

Using an ATR Accessory to Obtain Spectra of Powdered Samples in an FTIR

Introduction

A Fourier-transfer infrared spectrometer (FTIR) measures the absorbance of infrared (IR) laser light by chemical bonds in a sample. The attenuated total reflectance (ATR) accessory shines an IR laser at the angle of total reflectance into a germanium crystal. At the point of the reflectance, some of the light escapes the crystal and reflects off a sample sitting on top of the crystal. The computer attached to the FTIR performs a Fourier transform on the energy reflected back through the crystal, with and without a sample, displaying the characteristic spectrum for the sample. Because water vapor and carbon dioxide have IR-active bonds that would show up as part of the sample absorbance, the system is purged with dry air. The sample must be solid with some crystal structure in order for useful information to be gained from the analysis of its spectrum. Fortunately, though the science responsible for the functioning of the FTIR is complicated, the operation of the instrument is relatively simple – do not be dissuaded by the many steps! If you do run into trouble, nonetheless, contact Patty Wurm, the instrument manager, at 314-935-4832 or pattywurm@seas.wustl.edu.



Figure 1: Computer, air flowmeter, log book, and FTIR as found in Urbauer 13.

Preparation

The three prerequisites for using the FTIR are training from Patty Wurm, an account with the EECE, and access to the Environmental Engineering Shared Instruments Lab, Brauer 014, which requires a numerical code for entry. The training will familiarize you with the instrument and important precautions. To log into the computer collecting the spectra, you must have an EECE account. Access to Urbauer 13 is granted once payment arrangements are made for using the instrument.

You will need the following items to collect the spectrum of your sample:

- sample (~0.5 g)
- mortar and pestle
- timer
- wax paper
- brown Teflon-coated spatula
- small plastic vial with attached cap
- deionized (DI) water squirt bottle
- Kimwipes™
- PC, connected to FTIR
- FTIR Log Book
- Thermo Nicolet Nexus® 470 spectrometer (FTIR)
- Harrick Praying Mantis™ accessory
- Thermo Nicolet Smart Performer® accessory
- Kleenex Viva™ paper towels

The overall procedure is outlined in the following steps:

1. Prepare the sample.
2. Remove the Praying Mantis accessory.
3. Install the Smart Performer accessory.
4. Collect the background reading.
5. Place the sample in the Smart Performer accessory.
6. Collect the sample spectrum.
7. Clean up.

As a reminder, the FTIR is an expensive piece of equipment. Careless handling of or dropping the accessories can lead to damage on the order of a few thousand dollars, so please exercise caution. The easiest piece to damage is the germanium crystal, which can be scratched by merely rubbing a paper towel on it. However, if you follow the steps outlined here and reinforced in your training, you will have no problem obtaining spectra without damaging the instrument.

1.0 Preparing the Sample

- 1.a. Place your sample in the mortar.
- 1.b. Set the timer to 2 minutes and start timing.
- 1.c. Grind the sample with the pestle until the time is up.
- 1.d. Fold a piece of wax paper in half, open it up, and place it on the lab bench.
- 1.e. Empty the contents of the mortar onto the wax paper. If necessary, use the brown spatula to scrape the sample off the mortar's surface.
- 1.f. Fold the wax paper at the crease, and dump the sample into a small plastic vial.
- 1.g. Rinse the mortar and pestle with DI water, and dry them with Kimwipes.



Figure 2: Mortar and pestle with sample, timer, and plastic vial.

- 1.h. Cap the vial, and carry it, along with a piece of wax paper and the brown spatula, to the bench near the FTIR in Urbauer 13, the Shared Instruments Lab.
- 1.i. Log into the computer using your CEC account.
- 1.j. Sign into the Log Book next to the instrument.

2.0 Removing the Praying Mantis

- 2.a. Open the lid of the FTIR as shown in Figure 3 below.

