

Supporting Information

A Convenient Route to Monocarba-*closo*-dodecaborate Anions

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1. Analytical Data for $\text{Me}_3\text{NH}^+\cdot\mathbf{3}$

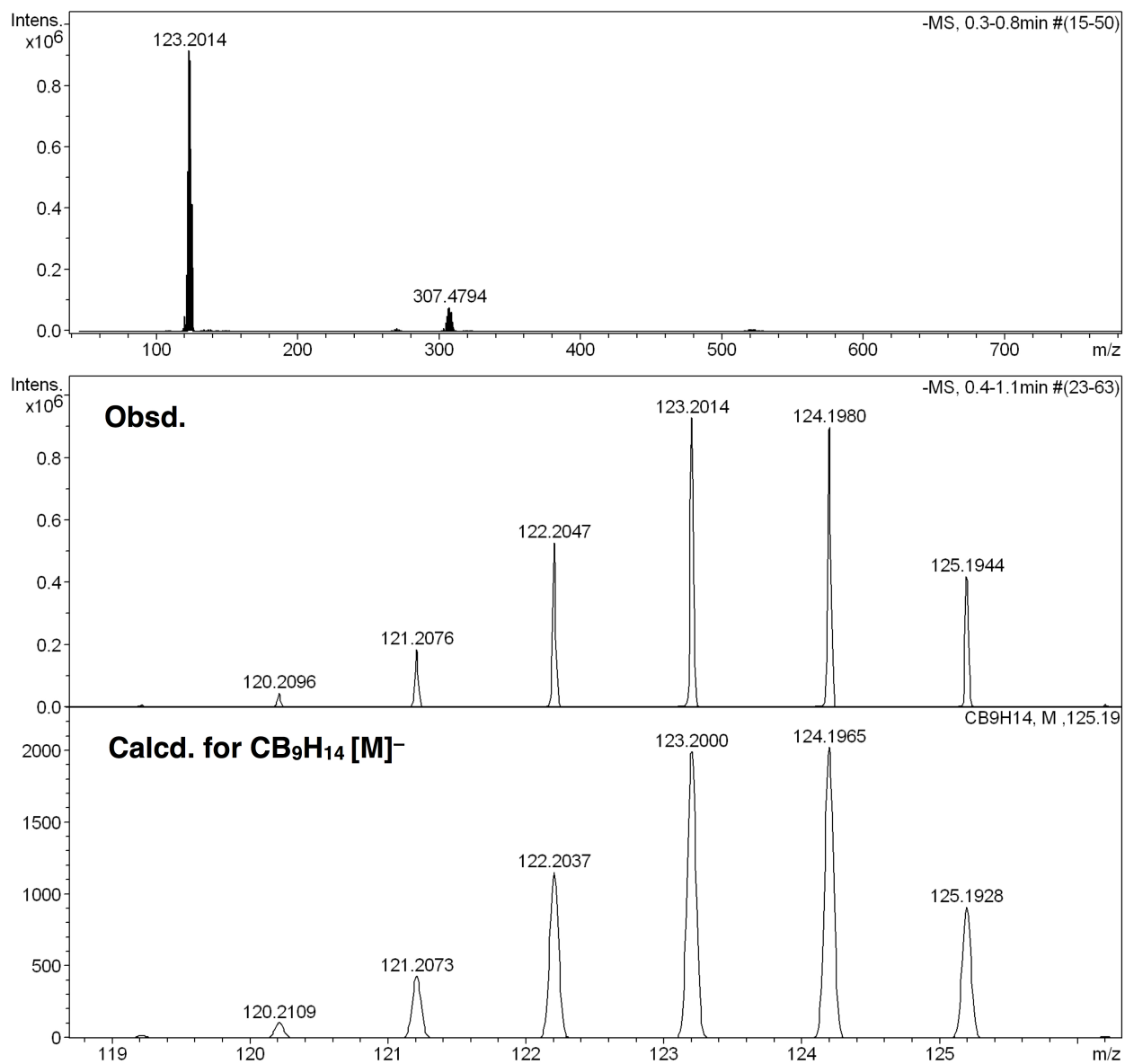


Figure S1. ESI-TOF MS spectrum of $\text{Me}_3\text{NH}^+\cdot\mathbf{3}$.

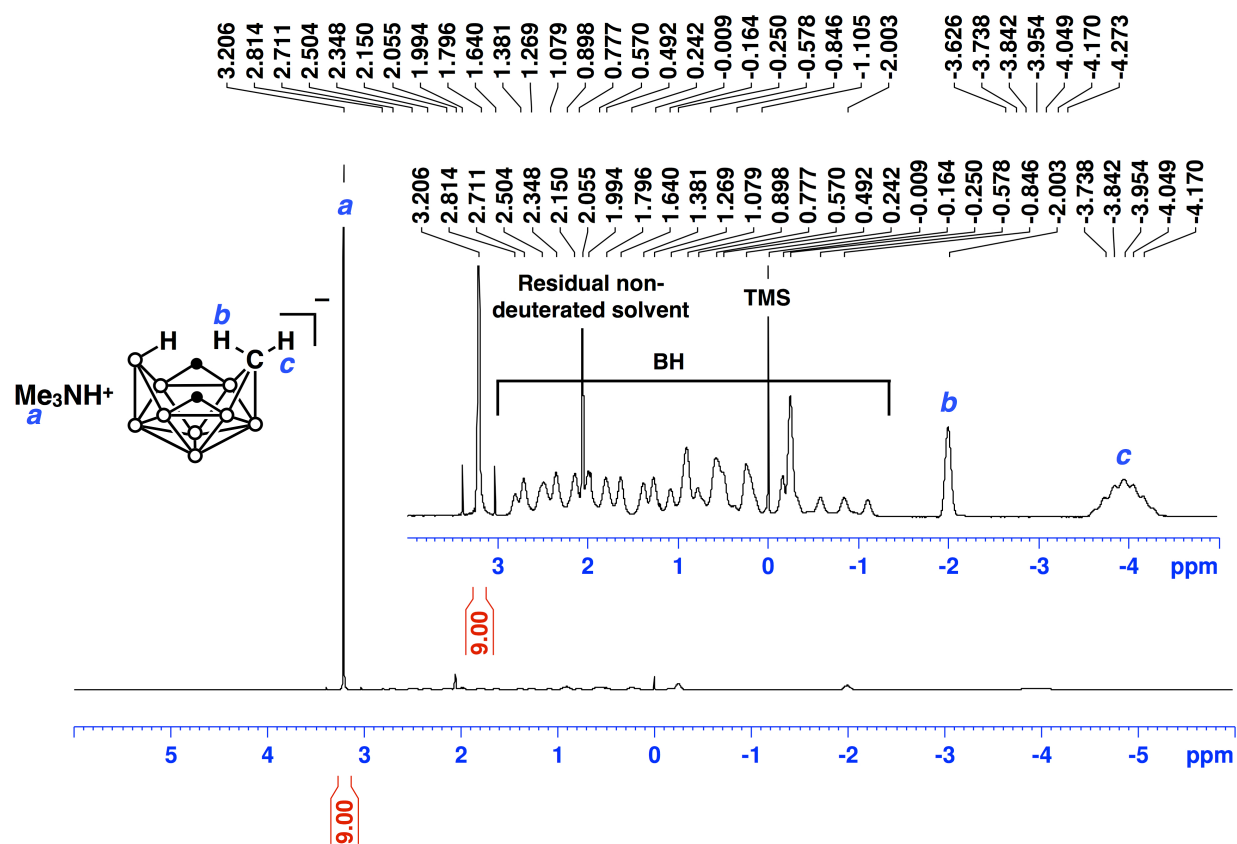


Figure S2. ^1H NMR spectrum (400 MHz) of $\text{Me}_3\text{NH}^+\cdot\mathbf{3}$ in acetone- d_6 at 25 °C.

