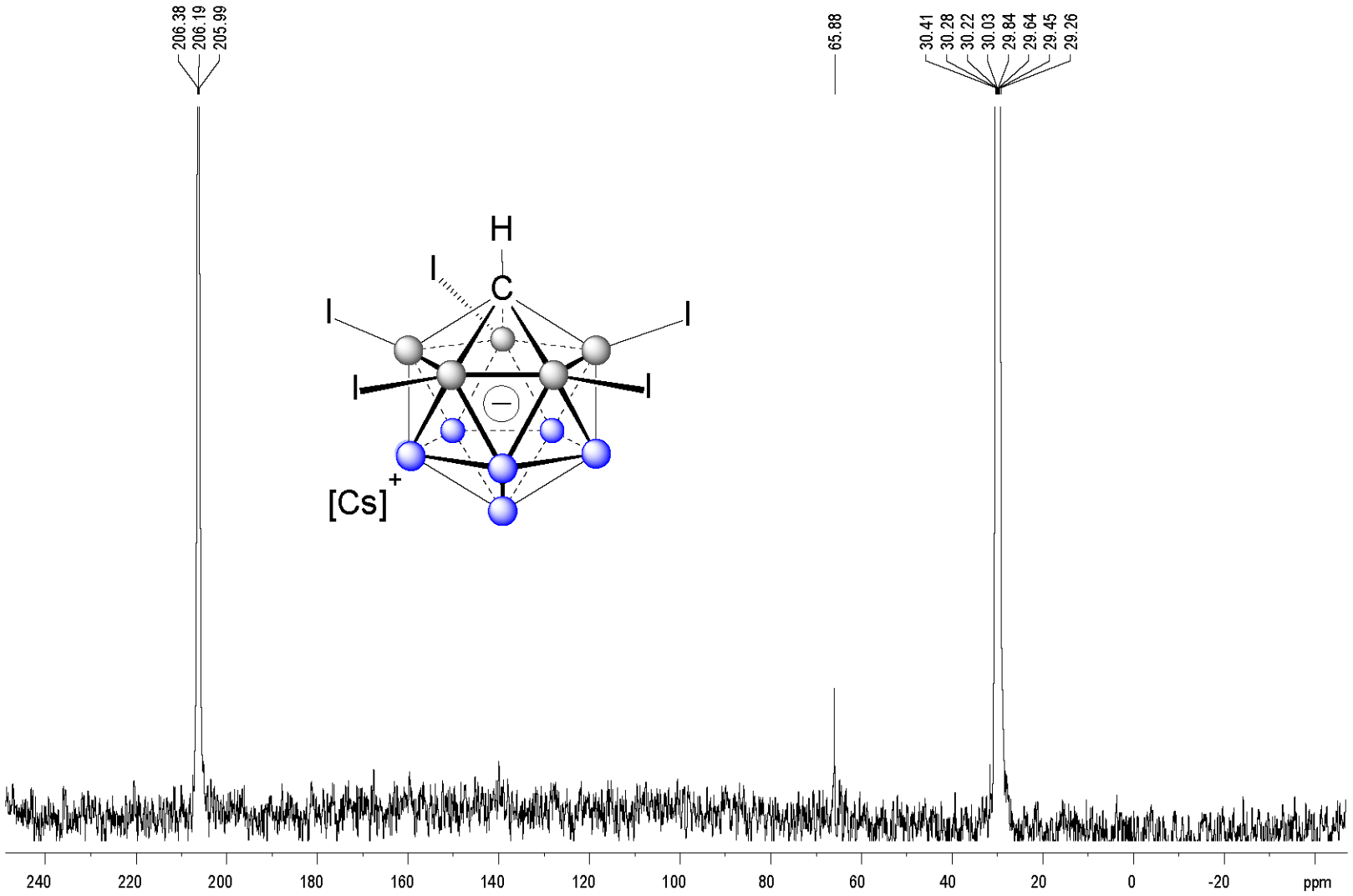
PULPROG              zgpg30
TD                    65536
SOLVENT             Acetone
NS                        128
DS                          4
SWH                25510.203 Hz
FIDRES             0.389255 Hz
AQ                1.2845056 sec
RG                    193.34
DW                    19.600 usec
DE                     6.50 usec
TE                    296.9 K
D1                1.00000000 sec
D11               0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      11B
P1                       9.93 usec
PLW1            52.96599960 W
SFO1           128.3776050 MHz

======== CHANNEL f2 ========
CPDPRG[2             waltz16
NUC2                      1H
PCPD2                  80.00 usec
PLW2            12.50000000 W
PLW12            0.43945000 W
PLW13            0.28125000 W
SFO2           400.1320007 MHz

F2 - Processing parameters
SI                        32768
SF             128.3776050 MHz
WDW                          EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```

[Cs][2,3,4,5,6-5I-CHB11H6]
101M,13C{1H} NMR,20mg,Acetone-d6*,296K




```
Current Data Parameters
NAME      400M-[Cs][2,3,4,5,6-5
EXPNO                        5
PROCNO                       1

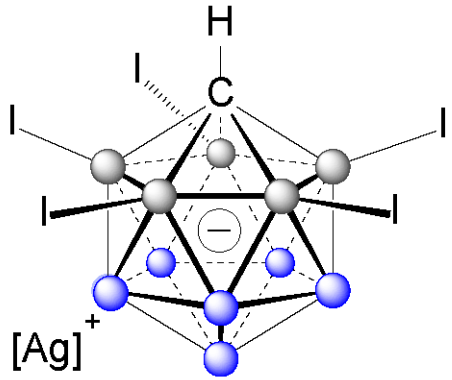
F2 - Acquisition Parameters
Date_              20221030
Time                 14.52
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG              zgpg30
TD                    65536
SOLVENT            Acetone
NS                      512
DS                        4
SWH               29761.904 Hz
FIDRES             0.454131 Hz
AQ                1.1010048 sec
RG                   193.34
DW                   16.800 usec
DE                    6.50 usec
TE                   296.8 K
D1                1.50000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                    10.00 usec
PLW1             53.00000000 W
SFO1            100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG[2             waltz16
NUC2                    1H
PCPD2                 80.00 usec
PLW2             12.50000000 W
PLW12             0.43945000 W
PLW13             0.28125000 W
SFO2            400.1316005 MHz

F2 - Processing parameters
SI                      32768
SF               100.6126827 MHz
WDW                        EM
SSB                         0
LB                    10.00 Hz
GB                          0
PC                     1.40
```

[Ag][2,3,4,5,6-5I-CHB11H6]
400M,1H{11B} NMR,20mg,Acetonitrile-d3*,296K




```
Current Data Parameters
NAME      400M-[Ag][2,3,4,5,6-5
EXPNO                         2
PROCNO                        1

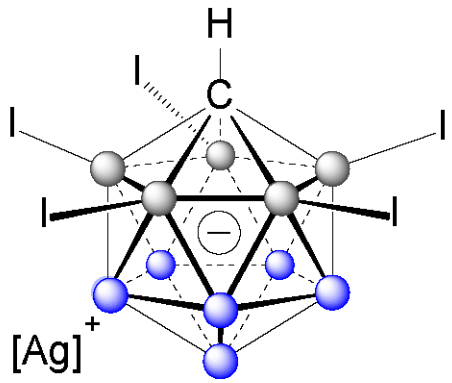
F2 - Acquisition Parameters
Date_                  20221031
Time_                     20.48
INSTRUM                   spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgig30
TD                       16384
SOLVENT                  CD3CN
NS                          16
DS                           4
SWH                   8012.820 Hz
FIDRES                0.489064 Hz
AQ                    1.0223616 sec
RG                       193.34
DW                       62.400 usec
DE                        6.50 usec
TE                       296.3 K
D1                    1.00000000 sec
D11                   0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                       15.00 usec
PLW1                12.50000000 W
SFO1                400.1320007 MHz

======== CHANNEL f2 ========
CPDPRG[2                 garp4
NUC2                        11B
PCPD2                    90.00 usec
PLW2                52.96599960 W
PLW12                0.64477998 W
SFO2                128.3776050 MHz

F2 - Processing parameters
SI                       32768
SF                  400.1300116 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```

[Ag][2,3,4,5,6-5I-CHB11H6]
128M,11B NMR,20mg,Acetonitrile-d3*,296K



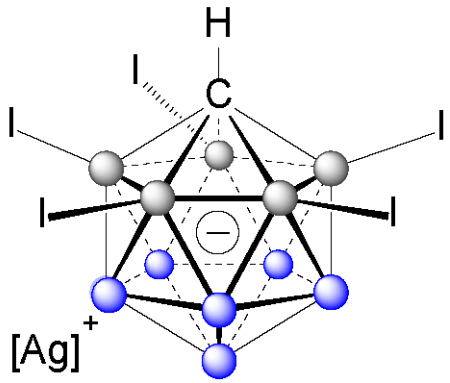

```
Current Data Parameters
NAME      400M-[Ag][2,3,4,5,6-5
EXPNO                        4
PROCNO                       1

F2 - Acquisition Parameters
Date_              20221030
Time                 13.42
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG                 zg
TD                   65536
SOLVENT              CD3CN
NS                     128
DS                       4
SWH              25510.203 Hz
FIDRES            0.389255 Hz
AQ               1.2845056 sec
RG                  193.34
DW                  19.600 usec
DE                   6.50 usec
TE                  296.5 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   11B
P1                    9.93 usec
PLW1          52.96599960 W
SFO1          128.3776052 MHz

F2 - Processing parameters
SI                   32768
SF            128.3776050 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

[Ag][2,3,4,5,6-5I-CHB11H6]
128M,11B{1H} NMR,20mg,Acetonitrile-d3*,296K




```
Current Data Parameters
NAME      400M-[Ag][2,3,4,5,6-
EXPNO                    3
PROCNO                   1

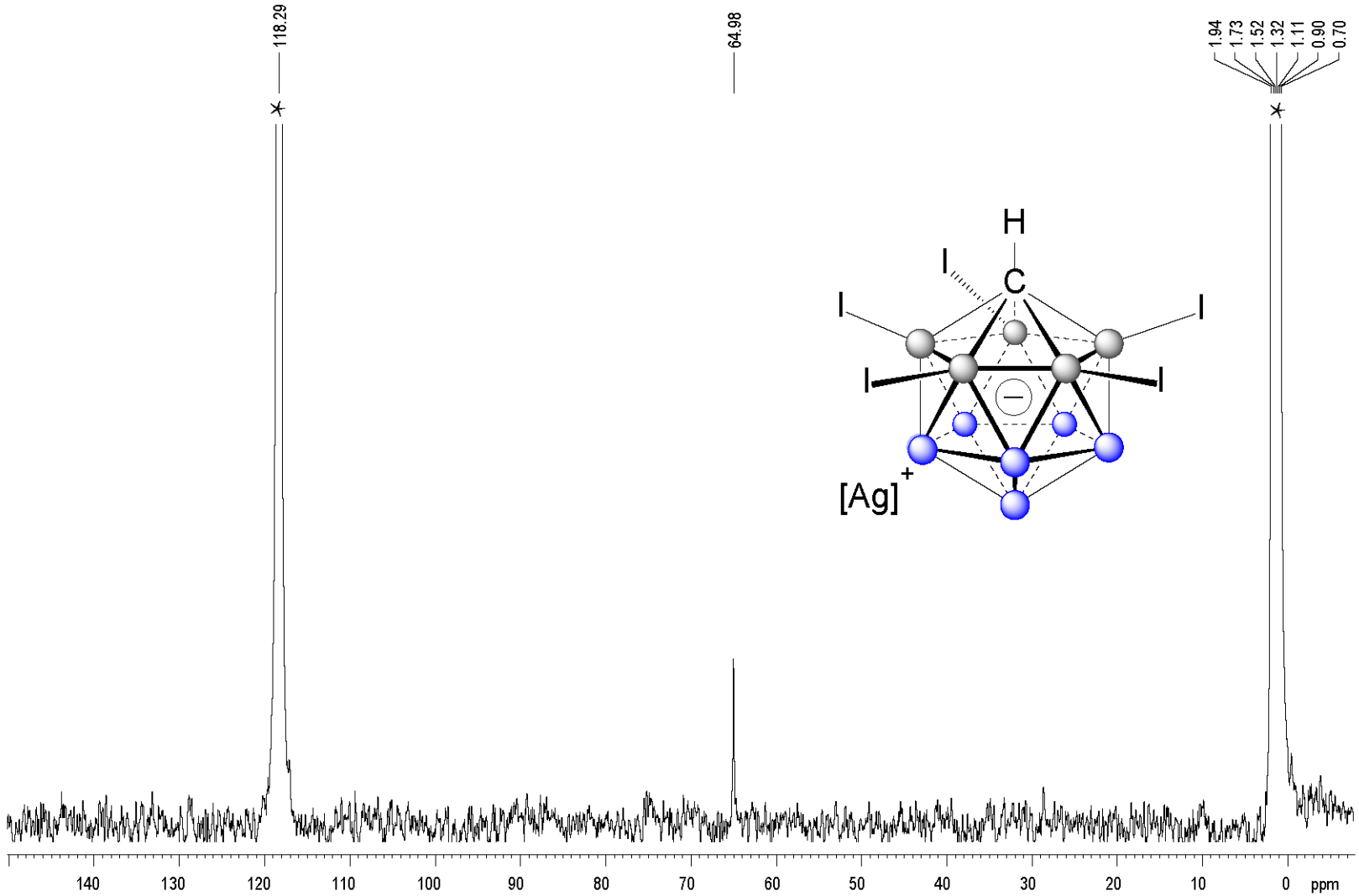
F2 - Acquisition Parameters
Date_           20221030
Time               13.36
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CD3CN
NS                  128
DS                    4
SWH           25510.203 Hz
FIDRES         0.389255 Hz
AQ            1.2845056 sec
RG               193.34
DW               19.600 usec
DE                6.50 usec
TE               297.5 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 11B
P1                  9.93 usec
PLW1        52.96599960 W
SFO1       128.3776050 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2              80.00 usec
PLW2        12.50000000 W
PLW12        0.43945000 W
PLW13        0.28125000 W
SFO2       400.1320007 MHz

F2 - Processing parameters
SI                32768
SF         128.3776050 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

[Ag][2,3,4,5,6-5I-CHB11H6]
101M,13C{1H} NMR,16mg dissolved in 0.6 mL Acetonitrile-d3*,296K

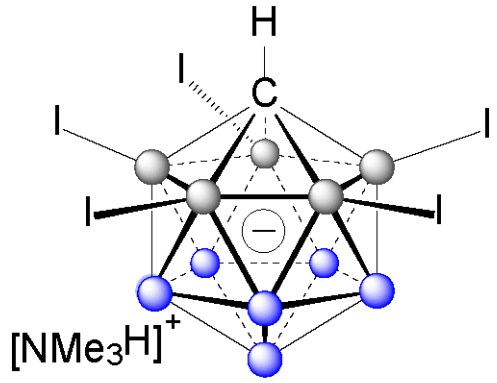



```
Current Data Parameters
NAME         0317-LCH-5I
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230318
Time                8.06 h
INSTRUM           Avance
PROBHD   Z163739_0656 (
PULPROG          zgpg30
TD                65536
SOLVENT            CD3CN
NS                   512
DS                     4
SWH            25906.736 Hz
FIDRES             0.790611 Hz
AQ              1.2648448 sec
RG                   101
DW                19.300 usec
DE                 6.50 usec
TE                 295.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1
SFO1          100.6248421 MHz
NUC1                 13C
P0                 2.67 usec
P1                 8.00 usec
PLW1          94.05000305 W
SFO2          400.1316005 MHz
NUC2                 1H
CPDPRG[2          waltz65
PCPD2             90.00 usec
PLW2          22.48500061 W
PLW12          0.17766000 W
PLW13          0.08936300 W

F2 - Processing parameters
SI                 32768
SF            100.6126746 MHz
WDW                   EM
SSB                    0
LB                 10.00 Hz
GB                     0
PC                  1.40
```

[NMe3H][2,3,4,5,6-5I-CHB11H6]
400M,1H{11B} NMR,20mg,Acetone-d6,296K




```
Current Data Parameters
NAME      400M-[NMe3H][2,3,4,5,
EXPNO                      2
PROCNO                     1

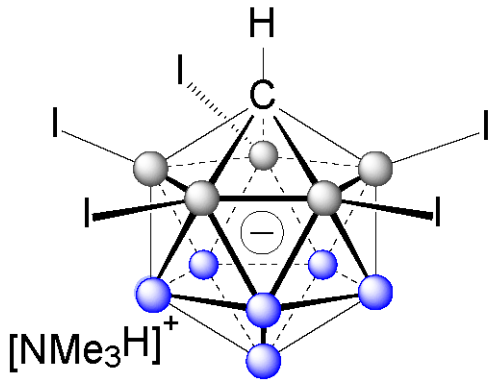
F2 - Acquisition Parameters
Date_              20221030
Time                  15.00
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG              zgig30
TD                    16384
SOLVENT           Acetone
NS                       16
DS                        4
SWH                8012.820 Hz
FIDRES             0.489064 Hz
AQ                1.0223616 sec
RG                   193.34
DW                   62.400 usec
DE                    6.50 usec
TE                   296.1 K
D1              1.00000000 sec
D11             0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                    15.00 usec
PLW1           12.50000000 W
SFO1          400.1320007 MHz

======== CHANNEL f2 ========
CPDPRG[2              garp4
NUC2                    11B
PCPD2                 90.00 usec
PLW2           52.96599960 W
PLW12           0.64477998 W
SFO2          128.3776050 MHz

F2 - Processing parameters
SI                    32768
SF            400.1300072 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

[NMe3H][2,3,4,5,6-5I-CHB11H6]
128M,11B NMR,20mg,Acetone-d6*,296K



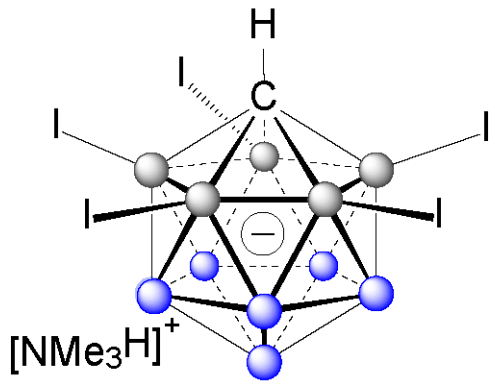

```
Current Data Parameters
NAME      400M-[NMe3H][2,3,4,5,6-
EXPNO                4
PROCNO               1

F2 - Acquisition Parameters
Date_           20221030
Time_             15.13
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG             zg
TD                65536
SOLVENT         Acetone
NS                  128
DS                    4
SWH           25510.203 Hz
FIDRES         0.389255 Hz
AQ            1.2845056 sec
RG               193.34
DW               19.600 usec
DE                6.50 usec
TE               296.0 K
D1            1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                11B
P1                 9.93 usec
PLW1        52.96599960 W
SFO1       128.3776052 MHz

F2 - Processing parameters
SI                32768
SF         128.3776050 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

[NMe3H][2,3,4,5,6-5I-CHB11H6]
128M,11B{1H} NMR,20mg,Acetone-d6*,296K



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```
Current Data Parameters
NAME      400M-[NMe3H][2,3,4,5,6-
EXPNO                 3
PROCNO                1

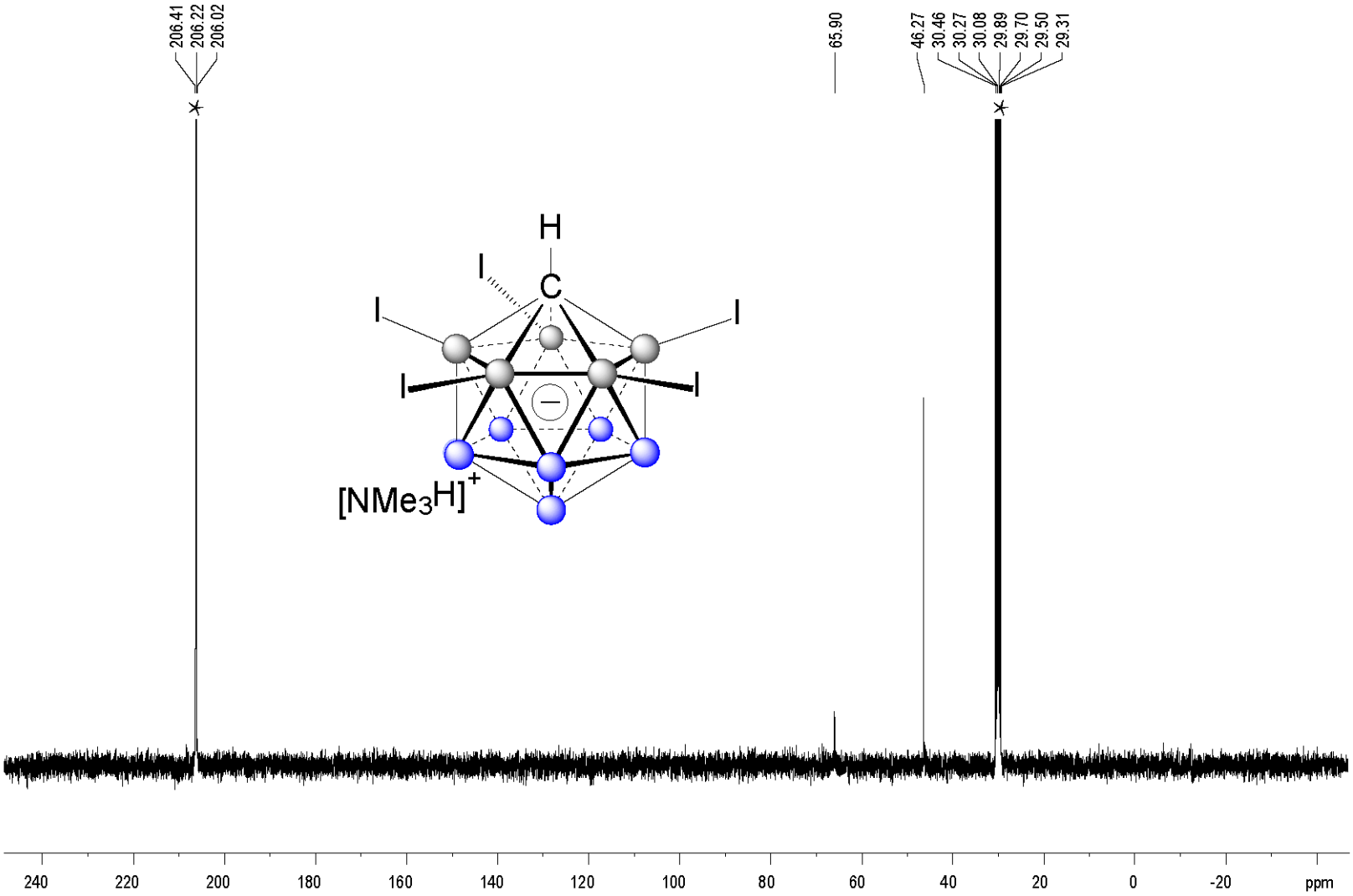
F2 - Acquisition Parameters
Date_          20221030
Time              15.07
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  128
DS                    4
SWH           25510.203 Hz
FIDRES         0.389255 Hz
AQ            1.2845056 sec
RG               193.34
DW               19.600 usec
DE                6.50 usec
TE               296.8 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 11B
P1                  9.93 usec
PLW1        52.96599960 W
SFO1       128.3776050 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PLW2        12.50000000 W
PLW12        0.43945000 W
PLW13        0.28125000 W
SFO2       400.1320007 MHz

F2 - Processing parameters
SI                32768
SF         128.3776050 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

[NMe3H][2,3,4,5,6-5I-CHB11H6]
101M,11C{1H} NMR,20mg,Acetone-d6*,296K




