Table of Contents

I General Information

Chemicals

If not otherwise specified, reagents and organic solvents were commercially available from the company Energy (China) and used without further purification. Acetone-d_6 and dimethyl sulfoxide- d_6 and CD_3CN and were purchased from Cambridge Isotope Laboratories. The anions $[CB_{11}H_{12}]^-$,[1] $[12\text{-I-}CB_{11}H_{11}]^-$,[2] $[12\text{-}CH_3\text{-}CB_{11}H_{11}]^-$,[3] $[1\text{-}CH_3\text{-}CB_{11}H_{11}]^-$,[4] [1-AcNH-$CB_{11}H_1^-$,[5] were prepared according to the literature.

Characterization

1) Thin-layer chromatography (TLC) was carried out using silica gel 60, F254 with a thickness of 0.25 mm. Column chromatography was performed on silica gel 60 (200-300 mesh).

2) NMR spectra were recorded on a Bruker AVANCE III 400 spectrometer (^{1}H NMR 400.13 MHz, ^{13}C NMR 100.62 MHz, ^{11}B NMR 128.38 MHz) or Bruker AVANCE III 500 spectrometer (1H NMR 500.13 MHz, 13C NMR 125.77 MHz, 11B NMR 160.46 MHz) at 23°C. 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 interpretation (where applicable). Signals were referenced against solvent peaks (^{1}H: residual C*H*$D_2C(O)CD_3$ = 2.05 ppm, C*H*D_2CN = 1.94 ppm, $^{13}C\{^1H\}$: *C*$D_3C(O)$*C*D_3 = 29.84 ppm, *C*D_3CN = 1.32 ppm). ^{11}B and $^{11}B\{^1H\}$ NMR spectra were calibrated against external $BF_3\cdot Et_2O$ = 0 ppm ($BF_3\cdot Et_2O$ capillary in C_6D_6).

Baseline correction for ^{11}B and $^{11}B\{^1H\}$ NMR spectra was carried out using the baseline correction function of the software TopSpin 4.3.0. (The NMR tube is quartz)

3) Low-resolution ESI-MS data were recorded on an Advion Expression CMS instrument, and high-resolution MS data were recorded using a Agilent 6545 Q-TOF MS instrument. The plots with a large *m/z* range demonstrate bulk purity. The zoomed-in plots show the observed isotopic pattern from the HRMS measurements.

The degree of substitution and bulk purity after purification could reliably be verified by full-range(–)-ESI-mass spectrometry because the prducts have a single negative charge and exhibit very little fragmentation.

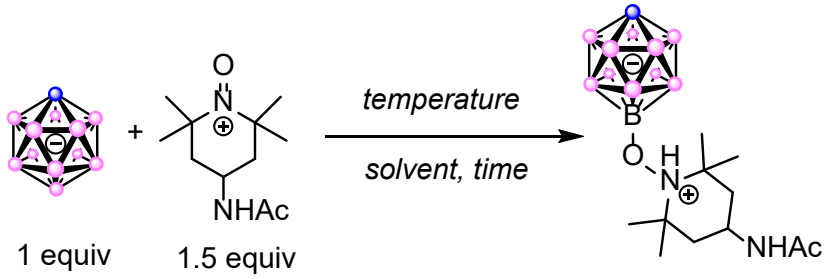
4) Single-crystal X-ray diffraction studies were performed on a Bruker D8 Venture instrument using MoK-α radiation.

5) Microwave reaction were performed on a CEM-Disover SP, focused single-mode microwave synthesizer.

6) Melting point was measured using the automated Optimelt melting point apparatus (USA).

II Experimental Section

Table S1 Optimization of reaction conditions.



Entry	T(℃)	Time (min)	Solvent[b]	Result[c]
1	60	30	MeCN	46%
2	60	60	MeCN	72%
3	60	120	MeCN	80%
4	60	180	MeCN	93%
5	80	30	MeCN	90%
6	80	60	MeCN	92%
7	80	180	MeCN	98%
8	80	180	CH_3OH	58%
9	80	180	DMF	11%
10	80	180	THF	84%
11	80	180	Acetone	23%
12	80	180	1,4-Dioxane	99%
13	80	180	DMSO	0%

[a]Reactions were conducted on a 0.18 mmol (50 mg) scale in 2 mL of solvent in a sealed vial under air; [b]MeCN = acetonitrile, DMF = dimethylformamide, THF = tetrahydrofuran, DMSO = dimethyl sulfoxide; [c]Results primarily based on NMR and ESI-MS; 1. 5 equivalents of commercial agent [AcNH-TEMPO][BF_4].

Synthesis of 2.

In a 10 ml vial, [Cs][$CB_{11}H_{12}$] (0.36 mmol, 100 mg), 4-acetylamino-2,2,6,6,-tetramethylpiperidine-1-oxoammonium tetrafluoroborate (0.54 mmol, 162 mg) were dissolved in 1, 4-dioxane (2 ml). The resulting mixture was stirred at 80 ℃ for 3 h until ESI-MS analysis showed no remaining [Cs][$CB_{11}H_{12}$]. After reaction, the mixture was concentrated on a rotary evaporator (H_2O bath, 40 ℃), most of the 1, 4-dioxane was removed. An aqueous solution of HCl (5 ml, 1 M) was added to the remaining solution, and white precipitate formed immediately. The precipitate was then collected by filtration through a fritted glass funnel (F porosity). The solid on the glass frit was washed with deionized water (3 x 5 mL, the crude product remained undissolved). The product was dried under vacuum at room temperature for 12 h to afford **2** as a white solid (124.9 mg, 97.5 %).

Synthesis of 3a.

In a 10 ml vial, [Cs][1-CH_3-$CB_{11}H_{11}$] (0.35 mmol, 100 mg), 4-acetylamino-2,2,6,6,-tetramethylpiperidine-1-oxoammonium tetrafluoroborate (0.53 mmol, 158 mg) were dissolved in 1, 4-dioxane (2 ml). The resulting mixture was stirred at 80 ℃ for 3 h until ESI-MS analysis showed no remaining [Cs][1-CH_3-$CB_{11}H_{11}$]. After reaction, the mixture was concentrated on a rotary evaporator (H_2O bath, 40 ℃), most of the 1, 4-dioxane was removed. An aqueous solution of HCl (5 ml, 1 M) was added to the remaining solution, and white precipitate formed immediately. The precipitate was then collected by filtration through a fritted glass funnel (F porosity). The solid on the glass frit was washed with deionized water (3 x 5 mL, the crude product remained undissolved). The product was dried under vacuum at room temperature for 12 h to afford **3a** as a white solid (93 mg, 72 %).

Synthesis of 3b.

In a 10 ml vial, [Cs][12-CH_3-$CB_{11}H_{11}$] (0.35 mmol, 100 mg), 4-acetylamino-2,2,6,6,-tetramethylpiperidine-1-oxoammonium tetrafluoroborate (0.53 mmol, 158 mg) were dissolved in 1, 4-dioxane (2 ml). The resulting mixture was stirred at 80 ℃ for 3 h until ESI-MS analysis showed no remaining [Cs][12-CH_3-$CB_{11}H_{11}$]. After cooling to room temperature, H_2O (2 mL) and NaOH solution (1 M in H_2O, 1 mL) was added and the resulting solution was stirred for 5 min at 25 ℃. The organic solvent was removed on a rotary evaporator (H_2O bath, 40 ℃). Et_4NBr solution (1M in H_2O, 1 mL) was added and the white precipitate formed immediately. The precipitate was then collected by filtration through a fritted glass funnel (F porosity), The solid on the glass frit was washed with deionized water (3 x 5 mL, the crude product remained undissolved). The product was dried under vacuum at 60 ℃ for 12 h to afford **3b** as a white solid (148 mg, 85%).

General procedure: synthesis of compound 4a, 4b, 4d and 4e.

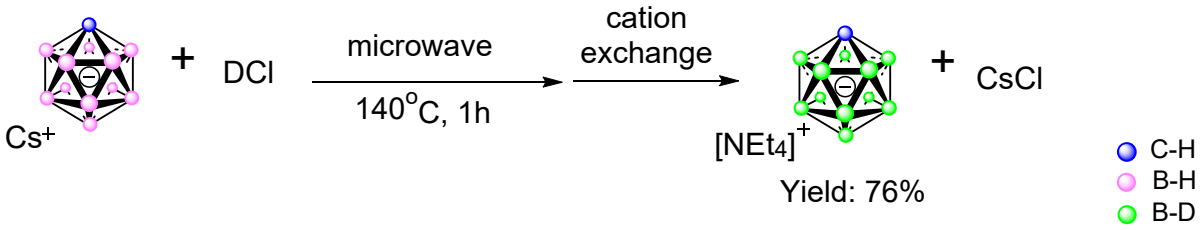
In a 10 ml vial, [Cs][$CB_{11}H_{12}$] (0.36 mmol, 100 mg), nitroxide radicals (0.9 mmol) and trifluoromethanesulfonic acid (0.9 mmol, 135 mg) were dissolved in 1, 4-dioxane (2 mL). The resulting mixture was stirred at 80 ℃ for 24 h until ESI-MS analysis showed no remaining [Cs][$CB_{11}H_{12}$]. After reaction, the mixture was concentrated on a rotary evaporator (H_2O bath, 40 ℃), most of the 1, 4-dioxane was removed. Added deionized water (5 ml) to the remaining solution, and white precipitate formed immediately. The precipitate was then collected by filtration through a fritted glass funnel (F porosity), the solid on the glass frit was washed with deionized water (3 x 5 mL, the crude product remained undissolved). The product was dried under vacuum at 60 ℃ for 12 h.

Synthesis of compound [Et_4N]•4c

The counter cation exchange to tetraethylammonium was carried out as follows:
In a 10 ml vial, [Cs] [$CB_{11}H_{12}$] (0.36 mmol, 100 mg), nitroxide radicals (0.9 mmol) and trifluoromethanesulfonic acid (0.9 mmol, 135 mg) were dissolved in 1, 4-dioxane (2 mL). The resulting mixture was stirred at 80 ℃ for 24 h until ESI-MS analysis showed no remaining [Cs][$CB_{11}H_{12}$]. After cooling to room temperature, H_2O (2 mL) and NaOH solution (1 M in H_2O, 1 mL) was added and the resulting solution was stirred for 5 min at 25 ℃. The organic solvent was removed on a rotary evaporator (H_2O bath, 40 ℃). Et_4NBr solution (1M in H_2O, 1 mL) was added and the white precipitate formed immediately. The precipitate was then collected by filtration through a fritted glass funnel (F porosity), The solid on the glass frit was washed with deionized water (3 x 5 mL, the crude product remained undissolved). The product was dried under vacuum at 60 ℃ for 12 h.

Synthesis of deuterated [Cs][$CB_{11}H_{12}$]

In a 10 mL microwave reaction tube, [Cs][$CB_{11}H_{12}$](0.36 mmol, 100 mg) was dissolved in 3 ml D_2O, followed by addition of DCl (2.3 mL, 20 wt.% in D_2O). The tube was treated in the microwave reactor at 140℃ for 40 min. After cooling to room temperature, 2 mL of aqueous Et_4NBr solution (0.4 M) was added into the tube, and white precipitate formed immediately. The generated precipitate was collected by filtration through a fritted glass funnel (F porosity). The solid on the glass frit was washed with deionized water (3 x 5 mL). The product was dried under vacuum at 60℃ for 12 h. The product was confirmed by $^{1}H\{^{11}B\}$, ^{11}B, $^{13}C\{^{1}H\}$ NMR, and HRMS.



Mechanistic study

1. The reaction of [AcNH-TEMPO][BF_4] with different boron clusters.

$Na_2B_{12}H_{12}$, *o*-$C_2B_{10}H_{12}$, *m*-$C_2B_{10}H_{12}$, and *p*-$C_2B_{10}H_{12}$ (0.36 mmol each) were used as substitutes for $[CB_{11}H_{12}]^-$, and reacted separately with 4-acetylamino-2,2,6,6-tetramethylpiperidine-1-oxoammonium tetrafluoroborate (0.54 mmol) in 2 mL of 1,4-dioxane. The resulting mixtures were stirred at 80°C for 3 hours and the outcome was assessed by ^{11}B and $^{11}B\{^1H\}$ NMR spectra. When the three kind of $\{C_2B_{10}\}$ boron clusters were used as the starting material, the NMR spectra remain unchanged before and after the reaciton. However, as the $Na_2B_{12}H_{12}$ was used, we could see multi subsitituted mixture formed accroding to the NMR spectra. The experiment results obtained obove show good agreement with the DFT calculation values.

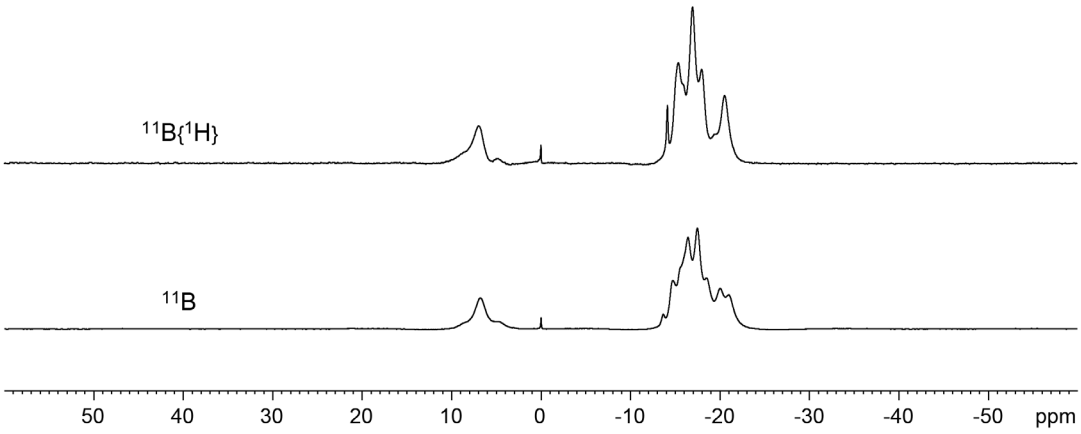


Figure S1. The ^{11}B and $^{11}B\{^1H\}$ NMR spectra of reaction mixture of $NaB_{12}H_{12}$ with [AcNH-TEMPO][BF_4] in C_6D_6.

2. Reactivity comparison experiments with different substituents of $[CB_{11}H_{12}]^-$ anion

12-I-$CB_{11}H_{12}$, 12-CH_3-$CB_{11}H_{12}$, 1-CH_3-$CB_{11}H_{12}$ and 1-AcNH-$CB_{11}H_{12}$ were used as substitutes for $[CB_{11}H_{12}]^-$, each reacted with 4-acetylamino-2,2,6,6-tetramethylpiperidine-1-oxoammonium tetrafluoroborate (0.54 mmol) dissolved in 2 mL of 1,4-dioxane, and the resulting mixtures were stirred at 80°C for 3 hours. When electron-donating –CH_3 and electron-withdrawing –NHAc substituents were introduced to the C vertex of $[CB_{11}H_{12}]^-$, the results aligned with our expectations: the desired product was obtained when [1-CH_3-$CB_{11}H_{11}]^-$ was used, while [1-AcNH-$CB_{11}H_{11}]^-$ did not undergo conversion. Additionally, when an electron-withdrawing –I group was placed at the B(12) position, the NMR spectra showed no change before and after the reaction. In contrast, when $[CB_{11}H_{12}]^-$ with an electron-donating –CH_3

group at position 12 was used, the B(7) position was activated, and the desired coupling product was detected by both NMR and HRMS.

3. Hydrogen kinetic isotope study (KIEs)

Hydrogen kinetic isotope effects (KIEs) can provide detailed information about the mechanism of chemical reactions. Because absolute rate measurements are rarely sufficiently precise, small KIEs are usually determined in competition reactions of isotopically labeled and unlabeled materials. A broadly useful alternative is to employ the high precision of isotope ratio mass spectrometry to study KIEs. As reaction proceeds, the starting materials are fractionatively enriched in isotopically slower-reacting components. The proportion of a minor isotopic component in recovered material compared to the original starting material (R/Ro) is related to the fractional conversion of reactants (F) and the KIE (relative rate for major/minor isotopic components) by eq1. As a reaction approaches completion ($F \rightarrow 1$), KIE approaches ∞, and KIEs become greatly magnified in the observable R/Ro.[6]

$$R/R_0 = (1 - F)^{(1/KIE)-1}$$

Following the general amination procedure I. $Cs[CB_{11}H_{12}]$ (10 mg), $Cs[H\text{-}CB_{11}D_{11}]$ (10 mg), 4-acetylamino-2,2,6,6,-tetramethylpiperidine-1-oxoammonium tetrafluoroborate (27 mg) were dissolved in MeCN (0.5 ml) in a NMR tube with a C_6D_6 caplliary. The resulting mixture was stirred at 80 ℃ for 60 min until conversion rate reached 95%. The ratio of D and H in the products was determined by high resolution mass spectrometry. R = 1.22, R_0 = 0.341, F = 0.95

Conv.	D (151.2537)	H (143.2035)	D/H
0 %	2185460.47	6955008.48	0.341
95 %	3007838.43	2474930.36	1.22

Table S2. Proportion of D and H at 0 % and 95 % conversion rates.

4. Electron Paramagnetic Resonance (EPR) Spectra

In a 10 ml vial, [Cs][$CB_{11}H_{12}$] (30 mg), 4-acetylamino-2,2,6,6,-tetramethylpiperidine-1-oxoammonium tetrafluoroborate (83.9 mg) were dissolved in MeCN (1.5 ml). The resulting mixture was stirred at 80 ℃. At 0, 10, 30, and 60 min, 50 μL of the reaction solution was sampled and dissolved in 1 mL of MeCN, followed by EPR signal measurement (Figure S3). Compared to the EPR signal of 0.01 mmol [AcNH-TEMPO] radicals, only a small amount of radical signal was detected in the reaction solution, suggesting the presence of minor radical impurities in [AcNH-TEMPO][BF_4] (9.3 mmol). The radical signal intensity gradually decreased as the reaction progressed, indicating that this reaction does not proceed via a radical mechanism.

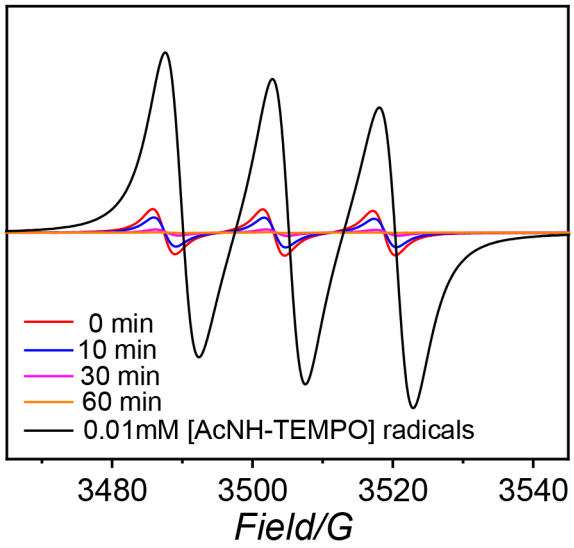


Figure S2. EPR spectra of 0.01 mM [AcNH-TEMPO] radicals and the EPR spectra measured during the reaction process. (requency: 9.841575 GHz)

5. The reaction of $[CB_{11}H_{12}]^-$ with Other nucleophilic trapping reagents

1). 4-methylpyridine

In a 5 ml vial, equipped with a magnetic stir bar, was charged with $Cs[CB_{11}H_{12}]$ (0.072 mmol, 20 mg), 4-acetylamino-2,2,6,6,-tetramethylpiperidine-1-oxoammonium tetrafluoroborate (0.108 mmol, 32.4 mg) trifluoromethanesulfonic acid (0.36 mmol, 54 mg) and 4-methylpyridine (0.36 mmol, 33.4 mg). 1, 4-dioxane (2 mL) was then added to the vial, and the resulting solution was stirred at 80 °C for 180 min. The 4-methylpyridine-substituted products were detected by high-resolution mass spectrometry.

HRMS ((–)-ESI): *m*/*z* calculated for $[C_8H_{18}B_{11}F_3NO_3S]^-$: 384.2056. Found: 384.2065.

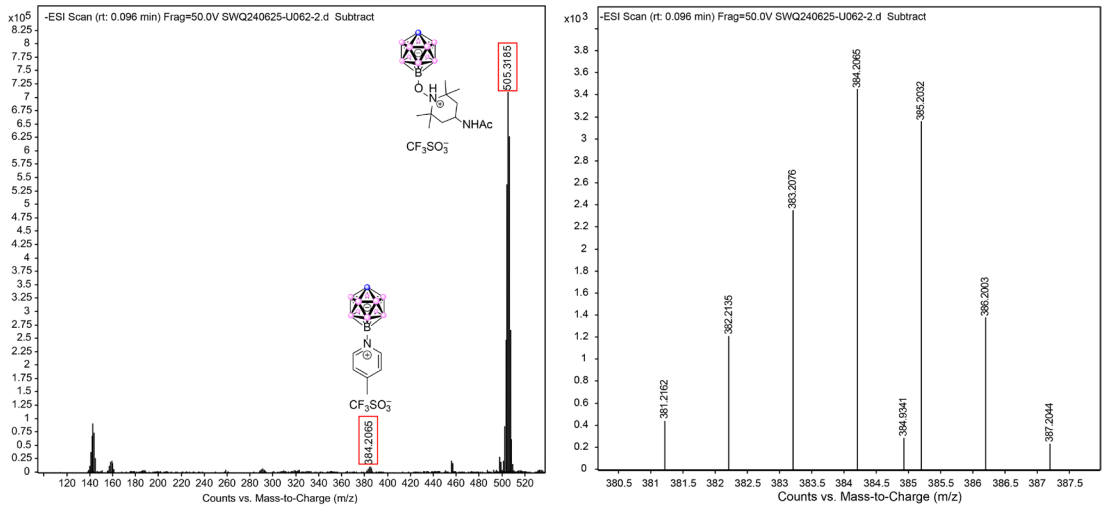


Figure S3. The HRMS ((–)-ESI) result of capture expriments using 4-methylpyridine.

2). 4-Methoxypyridine

In a 5 ml vial, equipped with a magnetic stir bar, was charged with $Cs[CB_{11}H_{12}]$ (0.072 mmol, 20 mg), 4-acetylamino-2,2,6,6,-tetramethylpiperidine-1-oxoammonium tetrafluoroborate (0.108 mmol, 32.4 mg) trifluoromethanesulfonic acid (0.36 mmol, 54 mg) and 4-Methoxypyridine (0.36 mmol, 39.3mg). 1, 4-dioxane (2 mL) was then added to the vial, and the resulting solution was stirred at 80 °C for 180 min. The 4-Methoxypyridine-substituted products were detected by high-resolution mass spectrometry.

HRMS ((–)-ESI): *m*/*z* calculated for $[C_8H_{18}B_{11}F_3NO_4S]^-$: 400.2005. Found: 400.2013.

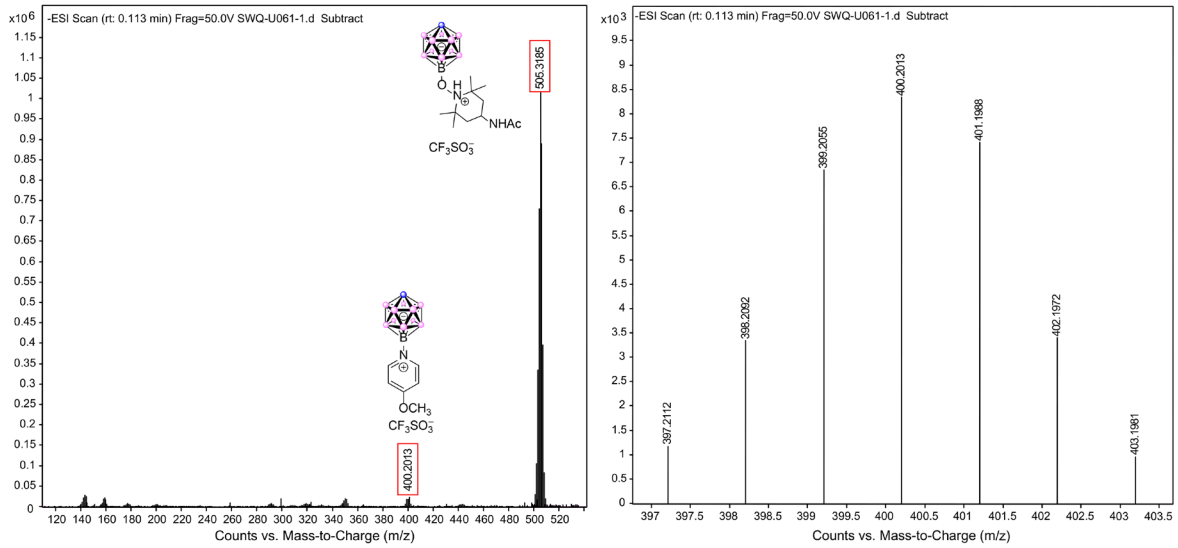


Figure S4. The HRMS ((–)-ESI) result of capture expriments using 4-Methoxypyridine.

3). H_2O

In a 5 ml vial, [Cs][$CB_{11}H_{12}$] (0.1 mmol, 27 mg), 4-acetylamino-2,2,6,6,-tetramethylpiperidine-1-oxoammonium tetrafluoroborate (0.15 mmol, 45mg) were dissolved in 2 mL of: 1) anhydrous 1,4-dioxane; 2) untreated commercial 1,4-dioxane; 3) 1,4-dioxane containing 10v% water. The resulting mixture was stirred at 80°C for 3 hours. The hydroxylamine-substituted B(12) products were detected using high-resolution mass spectrometry.

HRMS ((–)-ESI): *m*/*z* calculated for [$CH_{12}B_{11}O$]$^-$: 159.1984. Found: 159.1992.

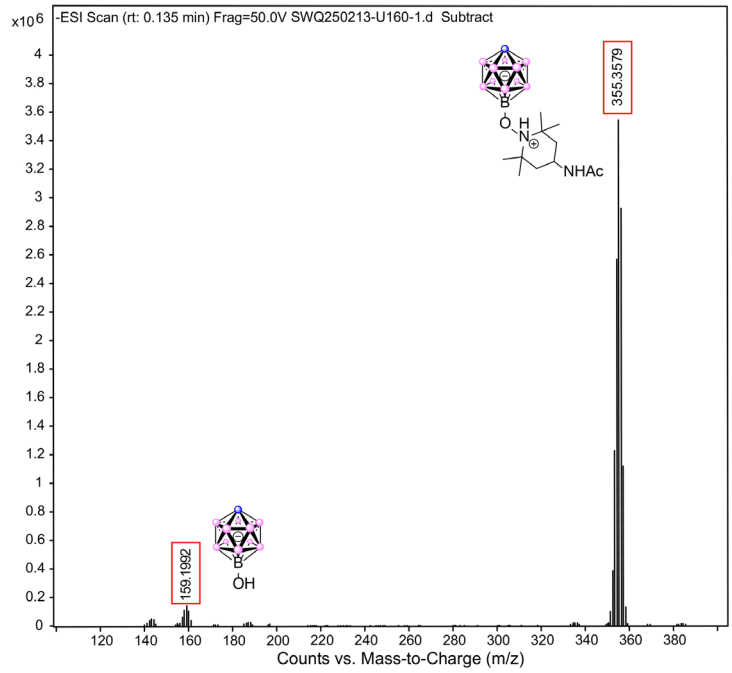


Figure S5. The HRMS ((–)-ESI) result of capture expriments with water in anhydrous 1,4-dioxane.

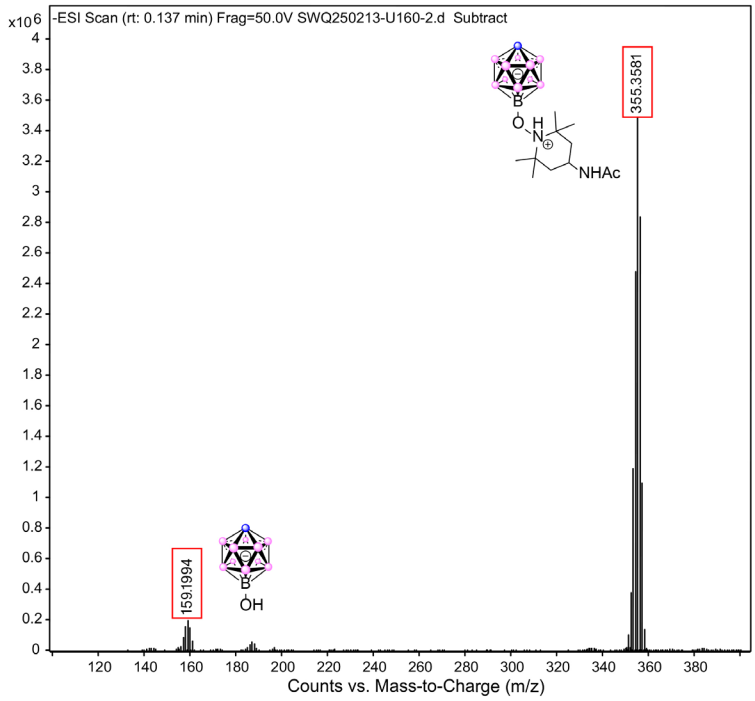


Figure S6. The HRMS ((–)-ESI) result of capture expriments with water in untreated commercial 1,4-dioxane.

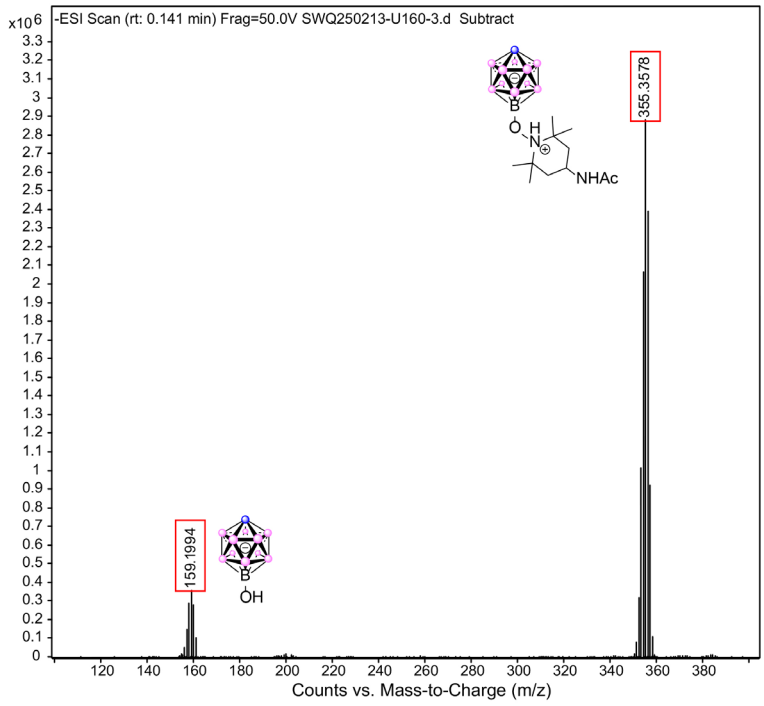
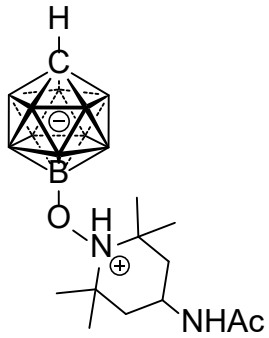


Figure S7. The HRMS ((–)-ESI) result of capture expriments with water in 1,4-dioxane with 10 v% water.

Spectroscopic Data

2



Reaction conditions: 3 h, 80 °C, 98 % yield, colorless solid, mp 268.5-269.3 °C;

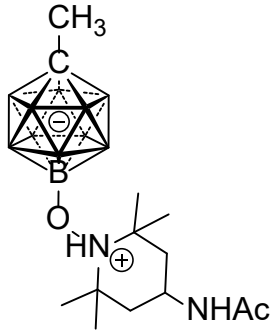
$^{1}H\{^{11}B\}$ NMR (400 MHz, DMSO-d_6, 23 °C): δ 9.77 (s, 1H, N-H), δ 7.86 (d, J = 6.90 Hz, 1H, BON-H), 4.07-3.92 (m, 1H, C-H), 2.46 (broad signal, 1H, CH of carborane), 1.87-1.81 (m, 4H, CH_2), 1.79 (s, 3H, $COCH_3$), 1.80-1.70 (broad signal, 5H, BH), 1.65-1.50 (broad signal, 5H, BH), 1.47 (s, 6H, CH_3), 1.29 (s, 6H, CH_3).

$^{11}B\{^{1}H\}$ NMR (128 MHz, Aacetone-d_6, 23 °C): δ 11.81 (1B, B-O), -14.43 (5B, B-H), -18.33 (5B, B-H).

$^{13}C\{^{1}H\}$ NMR (100 MHz, DMSO-d_6, 23 °C): δ 168.69 (CO), 70.27 ($C(CH_3)$), 41.55 (CH_2), 40.35 (cage C), 38.42 (CH), 29.06 ($COCH_3$), 22.59, 20.35 ($C(CH_3)_2$).

HRMS ((–)-ESI): m/z calculated for $[C_{12}H_{32}B_{11}N_2O_2]^-$: 355.3560. Found: 355.3572.

3a



Reaction conditions: 3 h, 80 °C, 72 % yield, colorless solid, mp 271.6-272.2 °C;

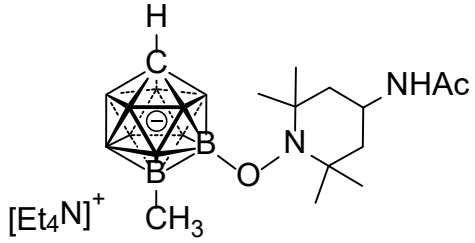
$^{1}H\{^{11}B\}$ NMR (500 MHz, DMSO-d_6, 23 °C): δ 9.67 (s, 1H, N-H), δ 7.86 (d, J = 6.90 Hz, 1H, BON-H), 4.02-3.95 (m, 1H, C-H), 2.05-1.74 (overlapping signals of CH_2 and $COCH_3$ and BH, 12H), 1.74-1.55 (broad signal, 5H, BH), 1.52 (s, 3H, cage C-CH_3), 1.43 (s, 6H, CH_3), 1.27 (s, 6H, CH_3).

$^{11}B\{^{1}H\}$ NMR (160 MHz, DMSO-d_6, 23 °C): δ 10.37 (1B, B-O), -13.92 (10B, B-H).

$^{13}C\{^{1}H\}$ NMR (126 MHz, DMSO-d_6, 23 °C): δ 168.68 (CO), 70.21 (C(CH_3)), 55.09 (cage C), 41.53 (CH_2), 38.40 (CH), 29.03 (CO$C$$H_3$), 24.54(cage C $C$$H_3$), 22.65, 20.34 (C($C$$H_3$)$_2$).

HRMS ((–)-ESI): m/z calculated for $[C_{13}H_{34}B_{11}N_2O_2]^-$: 369.3717. Found: 369.3731.

3b



Reaction conditions: 3 h, 80 °C, 85 % yield, colorless solid, mp 267.9-268.7 °C;

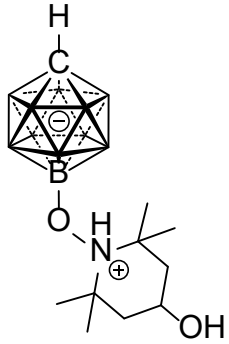
$^{1}H\{^{11}B\}$ NMR (400 MHz, CD_3CN, 23 °C):δ 6.08 (s, 1H, N-H), 4.09-3.86 (m, 1H, C-H), 3.16 (q, J = 7.2 Hz, 8H, CH_2 of cation), 1.91 (broad signal, 1H, CH of carborane), 1.80 (s, 3H, $COCH_3$), 1.77-1.57 (m, 6H, overlapping signals of CH_2 and BH), 1.40-1.17 (m, 25H, overlapping signals of CH_2, CH_3, CH_3 of cation and BH), 1.14 (s, 6H, CCH_3), 0.04 (s, 3H, B-CH_3).

$^{11}B\{^{1}H\}$ NMR (128 MHz, CD_3CN, 23 °C): δ 8.42 (1B, B-O), 0.04 (1B, B-C), -12.17 (2B, B-H), -15.35 (2B, B-H), -18.58 (2B, B-H), -20.26 (2B, B-H), -24.17 (1B, B-H).

$^{13}C\{^{1}H\}$ NMR (100 MHz, CD_3CN, 23 °C): δ 169.84 (C-O), 61.04 (C-N), 53.14 (CH_2 of cation), 47.03 (CH_2), 41.65 (CH), 41.11 (cage C), 35.50 ($CO\mathit{C}H_3$), 23.24, 21.00 (CH_3), 7.68 (CH_3 of cation), the signal for B12-C could not be detected due to severe broadening and splitting of the resonance by ^{11}B and ^{10}B.

HRMS (–)-ESI): *m*/*z* calculated for $[C_{13}H_{35}B_{11}N_2O^-$: 369.3722. Found: 369.3736.

