Anasazi Eft-60 Nuclear Magnetic Resonance Spectrometer
Acquisition of 1-D, ^{1}H-NMR Spectra

The Eft-60 is a 60 MHz pulsed Fourier transform NMR spectrometer that makes use of a permanent magnet (Varian EM360) and data acquisition and control software by Anasazi Instruments to allow rapid collection of 1-D and 2-D proton and carbon-13 spectra.

Provided here are basic instructions for acquisition of 1-dimensional, ^{1}H-NMR spectra.[1] For more advanced applications, the user should consult the *ft Spectrometer Guides* at: http://www.aiinmr.com/documents/Guide_041019_rev_P.pdf. The user will also wish to consult one or more of the following sources to secure a fundamental understanding of the principles on which NMR spectroscopy is based.

- McMurray, J. Structure Determination: Nuclear Magnetic Resonance Spectroscopy (CH 13). In *Organic Chemistry, 6th ed.*; Brooks-Cole: Canada, 2004.
- Skoog, D.A.; Holler, F.J.; Crouch, S.R. Nuclear Magnetic Resonance Spectroscopy. In *Principles of Instrumental Methods, 6th ed.,* Brooks-Cole: Ontario, Canada, 2006.
- Rubinson, K.A.; Rubinson, J.F. Nuclear Magnetic Resonance Spectrometry (CH 11). In *Contemporary Instrumental Analysis*, Prentice-Hall: Upper Saddle River, NJ, 2000.
- Pavia, D.L.; Lampman, G.M.; Kriz, G.S. Nuclear Magnetic Resonance Spectroscopy, Part One: Basic Concepts (CH 3). In *Introduction to Spectroscopy, 3rd ed.*, Brooks/Cole: Singapore, China, 2001.

BASIC OPERATING PROCEDURES:

1. Preparing the Bulk Solvent

The solvent used for ^{1}H-NMR should not produce hydrogen signals that will complicate the spectrum for your sample. Typically, proton-free solvents like carbon disulfide, CS_2, or carbon tetrachloride, CCl_4, or deuterated solvents such as deuterochloroform, $CDCl_3$, or deuteroacetone, CD_3COCD_3, are utilized. Also added to the bulk solvent is the internal standard tetramethylsilane (TMS). Commercially available solvents often are spiked with the TMS reference, but if not, you will need to prepare your bulk solvent of choice with 1-2% TMS, which can be stored for extended periods in a laboratory refrigerator. You will wish to read the relevant Material Safety Data Sheets to familiarize yourself with potential hazards when working with these solvents.

How do you select an appropriate solvent for use? It may be that your instructor suggests to you the most useful solvent for your sample, but if not, you will likely wish to consider solubility of

[1] It is assumed that the instrument has already been prepared for use by completing Sections A-D in the Instrument Setup Guide using a sample that is similar to those which will be measured.

your sample in the solvent, cost, and safety as you select a bulk solvent. Since deuterated samples are very expensive, it is wise to test the solubility of your sample in the non-deuterated solvent before attempting to prepare the deuterated sample solution.

2. Preparing the Sample

a. Solid Samples: Deposit roughly 25 to 50 mg of your sample into a clean glass vial, and add about 2 mL of bulk solvent containing 1-2% TMS. Mix well to assure complete dissolution of your solid sample. Filter out any particulates or undissolved materials using a Pasteur pipet packed with a small amount of glass wool. Add about 0.5-1.0 mL of the filtered solution to a clean NMR tube, and cap and label the tube. (A helpful series of images showing this process can be found at: **http://www.csi.edu/ip/physci/faculty/rex/Nsolsprp.htm**)

b. Liquid Samples: Deposit roughly 1 drop of neat liquid into a clean glass vial and add about 2 mL of bulk solvent containing 1-2% TMS. Mix well and filter out any particulates using a Pasteur pipet packed with a small amount of glass wool. Add about 0.5-1.0 mL of the filtered solution to a clean NMR tube, and cap and label the tube.

3. Inserting the Sample into the Spectrometer

a. Assure that an entry has been made in the instrument log to indicate your use of the instrument. It is possible that your instructor may have done this for the class.
b. Wipe your sample tube with a Kimwipe to remove all fingerprints.
c. Lift the lid on top of the magnet to reveal the sample compartment. Press the eject button (on the inside front left-hand corner). If a sample tube is already present in the sample chamber, set your sample tube aside momentarily, and remove the sample tube from the sample compartment by grasping the tube near the cap. Gently, using a Kimwipe, remove the spinner (the white holder around the tube). Set the old sample aside.
d. While holding the spinner with a Kimwipe, gently insert your sample tube into the spinner such that the bottom of your tube sticks out about a couple of centimeters.
e. Carefully insert your tube into the black depth gauge on the front of the magnet until you feel the bottom of your tube hit the bottom of the gauge. (The spinner will slide up the tube to the appropriate height.)
f. Again wipe the spinner and your tube with a Kimwipe to remove all fingerprints.
g. Turn the spinner off by flipping the black lever found next to the eject button to the up position.
h. Push the eject button in, gently place your sample into the sample chamber, and then release the eject button to allow your sample tube to sink into the probe.
i. Turn the spinner on by flipping the black lever down, and assure that you hear your sample tube spinning. You are now ready to obtain your spectrum!

