The Perkin-Elmer Paragon 1000 FT-IR spectrometer is a bench-top instrument that provides interferometer alignment ports, a screen, a sample compartment, a disk drive, and a power switch in one self-contained unit.

Warning!

DO keep the Paragon 1000 dry. Avoid spilling liquid into the instrument, especially into the top of the display-screen housing, which contains a high-voltage supply. Clean all external spills immediately. If anything that is spilled enters the main body of the Paragon 1000, switch off the power and call a Perkin-Elmer Service Engineering.

DO NOT stare into the laser beam. The Paragon 1000 contains a low power, visible (red) laser; momentary exposure to the beam is not dangerous, but deliberate, direct viewing of the beam along its axis could damage your eye.

Please read section 12.7 of the textbook. We will study the theory of Infrared Spectroscopy during the last week of class, but in lab we will learn to obtain an experimental spectrum much earlier. The following web sites will provide you with additional information:

http://www.brynmawr.edu/Acads/Chem/mnerzsto/study_aids.htm

http://www.forumsci.co.il/HPLC/FTIR_page.html

http://www.ftir.cz/


http://www.spectroscopynow.com/Spy/basehtml/SpyH/1,,3-4-9806-0-9806-directories--0,00.html

http://www.wpi.edu/Academics/Depts/Chemistry/Courses/CH2670/infrared.html

For preparation of samples – KBr


http://www.dq.fct.unl.pt/qoa/jas/ir.html

Infrared radiation, which is electromagnetic radiation of longer wavelength than visible light, is detected not with the eyes but by a feeling of warmth on the skin. When
absorbed by molecules, radiation of this wavelength (typically 2.5 to 25 um) increases the amplitude of vibrations of the chemical bonds joining atoms. The presence and also the environment of functional groups in organic molecules can be identified by infrared spectroscopy. The small quantity of sample needed, the speed with which a spectrum can be obtained, and the wide application of the method combine to make infrared spectroscopy one of the most useful tools available to the organic chemist.

**Scanning and Viewing:**

1. Before the Paragon 1000 can collect a sample spectrum it needs a valid background spectrum against which it can ratio the sample scan. Make sure that the sample slide is empty and press the green long SCAN button, then BACKG, and finally the button below EXECUTE on the screen. When the scan is complete, the background spectrum is displayed briefly. It is immediately replaced with the default region. Default region: The Paragon 1000 contains four memory regions for the temporary storage of spectral data. These regions are called spectrum X, Y, Z, and Backg. The Paragon 1000 always designates one to be the default region. We use region X. To display the background spectrum, press BACKG.

2. Open the sample compartment by lifting the cover using the recess at the front. Place the sample on the sample holder and close the sample compartment.

3. Press the green scan button, choose a region W, Y, or Z, indicate 4 scans, and press the button below execute. Wait until the four scans are done and the computer has finished calculating and has returned to “ready.”

**Viewing More Than One Spectrum**

The Paragon 1000 can display up to three spectra at one time. This helps you to compare spectra, for example, when you are comparing the product to the starting material.
Press the Z button and hold it down while you press X. Release both keys. Both X and Z spectrum regions are displayed, overlaid. The two spectra are displayed in different colors.

**Using the Vertical Cursor and Mark Buttons**

You can use the vertical cursor to determine the wavenumber and ordinate value of a peak and the mark button to display the values on the printed spectrum.

1. Press shift then vcursor (vertical cursor). A vertical line appears.
2. Move the line with the arrow buttons to the peak you want to mark.
3. Press shift then mark and go to the next peak.
4. Before plotting the spectrum, remove the vertical line by pressing shift then vcursor again.

**Automatic Expansions**

The autex button (automatic expansion) button will expand the spectrum vertically to fill the display area. This only changes how the spectrum appears on the screen, the data is not changed. To use the automatic expansion: press shift then autex.

**Plotting the Spectrum**

To plot the spectrum, press plot. The print button will print the entire screen.

**Spectra of Neat Liquids:**

To run a spectrum of a neat (free of water!) liquid, remove the NaCl plates from the dessicator. Be sure to handle the NaCl plates very carefully. They are very expensive, very breakable, and not easy to replace. Pick up the plates by the edge only. You do not want an IR spectrum of your oily fingerprint. Place a drop of the liquid on one plate, and carefully place the other NaCl plate on top. Press the plates together gently to remove any air bubbles. Place the plates in a vertical position on the sample holder and obtain a spectrum. Mark and expand your spectrum as necessary and then plot. While your spectrum is plotting, carefully remove the NaCl plates and rinse them with acetone. Replace the dry plates in the envelopes and dessicator for the next student.
**Potassium Bromide (KBr) Disk**

The spectrum of a solid sample can be run by incorporating the sample in a potassium bromide (KBr) disk. This procedure needs only one disk to cover the entire spectral range, since KBr is completely transparent to infrared radiation. Although very little sample is required, making the disk calls for special equipment and time to prepare it. Since KBr is hygroscopic, water is a problem.

**Recommended Method**

1. Transfer weighed amounts of sample, approx 2 mg, and KBr powder, approx. 200 mg into an agate mortar. The KBr powder must be of spectroscopic grade purity, and be spectroscopically dry.

2. Grind the powders together, with an agate pestle, until the sample is well dispersed and the mixture has the consistency of fine flour.
3. Gather all the equipment for making the KBr pellet.

4. Place one of the screws into the bolt and add one small scope of the KBr mixture.

5. Place the second screw into the bolt and hold by the vise clamp.
6. Apply pressure to the KBr sample by tightening the second screw.

7. Remove both of the screws and place the bolt on the sample holder in the sample compartment of the FT-IR. The KBr should transform into a translucent window.

_The spectrum quality is affected by the quality of the disk. The flatness of the baseline is dependent on the particle size and dispersion of the sample in the KBr powder. Check the disk and spectrum for the following faults:_

A. If the disk breaks on removal from the die, this indicates that the disk is too thin caused by too little powder, or too much pressure for too long. Remedy this effect by increasing the sample load.

B. If the disk is not translucent, this can have numerous causes.

1. Uneven distribution of powder in die
2. Too much sample
3. Too much KBr powder
4. Poorly dispersed sample
5. Water in disk
6. Pressed at too low pressure or for too short a time

All except the water in the disk can be remedied by re-grinding and pressing with adjusted amounts. Water in the disk can be present due to a wet sample or wet KBr powder. Heating the disk at approx 100°C for a few minutes and repressing
the disk will sometimes remove residual water.

C. Truncated Bands. If the spectrum contains bands which have a flattened turning point and do not reach 0 % T, this is caused by a poorly dispersed sample or holes in the disk. Check the disk visually and if necessary repeat the preparation.

D. Sloping baseline. This is usually due to a poorly dispersed sample.

The faults listed above have been commonly found, sometimes in publications, but is not necessarily exhaustive. Use of other halide salts can overcome some effects, or extend the range of the spectrum examined, e.g. CsI extends the lower wavenumber range from 400 to 200 cm⁻¹. Poor sample preparation can lead to avoidable errors in interpretation of the resultant spectrum. A little care can avoid the need for repetitious sample preparation or embarrassing errors in results.