```
Current Data Parameters
NAME      400M-[NMe3H][2,3,4,5,6
EXPNO                          5
PROCNO                         1

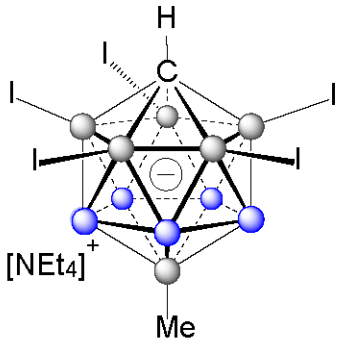
F2 - Acquisition Parameters
Date_                   20221030
Time                       15.37
INSTRUM                    spect
PROBHD    5 mm PABBO BB/
PULPROG                   zgpg30
TD                         65536
SOLVENT                  Acetone
NS                             512
DS                               4
SWH                    29761.904 Hz
FIDRES                 0.454131 Hz
AQ                     1.1010048 sec
RG                         193.34
DW                         16.800 usec
DE                          6.50 usec
TE                         296.6 K
D1                    1.50000000 sec
D11                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         13C
P1                         10.00 usec
PLW1                53.00000000 W
SFO1               100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                         1H
PCPD2                      80.00 usec
PLW2                12.50000000 W
PLW12                0.43945000 W
PLW13                0.28125000 W
SFO2               400.1316005 MHz

F2 - Processing parameters
SI                           32768
SF                 100.6126784 MHz
WDW                             EM
SSB                              0
LB                            1.00 Hz
GB                               0
PC                            1.40
```

[NEt4][2,3,4,5,6-5I-12-Me-CB11H6]
500MHz,1H{11B} NMR,20mg,0.5 mL acetonitrile-d3*,296k



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```
Current Data Parameters
NAME      1H{11B} NMR,500M,Acetonitrile
EXPNO                 4
PROCNO                1

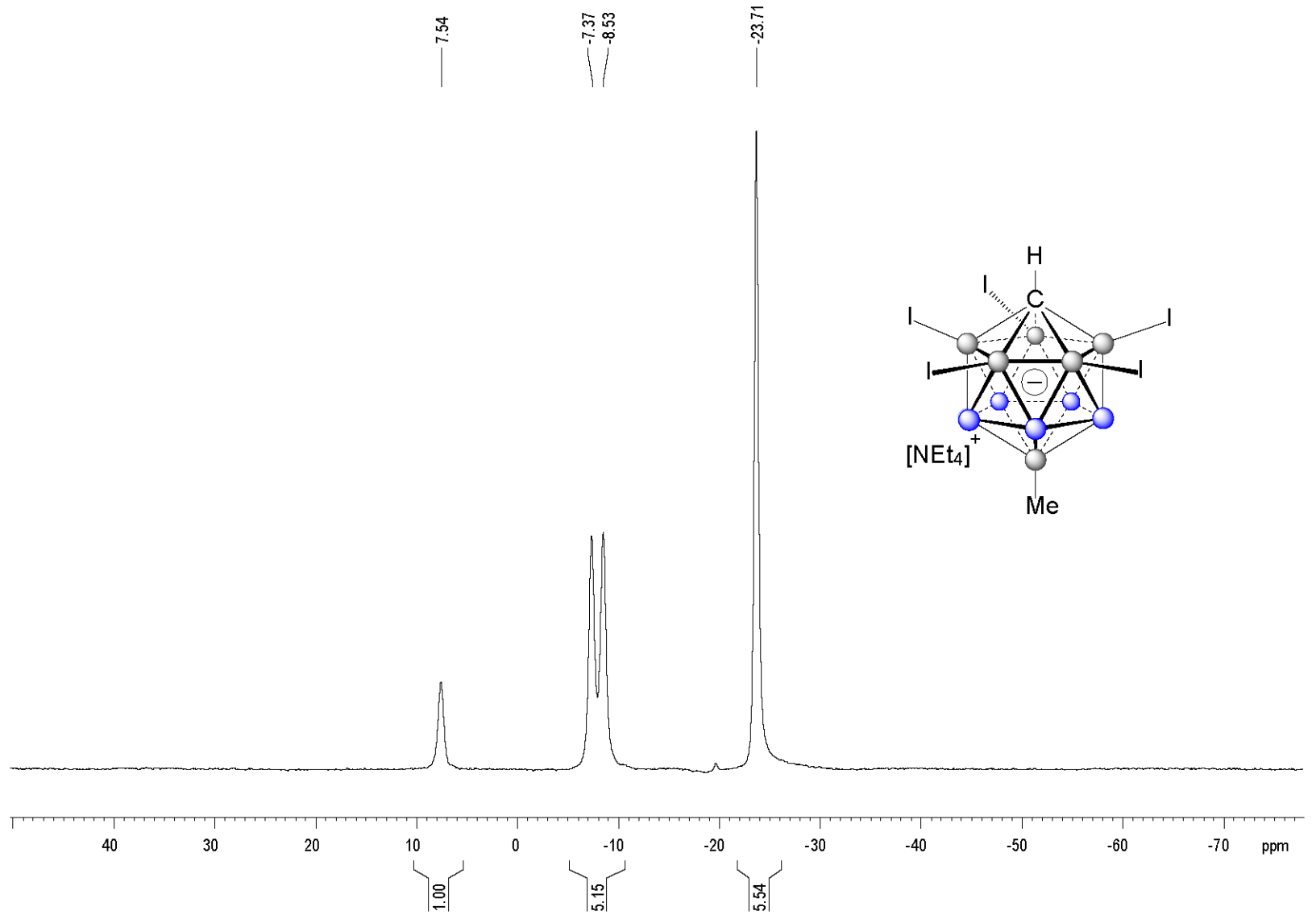
F2 - Acquisition Parameters
Date_           20220618
Time               0.17
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgig30
TD                65536
SOLVENT           CD3CN
NS                   16
DS                    0
SWH           12500.000 Hz
FIDRES        0.190735 Hz
AQ            2.6214399 sec
RG                  203
DW               40.000 usec
DE                6.50 usec
TE               296.9 K
D1            5.00000000 sec
D11           0.03000000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                11.70 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           500.1300153 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.00
```

[NEt4][2,3,4,5,6-5I-12-Me-CB11H6]
128MHz,11B NMR,6mg,0.5 mL acetone-d6*,296K



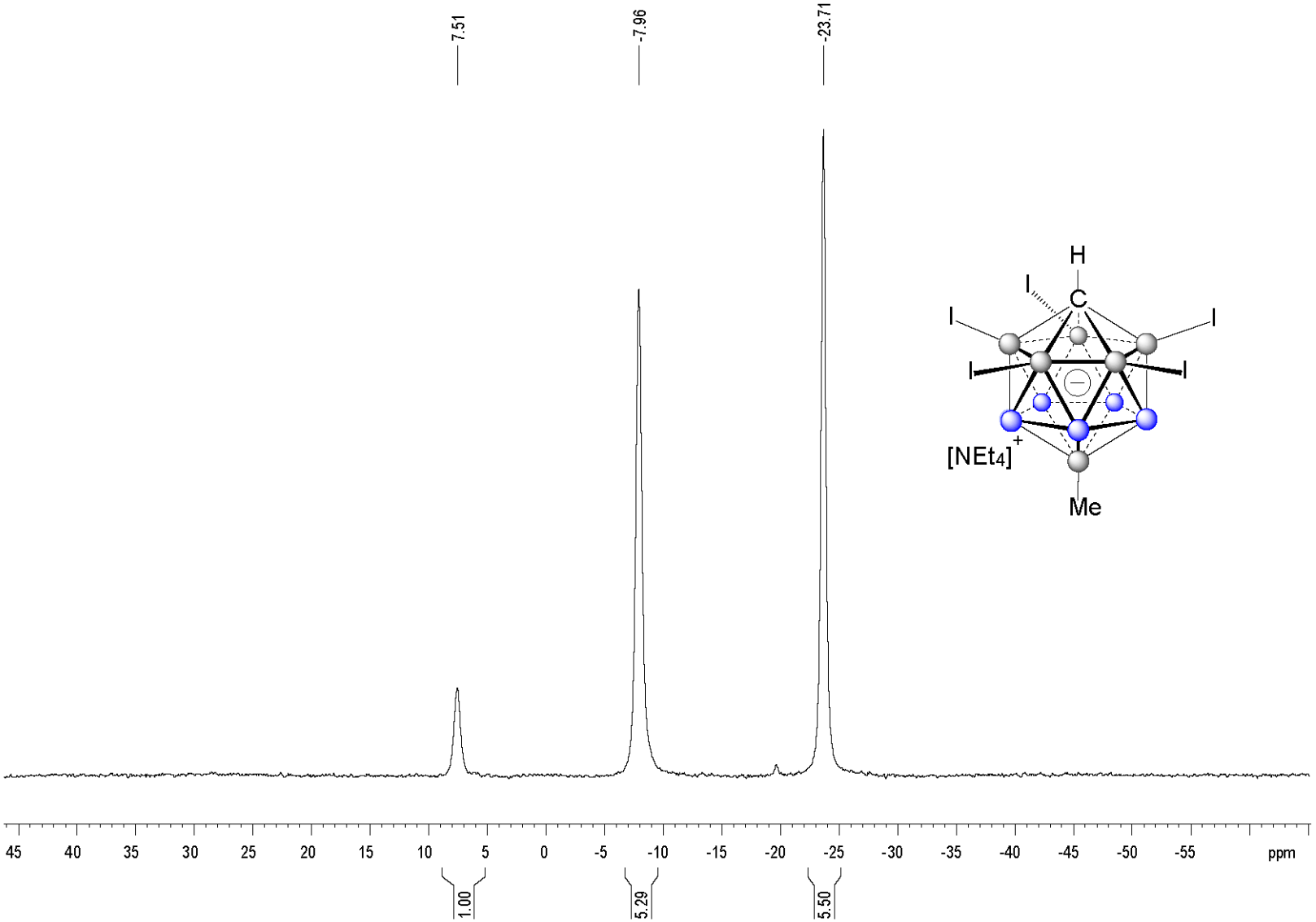

```
Current Data Parameters
NAME      11B NMR,400M,Acetone
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20221008
Time              8.26
INSTRUM          spect
PROBHD   5 mm PABBO BB/
PULPROG             zg
TD                65536
SOLVENT         Acetone
NS                  128
DS                    4
SWH           25510.203 Hz
FIDRES         0.389255 Hz
AQ            1.2845056 sec
RG               193.34
DW               19.600 usec
DE                6.50 usec
TE               296.0 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 11B
P1                  9.93 usec
PLW1         52.96599960 W
SFO1        128.3776052 MHz

F2 - Processing parameters
SI                32768
SF          128.3776050 MHz
WDW                  EM
SSB                   0
LB                10.00 Hz
GB                    0
PC                 1.40
```

[NEt4][2,3,4,5,6-5I-12-Me-CB11H6]
128MHz,11B{1H} NMR,6mg,0.5 mL acetone-d6*,296K




```
Current Data Parameters
NAME      11B{1H} NMR,400M,Aceton
EXPNO                1
PROCNO               1

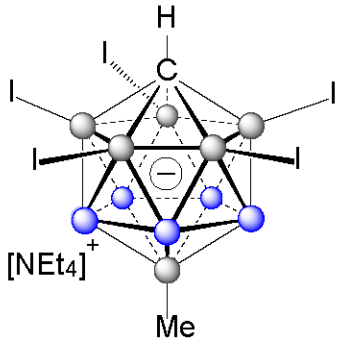
F2 - Acquisition Parameters
Date_          20221008
Time               8.21
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  128
DS                    4
SWH           25510.203 Hz
FIDRES         0.389255 Hz
AQ            1.2845056 sec
RG               193.34
DW               19.600 usec
DE                6.50 usec
TE               296.9 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                11B
P1                 9.93 usec
PLW1        52.96599960 W
SFO1       128.3776050 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2            80.00 usec
PLW2        12.50000000 W
PLW12        0.43945000 W
PLW13        0.28125000 W
SFO2       400.1320007 MHz

F2 - Processing parameters
SI                 32768
SF         128.3776050 MHz
WDW                EM
SSB                 0
LB                10.00 Hz
GB                  0
PC                1.40
```

[NEt4][2,3,4,5,6-5I-12-Me-CB11H6]
101MHz,13C{1H} NMR,10mg,0.5 mL acetone-d6*,296K




```
Current Data Parameters
NAME      13C{1H} NMR400M,Acetone
EXPNO                1
PROCNO               1

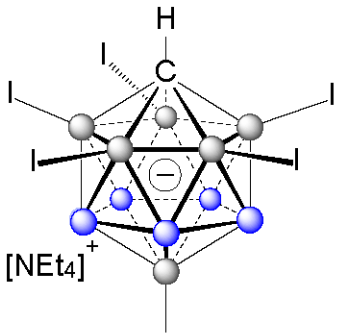
F2 - Acquisition Parameters
Date_          20221010
Time             18.58
INSTRUM          spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT        Acetone
NS                1024
DS                   4
SWH          29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG               193.34
DW               16.800 usec
DE                6.50 usec
TE               297.6 K
D1           1.50000000 sec
D11          0.03000000 sec
TD0                  1

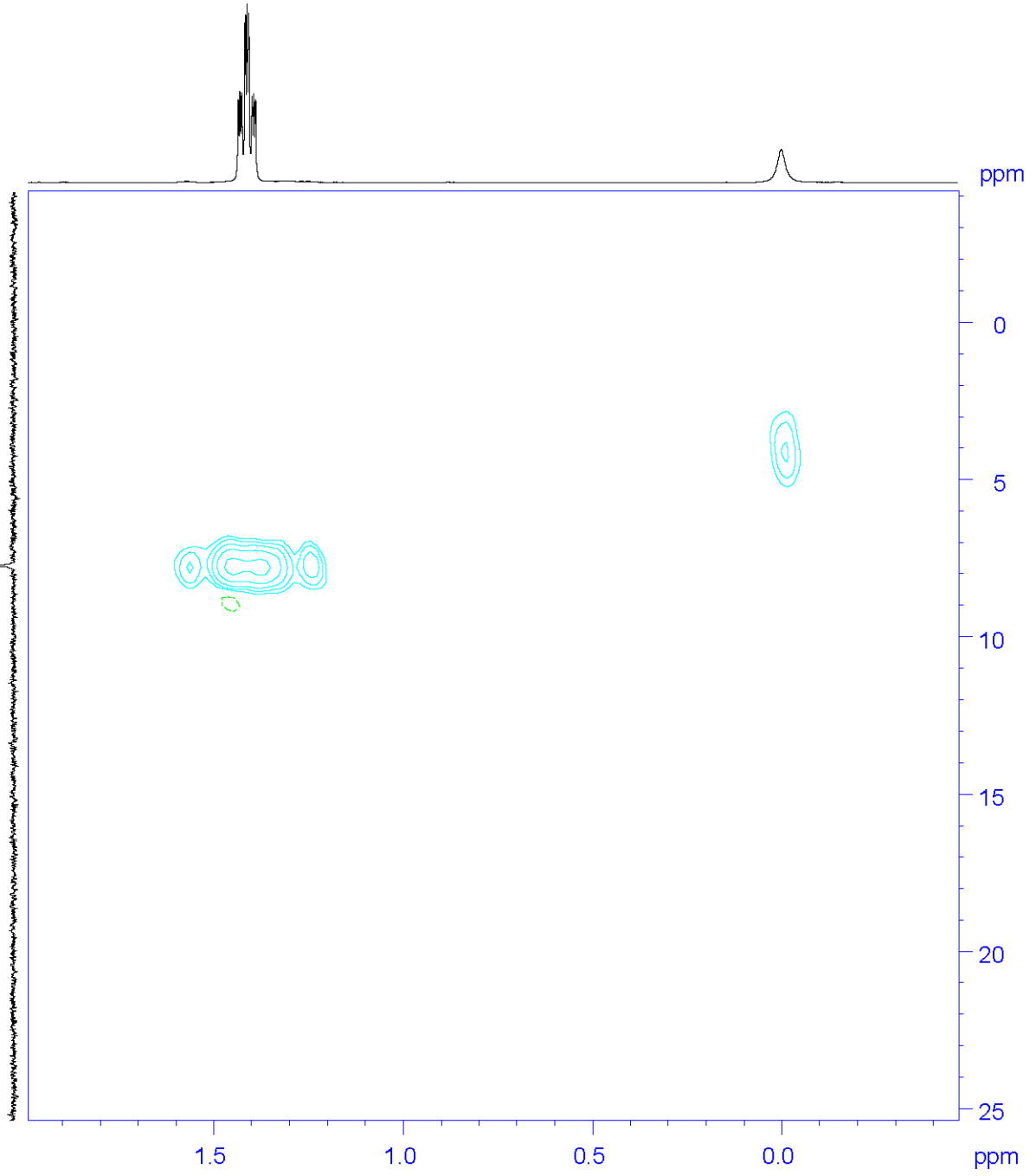
======== CHANNEL f1 ========
NUC1                13C
P1                10.00 usec
PLW1        53.00000000 W
SFO1       100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2            80.00 usec
PLW2        12.50000000 W
PLW12        0.43945000 W
PLW13        0.28125000 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6126835 MHz
WDW                EM
SSB                 0
LB                10.00 Hz
GB                  0
PC                1.40
```