4a



Reaction conditions: 24 h, 80 °C, 96 % yield, colorless solid, mp 232.0-232.5 °C;

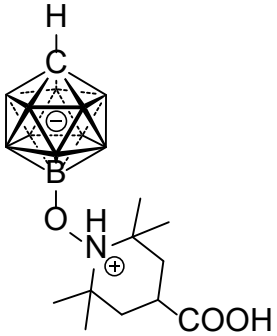
$^{1}H\{^{11}B\}$ NMR (400 MHz, CD_3CN, 23 °C): δ 7.53 (s, 1H, N-H), 4.01 (m, 1H, C-H), 3.03 (s, 1H, O-H), 2.35 (broad signal, 1H, CH of carborane), 2.03-1.97 (m, 2H, CH_2), 1.81 (broad signal, 5H, BH), 1.79-1.73 (m, 2H, CH_2), 1.63-1.50 (m, 11H, over lapping, signals of CH_3 and BH), 1.04 (s, 6H, CH_3).

$^{11}B\{^{1}H\}$ NMR (128 MHz, CD_3CN, 23 °C): δ 11.75 (1B, B-O), -14.40 (5B, B-H), -18.36 (5B, B-H).

$^{13}C\{^{1}H\}$ NMR (100 MHz, CD_3CN, 23 °C): δ 73.34 (C-N), 60.47 (C-OH), 46.01 (CH_2), 42.32 (cage C), 30.67, 21.47 (CH_3).

HRMS ((–)-ESI): *m*/*z* calculated for $[C_{10}H_{29}B_{11}NO^-$: 314.3295. Found: 314.3305.

4b



Reaction conditions: 24 h, 80 °C, 78 % yield, colorless solid, mp 238.6-238.9 °C;

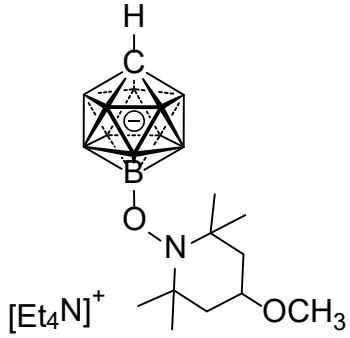
$^{1}H\{^{11}B\}$ NMR (400 MHz, CD_3CN, 23 °C): δ 9.19 (broad signal, 1H, COOH), 7.59 (broad signal, 1H, N-H), 2.84 (tt, *J* = 12.54 Hz, 3.69 Hz, 1H, C-H), 2.35 (broad signal, 1H, CH of carborane), 2.07-1.97 (m, 4H, overlapping signals of CH_2), 1.82 (broad signal, 5H, BH), 1.69-1.47 (m, 11H, over lapping, signals of CH_3 and BH), 1.35 (s, 6H, CH_3).

$^{11}B\{^{1}H\}$ NMR (128 MHz, CD_3CN, 23 °C): δ 11.74 (1B, B-O), -14.41 (5B, B-H), -18.33 (5B, B-H).

$^{13}C\{^{1}H\}$ NMR (100 MHz, CD_3CN, 23 °C): δ 175.01 (COOH), 72.47 (C-N), 42.29 (cage C), 39.93 (CH_2), 33.44 (C-H), 30.33, 20.77 (CH_3).

HRMS ((–)-ESI): *m*/*z* calculated for $[C_{11}H_{29}B_{11}NO_3]^-$: 342.3244. Found: 342.3258.

4c



Reaction conditions: 24 h, 80 °C, 88 % yield, colorless solid, mp 210.2-211.7 °C;

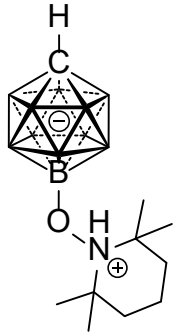
$^{1}H\{^{11}B\}$ NMR (400 MHz, acetone-d_6, 23 °C): δ 3.50 (q, J = 7.15 Hz, 8H, CH_2 of cation), 3.40-3.30 (m, 1H, CH), 3.21 (s, 3H, OCH_3), 1.98-1.62 (m, 8H, overlapping signals of CH_2, CH of carborane and BH), 1.61-1.33 (m, 17H, overlapping signals of BH and CH_3 of cation), 1.33-1.13 (m, 8H, overlapping signals of CH_2 and CH_3), 1.06 (s, 6H, CH_3).

$^{11}B\{^{1}H\}$ NMR (128 MHz, CD_3CN, 23 °C): δ 13.56 (1B, B-O), -14.18 (5B, B-H), -19.34 (5B, B-H).

$^{13}C\{^{1}H\}$ NMR (100 MHz, CD_3CN, 23 °C): δ 72.77 (C-N), 60.91 (O-CH), 55.65 (O-CH_3), 53.17 (t, J =2.7 HZ, CH_2 of cation), 46.19 (CH_2), 38.55 (cage C), 35.63, 21.50 (CH_3), 7.71 (CH_3 of cation).

HRMS ((–)-ESI): m/z calculated for $[C_{11}H_{31}B_{11}NO_2]^-$: 328.3451. Found: 328.3472.

4d



Reaction conditions: 24 h, 80 °C, 55% yield, colorless solid, mp 222.5-224.0 °C;

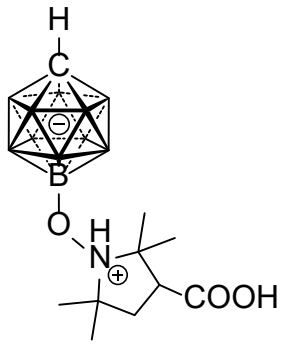
$^{1}H\{^{11}B\}$ NMR (400 MHz, CD_3CN, 23 °C): δ 7.42 (s, 1H, N-H), 2.34 (broad signal, 1H, CH of carborane), 1.92-1.68 (m, 11H, overlapping signals of CH_2 and BH), 1.61-1.49 (m, 11H, over lapping, signals of CH_3 and BH), 1.32 (s, 6H, CH_3).

$^{11}B\{^{1}H\}$ NMR (128 MHz, CD_3CN, 23 °C): δ 11.78 (1B, B-O), -14.42 (5B, B-H), -18.41 (5B, B-H).

$^{13}C\{^{1}H\}$ NMR (100 MHz, CD_3CN, 23 °C): δ 72.91 (C-N), 42.22 (cage C), 37.73 (CH_2), 30.57, 20.57 (CH_3), 16.02 (CH_2).

HRMS ((–)-ESI): *m*/*z* calculated for $[C_{10}H_{29}B_{11}NO]^-$: 298.3345. Found: 298.3358.

4e



Reaction conditions: 24 h, 80 °C, 92 % yield, colorless solid, mp 236.9-238.4 °C;

$^{1}H\{^{11}B\}$ NMR (500 MHz, CD_3CN, 23 °C): δ 9.76 (broad signal, 1H, COOH, 7.51, 6.98 (s, 1H, NH), 3.25-3.10 (m, 1H, CH), 2.43-2.32 (m, 2H, overlapping of cage CH and CH_2), 2.24-2.09 (m, 1H, CH_2), 1.77 (broad signal, 5H, BH), 1.62 (broad signal, 5H, BH), 1.71, 1.65, 1.64, 1.55, 1.44, 1.39, 1.33, 1.21 (s, 12H, CH_3).

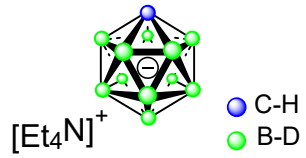
$^{11}B\{^{1}H\}$ NMR (160 MHz, CD_3CN, 23 °C): δ 11.25 (1B, B-O), -15.00 (5B, BH), -18.04 (5B, BH).

$^{13}C\{^{1}H\}$ NMR (126 MHz, CD_3CN, 23 °C): 173.7, 170.1 (COOH), 78.1, 77.8 ($C(CH_3)_2$), 76.5, 75.8 ($C(CH_3)$, 49.9, 47.9 (CH(COOH)), 42.2 (cage C), 39.2, 38.1 (CH_2), 28.3, 27.9, 25.4 (overlapping of two signals), 23.9, 23.3, 23.1, 17.3 (CH_3).

HRMS ((–)-ESI): *m*/*z* calculated for $[C_{10}H_{27}B_{11}NO_3]^-$: 328.3087. Found: 328.3099.

5

$[NEt_4]$ $[H\text{-}CB_{11}D_{11}]$



Reaction conditions: microwave, 1 h, 80 °C, 85 % yield, colorless solid;

$^{1}H\{^{11}B\}$ NMR (400 MHz, CD_3CN, 23 °C): δ 3.16 (q, J = 7.2 Hz, 8H, CH_2 of cation), 2.33 (broad signal, 1H, CH of carborane), 1.21 (broad signal, J = 6.55 Hz, 12H, CH_3 of cation).

^{11}B NMR (128 MHz, CD_3CN, 23 °C): δ -7.22 (1B, B-D), -13.54 (5B, B-D), -16.38 (5B, B-D).

$^{13}C\{^{1}H\}$ NMR (100 MHz, CD_3CN, 23 °C): δ 53.13 (CH_2 of cation), 51.74 (cage C), 7.68 (CH_3 of cation).

HRMS (–)-ESI): *m*/*z* calculated for $[HCB_{11}D_{11}]^-$: 154.2726. Found: 154.2706.

III Computational detail

All the DFT calculations were performed in Gaussian 09 D.01.[7] The geometries were fully optimized using the dispersion-corrected density functional method UB3LYP-D3[8] (with a Becke−Johnson (BJ) damping function[9]) with 6-31G(d) basis set.[10] The frequency analyses were performed at the same level to confirm that the structure was a minimum or a transition state, and to evaluate their zero-point vibrational energy and thermal corrections at 298 K. IRC analysis was applied to confirm the transition state.[11] Using the gas-phase optimized structures, the single-point energies were computed at the M06-2X[12] level with D3 dispersion correction,[13] where 6-311+G(d,p) basis set[10c, 10d, 14] was used and the solvation by MeCN was included under the SMD model.[15] The intrinsic bond orbitals are illustrated with IboView.[16] The computed structures are illustrated using CYLView software.[17]

Calculated Energies

Structure		Name	E(elec-B3LPY)[a]	G(corr-B3LYP)[b]	E(elec-M06-2X)[c]	ΔΔG (kcal/mol)	Imaginary Frequency
$[B_{12}H_{12}]^{2-}$		$[B_{12}H_{12}]^{2-}$	-305.690296	0.136479	-305.932542	0.0	None
	H^-	$[B_{12}H_{11}]^-$	-305.09794	0.127479	-305.134604	-12.2	None
	H^+	$[B_{12}H_{11}]^{3-}$	-304.704079	0.120308	-305.338728	8.3	None
$[CB_{11}H_{12}]^-$		$[CB_{11}H_{12}]^-$	-318.99442	0.142708	-319.030178	0.0	None
	H^-	*p*-$[CB_{11}H_{11}]$	-318.240096	0.132506	-318.211607	0.0	None
	H^+	*p*-$[CB_{11}H_{11}]^{2-}$	-318.15774	0.12667	-318.449694	0.0	None
	H^-	*m*-$[CB_{11}H_{11}]$	-318.231858	0.132274	-318.204835	4.1	None
	H^+	*m*-$[CB_{11}H_{11}]^{2-}$	-318.166232	0.126836	-318.452982	-2.0	None
	H^-	*o*-$[CB_{11}H_{11}]$	-318.217489	0.131327	-318.193659	10.5	None
	H^+	*o*-$[CB_{11}H_{11}]^{2-}$	-318.178425	0.127338	-318.457062	-4.2	None
$[12\text{-}CH_3\text{-}CB_{11}H_{11}]^-$		$[12\text{-}CH_3\text{-}CB_{11}H_{11}]^-$	-358.315364	0.166792	-358.343395	0.0	None
	H^-	*m*-$[12\text{-}CH_3\text{-}CB_{11}H_{10}]$	-357.554951	0.156987	-357.520133	3.2	None
	H^+	*m*-$[12\text{-}CH_3\text{-}CB_{11}H_{10}]^{2-}$	-357.489793	0.152336	-357.765634	-0.7	None
$C_2B_{10}H_{12}$		$C_2B_{10}H_{12}$	-332.130515	0.147545	-332.10266	0.0	None
	H^-	*m*-$[C_2B_{10}H_{11}]^+$	-331.19165	0.135504	-331.247266	22.0	None
	H^+	*m*-$[C_2B_{10}H_{11}]^-$	-331.466876	0.130808	-331.537183	-9.9	None
	H^-	o_1-$[C_2B_{10}H_{11}]^+$	-331.184865	0.134745	-331.242531	24.4	None
	H^+	o_1-$[C_2B_{10}H_{11}]^-$	-331.469655	0.132088	-331.542824	-12.6	None
	H^-	o_2-$[C_2B_{10}H_{11}]^+$	-331.160882	0.133536	-331.222105	36.5	None
	H^+	o_2-$[C_2B_{10}H_{11}]^-$	-331.49223	0.13279	-331.553667	-19.0	None
TS-1		B(12)-H-T	-1010.71654	0.436498	-1010.46416	None	-398.02
TS-2		B(7)-H-T	-1010.711731	0.436218	-1010.460205	None	-231.07

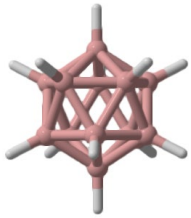
[a]Calculated electronic energies by B3LYP at gas phase. [b]Calculated thermal correction to free energies by B3LYP at gas phase. [c]Calculated electronic energies by M06-2X in MeCN.

3.1) Cartesian Coordinates and Energies

G (Solv): B3LYP-D3(BJ)/6-311++G(d,p)//B3LYP/6-31+G(d,p) Gibbs free energy including solvation by MeCN at 298 K.

All energies are given in Hartree.

$[B_{12}H_{12}]^{2-}$



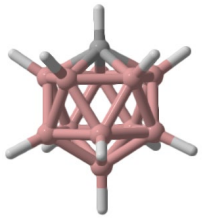
G(Solv) = -305.796063

0 imaginary frequencies

B	0.0005690	-0.0012130	-1.7450550
B	0.7951390	-1.2956750	-0.8041140
H	1.3591800	-2.2166720	-1.3443390
B	-1.4042300	0.5802310	-0.8061490
H	-2.4016630	0.9927880	-1.3476960
B	-0.7955400	1.2962680	0.7122870
H	-1.3595730	2.2172200	1.2523420
B	-0.9858430	-1.1563300	-0.8045830
H	-1.6867060	-1.9775660	-1.3455530
B	1.4049800	-0.5806140	0.7142690
H	2.4024100	-0.9931680	1.2555680
B	-1.4786540	-0.3554410	0.7136210
H	-2.5286060	-0.6073590	1.2541030
B	0.1183440	1.5144560	-0.8066160
H	0.2026730	2.5904810	-1.3481830
B	1.4779140	0.3551880	-0.8054680
H	2.5278800	0.6071100	-1.3461750
B	-0.1183980	-1.5153000	0.7147890
H	-0.2027510	-2.5912770	1.2561890
B	0.9863740	1.1568450	0.7127500

H	1.6872440	1.9780330	1.2535240
B	-0.0005590	0.0011380	1.6533040
H	-0.0007490	0.0019860	2.8608400
H	0.0007630	-0.0020570	-2.9520610

$[CB_{11}H_{12}]^-$

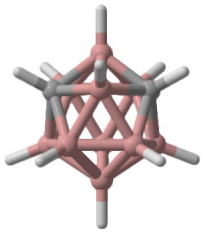


G(Solv) = -318.88747

0 imaginary frequencies

C	0.0001830	0.0017640	1.4771520
H	0.0005120	0.0029170	2.5618200
B	-0.0005310	-0.0017480	-1.7505350
B	-0.2120470	1.5071310	-0.8175190
H	-0.3622520	2.5770650	-1.3293010
B	1.0570580	-1.0959230	-0.8150460
H	1.8079390	-1.8742490	-1.3247890
B	0.2108110	-1.4993640	0.6909280
H	0.3492990	-2.4821920	1.3523230
B	1.3681200	0.6661210	-0.8173030
H	2.3399030	1.1388230	-1.3287120
B	-1.0520410	1.0904770	0.6889750
H	-1.7417520	1.8055560	1.3488580
B	1.4917250	-0.2626510	0.6895170
H	2.4698260	-0.4343080	1.3501810
B	-0.7149150	-1.3447050	-0.8149010
H	-1.2224490	-2.2995310	-1.3248930
B	-1.4996000	0.2636260	-0.8157490
H	-2.5643010	0.4509180	-1.3262830
B	0.7116830	1.3381410	0.6878180
H	1.1785030	2.2155160	1.3472390
B	-1.3613760	-0.6636270	0.6907050
H	-2.2539240	-1.0983570	1.3516570
H	-0.0006590	-0.0031260	-2.9468240

$C_2B_{10}H_{12}$

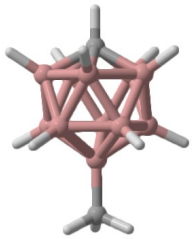


G(Solv) = -331.955115

0 imaginary frequencies

C	0.2627690	0.0025570	1.4798450
H	0.4852810	0.0046880	2.5406930
B	-0.3638460	-0.0034500	-1.6824000
B	-1.3825370	-0.9093480	-0.5420020
H	-2.3133810	-1.5543510	-0.8971900
B	-1.3781670	-0.0123770	0.9930980
H	-2.2239760	-0.0199480	1.8229840
B	0.2864210	-1.4439990	-0.8577890
H	0.6363350	-2.3916710	-1.4763920
B	1.2845180	0.9064840	0.4764690
H	2.2666540	1.4474050	0.8505540
B	-0.3422270	-1.4503030	0.7979630
H	-0.4891620	-2.4020190	1.4877360
B	-1.3988180	0.8839750	-0.5422280
H	-2.3411990	1.5119280	-0.8975420
B	0.2601680	1.4487480	-0.8581030
H	0.5928990	2.4024920	-1.4768570
B	1.3007560	-0.8829340	0.4766910
H	2.2925420	-1.4058840	0.8508770
B	-0.3684990	1.4440200	0.7975930
H	-0.5326560	2.3930670	1.4871630
C	1.1882450	0.0106580	-0.9577610
H	2.0587880	0.0184950	-1.6035270
H	-0.4464020	-0.0043120	-2.8645230

[12-CH_3-$CB_{11}H_{11}$]$^-$



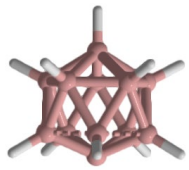
G(Solv) = -358.176603

0 imaginary frequencies

C	0.0116460	0.0102970	1.5289640
H	0.0195730	0.0176810	2.6133810
B	-0.0131690	-0.0118110	-1.7227680
B	1.4863870	-0.2898570	-0.7742210
H	2.5425580	-0.4982800	-1.2957490
B	-1.3806760	-0.6516050	-0.7495180
H	-2.3611910	-1.1121590	-1.2570420
B	-1.4816010	0.2897710	0.7487440
H	-2.4528710	0.4820680	1.4137540
B	0.1830470	-1.5127120	-0.7559730
H	0.3151920	-2.5872180	-1.2649040
B	1.3763460	0.6492970	0.7259810
H	2.2809320	1.0757210	1.3758740
B	-0.7249970	-1.3217450	0.7546570
H	-1.1988830	-2.1881170	1.4235650
B	-1.0450600	1.1024430	-0.7652360
H	-1.7883730	1.8873490	-1.2777560
B	0.7269890	1.3256670	-0.7792480
H	1.2420040	2.2669650	-1.3080360
B	1.0415200	-1.0992650	0.7396380
H	1.7267150	-1.8202650	1.3977860
B	-0.1828570	1.5074060	0.7309770
H	-0.3029940	2.4981570	1.3841030
C	-0.0252640	-0.0230550	-3.3331070
H	0.2816780	0.9455290	-3.7504430

H	0.6580800	-0.7800020	-3.7411590
H	-1.0243110	-0.2433210	-3.7331910

$[B_{12}H_{11}]^-$

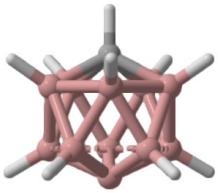


G(Solv) = -305.007125

0 imaginary frequencies

B	2.6491160	-0.4822180	0.0093440
B	4.2672350	-0.9660170	0.1554650
H	4.6735900	-2.0837610	0.2576340
B	2.0481050	0.8938520	0.7967700
H	0.9835760	1.0097730	1.3243130
B	2.8781870	2.1678470	-0.1487400
H	2.3979420	3.2577860	-0.2542070
B	3.3402630	-0.1810710	1.5283860
H	3.1323200	-0.7782230	2.5409050
B	5.0571980	0.3413350	-0.7786500
H	6.1033270	0.1512020	-1.3255890
B	3.5833960	1.5883640	1.4019300
H	3.5973460	2.2725190	2.3824940
B	2.1776250	0.7742610	-1.0275860
H	1.1986210	0.8105070	-1.7096210
B	3.5491150	-0.3755090	-1.4238180
H	3.4793550	-1.1015990	-2.3688000
B	4.9305600	0.4596360	1.0131470
H	5.8880490	0.3525060	1.7212190
B	3.7893100	1.3978900	-1.4966710
H	3.9472230	1.9481550	-2.5464090
B	4.6314090	1.8885650	-0.0063670
H	5.3990450	2.8059360	-0.0123570

***p*-[$CB_{11}H_{11}$]**

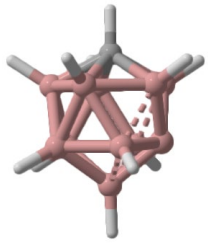


G(Solv) = -318.079101

0 imaginary frequencies

C	4.5213350	1.7566560	-0.0054410
H	5.2166780	2.5880530	-0.0108340
B	2.6419870	-0.4897080	0.0091460
B	4.2624850	-0.9773940	0.1560380
H	4.6895120	-2.0790920	0.2585640
B	2.0381270	0.8876600	0.7988170
H	0.9862340	1.0259850	1.3289480
B	2.8659300	2.1460870	-0.1483880
H	2.5255060	3.2766250	-0.2467810
B	3.3332570	-0.1902730	1.5321550
H	3.1422850	-0.7688110	2.5497370
B	5.0391240	0.3251460	-0.7759920
H	6.1091720	0.2732210	-1.2819280
B	3.5698410	1.5684940	1.3982930
H	3.6860650	2.3238100	2.3036490
B	2.1675370	0.7667830	-1.0300610
H	1.2016460	0.8248500	-1.7158640
B	3.5430270	-0.3849500	-1.4275400
H	3.4911990	-1.0929280	-2.3776960
B	4.9125000	0.4424890	1.0101850
H	5.9004270	0.4668710	1.6636730
B	3.7743410	1.3782160	-1.4921600
H	4.0230720	2.0100470	-2.4630480

***m*-[$CB_{11}H_{11}$]**

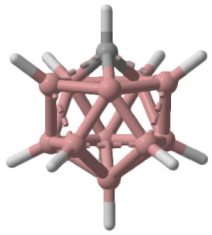


G(Solv) = -318.072561

0 imaginary frequencies

C	0.0031310	-0.0013800	1.4959290
H	-0.0030070	0.0063300	2.5800520
B	-0.0129590	0.0111720	-1.7854170
B	-0.2057330	1.5151170	-0.8217040
H	-0.3508620	2.5721240	-1.3424860
B	0.8893450	-0.9220470	-0.7064860
B	0.1926160	-1.5160640	0.7316620
H	0.3806310	-2.5208190	1.3281350
B	1.3903420	0.6972410	-0.8161350
H	2.3766550	1.1040440	-1.3360300
B	-1.0373940	1.0753400	0.6816020
H	-1.7300280	1.7935400	1.3223210
B	1.5092610	-0.2448830	0.7300720
H	2.5075460	-0.4673520	1.3255550
B	-0.7466780	-1.3659880	-0.8135560
H	-1.1885250	-2.3380260	-1.3317290
B	-1.5079930	0.2578240	-0.8201280
H	-2.5599020	0.4393690	-1.3398100
B	0.7174750	1.3510990	0.6997580
H	1.1850320	2.2247770	1.3492330
B	-1.3745990	-0.6687340	0.7022860
H	-2.2633500	-1.1045230	1.3533980
H	0.0215870	-0.0260990	-2.9711820

***o*-[$CB_{11}H_{11}$]**

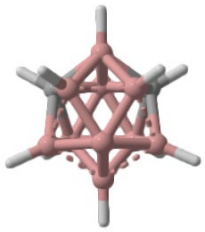


G(Solv) = -318.062332

0 imaginary frequencies

C	-0.0287140	0.0070950	1.5015270
H	0.0299920	-0.0017360	2.5842090
B	0.0020230	-0.0028540	-1.7511510
B	-0.2113630	1.5232090	-0.8151240
H	-0.3592820	2.5838010	-1.3279380
B	1.0642000	-1.1347930	-0.8400900
B	0.1920630	-1.5421830	0.7007900
H	0.3935250	-2.4873750	1.3875570
B	1.3878690	0.6997090	-0.8426520
H	2.3801930	1.1361910	-1.3228980
B	-1.0678130	1.1032530	0.7005520
H	-1.7494640	1.8172510	1.3555830
B	1.2619210	-0.2219300	0.5597020
B	-0.7202830	-1.3612120	-0.8111390
H	-1.2224920	-2.3085410	-1.3211070
B	-1.4751510	0.2591090	-0.8004100
H	-2.5244150	0.4435100	-1.3285150
B	0.7085600	1.3851080	0.6967210
H	1.2216420	2.2061170	1.3810600
B	-1.3805930	-0.6694250	0.7030150
H	-2.2651020	-1.1052830	1.3596520
H	0.0375380	-0.0107430	-2.9387750
H	1.8470070	-1.8857580	-1.3187540

***m*-[$C_2B_{10}H_{11}$]$^+$**

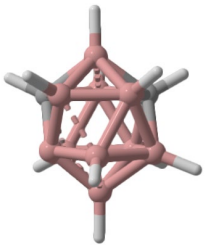


G(Solv) = -331.111762

0 imaginary frequencies

C	4.5399820	1.7979580	0.0035420
H	5.2516690	2.6177020	-0.0049610
B	2.4513770	-0.7244780	-0.0101480
B	2.2669610	0.8290760	0.6548270
B	2.8731610	2.1871050	-0.1690800
H	2.4794680	3.2973710	-0.2337630
B	3.3377410	-0.1942600	1.5145460
B	4.9969300	0.3558840	-0.7683170
H	6.0706390	0.1753220	-1.2214970
B	3.5967410	1.5932320	1.4167750
H	3.6457540	2.3130860	2.3504490
B	2.1673930	0.7512220	-1.0413810
H	1.1886670	0.8568870	-1.6983760
B	3.5152490	-0.3684120	-1.4063210
H	3.5719970	-1.1336860	-2.3027270
B	4.9058890	0.4662920	1.0131770
H	5.9038450	0.3554450	1.6290440
B	3.7711920	1.3977110	-1.5024910
H	4.0287780	2.0151280	-2.4745410
C	4.1623780	-0.8074890	0.1458040
H	4.6127200	-1.7911780	0.2355440
H	1.7582600	-1.6780220	0.0369420
H	3.1954550	-0.7945700	2.5203830

***o-1*-$[C_2B_{10}H_{11}]^+$**

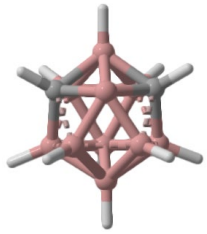


G(Solv) = -331.107786

0 imaginary frequencies

C	4.5247200	1.7632610	-0.0056760
H	5.2210120	2.5965220	-0.0112150
B	2.6297590	-0.4732630	0.0073320
B	2.0265700	0.8854950	0.8114050
H	0.9775950	1.0133780	1.3400680
B	2.8657410	2.1441410	-0.1482170
H	2.5025040	3.2632450	-0.2475820
B	3.3258600	-0.1720730	1.5410960
H	3.2152940	-0.8715110	2.4868680
B	5.0374570	0.3484280	-0.7866970
H	6.1119270	0.1677520	-1.2341760
B	3.5691390	1.5800780	1.4099620
H	3.6958080	2.3212650	2.3195090
B	2.1578980	0.7634000	-1.0436490
H	1.1946840	0.8115640	-1.7263540
B	3.5368970	-0.3682250	-1.4396640
H	3.5536830	-1.1860140	-2.2922260
B	4.9097430	0.4671370	1.0173060
H	5.9090990	0.3562740	1.6307910
B	3.7755400	1.3882280	-1.5053010
H	4.0354250	2.0056280	-2.4768760
C	4.2058230	-0.8066230	0.1408580
H	4.6021600	-1.8133660	0.2351680

***o*$_2$-[$C_2B_{10}H_{11}$]$^+$**

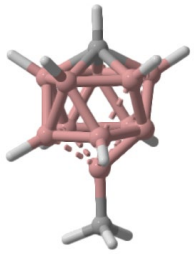


G(Solv) = -331.088569

0 imaginary frequencies

C	4.5517650	1.8028860	0.0208040
H	5.2873740	2.6009790	-0.0168490
B	2.4449990	-0.6981390	0.0143960
B	2.0782220	0.8631480	0.7637120
B	2.8693180	2.1595220	-0.1464040
H	2.5548170	3.2911700	-0.2658120
B	3.3059940	-0.1855980	1.5156470
B	4.7870520	0.3804480	-0.6700370
B	3.5695310	1.5877210	1.4157810
H	3.6971280	2.3250830	2.3281840
B	2.1752780	0.7483540	-1.0224860
H	1.2341620	0.8486820	-1.7315430
B	3.4913100	-0.3887130	-1.4371080
H	3.6089040	-1.1674420	-2.3176120
B	4.9005130	0.4608670	1.0576750
H	5.9499250	0.3347880	1.5864240
B	3.7575020	1.4034630	-1.5376550
H	4.0814640	2.0126930	-2.4965240
C	4.1610230	-0.8279640	0.1687420
H	4.6333500	-1.8039420	0.2308600
H	1.8144650	-1.6962500	0.0144530
H	3.2299930	-0.8239070	2.5052260
H	1.0359880	1.0485130	1.2988760

***m*-[12-CH_3-$CB_{11}H_{10}$]**



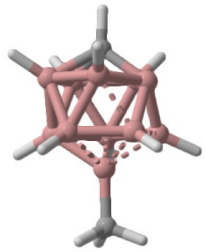
G(Solv) = -357.363146

0 imaginary frequencies

C	0.0054360	0.0055550	1.5496170
H	0.0194320	0.0167860	2.6333940
B	0.0085490	-0.0055130	-1.7681860
B	1.5007860	-0.2591610	-0.7743160
H	2.5515820	-0.4328000	-1.3014780
B	-1.1367380	-0.5807110	-0.6516980
B	-1.4984750	0.2654810	0.7844500
H	-2.5138590	0.3831860	1.3814890
B	0.2566410	-1.5414460	-0.7534500
H	0.3533260	-2.6066270	-1.2694240
B	1.3376940	0.6763810	0.7216260
H	2.2331930	1.1317150	1.3518270
B	-0.6738370	-1.3645460	0.7907510
H	-1.1824470	-2.2486260	1.3916160
B	-1.0848300	1.1104380	-0.7636420
H	-1.8842110	1.8160870	-1.2863690
B	0.6852600	1.3529370	-0.7802150
H	1.1693800	2.2988350	-1.3122900
B	1.0836970	-1.0803480	0.7561160
H	1.7848540	-1.7802000	1.4062660
B	-0.2281480	1.5127380	0.7463310
H	-0.3785420	2.4960900	1.3901540
C	-0.0308780	-0.0310290	-3.3632860
H	0.8543880	0.4627490	-3.7779040
H	-0.0464230	-1.0541870	-3.7559640

H	-0.9092760	0.4902450	-3.7609280

***o*-[12-CH_3-$CB_{11}H_{10}$]**



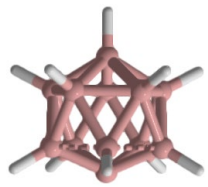
G(Solv) = - 357.352118

0 imaginary frequencies

C	0.0371550	0.0036810	1.5495460
H	-0.0204550	0.0218650	2.6320020
B	-0.0024230	-0.0132610	-1.7361850
B	1.4698950	-0.2692850	-0.7547750
H	2.5161850	-0.4608410	-1.2876930
B	-1.3868450	-0.7032710	-0.7834290
B	-1.2599290	0.2286740	0.6088860
B	0.2050010	-1.5280650	-0.7549920
H	0.3500760	-2.5924390	-1.2629970
B	1.3853170	0.6699150	0.7400160
H	2.2746350	1.1094120	1.3879470
B	-0.7084960	-1.3773550	0.7591230
H	-1.2189910	-2.1931080	1.4519080
B	-1.0596200	1.1269480	-0.7969610
H	-1.8430620	1.8734950	-1.2832250
B	0.7198130	1.3493560	-0.7758200
H	1.2228110	2.2902350	-1.2989930
B	1.0687210	-1.0998880	0.7528130
H	1.7521740	-1.8113610	1.4089840
B	-0.1855290	1.5460500	0.7376610
H	-0.3803800	2.4978910	1.4174090
C	-0.0388020	-0.0186920	-3.3335210
H	0.5529810	0.8060100	-3.7479300

H	0.3725740	-0.9520770	-3.7358540
H	-1.0561150	0.0824020	-3.7289970
H	-2.3815240	-1.1385250	-1.2615780

$[B_{12}H_{11}]^{3-}$

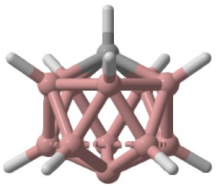


G(Solv) = -305.21842

0 imaginary frequencies

B	2.3532070	-0.8359890	0.0115190
B	4.1915910	-0.9592780	0.1497920
H	4.7786420	-2.0339390	0.2621270
B	2.0615140	0.8260310	0.7653380
H	1.0189600	1.1177890	1.3484760
B	2.8878800	2.1540380	-0.1472530
H	2.4029390	3.2726050	-0.2551940
B	3.3014250	-0.2060580	1.4671780
H	3.2069870	-0.7046220	2.5872630
B	5.0442230	0.3472910	-0.7702890
H	6.1171960	0.1598460	-1.3285270
B	3.5860950	1.5806540	1.3871850
H	3.6054840	2.2846840	2.3881980
B	2.1855750	0.7106600	-0.9857660
H	1.2379010	0.9143430	-1.7424230
B	3.5020840	-0.3926430	-1.3663420
H	3.5609570	-1.0338790	-2.4139340
B	4.9186500	0.4640430	1.0022470
H	5.9009210	0.3609560	1.7251270
B	3.7891410	1.3919320	-1.4805260
H	3.9551940	1.9596360	-2.5520610
B	4.6553470	1.9174660	-0.0065620
H	5.4400000	2.8561730	-0.0127840

***p*-[$CB_{11}H_{11}$]$^{2-}$**

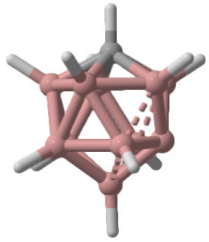


G(Solv) = -318.323024

0 imaginary frequencies

C	4.5383610	1.7771220	-0.0056850
H	5.2354420	2.6111960	-0.0110080
B	2.3630540	-0.8244560	0.0113180
B	4.1888140	-0.9674220	0.1503200
H	4.7827660	-2.0205990	0.2616920
B	2.0543500	0.8213570	0.7665920
H	1.0308080	1.1245650	1.3450550
B	2.8736420	2.1303310	-0.1466670
H	2.5449070	3.2868250	-0.2461440
B	3.2964390	-0.2132210	1.4698750
H	3.2150180	-0.6939460	2.5818420
B	5.0233340	0.3277160	-0.7681480
H	6.1158220	0.2925030	-1.2787260
B	3.5689910	1.5578930	1.3831560
H	3.7000810	2.3361840	2.2956300
B	2.1791890	0.7063270	-0.9872580
H	1.2496930	0.9216420	-1.7382010
B	3.4976360	-0.3991950	-1.3682280
H	3.5678750	-1.0217480	-2.4083980
B	4.8983900	0.4446000	0.9995950
H	5.9081640	0.4865110	1.6583830
B	3.7716830	1.3699170	-1.4764830
H	4.0368270	2.0237390	-2.4550400

***m*-[$CB_{11}H_{11}$]$^{2-}$**

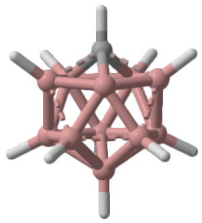


G(Solv) = -318.326146

0 imaginary frequencies

C	-0.0177560	0.0202470	1.4855550
H	-0.0192930	0.0230600	2.5762000
B	0.0226890	-0.0255940	-1.7364860
B	-0.2114550	1.5021380	-0.8120280
H	-0.3617260	2.5823350	-1.3312490
B	1.1535060	-1.1959640	-0.8629570
B	0.2499170	-1.5081160	0.6570040
H	0.3239290	-2.4564800	1.4061420
B	1.3772970	0.6237270	-0.8225450
H	2.3214800	1.2047210	-1.3157330
B	-1.0618200	1.1006490	0.6917840
H	-1.7650050	1.8299940	1.3433010
B	1.4993090	-0.3019010	0.6553990
H	2.4452820	-0.4083350	1.4036700
B	-0.6727610	-1.3557380	-0.8201960
H	-1.2871430	-2.2794920	-1.3114930
B	-1.4948440	0.2629710	-0.8104610
H	-2.5696970	0.4505880	-1.3286220
B	0.7082010	1.3252200	0.6901450
H	1.1846910	2.2100430	1.3538810
B	-1.3484140	-0.6603320	0.6928360
H	-2.2486080	-1.1046130	1.3582350
H	-0.0351900	0.0329350	-2.9470410

