## Characterizing Compounds by Infrared Spectroscopy (IR)

There are many different kinds of spectroscopy. All of them have to do with getting information from the interaction of matter and light. Infrared spectroscopy (IR) uses the infrared portion of the electromagnetic spectrum.

The bonds in a molecule are constantly vibrating in all directions. When the frequency of light hitting a molecule corresponds to the frequency of one of its vibrations, the light is absorbed. So when infrared light shines through a compound, specific frequencies are absorbed which correspond to the type of vibrations present in the molecule. This gives us information about what kinds of bonds are present, which we can put together to deduce the functional group.

An infrared spectrometer consists of a tunable laser that provides light of specific infrared frequencies, a sample chamber that lets light go through the compound, a detector that senses the light hitting it, and a computer that manipulates the data to generate a spectrum. When IR spectrometers were first invented, the laser would go through the IR frequency range a little at a time and the detector would determine how much of each frequency was absorbed. Now, however, the desired frequencies are all used at once, and the computer uses a complex mathematical process called a Fourier transform to sort the frequencies out. This speeds up the process considerably – from 30 minutes to a few seconds to obtain a spectrum. Therefore you will often see the abbreviation FTIR to stand for Fourier Transform Infrared Spectroscopy.



Since the IR spectrometer is by far the most expensive piece of equipment in the lab (on the order of \$20,000), please pay attention to the instructions and make sure that you are using it properly. Ask for help if you are unsure about what to do at any point.

The following table can help you identify IR bands.

О-Н	3400-3200 cm <sup>-1</sup> (smooth and broad)
N-H	3400-3200 cm <sup>-1</sup> (NH <sub>2</sub> 2 bands, NH 1 band)
C-H on CC	3300 cm <sup>-1</sup>
O-H on C=O	3400-2500 cm-1 (very broad)
C-H on C=C	3100-3000 cm <sup>-1</sup>
С-Н	3000-2850 cm <sup>-1</sup>
C-H on C=O	2900, 2700 cm <sup>-1</sup> (2 bands, rather small)
COOH dimer	$2700 \text{ cm}^{-1}$ (may be 2 bands)
$C=O \text{ on } CO_2$	3200 cm <sup>-1</sup>
CN	2260-2200 cm <sup>-1</sup> (sharp and long)
СС	2220-2100 cm <sup>-1</sup> (sharp, small to medium)
overtones, monosubstituted aromatic ring	2000-1660 cm <sup>-1</sup> (four small humps)
C=0	1800-1650 cm <sup>-1</sup>
C=C	$1680-1620 \text{ cm}^{-1}$ (smaller than C=O)
C=C aromatic	1600, 1500 cm <sup>-1</sup> (1 or 2 bands)
NH <sub>2</sub> bending	around 1600 cm <sup>-1</sup>
CH <sub>2</sub> bending	1400, 1380 cm-1
C-0	1300-1000 cm <sup>-1</sup>
C=C bending	980-675 cm <sup>-1</sup>
С-Х	850-500 cm <sup>-1</sup>

## Procedure:

- Turn on the computer and open the IR software (EZ Omnic) if this has not been done. If a spectrum is already displayed, click "clear" to get rid of it.
- Click on "collect sample" and hit return. (Do NOT hit "collect background" this will only collect a background, not collect one and subtract it from a sample.)
- The program is set to collect a background spectrum before each sample. Make sure the sample well is empty, then click "OK".
- When the background is done, use a plastic pipet to put 2-4 drops of your sample onto the spectrometer crystal enough to cover it well but not overflow. Click "OK" to collect the sample.
- After the sample has been collected, click "yes" to add it to the window. If you don't do this, the computer won't be able to process or print the spectrum.
- If you wish to do so, use the "T" tool to mark the peaks that you feel are important, then go back to the arrow cursor.
- If you observe peaks that are over 100% transmittance (other than CO<sub>2</sub>), this means that the last person to clean the IR didn't do a good job. If peaks are present in the background but are not in your sample, they will be subtracted out and come out as negative peaks! The only solution is to clean more thoroughly and start over.
- Click "Print" to print out a copy of the IR.
- Clean IR crystal by first unscrewing the gray ring, then removing the metal disk underneath. Rinse the disk with isopropanol over the waste container and wipe it with a small, clean piece of cotton. Then drip some isopropanol onto the crystal itself and wipe it gently with a piece of cotton (be absolutely certain you do not scratch the crystal), and repeat (with clean cotton). Replace the disk and ring carefully. Note that it is the solvent that is doing the cleaning, not the wiping action of the cotton. Use the solvent to dissolve the compound, then wipe it away with the cotton; don't rub the cotton around a lot or you're just smearing the compound around.