

Operation of the Perkin-Elmer 1600 Fourier-Transform Infrared Spectrophotometer with Spectrum 2.0 Software

A. Instrument Setup

1. The Perkin-Elmer 1600 Fourier-transform infrared (FT-IR) spectrophotometer and its computer are left on continuously.
2. Use the **intensity knob** behind the spectrophotometer's monitor to increase the screen brightness.
3. Press the blue **enter** button on the spectrophotometer control pad to begin a data acquisition session.
4. Go to the computer and double-click on the **Spectrum 2.0** icon to start the program.

B. Background Acquisition

1. Open the door to the spectrophotometer to verify that **no sample** is in the path of the laser beam. Then close the **door**.
2. Select the **Instrument > Scan Background** menu item to bring up the Scan Background window.
 - a. Spectrum Details
 - i. Fileame: **bkg.sp**
 - ii. **Single beam** selected.
 - b. Scan Parameters
 - i. Range: **4000–400 cm⁻¹**
 - ii. Number of Scans: **4**
 - iii. Resolution: **4.0 cm⁻¹**
 - iv. Interval: **1.0 cm⁻¹**
 - v. Apodization: **Weak**
 - c. Click on the **OK** button to initiate the background acquisition.
3. The background spectrum will appear after the acquisition is complete.
4. The Instrument-Auto Save window will appear. Click on the **Overwrite** button to save the newly-acquired background file.

C. Sample Acquisition

1. Obtain a pair of clean, sodium chloride salt plates from a desiccator. Be certain that the salt plates are clean. If they are not, clean and polish them with methanol and a paper towel.
2. Place your sample between two sodium chloride salt plates.
 - a. For a liquid sample, use a Pasteur pipet to put one or two drops of the sample on one of the plates. Then gently place the second plate on top of the first plate, so that the sample forms a thin film between the two plates.
 - b. For a solid sample, use a small mortar and pestle, approximately 5 mg of your sample and the smallest drop possible of nujol. Use the mortar and pestle to grind your sample with the nujol to make a mull. Then use a spatula to place the mull on one of the salt plates. Gently place the second plate on top of the first plate, so that the sample forms a thin film between the two plates. Alternatively, prepare a KBr pellet.
3. Open the door to the spectrophotometer. Place the **prepared sample** onto the sample holder. The laser beam should pass directly through the sample. Then close the **door**.
4. Select the **Instrument > Scan Sample** menu item to bring up the Scan Sample window.
 - a. Spectrum Details
 - i. Filename: Choose a **filename.sp**. Use your initials, and book and page identifiers. e.g., aa127a.sp for the first sample in Anthony Aldick's notebook 1, page 27).
 - ii. **Ratio** selected.
 - b. Scan Parameters
 - i. Region: **X**
 - ii. Range: **4000–400 cm⁻¹**
 - iii. Number of Scans: **4**
 - iv. Resolution: **4.0 cm⁻¹**
 - v. Interval: **1.0 cm⁻¹**
 - vi. Apodization: **Weak**
 - vii. Units: **%T**
 - c. Click on the **OK** button to initiate the data acquisition for your sample.
5. The instrument will collect four scans, perform a Fourier transform, and subtract the background spectrum. Both the spectra of your sample and the background will appear. Click on the **bkg.sp** label in the lower left corner to select the background scan. The label will be underlined once it is selected. Press the **delete** key on the keyboard to remove the background spectrum from the window.

D. Data Analysis

1. Click on the **filename.sp** label in the lower left corner to select your data file. The label will be underlined once it is selected.
2. Click on the **AutoX** and **AutoY** toolbar buttons to fill the entire data window with your spectrum.
3. Select the **Process > Peak Table** menu item to open the Peak Table window.
 - a. Threshold: Enter a value **between 5 and 15** %T.
 - b. Click on the **OK** button.
 - c. A numerical peak table window will appear. **Close it.**
4. **Maximize** the spectrum window.
5. Click on the **Peaks** toolbar button to label the peaks.
6. Click on the **Text** toolbar button to add a title to the printed spectrum. Enter your **title** and click on the **OK** button. **Position the box** where you want the title to appear on the spectrum.
7. Click on the **Print** toolbar button to print your spectrum.
8. After the spectrum has been printed, use the **File > Exit** menu item to close the Spectrum 2.0 program.

E. Instrument Standby

1. Use the **intensity knob** behind the spectrophotometer's monitor to decrease the screen brightness.
2. Open the door to the spectrophotometer, remove the **salt plates** and close the door. Clean the salt plates with a Kimwipe and methanol. Then return the salt plates to the desiccator.
3. Remember to take your **spectrum** and the rest of your **sample**.