

Nuclear Magnetic Resonance (NMR). Identification of an Unknown Substance.

Reading Techniques 26 and 27 in Pavia (3rd or 5th edition), Chapter 15 in Klein (3rd edition)
Find lots of resources in the following handouts at the website: Online NMR Resources.
¹H (Quick Guide) and ¹³C (modified) 1D Spectrum Guides

Homework Pavia (5th edition): 1-10 (pp. 927-933)
Klein (3rd edition), In-text: 15.1-15.31; End-of-chapter: 15.32-15.62; Integrated Problems: 15.63-15.77 Challenge Problems: 16.67

Description

You work for Crime Scene Investigator (CSI) – Laney. You and your partner have recently discovered an unknown substance at a crime scene (used, undoubtedly, for some adverse or otherwise devious act) that is believed to be among the following list of possible compounds:

toluene
o-, *m*-, or *p*-xylene
2-butanol
ethyl benzoate
aspirin
methyl salicylate
diisopropylethylamine
ethyl methacrylate
p-anisidine

As one of the most reliable methods of structure analysis found in the CSI-Laney laboratory, NMR spectroscopy, you reason, is the appropriate method to reveal the identity of the substance (aside from a simple melting point or boiling point; you would use the GC/MS but it is down for maintenance).

Prelab

Add the usual entries in your notebook, **Name**, **Date**, and **Title**. In the provided worksheet, show the structure for each of the compounds listed above. For each compound indicate the total number of *chemically equivalent* protons (¹H) and carbons (¹³C). Read through the Techniques in Pavia and/or Klein for the background on the NMR theory and how to determine the number of expected peaks and reach through the NMR guides above for the actual operation of the instrument.

Procedure

Your instructor will demonstrate how to run the NMR instrument during laboratory. Choose one of the unknown samples and note the code in your notebook (The samples have already been prepared for the NMR experiment). Obtain the proton NMR spectrum, use the guide to phase the spectrum and integrate the peaks, then print. Also obtain the carbon NMR spectrum and print.

To Complete the Experiment – Partial Report

Analyze the NMR spectra and determine the identity of the unknown compound. Report the chemical shifts for each type of proton and carbon present in your sample and report the multiplicity and integration using the appropriate notation. Be sure to show the structure of your identified unknown.

Chemical shifts (δ) in units of ppm are reported from lowest to highest values using the following notation:

¹H NMR (ppm) δ : value1 (*m*, *x* H), value1 (*m*, *y* H),(where *m* is the splitting and *x* and *y* are the integration values – e.g. 3 H).