

FT-IR Training Notebook: ATR

Lab Manager: Dr. Perry Cheung
MSE Fee-For-Service Facility
Materials Science and Engineering
University of California, Riverside

September 20, 2019 (rev. 2)

Before you begin...

- ☐ Receive a user name and temporary password for Faces scheduling
- ☐ Identify your ENGR username and Password from Systems
 - ☐ If you don't have an ENGR account, send me the following:
 - ☐ Full name
 - ☐ Principal Investigator (PI)
 - ☐ UCR NetID
 - ☐ email
- ☐ Coordinate a time with the lab manager for training
- ☐ Schedule a 1 hour block on Faces for your training

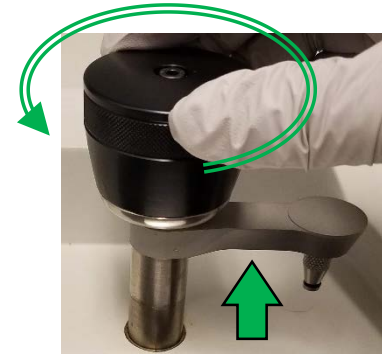
FT-IR Operation

- I. Pressure Tower Setup
- II. Initiate Software
- III. Collect Background
- IV. Sample Prep: Solids
- V. Sample Prep: Liquids
- VI. Collect Sample
- VII. Saving Data
- VIII. Peak Identification
- IX. Cleanup
- X. Library Search
- XI. Smart Transmission Accessory

I. Pressure Tower Setup – 1/2

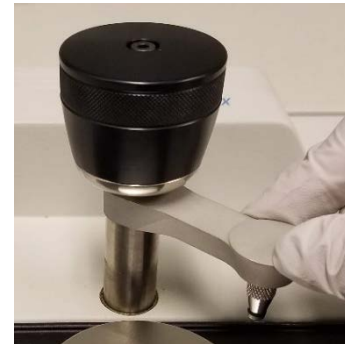
1. To adjust the position of **Pressure Tower**:

- Turn **Knob counter-clockwise = raise Tower**
- Turn **Knob clockwise = lower Tower**



2. Inspect the **Pressure Tip** by moving **Tower Arm** to **Cleaning Position**

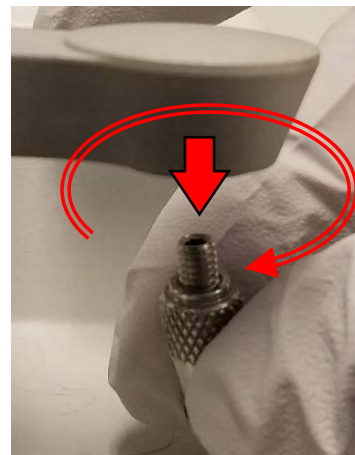
- Move **Tower Arm** to the right until it stops



Cleaning Position

3. Clean the **Pressure Tip** (remove if necessary) with appropriate solvent

- Recommend **Water** and **IPA**
- **DO NOT USE ACETONE!**



4. To remove/install **Pressure Tip**:

- Rotate **Tip clockwise = remove**
- Rotate **Tip counter-clockwise = install**

I. Pressure Tower Setup – 2/2

4. Identify appropriate **Pressure Tip** for your sample

- **Flat** – for flat samples such as polymer films
- **Concave** – for powders and curved surfaces
- **Volatiles Cover** – for volatile liquids



Flat



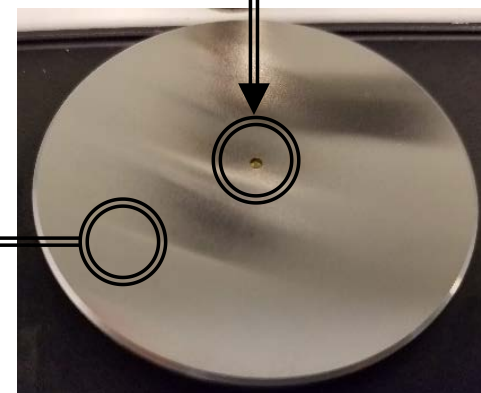
Concave



Cover

5. Use provided **Q-Tips** and appropriate solvent to clean the **Diamond Crystal**

- Recommend **Water** and **IPA**
- **DO NOT USE ACETONE!**
- **DO NOT USE KIM WIPES!**