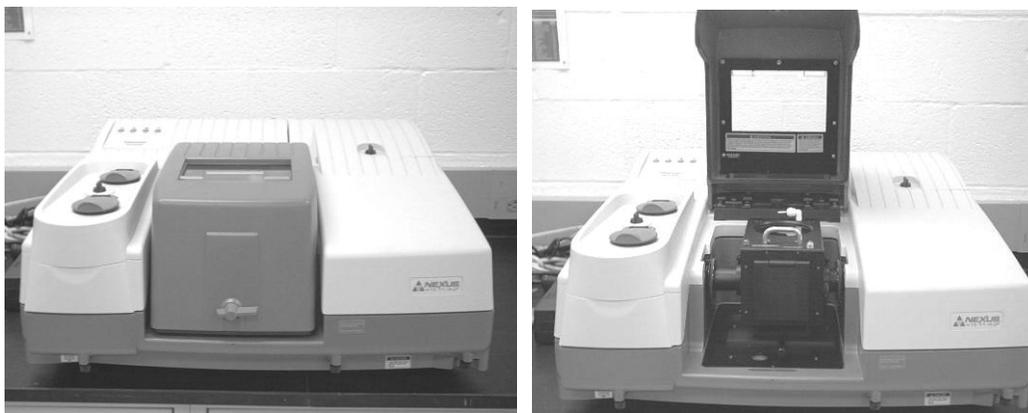


Figure 3: FTIR with the lid closed and open. The knob is at the base of the open lid.

- 2.b. Remove the lid via the knob behind the accessory compartment.
- 2.c. Loosen the thumbscrews holding the purge sleeves of the Praying Mantis in place, as seen in Figure 4 below.

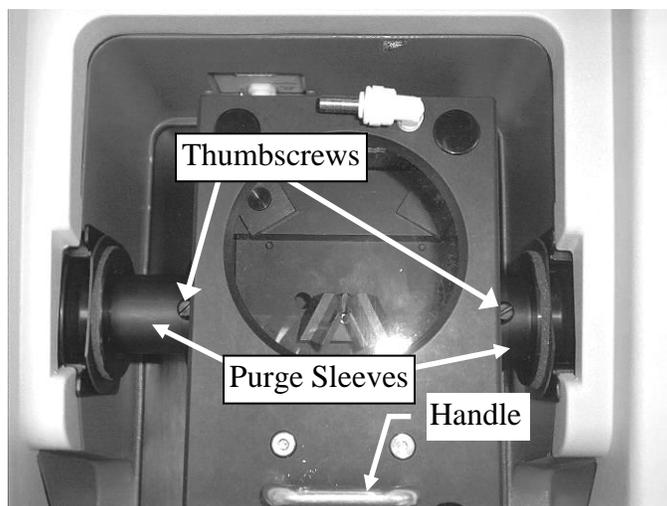


Figure 4: View of the Praying Mantis from above.

- 2.d. Push the purge sleeves into the body of the accessory.

NOTE: The base plate of the accessory is held in place by a magnet hidden from view. The initial force required to lift the accessory is therefore more than the weight of the accessory.

- 2.e. Lift the accessory out of the compartment, holding the handle on top and the body of the accessory at the back.

NOTE: Carry all accessories carefully. Do NOT drop them!

- 2.f. Set the accessory on the bench to the right of the instrument.
- 2.g. Remove the ears of the accessory from both holes, and place them in the bottom of the compartment, as shown in Figure 5.

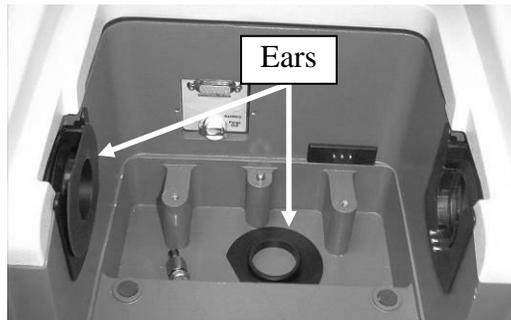


Figure 5: The accessory compartment with one ear inside and one ear not yet removed.

3.0 Installing the Smart Performer

- 3.a. As seen in Figure 6, retrieve the Smart Performer from the cabinet below the instrument. Place it on the bench opposite the FTIR.

