

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

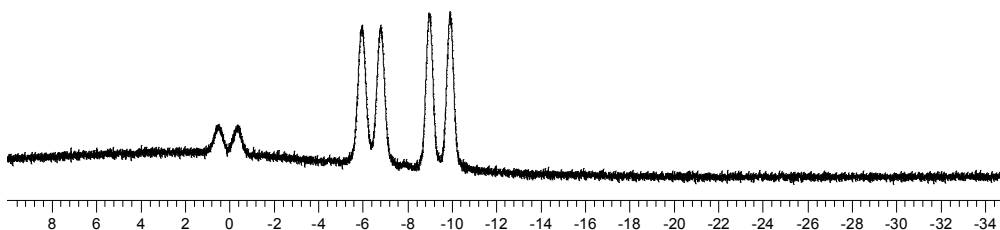
A few grams (typically less than 5g) of  $[\text{Cs}^+][\text{CB}_{11}\text{H}_{12}^-]$  (FWT=275.9) are placed in a 250 mL three-neck round-bottom flask containing a magnetic stirring bar, and fitted with:

- 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) a septum or stopper to close the third neck.

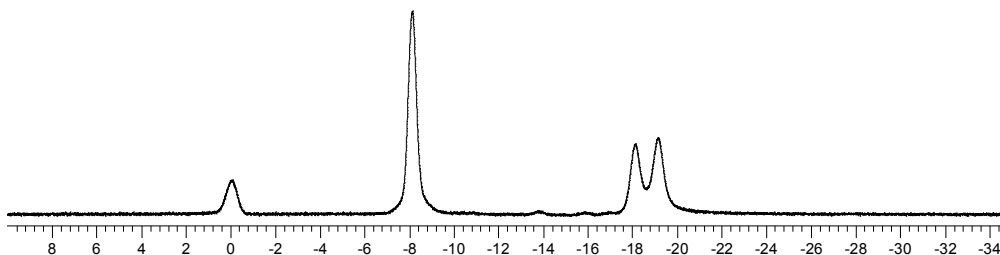
The flask is submerged in a temperature regulated bath in a fumehood. The carborane salt is covered with enough glacial acetic acid (ca. 50 mL) to cover completely, and 20 molar equivalents of bromine (FWT=159.8 density=3.1 g/mL) are **very slowly** added to the flask. **\*\*The bromine addition is exothermic and quick addition will raise the temperature enough to cause the bromine to boil violently and be lost.\*\***

Once the bromine is completely added, stirring is started, and the reaction is heated to 80 °C. The condenser should be sufficient to minimize the loss of bromine and acetic acid to the atmosphere, however, to capture additional exhaust, a hose adaptor can be fitted to the condenser with a tube leading to a trap followed by a vent hose. The vent hose is attached to a funnel whose mouth is submerged in an aqueous solution of sodium hydroxide and a reducing agent (e.g. sodium sulfite). The reaction is monitored by collecting the  $^{11}\text{B}$  NMR spectrum of an aliquot every 12 hours. The reaction typically requires a minimum of 24 hours to complete.

Once the reaction is complete, the product is isolated by removing the acetic acid and bromine by evaporation (rotavap or distillation under vacuum). Recrystallize the crude product from a minimum amount of boiling water. The product is somewhat 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  $[\text{Cs}^+][\text{CB}_{11}\text{H}_6\text{Br}_6^-]$