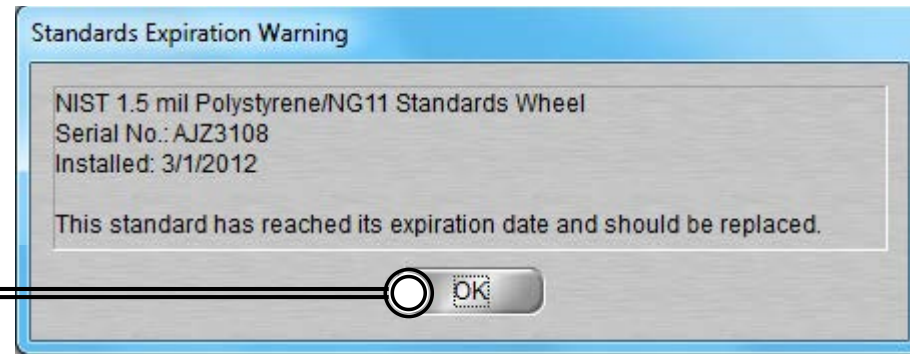
6. You may use **Kim Wipes** with **Water** and **IPA** to clean **Metal Surface** afterwards but avoid scratching the **Diamond Crystal**

II. Initiate Software – 1/10

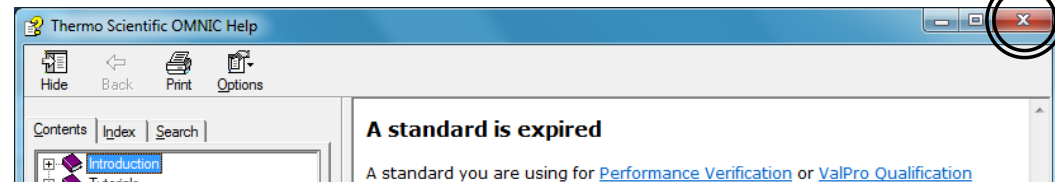


1. Double left-click on the **OMNIC software icon** for FT-IR

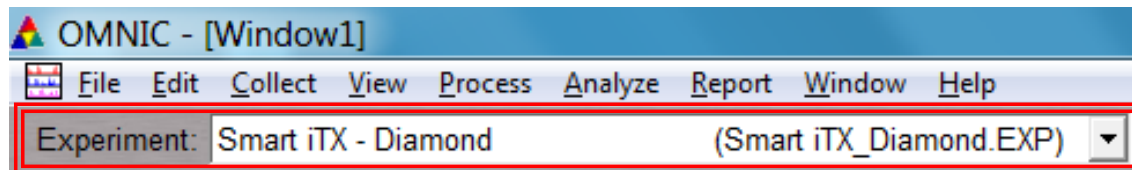
2. Ignore the **Standards Expiration Warning** and click **OK**



3. Close the **Thermo Scientific OMNIC Help** popup window



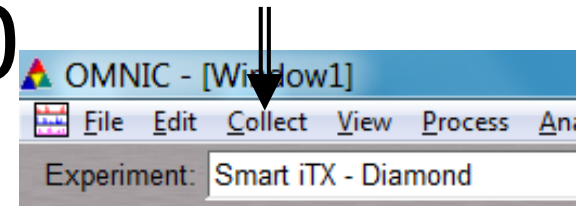
4. The **OMNIC main window** will now appear