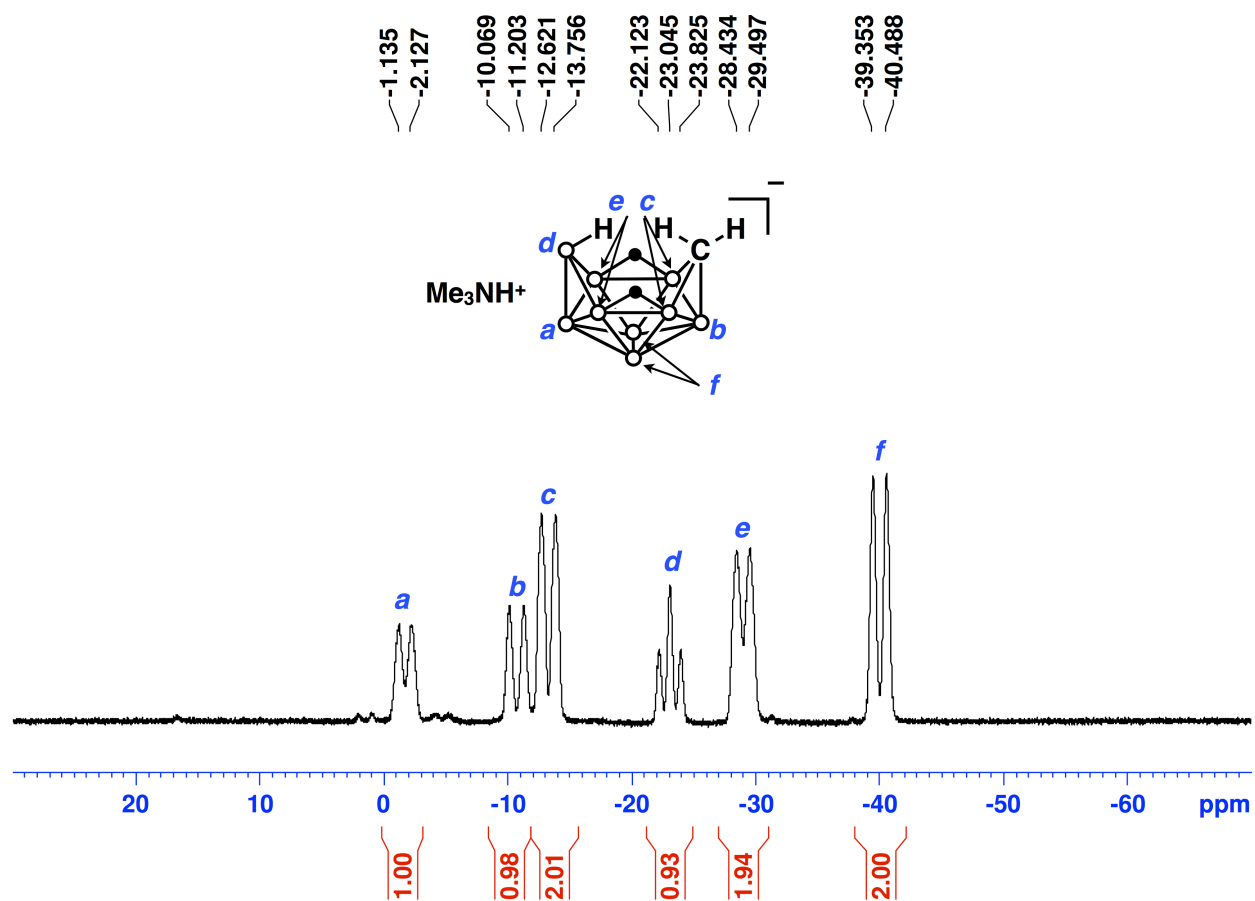


Figure S3. ^{11}B NMR spectrum (128 MHz) of $\text{Me}_3\text{NH}^+\cdot\mathbf{3}$ in acetone- d_6 at 25 °C.

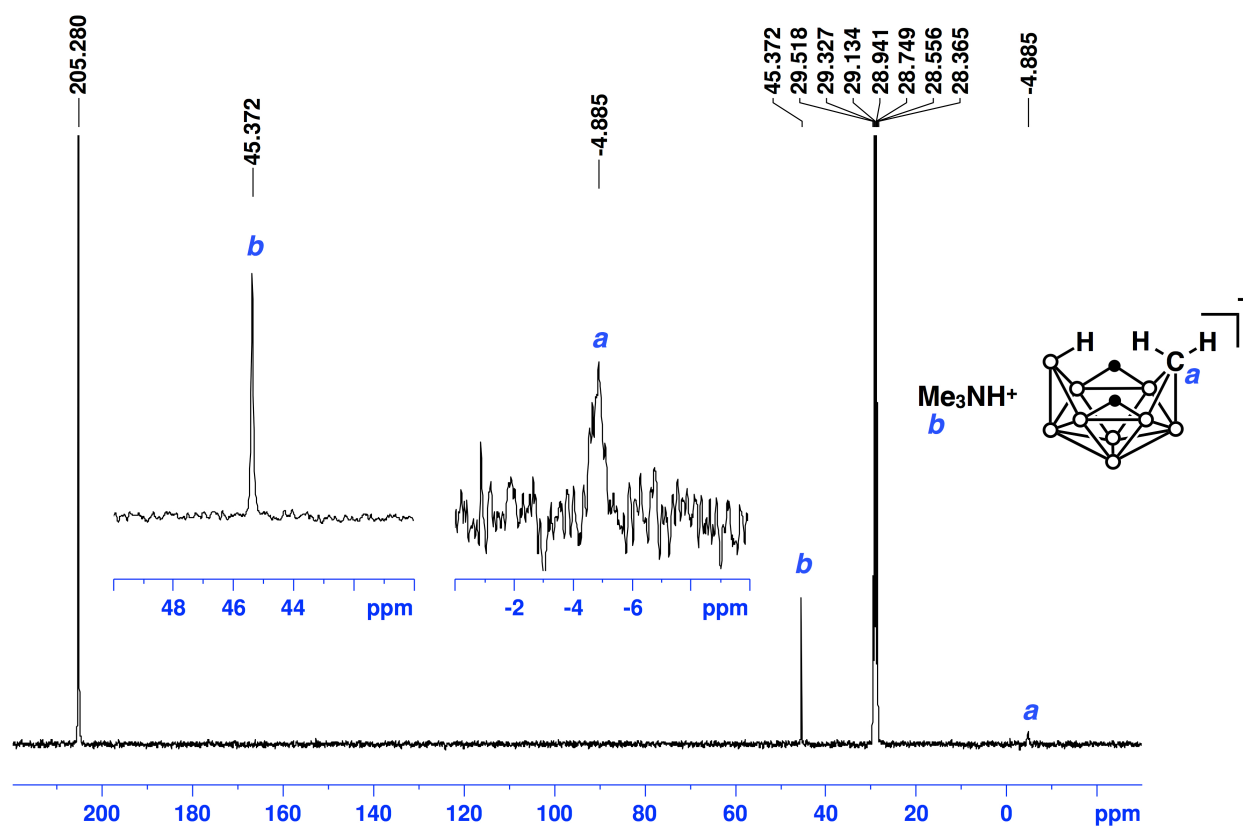


Figure S4. ^{13}C NMR spectrum (100 MHz) of $\text{Me}_3\text{NH}^+\cdot\mathbf{3}$ in $\text{acetone-}d_6$ at 25°C .

