Use of the Midac FT-IR Spectrometer

How to ruin the expensive salt plates:

NaCl plates are used as the sample cell. (1) Water is bad. NaCl is water soluble so samples contaminated with water will ruin the plates. Always check the sample to be sure water is not present. If a sample is cloudy it is likely that the sample is contaminated with water. Water will dissolve/ruin the expensive salt plates. Dry the sample further with drying agent if necessary. Handle the plate by the edges - the water in your fingers can ruin the surface of the plate.

(2) NaCl plates are very brittle and easily scratched. Use a polyethylene pipet to transfer the sample onto the plate, or if it is necessary to use a glass pipet be very carefull to not scratch the plate. Dropping a plate will break it.

Prepare the Sample:

(always use clean plates - see below for cleaning – for the cyclohexene experiment, to save time, just wipe off the surface with a Kim Wipe – the volative sample will evaporate quickly enough)

For a liquid sample, place a drop or two onto a salt plate then place a second salt plate onto this, forming a thin layer of sample between the plates. If the sample is low-boiling as is cyclohexene it will evaporate readily so work quickly.

Solids samples are done differently, usually by making a mull or KBr pellet. These are described elsewhere.

Place the Sample into the Instrument:

Open the cover on the FT-IR instrument and place the salt plate/sample sandwich onto the sample holder (V-shaped plastic holder). Be sure to keep the plates pushed together. otherwise the sample will evaporate more quickly. Close the cover of the instrument.

To Collect the Spectrum.

With "Sample" selected, click on "START."

The spectrum will take several seconds to finish. (On the lower right you will see an indicator of the scanning process.)

To mark the frequency of a peak of interest,

Under "TOOLS" in the menu bar, move the cursor to "ANALYSIS" and select "MANUAL PEAK PICKING"

Move the cursor to the peak of interest and right click on the peak. The frequency will be written next to the peak.

Label any relevant peaks. (E.g., for cyclohexene relevant peaks would include the C=C stretch (1650), and the C-H stretch of C=C-H (>3000).

Print the Spectrum.

Cleaning salt plates:

In the fume hood, using the halogenated waste bottle with a plastic funnel inserted into the top, place a salt plate into the funnel and rinse it with a little dichloromethane (methylene chloride). Allow it to dry (this is quick) and remove it by handling the edges of the salt plate only. Dichloromethane is toxic. Keep it off of your hands and do not breath its vapors.

If using mulls, the mortar and pestle may be cleaned in the same way.