[NEt4][2,3,4,5,6-5I-12-Me-CHB11H5]
400MHz,101MHz,HSQC NMR,20mg,0.6mL acetone-d6,296K






```
Current Data Parameters
NAME                  Me
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20221015
Time_              17.40
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG     hsqcetgpsi2
TD                 1024
SOLVENT         Acetone
NS                     2
DS                    16
SWH             6009.615 Hz
FIDRES          5.868765 Hz
AQ              0.0851968 sec
RG                 193.34
DW                 83.200 usec
DE                   6.50 usec
TE                 295.7 K
CNST2          145.0000000
D0              0.00000300 sec
D1              1.50000000 sec
D4              0.00172414 sec
D11             0.03000000 sec
D16             0.00020000 sec
D24             0.00086207 sec
IN0             0.00001990 sec
ZGOPTNS

======== CHANNEL f1 ========
NUC1                   1H
P1                  15.00 usec
P2                  30.00 usec
P28               1000.00 usec
PLW1           12.50000000 W
SFO1          400.1328009 MHz

======== CHANNEL f2 ========
CPDPRG[2              garp
NUC2                  13C
P3                  10.00 usec
P4                  20.00 usec
PCPD2               70.00 usec
PLW2           53.00000000 W
PLW12           1.08159995 W
SFO2          100.6238364 MHz

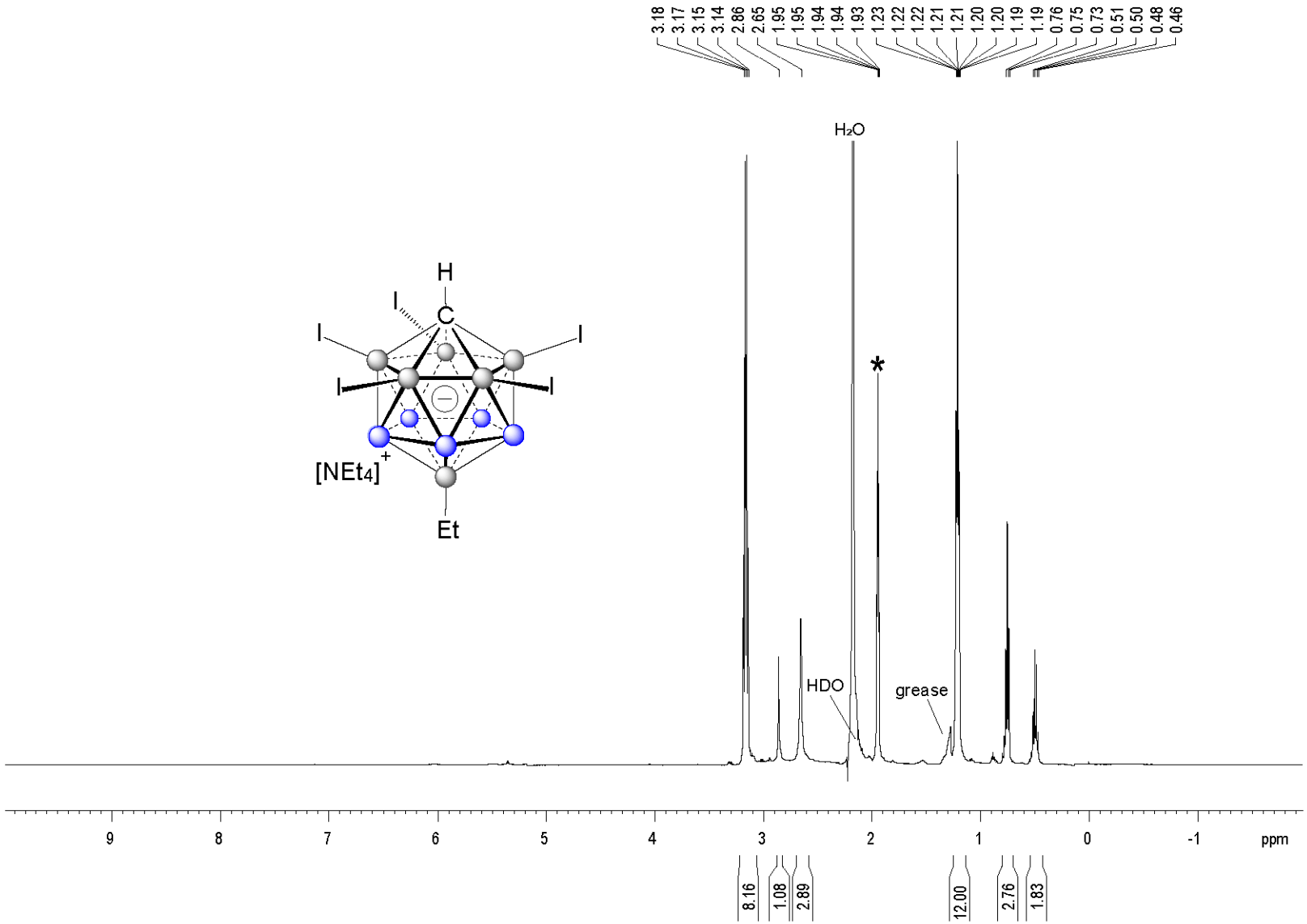
====== GRADIENT CHANNEL ====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPNAM[4]      SMSQ10.100
GPZ1                80.00 %
GPZ2                20.10 %
GPZ3                11.00 %
GPZ4                -5.00 %
P16               1000.00 usec
P19                600.00 usec

F1 - Acquisition parameters
TD                   256
SFO1          100.6238 MHz
FIDRES       196.524048 Hz
SW              249.991 ppm
FnMODE    Echo-Antiecho

F2 - Processing parameters
SI                  1024
SF           400.1300085 MHz
WDW                 QSINE
SSB                     2
LB                      0 Hz
GB                      0
PC                   1.40

F1 - Processing parameters
SI                  1024
MC2        echo-antiecho
SF           100.6126655 MHz
WDW                 QSINE
SSB                     2
LB                      0 Hz
GB                      0
```

[NEt4][2,3,4,5,6-5I-12-Et-CB11H6]
500MHz,1H{11B} NMR,20mg,0.5 mL acetonitrile-d3*,296K




```
Current Data Parameters
NAME      1H{11B} NMR,500M,Aceto
EXPNO                          4
PROCNO                         1

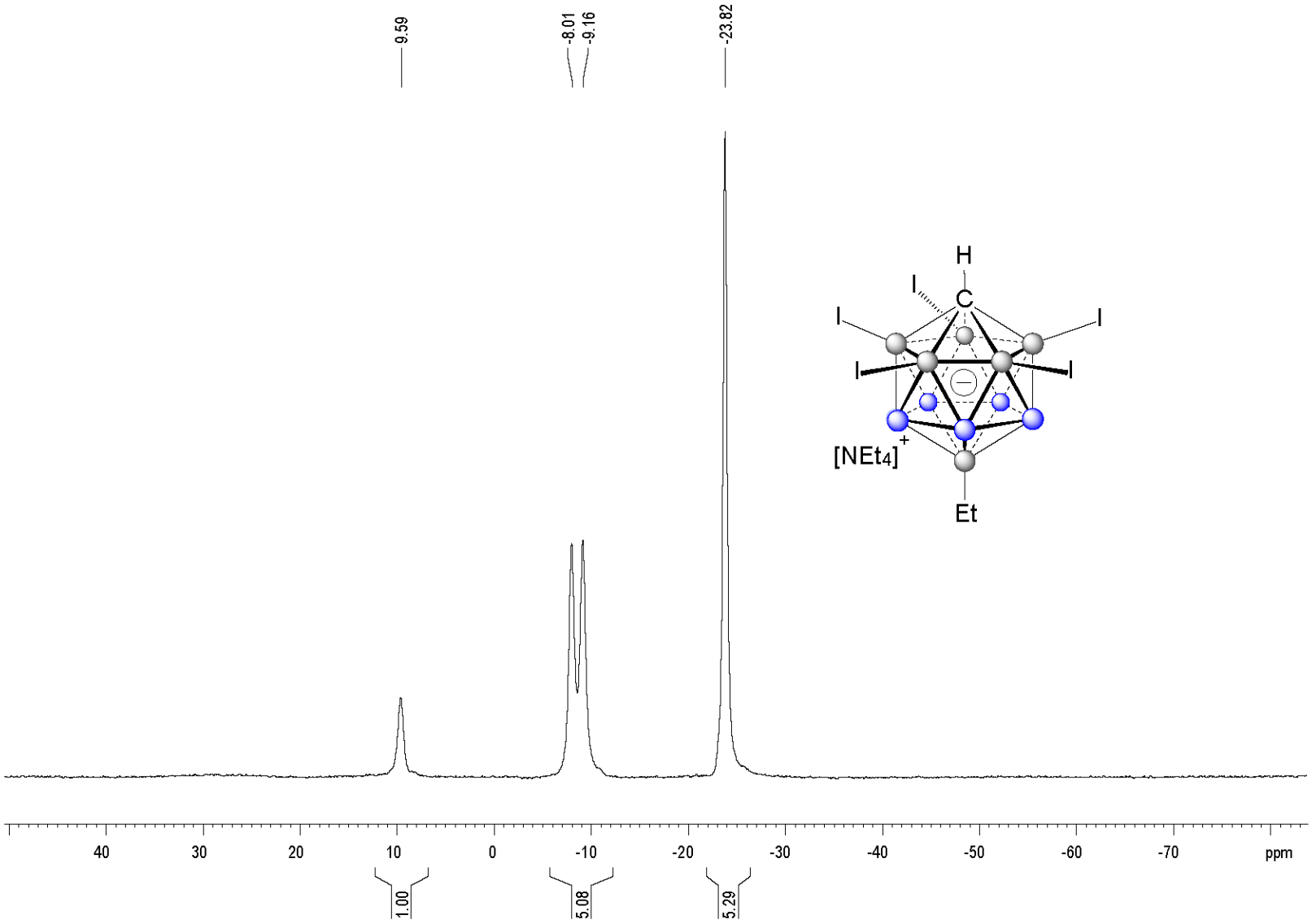
F2 - Acquisition Parameters
Date_                   20220618
Time                        0.31
INSTRUM                    spect
PROBHD    5 mm PABBO BB-
PULPROG                   zgig30
TD                         65536
SOLVENT                    CD3CN
NS                            16
DS                             0
SWH                    12500.000 Hz
FIDRES                 0.190735 Hz
AQ                     2.6214399 sec
RG                           114
DW                        40.000 usec
DE                         6.50 usec
TE                        297.1 K
D1                   5.00000000 sec
D11                  0.03000000 sec

======== CHANNEL f1 ========
NUC1                          1H
P1                        11.70 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                  500.1300154 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.00
```

[NEt4][2,3,4,5,6-5I-12-Et-CHB11H5]
128MHz,11B NMR,20mg,0.6mL acetone-d6*,296K



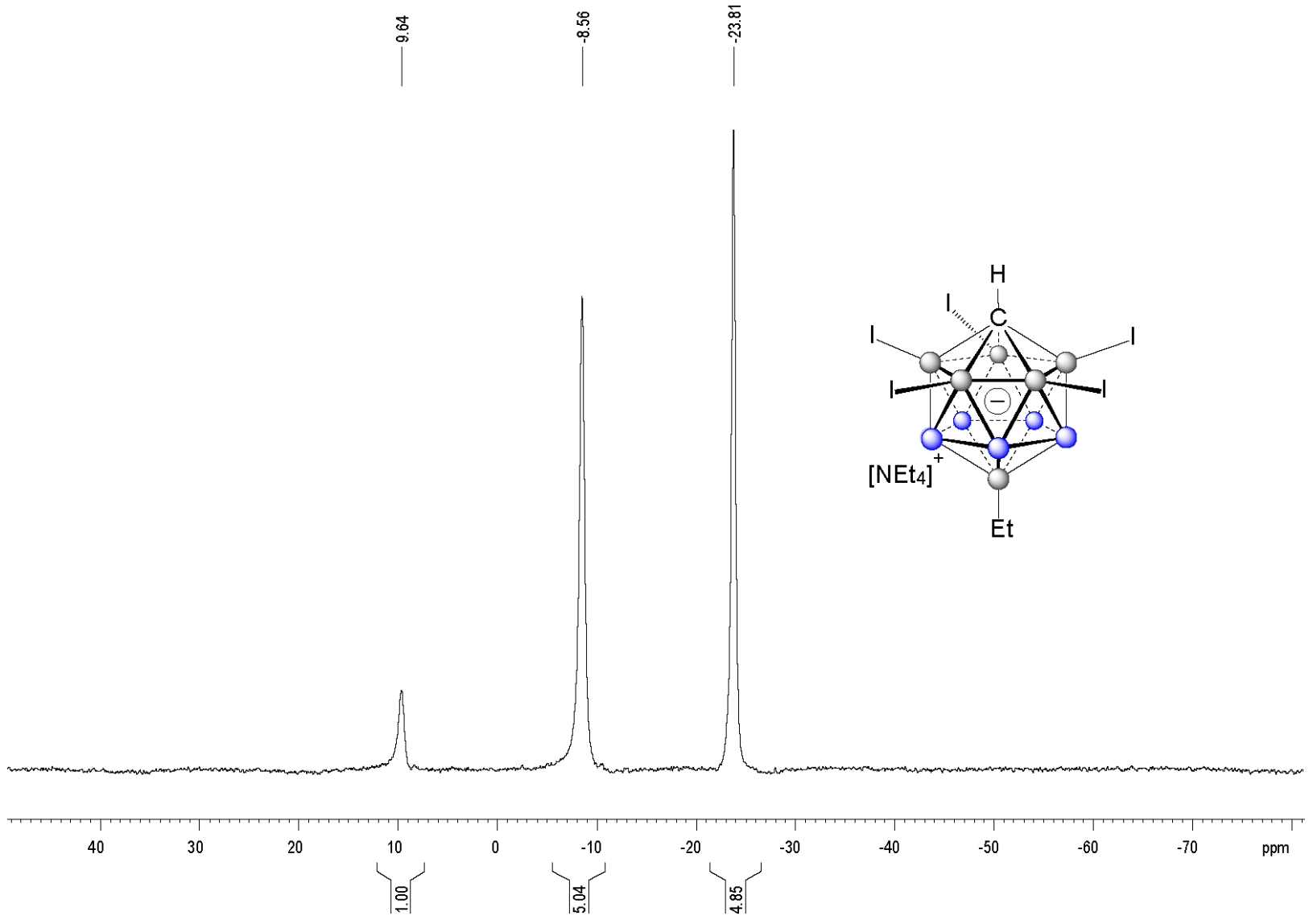

```
Current Data Parameters
NAME      11B NMR,400M,Aceton
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_           20221027
Time              13.24
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG             zg
TD                65536
SOLVENT         Acetone
NS                  128
DS                    4
SWH           25510.203 Hz
FIDRES         0.389255 Hz
AQ            1.2845056 sec
RG               193.34
DW               19.600 usec
DE                6.50 usec
TE               295.6 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 11B
P1                 9.93 usec
PLW1        52.96599960 W
SFO1       128.3776052 MHz

F2 - Processing parameters
SI                32768
SF         128.3776050 MHz
WDW                  EM
SSB                   0
LB                10.00 Hz
GB                    0
PC                 1.40
```

[NEt4][2,3,4,5,6-5I-12-Et-CHB11H5]
128MHz,11B{1H} NMR,20mg,0.6mL acetone-d6*,296K



47

```
Current Data Parameters
NAME      11B{1H} NMR,400M,Aceton
EXPNO                1
PROCNO               1

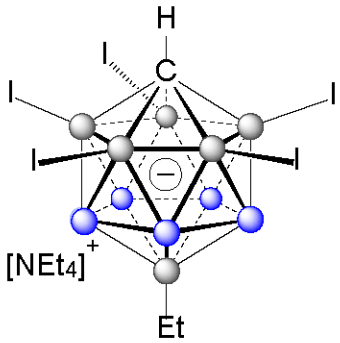
F2 - Acquisition Parameters
Date_          20221027
Time              13.18
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  128
DS                    4
SWH           25510.203 Hz
FIDRES         0.389255 Hz
AQ            1.2845056 sec
RG               193.34
DW               19.600 usec
DE                6.50 usec
TE               296.3 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                11B
P1                 9.93 usec
PLW1        52.96599960 W
SFO1       128.3776050 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2            80.00 usec
PLW2        12.50000000 W
PLW12        0.43945000 W
PLW13        0.28125000 W
SFO2       400.1320007 MHz

F2 - Processing parameters
SI                 32768
SF         128.3776050 MHz
WDW                  EM
SSB                   0
LB                10.00 Hz
GB                    0
PC                 1.40
```

[NEt4][2,3,4,5,6-5I-12-Et-CB11H6]
101M,13C{1H} NMR,20mg,0.5 mL acetone-d6*,296K



48

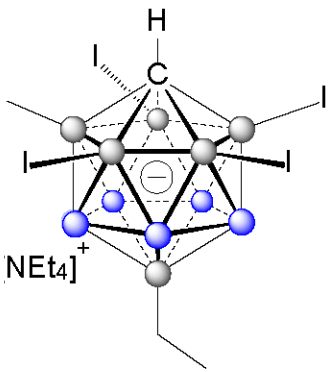
```
Current Data Parameters
NAME      13C{1H} NMR,400M,Aceton
EXPNO                5
PROCNO               1

F2 - Acquisition Parameters
Date_          20221010
Time               6.18
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT        Acetone
NS                1024
DS                   4
SWH          29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.1010048 sec
RG                193.34
DW                16.800 usec
DE                 6.50 usec
TE                298.7 K
D1           1.50000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1                10.00 usec
PLW1        53.00000000 W
SFO1       100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2              80.00 usec
PLW2        12.50000000 W
PLW12        0.43945000 W
PLW13        0.28125000 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF         100.6126837 MHz
WDW                   EM
SSB                    0
LB                 10.00 Hz
GB                     0
PC                  1.40
```



[NEt4][2,3,4,5,6-5I-12-Et-CHB11H5]
400MHz,101MHz,HSQC NMR,20mg,0.6mL CD3CN,296K


```
Current Data Parameters
NAME                  Et
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20221101
Time_              21.20
INSTRUM            spect
PROBHD    5 mm PABBO BB/
PULPROG     hsqcetgpsi2
TD                 1024
SOLVENT           CD3CN
NS                    2
DS                   16
SWH            6009.615 Hz
FIDRES         5.868765 Hz
AQ            0.0851968 sec
RG                193.34
DW                83.200 usec
DE                 6.50 usec
TE               296.5 K
CNST2        145.0000000
D0            0.00000300 sec
D1            1.50000000 sec
D4            0.00172414 sec
D11           0.03000000 sec
D16           0.00020000 sec
D24           0.00086207 sec
IN0           0.00001990 sec
ZGOPTNS

======== CHANNEL f1 ========
NUC1                  1H
P1                 15.00 usec
P2                 30.00 usec
P28              1000.00 usec
PLW1         12.50000000 W
SFO1         400.1328009 MHz

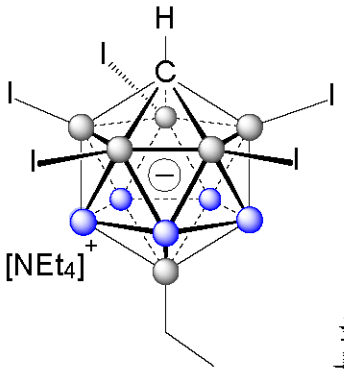
======== CHANNEL f2 ========
CPDPRG[2              garp
NUC2                  13C
P3                 10.00 usec
P4                 20.00 usec
PCPD2              70.00 usec
PLW2         53.00000000 W
PLW12         1.08159995 W
SFO2         100.6238364 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPNAM[4]      SMSQ10.100
GPZ1               80.00 %
GPZ2               20.10 %
GPZ3               11.00 %
GPZ4               -5.00 %
P16              1000.00 usec
P19               600.00 usec

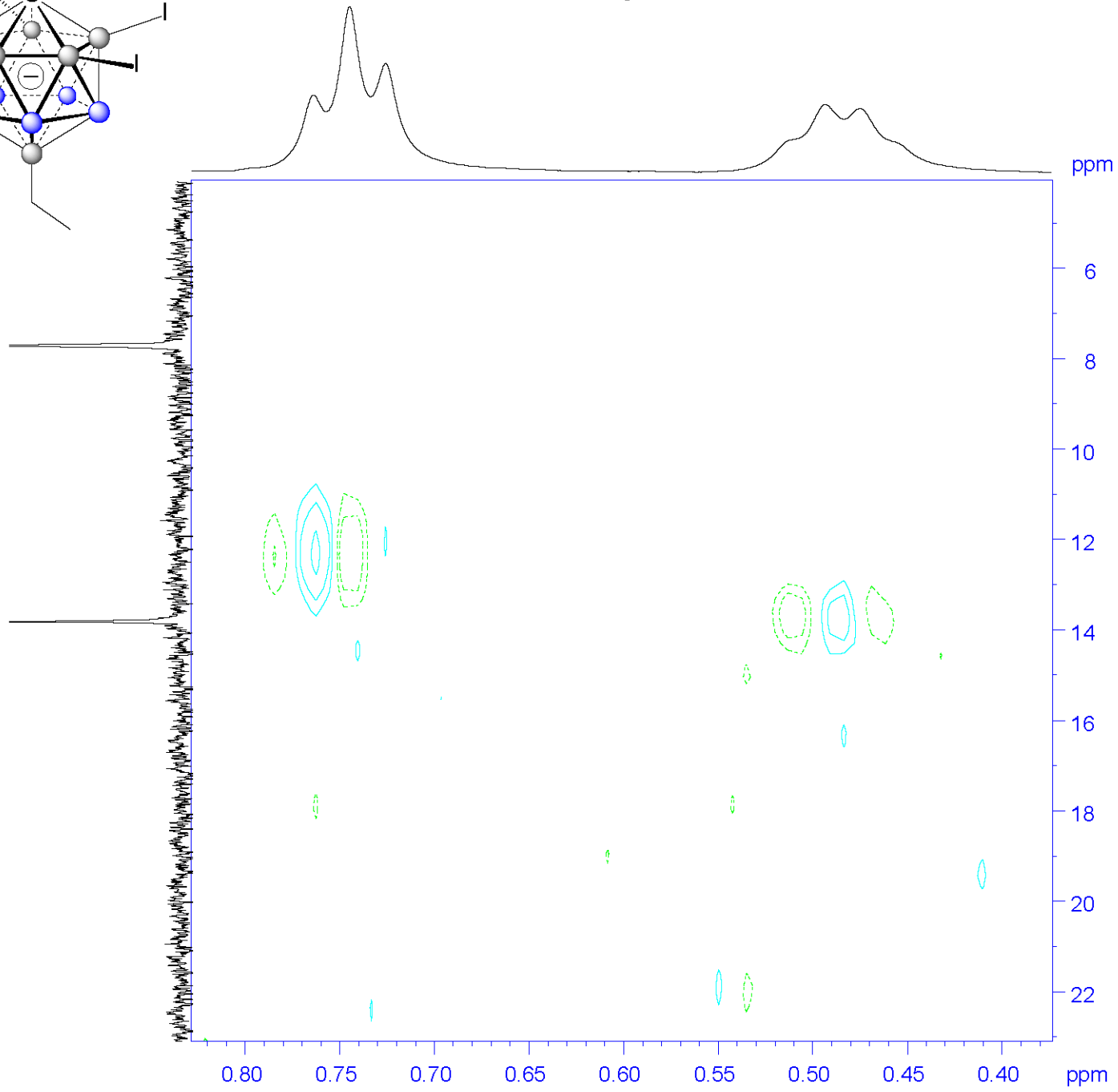
F1 - Acquisition parameters
TD                   256
SFO1          100.6238 MHz
FIDRES        196.524048 Hz
SW               249.991 ppm
FnMODE    Echo-Antiecho

F2 - Processing parameters
SI                  1024
SF           400.1300107 MHz
WDW                 QSINE
SSB                    2
LB                     0 Hz
GB                     0
PC                  1.40

F1 - Processing parameters
SI                  1024
MC2         echo-antiecho
SF           100.6126718 MHz
WDW                 QSINE
SSB                    2
LB                     0 Hz
GB                     0
```



