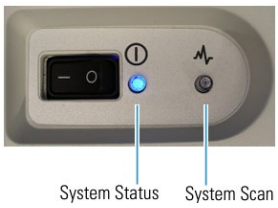
OPT5: Operating Procedure for ATR Spectral Data
Thermo Fisher Nicolet iS50 FT-IR Spectrometer

Refer to the OMNIC program help menus

Note: Right clicking on selections from the various pull-down menus in the software will bring up a help menu for that selection

1. **Ensure you have an active OPT5 reservation.**

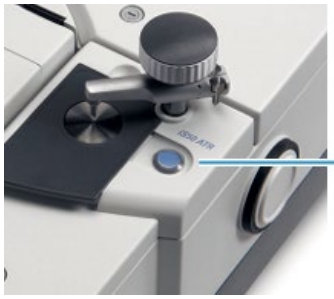
2. **Verify that the system is on.** There should be a blue power light on the back left of the instrument. If the light is any other color than blue, please do not use the instrument and notify SMIF staff. The system scan indicator may be flashing blue, which is acceptable.

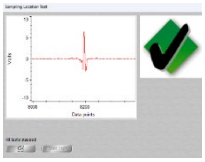


3. **Check the desiccant.** The round indicator on the desiccant compartment cap should be blue. If the indicator has turned pink or white, notify SMIF staff and do not use the instrument.

4. **Turn on the OMNIC computer.** The black box on top of the two instrument computers has a red light that indicates which computer is active. If the RaptIR+ computer (2) is active, ensure that the Paradigm software is closed and press the gray "SELECT" button to switch to the computer for the iS50 and ATR (1), which runs OMNIC software.

5. **Open the OMNIC software** on the computer by double clicking on the desk top icon. If the ATR **touch point** is not already lit solid blue, touch the touch point. It will flash blue for several seconds while the system reconfigures and remains lit when the system recognizes that the sample compartment is ready to be utilized.





6. **The instrument will perform several self-checks.** Once the instrument shows an interferogram with a green check mark and states "All tests passed", hit OK.

7. **Load the default file.** For the ATR accessory, the file is **"Default – ATR".**

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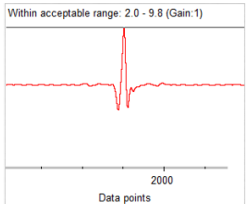
8. **Verify or modify the experiment settings** by clicking on the "Experiment Set-Up" icon in the top left (or by pressing Ctrl+E)

    a. For an explanation of the settings, find "Experiment" in the Index of the OMNIC help topics

    b. **Select the Collect tab and enter the desired values for:**
        i. **Number of Scans** (32 is typical, increasing this number reduces the noise level and makes smaller absorptions easier to distinguish)
        ii. **Resolution** (4 is typical and recommended)
        iii. **Final Format (**either %Reflectance or Log(1/R) are recommended for ATR)
            1. **%Reflectance** is widely used for reflection techniques like ATR
            2. **Log (1/R)** units are for spectra collected using reflection techniques for quantitative comparisons
        iv. **Correction** (none)
        v. **Atmospheric Suppression** (For known samples, this is recommended. Atmospheric Suppression may mask peaks in unknown samples.)
        vi. **File Handling** (It is strongly recommended that you save interferograms. With this, you can restore original data after processing and keep an archive of original data.)
        vii. **Background Handling** (The default is to collect a background before every sample. For most applications, you don't need to collect a new background spectrum for each sample spectrum if parameters have not changed.