***o*-$[CB_{11}H_{11}]^{2-}$**

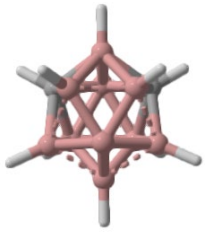


G(Solv) = -318.329724

0 imaginary frequencies

C	0.0012100	0.0018180	1.4683120
H	-0.0472680	0.0118580	2.5556880
B	-0.0128820	-0.0002730	-1.7504790
B	-0.2059660	1.5003020	-0.8074840
H	-0.3469230	2.5841310	-1.3206990
B	1.0681510	-1.0795020	-0.7935230
B	0.2720040	-1.4665420	0.7227470
H	0.2765250	-2.5038020	1.3434860
B	1.3726230	0.6464420	-0.7959570
H	2.3064510	1.1573410	-1.3710250
B	-1.0578740	1.0871510	0.6786150
H	-1.7543220	1.8149640	1.3379200
B	1.6736220	-0.2942440	0.7715370
B	-0.7073580	-1.3415330	-0.8035130
H	-1.2109650	-2.3130510	-1.3139140
B	-1.5101440	0.2652080	-0.8234830
H	-2.5859680	0.4543210	-1.3393550
B	0.7578520	1.2868260	0.7188320
H	1.1174570	2.2614800	1.3368890
B	-1.3657770	-0.6577430	0.6810780
H	-2.2688940	-1.1015470	1.3420190
H	-0.0202790	-0.0005220	-2.9590060
H	1.7705860	-1.8805610	-1.3668720

***m*-[$C_2B_{10}H_{11}$]$^-$**

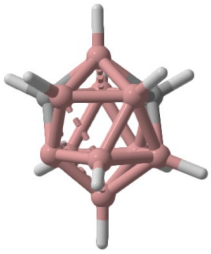


G(Solv) = -331.406375

0 imaginary frequencies

C	4.5872880	1.7627170	-0.0417630
H	5.2967980	2.5848660	-0.0462060
B	2.4824490	-0.6647130	0.0364730
B	1.9698060	0.8823490	0.8231810
B	2.8874420	2.1263840	-0.1157270
H	2.5992570	3.2822860	-0.2421650
B	3.2065110	-0.1332920	1.5224240
B	5.0339500	0.3480680	-0.8016220
H	6.1132070	0.1654700	-1.2734170
B	3.4530230	1.5736300	1.4294650
H	3.7222470	2.3597430	2.2916350
B	2.1313080	0.7615980	-0.9448400
H	1.2785590	0.8393990	-1.7857310
B	3.5163110	-0.3573870	-1.3796260
H	3.5498440	-1.1302150	-2.2875760
B	4.8401780	0.4758260	1.0042700
H	5.8395340	0.3655950	1.6435840
B	3.7693320	1.3893390	-1.4749690
H	4.0070760	2.0185360	-2.4600600
C	4.2154050	-0.7908730	0.0991340
H	4.6619500	-1.7762760	0.1939410
H	1.8761950	-1.6978740	0.0301990
H	3.2545750	-0.8678540	2.4668260

***o-*[$C_2B_{10}H_{11}$]$^-$**

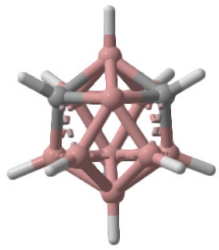


G(Solv) = -331.410736

0 imaginary frequencies

C	4.5429100	1.7943280	-0.0063940
H	5.2471860	2.6201030	-0.0108140
B	2.3325770	-0.8196250	0.0091250
B	2.0603760	0.8472330	0.7599290
H	1.0362270	1.0903420	1.3319870
B	2.8894030	2.1479550	-0.1467900
H	2.5656830	3.2936060	-0.2451650
B	3.2740380	-0.2387600	1.4429510
H	3.3029060	-0.7858980	2.5085080
B	5.0151960	0.3685100	-0.7740690
H	6.1061010	0.2033020	-1.2217170
B	3.5599900	1.5549130	1.3953850
H	3.6815370	2.3026970	2.3179520
B	2.1838480	0.7324290	-0.9836050
H	1.2522960	0.8892480	-1.7203610
B	3.4712100	-0.4220810	-1.3417120
H	3.6441510	-1.1029440	-2.3123020
B	4.8896830	0.4851650	0.9991350
H	5.9053850	0.3898900	1.6131570
B	3.7641800	1.3651780	-1.4890180
H	4.0208260	1.9874790	-2.4749050
C	4.1800440	-0.7741270	0.1369210
H	4.6585860	-1.7442240	0.2345320

***O*$_2$-[$C_2B_{10}H_{11}$]$^-$**

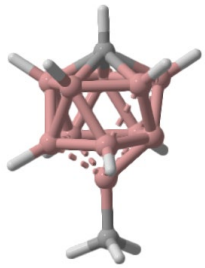


G(Solv) = -331.420877

0 imaginary frequencies

C	4.5200710	1.7736550	-0.0080950
H	5.2101650	2.6108160	0.0061250
B	2.4391340	-0.6724210	0.0208200
B	2.0409050	0.8707080	0.7827460
B	2.8571700	2.1378900	-0.1380660
H	2.5108150	3.2749380	-0.2401830
B	3.3093940	-0.1659020	1.4824320
B	5.2088700	0.3057280	-0.8705970
B	3.5667710	1.5650330	1.3845730
H	3.7034020	2.3031120	2.3111950
B	2.1995480	0.7456390	-1.0118270
H	1.2614920	0.8434070	-1.7457130
B	3.5666810	-0.3656580	-1.3684480
H	3.5214590	-1.1445720	-2.2723710
B	4.9421850	0.4462200	0.9114930
H	5.8370890	0.3577890	1.6965530
B	3.8242260	1.3657530	-1.4663880
H	3.9927550	2.0197420	-2.4511190
C	4.1380430	-0.7944950	0.1371120
H	4.5554530	-1.7890710	0.2548560
H	1.7756720	-1.6637600	0.0389060
H	3.2407820	-0.8049120	2.4869250
H	0.9979950	1.0567260	1.3378200

***m*-[12-CH_3-$CB_{11}H_{10}$]$^{2-}$**



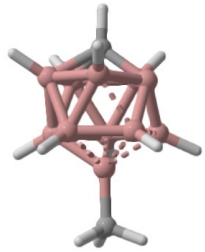
G(Solv) = -357.613298

0 imaginary frequencies

C	0.0352810	0.0220930	1.5330060
H	0.0477510	0.0314130	2.6226630
B	-0.0414850	-0.0238400	-1.7063900
B	1.4788470	-0.3023030	-0.7720520
H	2.5427070	-0.5203950	-1.3006430
B	-1.5165800	-0.6990330	-0.8004300
B	-1.5015210	0.2677660	0.7140640
H	-2.4172100	0.5177430	1.4646260
B	0.1231150	-1.5098840	-0.7661790
H	0.3576910	-2.5942610	-1.2570720
B	1.3911150	0.6427680	0.7241590
H	2.3160060	1.0701030	1.3656470
B	-0.7766050	-1.3086800	0.7193580
H	-1.1849900	-2.1624360	1.4734420
B	-1.0645770	1.0736710	-0.7746900
H	-1.7337490	1.9549650	-1.2720850
B	0.7346560	1.3165050	-0.7773580
H	1.2630610	2.2631310	-1.3097610
B	1.0182070	-1.1022000	0.7376140
H	1.7000830	-1.8407190	1.4006000
B	-0.1762320	1.4957680	0.7290730
H	-0.2945040	2.4977460	1.3863190
C	-0.0041140	-0.0120290	-3.3317910
H	0.0900020	1.0032070	-3.7516320

H	0.8285620	-0.6042030	-3.7462920
H	-0.9389660	-0.4428680	-3.7197140

***o*-[12-CH_3-$CB_{11}H_{10}$]$^{2-}$**



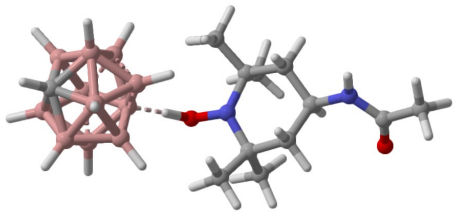
G(Solv) = -231.07

0 imaginary frequencies

C	0.0093760	0.0095840	1.5192830
H	0.0645780	0.0078110	2.6060870
B	0.0006000	-0.0136260	-1.7155740
B	1.4986880	-0.2825000	-0.7820150
H	2.5662670	-0.4847090	-1.3108340
B	-1.3811550	-0.6409210	-0.7291100
B	-1.6660920	0.3130420	0.8288070
B	0.1868310	-1.5056430	-0.7455240
H	0.3185400	-2.5898420	-1.2613290
B	1.3748580	0.6528020	0.7164780
H	2.2873830	1.0929430	1.3663330
B	-0.7618990	-1.2760680	0.7839210
H	-1.1263150	-2.2420500	1.4120460
B	-1.0631680	1.0793020	-0.7447560
H	-1.7626080	1.8781910	-1.3258640
B	0.7101720	1.3275430	-0.7666880
H	1.2160550	2.2852420	-1.3013570
B	1.0536440	-1.0899820	0.7294530
H	1.7510510	-1.8175140	1.3875240
B	-0.2558780	1.4746640	0.7624150
H	-0.2501120	2.5168010	1.3742750
C	-0.0119990	-0.0240350	-3.3378610

H	0.4578550	0.8766290	-3.7634840
H	0.5249160	-0.8900080	-3.7566720
H	-1.0375100	-0.0649190	-3.7367690
H	-2.3189140	-1.1449740	-1.3054380

TS-1



G(Solv) = -1010.027662

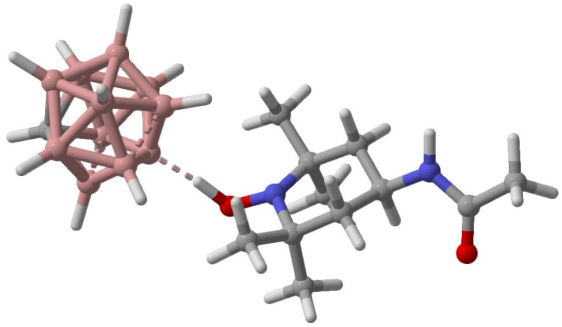
1 imaginary frequencies: -398.02

O	-0.4162760	-0.7333940	1.4444140
N	0.6793510	-0.4890000	0.7636460
H	-1.3499340	-0.4007520	0.8954790
C	1.4240460	0.7282330	1.2001210
C	2.5210610	1.0340640	0.1676370
H	2.0629800	1.4857240	-0.7218510
H	3.2056210	1.7690720	0.6001840
C	0.4422230	1.9048140	1.2488400
H	-0.0376190	2.0541460	0.2789970
H	0.9952850	2.8137030	1.5049810
H	-0.3357650	1.7558420	2.0009130
C	3.3113090	-0.2018140	-0.2702680
H	3.8147490	-0.6370710	0.5964250
C	-5.3193210	0.6247030	-1.0180480
H	-6.2577790	0.8720610	-1.4994860
C	2.0190790	0.4907170	2.6029310
H	1.2321750	0.1819230	3.2967480
H	2.4639450	1.4200580	2.9721090
H	2.8002010	-0.2730600	2.5976830
B	-2.6813990	-0.0684530	0.3307000
B	-2.9762160	1.6050010	-0.0004030
H	-2.2543870	2.5127190	0.2460790
B	-4.0559090	-1.0064790	0.7896060
H	-4.0562880	-1.8935300	1.5793720
B	-5.0369250	-1.0206670	-0.6899720

H	-5.8471360	-1.8327230	-0.9875260
B	-3.8661850	0.7217750	1.3068630
H	-3.7374990	1.0172500	2.4500480
B	-3.9617980	1.5482590	-1.4679230
H	-4.0712490	2.4118580	-2.2723890
B	-5.3941300	0.1432730	0.6162800
H	-6.4417680	0.0930000	1.1677600
C	1.2487760	-1.6760200	0.0639550
C	2.3533660	-1.2071150	-0.8985950
H	1.8814090	-0.7463660	-1.7777860
H	2.8982270	-2.0912570	-1.2466900
C	0.1328670	-2.3461920	-0.7469780
H	-0.3332570	-1.6369490	-1.4354110
H	0.5642590	-3.1654540	-1.3306620
H	-0.6426790	-2.7607160	-0.1008570
C	1.7963360	-2.6710970	1.1074790
H	1.0170750	-2.9123210	1.8354780
H	2.1017070	-3.5962910	0.6077750
H	2.6618030	-2.2737720	1.6433660
B	-3.2820460	-1.1936150	-0.8371260
H	-2.7670740	-2.2108290	-1.1638730
B	-2.6017170	0.4183340	-1.3209310
H	-1.6073760	0.5065510	-1.9702810
B	-4.7362530	1.7315240	0.1332620
H	-5.3486710	2.7172300	0.3733110
B	-4.1488580	-0.1550010	-1.9779640
H	-4.3804190	-0.3999930	-3.1143140
N	4.3719520	0.1402540	-1.2062290
H	4.1452440	0.2000100	-2.1892070
C	5.5655580	0.6431440	-0.7615710
O	5.8234510	0.7412740	0.4323830
C	6.5496580	1.0554050	-1.8426130
H	7.5001800	0.5451840	-1.6633520
H	6.2088340	0.8292440	-2.8576220

H	6.7361200	2.1308780	-1.7609920

TS-2



G(Solv) = -1010.023987

1 imaginary frequencies: -231.07

O	0.4137660	0.7829070	1.4304870
N	-0.6605590	0.4926590	0.7012560
H	1.3281860	0.4724560	0.9128990
C	-1.4061670	-0.7030640	1.1881170
C	-2.5125470	-1.0307500	0.1723850
H	-2.0604740	-1.4895480	-0.7165480
H	-3.1863450	-1.7656490	0.6219920
C	-0.4284960	-1.8827120	1.2556670
H	0.0581610	-2.0416290	0.2903180
H	-0.9828290	-2.7885250	1.5202470
H	0.3440040	-1.7248410	2.0112870
C	-3.3167220	0.1941360	-0.2702510
H	-3.8175410	0.6322040	0.5963010
C	-1.9883120	-0.4390220	2.5918610
H	-1.1984570	-0.1002010	3.2680010
H	-2.4123290	-1.3659310	2.9906570
H	-2.7829490	0.3102260	2.5798470
B	2.7171460	0.1222730	0.3560710
B	3.0475650	-1.5704820	0.2645840
H	2.3743590	-2.4316680	0.7235380
B	4.0872980	1.1412540	0.5717430

H	4.2136850	2.0947220	1.2629760
B	4.9788800	0.9612250	-0.9582890
H	5.7433190	1.8091430	-1.2752580
B	3.9862640	-0.4903450	1.3445660
H	4.0477920	-0.5688540	2.5247280
B	3.9391240	-1.7232680	-1.2732860
H	3.9034460	-2.7235540	-1.9123560
C	-1.2599660	1.6819240	0.0346790
C	-2.3729040	1.2032870	-0.9137650
H	-1.9073210	0.7417970	-1.7958830
H	-2.9278800	2.0822470	-1.2592070
C	-0.1690330	2.3711420	-0.7944590
H	0.2894440	1.6692350	-1.4952020
H	-0.6182190	3.1906680	-1.3642710
H	0.6156250	2.7892390	-0.1608490
C	-1.8034070	2.6687740	1.0882410
H	-1.0203040	2.9087790	1.8124400
H	-2.1152860	3.5963190	0.5969350
H	-2.6641200	2.2671620	1.6279790
B	3.2127560	1.0820100	-0.9913720
H	2.6528950	2.0454160	-1.3966170
B	2.5481250	-0.5976490	-1.1871590
H	1.5067290	-0.7916500	-1.7328370
B	4.8163750	-1.6471800	0.2775960
H	5.4751930	-2.5017600	0.7669090
B	4.0403220	-0.0982250	-2.0433700
H	4.0758340	0.0408450	-3.2222880
N	-4.3828360	-0.1652400	-1.1938200
H	-4.1599560	-0.2462920	-2.1762110
C	-5.5713220	-0.6660830	-0.7337090
O	-5.8241300	-0.7427510	0.4629640
C	-6.5576170	-1.1059920	-1.8019650
H	-7.5145810	-0.6088970	-1.6204950
H	-6.2284180	-0.8863440	-2.8222280

H	-6.7262580	-2.1833880	-1.7067580
B	5.4092080	-0.7488230	-1.1292020
C	5.3260650	-0.0157750	0.4064120
H	6.4720340	-1.0271140	-1.5762130
H	6.2564890	0.1730730	0.9278180

IV. X-ray Crystallography

The structures were solved with the dual-space algorithm using *SHELXT*[18] and were refined by full-matrix least-squares methods on *F*2 with *SHELXL-2014*[19] using the GUI *OLEX2*[20]. The graphical output was produced with the help of the program *Mercury*[21]. The hydrogen atoms of the carboranes bound to a boron atom were refined with the AFIX 153 command, fixing the B-H distance to 1.10 Å. Hydrogen atoms of the carboranes bound to a carbon atom were refined with the AFIX 154 command, allowing the C-H distance to vary. If the distances became too short, they were fixed at a minimal distance of 0.96 Å.

Crystal structure of 2 (CCDC: 2407119)

20 mg of the Protonated salt of the product was dissolved in acetone (0.6 mL) and filtered into an NMR tube (5 mm diameter, 18 cm length) via slow evaporation, Crystals suitable for X-ray diffraction grew within 1 week at 25 °C.

Table 1 Crystal data and structure refinement for 231024_OR.

Identification code	231024_OR
Empirical formula	$C_{12}H_{33}B_{11}N_2O_2$
Formula weight	356.31
Temperature/K	170.00
Crystal system	orthorhombic
Space group	Pnma
a/Å	10.0719(4)
b/Å	10.9585(5)
c/Å	18.5142(8)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	2043.47(15)
Z	4
ρ_{calc}g/cm^3	1.158
μ/mm^{-1}	0.314
F(000)	760.0
Crystal size/mm^3	0.19 × 0.04 × 0.02
Radiation	GaKα (λ = 1.34139)
2Θ range for data collection/°	8.156 to 121.23
Index ranges	$-13 \leq h \leq 13$, $-14 \leq k \leq 14$, $-23 \leq l \leq 24$
Reflections collected	26841

Independent reflections	2466 [R_{int} = 0.0845, R_{sigma} = 0.0481]
Data/restraints/parameters	2466/0/142
Goodness-of-fit on F^2	1.053
Final R indexes [I>=2σ (I)]	R_1 = 0.0562, wR_2 = 0.1574
Final R indexes [all data]	R_1 = 0.0746, wR_2 = 0.1719
Largest diff. peak/hole / e $Å^{-3}$	0.37/-0.23

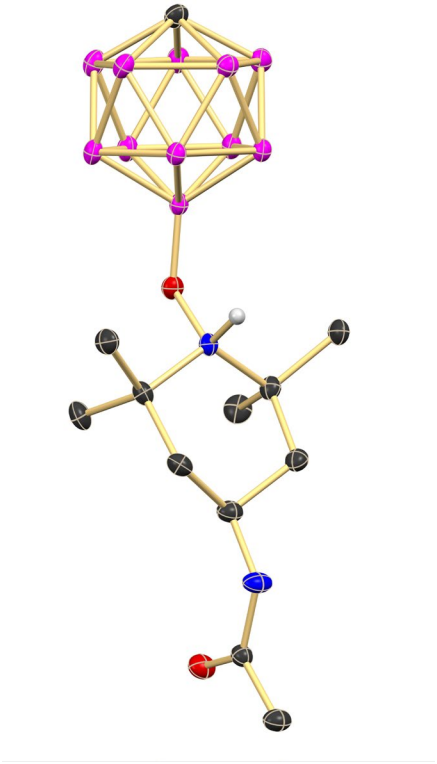


Figure S4. ORTEP representation of 2. Hydrogen atoms omitted for clarity; 30% displacement ellipsoids.

Crystal structure of 3b (CCDC: 2407120)

Table 2 Crystal data and structure refinement for 3.

Identification code	241017_OR_CH_3_TEMPO
Empirical formula	$C_{21}H_{54}B_{11}N_3O_2$
Formula weight	499.58
Temperature/K	170.00
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	13.1967(5)
b/Å	22.4636(8)
c/Å	20.8837(8)
α/°	90
β/°	92.836(2)
γ/°	90
Volume/Å^3	6183.3(4)
Z	8
ρ_{calc}g/cm^3	1.073
μ/mm^{-1}	0.290
F(000)	2176.0
Crystal size/mm^3	0.13 × 0.09 × 0.07
Radiation	GaKα (λ = 1.34139)
2Θ range for data collection/°	5.834 to 121.266
Index ranges	$-17 \leq h \leq 17$, $-29 \leq k \leq 23$, $-20 \leq l \leq 27$
Reflections collected	67077
Independent reflections	14162 [R_{int} = 0.0425, R_{sigma} = 0.0383]
Data/restraints/parameters	14162/0/687
Goodness-of-fit on F^2	1.037
Final R indexes [I>=2σ (I)]	R_1 = 0.0457, wR_2 = 0.1216
Final R indexes [all data]	R_1 = 0.0579, wR_2 = 0.1305
Largest diff. peak/hole / e Å^{-3}	0.23/-0.23

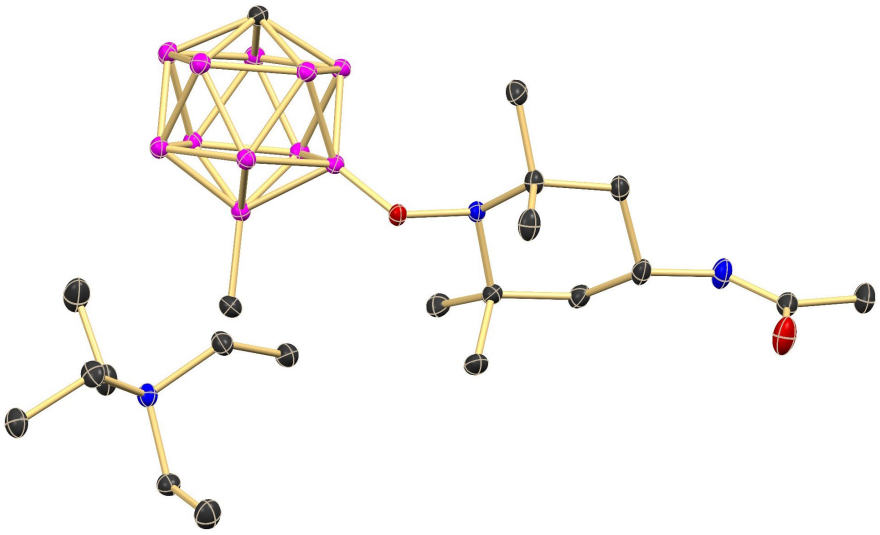


Figure S5. ORTEP representation of 3. Hydrogen atoms omitted for clarity; 30% displacement ellipsoids.

Crystal structure of 4b (CCDC: 2407121)

Table 3 Crystal data and structure refinement for 4b.

Identification code	240103_OR_COOH_0m
Empirical formula	$C_{24}H_{55}B_{11}N_2O_7$
Formula weight	602.61
Temperature/K	170.00
Crystal system	triclinic
Space group	P-1
a/Å	10.4232(6)
b/Å	11.4832(8)
c/Å	14.7581(9)
α/°	93.391(4)
β/°	94.642(4)
γ/°	91.388(4)
Volume/Å^3	1756.80(19)
Z	2
ρ_{calc}g/cm^3	1.139
μ/mm^{-1}	0.372
F(000)	648.0
Crystal size/mm^3	0.05 × 0.03 × 0.02
Radiation	GaKα (λ = 1.34139)
2Θ range for data collection/°	5.236 to 114.226
Index ranges	-6 ≤ h ≤ 13, -14 ≤ k ≤ 14, -18 ≤ l ≤ 18
Reflections collected	19898
Independent reflections	7117 [R_{int} = 0.1010, R_{sigma} = 0.1425]
Data/restraints/parameters	7117/0/409
Goodness-of-fit on F^2	0.940
Final R indexes [I>=2σ (I)]	R_1 = 0.0781, wR_2 = 0.1772
Final R indexes [all data]	R_1 = 0.1875, wR_2 = 0.2293
Largest diff. peak/hole / e Å^{-3}	0.24/-0.31

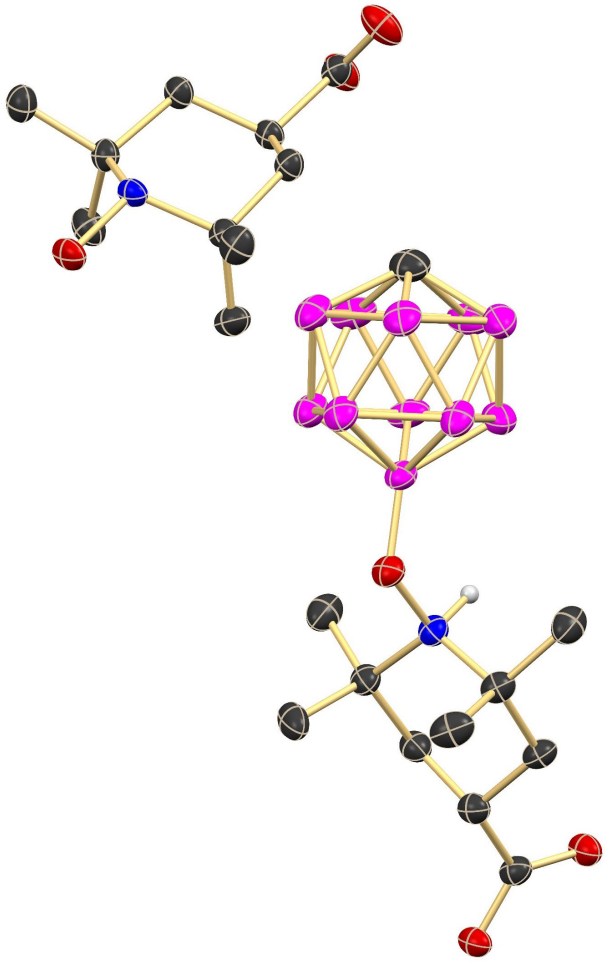


Figure S6. ORTEP representation of 4b. Hydrogen atoms omitted for clarity; 30% displacement ellipsoids.

Crystal structure of 4c (CCDC: 2407122)

Table 4 Crystal data and structure refinement for 240322_OR_OCH$_3$.

Identification code	240322_OR_OCH$_3$
Empirical formula	$C_{21}H_{53}B_{11}Cl_6N_2O_2$
Formula weight	697.26
Temperature/K	170.00
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	23.2378(9)
b/Å	9.4437(4)
c/Å	17.9211(7)
α/°	90
β/°	109.4290(10)
γ/°	90
Volume/Å^3	3708.8(3)
Z	4
ρ_{calc}g/cm^3	1.249
μ/mm^{-1}	2.923
F(000)	1464.0
Crystal size/mm^3	0.09 × 0.05 × 0.03
Radiation	GaKα (λ = 1.34139)
2Θ range for data collection/°	7.018 to 121.278
Index ranges	$-30 \leq h \leq 30$, $-11 \leq k \leq 12$, $-23 \leq l \leq 23$
Reflections collected	40697
Independent reflections	8510 [R_{int} = 0.0417, R_{sigma} = 0.0357]
Data/restraints/parameters	8510/0/388
Goodness-of-fit on F^2	1.053
Final R indexes [I>=2σ (I)]	R_1 = 0.0445, wR_2 = 0.1182
Final R indexes [all data]	R_1 = 0.0545, wR_2 = 0.1248
Largest diff. peak/hole / e Å^{-3}	0.69/-0.70

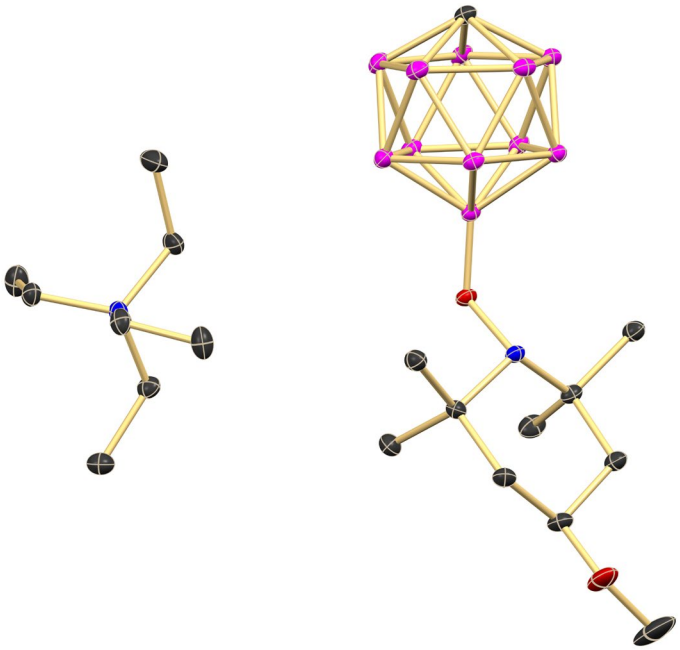


Figure S7. ORTEP representation of 4c. Hydrogen atoms omitted for clarity; 30% displacement ellipsoids.

V References

[1] C. A. Reed, *Acc. Chem. Res.* **2010**, *43*, 121-128.

[2] T. Jeljnek, J. Plesek, S. Hermanek, B. Stibr, *Collection Czechoslovak Chem. Commun.* **1986**, *51*, 819-829.

[3] B. r. Gruner, Z. k. Janousˇek, B. T. King, J. N. Woodford, C. H. Wang, V. c. Vsˇetecˇka, J. Michl, *J. Am. Chem. Soc.* **1999**, *121*, 3122-3126.

[4] B. T. King, S. Ko¨rbe, P. J. Schreiber, J. Clayton, A. Neˇmcova´, Z. Havlas, K. Vyakaranam, M. G. Fete, I. Zharov, J. Ceremuga, J. Michl, *J.Am.Chem.Soc.* **2007**, *129*, 12960-12980.

[5] D. S. Wilbur, D. K. Hamlin, R. R. Srivastava, M.-K. Chyan, *Nuclear Medicine and Biology* **2004**, *31*, 523-530.

[6] D. A. Singleton, A. A. Thomas, *J. Am. Chem. Soc* **1995**, *117*, 9357-9358.

[7] G. W. T. M. J. Frisch, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, G. Scalmani, V. Barone, B. Mennucci, G. A. Petersson, H. Nakatsuji, M. Caricato, X. Li, H. P. Hratchian, A. F. Izmaylov, J. Bloino, G. Zheng, J. L. Sonnenberg, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, T. Vreven, J. A. Montgomery, Jr., J. E. Peralta, F. Ogliaro, M. Bearpark, J. J. Heyd, E. Brothers, K. N. Kudin, V. N. Staroverov, T. Keith, R. Kobayashi, J. Normand, K. Raghavachari, A. Rendell, J. C. Burant, S. S. Iyengar, J. Tomasi, M. Cossi, N. Rega, J. M. Millam, M. Klene, J. E. Knox, J. B. Cross, V. Bakken, C. Adamo, J. Jaramillo, R. Gomperts, R. E. Stratmann, O. Yazyev, A. J. Austin, R. Cammi, C. Pomelli, J. W. Ochterski, R. L. Martin, K. Morokuma, V. G. Zakrzewski, G. A. Voth, P. Salvador, J. J. Dannenberg, S. Dapprich, A. D. Daniels, O. Farkas, J. B. Foresman, J. V. Ortiz, J. Cioslowski, and D. J. Fox, Revision D.01 ed., Gaussian, Inc., Wallingford, CT, **2013**.

[8] aA. D. Becke, *J. Chem. Phys.* **1993**, *98*, 5648-5652; bC. Lee, W. Yang, R. G. Parr, *Phys. Rev. B* **1988**, *37*, 785-789.

[9] S. Grimme, S. Ehrlich, L. Goerigk, *J. Comput. Chem.* **2011**, *32*, 1456-1465.

[10] aR. Ditchfield, W. J. Hehre, J. A. Pople, *J. Chem. Phys.* **1971**, *54*, 724-728; bW. J. Hehre, *J. Chem. Phys.* **1972**, *56*, 2257; cT. Clark, J. Chandrasekhar, G. W. Spitznagel, P. V. R. Schleyer, *J. Comput. Chem.* **1983**, *4*, 294-301; dM. J. Frisch, J. A. Pople, J. S. Binkley, *J. Chem. Phys.* **1984**, *80*, 3265.

[11] K. Fukui, *J. Phys. Chem.* **1970**, *74*, 4161-4163.

[12] Y. Zhao, D. G. Truhlar, *Theoretical Chemistry Accounts* **2008**, *120*, 215-241.

[13] S. Grimme, J. Antony, S. Ehrlich, H. Krieg, *J. Chem. Phys.* **2010**, *132*, 154104.

[14] R. Krishnan, J. S. Binkley, R. Seeger, J. A. Pople, *J. Chem. Phys.* **1980**, *72*, 650-654.

[15] A. V. Marenich, C. J. Cramer, D. G. Truhlar, *J. Phys. Chem. B* **2009**, *113*, 6378-6396.

[16] G. Knizia, J. E. Klein, *Angew. Chem. Int. Ed.* **2015**, *54*, 5518-5522.

[17] C. Y. Legault, Université de Sherbrooke, Canada, **2020**.

[18] G. M. Sheldrick, *Acta Cryst.* **2015**, *A71*, 3.

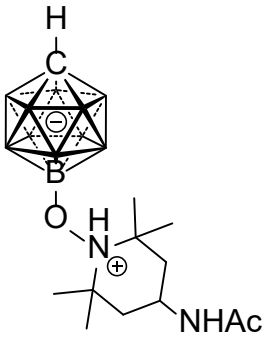
[19] G. M. Sheldrick, *Acta Cryst.* **2015**, *C71*, 3.

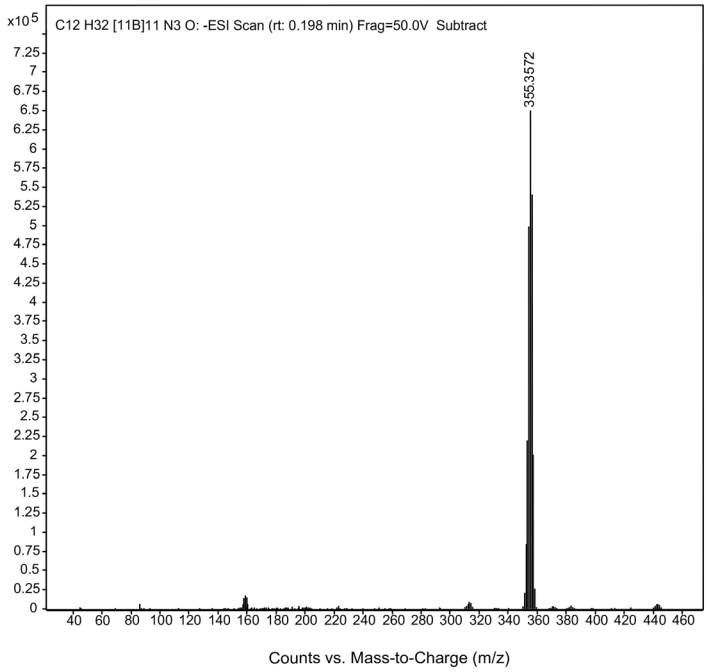
[20] O. V. Dolomanov, L. J. Bourhis, R. J. Gildea, J. A. K. Howard, H. Puschmann, *J.Appl. Cryst.* **2009**, *42*, 339.

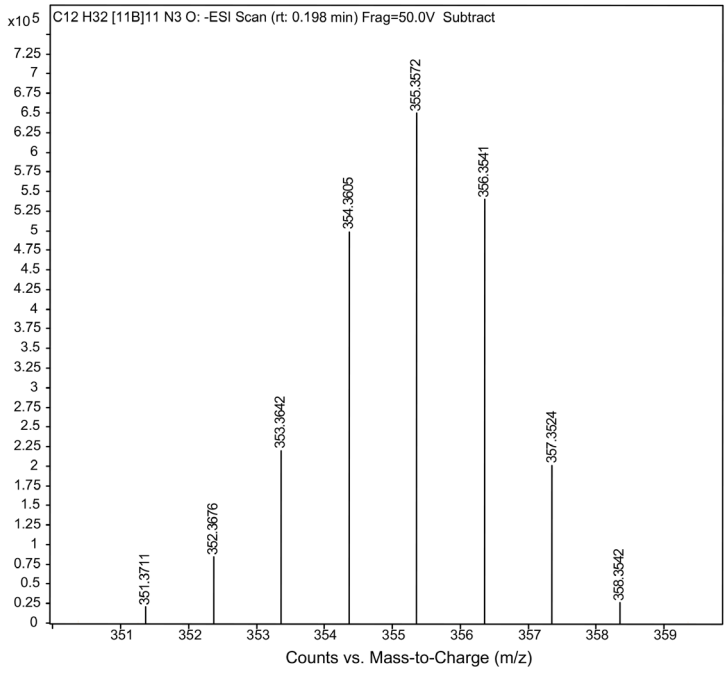
[21] C. F. Macrae, I. J. Bruno, J. A. Chisholm, P. R. Edgington, P. McCabe, E. Pidcock, L. Rodriguez-Monge, R. Taylor, J. vandeStreek, P. A. Wood, *J. Appl. Cryst.* **2008**, *41*, 466.