[NEt4][2,3,4,5,6-5I-12-Et-CHB11H5]
400MHz,101MHz,HMBC NMR,20mg,0.6mL CD3CN,296K



50

```
Current Data Parameters
NAME                  Et
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20221101
Time_              21.35
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG    hmbcetgpl3nd
TD                 2048
SOLVENT           CD3CN
NS                    4
DS                   16
SWH            6009.615 Hz
FIDRES         2.934382 Hz
AQ            0.1703936 sec
RG               193.34
DW               83.200 usec
DE                6.50 usec
TE                296.2 K
CNST6       120.0000000
CNST7       170.0000000
CNST13        8.0000000
CNST30        0.5981128
D0          0.00000300 sec
D1          2.00000000 sec
D6          0.06250000 sec
D16         0.00020000 sec
IN0         0.00001990 sec

======== CHANNEL f1 ========
NUC1                1H
P1                15.00 usec
P2                30.00 usec
PLW1        12.50000000 W
SFO1       400.1328009 MHz

======== CHANNEL f2 ========
NUC2               13C
P3               10.00 usec
P24            2000.00 usec
PLW2        53.00000000 W
SFO2       100.6238364 MHz
SPNAM[7]    Crp60comp.4
SPOAL7           0.500
SPOFFS7              0 Hz
SPW7        8.09780025 W

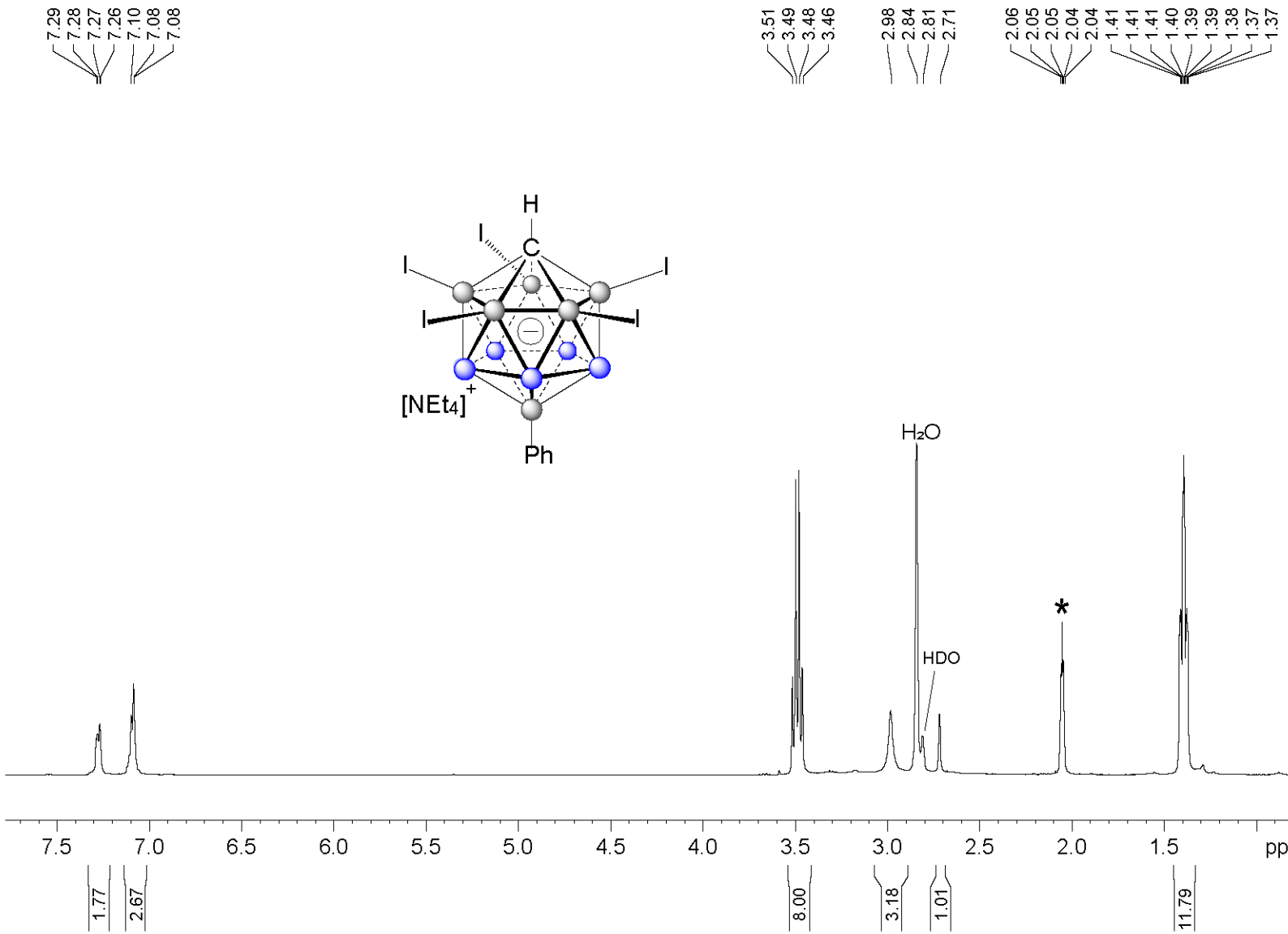
====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPNAM[4]      SMSQ10.100
GPNAM[5]      SMSQ10.100
GPNAM[6]      SMSQ10.100
GPZ1             80.00 %
GPZ3             14.00 %
GPZ4             -8.00 %
GPZ5             -4.00 %
GPZ6             -2.00 %
P16            1000.00 usec

F1 - Acquisition parameters
TD                  256
SFO1          100.6238 MHz
FIDRES       196.528931 Hz
SW           249.997 ppm
FnMODE    Echo-Antiecho

F2 - Processing parameters
SI                 2048
SF          400.1300088 MHz
WDW                SINE
SSB                   4
LB                    0 Hz
GB                    0
PC                 1.40

F1 - Processing parameters
SI                 1024
MC2       echo-antiecho
SF          100.6126593 MHz
WDW               QSINE
SSB                   2
LB                    0 Hz
GB                    0
```

[NEt4][2,3,4,5,6-5I-12-Ph-CB11H6]
400M,1H{11B} NMR,4mg,0.5 mL acetone-d6*,296K




```
Current Data Parameters
NAME      1H{11B} NMR,400M,Acetor
EXPNO                2
PROCNO               1

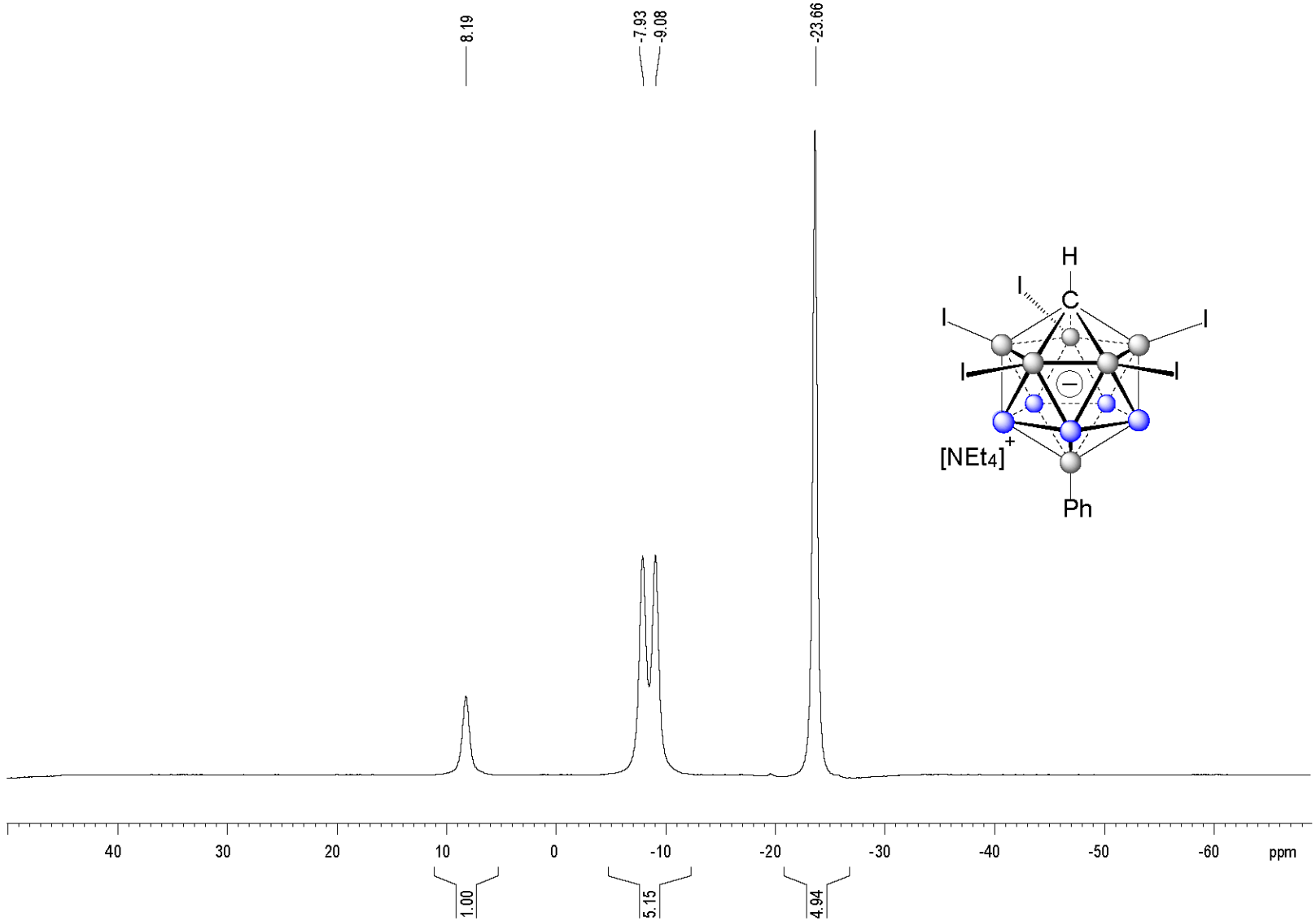
F2 - Acquisition Parameters
Date_          20221010
Time               2.24
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgig30
TD               16384
SOLVENT        Acetone
NS                  16
DS                   4
SWH           8012.820 Hz
FIDRES        0.489064 Hz
AQ            1.0223616 sec
RG               193.34
DW                62.400 usec
DE                 6.50 usec
TE                296.2 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                 1H
P1                15.00 usec
PLW1         12.50000000 W
SFO1        400.1320007 MHz

======== CHANNEL f2 ========
CPDPRG[2          garp4
NUC2                11B
PCPD2             90.00 usec
PLW2         52.96599960 W
PLW12         0.64477998 W
SFO2        128.3776050 MHz

F2 - Processing parameters
SI                32768
SF          400.1300073 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

[NEt4][2,3,4,5,6-5I-12-Ph-CHB11H5]
128MHz,11BNMR,19 mg,0.6mL Acetone-d6*,296K



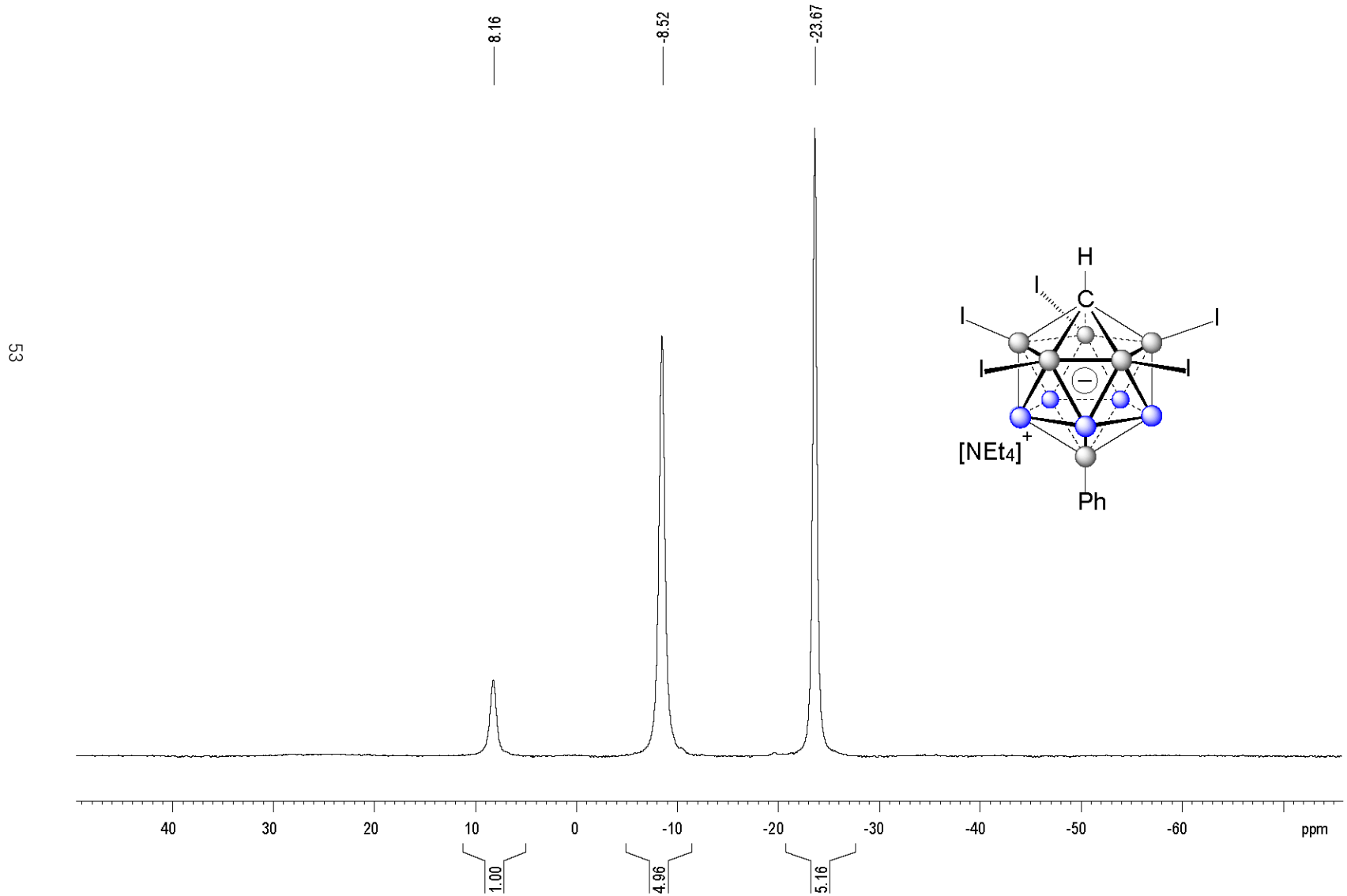

```
Current Data Parameters
NAME      11B NMR,400M,Acetone
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20221026
Time                 21.32
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG                 zg
TD                   65536
SOLVENT            Acetone
NS                       128
DS                         4
SWH                25510.203 Hz
FIDRES              0.389255 Hz
AQ                 1.2845056 sec
RG                    193.34
DW                    19.600 usec
DE                     6.50 usec
TE                    295.8 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     11B
P1                      9.93 usec
PLW1           52.96599960 W
SFO1           128.3776052 MHz

F2 - Processing parameters
SI                       32768
SF             128.3776050 MHz
WDW                        EM
SSB                         0
LB                      10.00 Hz
GB                          0
PC                      1.40
```

[NEt4][2,3,4,5,6-5I-12-Ph-CHB11H5]
128MHz,11B{1H} NMR,19 mg,0.6mL Acetone-d6*,296k



53

```
Current Data Parameters
NAME      11B{1H} NMR,400M,Acetone
EXPNO                 1
PROCNO                1

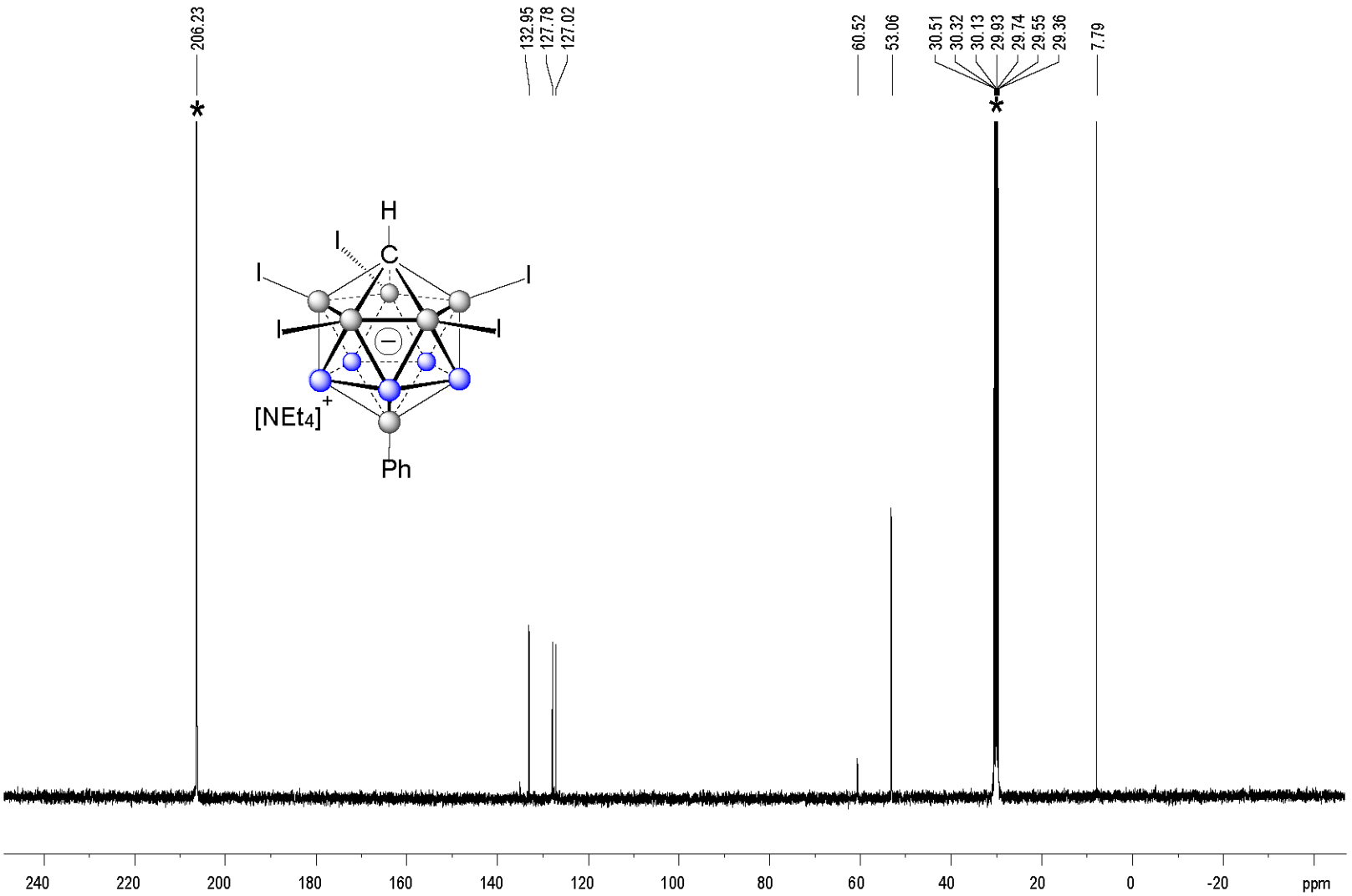
F2 - Acquisition Parameters
Date_           20221026
Time              21.26
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  128
DS                    4
SWH           25510.203 Hz
FIDRES         0.389255 Hz
AQ            1.2845056 sec
RG               193.34
DW               19.600 usec
DE                6.50 usec
TE               296.6 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 11B
P1                  9.93 usec
PLW1        52.96599960 W
SFO1       128.3776050 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PLW2        12.50000000 W
PLW12        0.43945000 W
PLW13        0.28125000 W
SFO2       400.1320007 MHz

F2 - Processing parameters
SI                32768
SF         128.3776050 MHz
WDW                  EM
SSB                   0
LB                10.00 Hz
GB                    0
PC                 1.40
```

[NEt4][2,3,4,5,6-5I-12-Ph-CHB11H5]
101MHz,13C{1H} NMR,60mg,0.6mL Acetone-d6*,296K



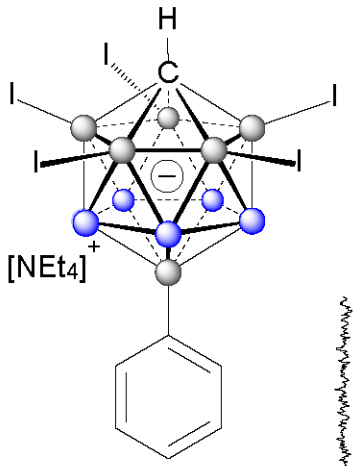

