How to Prepare Samples for IR Spectroscopy

Reading: Pavia *A Microscale Approach to Organic Laboratory Techniqes*, 5th edition, Technique 25

Salt (NaCl) plates (for liquids and solids)

Rule 1: ***ONLY*** clean NaCl plates with dichloromethane (aka, methylene chloride, CH_2Cl_2) and cyclohexane (C_6H_{12}). ***NEVER*** use water (H_2O) to clean a salt plate as it will ruin the plate, permanently!

Rule 2: ***NEVER*** hold the shiny side of a plate between your fingers (to avoid fingerprints). ***ONLY*** hold it by the edges.

Rule 3: Never scratch a plate with an item such as a spatula or other sharp object (expensive!)

Rule 4: Do not drop the salt plates, as they are quite fragile and will break easily (expensive!).

1. For *liquid* compounds, apply 1 drops of liquid compound in the center of one plate then place a second plate on top, then press together using a soft tissue and rotate slightly to give a thin film of compound.

   For *solid* compounds, prepare a Nujol (mineral oil) mull. Add ~10-15 mg of solid sample, then mineral oil (3-4 drops) to a mortar and pestle and grind the mixture to a thick paste (like that of honey). Dab a small amount (without scratching the NaCl plates!) of the mull on a salt plate and cover with a second plate. (Note that the IR absorptions from the mineral oil will always be present in the resulting spectrum). Alternatively, the "dry film" method can be used (see Section 25.4 in Pavia).

2. Place the salt plates in the holder that is in the sample chamber of the IR spectrophotometer and obtain the spectrum (see IR instructions for use.)

3. Immediately after use, clean the NaCl plates with dichloromethane (aka, methylene chloride, CH_2Cl_2) and/or cyclohexane (C_6H_{12}) and return them to the oven or the dessicator (ask instructor which one). Hold them only with forceps when washing with CH_2Cl_2 then wipe each plate dry with a clean soft tissue (like a Kimwipe).

Thin Film Method using salt (NaCl) plates

1. Dissolve ~15 mg of sample in ~1-2 drops of dichloromethane (CH_2Cl_2, aka methylene chloride)

2. Place 1 drop of this solution of one salt plate (only), let the solution evaporate, then proceed as above starting with step 2. (Don't forget about Rules 1-4 above)

KBr pellet press (for solids only). See the specific instructions under "Making a Pellet with a KBr Minipress" in Pavia (Technique 25.5 A)

1. For *solid* compounds, grind ~1-2 mg of the unknown in the special mortar and pestle (both made of agate, expensive!) until it is shiny (the goal it grind the sample to a particle size of <2 μm, which takes at least 2 minutes). Then add ~80 mg of anhydrous KBr and mix quickly with the sample (since KBr will absorb moisture from the air, therefore always keep the lid of bottle of KBr tightly sealed since it quickly absorbs moisture).

2. Add ~half of the contents of the ground mixture to the press with one of the bolts screwed *halfway* in the press. Screw in the other bolt and finger tighten. Using the correct wrench and the vise, slowly tighten the bolts to approx 20 ft-lbs of torque (which is pretty tight and probably about as tight as your hands can tighten it with the provided wrenches). Be sure that the wrench is used correctly to avoid stripping the bolt head. Leave the sample under pressure for 30 seconds before carefully removing one of the bolts. A successfully formed pellet is almost translucent [not cloudy--if it's a little cloudy the pellet will still work although the resolution will be less (peaks will be broad)].

3. Obtain the IR spectrum by carefully placing the KBr press in the holder in the sample chamber of the IR. (see IR instructions for use)

4. Poke out the KBr pellet with the eraser of a pencil. Do not use a spatula or scratch the press or bolt, since it might damage the smooth surface. Clean the mortar and pestle with distilled water, then finally acetone (back and forth until all of the inorganic (KBr) and organic materials have been removed), and dry with a tissue inside and out and then return to the oven to dry. (Both acetone and water will adversely affect the quality of your IR spectrum, so the KBr press must be completely dry and solvent-free before use.)

Using the QuickPress® KBr pellet maker

1. Perform step 1. as above for the MiniPress.

2. Add ~1/3 of the sample the special die (see instructor for directions).

3. Place die in the Quickpress and close the handle until it "clicks" closed.

4. Wait ~15 seconds, then open QuickPress and carfully remove the die.

5. Remove both ends of the die and place the central part of the die in the Spectrometer.

6. Take spectrum as described below.

7. Clean the die by using the longer end of the die to poke out pellet, then clean with a dry Kimwipe.

FT IR Spectrophotomer. Instructions for Use.
Obtaining the Spectrum and Printing

1. Place your sample in the appropriate holder in the sample chamber (the NaCl plates and the KBr press use the same holder). Make sure the sample is squarely in-line with the source (a red laser beam should be visible through the sample).

2. Press the **SURVEY** softkey (the button at the top of the keypad that corresponds to the button shown on the screen). This will take one scan and place it in the B register.

   (If more scans are desired, then press the **X** button followed by the **SCAN** button. Press the number of scans desired on the softkeys shown on the screen. Usually, no more than 4 scans are required to obtain an adequate spectrum and is usually the number of background scans stored in memory. See below for more information about the background.)

3. Make sure that the scale (4000-650 cm^{-1}) and the range (100-0%T) are correct. (Note that many of the following commands require the use of the **SHIFT** key to access the functions written in small type at the top of the buttons, just like on a computer keypad) To do this press **RESCALE** or **RERANGE**, whichever is appropriate to correct. Usually only the **RERANGE** command needs to be used. To get the spectrum to fill the range (%T) of the screen, press **AUTOEX**.

4. Press the gray **PLOT** button to print the spectrum on the printer.

5. To obtain a list of peaks, press the green key for **PEAKTABLE** key (another **SHIFT** function, above the X button). Place the paper with the spectrum that you just printed face up in the paper feeder tray, then press the **PRINT** softkey (the one at the top of the keypad by the screen, ***not*** the green colored key next to the **PLOT** button).

6. Don't forget to remove your sample and clean up the NaCl plates or KBr mini press as described in the "How to prepare IR Samples" section.

Background Scans

1. Background scans are usually performed at the start of each laboratory session or at the beginning of each day that the instrument will be used.

2. To take a background scan press the green BACKG (background) button and then the green SCAN key. Choose the number of scans using the softkeys at the top of the keypad. The standard number is 4 scans. Once the background scans are complete the screen should automatically return to the X register.

3. If the CO_2 air peaks shows either a positive (or negative peak) at 2200 cm^{-1} then ask the instructor to take more background scans

Troubleshooting

1. If the spectrum appears to have very broad peaks for everything and all of the absorptions are very intense, then you probably have too much compound in your sample. If you are analyzing a liquid, then simply wipe off some of the compound using a soft tissue and rescan the sample. If you made a KBr pellet, it will have to be remade and then re-scanned. If you made a thin film on a NaCl plate, then rinse the plate with CH_2Cl_2 and start over and add less sample to the plate.

2. If the spectrum appears to be off-scale, then make sure the transmittance scale reads from 0% (on the bottom) to 100% on the top. If not press the SHIFT, then **RESCALE** , as mentioned above

3. If you spectrum appears to "lean," typically the left side is lower than the right, which is common with KBr pellets, then press the FLAT key in the middle of the keypad. Use the softkeys on top of the keypad to "flatten" the spectrum with the MORE or LESS keys. If the adjustment goes too far then "flatten" in the other direction and press the SLOWER key and then try the MORE or LESS keys. Once you are finished with this adjustment, then press the **EXECUTE** key and proceed with the printing as described above.