Exploring FT- IR

FT-IR spectroscopy is rather versatile and great for Qualitative analysis. In this lab you will analyze different samples by some of the sampling methods available to us and you will perform a few investigations of the FT-IR instrument itself. IR can be run in transmittance mode, using salt plates, gas cells or liquid cells; on solids via transmittance (pellet) or reflectance; or in a mode called attenuated total internal reflectance. You will also investigate IR fine structure and the effect of mirror movement on resolution. In your report, you should describe the instrumentation in the FT-IR and discuss how the FT-IR works.

IR Fine structure: Water Vapor and CO_2

1.) Go to Instrument, Scan
2.) Go to the Instrument tab and change the resolution to 2 cm^{-1}.
3.) Hit the Advanced Button and unclick the CO2/H2O correction.
4.) Go back to the scan tab and change the Sample to Background.
5.) Scan a background of CO_2 and water vapor.
6.) Identify the peaks for CO2. Identify the stretch involved and the reason for the doublet in appearance.
7.) Identify the water vapor peaks (you should find a symmetric, asymmetric and bending modes for water vapor).
8.) Calculate the difference in wave number between any 2 adjacent peaks near 1600 cm^{-1} and explain the appearance of multiple bands for the H_2O bending vibration (that helps answer # 7 huh?).
9.) Based solely on your IR data, explain why water in a lake or an ocean looks blue. Specifically, determine where pure liquid water would absorb in the VISIBLE region. How is this affected by hydrogen bonding? Hint: it has something to do with the peaks you identified above…think about it and do a little math.

THE FT in FT-IR

1.) Go to Instrument, Scan
2.) Go to the Instrument tab and change the resolution to 16 cm^{-1}.
3.) Use a polystyrene IR card and obtain a spectrum.
4.) Print the spectrum.
5.) Go to Instrument, Scan
6.) On the Scan tab, select Interferogram and collect an interferogram and print it.
7.) Repeat this process for a resolution at 2 cm^{-1}.
8.) Identify and label the peaks in polystyrene (include the fingerprint region).
9.) Calculate the length of the mirror drive in both cases.
10.) Comment on the appearance of both the interferogram and the spectra in both cases.

Salt plates, liquid cell. If actual liquid cell is not available, sandwich 2 salt plates together.

a. Collect a spectrum of neat o-xylene, m-xylene and p-xylene (remember, IR is very

path length dependent, be sure you have a small coating of sample). Clearly label ALL peaks and indicate the stretch that is responsible for the peak. For o,m and p-xylene, you MUST investigate the fingerprint region in order to distinguish between the compounds. WHY??? Why do the peaks shift when the substitution is changed?

b. Obtain a mixed xylene sample. Take it's IR spectrum. Determine the relative composition of the mixed sample, using the following multicomponent absorbance equations. The values for concentration are in g/L. Give a brief discussion on the origin of these equations (you don't have to derive them, just understand how to use them and where they come from). Why is there no "b" term in the equations? How will your calculations be affected if your baseline is not flat?

$$A_{\lambda p} = 1.506c_p + 0.048c_m + 0.000c_o$$
$$A_{\lambda m} = 0.025c_p + 1.440c_m + 0.000c_o$$
$$A_{\lambda o} = 0.032c_p + 0.033c_m + 2.405c_o$$

c. Question: Why is IR one of the best methods for resolving these isomers in a mixture? It is even better than MS, WHY???

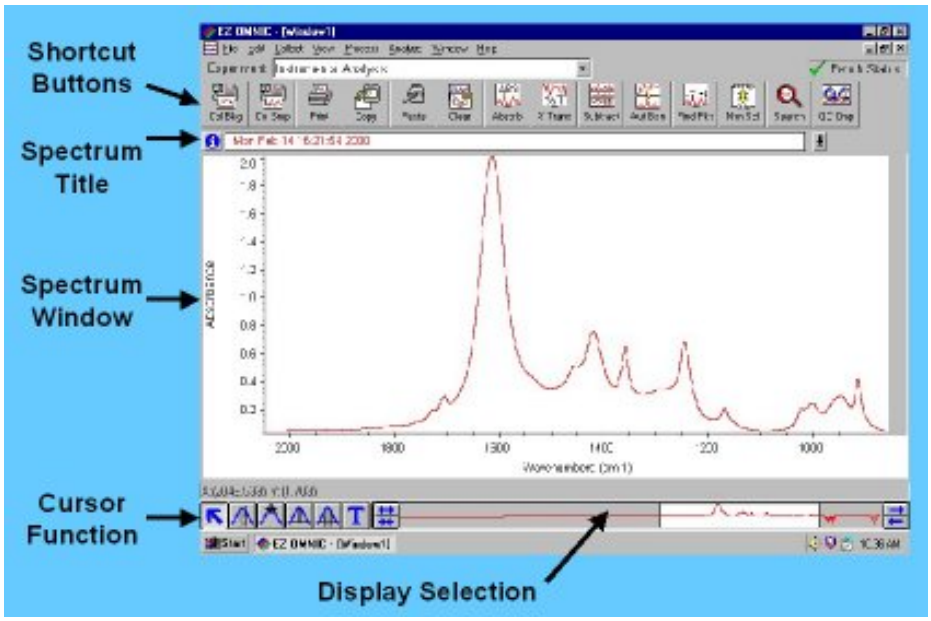
ATR-IR of paint and polymer films.