5. Confirm that **Smart iTX – Diamond (Smart iTX_Diamond.EXP)** appears in the Experiment window

II. Initiate Software – 2/10

6. Select **Collect -> Experiment Setup** at the top window



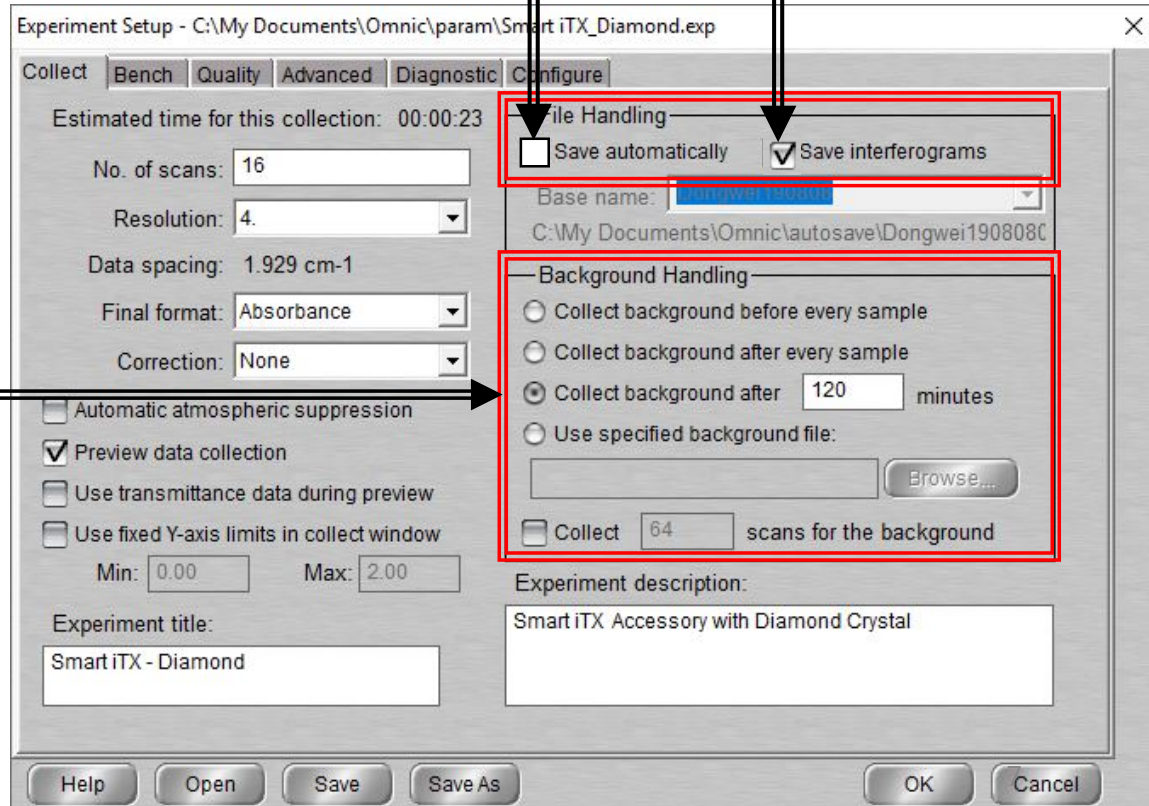
7. Confirm that **Save interferograms** is **checked**

- Saving interferogram data lets you reprocess in case you want to restore the original data, even using a different background or changing parameter settings used

8. Confirm that **Save automatically** is **unchecked**

9. Set preferred **Background Handling** settings

- Before every sample
- After every sample
- **After 120 minutes (default)**
- Use specific file



Note: A new background will be requested if there is a change in resolution or data spacing of your sample spectrum!

II. Initiate Software – 3/10

10. Select desired **No. of scans** – recommend starting at 16 scans

- Increase to optimize desired spectrum signal/noise
- Recommend increments of powers of 4 (e.g. 16, 64, 256, 1024,...)

11. Select desired **Resolution value** – recommend 8 or 4

- Decrease value to increase spectrum resolution
- Decreasing value too much may result in increased noise!

Note: **Aperture = High resolution** if Resolution value is ≤ 2

12. Check **Estimated time for collection**

- Time dependent on **No. of scans** and **Resolution**

13. Select desired **Final format**

- % Transmittance
- **Absorbance (default)**
- Etc...

