

## Module 1

## Experiment 1: Estimates of hybridization using $^1\text{H}-^{13}\text{C}$ coupling constants.

## Lab Assignment

A .pdf file of Mosher, M.D. and Ojha, S. *J. Chem. Ed.* **75**, 1998, 888-890 will be provided. It is suggested that you review hybridization models from any quality organic chemistry text.

**Samples:** Use 50  $\mu\text{L}$  of each unknown diluted with 600  $\mu\text{L}$  of  $\text{CDCl}_3$  in a clean NMR tube. Turn the decoupler off and run a carbon spectrum. When printing the spectra be sure to express the chemical shift in Hz NOT ppm.

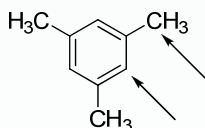
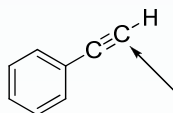
**Data:** The following data should be included in your report:

$^1\text{H}$ -coupled  $^{13}\text{C}$  NMR shifts ( $\delta$ ), coupling constants ( $^1J_{\text{HC}}$ , Hz) and raw spectral data for:

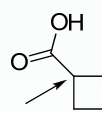
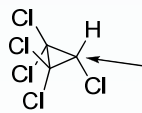
- mesitylene
- phenylacetylene
- pentachlorocyclopropane
- cyclobutane carboxylic acid
- A unique natural product for each student to estimate hybridization of each atom

You will run each spectrum to determine values for  $^1J_{\text{HC}}$ .

**Data Analysis:** Calibration curve, plotting average  $^1J_{\text{HC}}$  vs. % s character of the C-H bond for the indicated hydrogens in phenylacetylene and mesitylene.



These two compounds are used to generate standard values for  $^1J_{\text{HC}}$  for  $sp$ ,  $sp^2$  and  $sp^3$



Discuss the calculated hybridization for these two compounds based on the standard values for  $^1J_{\text{HC}}$ .

- The spreadsheet should be organized and clearly labeled. A least squares linear regression should be performed to obtain the  $R^2$  value.
- Calculate the “hybridization” of the indicated carbons in pentachlorocyclopropane and cyclobutane carboxylic acid and each carbon in your unique compound. Discuss.
- Based on % s-character, calculate the predicted bond angles about each indicated carbon in these two compounds as well as in your unique compound. Discuss.

### Other Topics for Report

- Is the calibration curve linear? What is  $R^2$ ? Is the intercept zero? From your plot, hypothesize about the sources of error.
- From what you have “discovered” about the hybridization of carbons in cyclopropanes, do you think the standard hybridization models are appropriate? Could there be any absolute standard?
- Do you think the addition of the five chlorine atoms on this cyclopropane system has any effect on your observation of hybridization? Compare to cyclobutane which does not have this effect.
- Using literature sources, what is the accepted bonding model (from *ab initio* studies, computer modeling and spectroscopic probes) for cyclopropane?