2. Analytical Data for $\text{Me}_3\text{NH}^+\cdot\mathbf{1}$

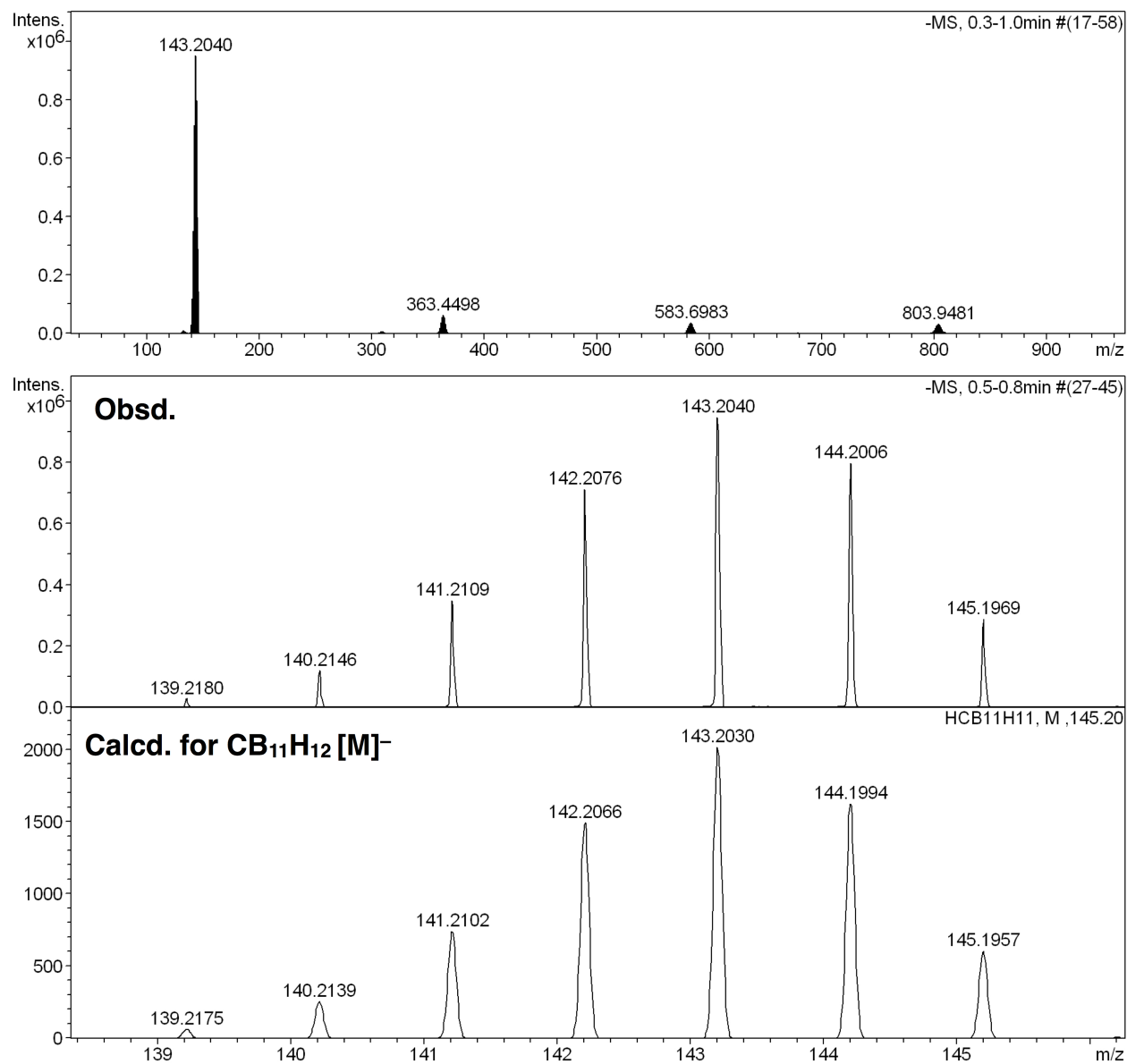


Figure S5. ESI-TOF MS spectrum of $\text{Me}_3\text{NH}^+\cdot\mathbf{1}$.

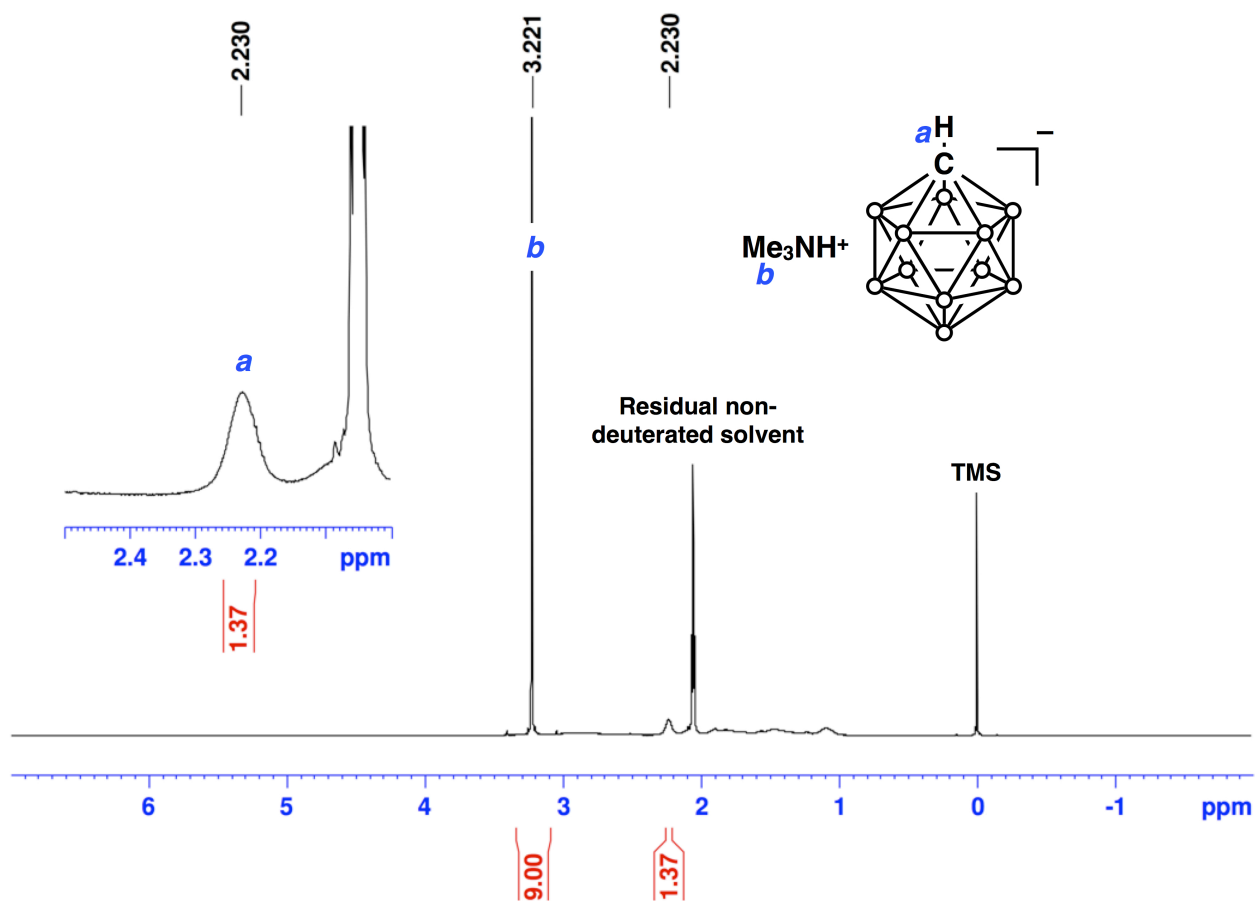


Figure S6. ^1H NMR spectrum (400 MHz) of $\text{Me}_3\text{NH}^+\cdot\mathbf{1}$ in $\text{acetone-}d_6$ at $25\text{ }^\circ\text{C}$.

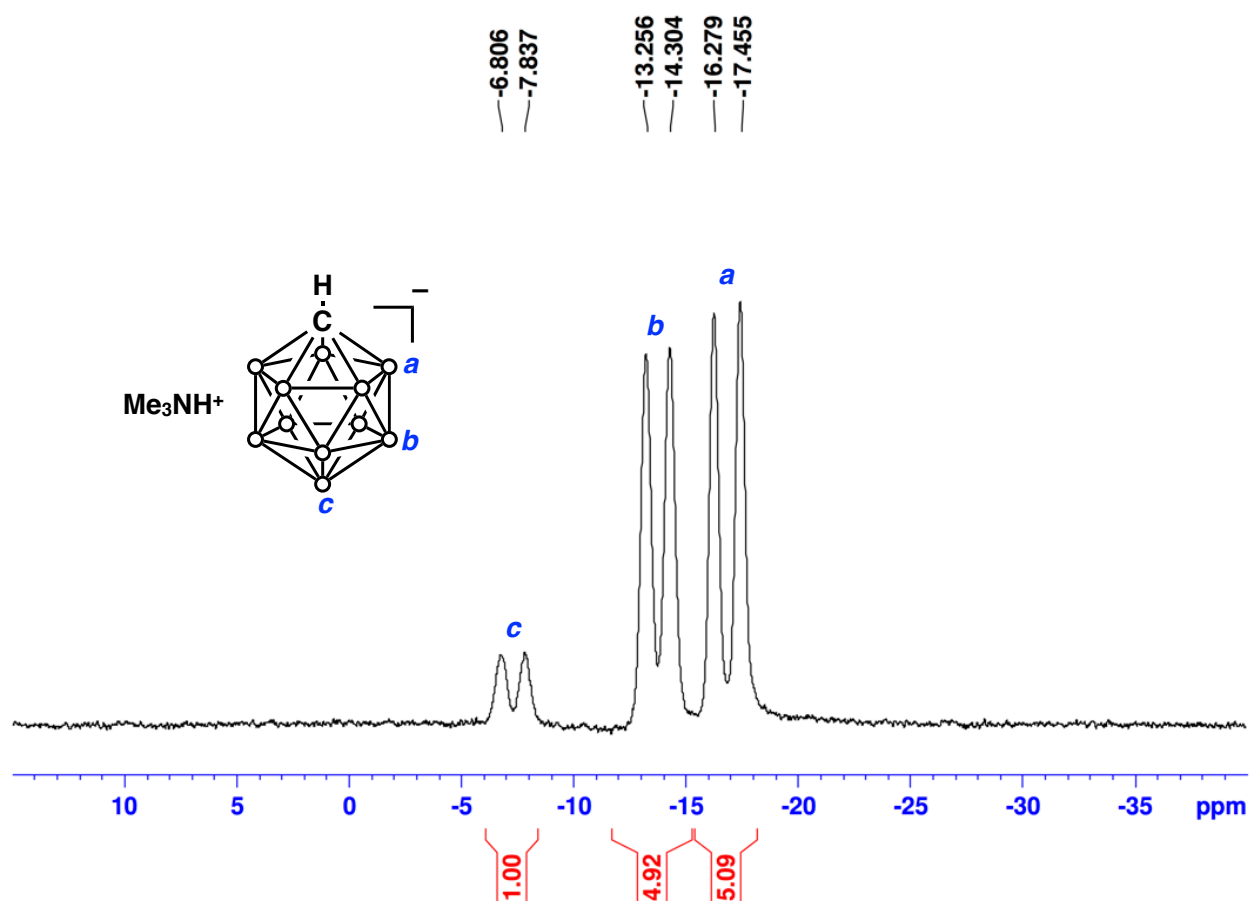


Figure S7. ^{11}B NMR spectrum (128 MHz) of $\text{Me}_3\text{NH}^+\bullet\mathbf{1}$ in $\text{acetone-}d_6$ at $25\text{ }^\circ\text{C}$.