VI Mass spectra

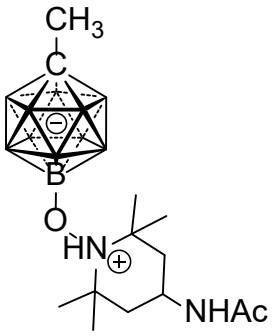
2

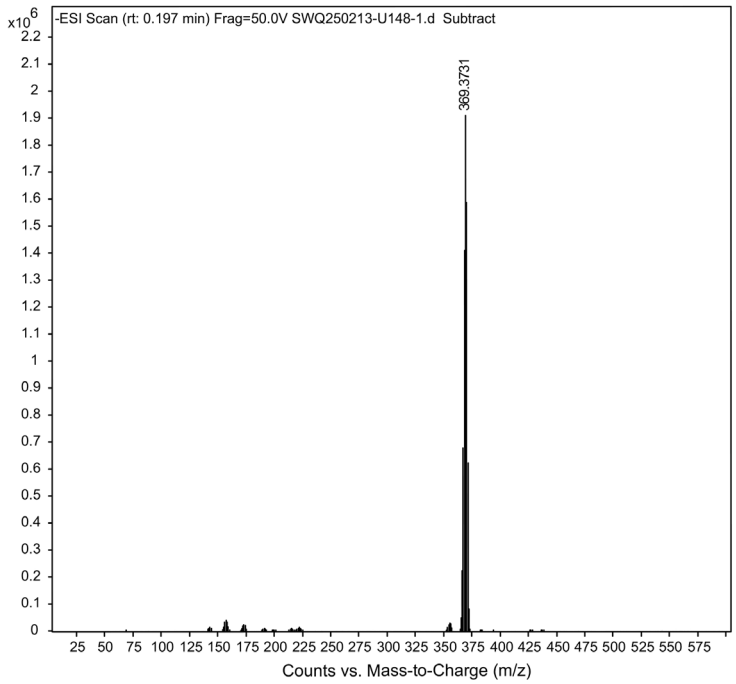


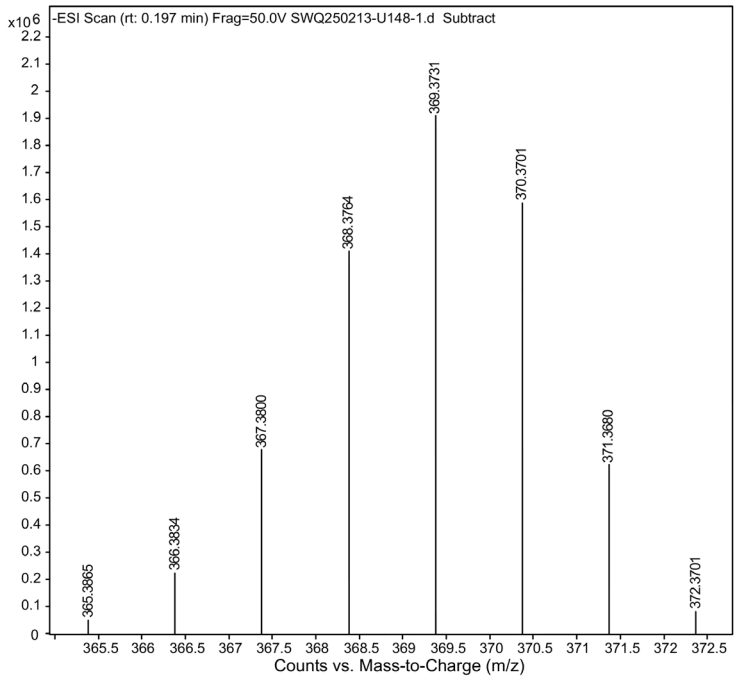




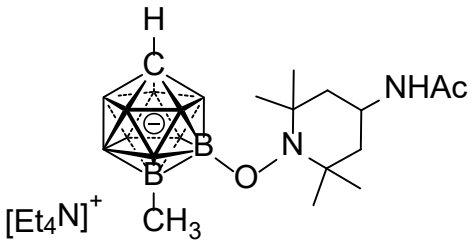
3a

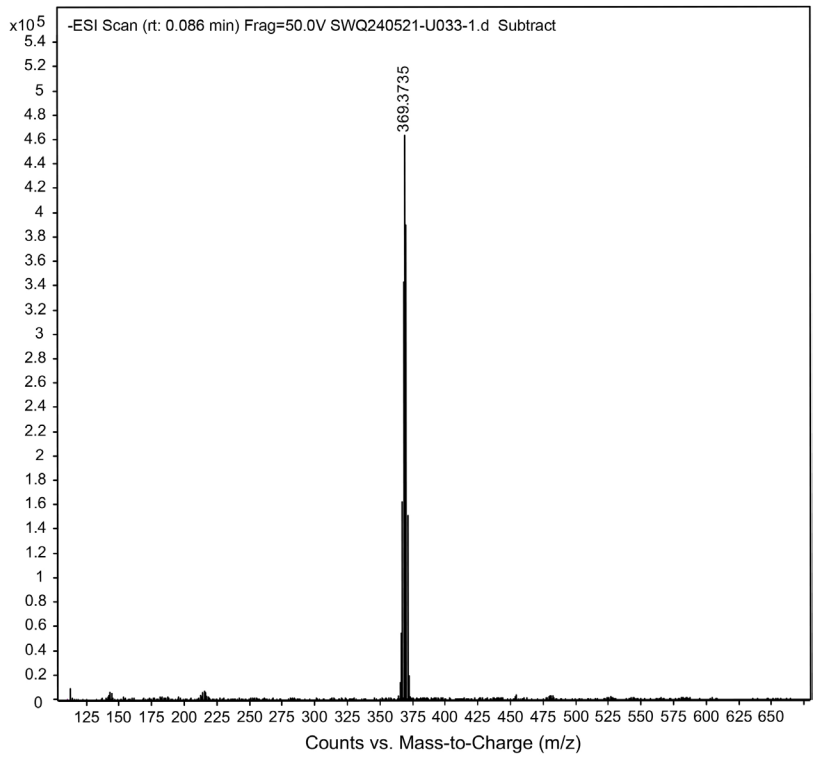

CH3
C
B
O
HN
NHAc

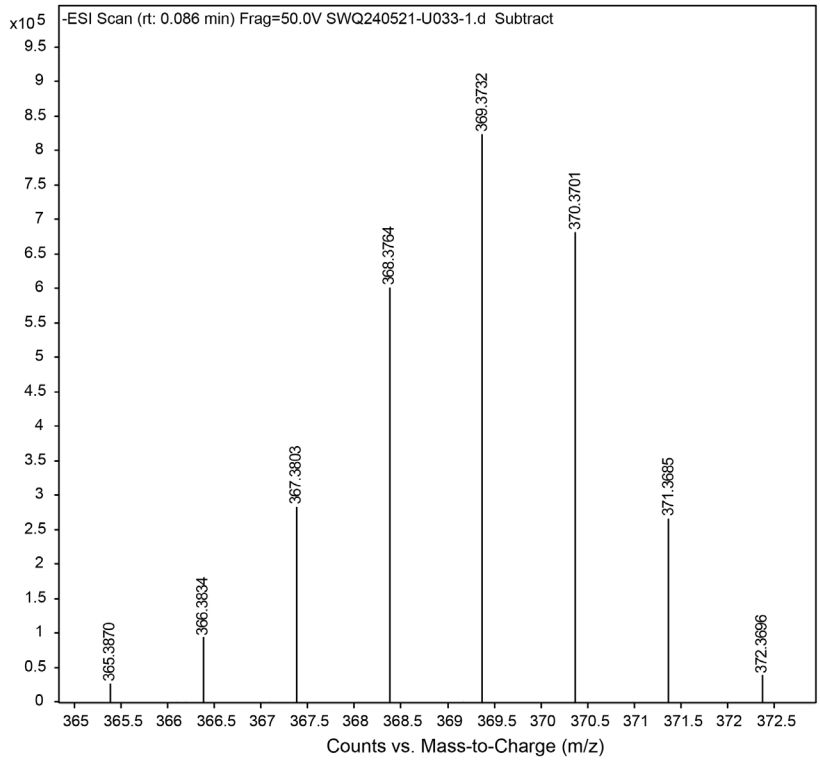

x10^6
-ESI Scan (rt: 0.197 min) Frag=50.0V SWQ250213-U148-1.d Subtract
369.3731
Counts vs. Mass-to-Charge (m/z)


x10^6
-ESI Scan (rt: 0.197 min) Frag=50.0V SWQ250213-U148-1.d Subtract
365.3865
366.3834
367.3800
368.3764
369.3731
370.3701
371.3680
372.3701
Counts vs. Mass-to-Charge (m/z)

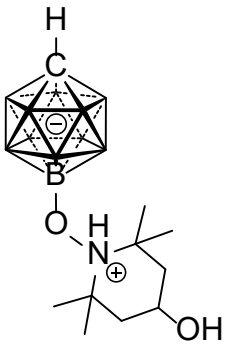
3b

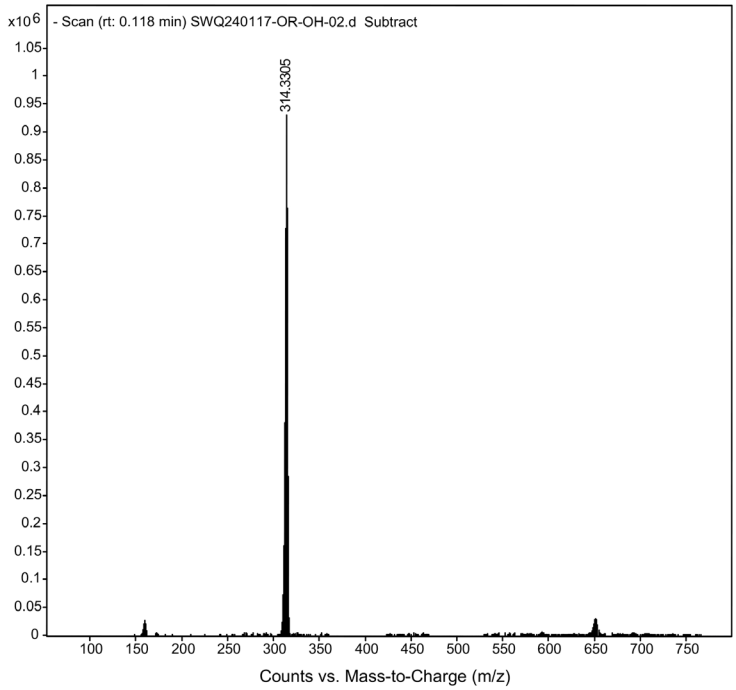

H
C
B
B
O
N
NHAc
[Et4N]+
CH3

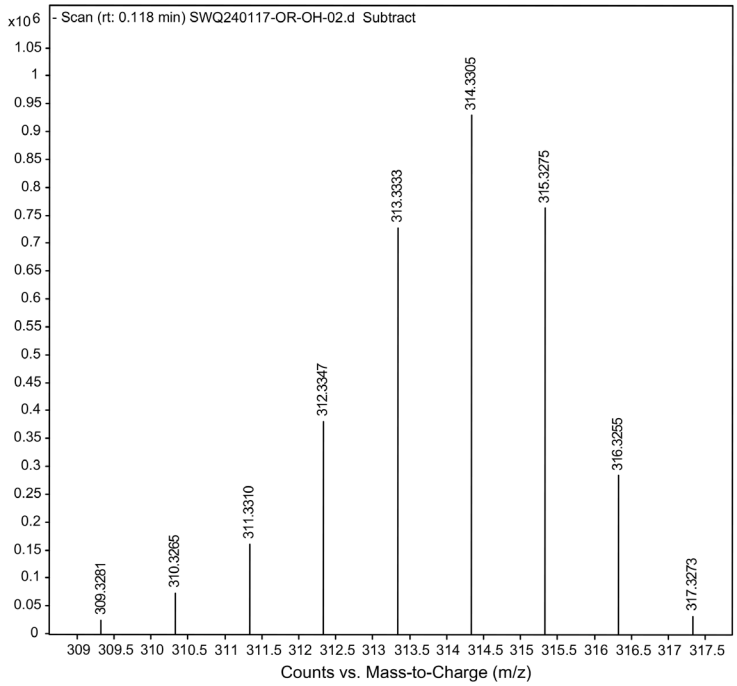

x10 5
-ESI Scan (rt: 0.086 min) Frag=50.0V SWQ240521-U033-1.d Subtract
369.3735
Counts vs. Mass-to-Charge (m/z)


x10 5
-ESI Scan (rt: 0.086 min) Frag=50.0V SWQ240521-U033-1.d Subtract
365.3870
366.3834
367.3803
368.3764
369.3732
370.3701
371.3685
372.3696
Counts vs. Mass-to-Charge (m/z)

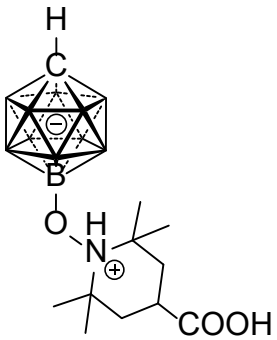
4a

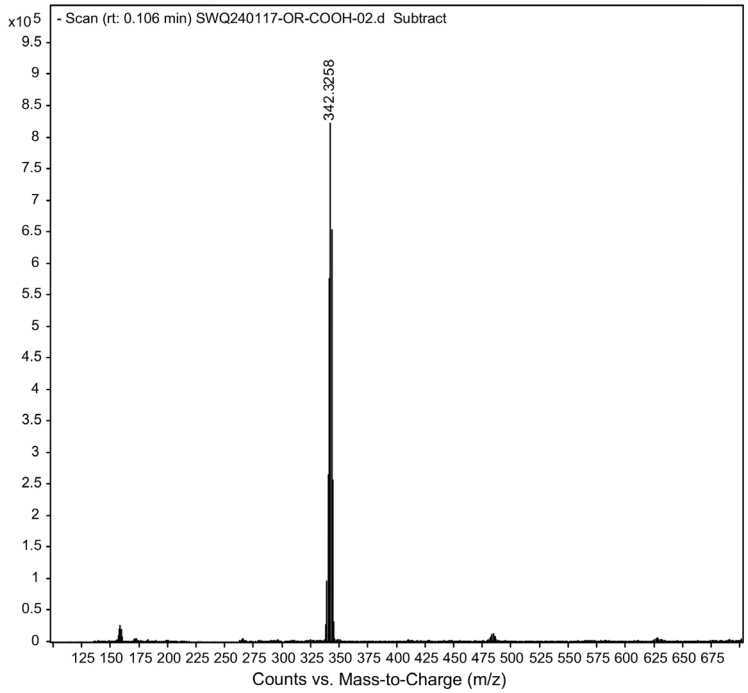

H
C
B
O
H
N
OH

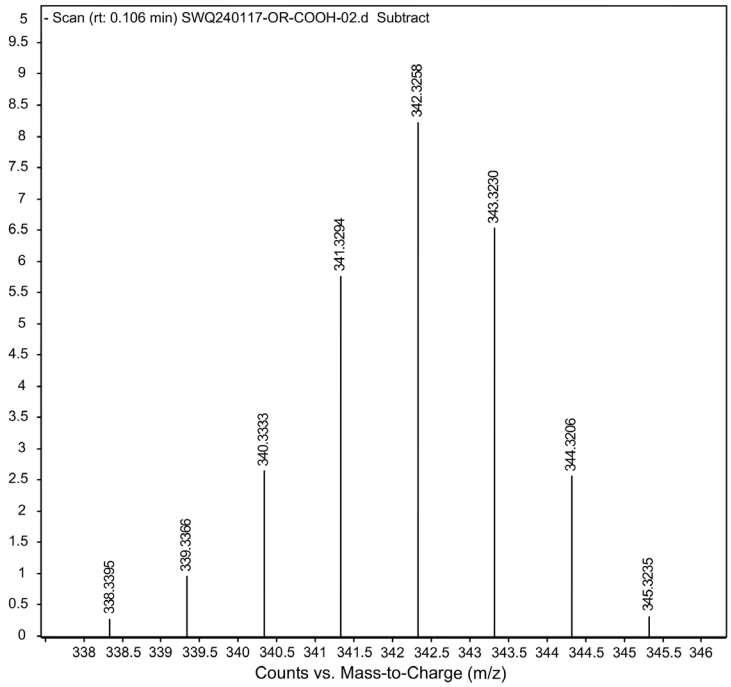

x10 6
- Scan (rt: 0.118 min) SWQ240117-OR-OH-02.d Subtract
314.3305
Counts vs. Mass-to-Charge (m/z)


x10 6
- Scan (rt: 0.118 min) SWQ240117-OR-OH-02.d Subtract
309.3281
310.3265
311.3310
312.3347
313.3333
314.3305
315.3275
316.3255
317.3273
Counts vs. Mass-to-Charge (m/z)

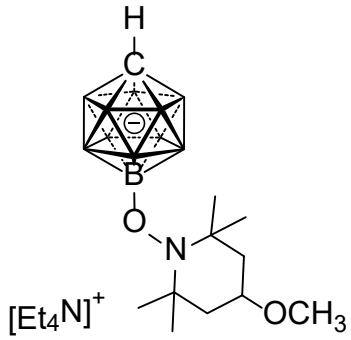
4b

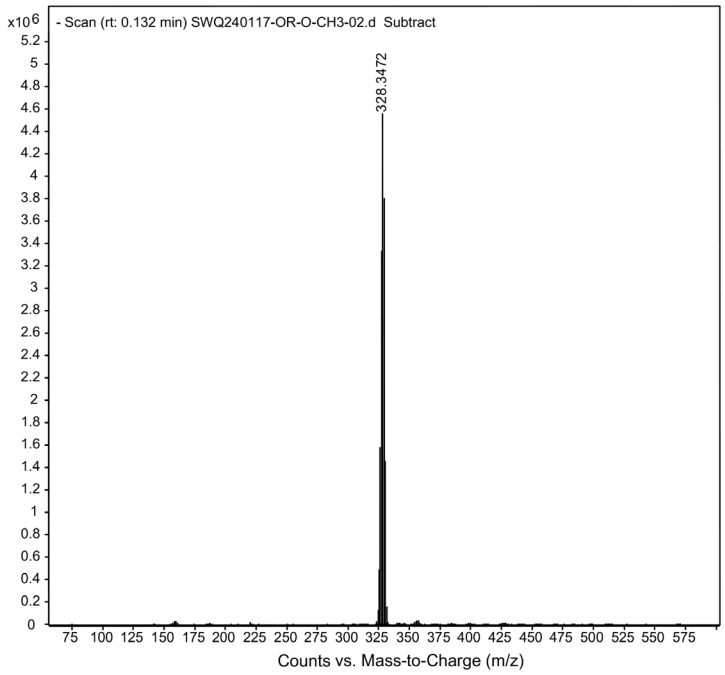

H
C
B
O
H
N
COOH

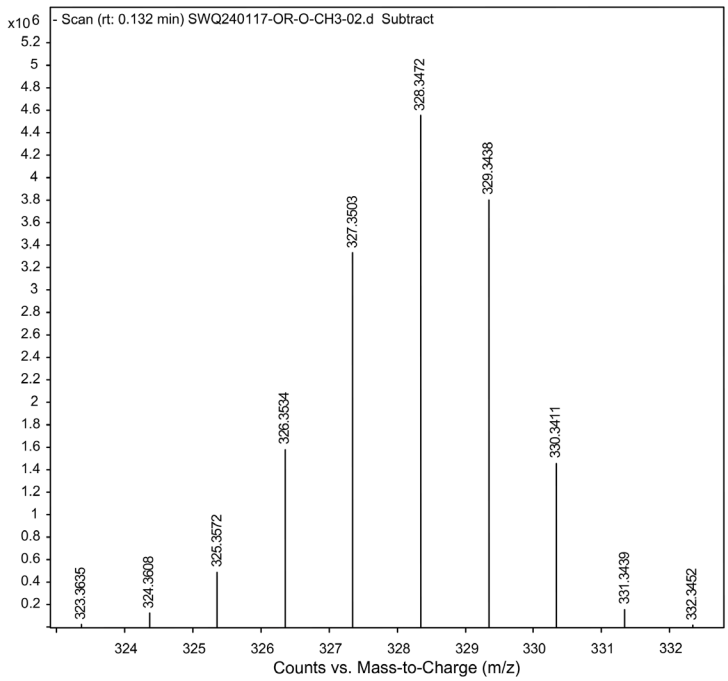

x10 5
- Scan (rt: 0.106 min) SWQ240117-OR-COOH-02.d Subtract
342.3258
Counts vs. Mass-to-Charge (m/z)


- Scan (rt: 0.106 min) SWQ240117-OR-COOH-02.d Subtract
338.3395
339.3366
340.3333
341.3294
342.3258
343.3230
344.3206
345.3235
Counts vs. Mass-to-Charge (m/z)

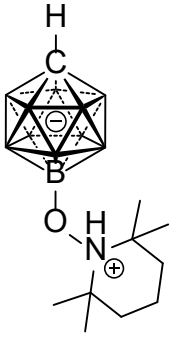
4c

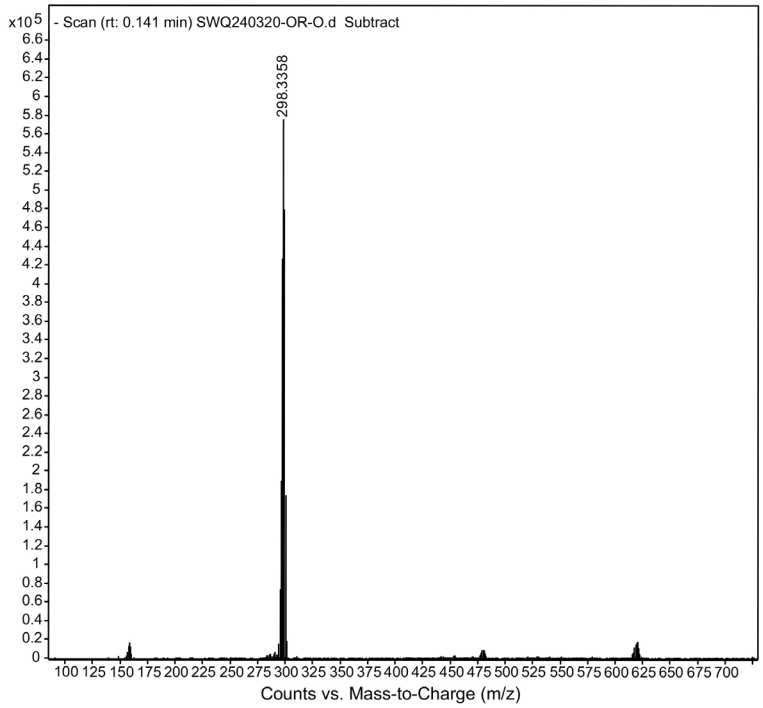

H
C
B
O
N
[Et4N]+
OCH3

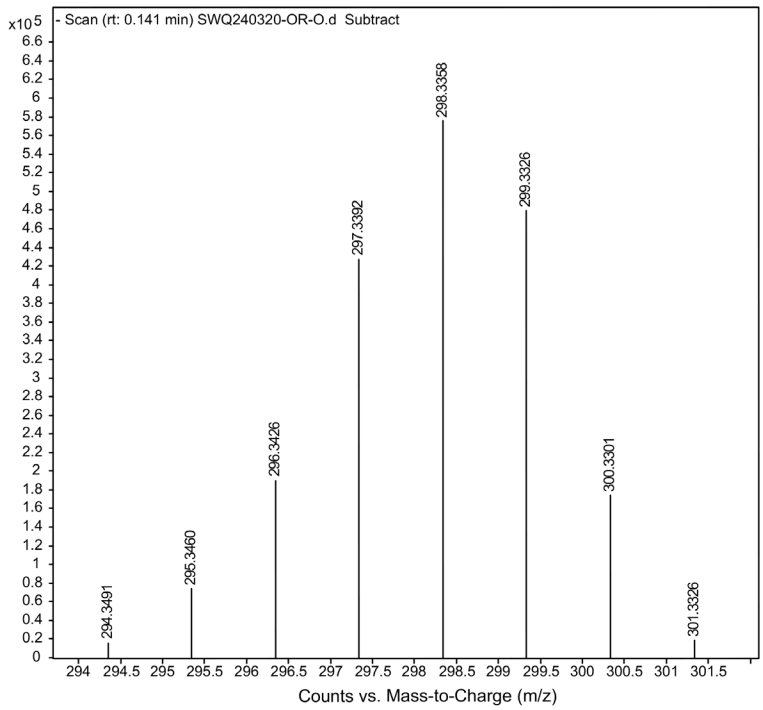

x10 6
- Scan (rt: 0.132 min) SWQ240117-OR-O-CH3-02.d Subtract
328.3472
Counts vs. Mass-to-Charge (m/z)


x10 6
- Scan (rt: 0.132 min) SWQ240117-OR-O-CH3-02.d Subtract
323.3635
324.3608
325.3572
326.3534
327.3503
328.3472
329.3438
330.3411
331.3439
332.3452
Counts vs. Mass-to-Charge (m/z)

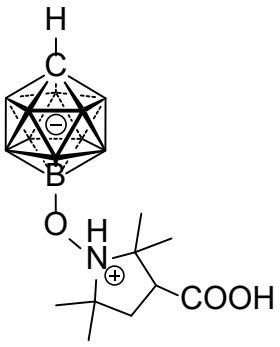
4d

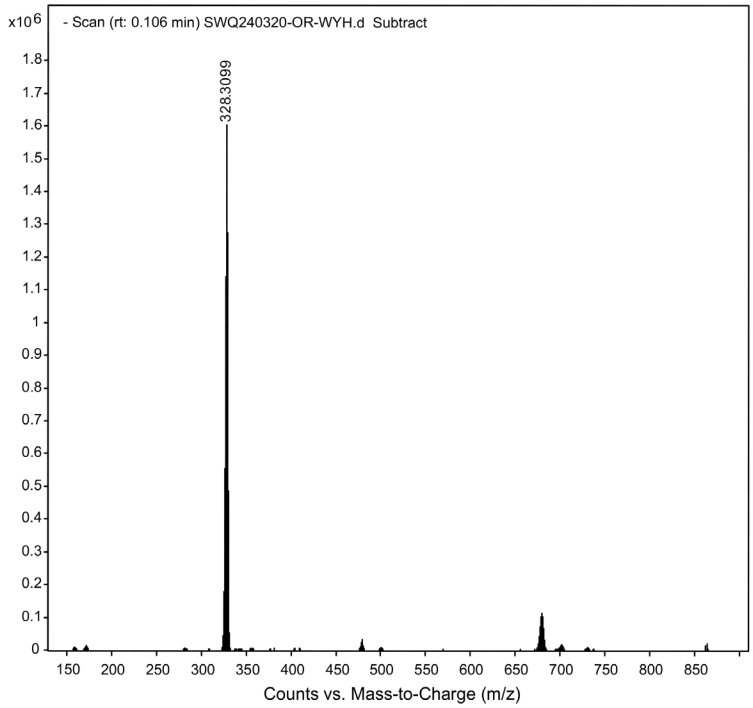

H
C
B
O
H
N

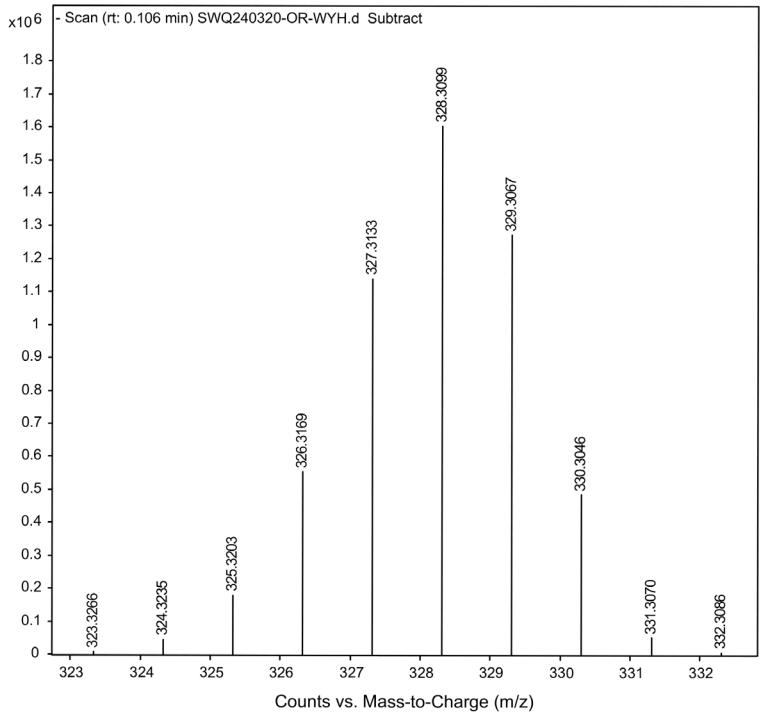

x10 5
- Scan (rt: 0.141 min) SWQ240320-OR-O.d Subtract
298.3358
Counts vs. Mass-to-Charge (m/z)


x10 5
- Scan (rt: 0.141 min) SWQ240320-OR-O.d Subtract
294.3491
295.3460
296.3426
297.3392
298.3358
299.3326
300.3301
301.3326
Counts vs. Mass-to-Charge (m/z)

4e

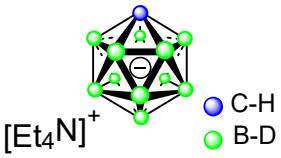

H
C
B
O
H
N
COOH

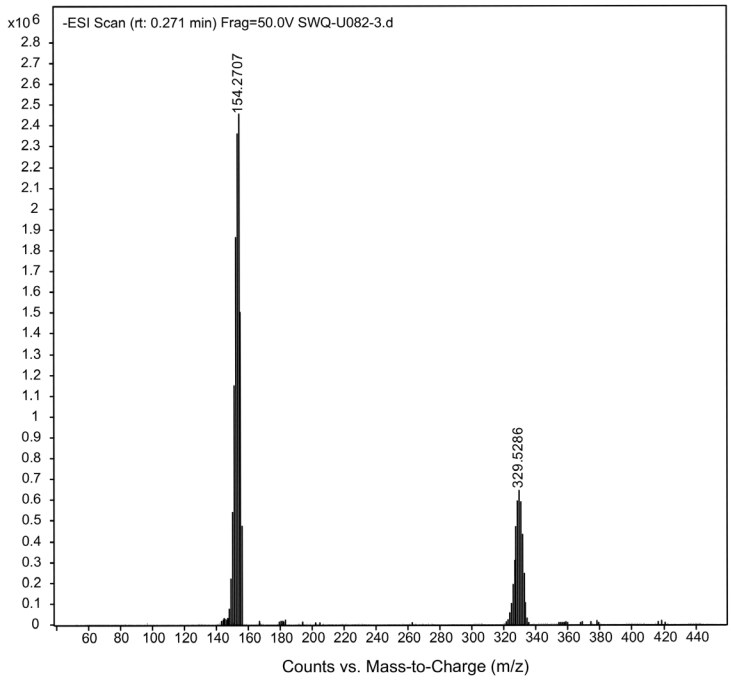

x10 6
- Scan (rt: 0.106 min) SWQ240320-OR-WYH.d Subtract
328.3099
Counts vs. Mass-to-Charge (m/z)

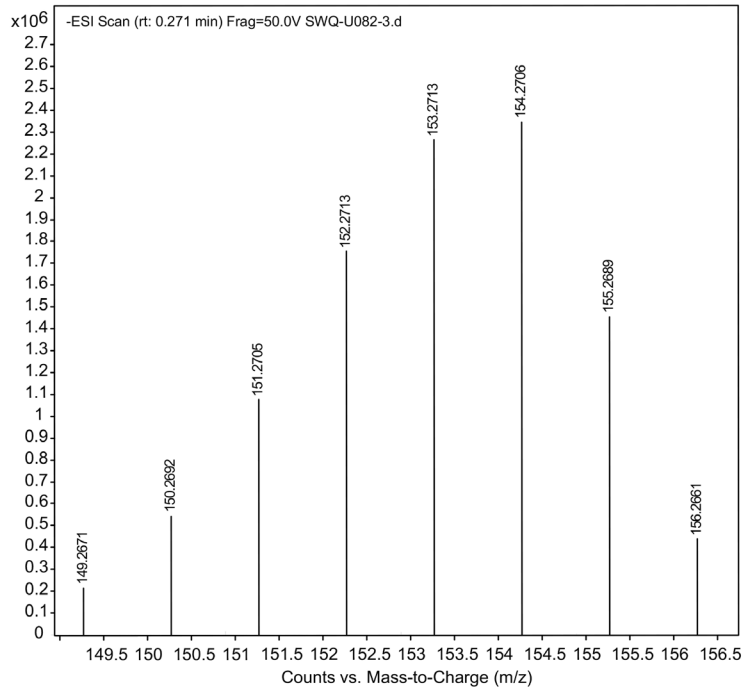

x10 6
- Scan (rt: 0.106 min) SWQ240320-OR-WYH.d Subtract
323.3266
324.3235
325.3203
326.3169
327.3133
328.3099
329.3067
330.3046
331.3070
332.3086
Counts vs. Mass-to-Charge (m/z)