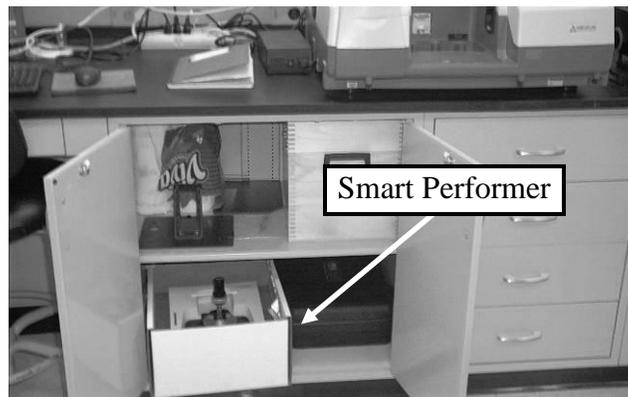


Figure 6: The Smart Performer is located in a white cardboard box in the cabinet below the FTIR.

- 3.b. Holding it by the handle and the front, remove the Smart Performer from the foam-filled cardboard box.

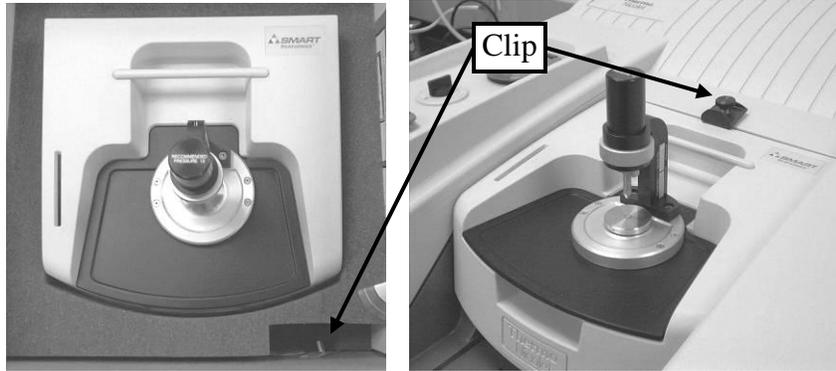


Figure 7: The Smart Performer in the foam box and in the instrument. The lock-down clip is found in the lower right compartment of the foam box and is installed as shown above.

- 3.c. Align the rigid purge sleeves of the Smart Performer with the holes you just removed the ears from, and insert the accessory into the instrument. When installed correctly, the accessory fits snugly in the instrument.
- 3.d. Screw in the lock-down clip, seen in Figure 7 above, into the hole where the lid's knob was fastened.

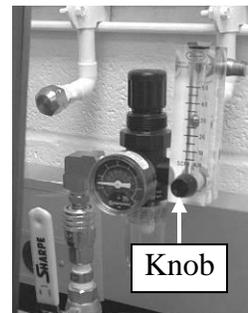


Figure 8: Air flowmeter.

4.0 Collecting the Background Reading

- 4.a. Check the flow rate of dry air into the system at the air flowmeter behind the computer monitor. Adjust the knob until the midsection of the ball hovers at 30 standard ft³/hr (scfh).
- 4.b. Open OMNIC on the computer by double-clicking the OMNIC desktop icon, seen at right.
- 4.c. Click the menu bar icon reading "Expt Set" to enter the *Experiment Setup* window.

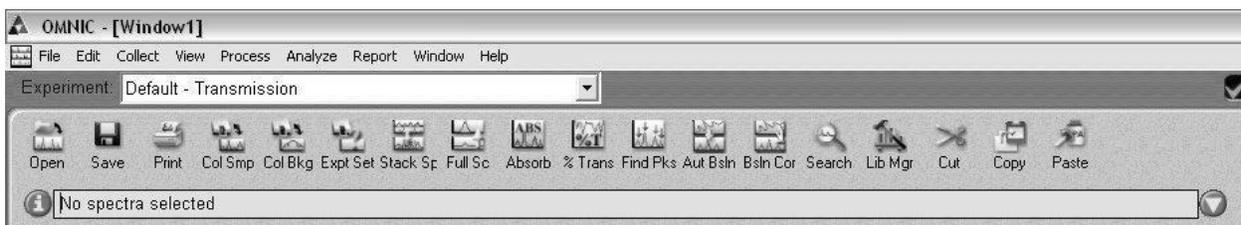


Figure 9: OMNIC Menu Bar.