Note: Convert to other Y-axis units in **Process** menu

Experiment Setup - C:\My Documents\Omnic\param\

Collect Bench Quality Advanced Diagnostic

Estimated time for this collection: 00:00:23

No. of scans: 16

Resolution: 4.

Data spacing: 1.929 cm⁻¹

Final format: Absorbance

Correction: None

☐ Automatic atmospheric suppression

☒ Preview data collection

☐ Use transmittance data during preview

☐ Use fixed Y-axis limits in collect window

Min: 0.00 Max: 2.00

Experiment title:

Smart iTX - Diamond

Help Open Save Save As

II. Initiate Software – 4/10

14. Select desired ***Correction type*** to ***None***

15. Decide if ***Automatic atmospheric suppression*** is desired

- Effects of water vapor and carbon dioxide will be automatically suppressed via quantitative model

NOTE: Do **NOT** use this feature if atmospheric conditions change very slowly, only use if conditions change rapidly

16. Check ***Preview data collection***

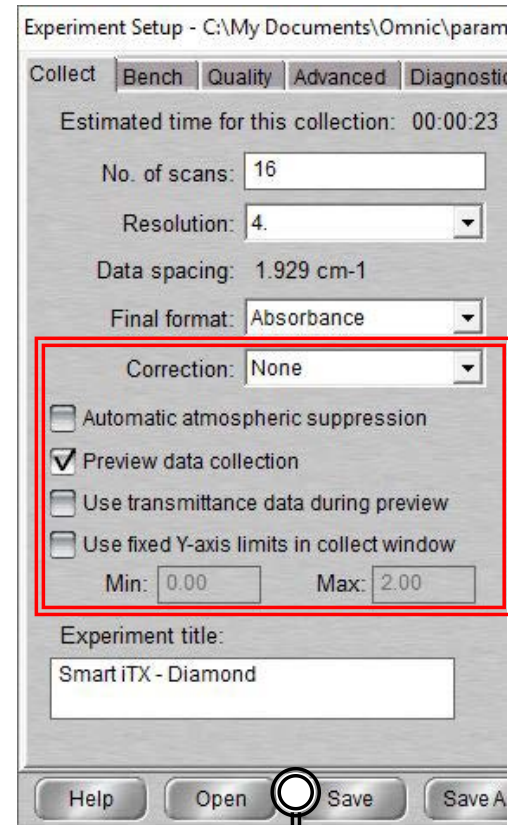
- Views preliminary data before start of sample for verification

17. Decide if you want to preview data collection using % transmittance

- May provide an improved preview of the data

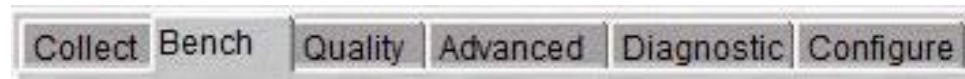
18. Decide if fixed Y-axis limits will be used in the preview

- Recommend using Min: -5% to Max: 105%



II. Initiate Software – 5/10

19. Select **Bench** tab



20. Confirm that the following are correct:

- **Source = IR**
- **Accessory = Smart iTX**
- **Window = Diamond**

21. Select desired **Max** and **Min** range limit for your scans

- Recommend using **Recommended range**

22. Select the **Gain** parameter

- Electronically amplifies signal – recommend **Autogain**
- **DO NOT** set to **Autogain** if performing quantitative analysis

23. Select the desired Aperture

- **High resolution** – used with resolution at 2 or less for better stability and accuracy
- **Medium resolution** – recommended with resolution 4 for better Signal/Noise

24. Confirm the **Screen wheel** is set to **Open**

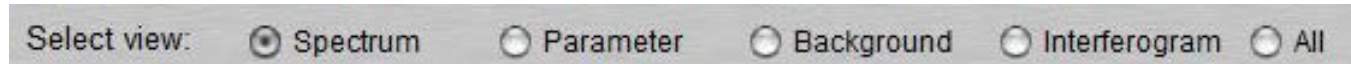
Parameter	Value
Sample compartment	Main
Detector	DTGS KBr
Beamsplitter	KBr
Source	IR
Accessory	Smart iTX
Window	Diamond
Recommended range	4000 ---- 525
Max range limit	4000
Min range limit	400
Gain:8	Autogain
Optical velocity	0.4747
Aperture	Medium resolution
Sample shuttle	<input type="checkbox"/>
Screen wheel	Open