```
Current Data Parameters
NAME      13C{1H} NMR,400M,Acetor
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_           20221025
Time_              7.58
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  512
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG               193.34
DW                16.800 usec
DE                 6.50 usec
TE                295.7 K
D1           1.50000000 sec
D11          0.03000000 sec
TD0                  1

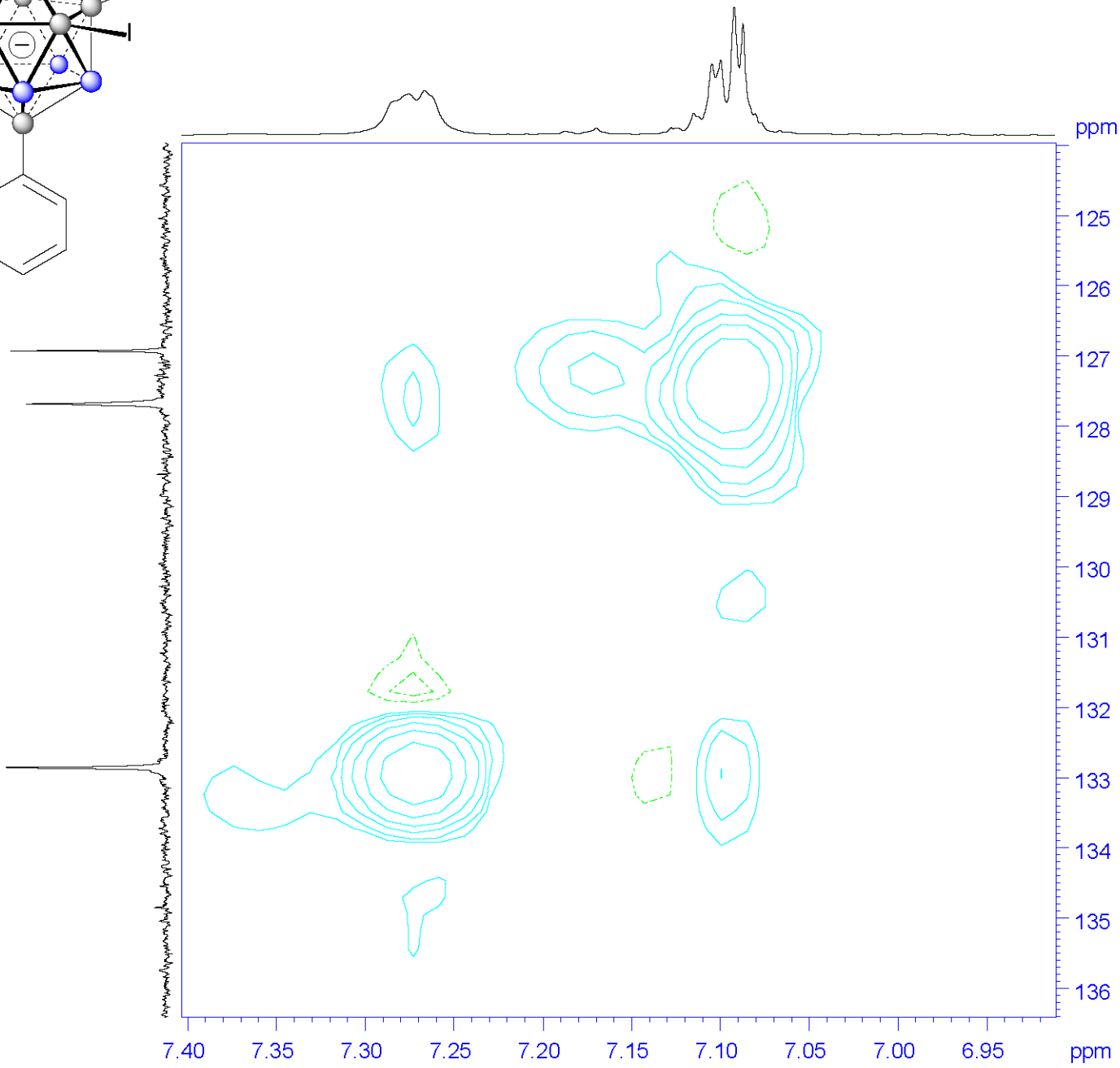
======== CHANNEL f1 ========
NUC1                13C
P1                10.00 usec
PLW1        53.00000000 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2             80.00 usec
PLW2        12.50000000 W
PLW12        0.43945000 W
PLW13        0.28125000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6126784 MHz
WDW                EM
SSB                 0
LB                1.00 Hz
GB                  0
PC                1.40
```



[NEt4][2,3,4,5,6-5I-12-Ph-CHB11H5]
400MHz,101MHz,HSQC NMR,20mg,0.6mg acetone-d6,296K




```
Current Data Parameters
NAME      HSQC[NEt4][2,3,4,5,6-5I-12-Ph-CB11H6
EXPNO                3
PROCNO               1

F2 - Acquisition Parameters
Date_          20221027
Time_             13.55
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG     hsqcetgpsi2
TD                1024
SOLVENT        Acetone
NS                   2
DS                  16
SWH           6009.615 Hz
FIDRES        5.868765 Hz
AQ            0.0851968 sec
RG               193.34
DW                83.200 usec
DE                 6.50 usec
TE                296.0 K
CNST2       145.0000000
D0           0.00000300 sec
D1           1.50000000 sec
D4           0.00172414 sec
D11          0.03000000 sec
D16          0.00020000 sec
D24          0.00086207 sec
IN0          0.00001990 sec
ZGOPTNS

======== CHANNEL f1 ========
NUC1                 1H
P1                15.00 usec
P2                30.00 usec
P28             1000.00 usec
PLW1         12.50000000 W
SFO1        400.1328009 MHz

======== CHANNEL f2 ========
CPDPRG[2             garp
NUC2                13C
P3                10.00 usec
P4                20.00 usec
PCPD2             70.00 usec
PLW2         53.00000000 W
PLW12         1.08159995 W
SFO2         100.6238364 MHz

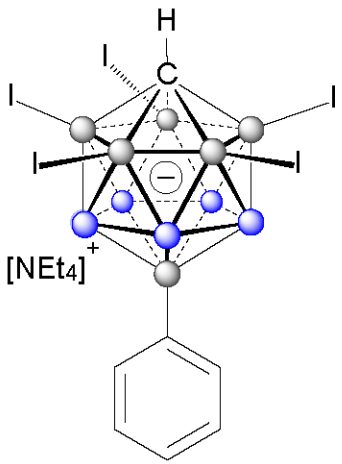
====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPNAM[4]      SMSQ10.100
GPZ1              80.00 %
GPZ2              20.10 %
GPZ3              11.00 %
GPZ4              -5.00 %
P16             1000.00 usec
P19              600.00 usec

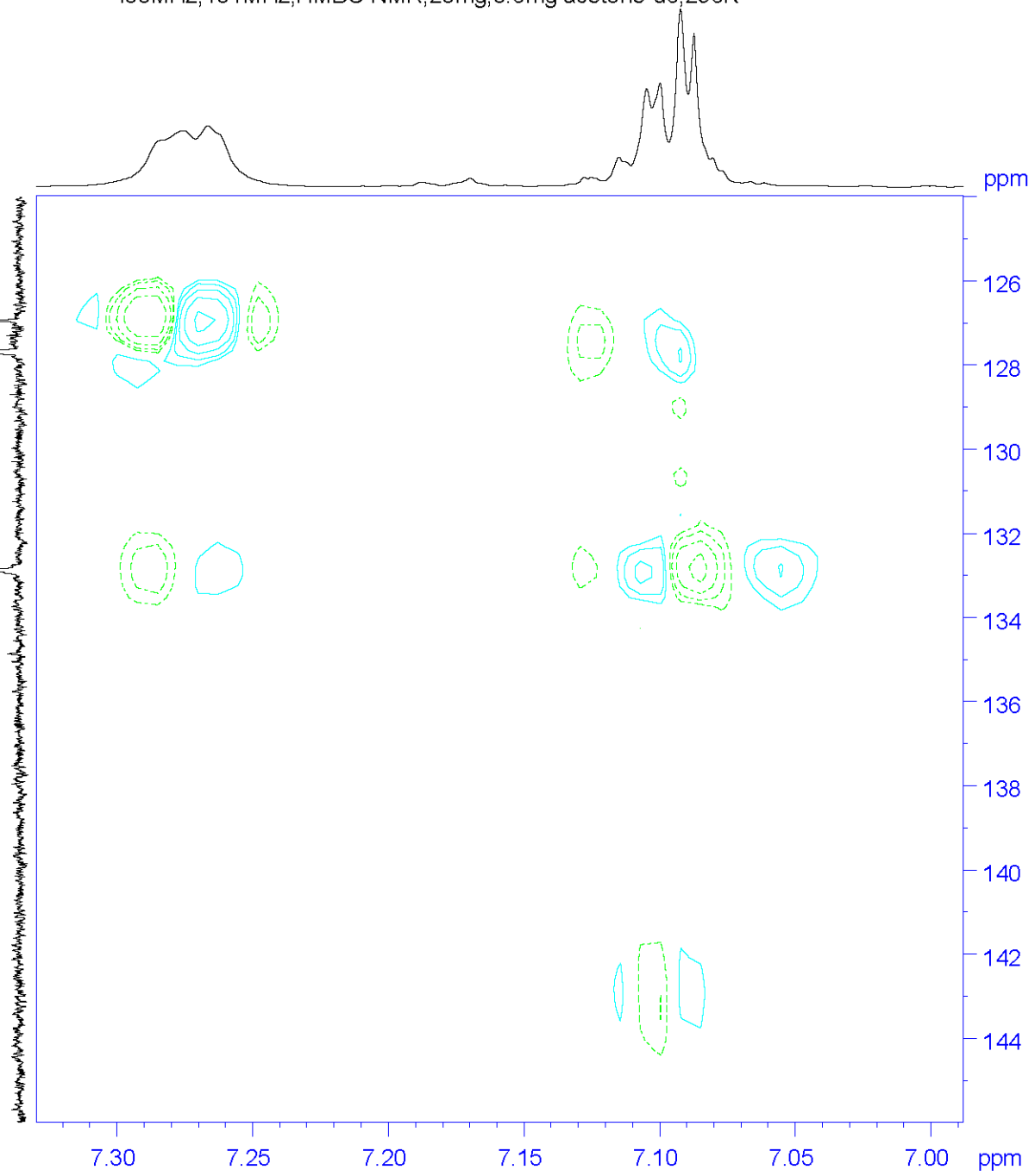
F1 - Acquisition parameters
TD                 256
SFO1          100.6238 MHz
FIDRES        196.524048 Hz
SW            249.991 ppm
FnMODE    Echo-Antiecho

F2 - Processing parameters
SI                1024
SF          400.1300079 MHz
WDW               QSINE
SSB                  2
LB                   0 Hz
GB                   0
PC                1.40

F1 - Processing parameters
SI                1024
MC2       echo-antiecho
SF          100.6127004 MHz
WDW               QSINE
SSB                  2
LB                   0 Hz
GB                   0
```

[NEt4][2,3,4,5,6-5I-12-Ph-CHB11H5]
400MHz,101MHz,HMBC NMR,20mg,0.6mg acetone-d6,296K





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```
Current Data Parameters
NAME      HSQC[NEt4][2,3,4,5,6-5I-12-Ph-CB11
EXPNO                4
PROCNO               1

F2 - Acquisition Parameters
Date_          20221027
Time_             14.11
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG    hmbcetgp13nd
TD                2048
SOLVENT        Acetone
NS                   4
DS                  16
SWH           6009.615 Hz
FIDRES        2.934382 Hz
AQ            0.1703936 sec
RG              193.34
DW               83.200 usec
DE                6.50 usec
TE              295.8 K
CNST6       120.0000000
CNST7       170.0000000
CNST13        8.0000000
CNST30        0.5981128
D0            0.00000300 sec
D1            2.00000000 sec
D6            0.06250000 sec
D16           0.00020000 sec
IN0           0.00001990 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                15.00 usec
P2                30.00 usec
PLW1         12.50000000 W
SFO1         400.1328009 MHz

======== CHANNEL f2 ========
NUC2                13C
P3               10.00 usec
P24            2000.00 usec
PLW2         53.00000000 W
SFO2         100.6238364 MHz
SPNAM[7]     Crp60comp.4
SPOAL7             0.500
SPOFFS7              0 Hz
SPW7          8.09780025 W

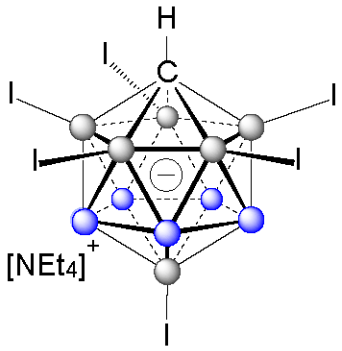
====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPNAM[4]      SMSQ10.100
GPNAM[5]      SMSQ10.100
GPNAM[6]      SMSQ10.100
GPZ1               80.00 %
GPZ3               14.00 %
GPZ4               -8.00 %
GPZ5               -4.00 %
GPZ6               -2.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                  256
SFO1          100.6238 MHz
FIDRES       196.528931 Hz
SW              249.997 ppm
FnMODE    Echo-Antiecho

F2 - Processing parameters
SI                 2048
SF           400.1300068 MHz
WDW                SINE
SSB                   4
LB                    0 Hz
GB                    0
PC                 1.40

F1 - Processing parameters
SI                 1024
MC2        echo-antiecho
SF           100.6126854 MHz
WDW               QSINE
SSB                   2
LB                    0 Hz
GB                    0
```

[NEt4][2,3,4,5,6-5I-12-I-CB11H6]
400MHz,1H{11B} NMR,20mg,0.5 mL acetone-d6*,296K




```
Current Data Parameters
NAME      1H(11B) NMR,400M,Aceto
EXPNO                          2
PROCNO                         1

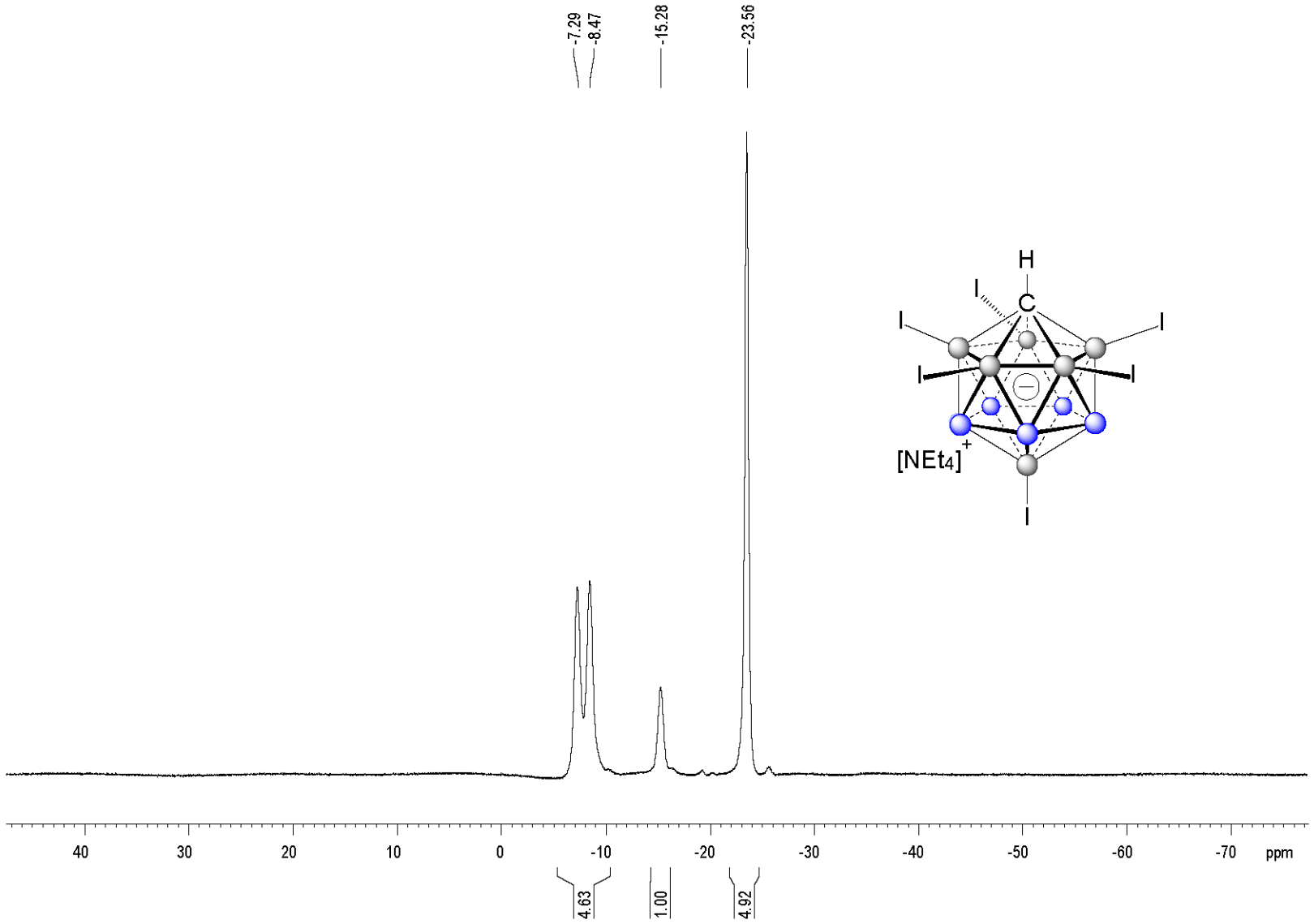
F2 - Acquisition Parameters
Date_                   20221009
Time                       15.56
INSTRUM                    spect
PROBHD    5 mm PABBO BB/
PULPROG                  zgig30
TD                        16384
SOLVENT                 Acetone
NS                             16
DS                              4
SWH                     8012.820 Hz
FIDRES                  0.489064 Hz
AQ                     1.0223616 sec
RG                        193.34
DW                        62.400 usec
DE                         6.50 usec
TE                        296.2 K
D1                   1.00000000 sec
D11                  0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                          1H
P1                        15.00 usec
PLW1                12.50000000 W
SFO1                400.1320007 MHz

======== CHANNEL f2 ========
CPDPRG[2                  garp4
NUC2                         11B
PCPD2                     90.00 usec
PLW2                52.96599960 W
PLW12                0.64477998 W
SFO2                128.3776050 MHz

F2 - Processing parameters
SI                         32768
SF                  400.1300069 MHz
WDW                           EM
SSB                            0
LB                         1.00 Hz
GB                             0
PC                         1.40
```

[NEt4][2,3,4,5,6-5I-12-I-CB11H6]
128M,11B NMR,20mg,0.5 mL acetone-d6*,296K



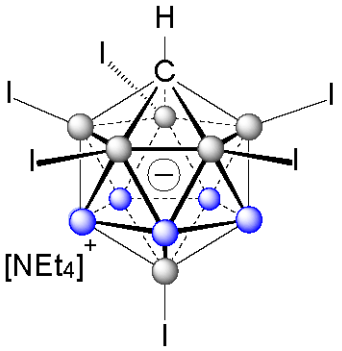

```
Current Data Parameters
NAME      11B NMR,400M,Acetor
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20221007
Time                 16.59
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg
TD                   65536
SOLVENT            Acetone
NS                       128
DS                         4
SWH                25510.203 Hz
FIDRES             0.389255 Hz
AQ                 1.2845056 sec
RG                    193.34
DW                    19.600 usec
DE                     6.50 usec
TE                    295.8 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     11B
P1                      9.93 usec
PLW1           52.96599960 W
SFO1           128.3776052 MHz

F2 - Processing parameters
SI                       32768
SF             128.3776050 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```

[NEt4][2,3,4,5,6-5I-12-I-CB11H6]
128M,11B{1H} NMR,20mg,0.5 mL acetone-d6*,296K




```
Current Data Parameters
NAME      11B{1H} NMR,400M,Acetone
EXPNO                1
PROCNO               1