- 4.d. Under the *Collect* tab, set the number of scans to 64 and the final format to absorbance. Check *Preview data collection* and click the radio button for *Collect background after 100 minutes*. Finally, enter a title for the experiment. Check that your window matches that shown in Figure 10.

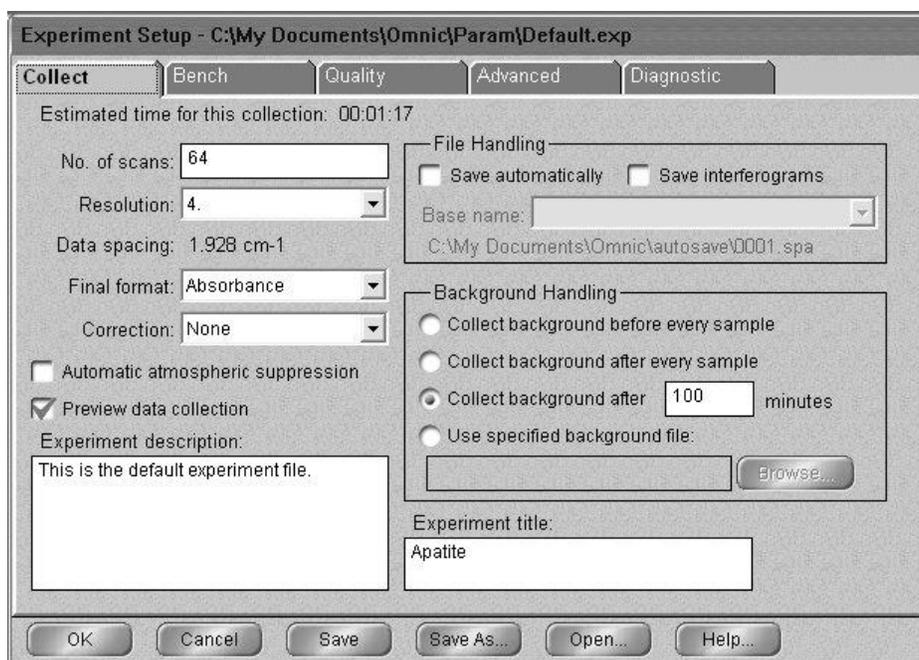


Figure 10: The Collect tab of the Experiment Setup window.

- 4.e. Under the *Bench* tab, set the accessory to *ATR*. In the Log Book, record the *Max*, *Min*, and *Loc* values found at the top of the window, seen below in Figure 11.

NOTE: If any of the three parameters you record in the Log Book is more than 10% off from other values recently recorded there, please notify Lawrence that instrument maintenance is needed.