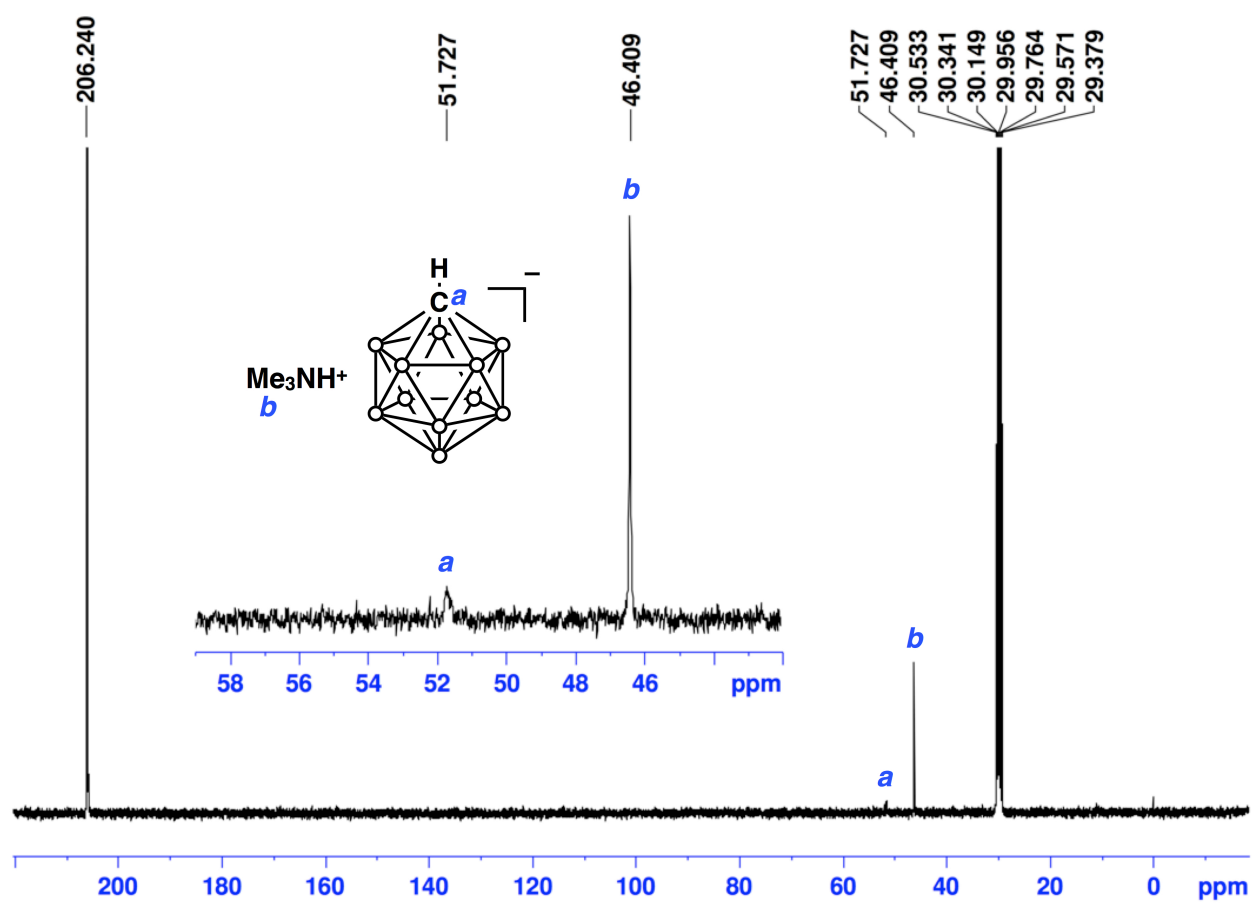


Figure S8. ^{13}C NMR spectrum (100 MHz) of $\text{Me}_3\text{NH}^+\cdot\mathbf{1}$ in $\text{acetone-}d_6$ at 25°C .

3. Analytical Data for $\text{Cs}^+\cdot\mathbf{1}$

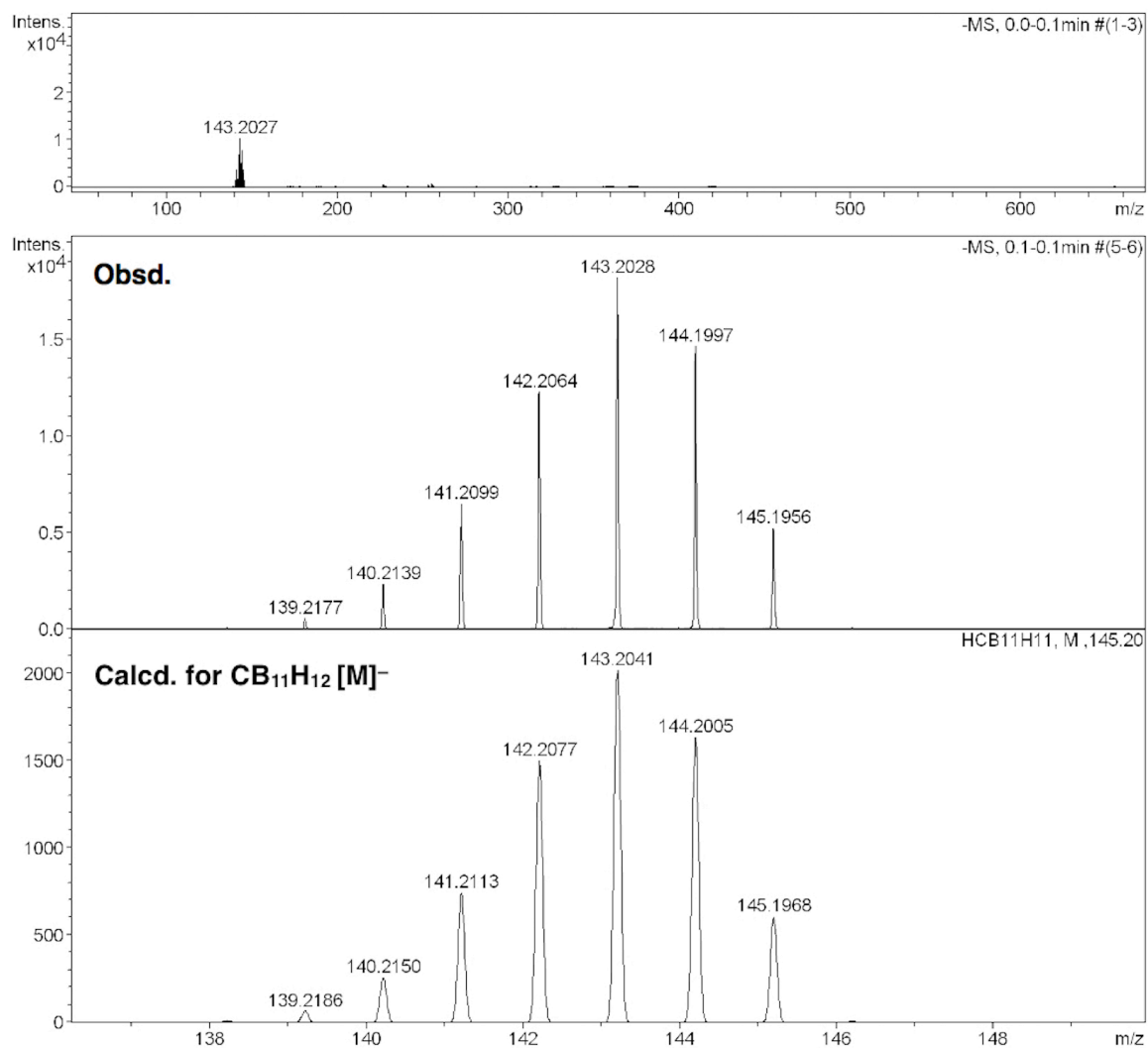


Figure S9. ESI-TOF MS spectrum of $\text{Cs}^+\cdot\mathbf{1}$.