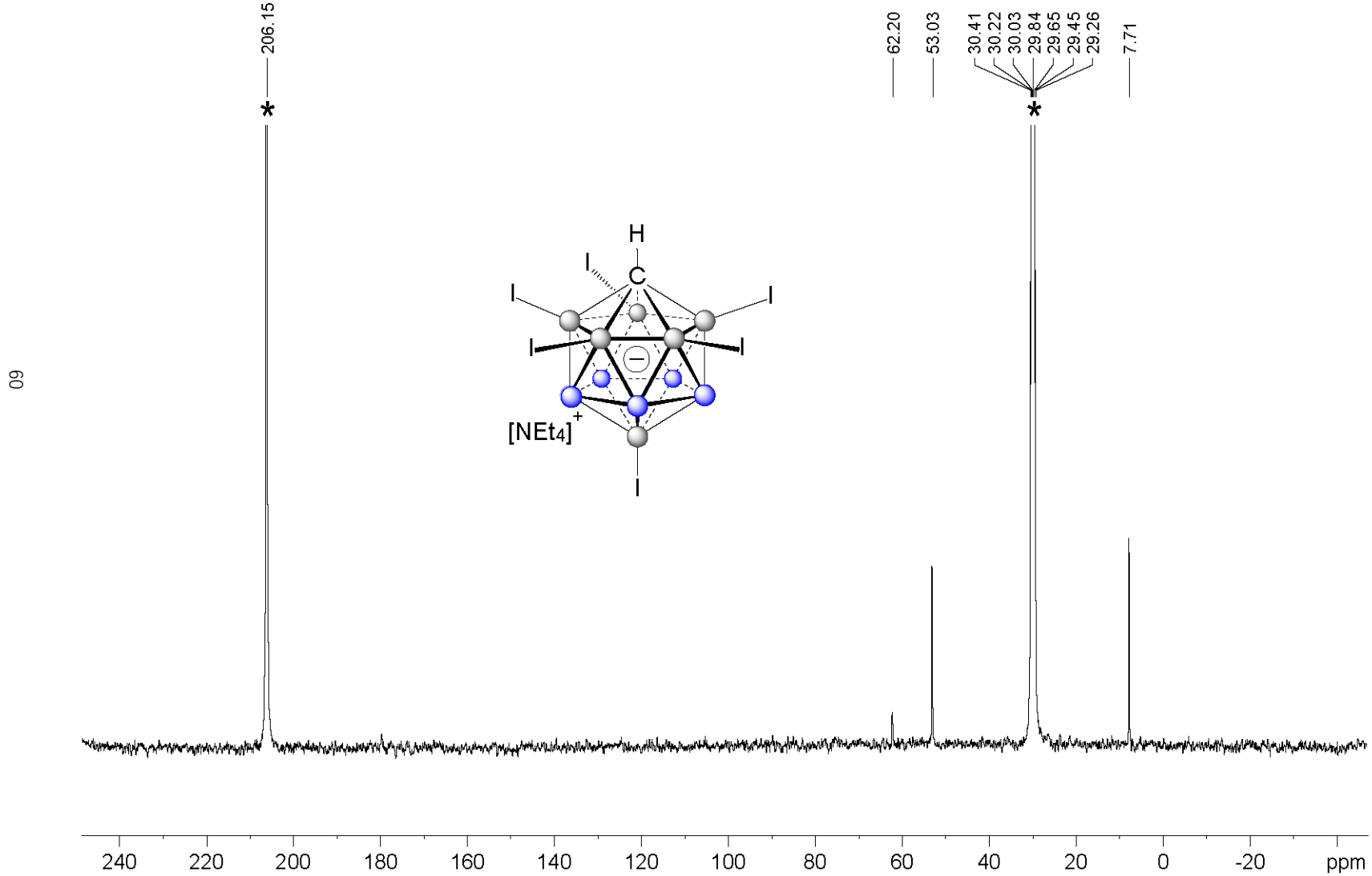
F2 - Acquisition Parameters
Date_           20221007
Time              16.53
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT        Acetone
NS                 128
DS                   4
SWH          25510.203 Hz
FIDRES        0.389255 Hz
AQ           1.2845056 sec
RG              193.34
DW              19.600 usec
DE               6.50 usec
TE              296.6 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                11B
P1                 9.93 usec
PLW1        52.96599960 W
SFO1       128.3776050 MHz

======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2                1H
PCPD2            80.00 usec
PLW2        12.50000000 W
PLW12        0.43945000 W
PLW13        0.28125000 W
SFO2       400.1320007 MHz

F2 - Processing parameters
SI                 32768
SF         128.3776050 MHz
WDW                  EM
SSB                  0
LB                10.00 Hz
GB                   0
PC                1.40
```

[NEt4][2,3,4,5,6-5I-12-I-CB11H6]
101M,13C{1H} NMR,20mg,0.5 mL acetone-d6*,296K




```
Current Data Parameters
NAME      13C{1H} NMR,400M,Acetone
EXPNO                 3
PROCNO                1

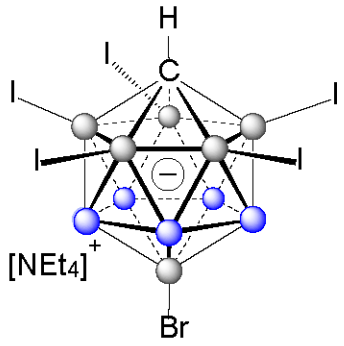
F2 - Acquisition Parameters
Date_          20221009
Time              16.44
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                 1024
DS                    4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010048 sec
RG               193.34
DW               16.800 usec
DE                6.50 usec
TE               297.1 K
D1           1.50000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PLW1        53.00000000 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PLW2        12.50000000 W
PLW12        0.43945000 W
PLW13        0.28125000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6126813 MHz
WDW                  EM
SSB                   0
LB                10.00 Hz
GB                    0
PC                1.40
```

[NEt4][2,3,4,5,6-5I-12-Br-CB11H6]
400MHz,1H{11B} NMR,5mg, 0.5mL Acetonitrile-d3*,296K



61

```
Current Data Parameters
NAME      1H{11B} NMR,400M,Aceto
EXPNO                          2
PROCNO                         1

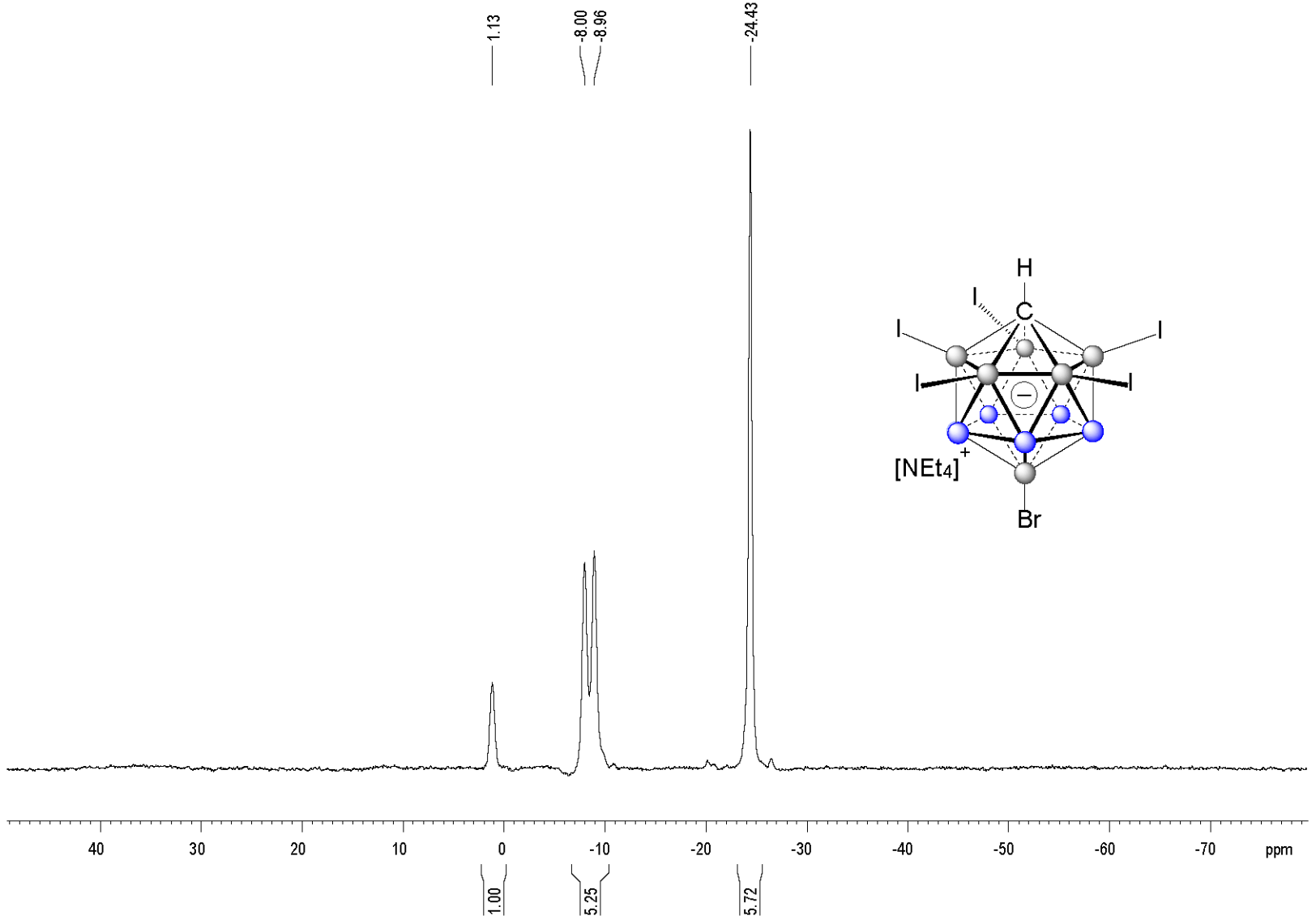
F2 - Acquisition Parameters
Date_                   20221011
Time                        7.17
INSTRUM                    spect
PROBHD    5 mm PABBO BB/
PULPROG                   zgig30
TD                         16384
SOLVENT                    CD3CN
NS                            16
DS                             4
SWH                     8012.820 Hz
FIDRES                  0.489064 Hz
AQ                     1.0223616 sec
RG                        193.34
DW                        62.400 usec
DE                         6.50 usec
TE                        295.0 K
D1                   1.00000000 sec
D11                  0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                          1H
P1                        15.00 usec
PLW1                12.50000000 W
SFO1                400.1320007 MHz

======== CHANNEL f2 ========
CPDPRG[2                  garp4
NUC2                         11B
PCPD2                     90.00 usec
PLW2                52.96599960 W
PLW12                0.64477998 W
SFO2                128.3776050 MHz

F2 - Processing parameters
SI                         32768
SF                  400.1300000 MHz
WDW                           EM
SSB                            0
LB                         1.00 Hz
GB                             0
PC                         1.40
```

[NEt4][2,3,4,5,6-5I-12-Br-CB11H6]
160MHz,11B NMR,5mg, 0.5mL Acetone-d6*,296K



62

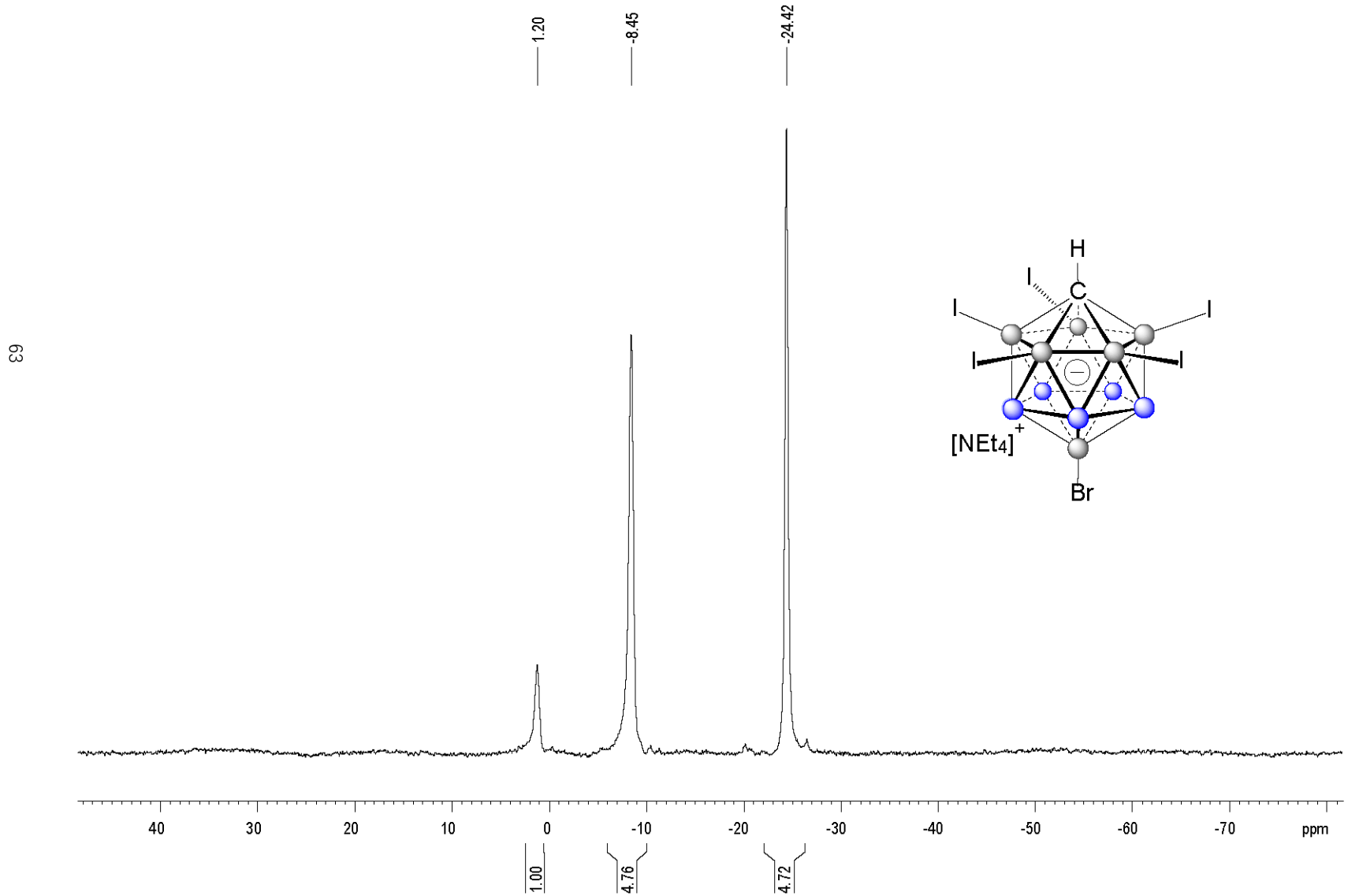
```
Current Data Parameters
NAME      11B NMR,500M,Acetone
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_              20220607
Time                  17.23
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                    296.1 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                    10.00 Hz
GB                          0
PC                     1.40
```

[NEt4][2,3,4,5,6-5I-12-Br-CB11H6]
160MHz,11B{1H} NMR,5mg,0.5mL Acetone-d6*,296K



63

```
Current Data Parameters
NAME      11B{1H} NMR,500M,Aceton
EXPNO                 4
PROCNO                1

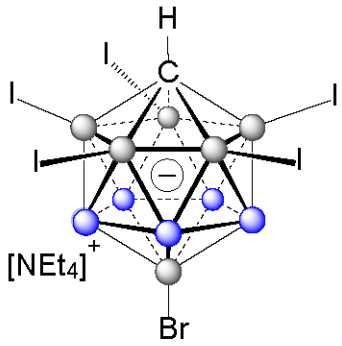
F2 - Acquisition Parameters
Date_           20220607
Time              17.27
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                   16
DS                    0
SWH           32051.281 Hz
FIDRES         0.489064 Hz
AQ            1.0223616 sec
RG                  203
DW               15.600 usec
DE                6.50 usec
TE               296.4 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         0.42394000 W
PLW13         0.27131999 W
SFO2        500.1325007 MHz