    c. **Select the Bench tab**

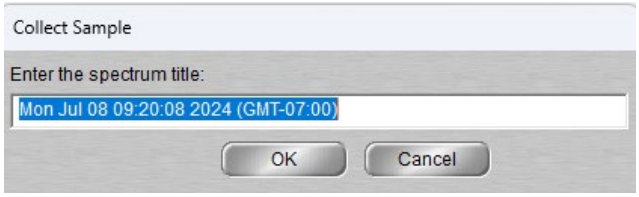


        i. Verify that the laser interferogram is present
        ii. Verify that the value for "Sample Compartment" is "iS50 ATR"
        iii. **Set the desired Max. and Min. range limit** for the measurement (in wavenumbers). To change them, double click the value and type the desired value. The range for the diamond ATR is 4000 – 400 wavenumbers
        iv. **Gain** amplifies the detector signal intensity and is helpful when the IR signal is weak. Autogain is suggested.
        v. **Optical Velocity** is the speed of the moving mirror in the interferometer. Slower velocities take longer to collect a spectrum and increase the spectrum intensity. 0.4747 is the suggested optical velocity.
        vi. **Aperture** controls the size of the angular size of the IR beam and thus the amount of radiation that reaches the sample and the optical resolution. For the DLaTGS detector, the aperture size should be 100.

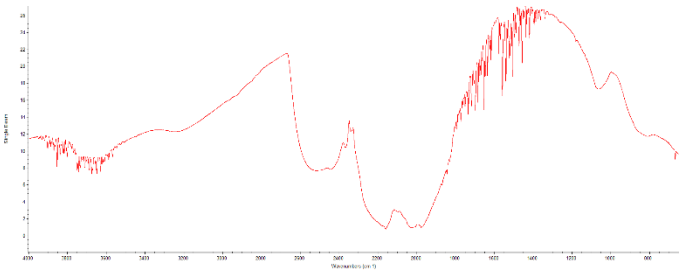
NOTICE

- **The diamond crystal, while very robust, can be broken** if small, very hard materials (like cement or metal beads) are compressed against it at maximum pressure.
- **Make sure your sample will not react with the diamond ATR crystal;** do not use the ATR module with concentrated sulfuric acid (H_2SO_4) or potassium dichromate ($K_2Cr_2O_7$).
- **Do not scrape the diamond crystal with hard materials** such as a metal spatula to remove particles.

9. **Begin the experiment by pressing the "Collect Sample" button** in the toolbar.
   a. Alternatively, you can press the **touch point** next to the ATR
   b. You will be prompted to **give a title to the spectrum**. Enter the title of the sample you plan to run. If you do not enter a title, the software will default to a name that details the date and time of the experiment. Hit OK or the **touch point** to move forward.



   c. **You will be prompted to collect the background. Ensure that the pressure tip is not contacting the diamond and no sample is present.** To raise the pressure tip, rotate the pressure control knob counterclockwise. Once raised, you can also rotate the pressure tower to move the tip out of the way. Once you have verified that nothing is contacting the diamond, hit OK or press the **touch point**.
   d. A background spectrum will appear in the Collect Sample window. A typical background spectrum will look like the spectrum pictured below (with absorptions from the diamond crystal appearing as low-level noise between 2300 cm^{-1} and 2000 $^{cm-1}$).

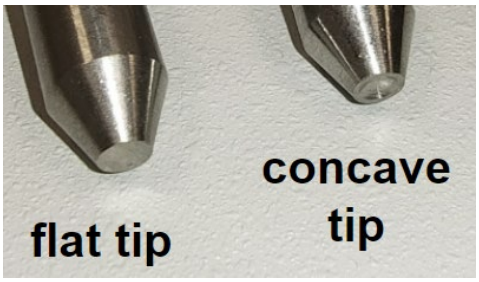


While the spectrum is being collected, you can view the status of the scans in the lower left of the software. The amount of background scans will match the amount of scans you specify for your sample.

10. **Once the background scan has been completed, do NOT hit "OK" until you have proceeded to the next relevant section and loaded your sample.**

Loading a Solid Sample

11. **Ensure that the appropriate pressure tip is installed** on the pressure tower. If needed, with the pressure tower fully raised, rotate the tip to loosen it and remove it from the pressure arm. Place the unneeded tip in the tip storage at the back of the arm and screw in the needed tip.
    a. The **flat tip** is ideal for measuring films, flat plastics, tiny shavings, and fibers. This is the general-purpose tip style.
    b. The **concave tip** allows for good contact with powders and curved solids such as a tablet or polymer bead.