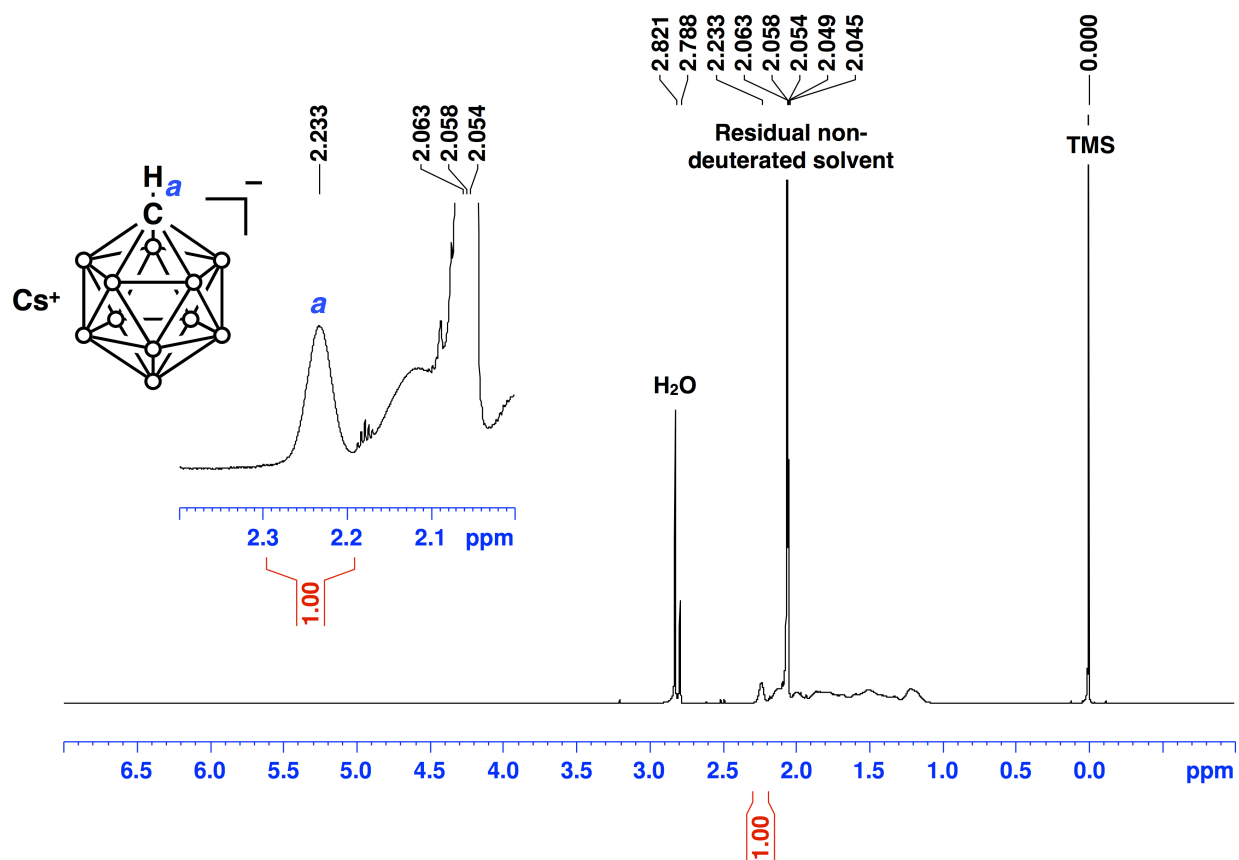


Figure S10. ^1H NMR spectrum (400 MHz) of $\text{Cs}^+\bullet\mathbf{1}$ in $\text{acetone-}d_6$ at $25\text{ }^\circ\text{C}$.

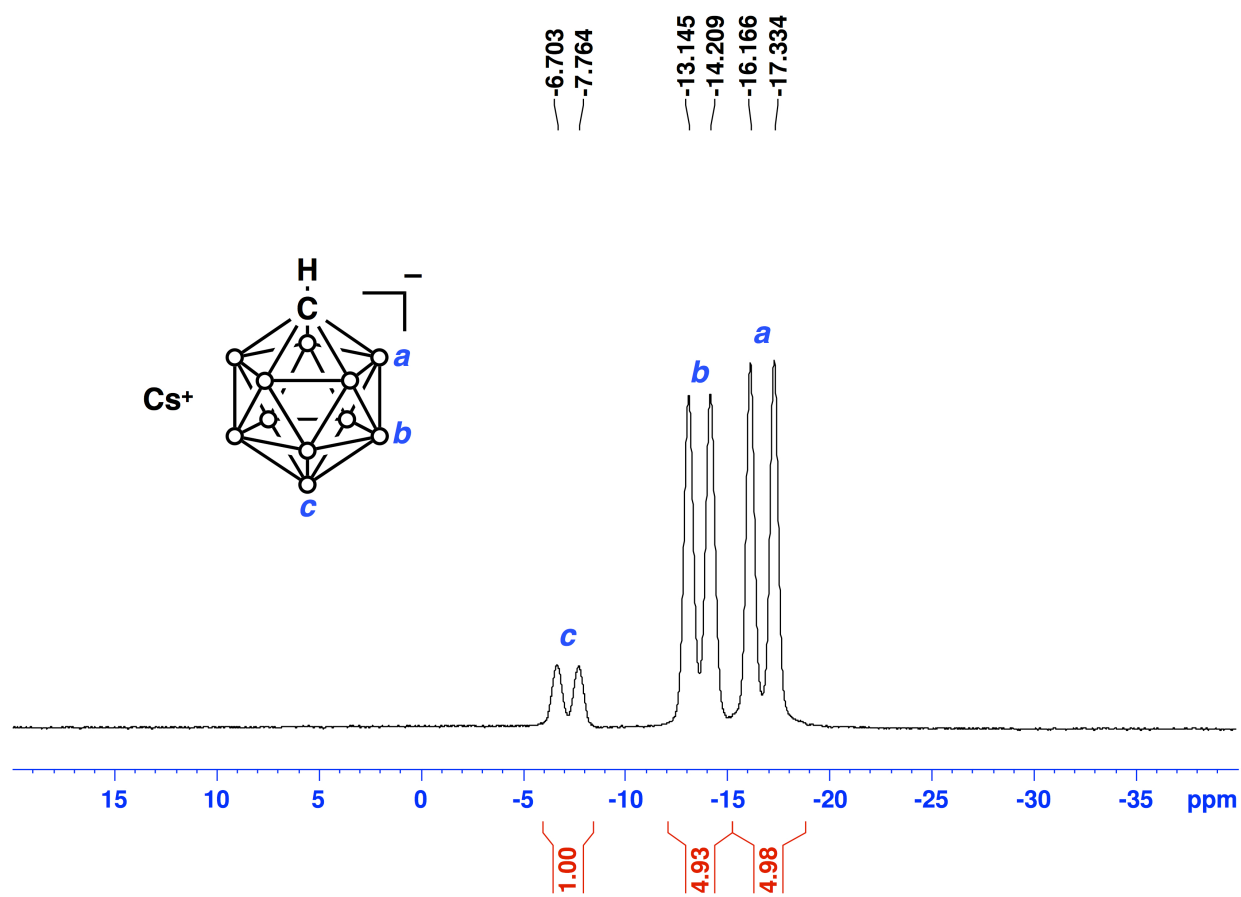


Figure S11. $^{\text{11}}\text{B}$ NMR spectrum (128 MHz) of $\text{Cs}^+\bullet\mathbf{1}$ in acetone- d_6 at 25 °C.