F2 - Processing parameters
SI                32768
SF          160.4615790 MHz
WDW                EM
SSB                 0
LB                10.00 Hz
GB                  0
PC                1.40
```

[NEt4][2,3,4,5,6-5I-12-Br-CB11H6]
101M,13C{1H} NMR,5mg,0.5 mL Acetone-d6*,296K



206.44 58.06 53.03 30.45 30.26 30.07 29.88 29.68 29.49 29.31 7.71

* *

64

220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

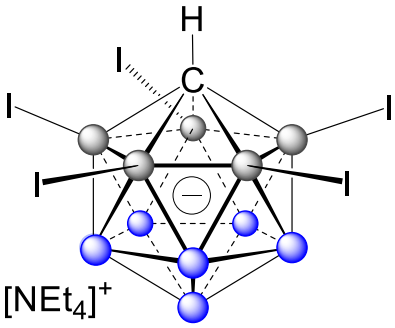
```
Current Data Parameters
NAME      13C{1H} NMR,400M,Acetone
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20220913
Time              14.56
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                 2048
DS                    4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010048 sec
RG               193.34
DW               16.800 usec
DE                6.50 usec
TE               296.9 K
D1            1.50000000 sec
D11           0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1                10.00 usec
PLW1         53.00000000 W
SFO1         100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2            80.00 usec
PLW2         12.50000000 W
PLW12         0.43945000 W
PLW13         0.28125000 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6126784 MHz
WDW                   EM
SSB                    0
LB                10.00 Hz
GB                     0
PC                 1.40
```


H
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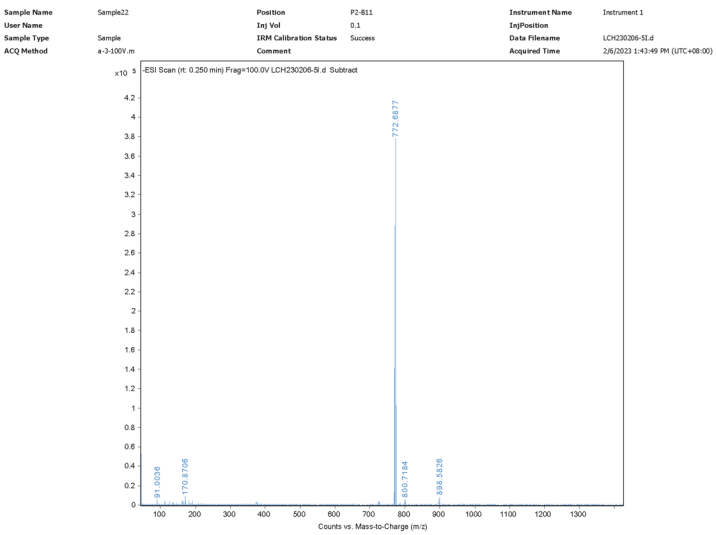
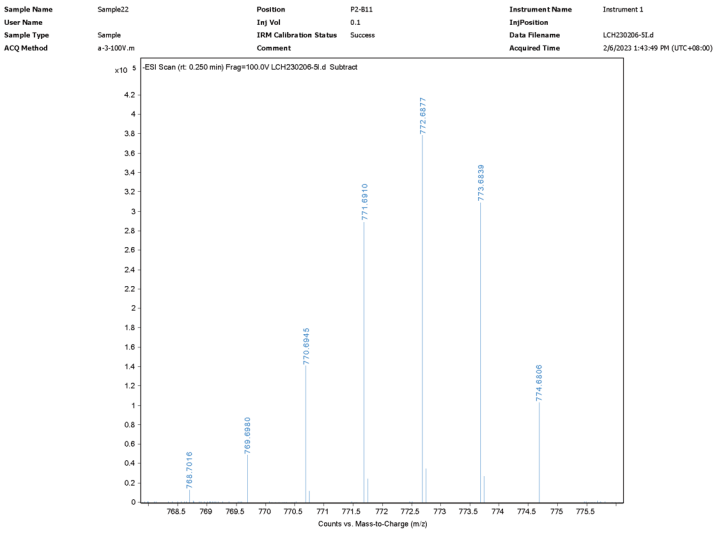
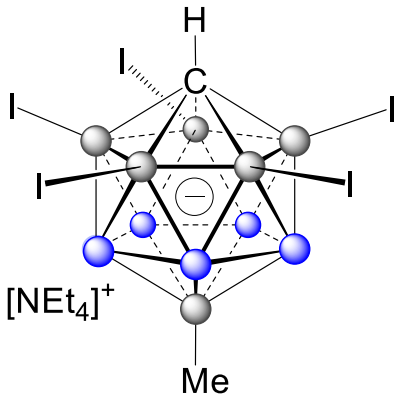
[NEt4]+

ESI-HRMS


Sample Name Sample22
User Name
Sample Type Sample
ACQ Method a-3-100V.m
Position P2-B11
Inj Vol 0.1
IRM Calibration Status Success
Comment
Instrument Name Instrument 1
InjPosition
Data Filename LCH230206-SI.d
Acquired Time 2/6/2023 1:43:49 PM (UTC+08:00)
-ESI Scan (rt: 0.250 min) Frag=100.0V LCH230206-SI.d Subtract
x10 5
772.6877
91.0038
170.8706
800.7184
868.5626
Counts vs. Mass-to-Charge (m/z)


Sample Name Sample22
User Name
Sample Type Sample
ACQ Method a-3-100V.m
Position P2-B11
Inj Vol 0.1
IRM Calibration Status Success
Comment
Instrument Name Instrument 1
InjPosition
Data Filename LCH230206-SI.d
Acquired Time 2/6/2023 1:43:49 PM (UTC+08:00)
-ESI Scan (rt: 0.250 min) Frag=100.0V LCH230206-SI.d Subtract
x10 5
772.6877
771.6910
773.6839
770.6845
769.6980
768.7016
774.6906
Counts vs. Mass-to-Charge (m/z)


H
I
C
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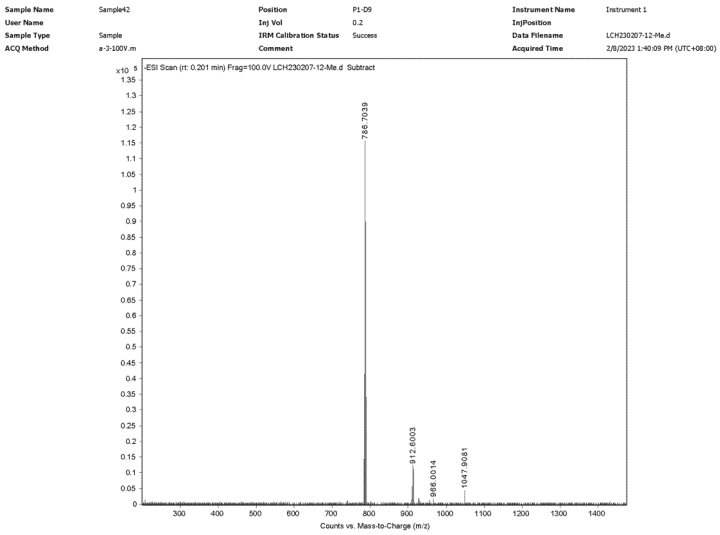
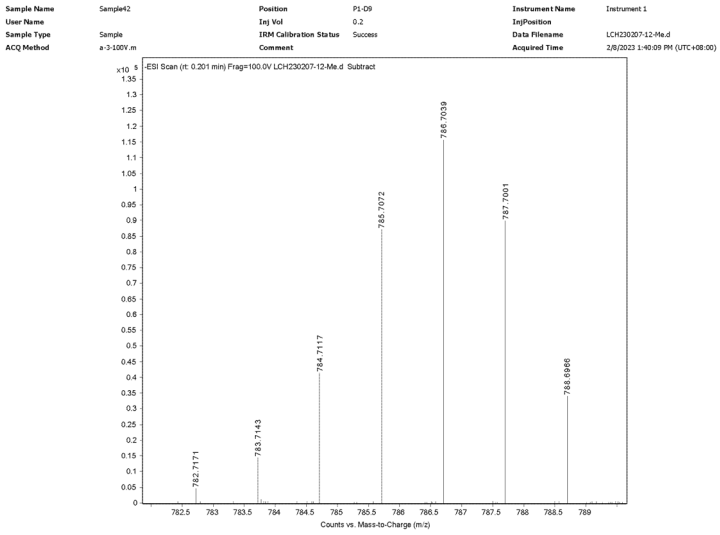
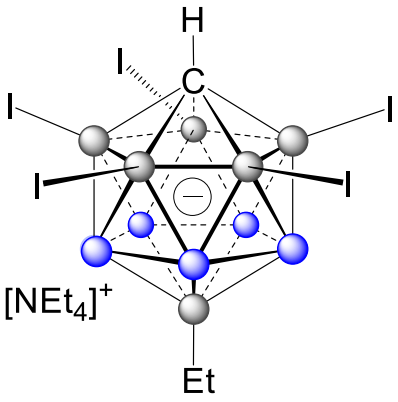
I
[NEt4]+
Me

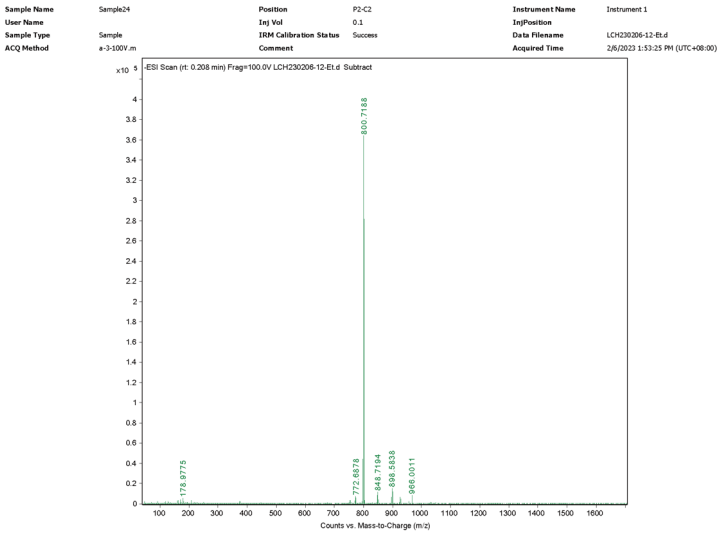
ESI-HRMS

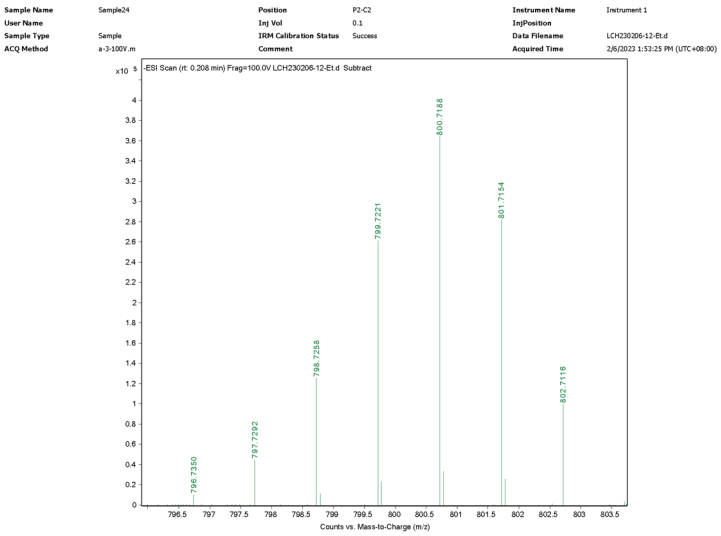

Sample Name Sample42
User Name
Sample Type Sample
ACQ Method a-3-100V.m
Position P1-D9
Inj Vol 0.2
IRM Calibration Status Success
Comment
Instrument Name Instrument 1
InjPosition
Data Filename LCH230207-12-Me.d
Acquired Time 2/8/2023 1:40:09 PM (UTC+08:00)
ESI Scan (rt: 0.201 min) Frag=100.0V LCH230207-12-Me.d Subtract
x10 5
786.7039
912.6003
966.0014
1047.9081
Counts vs. Mass-to-Charge (m/z)

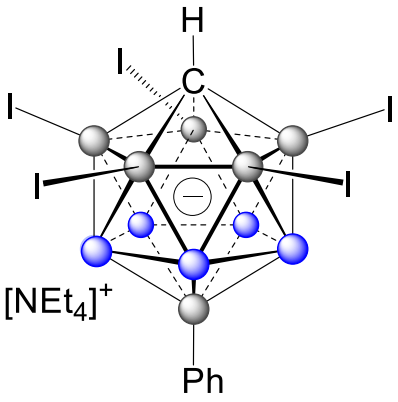

Sample Name Sample42
User Name
Sample Type Sample
ACQ Method a-3-100V.m
Position P1-D9
Inj Vol 0.2
IRM Calibration Status Success
Comment
Instrument Name Instrument 1
InjPosition
Data Filename LCH230207-12-Me.d
Acquired Time 2/8/2023 1:40:09 PM (UTC+08:00)
ESI Scan (rt: 0.201 min) Frag=100.0V LCH230207-12-Me.d Subtract
x10 5
782.7171
783.7143
784.7117
785.7072
786.7039
787.7001
788.6966
Counts vs. Mass-to-Charge (m/z)


H
I
C
I
I
I
I
[NEt4]+
Et

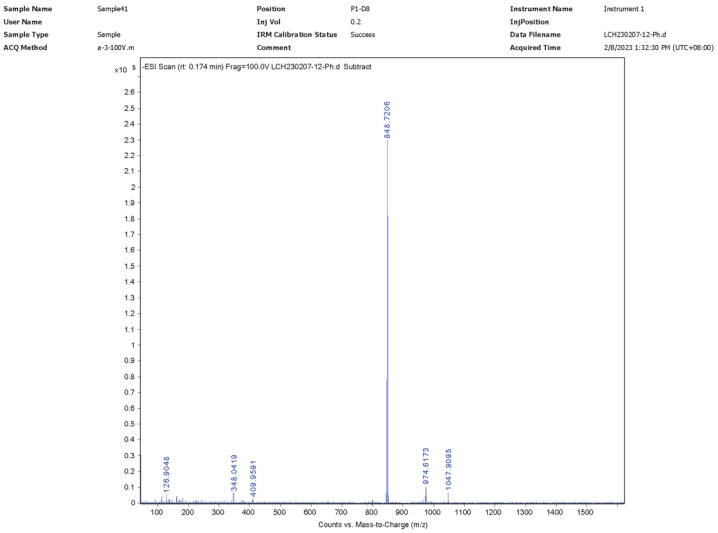
ESI-HRMS

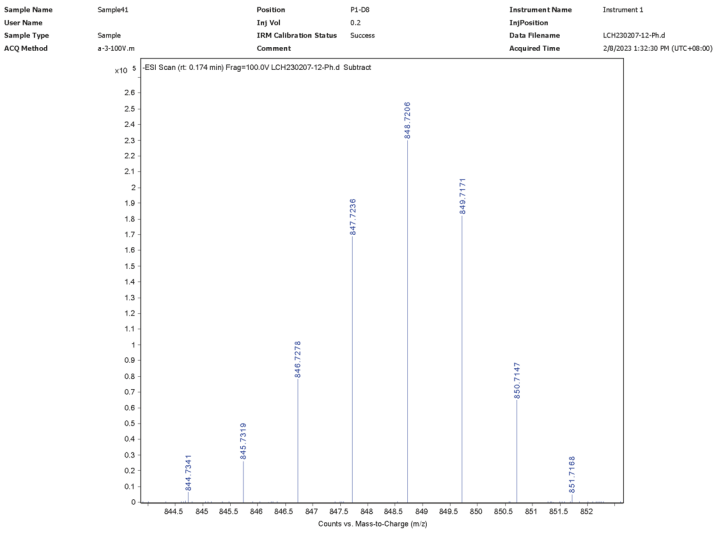

Sample Name Sample24
Position P2-C2
Instrument Name Instrument 1
User Name
Inj Vol 0.1
InjPosition
Sample Type Sample
IRM Calibration Status Success
Data Filename LCH230206-12-Et.d
ACQ Method a-3-100V.m
Comment
Acquired Time 2/6/2023 1:53:25 PM (UTC+08:00)
ESI Scan (rt: 0.208 min) Frag=100.0V LCH230206-12-Et.d Subtract
x10 5
800.7188
776.8775
772.6878
848.7184
898.5838
966.0011
Counts vs. Mass-to-Charge (m/z)

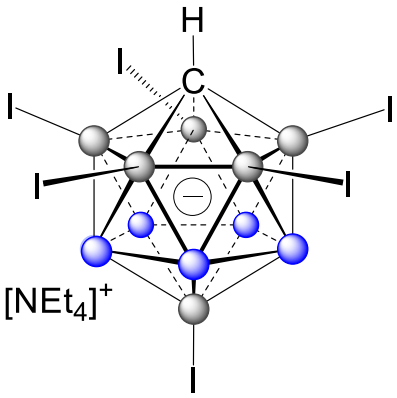

Sample Name Sample24
Position P2-C2
Instrument Name Instrument 1
User Name
Inj Vol 0.1
InjPosition
Sample Type Sample
IRM Calibration Status Success
Data Filename LCH230206-12-Et.d
ACQ Method a-3-100V.m
Comment
Acquired Time 2/6/2023 1:53:25 PM (UTC+08:00)
ESI Scan (rt: 0.208 min) Frag=100.0V LCH230206-12-Et.d Subtract
x10 5
800.7188
801.7154
799.7221
798.7258
797.7292
796.7350
802.7116
Counts vs. Mass-to-Charge (m/z)


H
I
C
I
I
I
I
[NEt4]+
Ph

ESI-HRMS


Sample Name Sample41
User Name
Sample Type Sample
ACQ Method a-3-100V.m
Position P1-D8
Inj Vol 0.2
IRM Calibration Status Success
Comment
Instrument Name Instrument 1
InjPosition
Data Filename LCH230207-12-Ph.d
Acquired Time 2/8/2023 1:32:30 PM (UTC+08:00)
ESI Scan (rt: 0.174 min) Frag=100.0V LCH230207-12-Ph.d Subtract
x10 5
848.7206
126.9048