12. **Place the sample in the center of the crystal directly under the pressure point.** For the best results, the sample should cover the crystal completely.

13. With the pressure arm in the sampling position (you will feel it lock into place upon rotation), **lower the pressure arm by rotating the large pressure control knob clockwise until you feel and hear it click.** The tip should be pressed firmly against the sample. Once you feel and hear the click, the maximum force (267 N or 60 lbs) is achieved, and additional rotation of the control knob has no effect.

    Proceed to measurement (step 16).

Loading a Liquid Sample

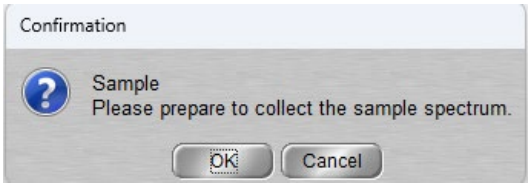
Pressure arm in resting position

14. **Raise the pressure arm and move it to the resting position.**

15. **Use a dropper or syringe to place a thin film of sample on the crystal.** For the best results, the sample should cover the crystal completely.

Measurement

16. **When you are ready to scan your sample, hit 'OK' or press the touch point.** As data is collected, the sample spectrum in the Collect Sample window is updated. The number of scans collected and the total number of scans for the collection are displayed to the right of the gauge at the bottom left of the screen.



17. Once the scan is complete, you will be prompted to add the spectrum to the viewing window. **Choose "Yes" to add the sample spectrum to the window.**

18. If needed, click the spectrum to select it. The active spectrum will be red. **To save the file, choose File > Save As and enter a file name.**
    a. Choose your personal directory within the UserData folder on the desktop for the file location.
    b. Select the file type. The most common file types to save are:
        i. Spectra file (.SPA) (OMNIC format, includes experimental conditions and interferogram / raw data)
        ii. Text file (.CSV)
        iii. Image file (.TIF)
    c. The experiment conditions under which the spectrum was collected can be viewed by clicking the "i" information button beside the spectrum list pull-down menu. These conditions can be saved by clicking the copy button and pasting into the Notepad program, then saving as a text file.

19. **Process the data.** (If desired.)
    a. The OMNIC software can be used to convert the spectrum units, find and label peaks, adjust the spectrum layout, and overlay or compare spectra using commands under the View, Process, and Analyze pull-down menus.
    b. Right click on any of the commands in these menus to view the online help file for that command.
    c. The command "IR Spectral Interpretation" in the Analyze pull-down menu can be useful for identifying various chemical (functional) groups in your sample.

20. **Compare the spectrum to known materials in the library.** (If desired.)
    a. Select the desired spectrum by clicking on it. The active spectrum will be red.
    b. Choose "Library Set-Up" from the Analyze pull-down menu.
    c. Choose the libraries for your search and then click on "search" to produce a set of matches.

21. **When finished:**

    a. Unload your sample and clean the crystal.

NOTICE

- **Use only water, mild soap, alcohol (isopropyl or methanol) or toluene to clean the crystal (IPA is provided).**
- **Avoid spilling solvent onto the ATR module or instrument.**
- **Avoid brushing powder into the space between the metal crystal plate and the plastic sampling plate.**

i. Dab the crystal with a delicate task wipe (such as a KimWipe) to remove most of the sample. Do NOT use a metal spatula to remove solid samples.
ii. Dampen another KimWipe with IPA and use it to wipe the crystal.
iii. If the sample is oily or viscous, place a drop of IPA (using a pipette, NOT the squirt bottle) on the crystal and wipe it with another clean KimWipe.

b. Exit the OMNIC software.
c. End your active OPT5 reservation.
d. ***Leave the FT-IR system power on.***