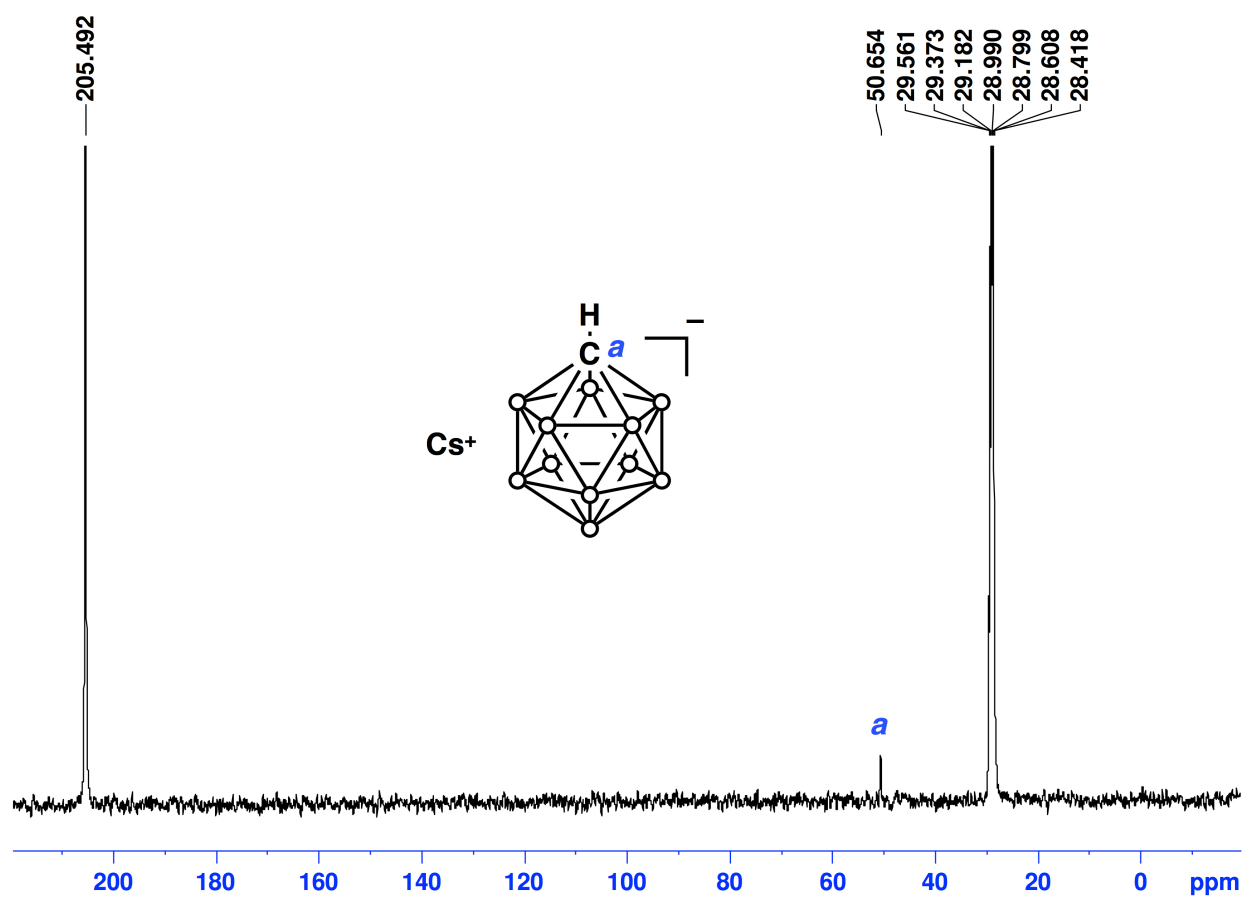


Figure S12. ^{13}C NMR spectrum (100 MHz) of $\text{Cs}^+\bullet\mathbf{1}$ in $\text{acetone-}d_6$ at 25°C .

4. Analytical Data for $\text{Cs}^+\bullet 4$

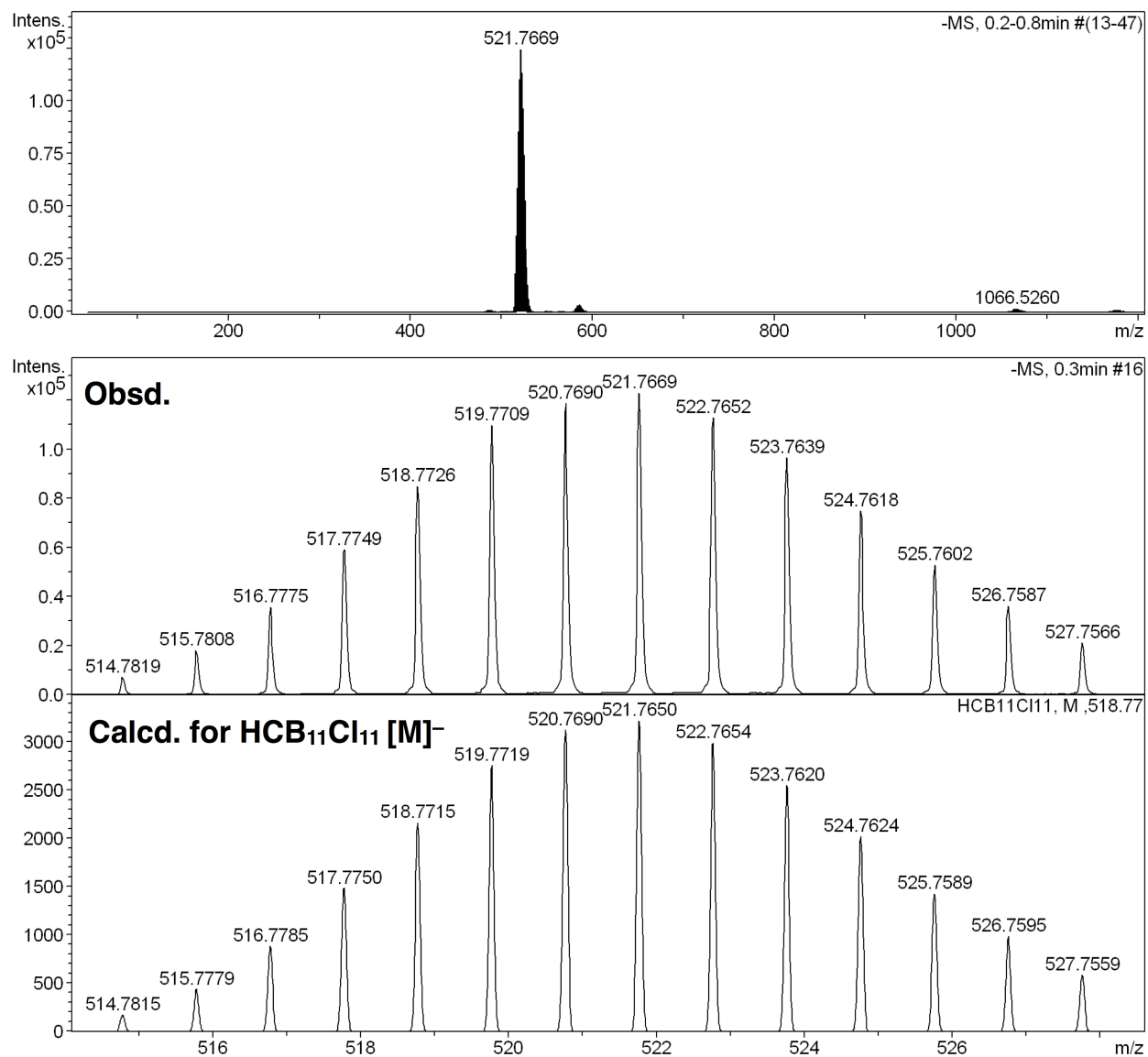


Figure S13. ESI-TOF MS spectrum of $\text{Cs}^+\bullet 4$.