348.0419
409.9591
974.6173
1047.9095
Counts vs. Mass-to-Charge (m/z)


Sample Name Sample41
User Name
Sample Type Sample
ACQ Method a-3-100V.m
Position P1-D8
Inj Vol 0.2
IRM Calibration Status Success
Comment
Instrument Name Instrument 1
InjPosition
Data Filename LCH230207-12-Ph.d
Acquired Time 2/8/2023 1:32:30 PM (UTC+08:00)
ESI Scan (rt: 0.174 min) Frag=100.0V LCH230207-12-Ph.d Subtract
x10 5
848.7206
847.7236
849.7171
846.7278
850.7147
845.7319
844.7341
851.7168
Counts vs. Mass-to-Charge (m/z)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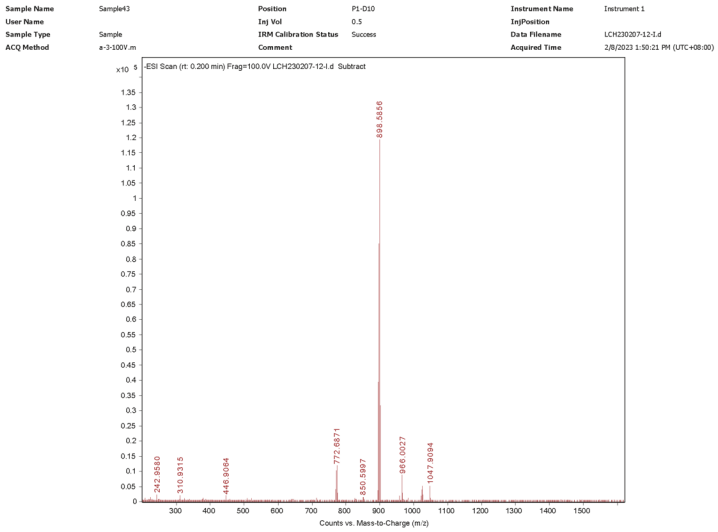
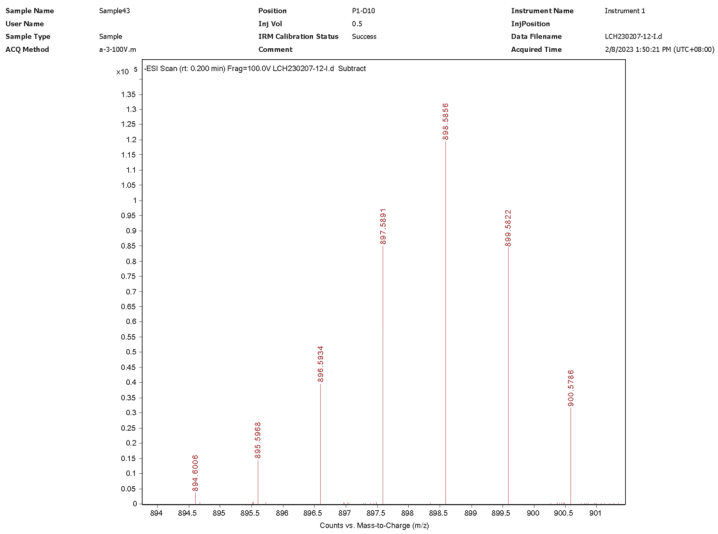
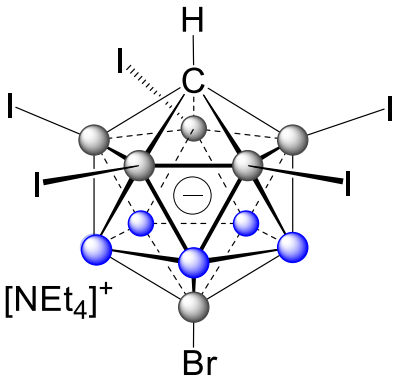

Sample Name Sample43
User Name
Sample Type Sample
ACQ Method a-3-100V.m
Position P1-D10
Inj Vol 0.5
IRM Calibration Status Success
Comment
Instrument Name Instrument 1
InjPosition
Data Filename LCH230207-12-I.d
Acquired Time 2/8/2023 1:50:21 PM (UTC+08:00)
-ESI Scan (rt: 0.200 min) Frag=100.0V LCH230207-12-I.d Subtract
x10 5
898.5856
772.6871
866.0027
1047.8094
650.5997
446.9084
310.8315
242.8580
Counts vs. Mass-to-Charge (m/z)


Sample Name Sample43
User Name
Sample Type Sample
ACQ Method a-3-100V.m
Position P1-D10
Inj Vol 0.5
IRM Calibration Status Success
Comment
Instrument Name Instrument 1
InjPosition
Data Filename LCH230207-12-I.d
Acquired Time 2/8/2023 1:50:21 PM (UTC+08:00)
-ESI Scan (rt: 0.200 min) Frag=100.0V LCH230207-12-I.d Subtract
x10 5
898.5856
897.5891
899.5822
896.5934
900.5768
895.5968
894.6006
Counts vs. Mass-to-Charge (m/z)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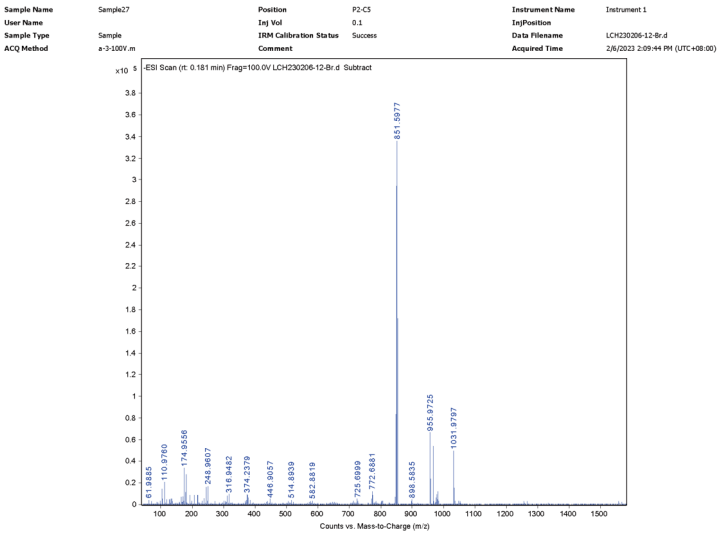
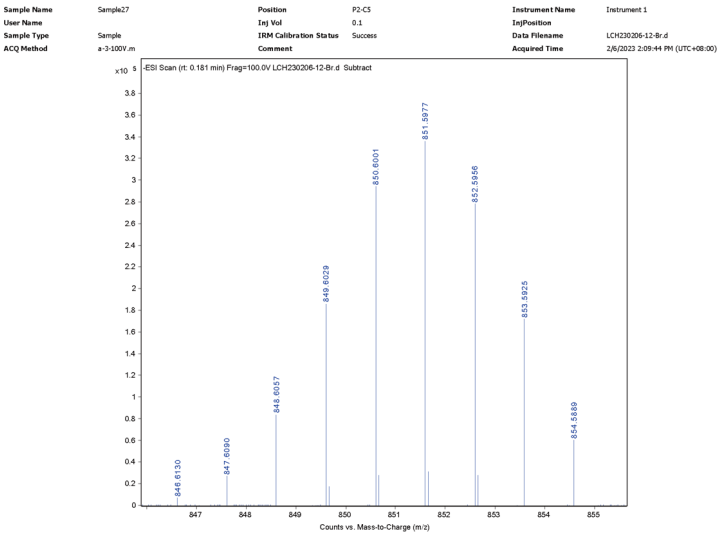
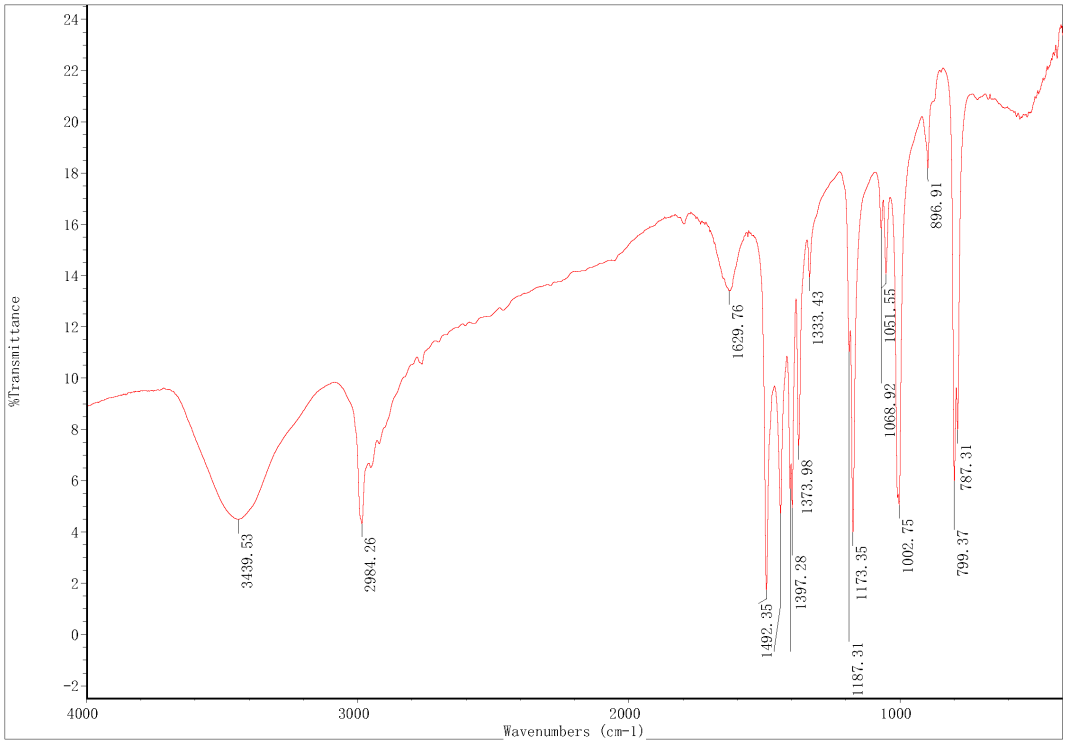
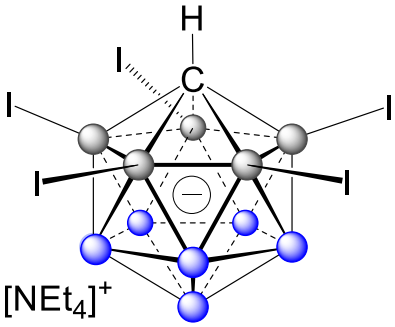
ESI-HRMS


Sample Name Sample27
User Name
Sample Type Sample
ACQ Method a-3-100V.m
Position P2-C5
Inj Vol 0.1
IRM Calibration Status Success
Comment
Instrument Name Instrument 1
InjPosition
Data Filename LCH230206-12-Br.d
Acquired Time 2/6/2023 2:09:44 PM (UTC+08:00)
-ESI Scan (rt: 0.181 min) Frag=100.0V LCH230206-12-Br.d Subtract
x10 5
851.5977
955.9725
1031.9797
888.5835
772.6881
725.6899
582.8819
514.8939
449.9057
374.2379
316.9482
248.9607
174.9556
110.9760
61.9885
Counts vs. Mass-to-Charge (m/z)


Sample Name Sample27
User Name
Sample Type Sample
ACQ Method a-3-100V.m
Position P2-C5
Inj Vol 0.1
IRM Calibration Status Success
Comment
Instrument Name Instrument 1
InjPosition
Data Filename LCH230206-12-Br.d
Acquired Time 2/6/2023 2:09:44 PM (UTC+08:00)
-ESI Scan (rt: 0.181 min) Frag=100.0V LCH230206-12-Br.d Subtract
x10 5
851.5977
850.6001
852.5956
849.6029
853.5925
848.6057
854.5889
847.6090
846.6130
Counts vs. Mass-to-Charge (m/z)

Reference IR spectrum of $[NEt_4]Br$

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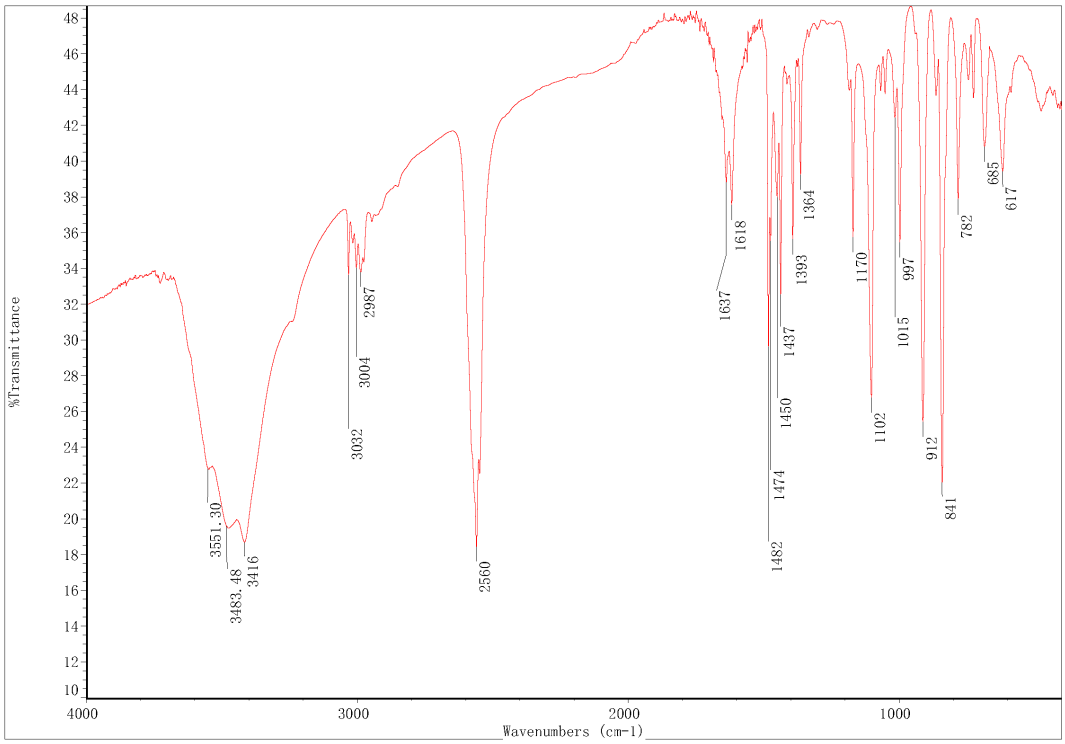
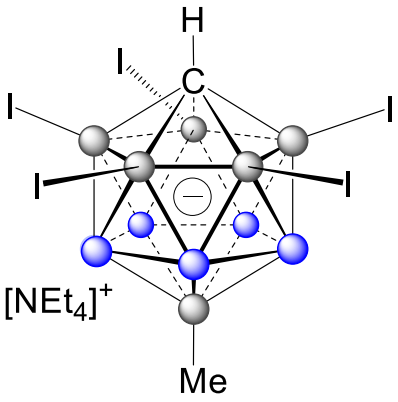

%Transmittance
Wavenumbers (cm-1)
4000
3000
2000
1000
3551.30
3483.48
3416
3032
3004
2987
2560
1637
1618
1482
1474
1450
1437
1393
1364
1170
1102
1015
997
912
841
782
685
617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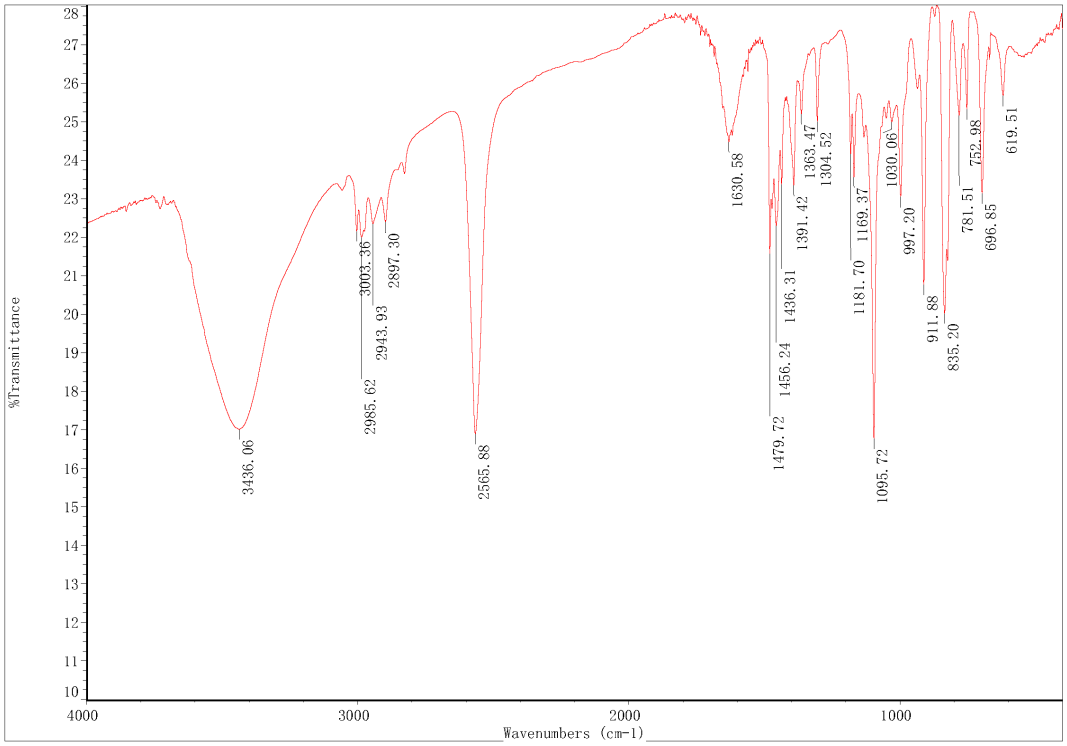
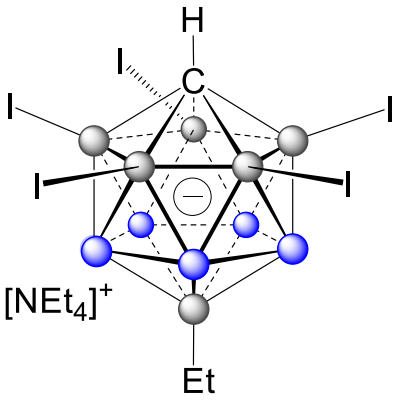

%Transmittance
Wavenumbers (cm-1)
4000
3000
2000
1000
3436.06
2985.62
3003.36
2943.93
2897.30
2565.88
1630.58
1479.72
1456.24
1436.31
1391.42
1363.47
1304.52
1181.70
1169.37
1095.72
1030.06
997.20
911.88
835.20
781.51
752.98
696.85
619.51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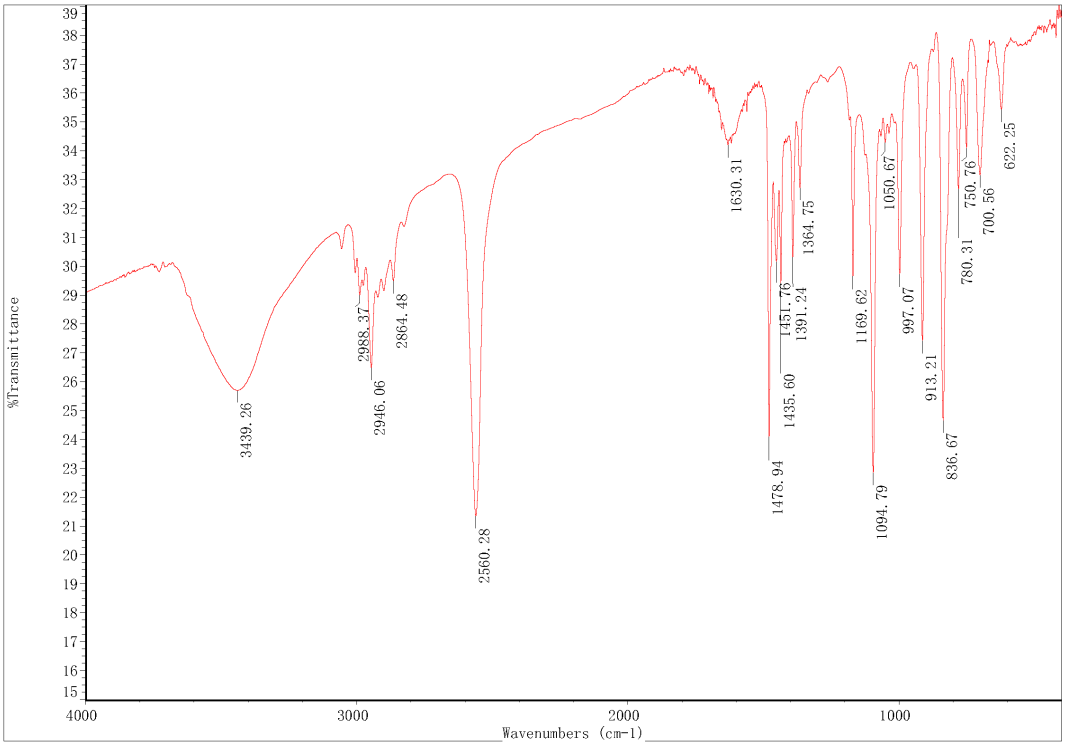
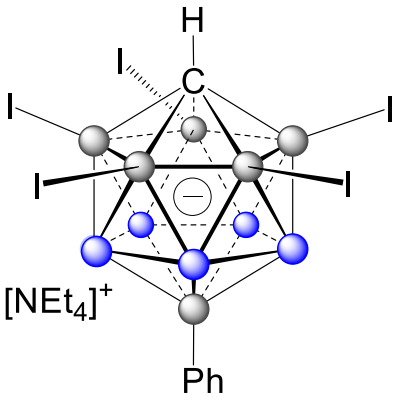

%Transmittance
Wavenumbers (cm-1)
4000
3000
2000
1000
3439.26
2988.37
2946.06
2864.48
2560.28
1630.31
1478.94
1435.60
1451.76
1391.24
1364.75
1169.62
1094.79
1050.67
997.07
913.21
836.67
780.31
750.76
700.56
622.25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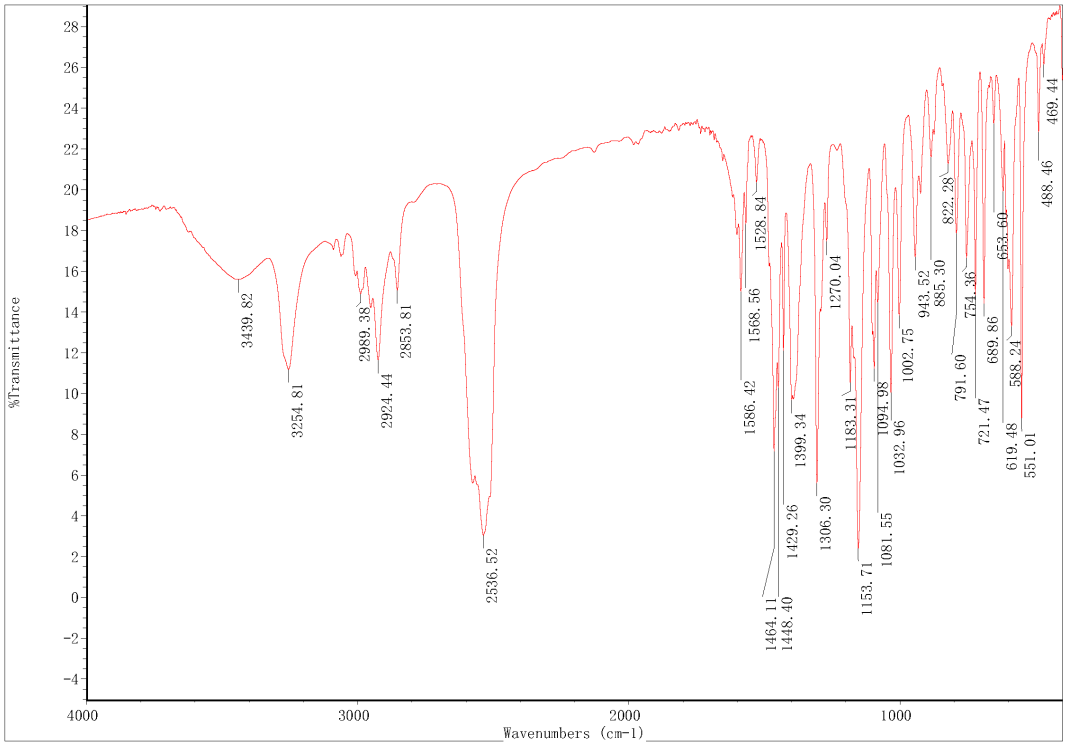
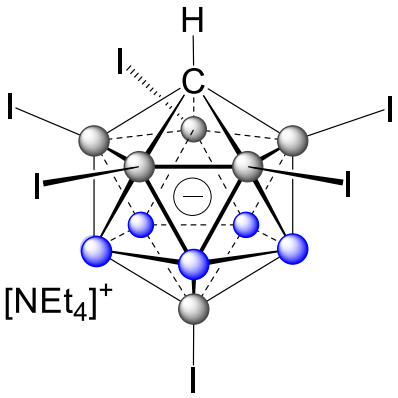

%Transmittance
Wavenumbers (cm-1)
4000
3000
2000
1000
3439.82
3254.81
2989.38
2924.44
2853.81
2536.52
1586.42
1568.56
1528.84
1464.11
1448.40
1429.26
1399.34
1306.30
1270.04
1183.31
1153.71
1094.98
1081.55
1032.96
1002.75
943.52
885.30
822.28
791.60
754.36
721.47
689.86
653.60
619.48
588.24
551.01
488.46
469.44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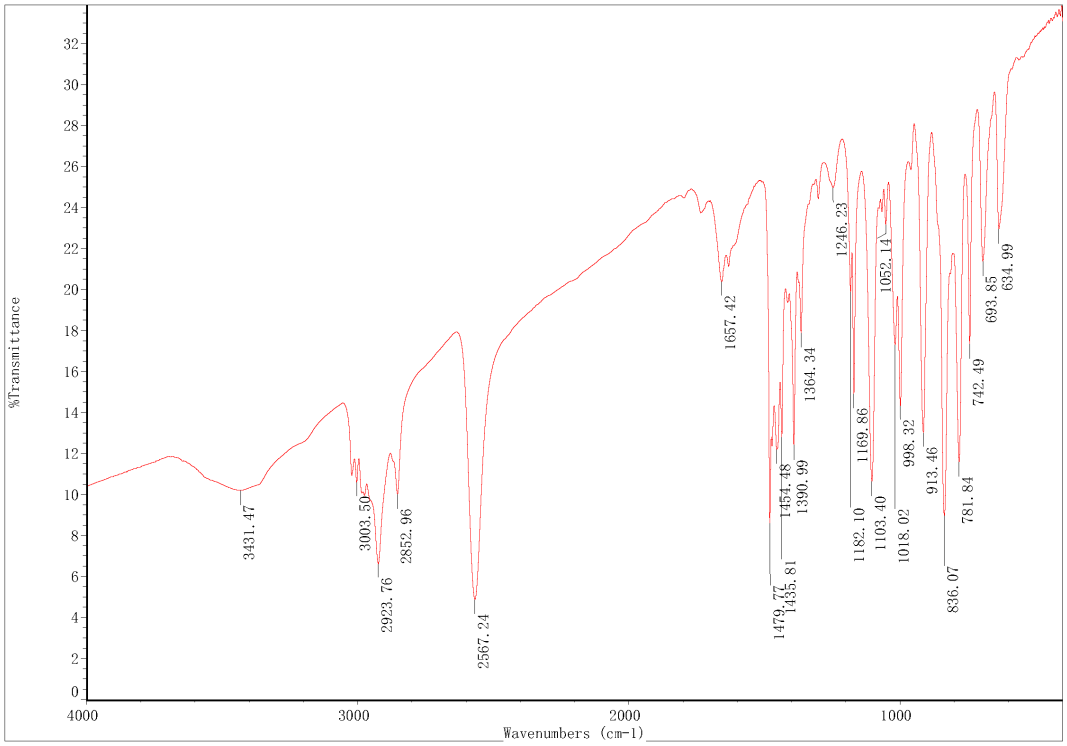
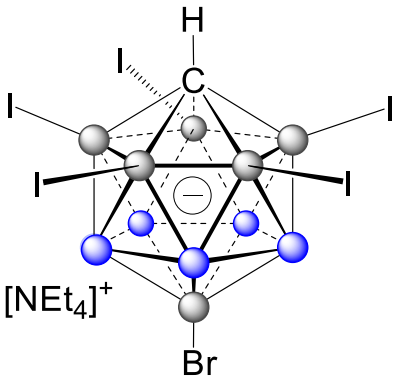

%Transmittance
Wavenumbers (cm-1)
4000
3000
2000
1000
3431.47
3003.50
2923.76
2852.96
2567.24
1657.42
1479.77
1454.48
1435.81
1390.99
1364.34
1246.23
1182.10
1169.86
1103.40
1052.14
1018.02
998.32
913.46
836.07
781.84
742.49
693.85
634.99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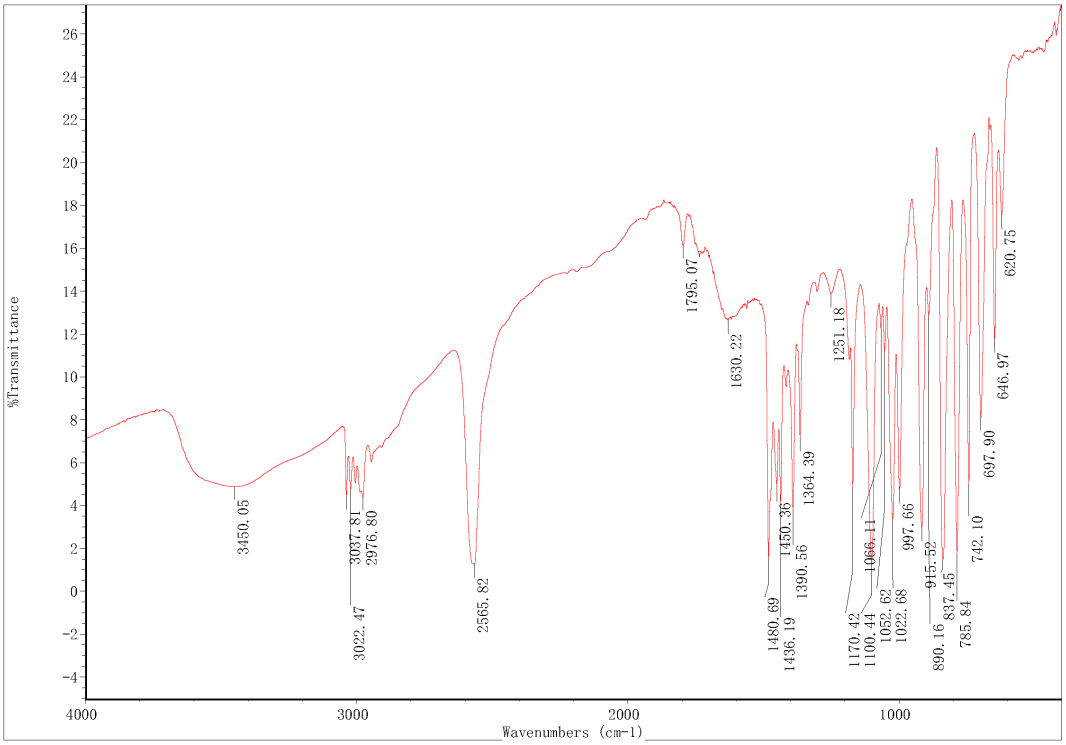
%Transmittance
Wavenumbers (cm-1)
4000
3000
2000
1000
3450.05
3037.81
2976.80
3022.47
2565.82
1795.07
1630.22
1480.69
1450.36
1436.19
1390.56
1364.39
1251.18
1170.42
1100.44
1066.11
1052.62
1022.68
997.66
915.52
890.16
837.45
785.84
742.10
697.90
646.97
620.75