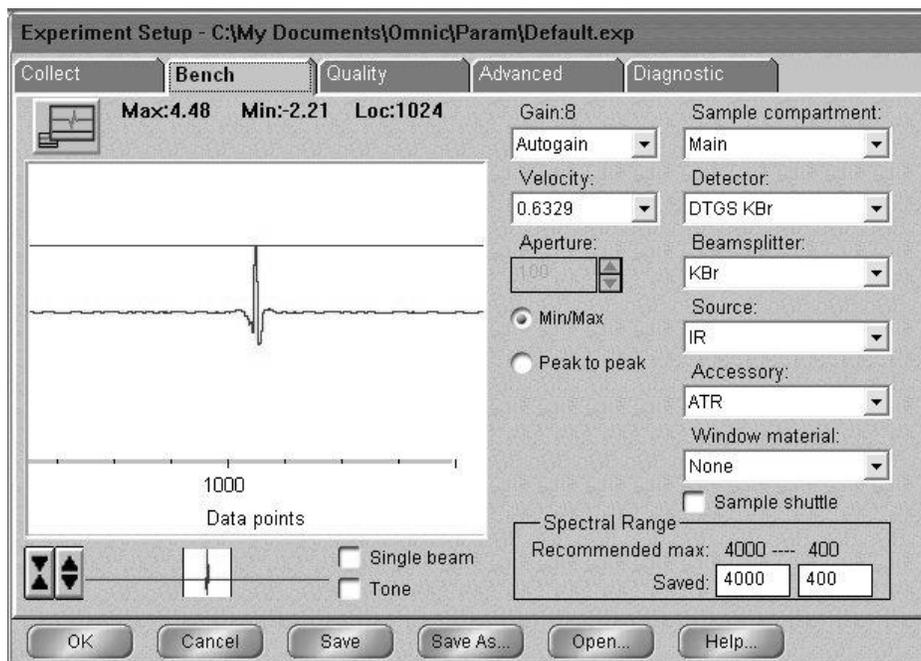


Figure 11: The Bench tab of the Experiment Setup window.

- 4.f. Click *OK*.
- 4.g. Click the menu bar icon reading “Col Bkg” to begin collecting the background.
- 4.h. Click *OK* to indicate you are ready to collect the background.
- 4.i. When the background looks stable – no major changes in peaks – click *Start Collection* in the top right corner of the window. An unstable background indicates that the instrument is not fully purged, so you should wait 1 minute for purging to finish.
- 4.j. Click *No* when prompted about adding the background to Window 1, unless you are troubleshooting the peaks of a spectrum already on hand.

5.0 Placing the Sample in the Smart Performer Accessory

- 5.a. Make sure that the pressure applicator’s tip is at its maximum distance from the crystal (approximately 1” above it).

NOTE: The crystal can be removed only by lifting straight up, because it is held in place by two pegs. Make sure the crystal does not hit the pressure applicator as you remove it.

- 5.b. Lifting straight up, remove the crystal from the accessory and place it on the piece of wax paper.

NOTE: The Smart Performer is open beneath the crystal’s base. Take care not to drop sample or other items in the accessory while the base is absent.

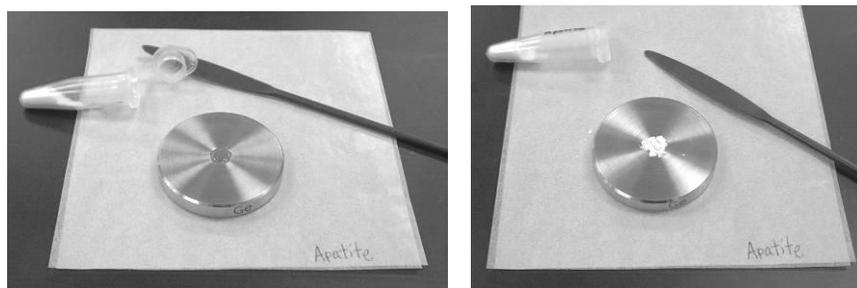


Figure 12: Sample placement on the crystal.

- 5.c. Use the brown spatula to add sample from the vial to the crystal’s surface, covering the crystal alone in a thin layer, as seen in Figure 12 above. The laser reaches only two microns of the sample layer on top of the crystal, so there is no need for a thick layer of sample or for sample to cover the rest of the crystal’s base.
- 5.d. Replace the crystal in the Smart Performer – match the two pegs of the instrument (seen in Figure 13 below) to the two holes in the crystal’s base by ensuring the “Ge” on the side of the base is centered in the front.

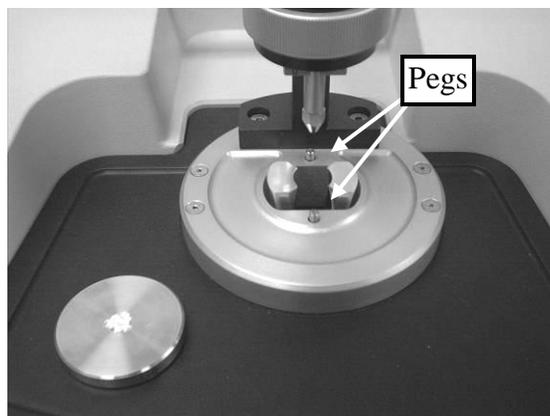


Figure 13: Replacing the crystal on the 2 pegs of the Smart Performer.