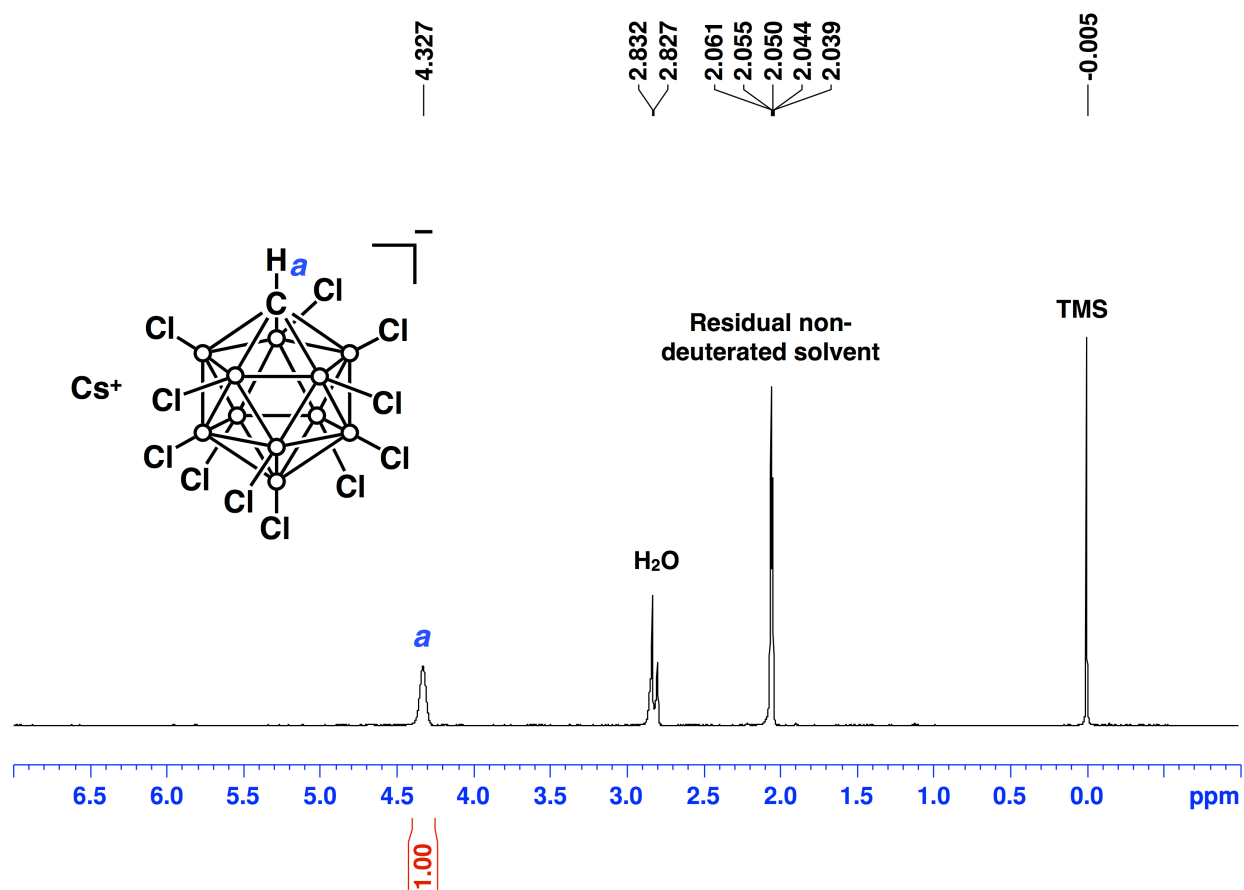


Figure S14. ^1H NMR spectrum (400 MHz) of $\text{Cs}^+\bullet\mathbf{4}$ in acetone- d_6 at 25 °C.

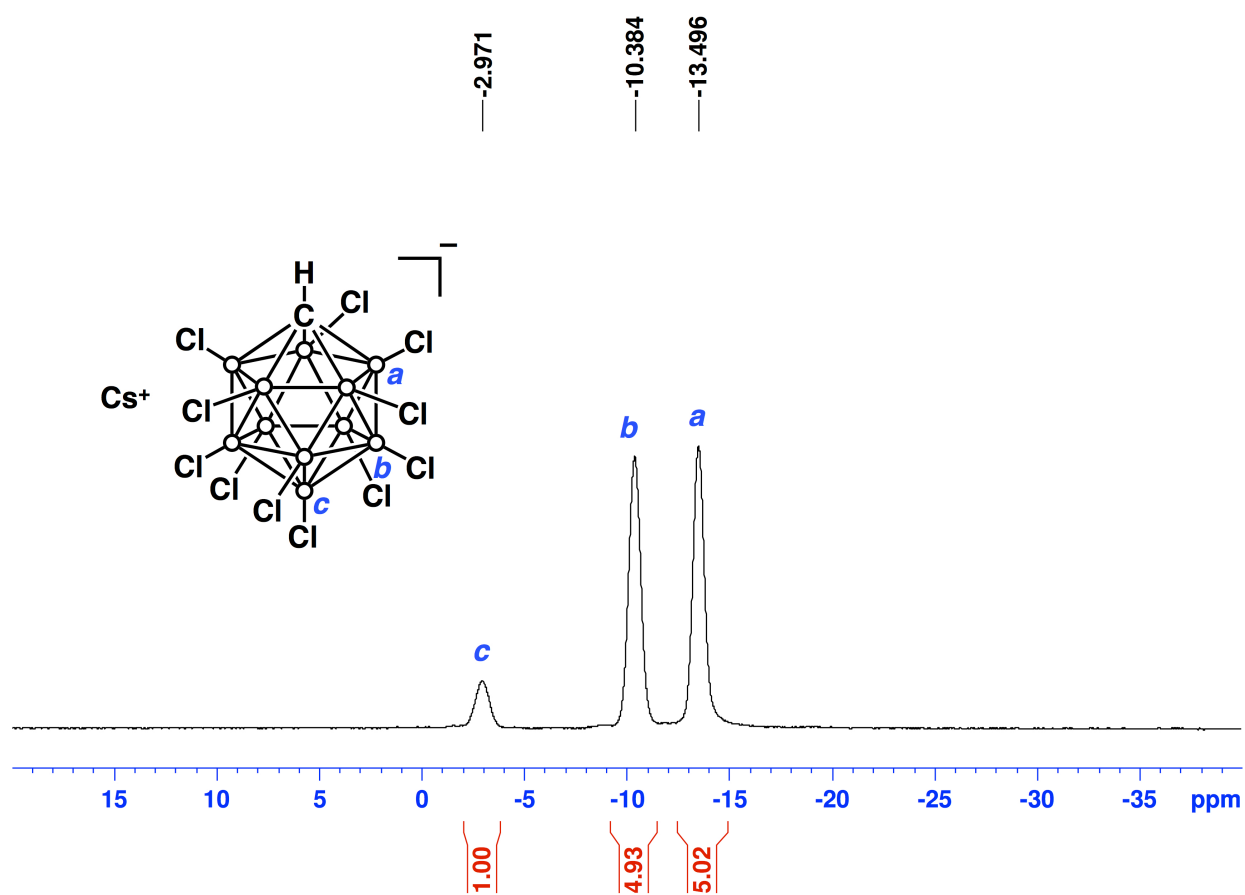


Figure S15. ^{11}B NMR spectrum (128 MHz) of $\text{Cs}^+ \cdot \mathbf{4}$ in acetone- d_6 at 25 °C.

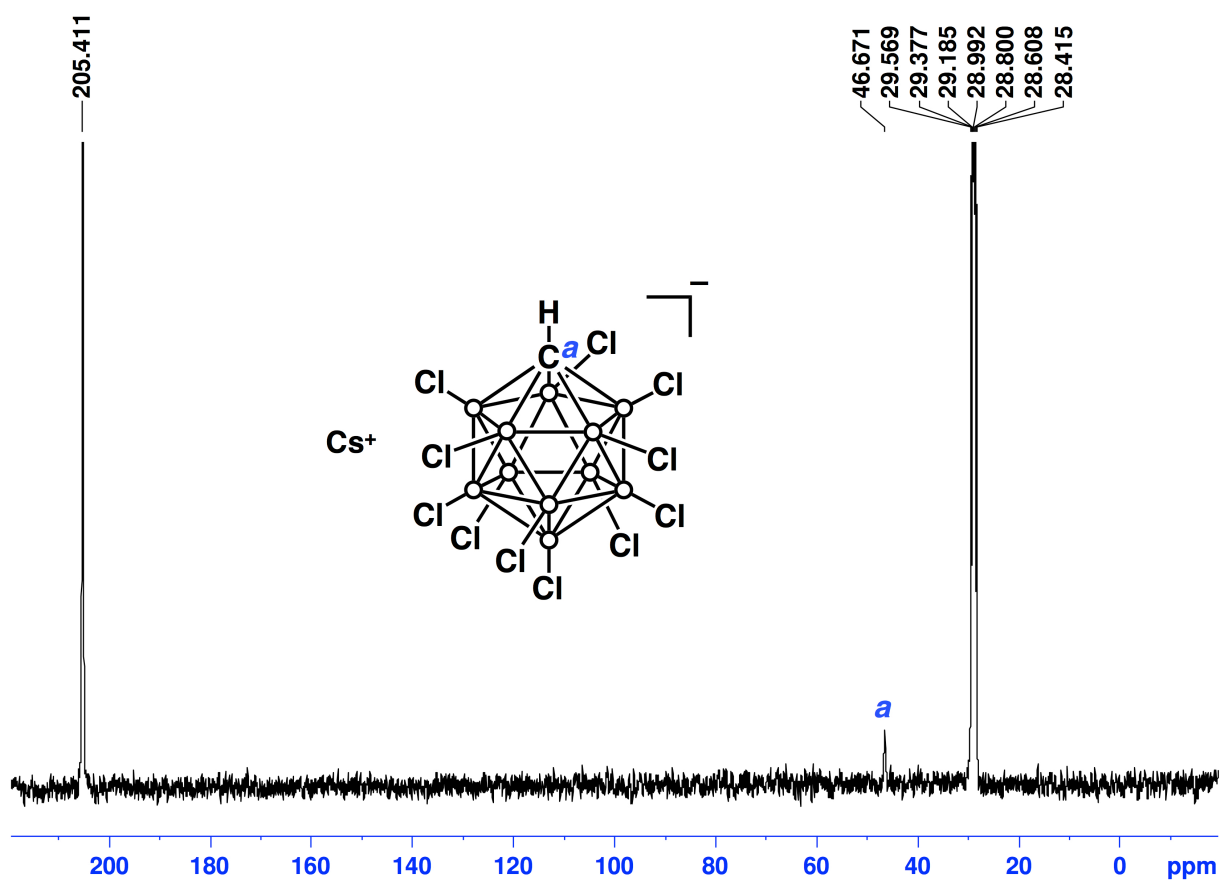


Figure S16. ^{13}C NMR spectrum (100 MHz) of $\text{Cs}^+\bullet\mathbf{4}$ in $\text{acetone-}d_6$ at $25\text{ }^\circ\text{C}$.

