

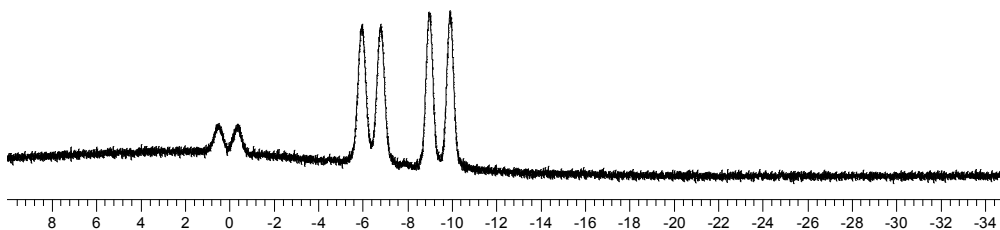
## Hexachlorination of $\text{CB}_{11}\text{H}_{12}^-$

A few grams (typically less than 5g) of  $[\text{Cs}^+][\text{CB}_{11}\text{H}_{12}^-]$  (FWT=275.9) are placed in a 250 mL three-neck round-bottom flask containing a magnetic stirring bar, and fitted with:

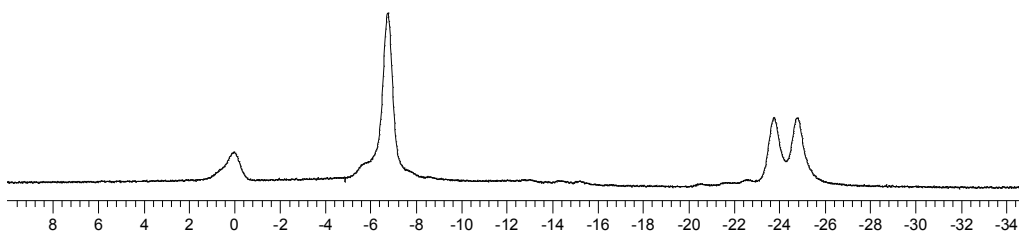
- 1) a water cooled condenser, topped with a hose adapter,
- 2) a thermometer fed through either a standard taper adapter or a slit septum,
- 3) and a piece of glass tubing (preferably end capped with sintered glass) also fed through a split septum.

The flask is submerged in a temperature regulated bath in a fumehood. The carborane salt is covered with enough glacial acetic acid (ca. 50 mL) to cover completely, and stirring is started. Chlorine gas is bubbled through glass tubing into the solution. The solution will warm slightly upon chlorine addition, and the temperature is further raised and held at 70 °C by the heating bath. Exhaust of chlorine gas/acetic acid should be scrubbed: A hose connected to a trap is attached to the hose adapter on the condenser, and a funnel is attached via a length of tubing to the exit of the trap. The mouth of the funnel is then submerged in an aqueous solution of sodium hydroxide and a reducing agent, e.g. sodium sulfite. After one hour has elapsed, the progress of the reaction is monitored by collecting the  $^{11}\text{B}$  NMR spectrum of an aliquot every 20 minutes. (See attached spectra). The reaction is typically complete in roughly 4-8 hours.

Once the reaction is complete, the product is isolated by removing the acetic acid and residual dissolved chlorine by evaporation (rotavap or distillation under vacuum). Recrystallize the crude product from a minimum amount of boiling water. The product is fairly soluble in room temperature water, so be sure to cool the recrystallization solution in an ice bath or refrigerator before filtering. Check the  $^{11}\text{B}$  and  $^1\text{H}$  NMR of the product to verify purity.



$^{11}\text{B}$  NMR spectrum of  $[\text{Cs}^+][\text{CB}_{11}\text{H}_{12}^-]$



$^{11}\text{B}$  NMR spectrum of nearly complete hexachlorination of  $[\text{Cs}^+][\text{CB}_{11}\text{H}_{12}^-]$  to  $[\text{Cs}^+][\text{CB}_{11}\text{H}_6\text{Cl}_6^-]$