6.0 Collecting the Sample Spectrum

- 6.a. Increase the pressure applied to the sample by turning the thumbscrew clockwise (as seen from above). Set the pressure to 4 (psi) initially (see Figure 14 below).

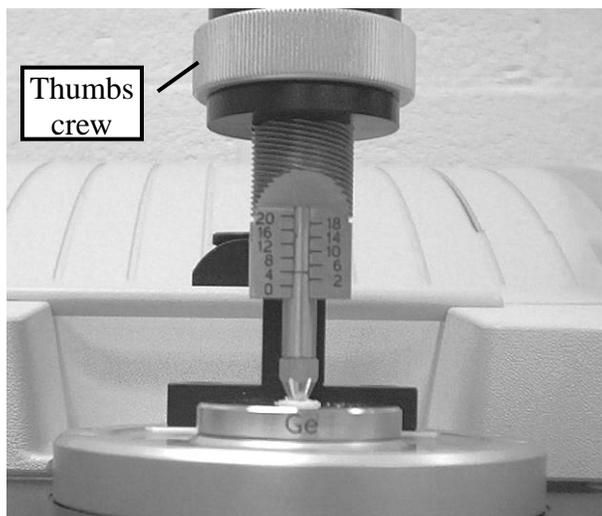


Figure 14: Application of pressure. As shown, the screw is turned to the left to increase pressure. Note that the "Ge" on the crystal's base is centered at the front.

- 6.b. Click the menu bar icon reading "Col Smp" to begin collecting the sample.
- 6.c. At the title prompt (see Figure 15 below), enter the sample's chemical name or formula in front of the default title (the current date and time). This way, identical samples run on different days will be distinguishable when saved.

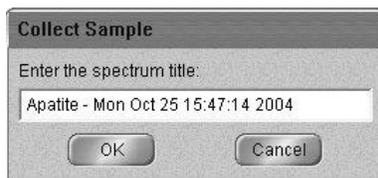


Figure 15: Spectrum Title window.

NOTE: After replacing the crystal in the accessory, you must allow one minute of purging time so that carbon dioxide and water vapor in the room air do not interfere with the sample's absorbance recorded in the instrument. Be sure that one minute has passed before continuing to the next step.

6.d. Click *OK* when prompted to prepare to collect the sample.

NOTE: For materials harder than the germanium crystal, like sand, application of pressure beyond 4 psi will cause the coarse particles to scratch the crystal. Hard material must be thoroughly ground in the mortar and pestle to use pressure greater than 4 psi. Relative hardness can be determined from the chart posted on the wall above the FTIR.

6.e. Watching the spectrum previewed on the computer screen, increase the applied pressure until the noise is minimal. For most samples this pressure will be 12 psi, the maximum pressure recommended.

6.f. Press *Start Collection* in the top right corner of the screen.

6.g. Unless the spectrum is completely unacceptable based on prior knowledge of your material's composition, click *Yes* when prompted *Add to Window1?* Unexpected peaks in the range of O-H or C-O bonds indicate the presence of H₂O or CO₂ (incomplete purging), and the background and sample should be re-run with ample time allowed for the system to purge.

6.h. Save the spectrum by clicking the *Save* icon. As seen in Figure 16, the *Save As* window will open. Note that if multiple spectra are open in a window, you will need to click on the desired spectrum before clicking *Save*.

6.i. Click *Set Filename to Title*, and save the file to either the "Spectra" file on the C drive of the FTIR computer or to a removable or network drive if you

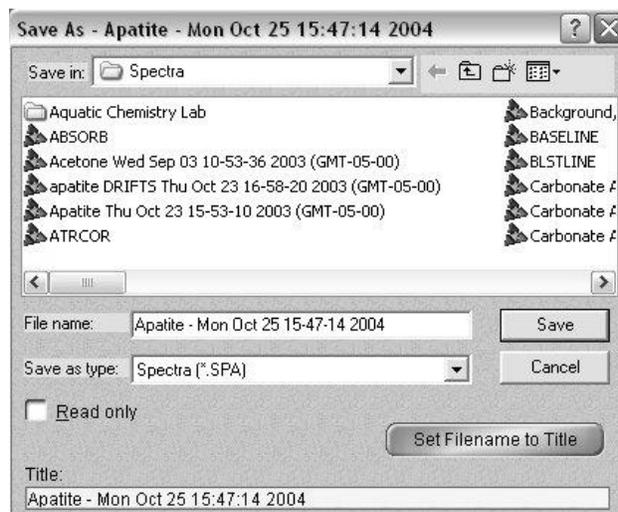


Figure 16: *Save As* window.

are planning on analyzing the spectrum at a different computer. The file can be saved as a spectrum file (*.spa) or a jpeg image (*.jpg), depending on your intended use of the spectrum.

