

Nicolet FT-IR Procedure

Jerry Hu (Lab Manager), ext. 7914 or 7940

jghu@mrl.ucsb.edu

Jaya Nolt (Lab Tech), ext. 4997

jaya@mrl.ucsb.edu

Materials Research Laboratory
University of California, Santa Barbara

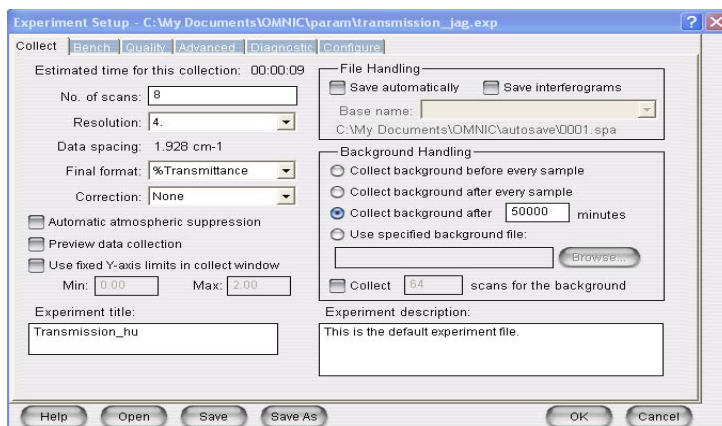
Getting Started

- 1.) Sign in on the Log Sheet. Record your full name, advisor's name, department, start time, and your recharge number.
- 2.) Log into your personal IR account.
- 3.) Open **OMNIC** by double clicking the icon:
Click "OK" for error messages that appear (should be two of them).

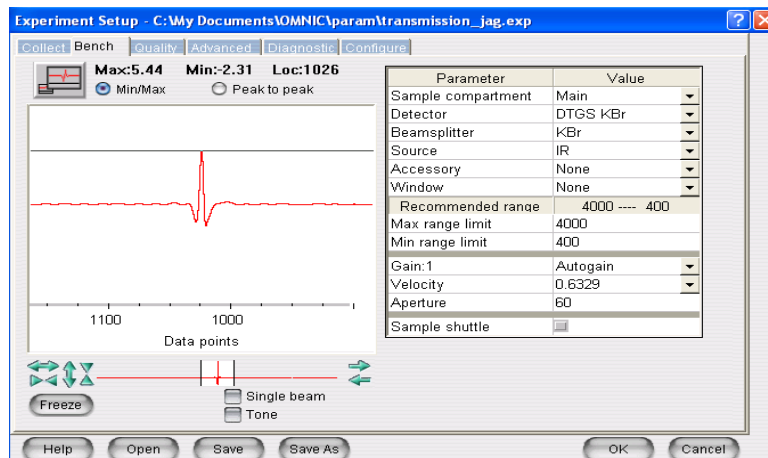


Instrument Setup

- 1.) All parameters for **routine FTIR experiments** are already set under "Default-Transmission" located under the *Experiment* drop down menu. If this option is not already highlighted then select it from drop down menu. (Note: if you wish to create your own parameters you are welcome to do so, but please save them under your own name and remember to set the parameters back to the default ones before logging out)
- 2.) Make sure: (a) that the sample chamber is empty (IMPORTANT: before opening the sample chamber make sure the shutters are closed – levers are up. Failing to do so will cause damage to the source and detector), the sample chamber door and main door are closed, and shutters to the source and detector are open (levers are down).
(b) that settings are correct by going to *Collect* => *Experiment Setup*
* Number of scans should be 8 and under "Background Handling" the third one down should be checked with "50000" entered into the box



Next go to the *Bench* tab: **Detector** should be set on DTGS KBr
Beam Splitter should be set on KBr
 You should also see a signal in the window:



If no signal is seen like the one above make sure the shutters are open (levers are down). If they are closed (levers are up), then open them and wait a few minutes for signal to appear (**Gain** should be at 1 or 2). If still no signal, then do the following:

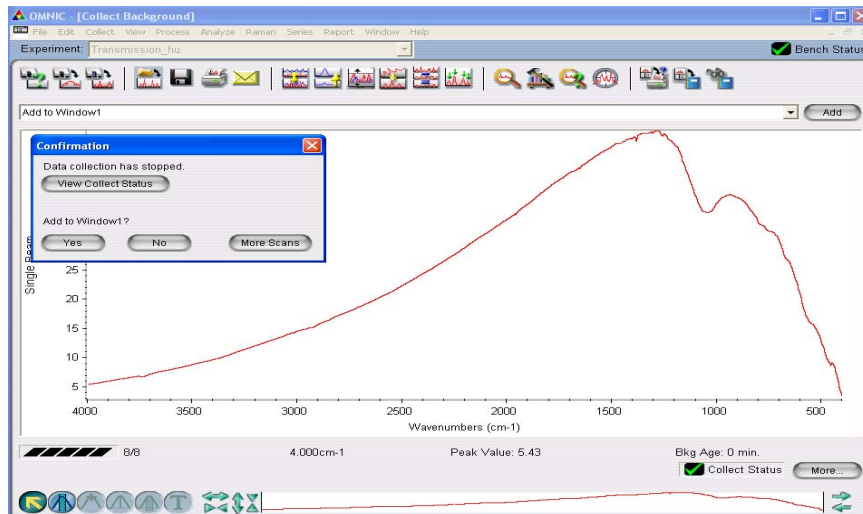
Go to *Diagnostics* tab and hit the “Align...” button to optimize the intensities. Please wait until the Align Bench comment box has disappeared from the screen before doing anything else.

