Introduction to ^{1}H NMR using the picoSpin 45 MHz NMR Spectrometer

CHM 245 Organic Chemistry Laboratory
Miami University Middletown
Professors Janet Marshall & Howard Vail

Purpose:

The purpose of this lab is to learn how to use a picoSpin 45 MHz NMR spectrometer and interpret experimentally-acquired ^{1}H NMR spectra. Each pair of students will obtain the ^{1}H NMR spectrum of an unknown organic compound (selected from several possible compounds) and determine its identity based upon a complete analysis of the spectrum. All students will present their results and conclusions to the class.

Pre-Lab Preparation:

1. Make sure you have read and reviewed the posted NMR lecture notes and Chapter 16 of the Klein (1st edition) text.
2. For each of the unknown compounds listed below, draw the chemical structure in your lab notebook and label all functional groups. Also determine the IHD (index of hydrogen deficiency) for each compound.
3. Instructions for using the picoSpin 45 MHz NMR spectrometer will be provided in lab.

Experimental Results and Class Presentation:

Students will work in pairs and run the ^{1}H NMR spectrum of an unknown organic compound chosen from those listed below. All of the compounds are liquids and the unknown samples will be spiked with TMS (tetramethylsilane) at approximately 10% (*v/v*). The TMS signal should be easily observed and will be used to normalize the spectrum of your unknown.

The spectra will be acquired during the first week of this lab. For the following week, each pair of students will present their experimental results and identification of their unknown to the class, including a complete analysis of the ^{1}H NMR spectrum. Bring a flash drive to lab so you can obtain an electronic copy of the spectrum of your unknown to use in the presentation.

<u>**Possible Unknowns**</u>

Ethyl acetate	2-Butanone
Ethyl benzene	4-Methoxybenzaldehyde
Methyl salicylate	2-Furaldehyde
1-Chloropropane	1-Chloro-2-methylpropane
trans-Cinnamaldehyde	Propanoic acid

In the class presentation, include the structure of the compound either neatly drawn by hand or using ChemDraw (chemical structure drawing software loaded on the iPads that are available for

student use from the library). On your drawing, show all hydrogen atoms and label each unique set A, B, C, etc. For the interpretation of your ^{1}H NMR spectrum, assign each observed signal using these labels. Discuss the number of signals, the chemical shift values, integration of the signals, and splitting (multiplicity), including coupling constants (J values) if observed and interpretable.

Turn in a copy of your ^{1}H NMR spectrum with the unknown code indicated along with your pre-lab notebook pages. Also prepare a table for the interpretation of your spectrum and identification of your unknown, as shown below. You do not need to write an experimental procedure, observations, or conclusion in your lab notebook. The class presentation, the labeled structure of your compound, and the table showing the interpretation of your spectrum will suffice.

Example of Data Presentation

2-Methylpropanoic acid ^{1}**H NMR Spectral Data**
(unknown Z)
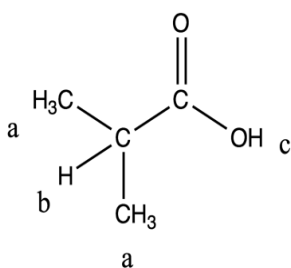
Chemical Shift (ppm)	Integration	Splitting	Coupling Constant	Assignment
1.16-1.24	6 H	doublet	J_{ab} = 6.7 Hz	a
2.44-2.75	1 H	septet	J_{ab} = 6.7 Hz	b
11.88	1 H	singlet		c

^{1}**H NMR Spectrum of 2-Methylpropanoic acid**

HSP-02-012