Infrared Spectroscopy

Infrared Spectroscopy is an analytical method that measures the absorbance of a select band of electromagnetic radiation by a sample. The wavelength of radiation that is absorbed is characteristic of different types of chemical bonds. The graphical representation of the absorption of the electromagnetic radiation by the sample makes an IR spectrum.

Organic chemists use infrared spectroscopy (IR) as a means of identifying functional groups within compounds and in what type of environment these groups exist. This information is used to determine the structure of compounds. IR is usually used in combination with other techniques, especially nuclear magnetic resonance spectroscopy (NMR).

In this class you will learn how to interpret an IR spectrum and how to operate a Perkin-Elmer infrared spectrometer. During this experiment you will obtain an IR spectrum of an unknown compound and use this information to identify the material.

PRE-EXPERIMENT ASSIGNMENT

Study this chapter of the manual, the lecture notes on the Chemistry Department web site, the Sample IR Spectra posted on the web site and the appropriate section in your lecture textbook. Do the first six parts of your notebook writeup.

A student who has prepared for the Infrared Spectroscopy experiment should be able to:

1. Identify the relative energy, frequency, and wavelength of infrared light compared with other types (including gamma rays, x rays, ultraviolet and visible light, microwaves and radio waves). Also, identify the change that occurs in molecules when infrared light is absorbed.
2. Explain the relationship between frequency, energy, wavelength and wavenumbers.
3. Define the 5 major absorbance regions in the IR. Which functional group(s) absorb in each of these 5 regions?
4. If given a functional group, chose in which of the 5 regions it will absorb.
5 Define, identify, recognize, and explain the use of each of the following: IR (infrared) spectroscopy, neat samples, salt plates (including how to clean them), Nujol mull, KBr pellet
6. Draw the structure given the name, or give the name from the structure, of the compounds used in the day's experiment.
7. Identify and explain safety considerations for this experiment.
8. Perform the day's experiment safely and successfully.

Quizzes given after the experiment has been performed may also include:

9. Give or recognize a probable absorption frequency in cm^{-1} in the IR spectrum of a compound containing any of the following functional groups; Amines, Alcohols, Alkynes, Terminal Alkynes, Nitriles, Alkanes, Alkenes, Aldehydes, Carboxylic Acids, Esters, Amides, Aldehydes, Ketones, and Ethers. (Detailed instructions of spectrum interpretation can be found in "Sample IR Spectra" located on the organic web site.
10. Draw a reasonable IR spectrum if given a compound name or structure.
11. Determine the presence (or absence) of functional groups in a sample from infrared spectral data. The data may be given in the form of a list of peak locations (see #6 above) or as a spectrum.

Safety Precautions

All of the compounds you will take IR spectra of in this experiment are at least slightly toxic and flammable. Wash your hands after conducting the experiment.

Infrared radiation is relatively (but not completely) harmless. The safety instructions on the IR spectrometer say "Do not stare into beam," because long exposures can be damaging.

Obtaining the Spectrum

Handle salt plates with care. The salt plates are very sensitive to moisture and will break easily. Each pair costs approximately $25. Obtain an unknown compound. When you turn arrives, bring your notebook and your unknown into the instrument room. Go to one of the four IR spectrometers. IF you are the first one to use the instrument, remove the plastic container containing the salt plates from the desiccator. Remove two salt plates from the container. Replace the container back in the desiccator, replace cover on desiccator.

Place a clean kim-wipe on the counter. Place the two salt plates on the kim-wipe. Place a few drops of acetone on each salt plate. Using a new kim-wipe, rub each salt plate with the acetone, dry the salt plate with a kim-wipe.

Place 2 drops of you unknown on one salt plate. Place the other salt plate on top, sandwiching the compound. Place these salt plates into the sample holder.

Open the IR spectrometer door. Place sample holder in slot. Close door. Press large green "X" button. Press green "SCAN" button. Press the soft "4" key- This is the one under the monitor, not on the normal key pad. Wait about 18 seconds while the 4 scans are being taken. During this time you should see a small countdown clock in the lower right corner. When complete and spectrum looks reasonable, press "PLOT". A copy of your spectrum will start to emerge from the printer.

Open IR spectrometer door. Remove sample holder. Remove salt plates. Open salt plates. Place a few drops of acetone on each salt plate. Using a new kim-wipe, rub each salt plate with the acetone, dry the salt plate with a kim wipe. If another student is waiting to run the spectrometer, help them go through the process. If you are the last user, replace each salt plate in a separate plastic envelope within the plastic container. Place the plastic container back in the desiccator.

Throw away any used kim-wipes.

Take your spectrum back into the laboratory. Use your new knowledge about IR interpretation to answer the questions on your datasheet.

POST-EXPERIMENT ASSIGNMENT

Complete the datasheet and turn in before leaving class. Ensure your IR spectrum is stapled to the datasheet. Turn in the white notebook pages from your lab notebook. Staple multiple sheets together. Tear off rough edges.

Prepare for the IR portion of the next quiz. Remember that you may be asked questions on anything you should have learned from assigned readings, pre-experiment lecture, or doing the experiment.