II. Initiate Software – 6/10

25. Select **Quality** tab



26. Determine if you want any spectral quality characteristics to be checked during your scans



- **Spectrum** – checks quality of the spectrum scan
- **Parameter** – checks the scan parameters
- **Background** – checks the quality of the background scan
- **Interferogram** – checks the raw interferogram signal
- **All** – checks all the above characteristics

27. If you choose to check **Spectrum**...

- **Peaks present?** – checks for peaks and if sample is positioned correctly, recommend **ON** at **50%**
- **Totally absorbing peaks** – checks for absorbing peaks, recommend **ON** at **50%**
- **Fringes or channeling** – checks for back reflection inside sample, recommend **ON** at **50%**
- **Derivative peaks** – checks for derivative-shaped peaks, recommend **ON** at **50%**
- **Baseline error** – checks for baseline problems, recommend **ON** at **50%**
- **CO₂ levels** – checks for CO₂ absorption, recommend **ON** at **50%**
- **H₂O levels** – checks for H₂O absorption, recommend **ON** at **50%**

II. Initiate Software – 7/10

28. If you chose to check **Parameters...**

- **Spectral range** – checks if spectral range is consistent for the hardware, recommend **ON**
- **Apodization correct** – checks apodization type is appropriate, recommend **ON**
- **Resolution** – checks if resolution is appropriate for the experiment, recommend **ON**

29. If you chose to check **Background...**

- **Contamination peaks** – checks for contaminants, recommend **ON** at **50%**
- **Detector icing** – checks signs of detector icing, recommend **NO**
- **CO₂ levels** – checks for CO₂ absorption, recommend **ON** at **50%**
- **H₂O levels** – checks for H₂O absorption, recommend **ON** at **50%**
- **Background correct for accessory** – checks background spectrum, recommend **ON** at **50%**

30. If you chose to check **Interferogram...**

- **Peak amplitude within range** – checks if amplitude is sufficient, recommend **ON**
 - **Interferogram minimum = 0.20** and **Interferogram maximum = 9.80**
- **Minimum peak above noise** – checks if peak signal is above noise level, recommend **ON**
 - **Peak Minimum = 10**

II. Initiate Software – 8/10

31. Select **Advanced** tab



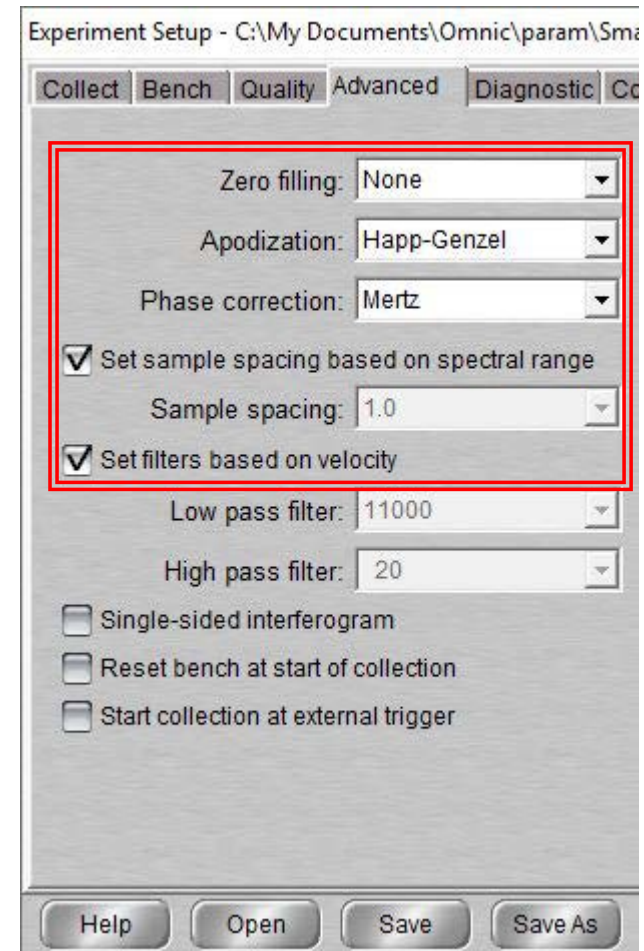
32. Confirm **Zero filling** is set to **None**

