

Collecting and Printing an FT-IR Spectrum

This guide tells you how to operate the ThermoNicolet IR100 spectrometer equipped with a Thunderdome attenuated total reflectance (ATR) sample accessory. An example of a typical IR spectrum is shown on the next page. The printout features:

- a “full” %Transmittance spectrum showing all peaks between 4000 and 850 cm^{-1}
- peak positions for the larger signals (these are also listed in a separate report)
- user-provided spectrum title

Typical operation involves the following steps:

1. Setup spectrometer configuration (p. 4)
 2. Collect background spectrum (p. 4)
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3. Apply sample to ATR accessory (p. 5)
 4. Collect sample spectrum (p. 5)
 5. Find major peaks (p. 6)
 6. Print spectrum (p. 7)
 7. Clear sample spectrum and report (p. 7)
 8. Clean ATR accessory (p. 8)

Most users should start at step 3. [Steps 1-2 are normally performed by the instructor at the start of the lab period.]

Spectrum goes here

Computer Mouse

The IR100 mouse has three buttons. Use the *LEFT* mouse button for all operations

Computer Menus

The IR100 computer is operated from pop-up menus. The menus are opened by clicking on the buttons located at the *bottom* of the display screen.

The menus are labeled: **File**, **Setup**, **Collect**, **View**, **Process**, and **Analyze**.

Computer Keyboard

Some IR100 commands require you to enter information from a computer “keyboard” that is displayed on the computer screen. Enter information by clicking on the “keys” and then click on the large green bent “Enter” arrow. The “Enter” arrow is not labeled, but it is on the right edge of the keyboard and is unmistakable.

The Thunderdome Attenuated Total Reflectance (ATR) Accessory

The Thunderdome attenuated total reflectance (ATR) accessory can be used to measure IR spectra for liquid and solid samples. Both types of samples can be prepared and cleaned up quickly, enabling a large number of students to use the IR100.

Although the ATR accessory is easy to use, proper care must be taken in its use. The heart of the accessory is an *expensive*, highly polished germanium crystal. The spectrometer directs IR radiation at the bottom of the crystal, and a portion of this radiation enters any sample lying on top of the crystal. Therefore, you will need to place your sample directly on this crystal and you will need to clean the crystal afterwards. It is possible to damage the crystal at either stage, so please read and obey all instructions concerning the ATR accessory. In particular, when you are finished with using the accessory, you must:

clean the accessory with an appropriate material, rotate the Pressure Tower counterclockwise to its maximum elevation, and lock the Tower in the tilt back position.

Failing to follow these instructions runs several risks. Compounds left on the crystal can etch the crystal surface (strong acids especially) or attack the adhesives that hold the crystal in place (acetone and chlorinated solvents). Leaving the Pressure Tower in contact with the crystal for an extended time period can damage the crystal surface or even crack the crystal. Failing to raise the Pressure Tower may mean that the next user inadvertently smashes the pressure tip into the crystal when the Tower is rotated forward.

Preparing the Spectrometer

Your instructor will set up the IR100 at the beginning of each lab period. However, if this hasn't been done, or if you need a new background spectrum, follow these steps:

Clean the Thunderdome ATR accessory

1. See p. X

Configure the spectrometer

2. Click on **Setup**, then click on **Open Configuration...**
3. Select **Collect4cm-1_ATR** and click **OK**

OK button is located in upper right hand portion of message window

This configuration differs from the default configuration in three respects. First, the Gain is set to 4 (default Gain = 1) to compensate for the smaller signals obtained from the ATR accessory. Second, the spectral windows is set to 4000-850 cm^{-1} (default window = 4000-400 cm^{-1}) because the germanium crystal in the ATR accessory blocks lower energy radiation. Third, the number of scans for the sample and background spectra have been set to 32 (default = 16) to reduce noise.

Show reports, and clear all displayed spectra and reports

4. Click on **View**, then click on **Clear All Spectra**

Message window appears warning you that some spectra have not been saved

5. Click on **No to all**
6. Click on **View**, then click on **Show Report**
7. Click on **View**, then click on **Clear Report**

Collect a new background spectrum

IR100 spectra are produced by calculating the *ratio* of two spectra: a sample spectrum and a background (no sample) spectrum. When this procedure works properly, it eliminates all signals due to the electrical properties of the IR100 spectrometer components, and signals produced by background gases, such as water and carbon dioxide.

The "ratio" procedure works best when the sample and background spectra are collected under identical conditions. Therefore, it is a good idea to collect a new background spectrum every few hours or so. A new background spectrum should also be collected whenever the spectrometer is reconfigured.

8. Click on **Collect**, then click on **Background**

window appears prompting your for a spectrum title

9. Enter a short title on the "keyboard" and click on the "Enter" key

Collect window appears showing you the number of completed scans

IR100 is ready for use when the Collect window closes

Apply Sample to ATR Accessory

The ATR accessory is easily damaged. Please read the instructions at the bottom of p. 3 regarding the ATR accessory before applying your sample.