5

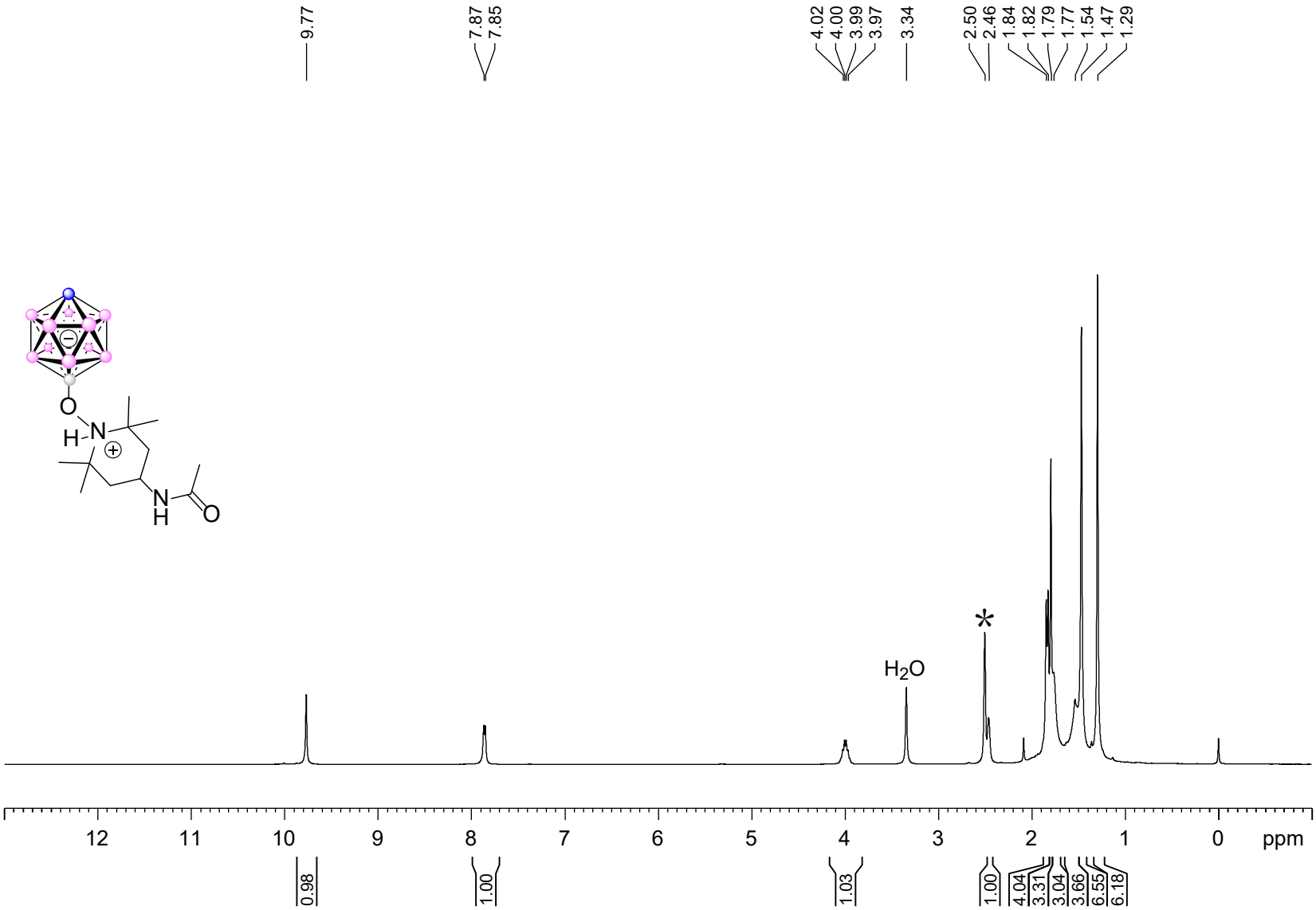
$[NEt_4]\ [H\text{-}CB_{11}D_{11}]$





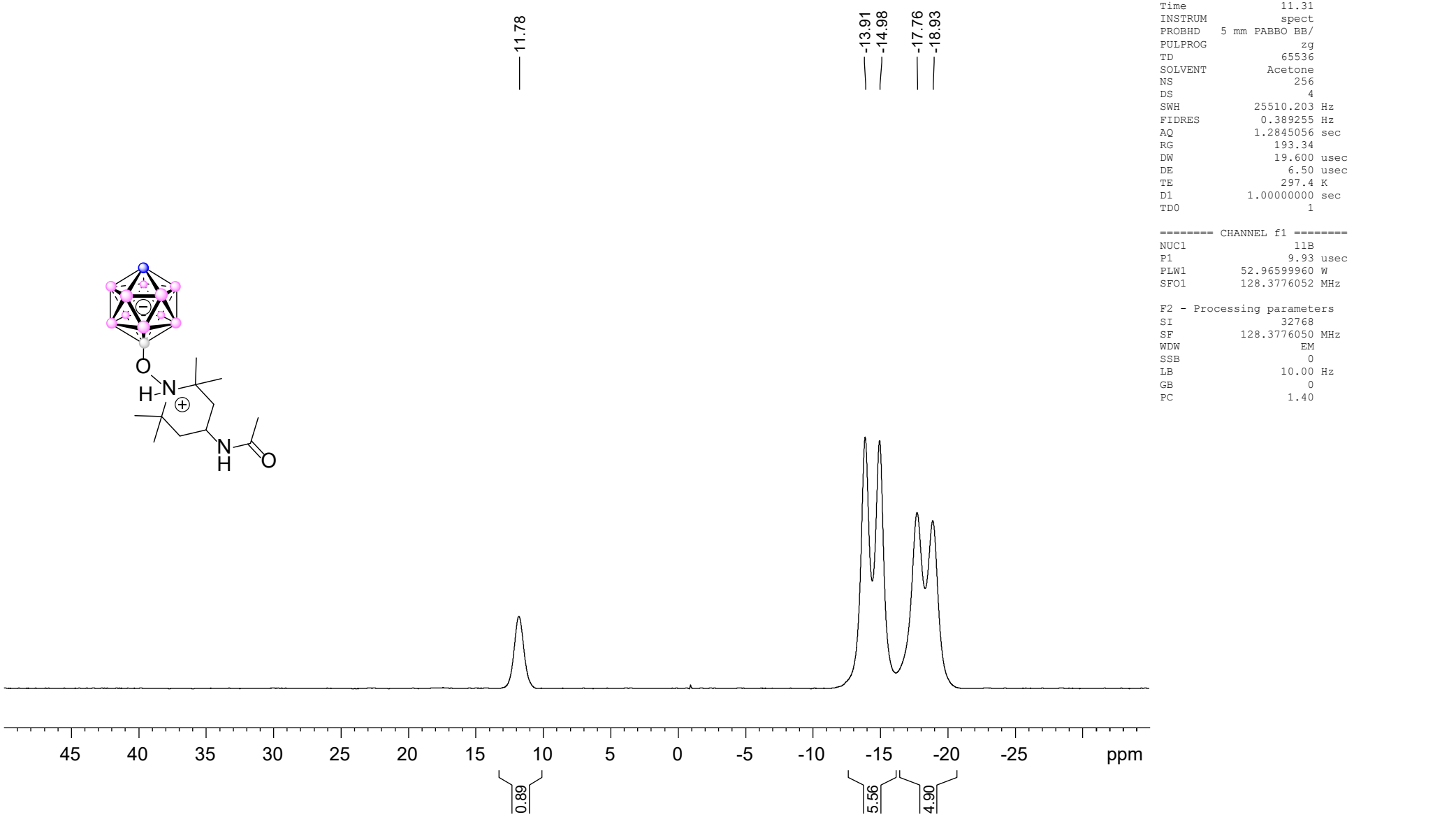


[H][12-NHAc-TEMPO-CB11H11]
1H{11B} 400MHZ DMSO T=23 C



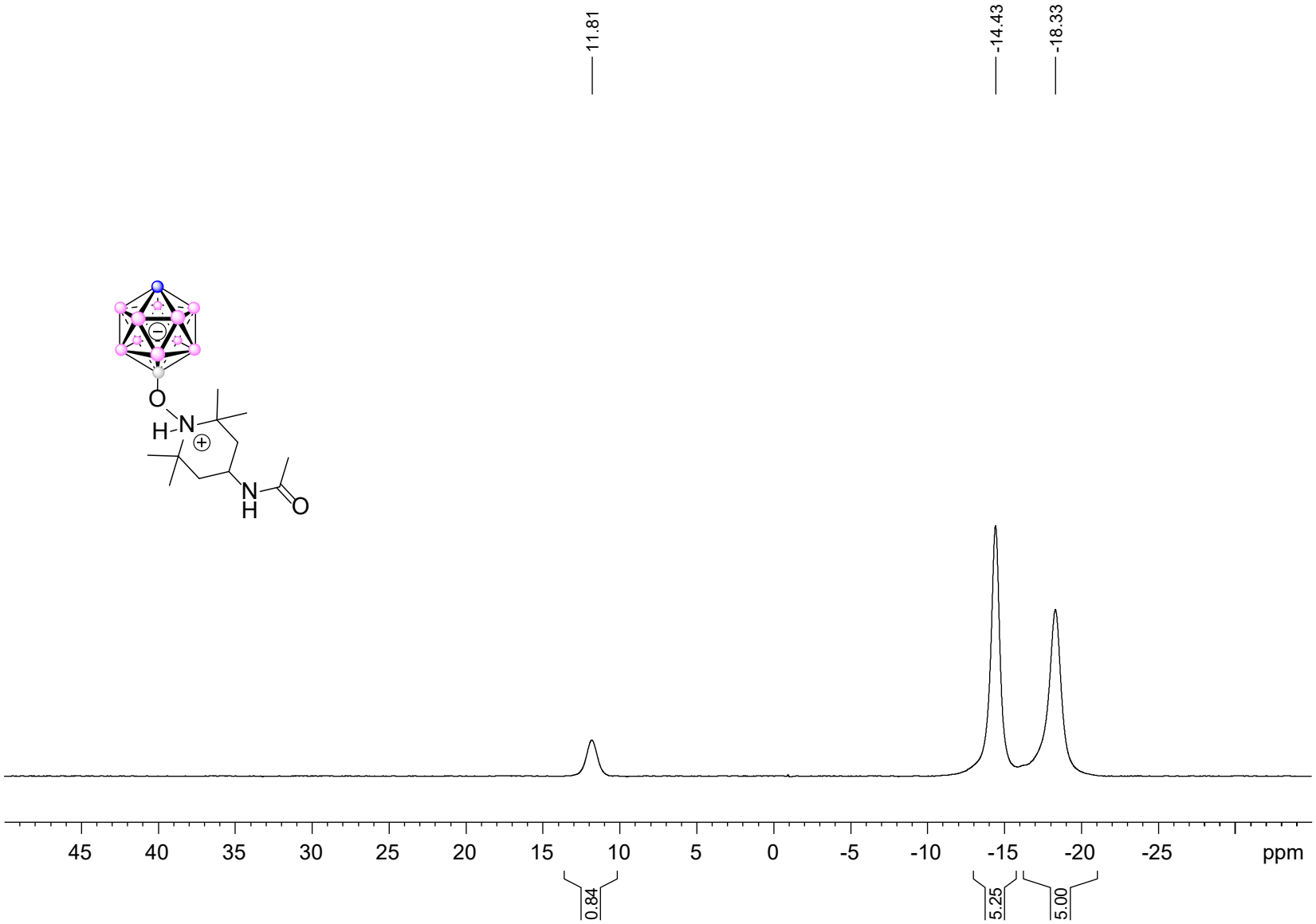

```
Current Data Parameters
NAME      20240323-OR-NHAc
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20240323
Time                 10.42
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG           zgig30
TD                 16384
SOLVENT             DMSO
NS                    16
DS                     4
SWH             8012.820 Hz
FIDRES          0.489064 Hz
AQ              1.0223616 sec
RG                 107.6
DW                62.400 usec
DE                 6.50 usec
TE                297.8 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 15.00 usec
PLW1         15.48099995 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          400.1300024 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

[H][12-NHAc-TEMPO-CB11H11]
11B 128MHZ Acetone T=23 C



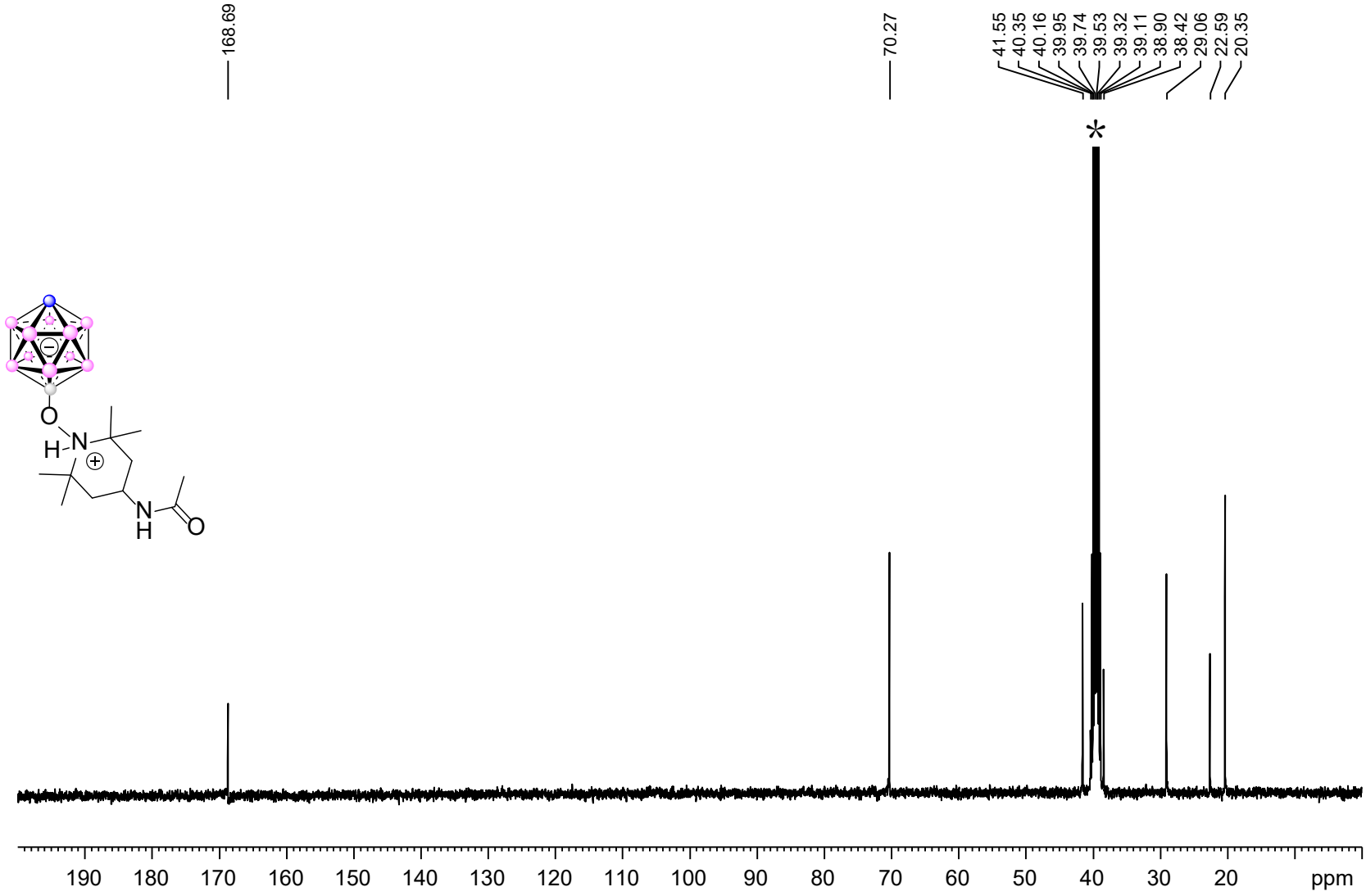

```
Current Data Parameters
NAME      20240326-NHAc-OR
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_           20240326
Time               11.31
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG               zg
TD                 65536
SOLVENT          Acetone
NS                   256
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                297.4 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
```

[H][12-NHAc-TEMPO-CB11H11]
11B{1H} 128MHZ Acetone T=23 C



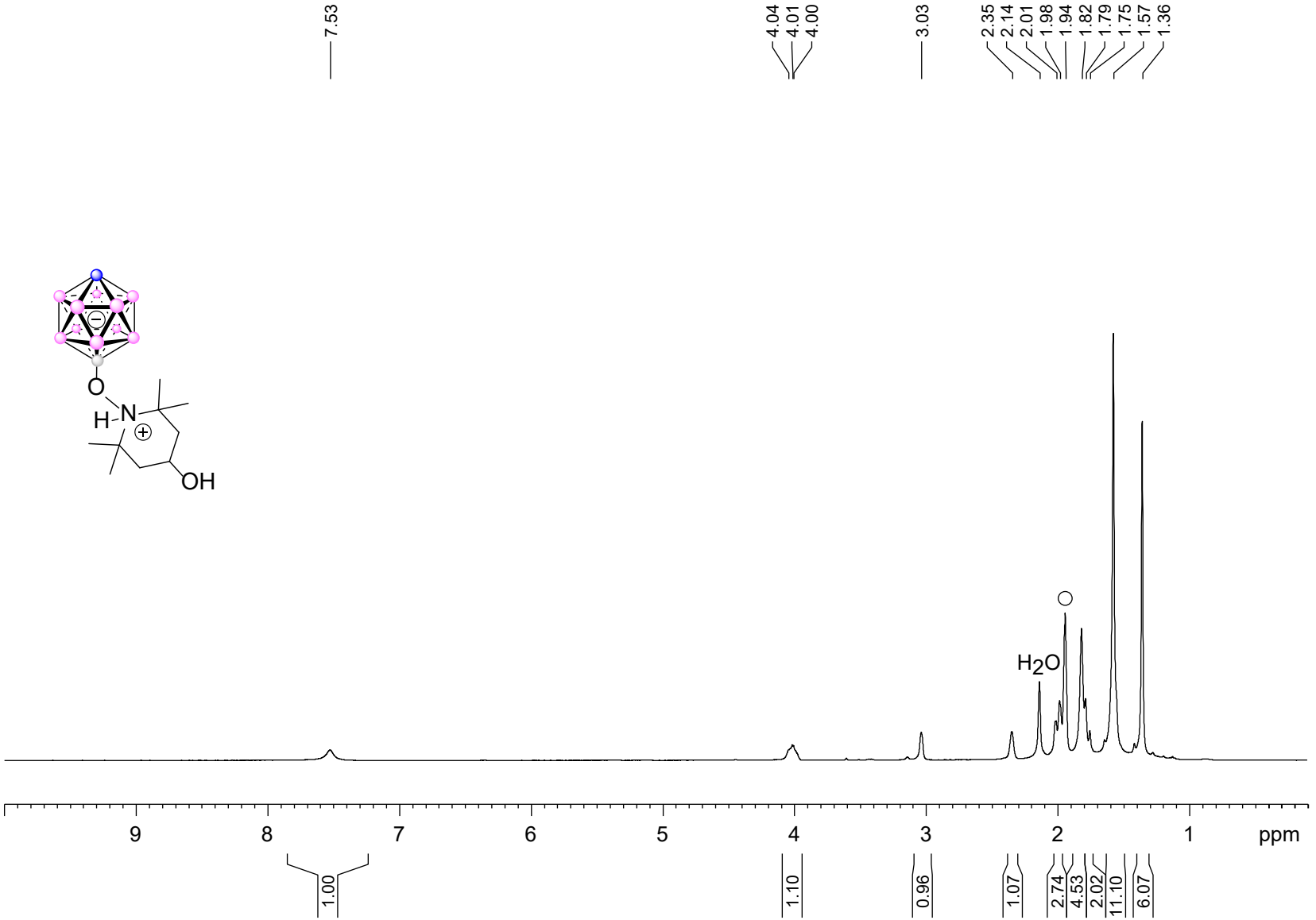

```
Current Data Parameters
NAME      20240326-NHAc-OR
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_             20240326
Time                 11.42
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                  65536
SOLVENT           Acetone
NS                    256
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                299.0 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         15.48099995 W
PLW12         0.54425001 W
PLW13         0.34832001 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
```

[H][12-NHAc-TEMPO-CB11H11]
13C{1H} 100MHZ DMSO T=23 C



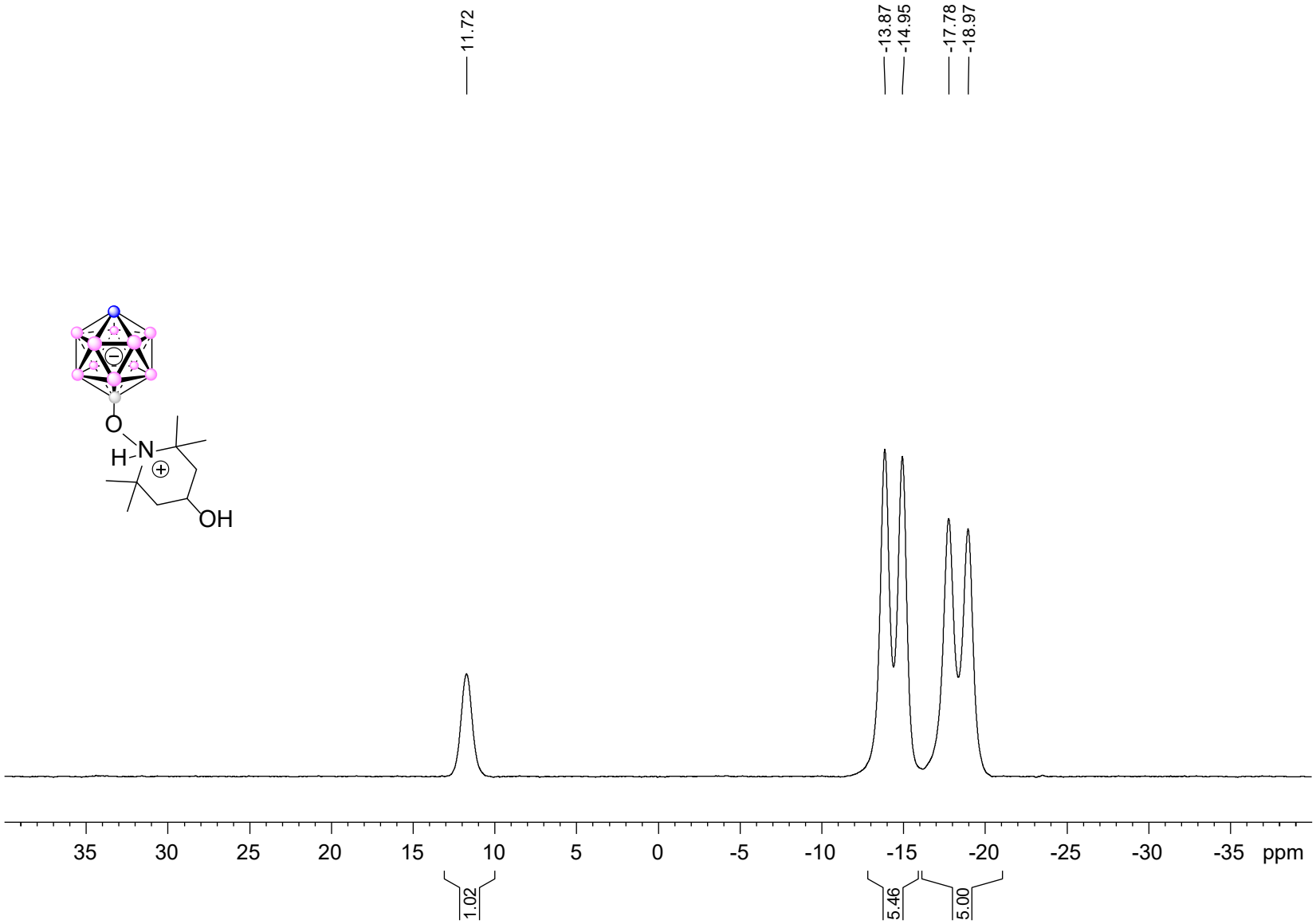

```
Current Data Parameters
NAME          S075-2-OR
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20231104
Time              16.22
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT            DMSO
NS                   512
DS                     4
SWH            29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.1010048 sec
RG                170.36
DW              16.800 usec
DE               6.50 usec
TE               301.7 K
D1          1.50000000 sec
D11         0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PLW1        66.13099670 W
SFO1       100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PLW2        15.48099995 W
PLW12        0.54425001 W
PLW13        0.34832001 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                32768
SF         100.6128193 MHz
WDW                  EM
SSB                   0
LB                 2.00 Hz
GB                    0
PC                 1.40
```

S123-1-3 [H][12-OH-TEMPO-CB11H11]
1H{11B} 400MHZ CD3CN T=23 C



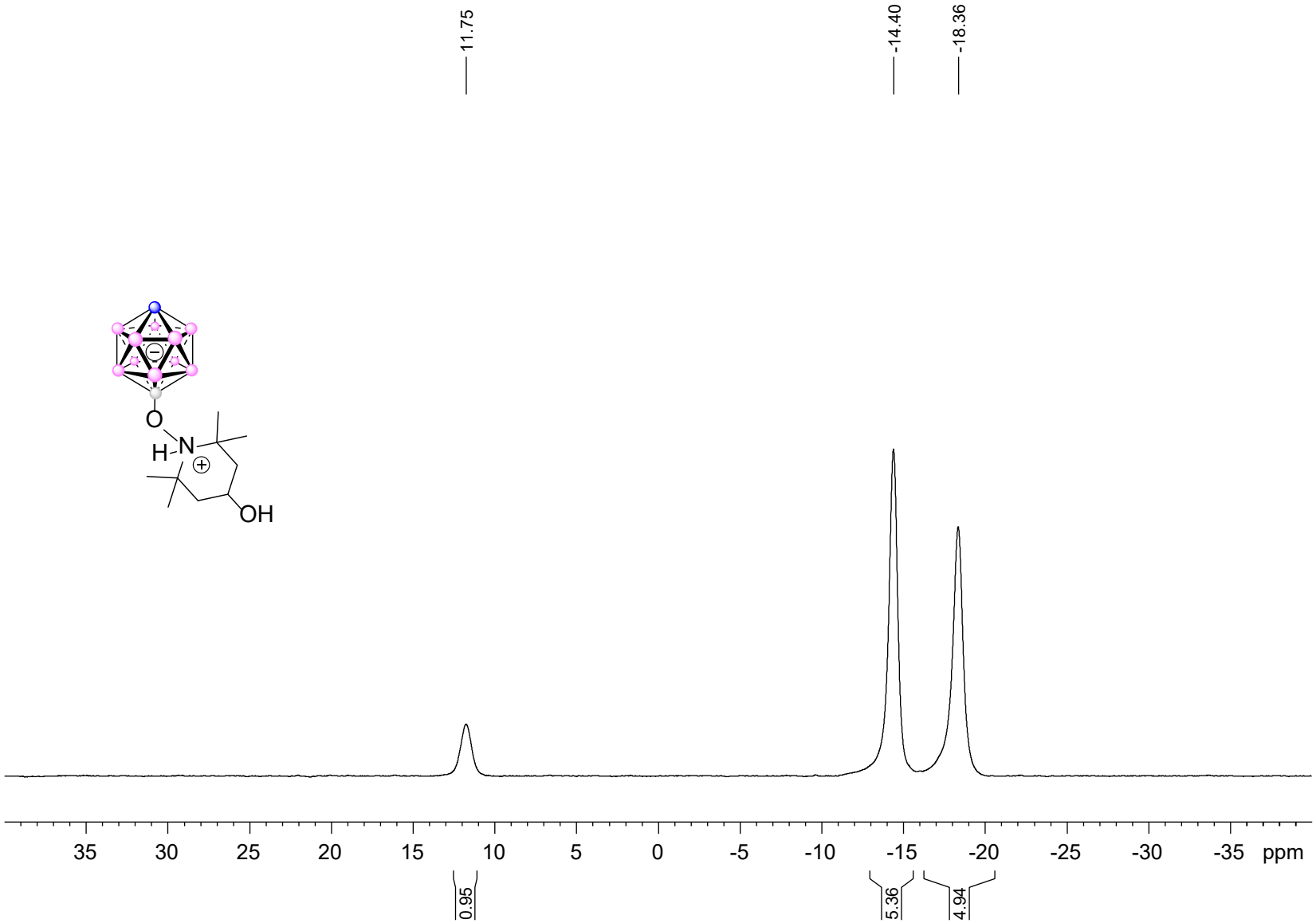

```
Current Data Parameters
NAME      S123-1-3-OR-OH
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240106
Time                2.01
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgig30
TD                16384
SOLVENT           CD3CN
NS                   64
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               298.1 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                15.00 usec
PLW1        15.48099995 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          400.1300093 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40
```

S123-1-3 [H][12-OH-TEMPO-CB11H11]
11B 128MHZ CD3CN T=23 C



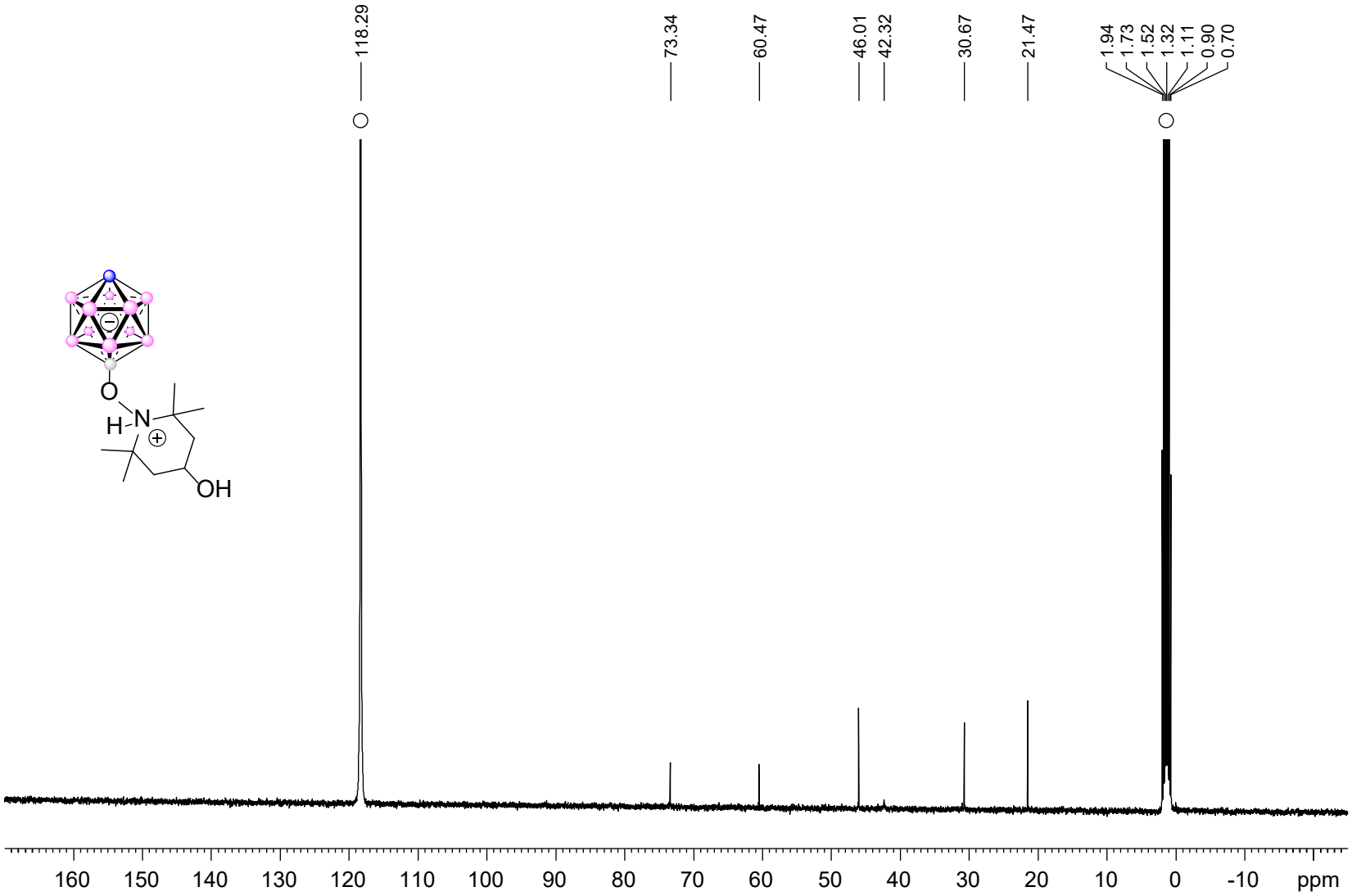

```
Current Data Parameters
NAME      S123-1-3-OR-OH
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240107
Time               22.06
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG               zg
TD                 65536
SOLVENT            CD3CN
NS                   254
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                298.1 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                10.00 Hz
GB                    0
PC                 1.40
```

S123-1-3 [H][12-OH-TEMPO-CB11H11]
11B{1H} 128MHZ CD3CN T=23 C



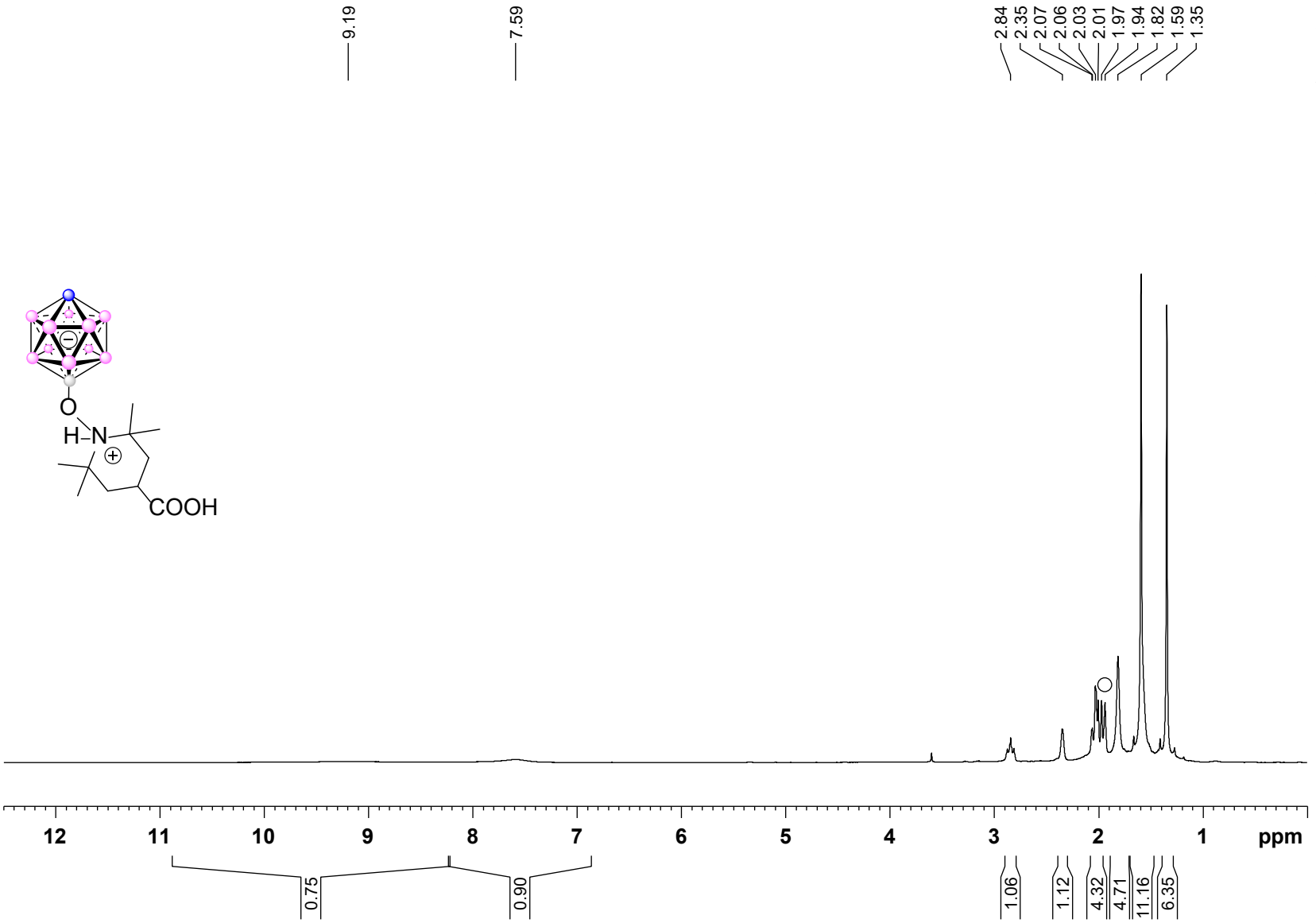

```
Current Data Parameters
NAME      S123-1-3-OR-OH
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240107
Time              22.17
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT           CD3CN
NS                  254
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              299.5 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        15.48099995 W
PLW12        0.54425001 W
PLW13        0.34832001 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
```

S123-1-3 [H][12-OH-TEMPO-CB11H11]
13C{1H} 128MHZ CD3CN T=23 C



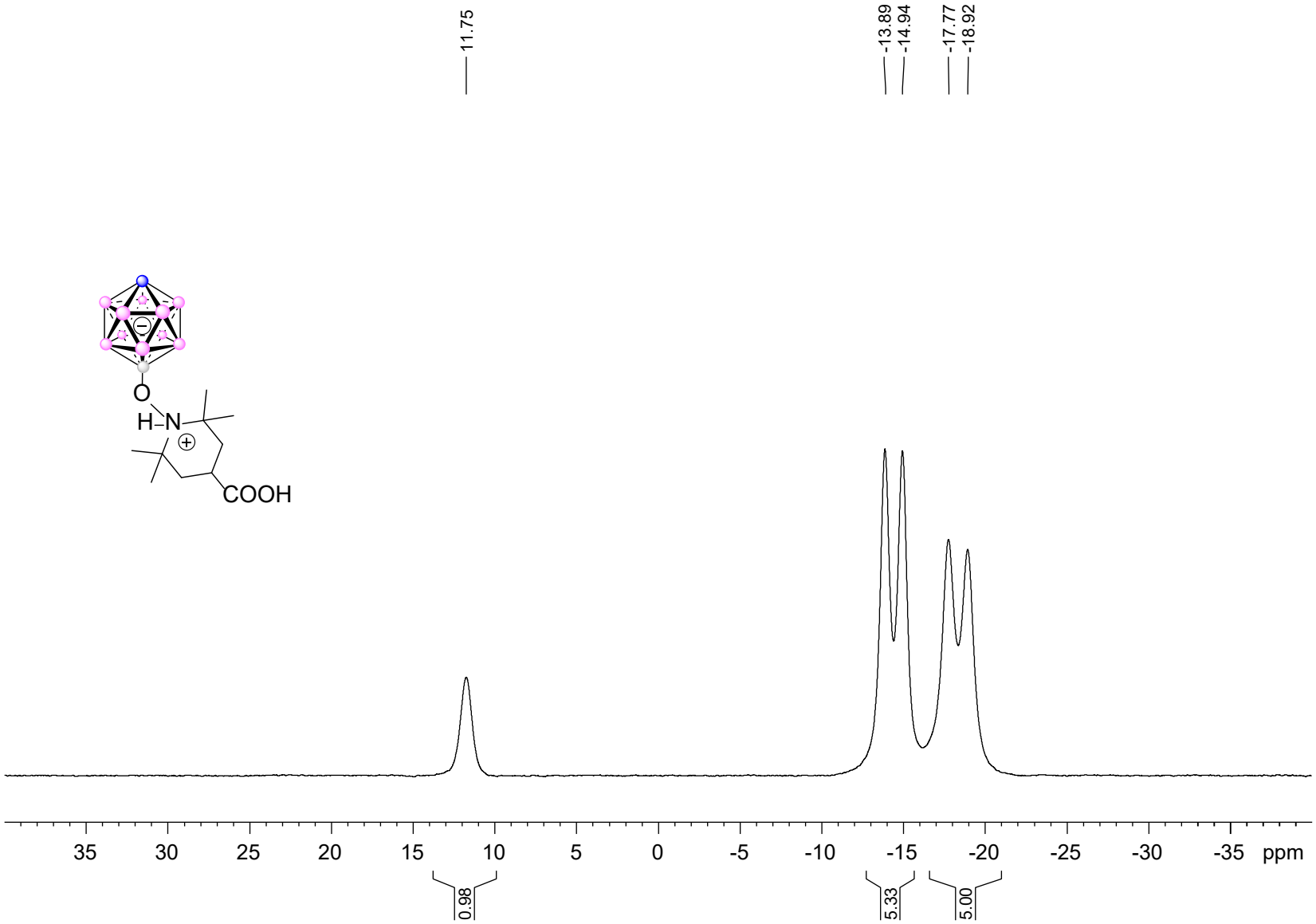

```
Current Data Parameters
NAME      S123-1-3-OR-OH
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20240107
Time              21.55
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT          CD3CN
NS                3072
DS                   4
SWH          29761.904 Hz
FIDRES        0.454131 Hz
AQ           1.1010048 sec
RG               107.6
DW              16.800 usec
DE               6.50 usec
TE              299.7 K
D1          1.50000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1                10.00 usec
PLW1        66.13099670 W
SFO1       100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2                1H
PCPD2            80.00 usec
PLW2        15.48099995 W
PLW12        0.54425001 W
PLW13        0.34832001 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6126648 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