7.0 Cleaning Up

- 7.a. Close OMNIC, and log off the computer.
- 7.b. Turn the thumbscrew counterclockwise to lift the pressure applicator to its maximum height.
- 7.c. Rip off a small piece of paper towel (about one eighth of a sheet). Twist it twice at its midsection, and wet it at the twist with DI water.

NOTE: *Inverting the next two steps risks having some sample that was stuck on the applicator tip fall into the accessory. Make sure you follow these steps in order.*

- 7.d. Rub the wetted section on the tip of the pressure applicator to remove any sample stuck to it, as shown in Figure 17.



Figure 17: Removing sample from the tip of the pressure applicator.

NOTE: *Make sure the crystal does not hit the pressure applicator as you remove it.*

- 7.e. Remove the crystal, and tap its base gently on the wax paper to dislodge the powder sample, shown below in Figure 18.

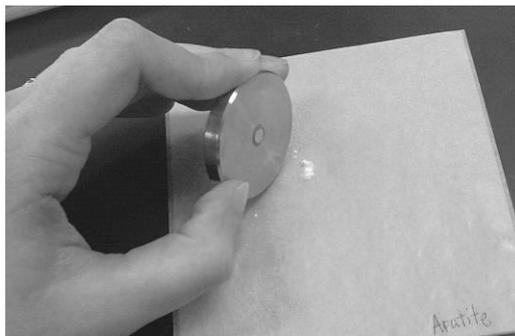


Figure 18: Dislodging sample from the crystal's surface.

NOTE: *Do not rub the paper towel on the crystal, as that will scratch it. Only dab at the crystal with the paper towel.*

- 7.f. Use another small piece of wetted paper towel to gently wipe clean the crystal's base and dab at the crystal.
- 7.g. Replace the crystal in the Smart Performer.
- 7.h. Use the wax paper to return the sample to the plastic vial, and then throw away the wax paper and the used paper towel.

NOTE: Carry the accessories carefully. Do NOT drop them!

- 7.i. Remove the lock-down clip and the Smart Performer from the instrument, and place them both in the foam of the white cardboard box. Replace the box in the cabinet.
- 7.j. Replace the ears of the Praying Mantis over the holes in the accessory compartment.
- 7.k. Replace the Praying Mantis in the compartment, aligning the two pegs in the bottom of the compartment with the two holes in the base plate of the accessory.
- 7.l. Extend the purge sleeves to the ears, and screw them in place.

NOTE: There is a latch at the bottom of the front of the lid, but it will not lock the cover in place with the Praying Mantis inside – do not try to force the latch closed over the Praying Mantis.

- 7.m. Screw the lid's knob back on the instrument, and close the lid.
- 7.n. Record the time out in the Log Book.

Conclusion

You now have an IR spectrum for your sample. This spectrum is unique to the chemical composition of the sample you analyzed, and its peaks can give you information about the chemical bonds comprising the sample. The peak locations for various compounds are often tabulated in books or given in research papers. You can further analyze the peak locations using a spectral analysis program such as GRAMS-AI, or you can simply publish your spectrum if you saved it as a jpeg image. If you would like to know more theory behind the operation of the FTIR, reference the three books listed below.

References:

- Ege, SN (1999). Organic Chemistry: Structure and Reactivity. D.C. Heath and Co. pp. 376-382.*
Gremlich, HG, and Gunzler, H (2002). IR Spectroscopy: An Introduction. Wiley. pp.24-28.
Smith, BC (1995). Fundamentals of Fourier Transform Infrared Spectroscopy. CRC. pp. 1-45.