Exit from the *Experiment Setup* box by pressing “OK.”

Collecting a Spectrum

- 1.) Always obtain a new **background spectrum** for each day:
 - a.) Purge sample chamber (only needs ~15 seconds)
 - b.) Go to *Collect* => *Collect Background...*

Should get a spectrum as follows:



Select “Yes” from *Confirmation* box if spectrum is the same; select “No” if not the same and repeat background collection. (Note: CO₂ is visible at ~2300cm⁻¹ and H₂O is visible at ~1400cm⁻¹ and 1800cm⁻¹; if these peaks are visible then allow more time to purge the sample chamber and repeat background collection)

2.) Collect **sample spectrum**:

- (a) Close the shutters before opening sample chamber.
Place sample in the chamber, making sure the target beam hits the sample and goes to the receiver.
Close the sample chamber, purge the sample chamber, and then open shutters.
- (b) Go to *Collect => Collect Sample...*
- (c) A *Spectrum Title* box will open; this title will appear on the spectrum itself. It can be anything you want; most people put the name of the sample, any necessary experiment details/parameters, etc.
If the spectrum is not acceptable then just hit “Cancel” on this box, and it will not save the spectrum. Repeat sample collection.
- (d) By clicking “Yes” to *Add to Window 1* the sample spectrum will be placed in same window as the background spectrum.

Saving Data

There are two ways to save data; you can do one or the other or both.

1.) To save data as an **OMNIC file**:

- (a) Highlight the data by clicking on the spectrum that you want to save with the left mouse button (the desired spectrum will turn red).
- (b) Go to *File => Save* (change to your own directory; if you do not have one then create one by clicking the right mouse button => *New => Folder*)
- (c) Type in the name of file in *File Name* box, keeping the *Spectra (*.spa)* extension in the *Save as Type* box, press “OK.”

2.) To save data as an **Excel/Text file**:

- (a) Highlight data desired to be saved
- (b) Go to *File => Save as...* (change to your own directory)
- (c) Type in the name of file in *File Name* box, change the *Spectra (*.spa)* extension to *CSV Text (*.CSV)* in the *Save as Type* box, press “OK.”

If you wish to **print a spectrum** go to *File => Print* and make sure that the *HP Laserjet 6L* is highlighted

When you are Done

- 1.) Under the bench tab in the top left corner there is a Max IR intensity number. Record that number on the log sheet.
- 2.) Exit OMNIC by going to *File => Exit*
- 3.) Log off your account by going to *Start => Log off* (lower right hand corner). Failing to do this may cause erroneous charges to be made to your account as stated in the **IR Policy** section.
- 3.) Leave the spectrometer with all sample chambers closed and the shutters open (levers down). Please return any equipment that you have used to its designated area. Do not leave your samples in the lab; we are not responsible for them.
- 4.) Complete the log sheet with your stop time and the actual time of usage. Please include any problems that you had with the instrument, if any. If you were able to solve the problem yourself, please include your explanation of how you did it; we greatly appreciate it.

The manual for FTIR and for the OMNIC software have clear and easy to follow directions for more complex/different experiments than the simple procedure provided here. The manuals are located on the counter on the other side of the room (directly behind you), or you can find them through online help.

PLEASE DO NOT REMOVE ANY MANUALS or EQUIPMENT FROM THE IR/RAMAN LAB!

PLEASE PUT ANYTHING THAT YOU GET OUT OR USE BACK IN ITS DESIGNATED AREA.

Thank you.

PLEASE NOTE:

Detector Options:

- Standard IR Detector
- Near IR Detector
- Far IR Detector

- Photo Acoustic (PAS) Detector – kept in left hand desk drawer. Dave Margolese regularly uses it.

Beam Splitter* Options:

- KBr – Mid IR
- CaF₂ – Near/Mid IR & Raman
- Substrate Solid – Far IR

*If you change the beam splitter:

- Be **extremely careful** not to drop it or touch the center
- Always put the **KBr** beam splitter back as it is the **default** one

Sample Options:

- **Solid Sample** – Make a KBr pellet
 - You may have to vary the mix of KBR and sample to get a good transparence. To start try ~5 parts dry KBr to ~1 part sample. Grind together in a mortar with pestle.
 - Load into the press cell, filling it to 1/4 - 1/3 of the way full. You may want to pull a vacuum on this to get any water out.
 - Align this cell in the Carver Press and compress to ~ 10,000 PSI on the press pressure gauge. **DO NOT EXCEED 12,000 PSI!!!** Also, **DO NOT** extend the stroke of the press past 5” – use spacers if necessary.
 - Leave under pressure at least 2 minutes
 - The final pellet should be ~ 0.5 – 1.0 mm thick
- **Liquid Sample** – Sandwich sample between two plates
- **ATR Accessory** – Used to measure the reflected spectrum of liquids and solids
 - without special sample handling.
- **DRIFTS Cell** – Reflection cell (In wood box labeled DRA)
 - Load powder into cell (may have to add KBr)
 - Align two small mirrors (do this every time)
 - Harrick Cell is used with the DRIFTS Cell to control the atmosphere around the sample.