S125-1-2 [H][12-COOH-TEMPO-CB11H11]
1H{11B} 400MHZ CD3CN T=23 C



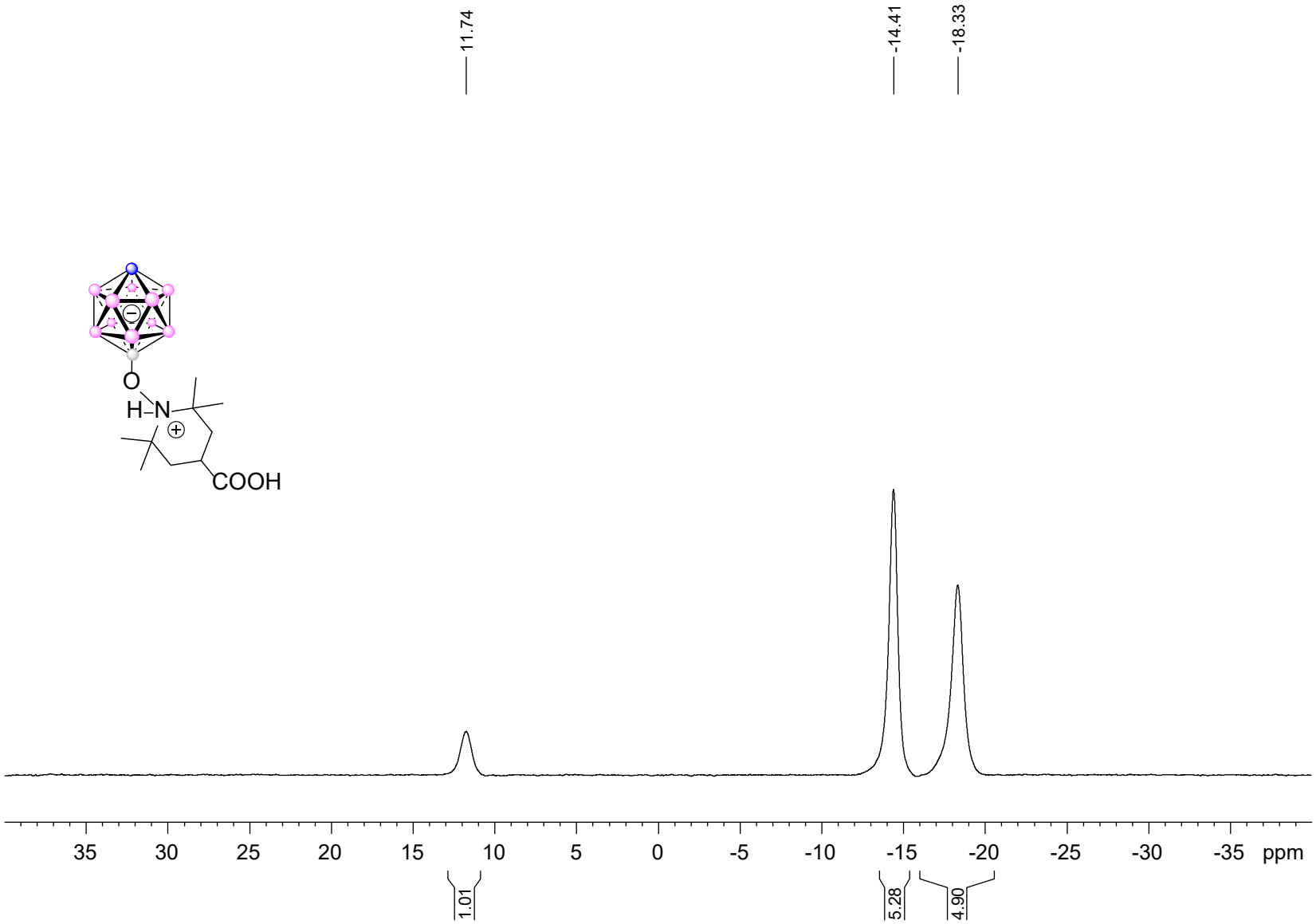

```
Current Data Parameters
NAME      20240320-OR-COOH
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_             20240320
Time                 19.32
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG             zgig30
TD                   16384
SOLVENT              CD3CN
NS                      64
DS                       4
SWH               8012.820 Hz
FIDRES            0.489064 Hz
AQ               1.0223616 sec
RG                   107.6
DW                  62.400 usec
DE                   6.50 usec
TE                  297.4 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   15.00 usec
PLW1          15.48099995 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
```

S125-1-2 [H][12-COOH-TEMPO-CB11H11]
11B 128MHZ CD3CN T=23 C



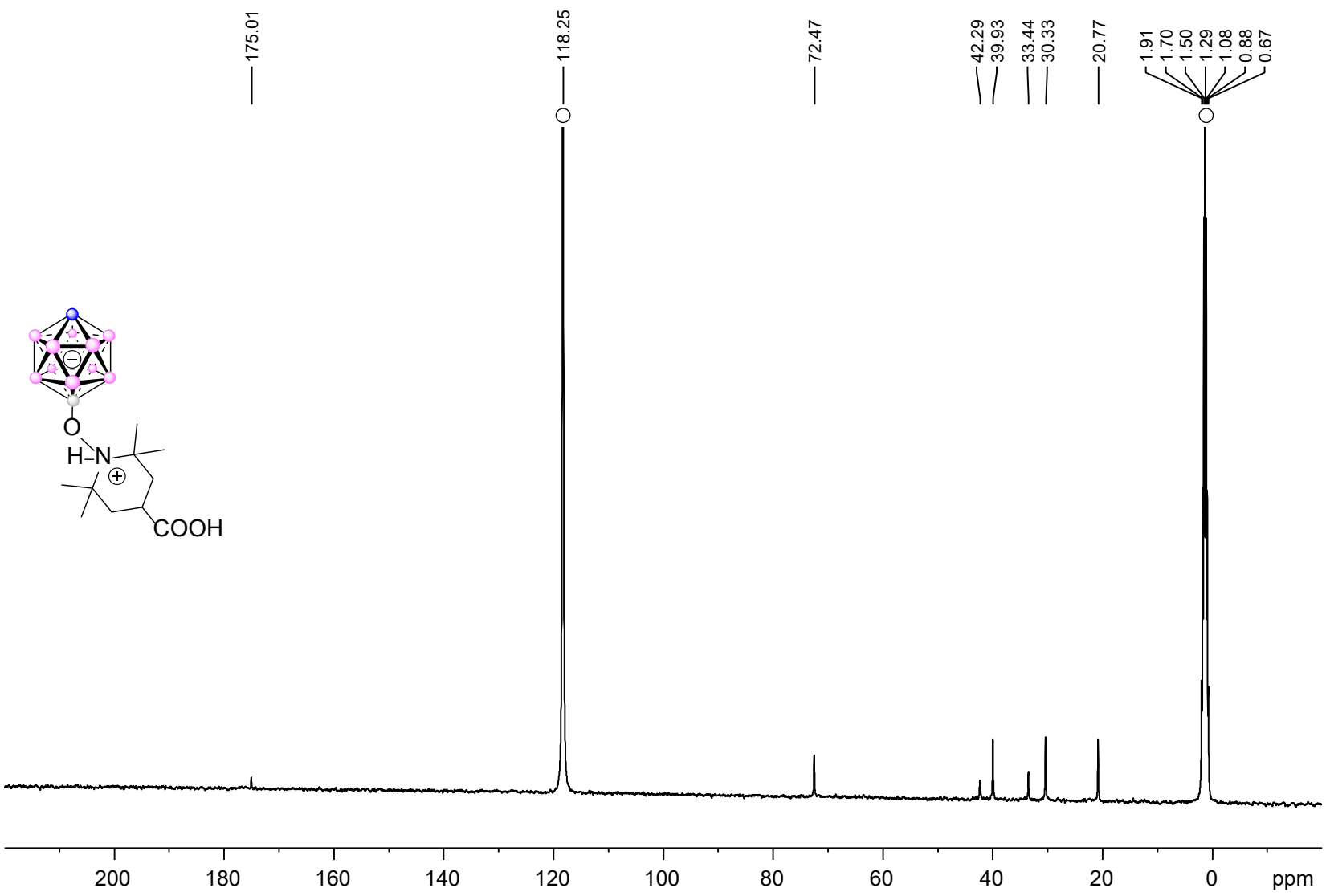

```
Current Data Parameters
NAME      S125-1-2-OR-COOH
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20240110
Time               20.16
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                298.0 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
```

S125-1-2 [H][12-COOH-TEMPO-CB11H11]
11B{1H} 128MHZ CD3CN T=23 C



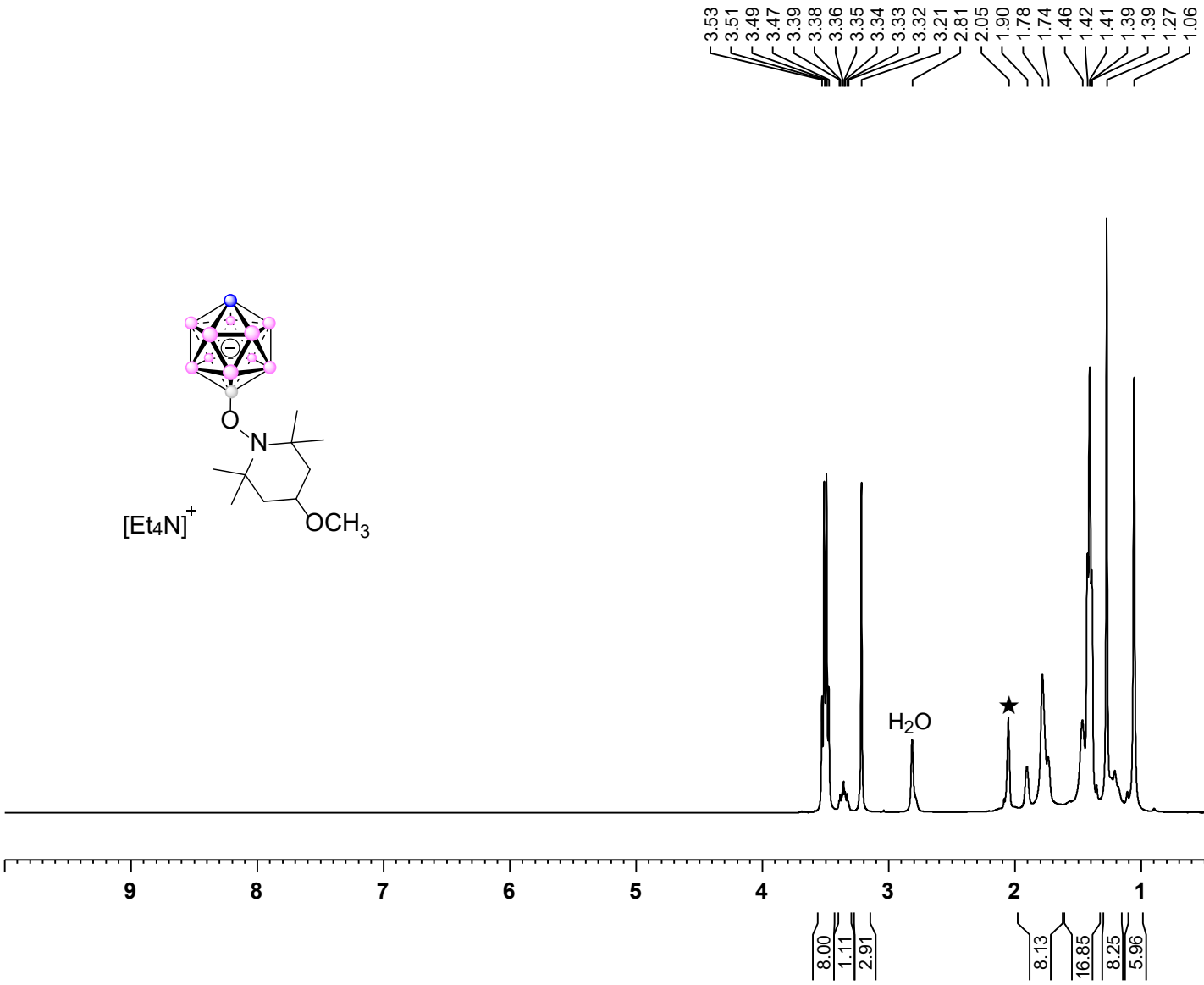

```
Current Data Parameters
NAME      S125-1-2-OR-COOH
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_             20240110
Time                 20.22
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                 299.1 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         15.48099995 W
PLW12         0.54425001 W
PLW13         0.34832001 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
```

S125-1 [H][12-COOH-TEMPO-CB11H11]
13C 100MHZ CD3CN T=23 C



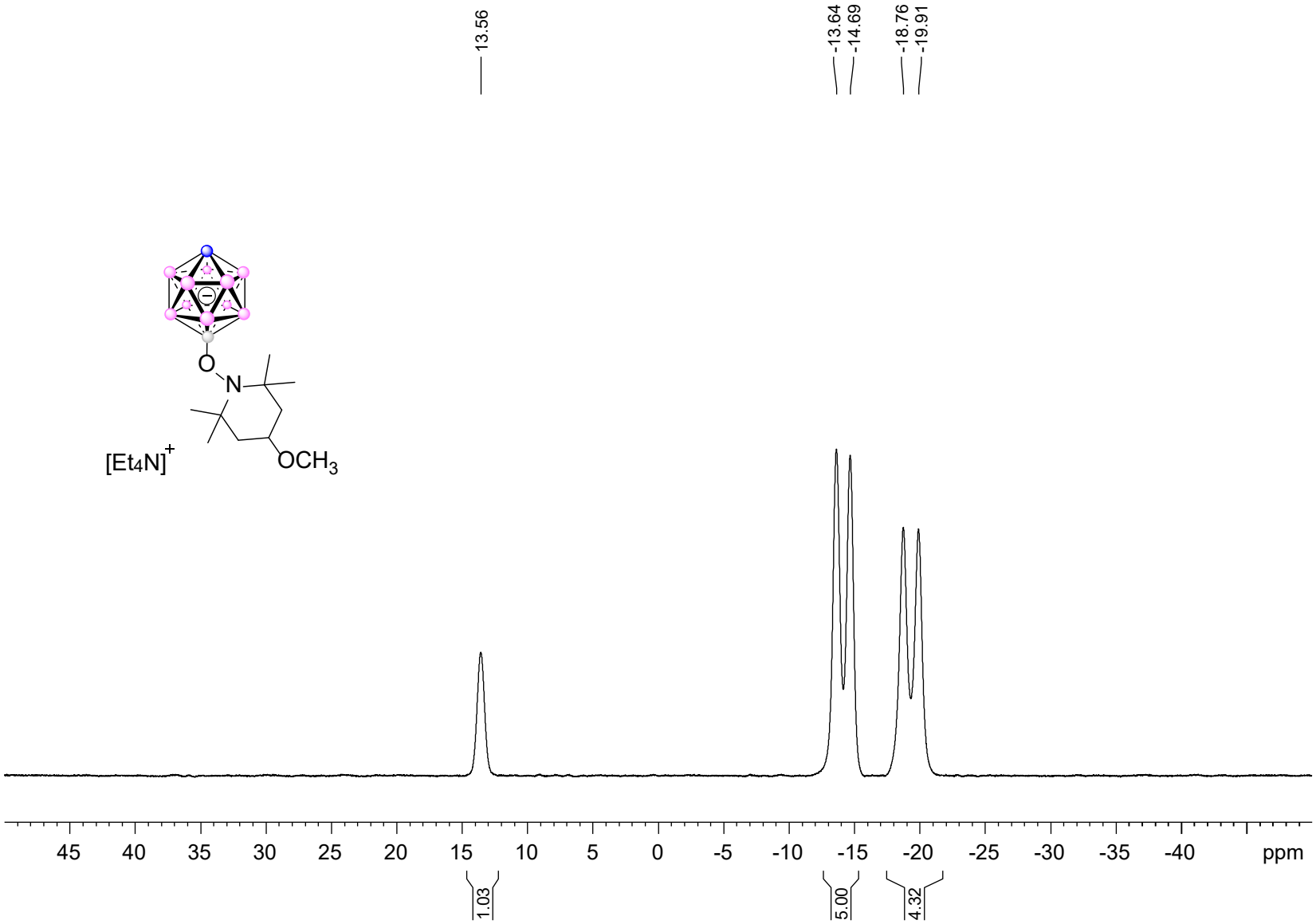

```
Current Data Parameters
NAME      20240904-OR-COOH
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_           20240904
Time              12.32 h
INSTRUM           spect
PROBHD   Z108618_0497 (
PULPROG          zgpg30
TD                65536
SOLVENT           CD3CN
NS                 1024
DS                    4
SWH           29761.904 Hz
FIDRES         0.908261 Hz
AQ             1.1010048 sec
RG                170.36
DW                16.800 usec
DE                 6.50 usec
TE                301.8 K
D1            1.50000000 sec
D11           0.03000000 sec
TD0                   1
SFO1         100.6228293 MHz
NUC1                13C
P1                10.00 usec
PLW1         66.13099670 W
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         15.48099995 W
PLW12         0.43002000 W
PLW13         0.21630000 W

F2 - Processing parameters
SI                 32768
SF           100.6126688 MHz
WDW                  EM
SSB                   0
LB                10.00 Hz
GB                    0
PC                 1.40
```

S105-1-3 [NEt4][12-CH3-O-TEMPO-CB11H11]
1H{11B} 400MHZ Acetone T=23 C



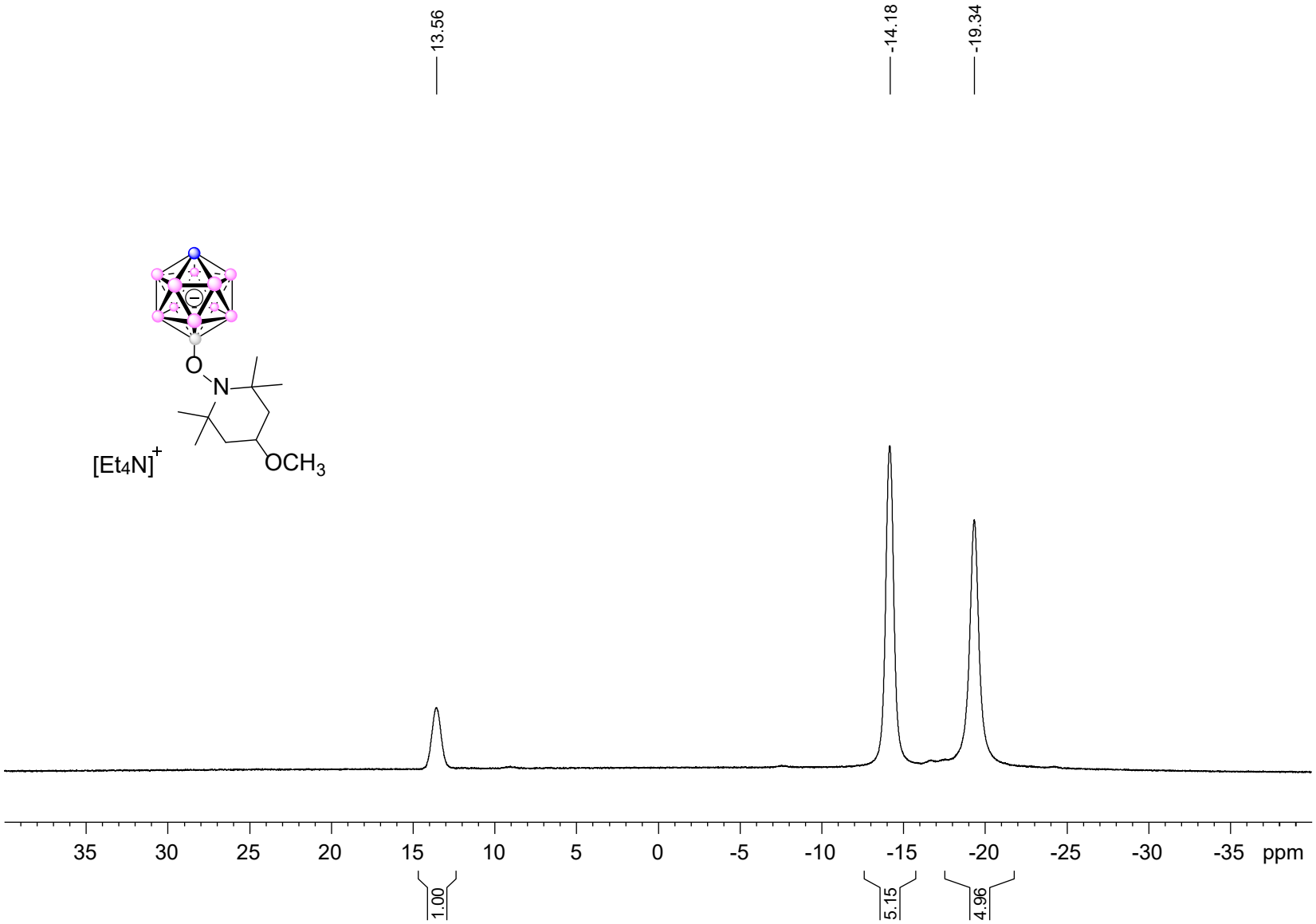

```
Current Data Parameters
NAME      20240323-OR-OCH3
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_            20240323
Time                21.06
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                   107.6
DW                  62.400 usec
DE                   6.50 usec
TE                  298.4 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  15.00 usec
PLW1          15.48099995 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
```

S105-1-3 [NEt4][12-CH3-O-TEMPO-CB11H11]
11B 128MHZ CD3CN T=23 C



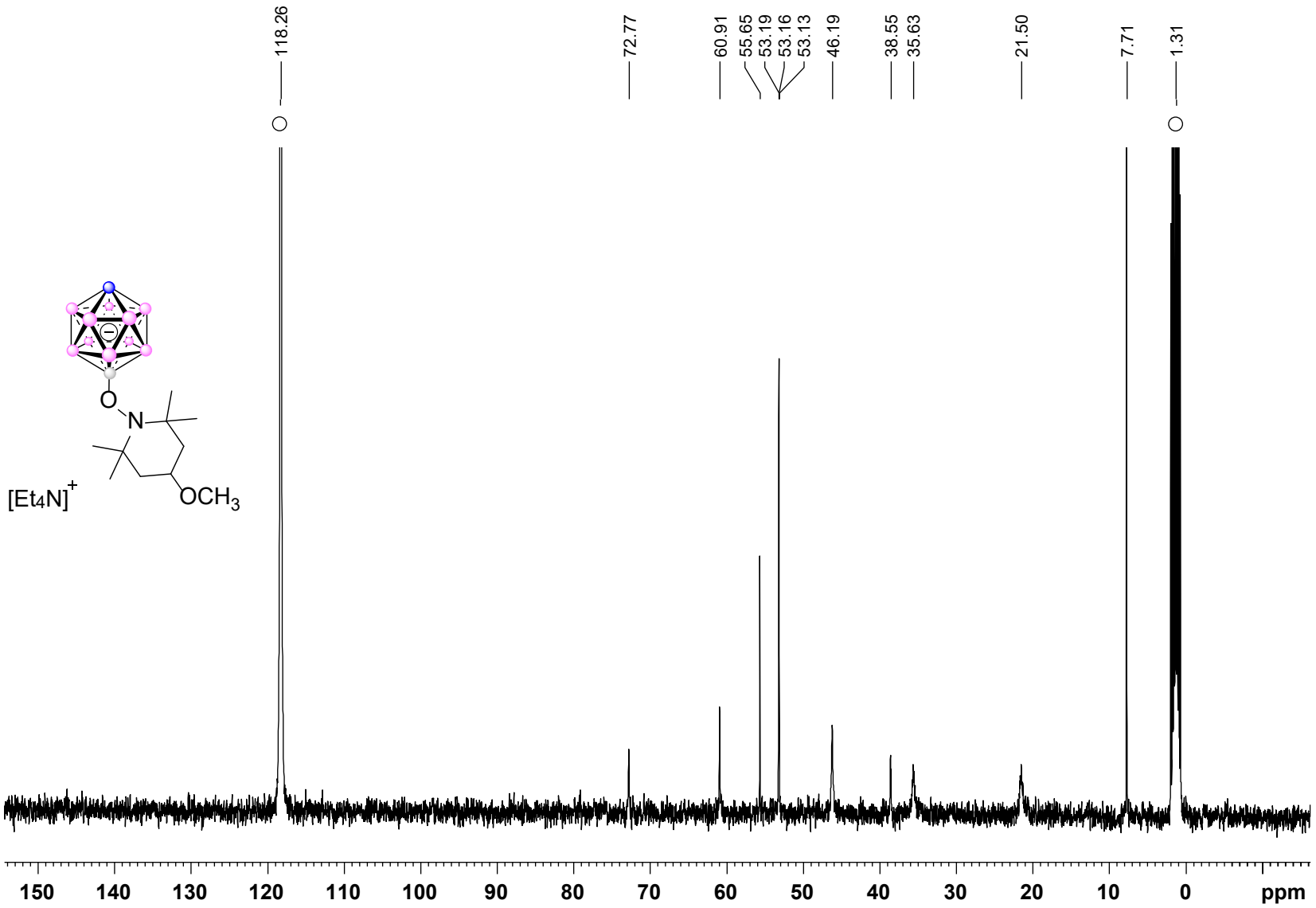

```
Current Data Parameters
NAME      S105-1-3-O-CH3
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240110
Time               19.38
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                298.1 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
```

S105-1-3 [NEt4][12-CH3-O-TEMPO-CB11H11]
11B{1H} 128MHZ CD3CN T=23 C



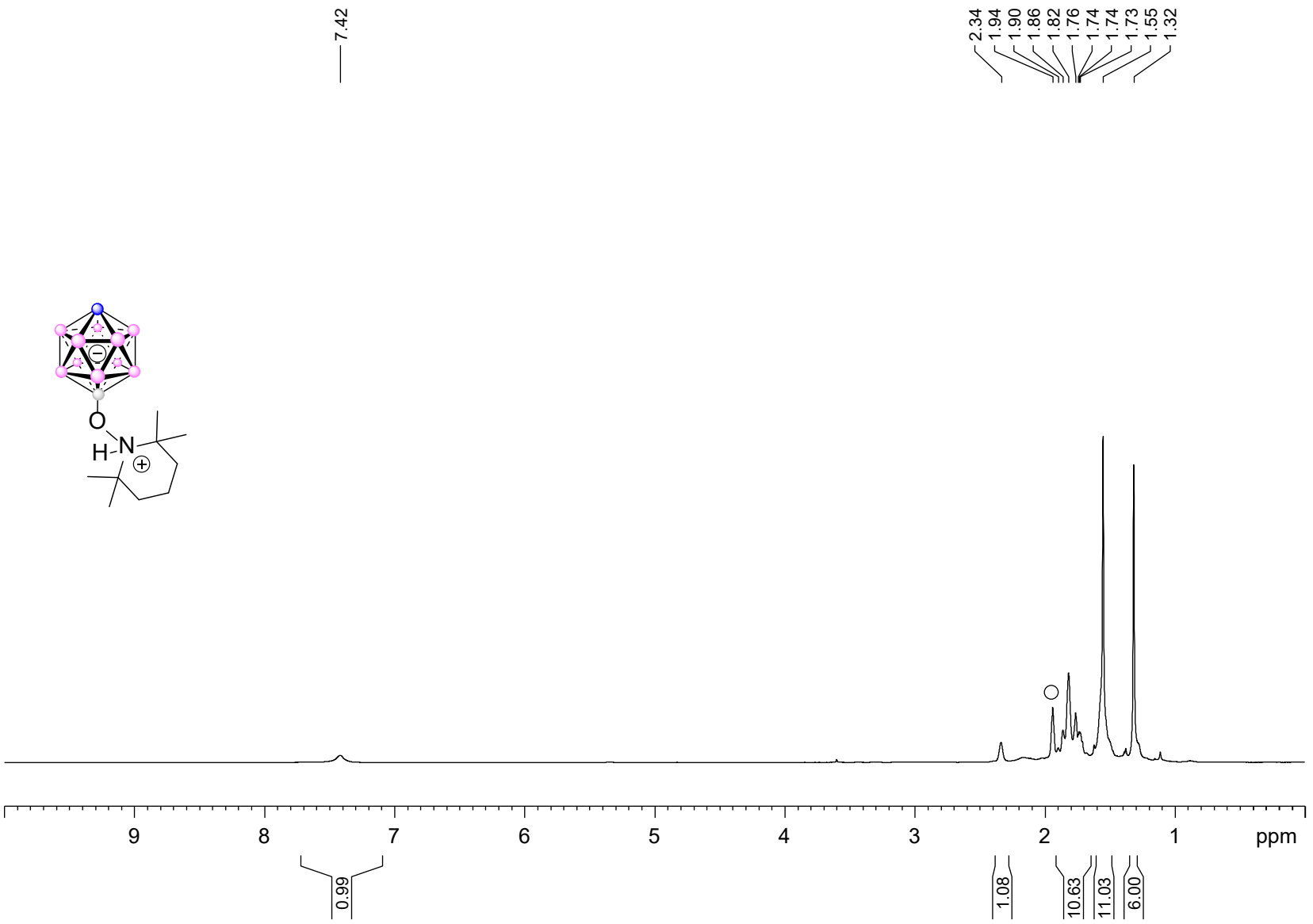

```
Current Data Parameters
NAME      S105-1-3-O-CH3
EXPNO                 4
PROCNO                1

F2 - Acquisition Parameters
Date_          20240110
Time              19.44
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              299.2 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        15.48099995 W
PLW12        0.54425001 W
PLW13        0.34832001 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
```

S105-1-3 [NEt4][12-CH3-O-TEMPO-CB11H11]
13C{1H} 100MHZ CD3CN T=23 C



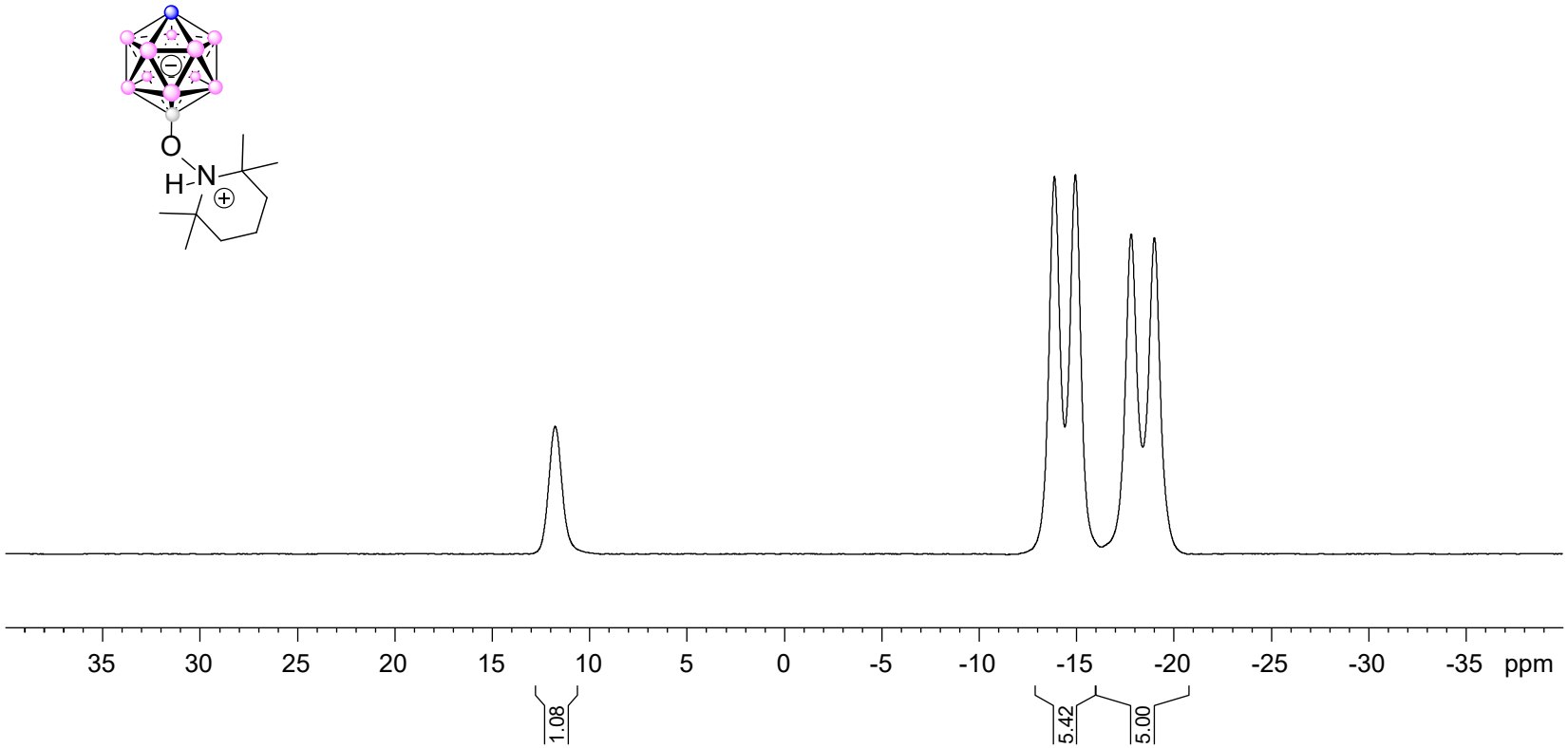

```
Current Data Parameters
NAME      S105-1-3-O-CH3
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240110
Time              20.08
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT          CD3CN
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              299.5 K
D1          1.50000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1               10.00 usec
PLW1        66.13099670 W
SFO1       100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2            80.00 usec
PLW2        15.48099995 W
PLW12        0.54425001 W
PLW13        0.34832001 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF         100.6126674 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.40
```

S144-4 [H][TEMPO-CB11H11]
1H{11B} 400MHZ CD3CN T=23 C



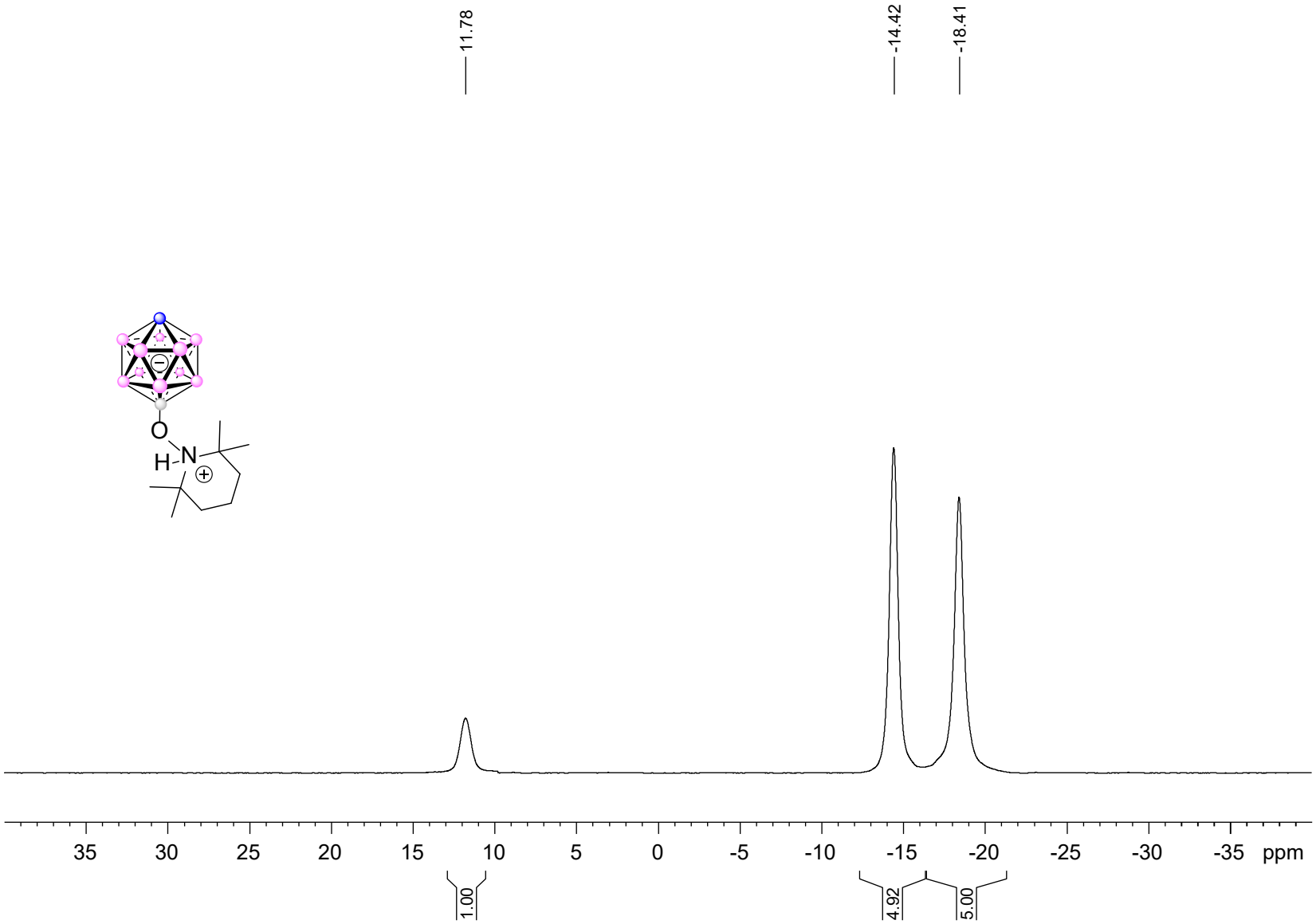

```
Current Data Parameters
NAME         S144-4-OR-0
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240307
Time               22.31
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                107.6
DW               62.400 usec
DE                6.50 usec
TE                298.4 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PLW1        15.48099995 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          400.1300108 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

S144-4 [H][TEMPO-CB11H11]
11B 128MHZ CD3CN T=23 C



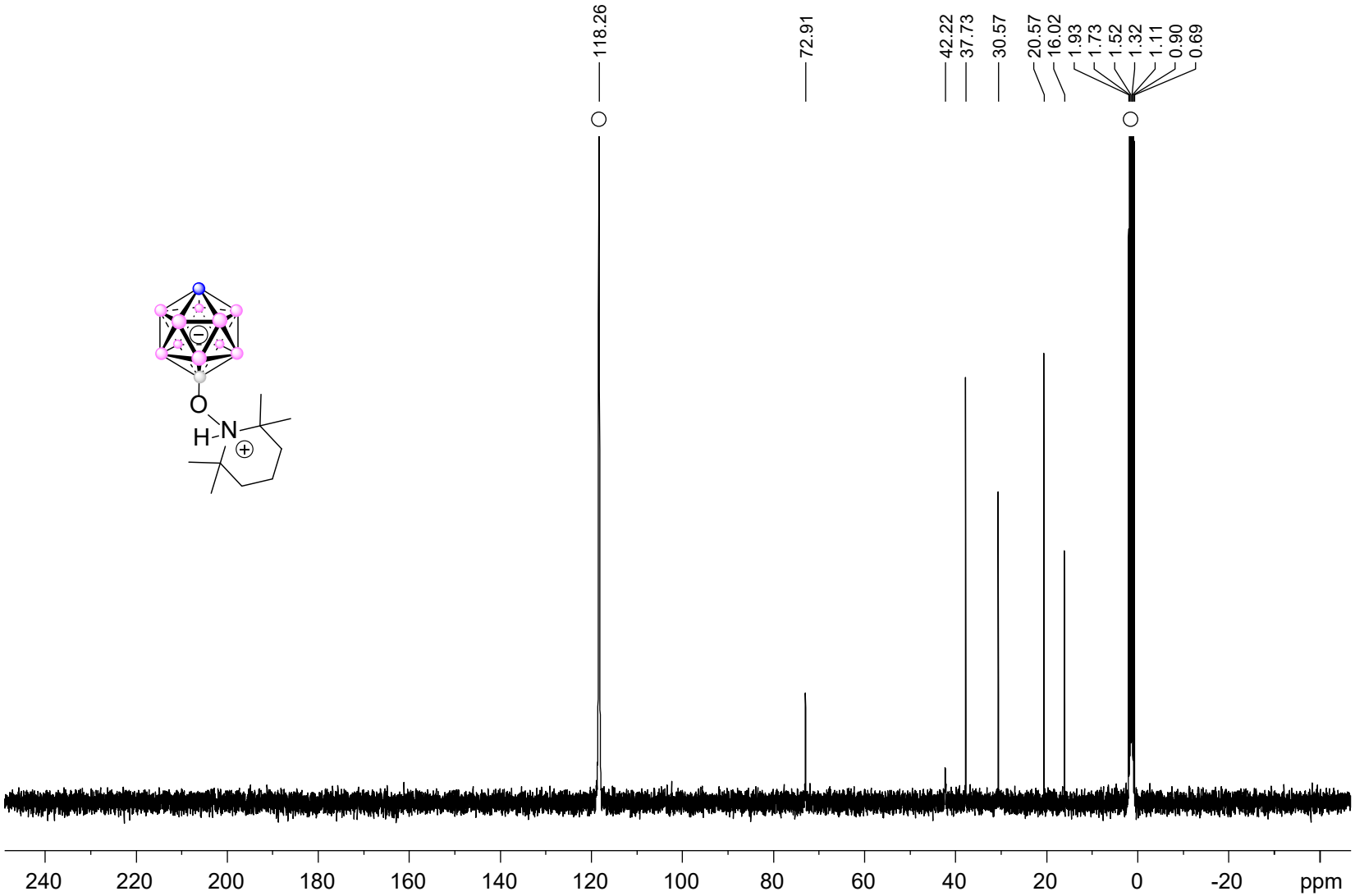