(A helpful series of images showing this process can be found at: **http://www.csi.edu/ip/physci/faculty/rex/Ntubeins.htm**)

4. Acquiring a Spectrum

a. Open the PNMR program, and verify that the H1 prompt is displayed at the bottom of the screen. If not, type nu H1, and hit <Enter>.
b. To *acquire* your data, type zg, and hit <Enter>.
c. When prompted to enter a file name, find and open the folder labeled with your course number and lab section by double clicking it with the mouse. Type a filename that includes the course number, your initials, the date, and a description of your sample (*e.g.*, CH113_LE_013107_ethylbenzene).
d. A gray box will appear in the upper right hand corner of the screen and will disappear when the data have been collected.

5. Processing a Spectrum

a. To *process* your data (*i.e.*, to reference TMS, pick peaks, integrate, accept spectral information, and print), open the NUTS program, and hit <CTRL+F1>.
b. In the window that pops up, enter the sample name into the comment box, enter your name into the User box, and click OK.
c. Assure that the printer is on, and then click OK again to print your processed spectrum.
d. NOTE: If you are not pleased with what you see (*e.g.*, too many peaks have been selected, the integration values seem unreasonable, etc.), it may be necessary to complete each processing step independently as follows:

Step-by-Step Processing

e. To *process* your data, open the NUTS program, and select <CTRL+F2>. Find and open your saved file in the list.
f. To *adjust the phasing* of your spectrum:
    i. Type zo to enter the zoom routine.
    ii. Select your first region of interest by dragging the cursor over a strong peak on the left and entering <1>. Drag the cursor over a second strong peak on the right of your spectrum and press <2> to assign a second region of interest.
    iii. Type pe to enter the phase trimming routine.
    iv. Next, phase the peak on the left side of your spectrum by pressing and holding the left mouse button while sliding the mouse from side to side. Repeat using the right mouse button to adjust the peak on the right hand side of the spectrum. Your goal is to align the tails of each peak in the spectrum with the baseline.
g. To *fit the baseline* of your spectrum:
    i. Type fb to enter the fit baseline routine.
    ii. Red stripes will appear on the spectrum. Click with the mouse to remove stripes that are overlapping with peaks or that appear to be too close to peaks in order that these data points are not included in the least squares fit of the baseline.

iii. Type the letter l (not the number 1) to execute the least squares fit of the baseline.
iv. Save the result and exit the baseline fitting routine by hitting <Enter>.

h. To *integrate peaks* in your spectrum:
   i. Type id to enter the integral display mode.
   ii. For each peak or set of peaks in your spectrum that you wish to integrate, double click the left mouse button twice on the left side of the peak and once on the right side.
   iii. If you wish to assign a relative integral value to which all others will be adjusted, place your cursor on the integral, click the left mouse button one time, type v for value, and enter the integral value.
   iv. Select <Enter> to exit the integral display mode.

i. To *pick peaks* in your spectrum:
   i. Automatically—Type pp, and vertical red lines will indicate the selected peaks. Use MH and RM to change the peaks that are selected.
   ii. Manually—Type dp and the cursor will change to a crosshair with the label DP. Type a to automatically pick peaks, c to clear all peaks, and k to remove a single peak nearest the cursor. Add a peak by clicking the left mouse button near the peak. When you have selected the desired peaks, type t to list the peak values in a table. When you have finished, type dp to exit the manual peak pick mode.

j. To plot your spectrum, type pl at the prompt.

6. Removing the Sample from the Spectrometer

a. Lift the lid on top of the magnet to again reveal the sample compartment. Press the eject button, and remove your sample tube from the sample compartment by grasping the tube near the cap.
b. Gently, using a Kimwipe, remove the spinner (the white holder around the tube), and carefully set it aside for the next user.
c. If you are the final user of the instrument, prepare in the spinner (as described previously in the inserting the sample section) and return to the sample chamber the water sample found next to the instrument.
d. Dispose of your waste and store your tube as detailed by your laboratory instructor.