33. Confirm **Apodization** is set to **Happ-Genzel**

34. Confirm **Phase correction** is set to **Mertz**

35. Confirm that the following are checked:

- **Set sample spacing based on spectral range**
- **Set filters based on velocity**





II. Initiate Software – 9/10

36. Select **Diagnostic** tab



37. Click on indicators to check spectrometer components

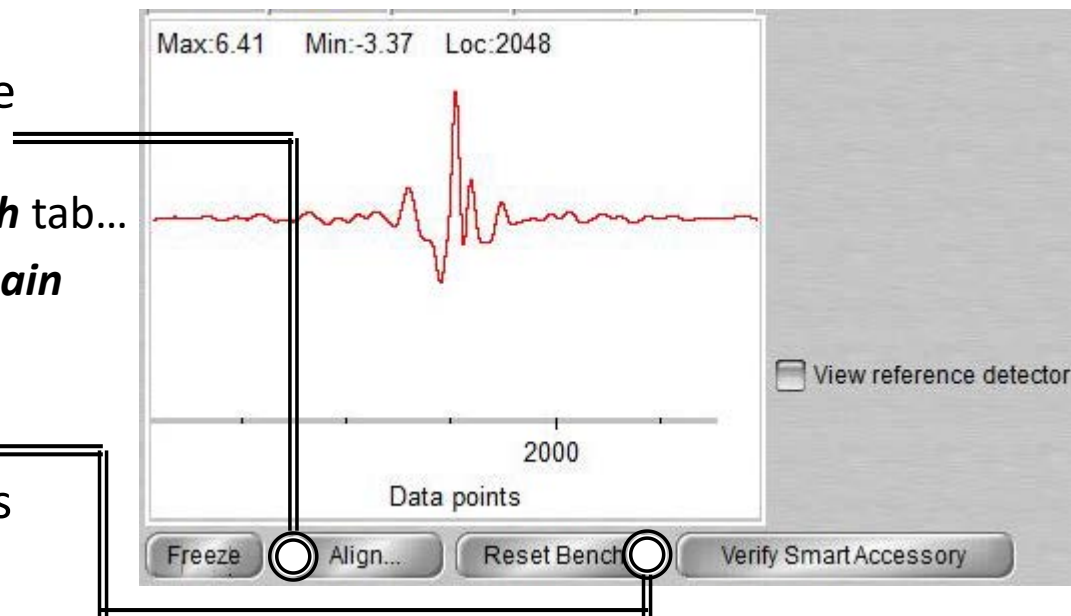


- If the values are within the Acceptable Range, they will appear as a 
- If any values show , contact the Lab Manager immediately!

38. Click on **Align** button to perform automatic alignment to maximize the detector signal

- Set **Gain = 1** before **Align** in **Bench** tab...
- Remember to reset **Gain = Autogain** afterwards