```
Current Data Parameters
NAME         S144-4-OR-0
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240307
Time               22.36
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                298.1 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                 10.00 Hz
GB                     0
PC                  1.40
```

S144-4 [H][TEMPO-CB11H11]
11B{1H} 128MHZ CD3CN T=23 C



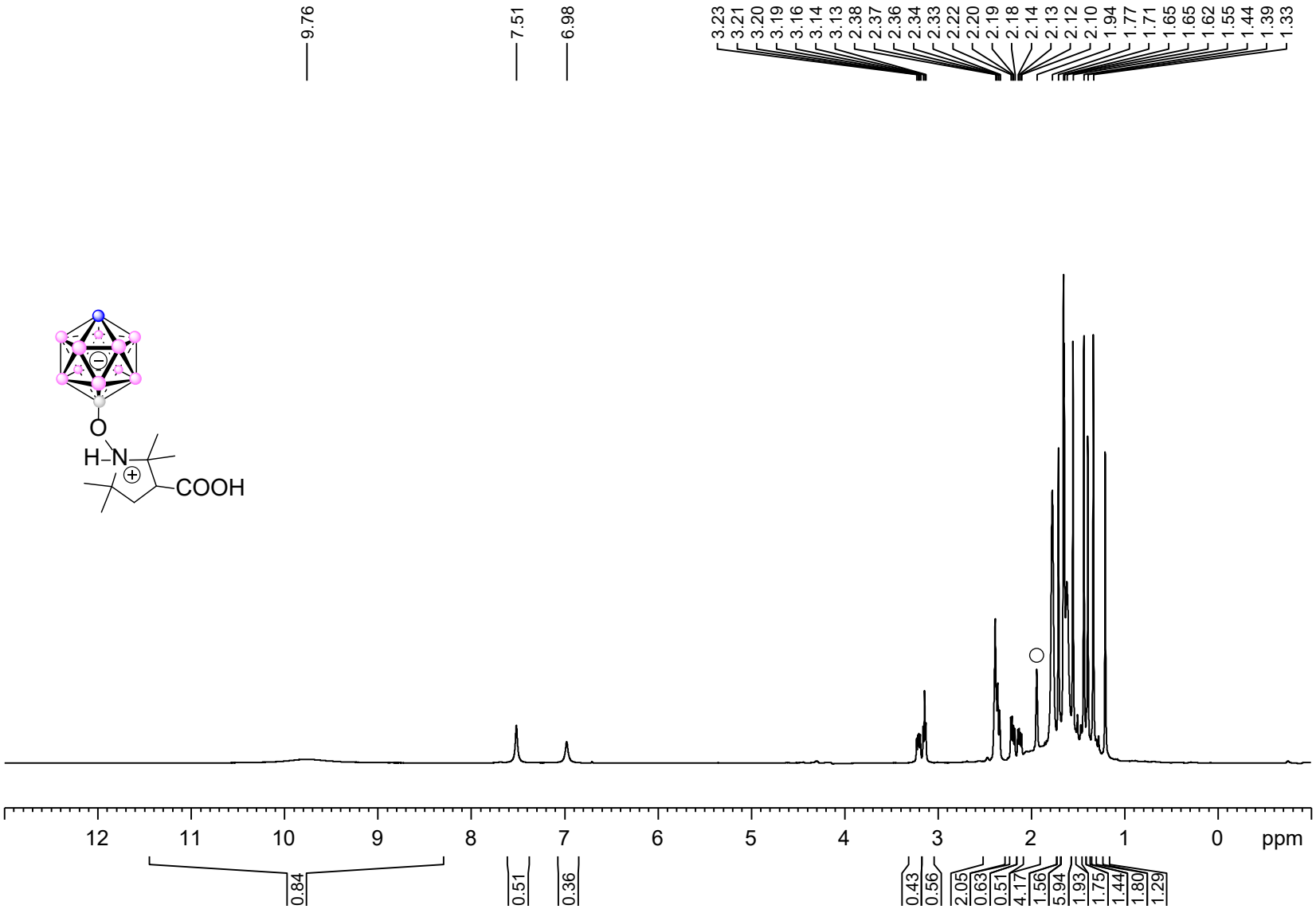

```
Current Data Parameters
NAME         S144-4-OR-0
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240307
Time              22.43
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              299.3 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        15.48099995 W
PLW12        0.54425001 W
PLW13        0.34832001 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
```

S144-4 [H][TEMPO-CB11H11]
13C{1H} 100MHZ CD3CN T=23 C



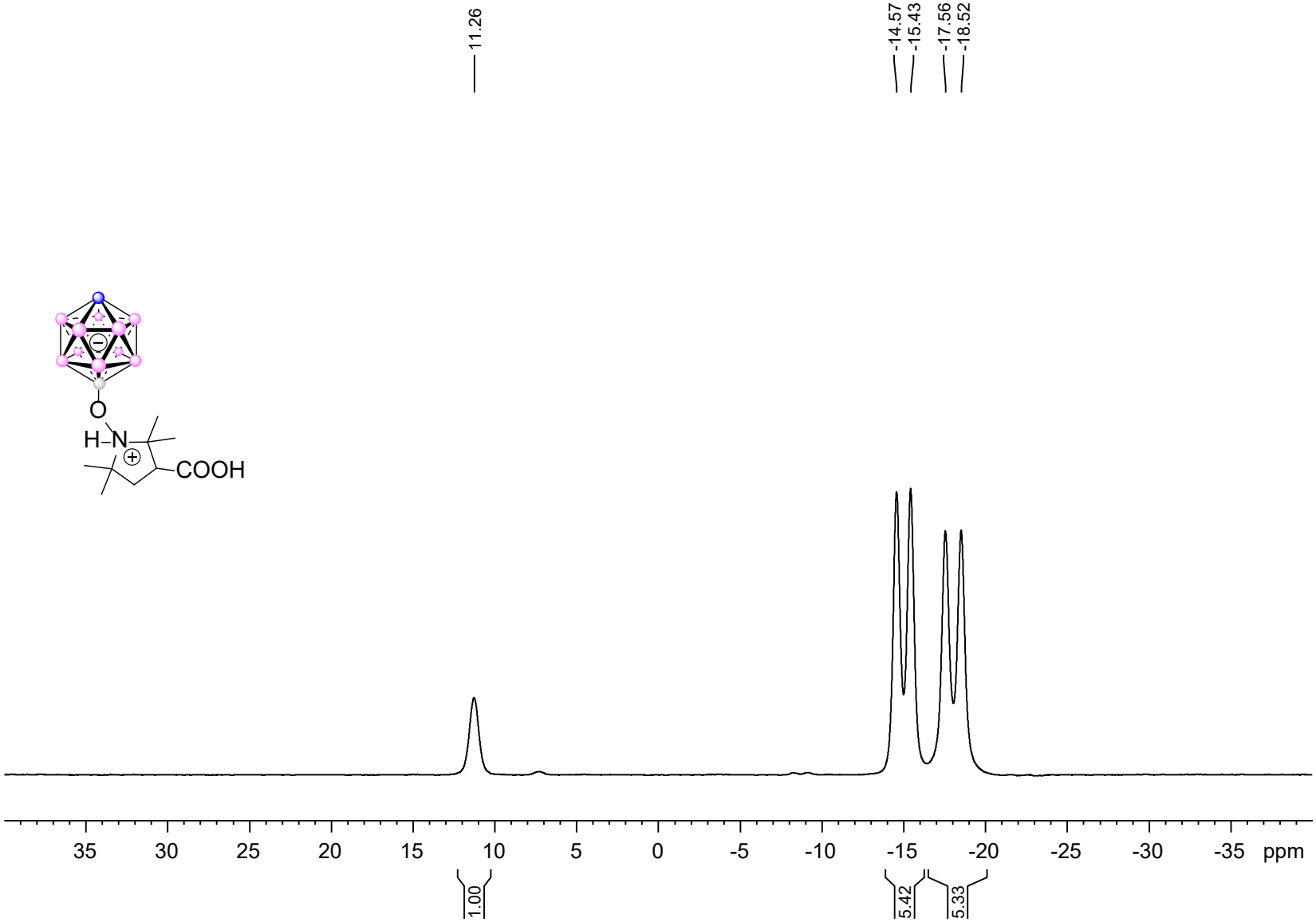

```
Current Data Parameters
NAME         S144-4-OR-0
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240313
Time              17.19
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT           CD3CN
NS                  512
DS                    4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010048 sec
RG               152.18
DW               16.800 usec
DE                6.50 usec
TE               299.5 K
D1          1.50000000 sec
D11         0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PLW1        66.13099670 W
SFO1       100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PLW2        15.48099995 W
PLW12        0.54425001 W
PLW13        0.34832001 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                32768
SF         100.6126684 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

U021-3 [H][12-COOH-PROXYL-CB11H11]
1H{11B} 500MHZ CD3CN T=23 C



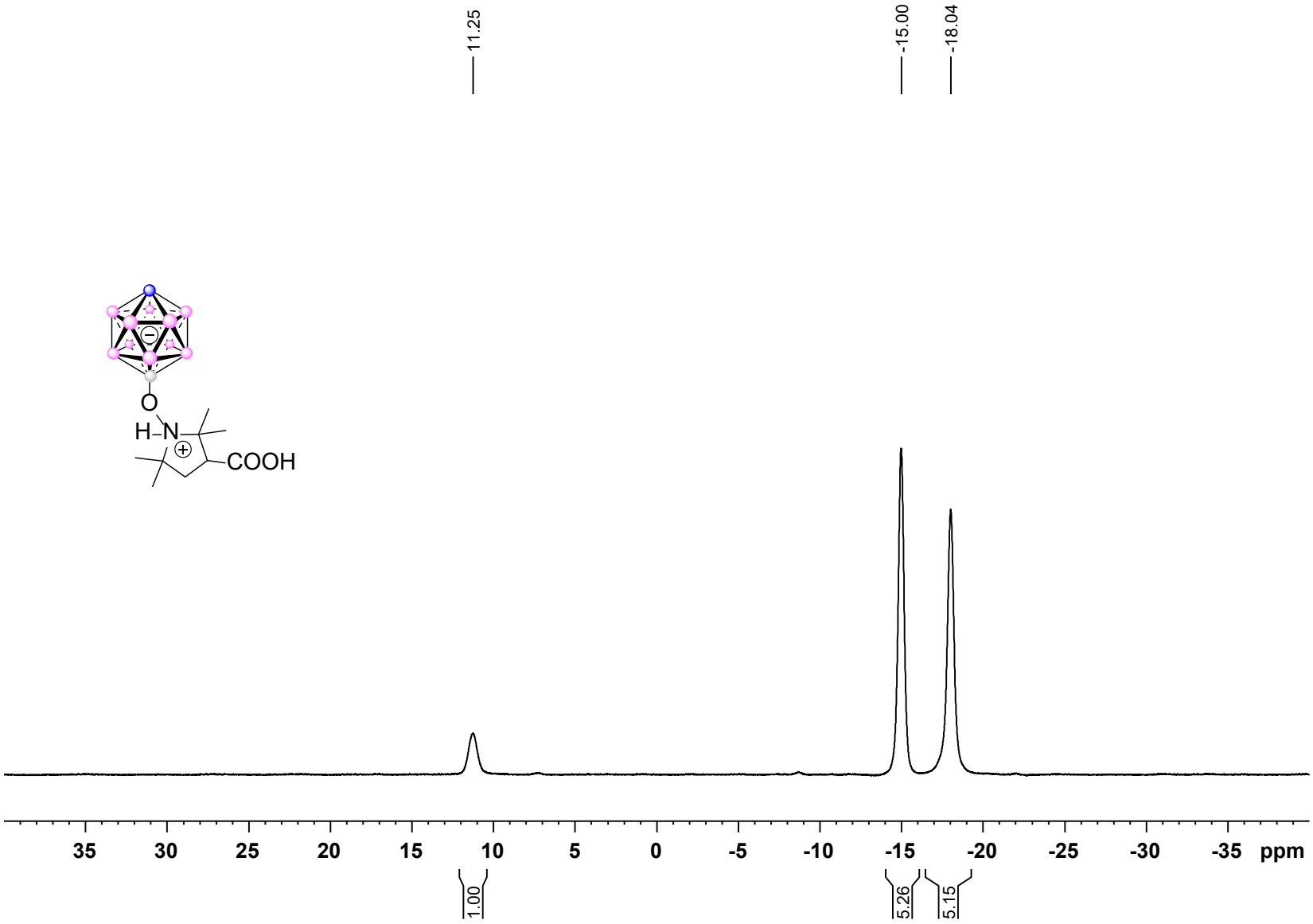

```
Current Data Parameters
NAME      U021-3-OR_PROXYL
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20240531
Time                13.57
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                  297.7 K
D1             5.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          500.1735009 MHz
NUC1                    1H
P1                   11.85 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            500.1700139 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.00
```

U021-3 [H][12-COOH-PROXYL-CB11H11]
11B 160MHZ CD3CN T=23 C



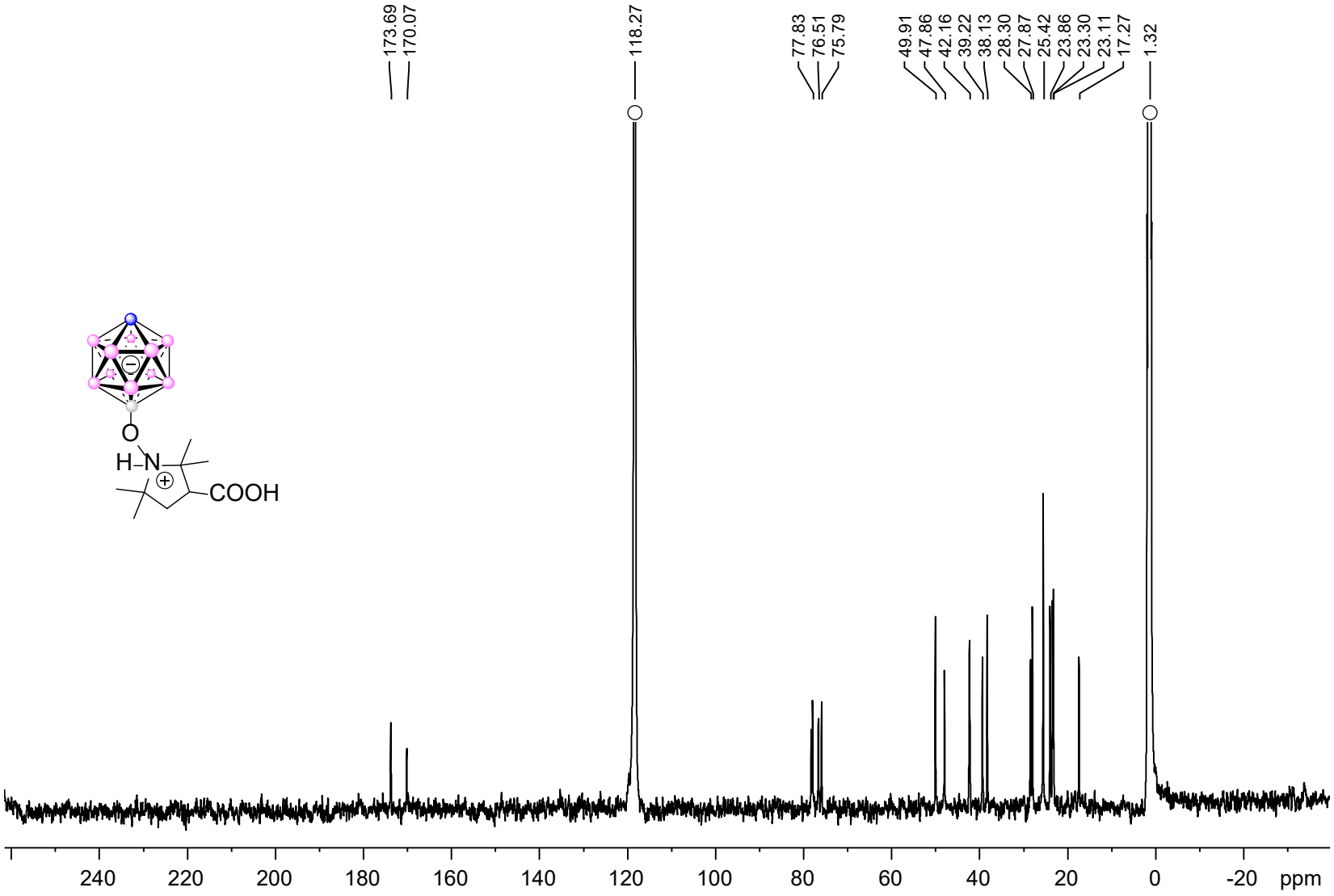

```
Current Data Parameters
NAME      U021-3-OR_PROXYL
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_           20240531
Time              14.01
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               64098
SOLVENT          CD3CN
NS                  96
DS                   0
SWH          32051.281 Hz
FIDRES        0.500036 Hz
AQ           0.9999288 sec
RG                 203
DW              15.600 usec
DE               6.50 usec
TE              297.6 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        160.4744132 MHz
NUC1               11B
P1              13.10 usec
PLW1        95.00000000 W

F2 - Processing parameters
SI               32768
SF          160.4744130 MHz
WDW                 EM
SSB                  0
LB              10.00 Hz
GB                   0
PC                1.40
```

U021-3 [H][12-COOH-PROXYL-CB11H11]
11B{1H} 160MHZ CD3CN T=23 C



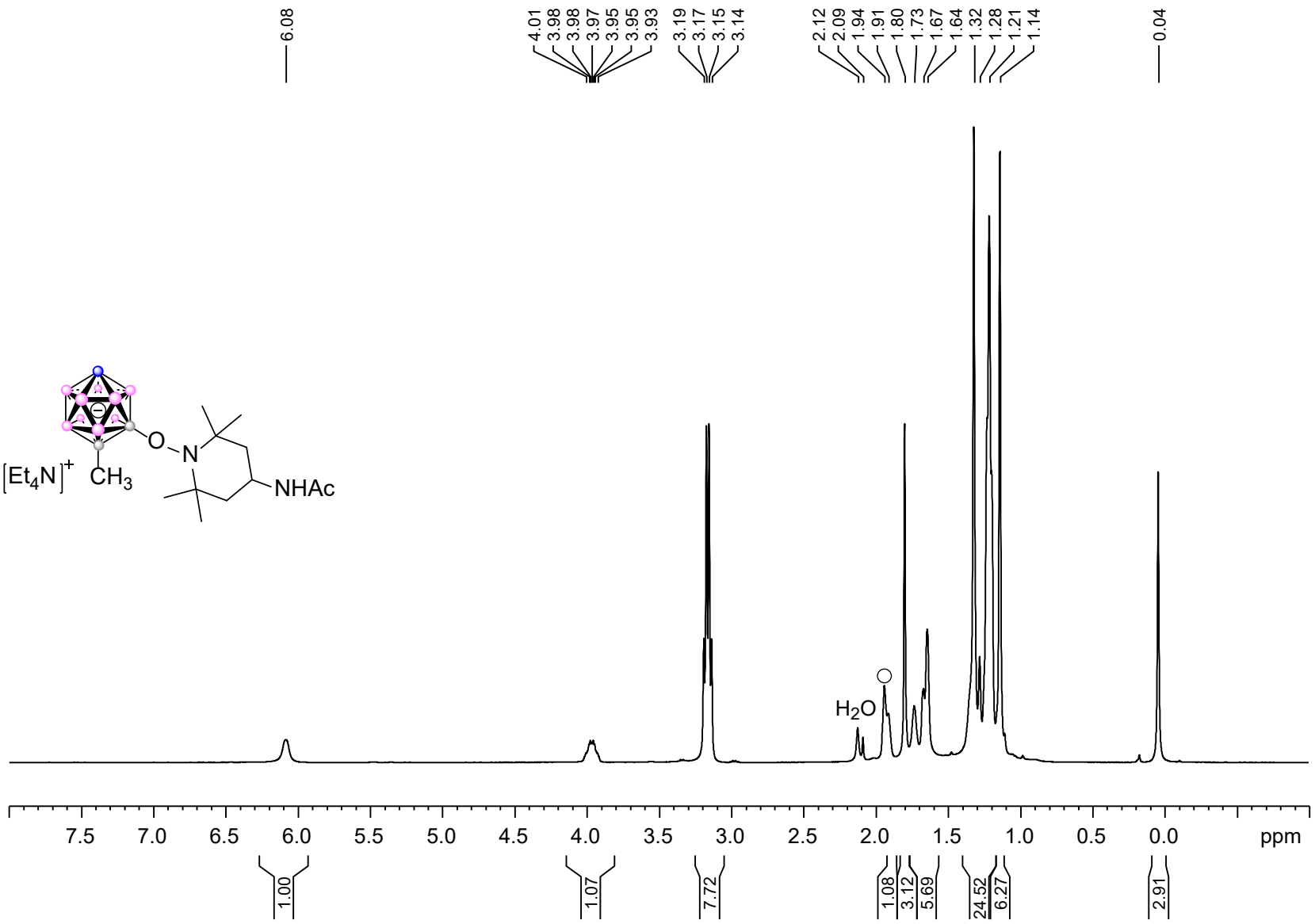

```
Current Data Parameters
NAME      U021-3-OR_PROXYL
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_            20240531
Time                14.05
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CD3CN
NS                     96
DS                      0
SWH             32051.281 Hz
FIDRES          0.489064 Hz
AQ              1.0223616 sec
RG                    203
DW                15.600 usec
DE                 6.50 usec
TE                297.9 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         160.4744130 MHz
NUC1                  11B
P1                  13.10 usec
PLW1         95.00000000 W

======== CHANNEL f2 ========
SFO2         500.1725007 MHz
NUC2                   1H
CPDPRG[2         waltz16
PCPD2              80.00 usec
PLW2         19.00000000 W
PLW12         0.40639001 W
PLW13         0.26008999 W

F2 - Processing parameters
SI                  32768
SF           160.4744130 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

U021-3 [H][12-COOH-PROXYL-CB11H11]
13C 126MHZ CD3CN T=23 C



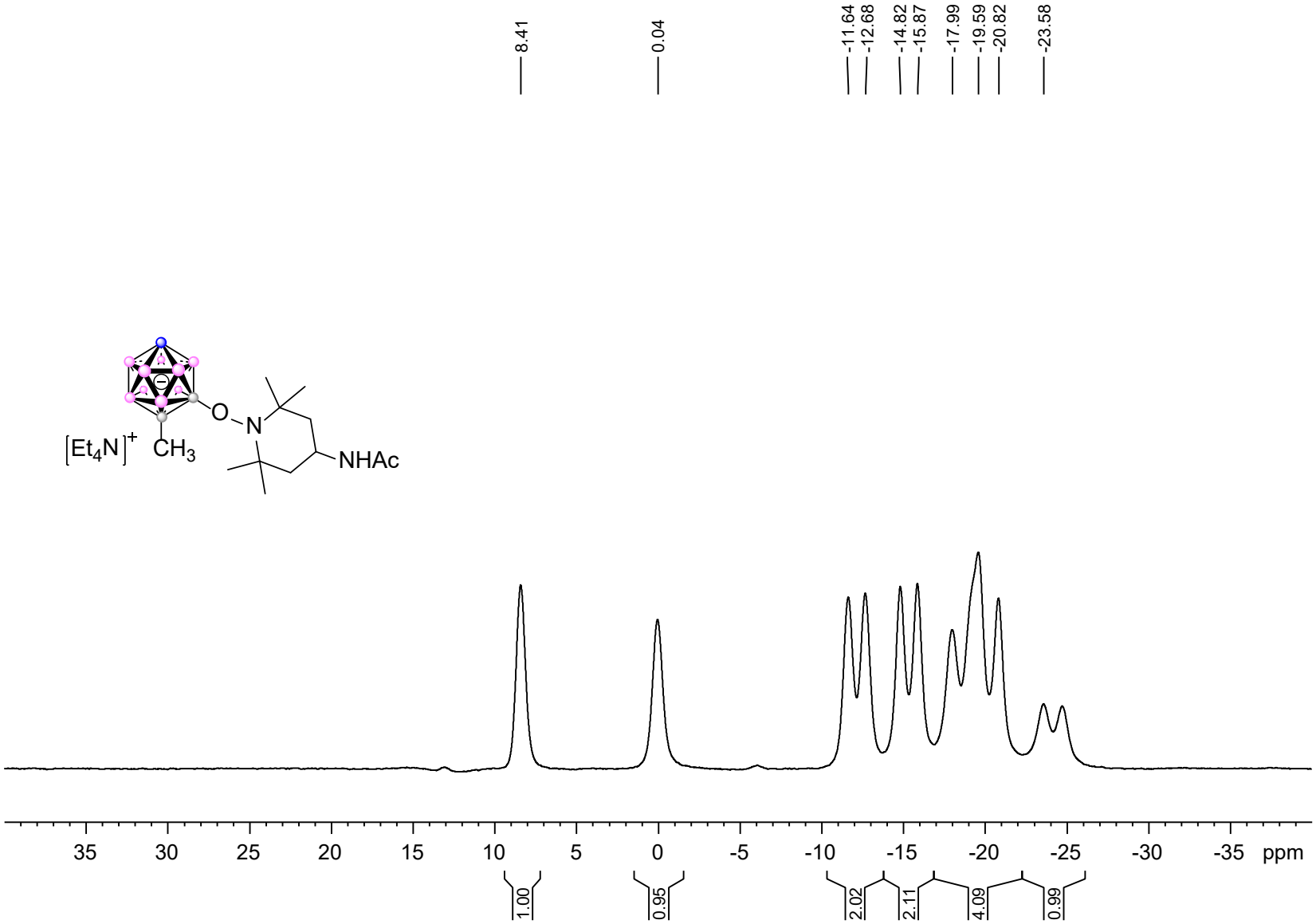

```
Current Data Parameters
NAME      U021-3-OR_PROXYL
EXPNO                    5
PROCNO                   1

F2 - Acquisition Parameters
Date_             20240531
Time                 14.26
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CD3CN
NS                     530
DS                       4
SWH              37878.789 Hz
FIDRES           0.577984 Hz
AQ               0.8650752 sec
RG                     203
DW                13.200 usec
DE                 6.50 usec
TE                 298.0 K
D1            1.50000000 sec
D11           0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1         125.7816804 MHz
NUC1                  13C
P1                 10.70 usec
PLW1         95.00000000 W

======== CHANNEL f2 ========
SFO2         500.1720005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2         19.00000000 W
PLW12         0.40639001 W
PLW13         0.26008999 W

F2 - Processing parameters
SI                   32768
SF           125.7677257 MHz
WDW                     EM
SSB                      0
LB                 10.00 Hz
GB                       0
PC                    1.40
```

U074-7 [NEt4][12-CH3-7-NHAc-TEMPO-CB11H11]
1H{11B} 400MHZ CD3CN T=23 C



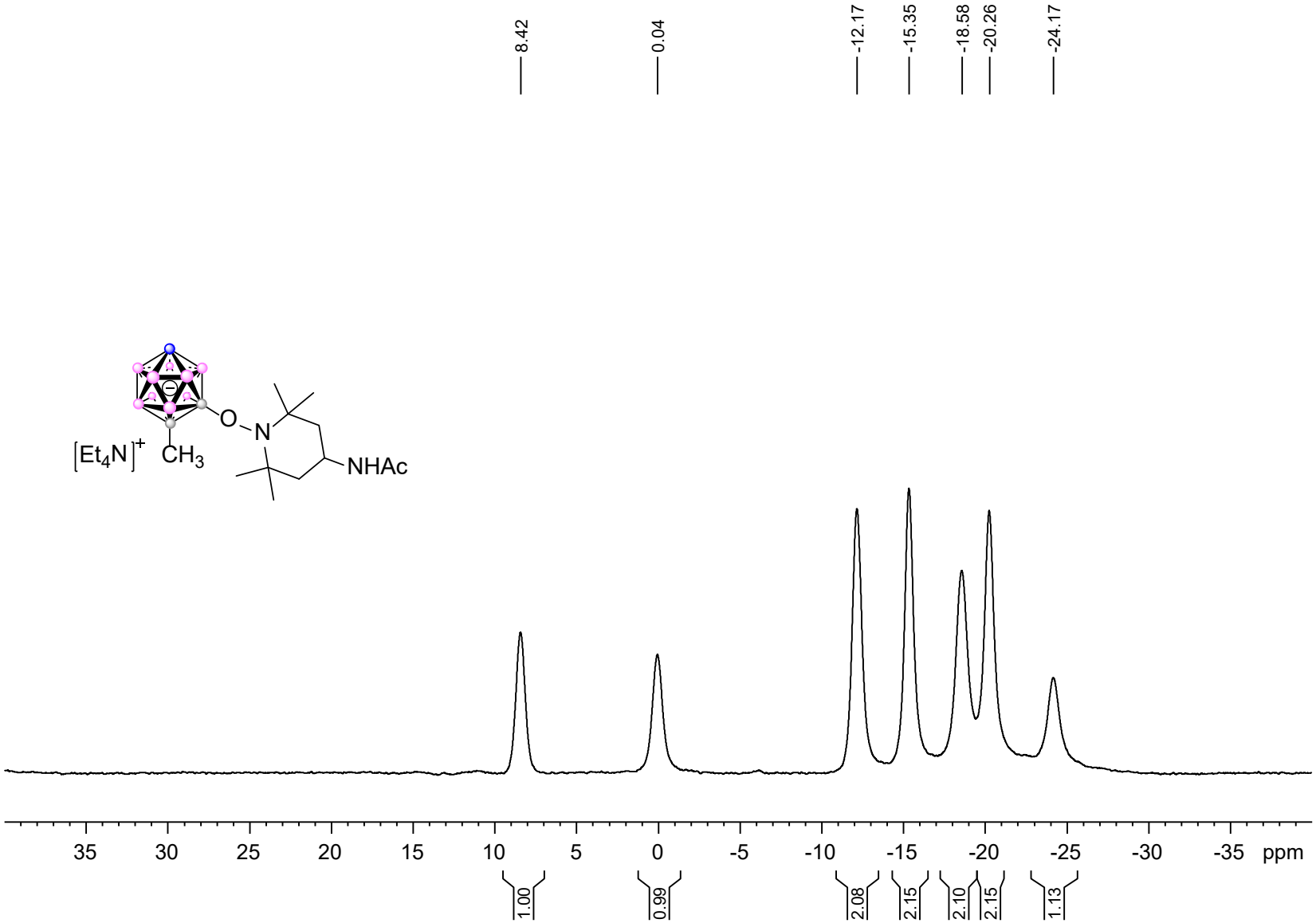

```
Current Data Parameters
NAME                  U074-7
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_              20240901
Time                  12.14 h
INSTRUM                spect
PROBHD    Z108618_0497 (
PULPROG               zgig30
TD                     16384
SOLVENT                CD3CN
NS                        16
DS                         4
SWH                8012.820 Hz
FIDRES            0.978127 Hz
AQ               1.0223616 sec
RG                     107.6
DW                  62.400 usec
DE                   6.50 usec
TE                   301.4 K
D1              1.00000000 sec
D11             0.03000000 sec
TD0                        1
SFO1           400.1320007 MHz
NUC1                      1H
P1                   15.00 usec
PLW1           15.48099995 W
SFO2           128.3776050 MHz
NUC2                      11B
CPDPRG[2               garp4
PCPD2                60.00 usec
PLW2           52.96599960 W
PLW12           1.45079994 W


SI                     32768

WDW                       EM
SSB                        0

GB                         0
PC                      1.40
```

U074-7 [NEt4][12-CH3-7-NHAc-TEMPO-CB11H11]
11B 128MHZ CD3CN T=23 C



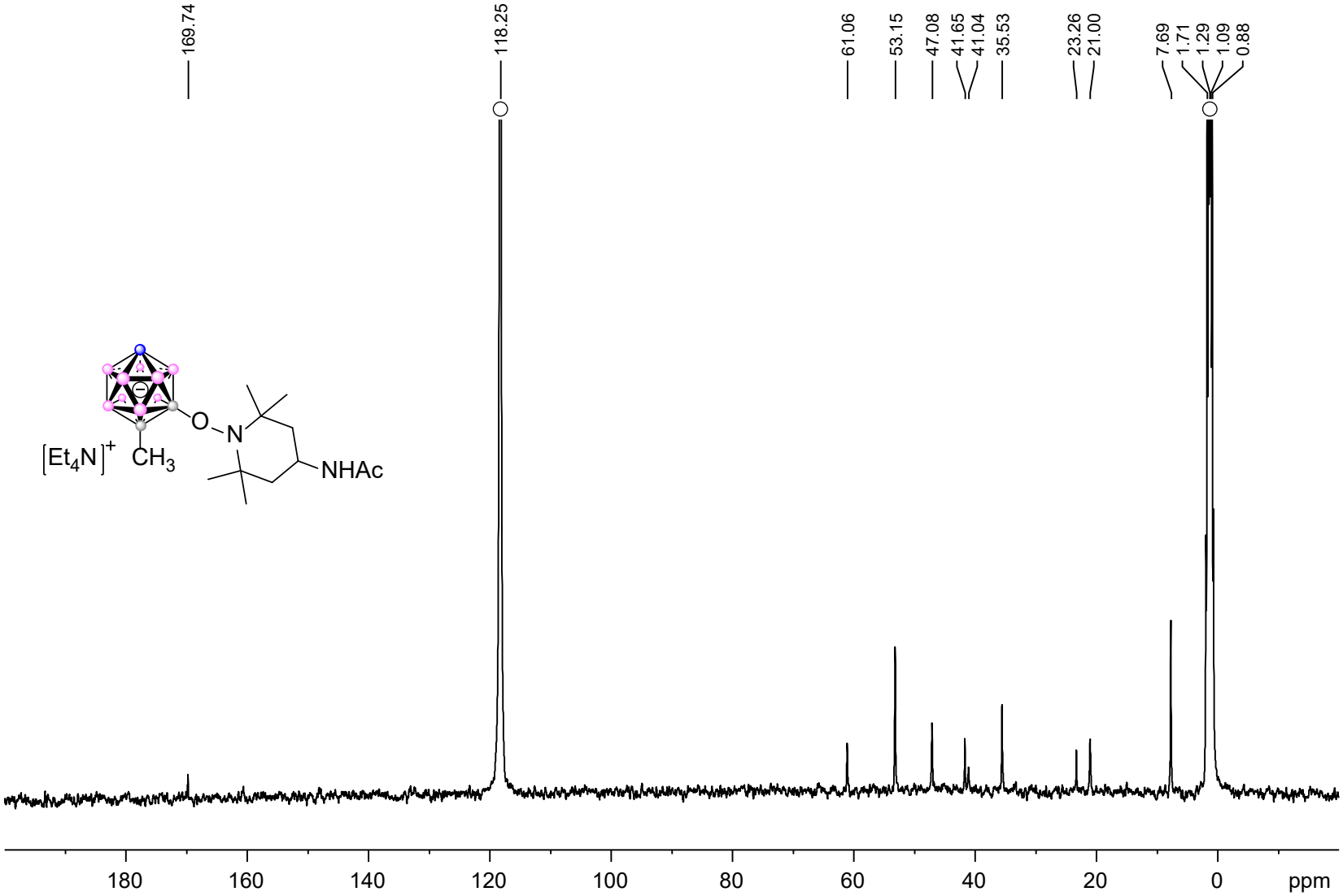

