University of Wisconsin
Chemistry 524
Fourier Transform Molecular Spectroscopy[i]

For this laboratory exercise, you will be introduced to two important molecular spectroscopic instruments and use each one for an analytical application. The two instruments are: a Fourier Transform Infrared Spectrometer (FTIR) and a Nuclear Magnetic Resonance (NMR) Spectrometer. These instruments are similar in that they are both based on Fourier transform techniques, but they are quite different in the instrumentation details and the types of information they provide.

NMR Experiment

In this experiment, you are going to use NMR to measure the ethanol content in a wine sample. The weight percent ethanol is determined by comparing the peak area due to water protons to the peak area due to ethanol protons in the sample.

The sample preparation is straightforward and has already been done for you. The wine was filtered through a glass wool plugged transfer pipet into an NMR tube to a height of about 4 cm. The tube has already been placed into the NMR.

There are two software programs running simultaneously on the computer. PNMR is the data acquisition program and NUTS is the processing software. You can alternate between the programs using Alt-Tab.

Verify the following acquisition parameters in PNMR. The parameters can be changed by entering the two letter command at the H1> prompt. No second dimension nucleus will be used.

		First dimension	
SI	16384	N1	H1
NS	4	F1	60.010 MHz
RG	2	W1	1000 Hz
RD	5 sec	O1	360 Hz
		PW	5 µsec

Collect the FID using “zg” at the H1> prompt. Switch to NUTS and process the data with the following commands:

zz	imports the FID into NUTS
bc	subtracts any baseline due to DC offset
mf	fits the data to the window
pl	prints your FID for your report
ft	computes the fourier transform
mf	fits the data to the window

To make the spectra look better you will need to "phase" it by:
highlight the water peak with the "zo" (zoom) command and type "1"
highlight the methyl protons and type "2".
Return to the main prompt using "enter"
Type "pe". In this routine the left mouse phases the region near the water peak, and the right mouse phases the region near the methyl peaks. After phasing, return to the main prompt.

The baseline adjusted using the "fb" (fit baseline) command. Highlight in pink the regions that you want to use for a baseline correction. Fit the baseline using "L", this applies a least squares polynomial fit to the baseline. Type "p" to see the fit on the display. Press "enter" to apply the fit. Type "bc" to correct for DC drift in the baseline.

To integrate the peaks, type "id". Remove the previous users integral lines by typing "c". Use the left mouse to select the region for integration. The integral is calculated and displayed on the plot. Integrate the –OH region and the methyl region. Print your spectrum showing the integrated intensities.

Use the integrated areas to determine the mass percent ethanol in the wine sample.

FTIR Experiment

In this experiment you will use the FTIR to measure the thickness of a thin polymer film. While doing this, you will look at how FTIR data is collected and processed. You will also use the attenuated total reflection accessory (ATR) to collect the FTIR spectra of the polymer.

Click on the OPUS icon to start the FTIR software. Login as the chem524 user with "op$$amp" password. Be sure the sample compartment is empty.

Click on the measurement icon, the green test tube, to set up the collection parameters. Load the interferogram.xpm experiment file. This experiment file is very basic. It takes only one scan and records the interferogram. With the sample compartment empty, take a background scan. When the background is finished, put in the polystyrene sample and take a sample scan. The interferogram of the sample should now be plotted in the window. Display the reference interferogram by changing its color to something visible. Use the "stacked" view and print the interferograms using the "quick print" tool. **Qualitatively describe how the reference interferogram compares to the sample interferogram.**

Use the "interferogram to spectrum" tool tool to fourier transform the sample and background data. Use the Power Spectrum phase correction and a boxcar apodization function with zero filling order of 1. Zoom in a region in the sample where the interference fringes are easily seen. You probably notice that the spectrum does not look very clean. Redo the FT using a Blackman-Harris 3 term apodization and a zero filling factor of 4. Did this clean up the spectrum? **Compare the background spectrum to the sample spectrum. How would an absorbance spectrum be calculated?**

Load the polystryrene.xpm file and recollect the background and sample spectrum of the polystyrene film. This experiment file automatically takes the FT and calculates the transmittance.

Measure the difference in wavenumber between the fringes and calculate the polystyrene film thickness. Page 406 in your text will help you with this calculation. The refractive index of polystyrene is 1.57.

Place the ATR sampling accessory in the sample compartment. Load the polystyreneATR.xpm experiment file. Collect the background. Place the polystyrene sample from a coffee cup on the ATR and collect the spectrum. **How does the ATR spectrum of the Styrofoam cup compare to the thin film?**

[i] These activities were developed by R. McClain, and updated in April 2010. The ethanol content activity was suggested by F. Contratto of Anasazi Instruments in 2004.