39. Click on **Reset Bench** button to reposition the peak if drift occurs



II. Initiate Software – 10/10

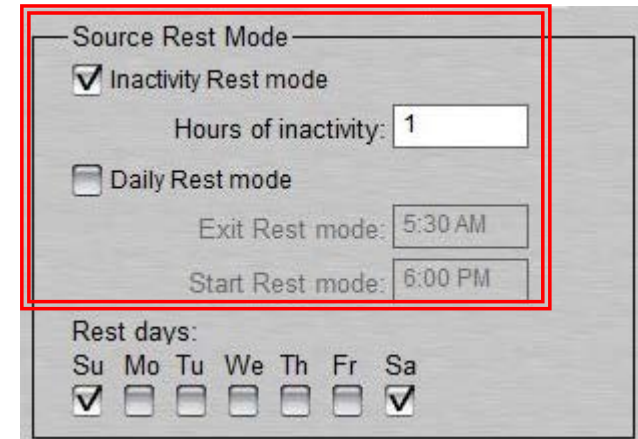
40. Select **Configure**



41. Confirm **Inactivity Rest mode** is checked

- Confirm **Hours of inactivity** is set to “1” hour

42. Confirm **Daily Rest mode** is not checked

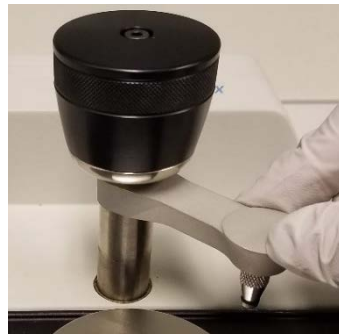


43. Click “**Save**” then “**OK**”

III. Collect Background – 1/2

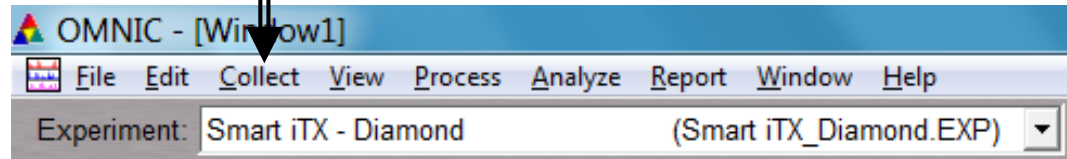
1. It is critical that the **Crystal** is cleaned **BEFORE Background** is collected!
2. A single **Background** can be used to analyze multiple samples, but it is recommended to collect new **Background** at least every **2 hours**

