

FTIR S.O.P

Instrument:

Nicolet iS50 FTIR from Thermo Fisher

Software:

Omnic & Omnic Specta- Both software are available for download as well as databases

Purpose:

To utilize the induced dipole moment from IR active materials, which is caused by the absorption of Infrared Light, to analyze vibrational modes, allowing for quantitative and qualitative analysis of materials.

To Begin:

➤ **Know the specifications of the instrument.**

Specifications	Nicolet iS50
Detector 1: MCT-A	Cryo, photon detector
Detector 2: DTGs	Thermal detector, “walk up”
Beam Splitter	Automated Beamsplitter Exchanger (ABX)
Source Type	Dual Source capable: Polaris™ long-life IR source
Spectral Range	15 to 27,000 cm ⁻¹
Spectral Resolution	Better than 0.09cm ⁻¹
Crystal	Diamond
Crystal Depth of Penetration	2 um
Crystal Refractive Index	2.4
Crystal Incident Angle	45 degrees

➤ **Steps to startup the instrument**

NOTE: The instrument stays on, there is a desiccator in the back left corner with a color indicator, if not blue moisture is trapped inside the instrument contact Facility Manager

1. The instrument should be on, if not there is a power switch in the back left corner
2. Log on to the computer
 - Computer Password is Ftir and is listed as a hint in case it ever changes

➤ **Open Omnic**

- Upon initialization of Omnic, a stability test will be performed to ensure internal optics are properly aligned and that the currently attached read mode fixture is properly in place.

➤ **Select Read Mode**

- Attenuated Total Reflectance
 - ATR crystal is diamond, which has the widest spectral range (25,000 cm⁻¹ -525 cm⁻¹) compatibility.
 - Diamond has a refractive index of 2.4
 - Diamond has a depth of penetration of 2 μm
- Transmission
 - Transmission holders are available for different samples
 - Flow cells are not provided
 - A background of the empty cell is required before testing samples

➤ **Setup experiment**

- Open Experiment Setup
 - Under Bench select the detector to be used.
 - MCT requires liquid nitrogen, contact Facility Manager, detector is very delicate, improper loading could result in damage to the detector which can be expensive and have a long down time.
 - DTGs is a “walk up” detector as it requires no sample preparation
 - Under Collect setup parameters for measurement
 - No. of Scans
 - Must be a multiple of 2 due to the Fourier Transform model
 - The more scans you do the more noise you can average out
 - Start with 16 scans and adjust from there
 - Resolution
 - Resolution selects the data spacing being used, the more datapoints being collected the longer the experiment.
 - Resolution of 4 is default and a good starting point.
 - Final Format
 - Not actually a final format, the overlay can be changed after a measurement
 - Correction
 - The instrument has little to no atmospheric suppression. Corrections can be applied based on read mode
 - For ATR use ATR correction to minimize excess noise below 800cm⁻¹
 - For Transmission use H₂O and CO₂
 - Automatic Atmospheric Suppression
 - It is recommended to leave this on especially for ATR in case the room is humid
 - Preview Data Collection
 - Enabling this allows for a start collection button to appear before a measurement is starting to ensure proper contact with the diamond is present.

- Background Handling
 - For typical runs, one background is taken at the start and the samples are collected after. If doing this select collect background after 180 minutes. Most runs will not take this long so you won't be asked to take a background within your reservation window.
 - If you have a flow cell or want to save a background, you can use the option above first, then save the background to use a specified background file.
- These are the recommended settings to change. You can adjust settings and save as a preset to not have to change this again.
- Load sample onto instrument
 - Before loading a sample make sure to have the sample loading area clean
 - For the ATR use IPA, methanol or water to clean the diamond with a Kim wipe
 - Also clean the compressor otherwise when compressing solid samples extra unwanted results may appear.
 - Click Collect Background
 - Once the background is finished you will be asked if you would like to add the background to the current window, it is recommended to do this in order to confirm the background peaks are not bleeding into the sample spectrum
 - Click Collect Sample
 - Name the sample you will be running.
 - If you name the sample how you will name the file you will save time later on when saving data.
 - Add the spectrum to window 1
- Process Data for best results.
 - It is recommended to take a baseline correction by clicking, auto baseline correct
 - Find Peaks will allow you to set a minimum for where to find peaks based on intensity
 - Click the bar all the way down to find all peaks in spectrum
 - Sensitivity bar on the left will allow you to find smaller peaks
 - Under Process you can switch from Absorbance, %Transmittance or other file types available.
 - Under Analyze IR Spectral Interpretation will categorize your functional groups for you
 - Keep in mind that functional groups can appear in similar regions so it is good to have a rough idea of what you are working with.
 - Under Analyze and Library Setup, different databases can be enabled. Select the databases you need or leave all selected.



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- Click Search and the software will go through all the databases to find the match to your sample.
 - % match will change based on database, experiment settings, and how the data was processed.
 - ATR correction is not enabled by all databases
 - Some databases will have different grades for different solvents
- For a more detailed walkthrough of data processing contact Facility Manager

To Finish:

- Make sure the area and instrument are clean for the next user
- Save Data
 - Data can be saved by going to **File...Save As**
 - In the directory **User Data** folder is located in the desktop
 - .SPA file can only be opened by *Omnice*, if you will be downloading software use this file.
 - For using data in other programs .CSV
- The software can now be closed