

# Nuclear Magnetic Resonance

Nuclear Magnetic Resonance (NMR) is a spectroscopic technique widely used by organic chemists. NMR provides details about the location, number and environment of specific types of atoms within a sample. The proper interpretation of this information allows detailed chemical structures to be discerned. The same basic instrumentation used in this week's experiment are used in physician offices when running MRI (magnetic resonance imaging) of body parts.

NMR is a relatively recent invention. The theory behind the process was first understood in the 50's with the 1952 Nobel prize in physics going to Felix Bloch and E. M. Purcell<sup>i</sup>. It took virtually 10 years until the first commercial NMR was produced in 1963<sup>ii</sup> and large research universities began acquiring and using NMR by the mid 1970's. Many universities still do not have their own NMR machines due to their significant expense. A state of the art 600 MHz NMR can easily cost 2 million dollars and a 'relatively inexpensive' educational 300 MHz may be obtained for approximately \$ 600,000.

Your instructor will give a lecture on the practical skills needed to properly interpret <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra. A number of problems will be worked through together in the class. A separate set of problems will be given during the class to solve on your own or in small groups. Your daily grade will be assigned based on this problem set and not on your pre-lab. (This is one of a few experiments this semester in which your pre-lab will not be graded. It is still a good idea to write a short prelab even though you will not be given points based on the work.) Each student will be assigned an unknown sample. Each student will run their unknown on the 60 MHz NMR and obtain a <sup>1</sup>H NMR spectra. The empirical formula, IR, and <sup>13</sup>C NMR will be provided for each unknown.

The interpretation of the data will result in a chemical structure being assigned to the unknown. In lieu of a lab report for this experiment, each student will complete a provided data sheet for their unknown. This data sheet is due at the beginning of the next lab.

## **PRE-EXPERIMENT ASSIGNMENT**

Read and study this chapter of the manual and the on-line notes on the Organic Chemistry web site. You may also find it helpful to read and study your organic lecture notes on NMR.

**A student who has prepared for the Nuclear Magnetic Resonance experiment should be able to:**

- 1) List the four main pieces of information that can be obtained from  $^1\text{H}$  NMR spectrum.
- 2) Understand and be able to list some atoms which are 'visible' in the NMR.
- 3) Simply explain how an NMR works.
- 4) Given a structure, predict how many non-equivalent signals will show in the  $^1\text{H}$  NMR (how many non-equivalent hydrogens).

Quizzes given after the experiment has been performed may include:

- 5) Predict the NMR shift of a specified hydrogen or carbon if given a chemical structure.
- 6) Predict the splitting pattern of a specified hydrogen in an NMR if given a chemical structure.
- 7) Predict the relative ratios of specified hydrogens seen in the NMR if given a chemical structure.
- 8) Give the chemical structure if given the  $^1\text{H}$  NMR,  $^{13}\text{C}$  NMR, IR and empirical formula.
- 9) Answer practical questions about the operation of an NMR.

Your instructor will begin by explaining how a NMR works and going over in some detail steps to properly and thoroughly explain NMR spectra. There is not one correct manner in approaching this problem. You may experience slightly different methodologies in lecture, drill and lab. Understand and utilize the method which works best for you and stick with it.

Your instructor will go over portions of problem set 1 as a group exercise. Unknowns will be assigned and students will begin to take their own spectrum. With 1 ½ hours left, problem set 2 will be distributed. You may work in small groups on problem set 2. The answers to problem set 2 will be turned in at the end of class. This will be used to assign the notebook grade (5 points) for today's class.

### **The experiment**

There is only one NMR in NCF 371 due to the cost (approximately \$100,000) of this instrument. Students will take turns running their assigned unknown number. When it is your turn, retrieve the NMR tube with your number from the hood. Keep the NMR tube in an upright position. Take your sample into 371.

You will insert your sample into the machine. Instruct the machine to analyze your sample. Wait while your sample is being analyzed, then instruct the machine to transform and display the data in a standard manner. The spectrum will be printed. The exact steps necessary to perform this sequence are written on a separate NMR procedure which

will be available adjacent to the NMR in the instrument room. There will be someone in 371 to assist you.

After you have successfully obtain a spectrum, replace the label on the NMR tube and return the sample in numerical order in the NMR tube rack in the hood. Be careful with the NMR tube, these are delicate and expensive.

Before you leave class, check with your instructor that the integration values obtained on your spectrum are adequate. Alternative integration values or entire spectra may be provided.

### **POST-EXPERIMENT ASSIGNMENT**

Work individually on interpreting your  $^1\text{H}$  NMR,  $^{13}\text{C}$  NMR, IR data. If help is needed, consult with your lecture notes, the on-line notes or your lab instructor. Complete the questions on the NMR Datasheet provided. Staple your spectra to the datasheet and turn this in at the start of the next class period.

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<sup>i</sup> Felix Bloch - Nobel Lecture". Nobelprize.org. [http://nobelprize.org/nobel\\_prizes/physics/laureates/1952/bloch-lecture.html](http://nobelprize.org/nobel_prizes/physics/laureates/1952/bloch-lecture.html) (December 30, 2010)

<sup>ii</sup> Bruker History <http://www.bruker.com/50years.html> (December 30, 2010)

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