```
Current Data Parameters
NAME                 U074-7
EXPNO                     3
PROCNO                    1

F2 - Acquisition Parameters
Date_              20240901
Time                 12.20 h
INSTRUM              spect
PROBHD     Z108618_0497 (
PULPROG                 zg
TD                   65536
SOLVENT              CD3CN
NS                     128
DS                       4
SWH              25510.203 Hz
FIDRES           0.778510 Hz
AQ               1.2845056 sec
RG                  193.34
DW                  19.600 usec
DE                   6.50 usec
TE                  301.2 K
D1              1.00000000 sec
TD0                       1
SFO1           128.3776052 MHz
NUC1                   11B
P1                    9.93 usec
PLW1           52.96599960 W

F2 - Processing parameters
SI                    32768
SF             128.3776052 MHz
WDW                     EM
SSB                      0
LB                   10.00 Hz
GB                       0
PC                    1.40
```

U074-7 [NEt4][12-CH3-7-NHAc-TEMPO-CB11H11]
11B{1H} 128MHZ CD3CN T=23 C



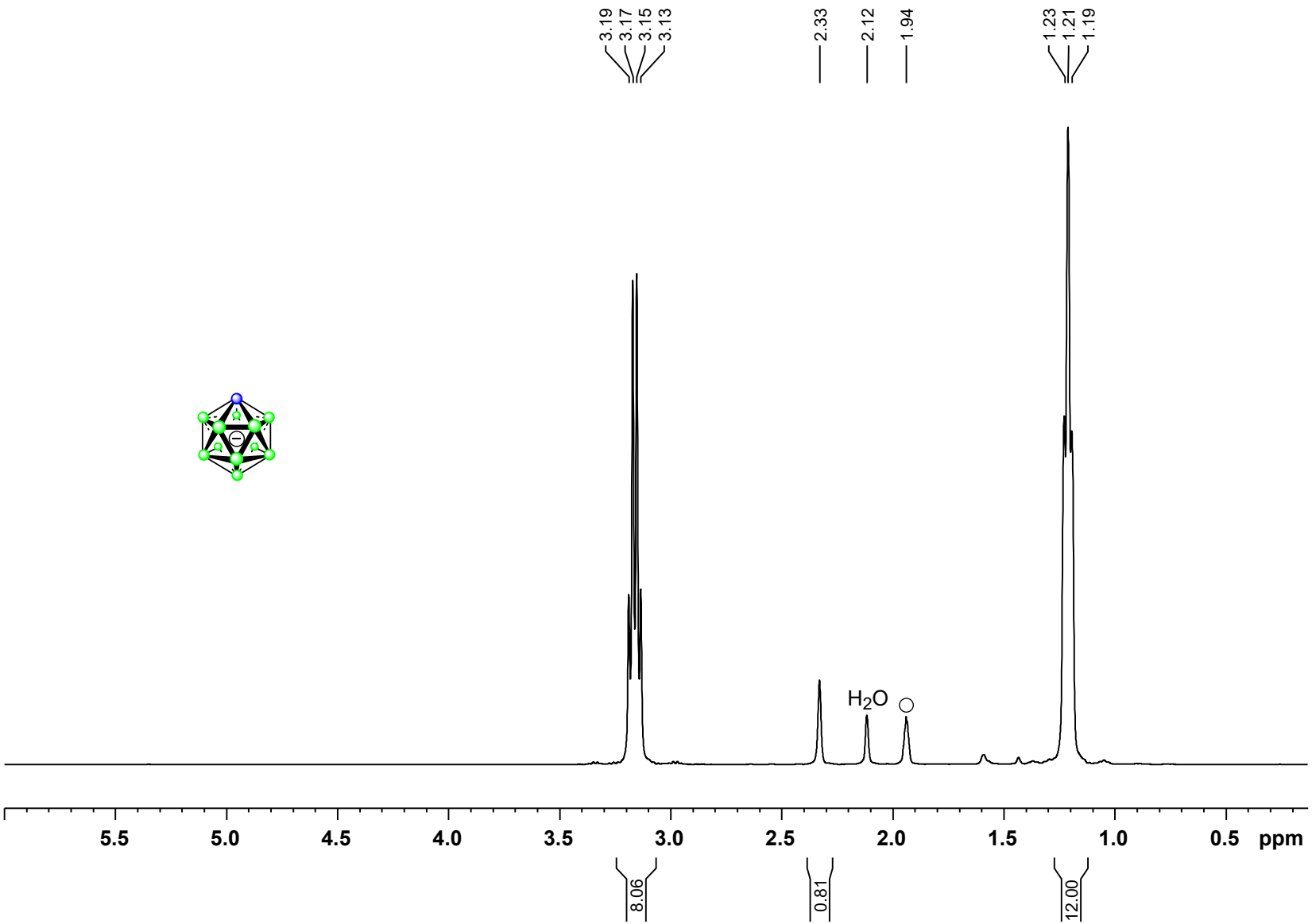

```
Current Data Parameters
NAME                  U074-7
EXPNO                      4
PROCNO                     1

F2 - Acquisition Parameters
Date_              20240901
Time                   12.26 h
INSTRUM                spect
PROBHD    Z108618_0497 (
PULPROG              zgpg30
TD                    65536
SOLVENT               CD3CN
NS                      128
DS                        4
SWH               25510.203 Hz
FIDRES             0.778510 Hz
AQ                 1.2845056 sec
RG                    193.34
DW                   19.600 usec
DE                    6.50 usec
TE                   302.0 K
D1                1.00000000 sec
D11               0.03000000 sec
TD0                       1
SFO1            128.3776050 MHz
NUC1                    11B
P1                    9.93 usec
PLW1             52.96599960 W
SFO2            400.1320007 MHz
NUC2                    1H
CPDPRG[2             waltz16
PCPD2                90.00 usec
PLW2             15.48099995 W
PLW12             0.43002000 W
PLW13             0.21630000 W

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

U074-7 [NEt4][12-CH3-7-NHAc-TEMPO-CB11H11]
13C 100MHZ CD3CN T=23 C



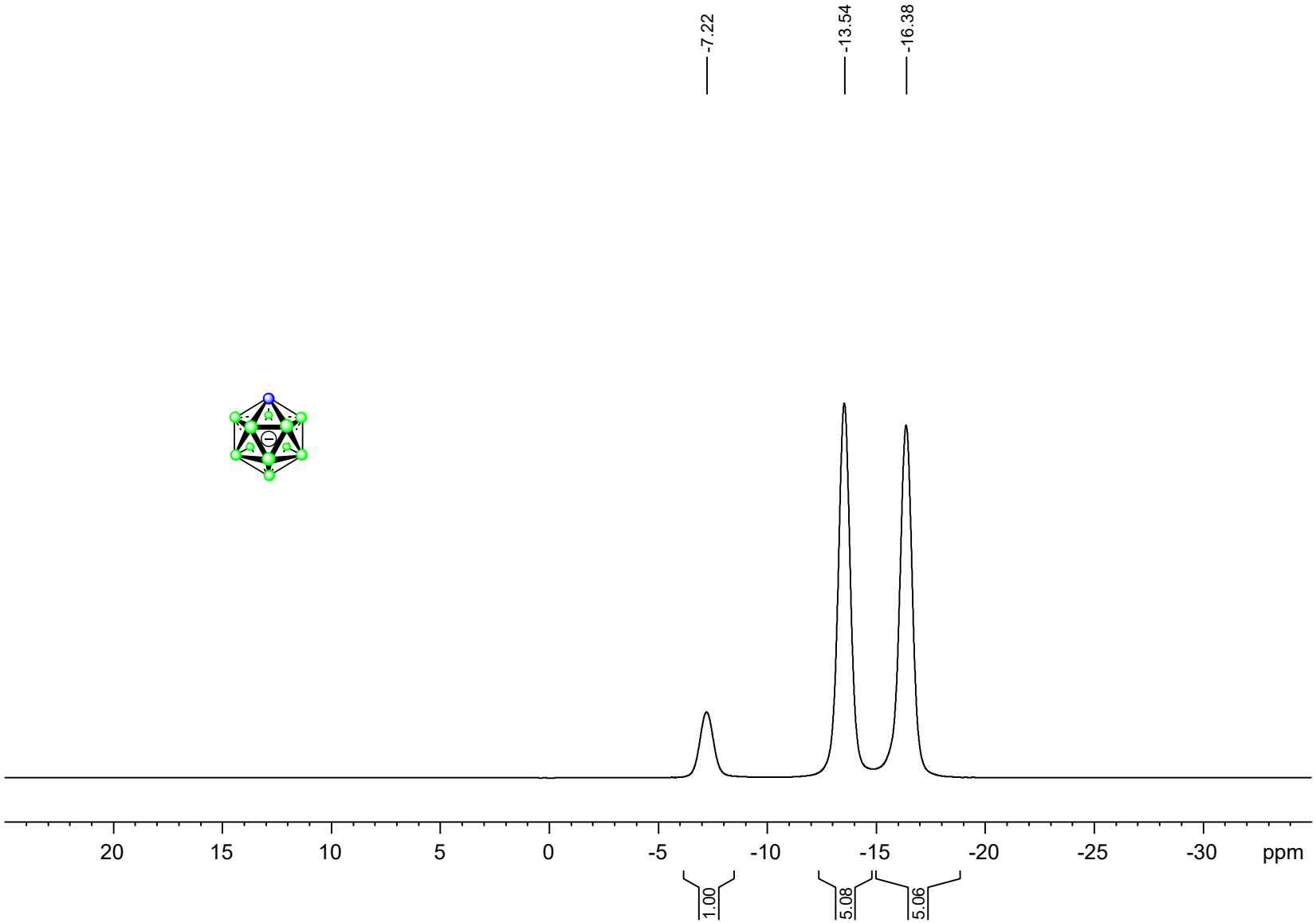

```
Current Data Parameters
NAME                  U074-7
EXPNO                      9
PROCNO                     1

F2 - Acquisition Parameters
Date_              20240901
Time                  20.38 h
INSTRUM                spect
PROBHD    Z108618_0497 (
PULPROG              zgpg30
TD                    65536
SOLVENT               CD3CN
NS                      512
DS                        4
SWH               29761.904 Hz
FIDRES             0.908261 Hz
AQ                1.1010048 sec
RG                   193.34
DW                   16.800 usec
DE                    6.50 usec
TE                   302.7 K
D1             1.50000000 sec
D11            0.03000000 sec
TD0                       1
SFO1          100.6228293 MHz
NUC1                   13C
P1                   10.00 usec
PLW1          66.13099670 W
SFO2          400.1316005 MHz
NUC2                   1H
CPDPRG[2           waltz16
PCPD2                90.00 usec
PLW2          15.48099995 W
PLW12          0.43002000 W
PLW13          0.21630000 W

F2 - Processing parameters
SI                    32768
SF            100.6126675 MHz
WDW                     EM
SSB                      0
LB                   10.00 Hz
GB                       0
PC                    1.40
```

[NEt4][H-CB11D11]
1H{11B} 400MHZ CD3CN T=23 C



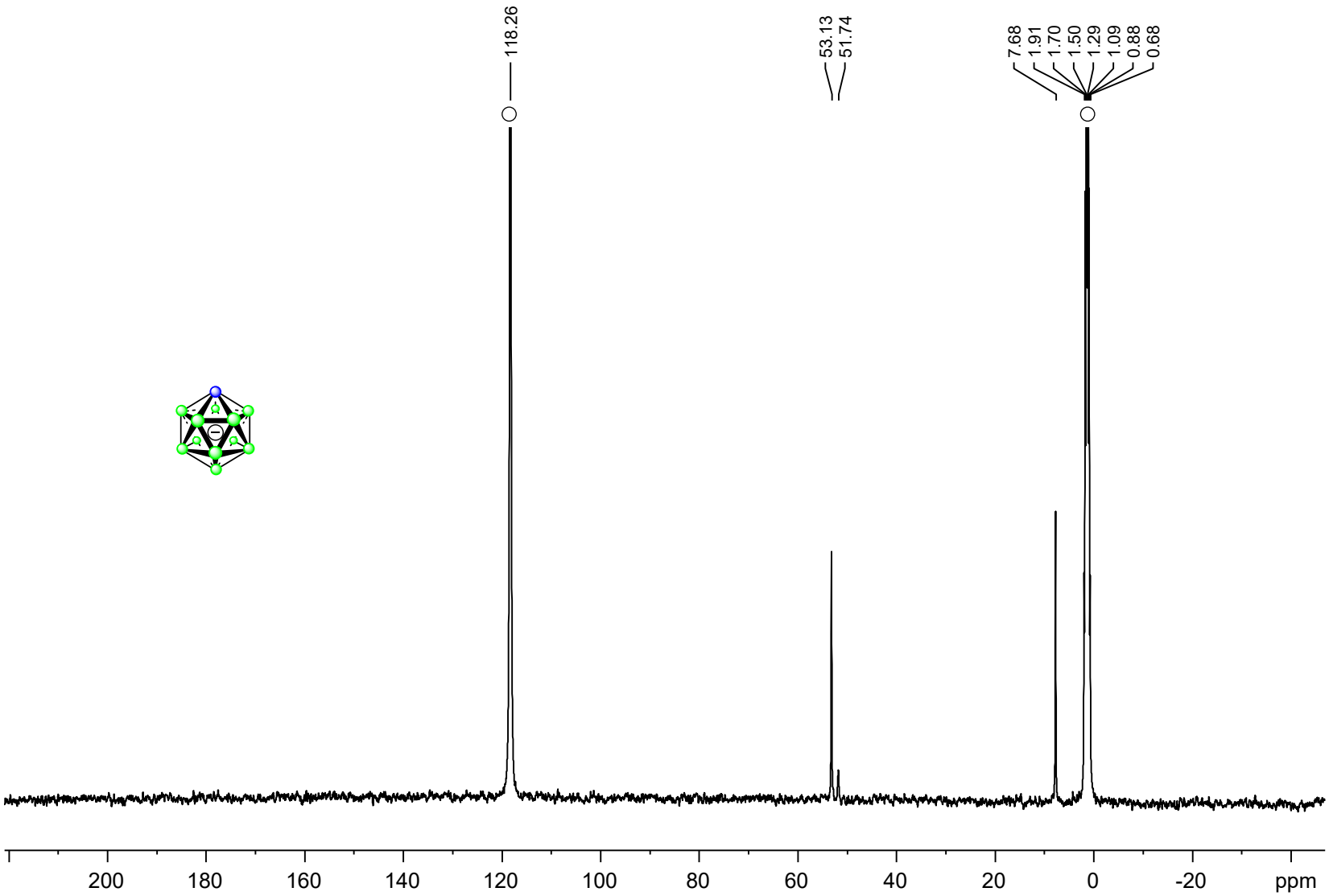

```
Current Data Parameters
NAME      U091-2-CB11D11
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20241101
Time               11.56 h
INSTRUM            spect
PROBHD   Z108618_0497 (
PULPROG          zgig30
TD                16384
SOLVENT           CD3CN
NS                   16
DS                    4
SWH             8012.820 Hz
FIDRES          0.978127 Hz
AQ             1.0223616 sec
RG                170.36
DW                62.400 usec
DE                 6.50 usec
TE                299.7 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1         400.1320007 MHz
NUC1                 1H
P1                15.00 usec
PLW1         15.48099995 W
SFO2         128.3776050 MHz
NUC2                 11B
CPDPRG[2           garp4

PLW2         52.96599960 W
PLW12         1.45079994 W

F2 - Processing parameters
SI                32768

WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

[NEt4][H-CB11D11]
11B 128MHZ CD3CN T=23 C



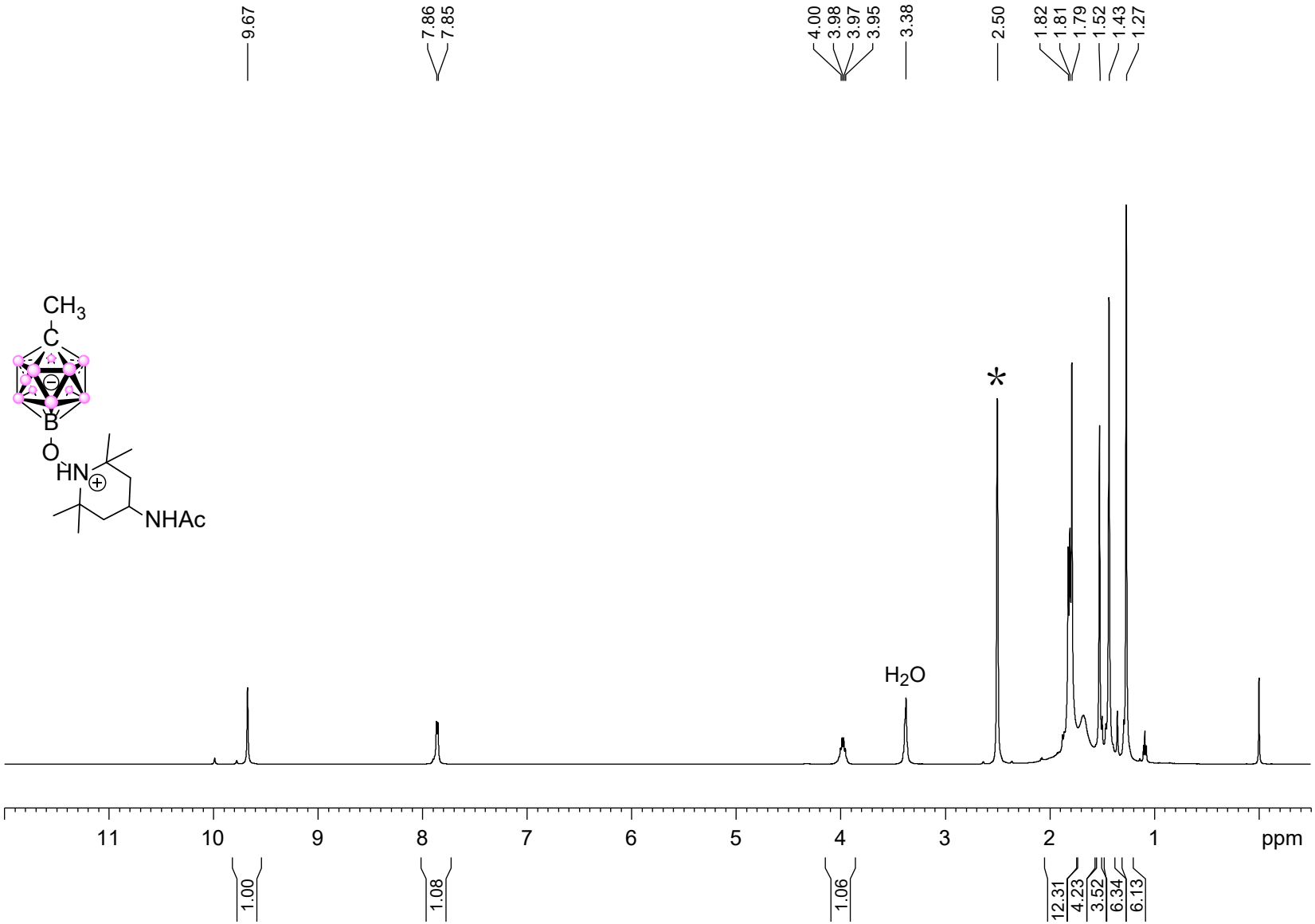

```
Current Data Parameters
NAME      U091-2-CB11D11
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20241101
Time              12.01 h
INSTRUM            spect
PROBHD   Z108618_0497 (
PULPROG              zg
TD                65536
SOLVENT           CD3CN
NS                  128
DS                    4
SWH           25510.203 Hz
FIDRES         0.778510 Hz
AQ             1.2845056 sec
RG                193.34
DW                19.600 usec
DE                 6.50 usec
TE                299.4 K
D1            1.00000000 sec
TD0                   1
SFO1        128.3776052 MHz
NUC1               11B
P1                 9.93 usec
PLW1        52.96599960 W

F2 - Processing parameters
SI                32768
SF          128.3776052 MHz
WDW                EM
SSB                0
LB                10.00 Hz
GB                 0
PC                1.40
```

[NEt4][H-CB11D11]
13C 100MHZ CD3CN T=23 C



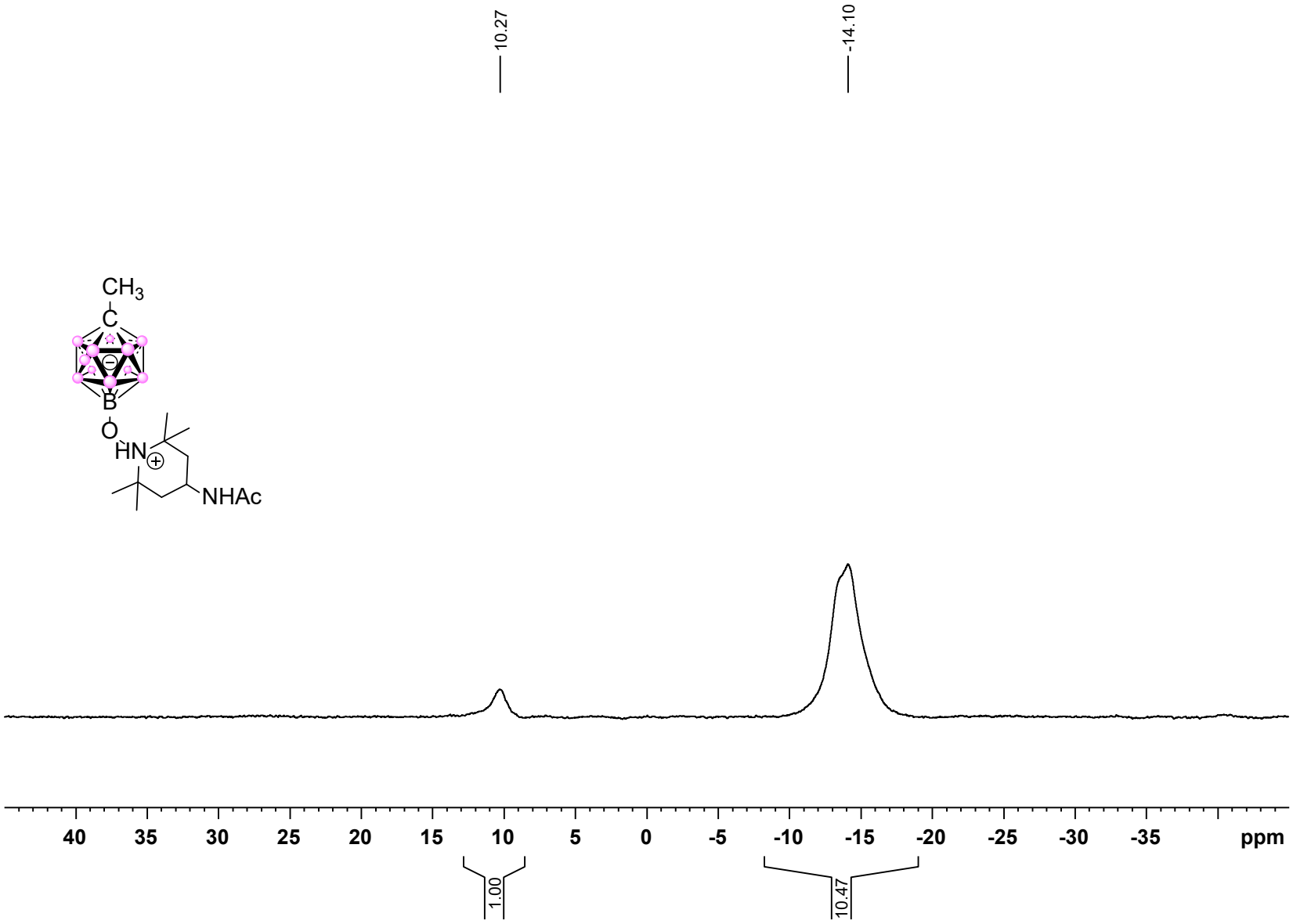

```
Current Data Parameters
NAME      U091-2-CB11D11
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20241101
Time              16.35 h
INSTRUM             spect
PROBHD   Z108618_0497 (
PULPROG          zgpg30
TD                65536
SOLVENT           CD3CN
NS                  512
DS                    4
SWH           29761.904 Hz
FIDRES         0.908261 Hz
AQ            1.1010048 sec
RG               193.34
DW               16.800 usec
DE                6.50 usec
TE               300.9 K
D1           1.50000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228293 MHz
NUC1                13C
P1                10.00 usec
PLW1        66.13099670 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        15.48099995 W
PLW12        0.43002000 W
PLW13        0.21630000 W

F2 - Processing parameters
SI                32768
SF          100.6126695 MHz
WDW                  EM
SSB                   0
LB                10.00 Hz
GB                    0
PC                 1.40
```

[H][1-CH3-12-NHAc-TEMPO-CB11H11]
1H{11B} 500MHZ DMSO T=23 C



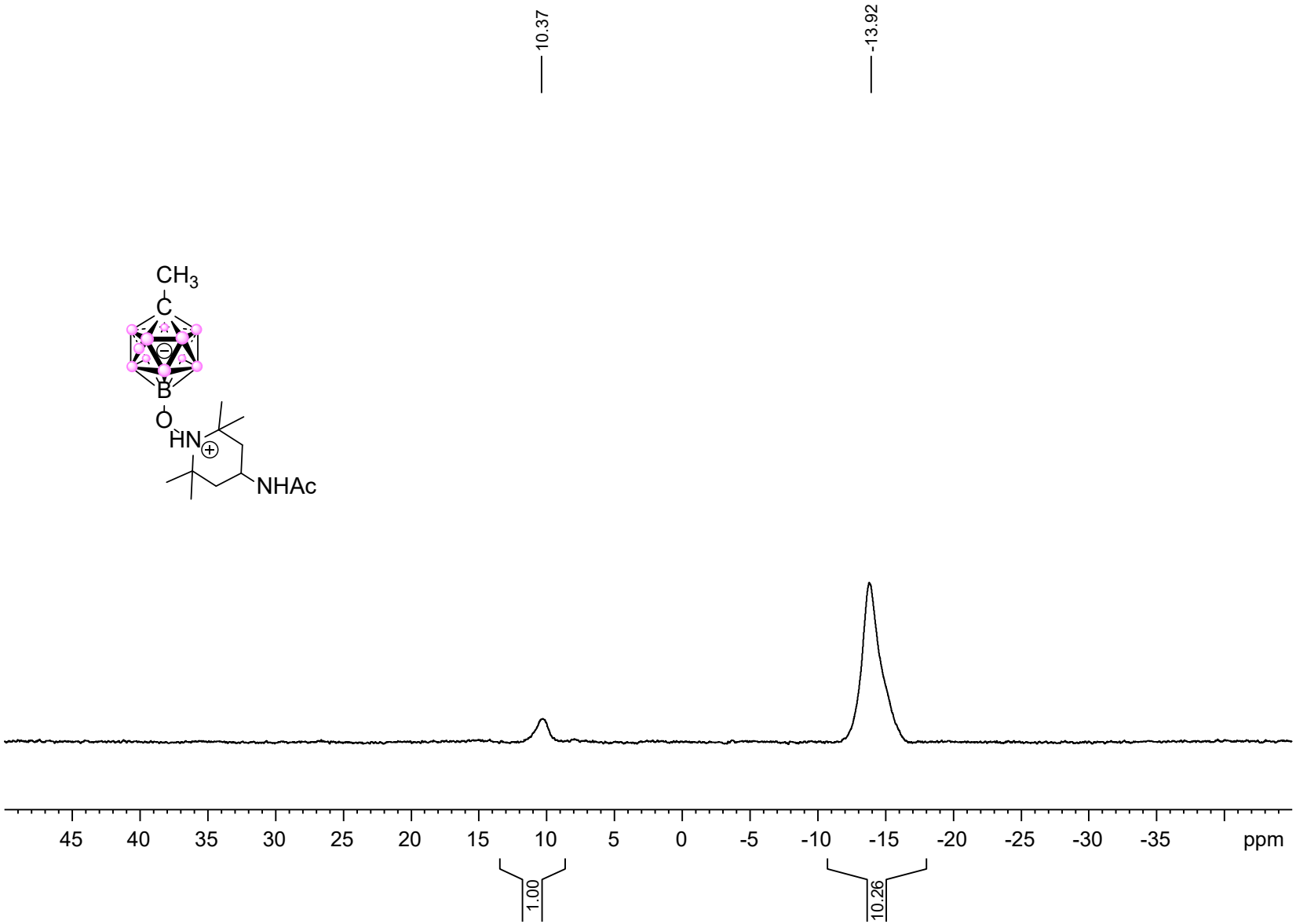

```
Current Data Parameters
NAME                  U148-1
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_               20250213
Time                   11.19
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                zgig30
TD                      65536
SOLVENT                  DMSO
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                     293.9 K
D1                  5.00000000 sec
D11                 0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               500.1735009 MHz
NUC1                       1H
P1                     11.16 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                 500.1700037 MHz
WDW                        EM
SSB                         0
LB                     1.00 Hz
GB                          0
PC                     1.00
```

[H][1-CH3-12-NHAc-TEMPO-CB11H11]
11B 160MHZ DMSO T=23 C



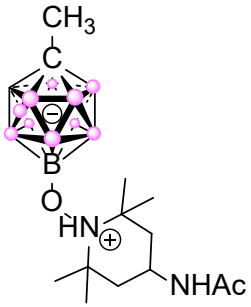

```
Current Data Parameters
NAME                  U148-1
EXPNO                      3
PROCNO                     1

F2 - Acquisition Parameters
Date_               20250213
Time                   11.21
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    64098
SOLVENT                DMSO
NS                       32
DS                        0
SWH               32051.281 Hz
FIDRES             0.500036 Hz
AQ                 0.9999288 sec
RG                       203
DW                    15.600 usec
DE                     6.50 usec
TE                    293.8 K
D1              1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1            160.4744132 MHz
NUC1                    11B
P1                    13.10 usec
PLW1           95.00000000 W

F2 - Processing parameters
SI                     32768
SF              160.4744130 MHz
WDW                       EM
SSB                        0
LB                    10.00 Hz
GB                         0
PC                     1.40
```

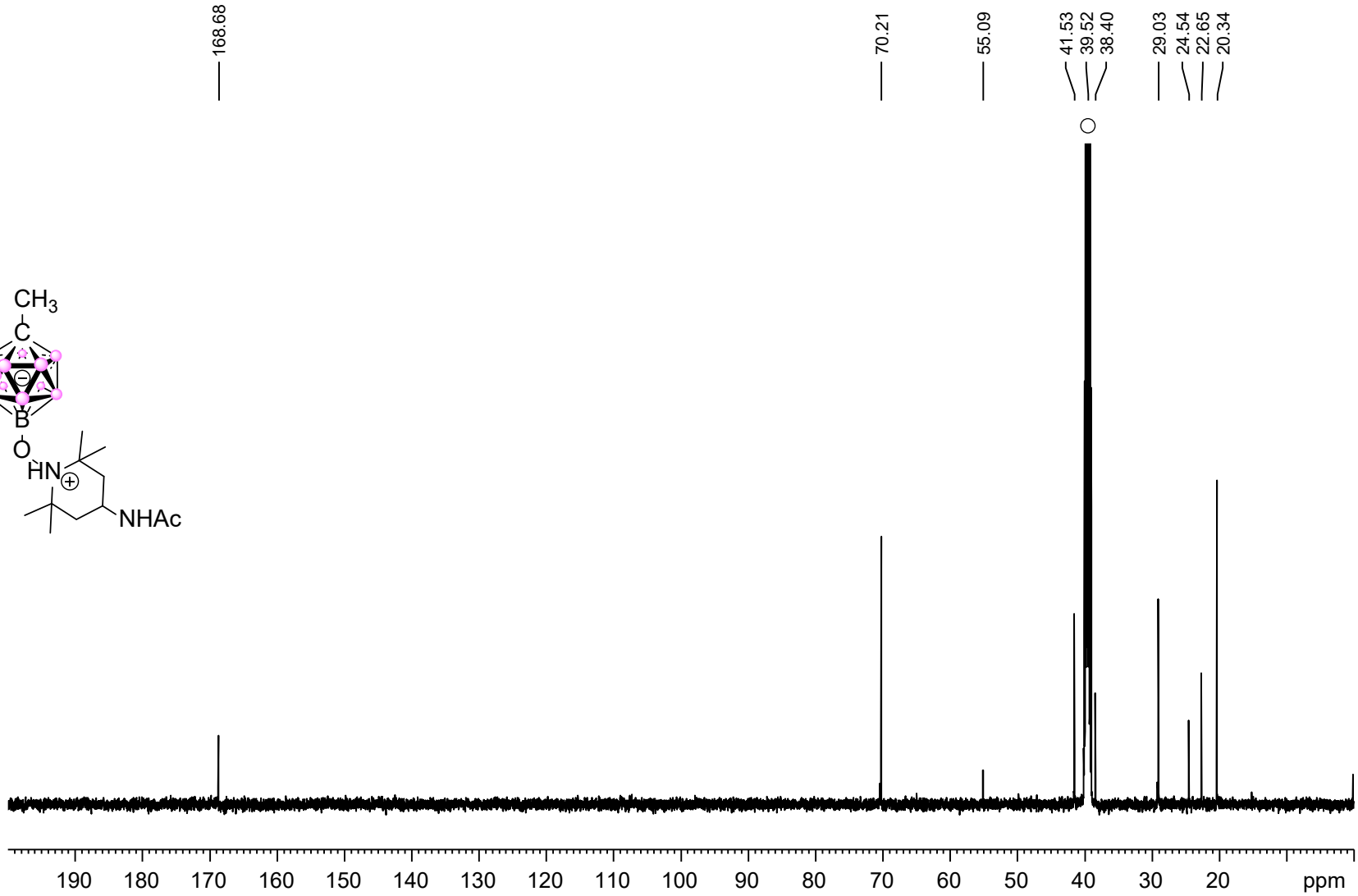
[H][1-CH3-12-NHAc-TEMPO-CB11H11]
11B{1H} 160MHZ DMSO T=23 C




```
Current Data Parameters
NAME                  U148-1
EXPNO                      4
PROCNO                     1

F2 - Acquisition Parameters
Date_               20250213
Time                   11.21
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT                DMSO
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                   294.0 K
D1                1.00000000 sec
D11               0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1            160.4744130 MHz
NUC1                    11B
P1                    13.10 usec
PLW1            95.00000000 W

======== CHANNEL f2 ========
SFO2            500.1725007 MHz
NUC2                    1H
CPDPRG[2             waltz16
PCPD2                 80.00 usec
PLW2            19.00000000 W
PLW12            0.40639001 W
PLW13            0.26008999 W

F2 - Processing parameters
SI                     32768
SF              160.4744130 MHz
WDW                       EM
SSB                        0
LB                    10.00 Hz
GB                         0
PC                     1.40
```

[H][1-CH3-12-NHAc-TEMPO-CB11H11]
13C 126MHZ DMSO T=23 C






```
Current Data Parameters
NAME                  U148-1
EXPNO                      5
PROCNO                     1

F2 - Acquisition Parameters
Date_              20250213
Time                  12.04
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT                DMSO
NS                      1024
DS                         4
SWH               37878.789 Hz
FIDRES             0.577984 Hz
AQ                0.8650752 sec
RG                       203
DW                  13.200 usec
DE                   6.50 usec
TE                   294.5 K
D1              1.50000000 sec
D11             0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1           125.7816804 MHz
NUC1                    13C
P1                  10.17 usec
PLW1           95.00000000 W

======== CHANNEL f2 ========
SFO2           500.1720005 MHz
NUC2                    1H
CPDPRG[2            waltz16
PCPD2                80.00 usec
PLW2           19.00000000 W
PLW12           0.40639001 W
PLW13           0.26008999 W

F2 - Processing parameters
SI                     32768
SF             125.7679021 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```