5. Supporting Figures

Data: <Untitled>.O19[c] 4 Jun 2014 17:03 Cal: 12 Mar 2010 9:40

Shimadzu Biotech Axima CFRplus 2.9.3.20110624: Mode Reflectron_neg, Power: 100, P.Ext. @ 521 (bin 57)

%Int. 171 mV[sum= 5141 mV] Profiles 1-30 Smooth Av 20

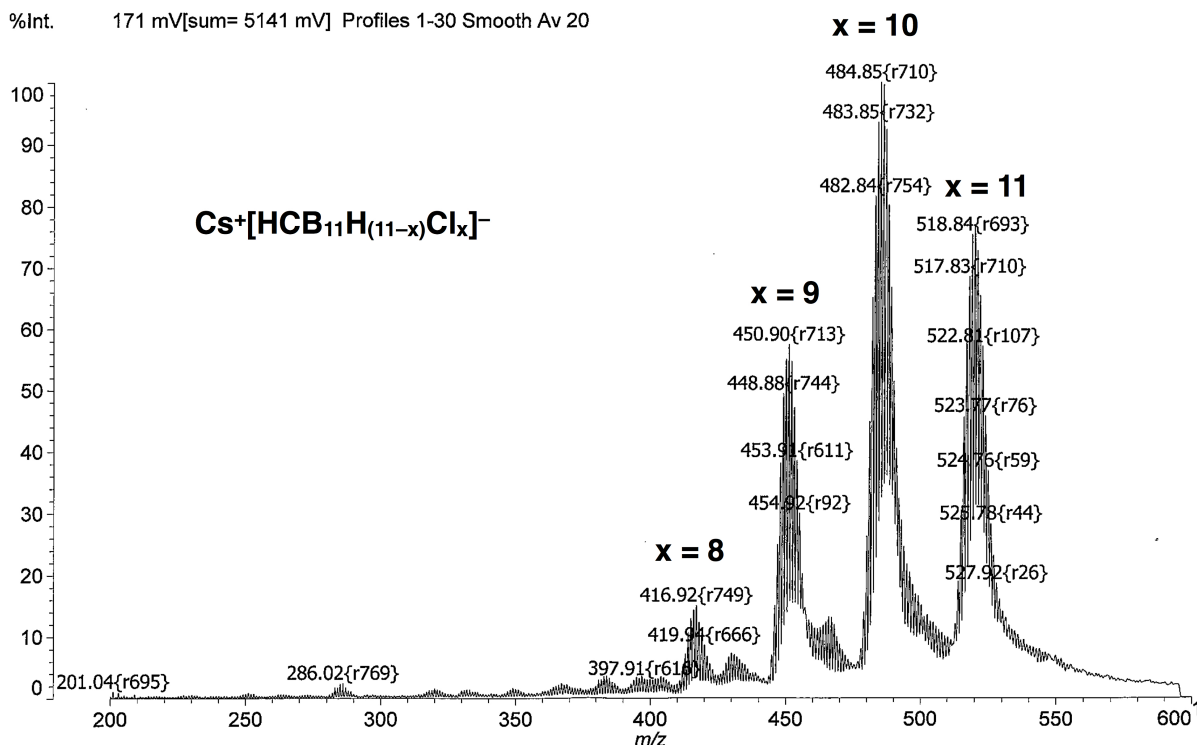


Figure S17. MALDI-TOF MS spectrum of a reaction mixture obtained after refluxing a SO_2Cl_2 solution of $\text{Cs}^+\cdot\mathbf{1}$ under argon for 1 week.

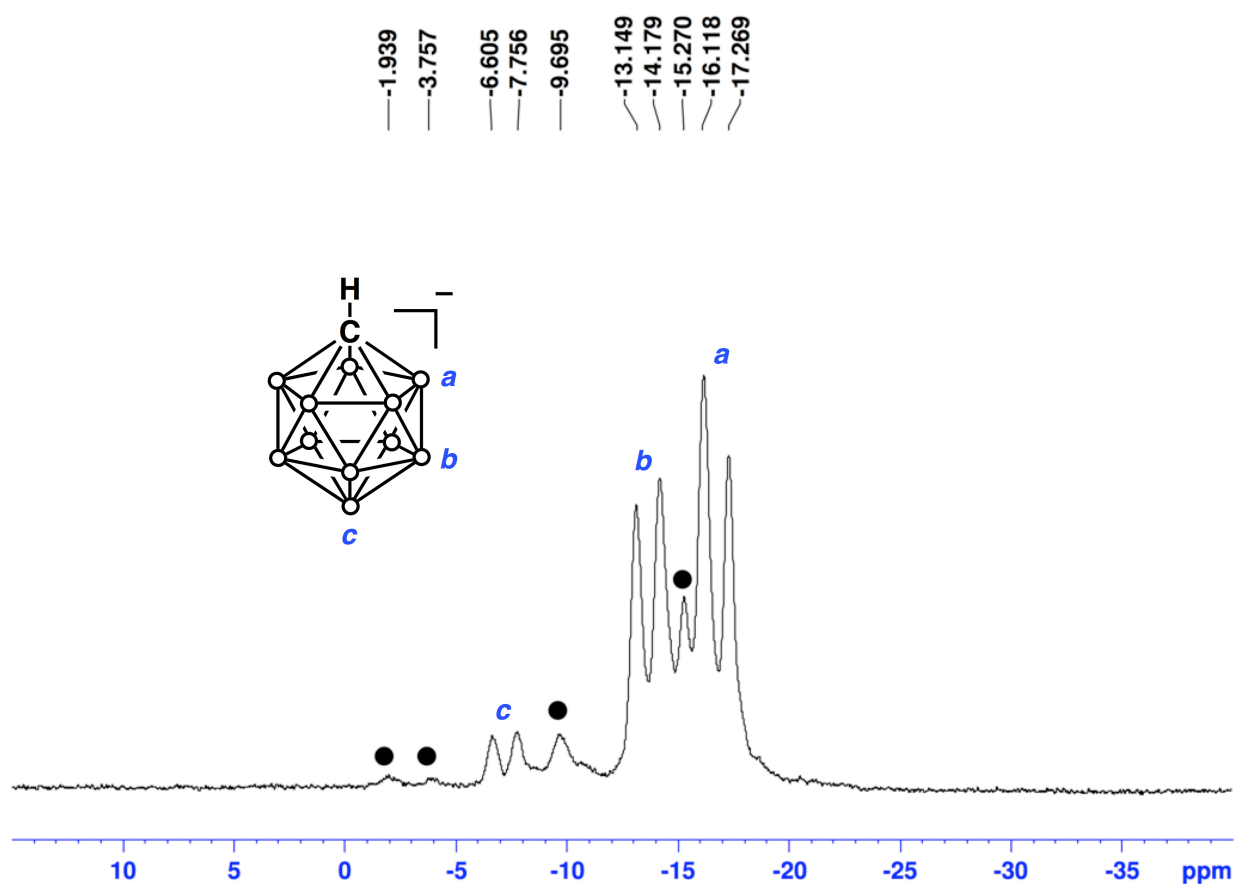


Figure S18. ^{11}B NMR spectrum (128 MHz) of a reaction mixture obtained after the reaction of $\text{Me}_3\text{NH}^+\cdot\mathbf{3}$ with $\text{BH}_3\cdot\text{SMe}_2$ followed by washing with hot water. Peaks associated with black circles (•) are due to a boron-containing byproduct.



Figure S19. Photograph of a 100 mL-volume pressure-tight autoclave reactor (TPR6-VS2-100, Taiatsu Techno®) used for the perchlorination reaction of CB₁₁H₁₂⁻ (1).