Liquid samples

1. Apply liquid to the center of the germanium crystal with a disposable pipet (**avoid contact between the pipet and crystal**). Use just 1-2 drops and allow the liquid to spread out to make a thin film. It is ok if the liquid spreads across the entire crystal
2. Leave the Pressure Tower tilted back

Solid samples

1. Place solids on the center of the germanium crystal with a microspatula (**avoid contact between the spatula and crystal**)
2. Carefully bring the Pressure Tower upright by pulling out the silver release knob and tilting the Tower forward (**do not let the tip fall on the crystal**)
3. Use your spatula to position your sample underneath the Tower's pressure tip
4. Rotate the knob on top of the Tower *clockwise* so that the pressure tip presses your sample onto the germanium crystal. Stop rotating the knob when you hear a "click". These "clicks" are created by a slip-clutch safety mechanism that prevents the tip from applying too much pressure to the crystal

Collect Sample Spectrum

1. Click on **Collect**, then click on **Sample**

window appears prompting you for a spectrum title

2. Type your title on the "keyboard" and click on the "Enter" key

Collect window appears showing you the number of completed scans

"ratio" sample spectrum is displayed in red

Adjust Display

Ideally, your “ratio” sample spectrum will look like the one on p. 2, that is, it will contain a “baseline” that runs across the top of the spectrum (100% Transmittance) and one or more narrow IR absorption “peaks” that drop downward.

In some cases, your spectrum will contain one or more signals that rise upward from the baseline. These signals are usually associated with water and CO₂ absorptions that have not been cancelled properly (see background spectrum). **If the rising signals are small, ignore them.** However, if the signals are large compared to your sample’s absorption peaks, adjust the display so that the rising signals are hidden.

1. Move the cursor into the lower left corner of the *sample* spectrum
2. Click-drag upwards and to the right. *Gray box appears*
3. Click inside the gray box *Display is adjusted so that only the region inside the box is seen*

These steps can be repeated. If you lose part of your spectrum, return to the full spectrum by following step 4.

4. Click on **View**, then click on **Full Display** *Full sample spectrum is seen*

Find Major Peaks

1. Click on **Analyze**, then click on **Find Peaks...**
New window appears with gray “threshold” line drawn horizontally across the spectrum
Peaks extending below the “threshold” are identified in two ways:
 - 1) they are listed in the table of peaks (the “Report”)*
 - 2) their positions are printed out on the spectrum:*
2. If it is necessary to change the number of labeled peaks, adjust the “threshold” lines height and angle by click-dragging the ▲ symbols located on the left and right sides of the spectrum
3. Click on **OK**

Note: Peak positions are listed to the closest 0.01 cm⁻¹. The actual resolution of the IR100 in its current configuration is much poorer than this (typically ± 4 cm⁻¹). Round off your peak positions to the closest multiple of 5 cm⁻¹ when you write your lab report.

Print Spectrum

1. Click **File**, then click on **Print...** *Printer dialog box appears*
 2. If necessary, select **Landscape**
 3. Click **OK** *Printer dialog box disappears*
- Your spectrum is printed on two pages: a spectrum + a report*

Clear Sample Spectrum and Report

1. Click on **View**, then click on **Clear Spectrum**
Message window appears warning you that some spectra have not been saved
2. Click on **No** *sample spectrum disappears, background spectrum appears red*
3. Click on **View**, then click on **Clear Report**

If there are multiple sample spectra, repeat steps 1-2 until display is empty.

Do not use 'Clear All Spectra' since this will clear the background spectrum.

Clean ATR Accessory

Solid samples require a few extra steps (steps 1-3) since it is necessary to retract the Pressure Tower before cleaning. If your sample is a liquid, start at step 4.

1. Rotate the knob on top of the Pressure Tower *counterclockwise* (NO clicks) as far as you can. This raises the pressure tip to its maximum elevation
2. Pull out the silver release knob and move the Pressure Tower back
3. Carefully release the silver knob to lock the Tower in place
4. Moisten an undyed, unscented cotton ball (or soft tissue) with alcohol

Never squirt cleaning solvents directly on the ATR accessory or the IR100

5. Gently wipe the pressure tip and crystal surface to remove your compound (repeat steps 4 and 5 as needed).

Place used cleaning materials, pipets, and unwanted printouts in the appropriate containers

>>> More Warnings <<<

No acetone, no chlorinated solvents. Alcohol and water are the best cleaning solvents. Acetone can be used in a pinch, but repeated use is not recommended because acetone will slowly dissolve the bonding adhesive used in the ATR accessory. Never use chlorinated solvents to clean the ATR accessory as they too will dissolve the adhesive.

No Kimwipes. Kimwipes are abrasive and can scratch the surface of the germanium crystal.

Quick Guide to IR100 Operation

Make sure ATR & spectrometer are ready for use

ATR should be clean and Pressure Tower should be tilted back.

IR100 display should be empty (the display should contain two regions, one for a spectrum and one for a report, but both regions should be empty)

Apply sample

Put 1-2 drops of liquid on germanium crystal. Try not to touch crystal with pipet.

Collect and print spectrum

Collect: Sample

Analyze: Find Peaks...

File: Print...

Clear sample spectrum and report for next user

View: Clear Spectrum

View: Clear Report

Clean ATR accessory

Moisten cotton ball with alcohol (**do not use Kimwipes, do not use acetone**). Gently wipe accessory with moistened ball.

Discard used pipets and cotton.