You have been asked by your employer, AABI (Armstrong Atlantic Bureau of Investigation) to construct a library of ATR-IR spectra of paint and polymer samples to help identify samples from crime scenes.

Have the instructor show you how to work the ATR cell and collect spectra of 2 paints and 1 polymer (Saran Wrap, gloves etc.). Name the files accordingly and identify the components of the sample.

Nicolet OMNIC Software

Our Nicolet Avatar FT-IR is controlled using OMNIC software. It is highly recommended that you take a few minutes to examine the OMNIC tutorial to get a better understanding of the software. The tutorial can be started by double clicking on the "OMNIC E.S.P Tutorial" icon on the Windows desktop. To start the data collection software click on the "EZ OMNIC E.S.P. 5.1" icon on the Windows desktop. Below is a screenshot of the OMNIC software. Click on a portion of the picture for a brief description.



Shortcut Buttons:

These useful shortcuts will save you some time navigating through the OMNIC menus. Particularly useful are the buttons to collect a new sample and collect a new background. Other buttons will change the display in the Spectrum Window.

Spectrum Title:

This pull-down list will allow you to see all of the active spectra and overlay multiple spectra in the Spectrum window. This is a very useful feature, but can result in a cluttered window unless you know where to find the "Hide Spectrum" command. Look in the "View" menu for this.

Nicolet Avatar FT-IR Barebones Guide

1. The IR and computer should already be turned on. If they are not, turn them on and let the laser warm up for about 30 min.

2. If you wish to use a nitrogen atmosphere for your sample, be sure the nitrogen tank valve is open and turn the knob on the flowmeter until the flow is around 7. Let the nitrogen purge for a couple minutes before running any samples.

3. Open the Omnic software (double-click icon on desktop).

4. Set the number of scans by clicking Collect->Experiment Setup. Click on the Collect tab and change the number of scans as needed (default is 32).

5. Put your background sample (either a clean salt plate or pressed KBR) on the sample holder. Click on the Collect Background icon  and click OK to start the scan.

6. After collecting the background you don't need to add it to the data window (ie. click no).

7. Put your sample in the holder. Click on the Collect Sample icon  . Type the name of your sample and click OK to start the scan.

8. When the sample is done being scanned, click YES to add the spectrum to the data window.

9. Save your spectrum by clicking on File->Save As and enter the filename and where you want to save it (hard drive, ZIP disk, etc).

Data Analysis:

1. To toggle between absorbance and transmittance mode, click on the appropriate icon:  .

2. To do a baseline correction, click on the  . You MUST be in absorbance mode to do baseline correction, but you may go back to transmittance after this correction completes.

3. To label your peaks click on the  icon (lower left). Click on each peak of interest and press enter to mark it with the wavenumber value. If you pick a wrong peak, click on the wavenumber value then press delete and then enter.

4. Your spectra can be printed by going to File->Print.

5. More detailed information about acquisition can be printed out with the spectra. The templates for these can be found under the Reports menu.

Spectrum Window:

Here's where the action is. Pretty self explanatory.

Cursor Function:

Selecting a different cursor function will allow you to integrate peaks, add text to the spectrum, determine peak height and position, as well as other useful actions.

Display Selection:

This portion of the window displays the entire spectrum that has been collected or that has been stored in a file (typically from 400-4000 cm^{-1}). By clicking and dragging on the vertical bars at the interface between the grey and white sections, you can easily change the region of the spectrum that you are viewing. You can also click and drag the entire white section along the x-axis to view a smaller portion of the spectrum.