3. Move the **Pressure Tower** to the **Cleaning Position**

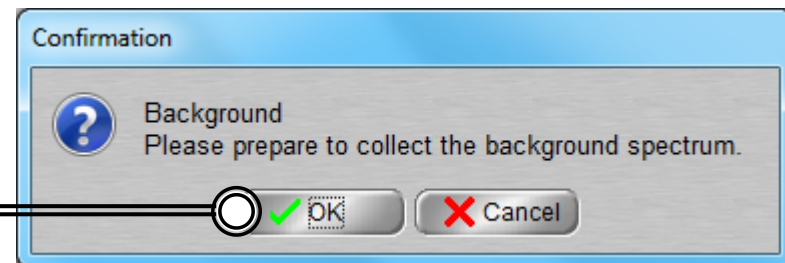


Cleaning Position

4. Select **Collect -> Collect Background**

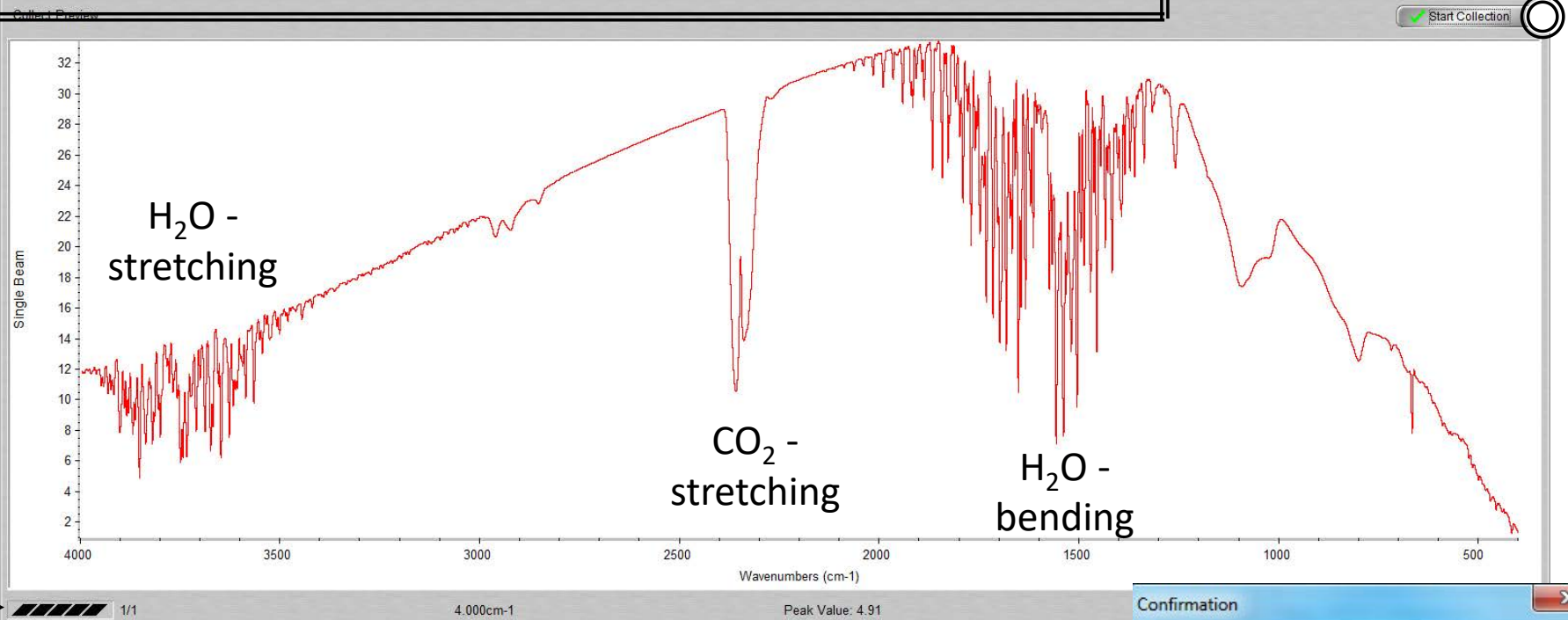


5. Confirm to collect background by clicking **OK**



III. Collect Background – 2/2

6. Preview **Background Collection** then click **Start Collection** to begin
7. The **Background Collection** will begin with the progress shown at the bottom



8. Confirmation of **Data Collection** will be shown
9. Click **Yes** to add data to current Window



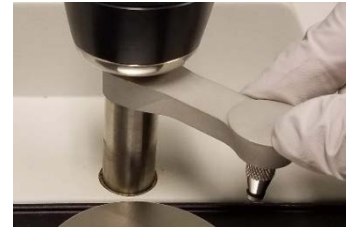
IV. Sample Prep: Solids – 1/1

1. For ***Solid*** and ***Thin Films*** use ***Flat Tip*** and for ***Powder*** use ***Concave Tip***...

2. Ensure the ***Flat*** or ***Concave Pressure Tip*** is installed first

3. Place sample onto ***Crystal***

4. Move the ***Pressure Tip*** into ***Sampling Position***



Sampling Position

5. Lower the ***Pressure Tower*** to press the ***Sample*** against the ***Crystal***

6. The ***Pressure Tower Knob*** will ***Click*** and ***Freely Rotate*** when the maximum pressure is reached

V. Sample Prep: Liquids – 1/1

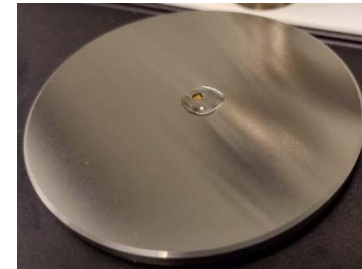
1. For ***Liquid, Paste, or Gel Sample...***

2. Move the ***Pressure Tip*** into ***Cleaning Position*** and dispense sample onto ***Crystal***



Cleaning Position

3. The sample should cover the ***Crystal*** but **DO NOT OVERFILL** or else the sample will run off the ***Crystal Plate***



4. For ***highly volatile samples***, place ***Volatiles Cover*** over sample to reduce of evaporation

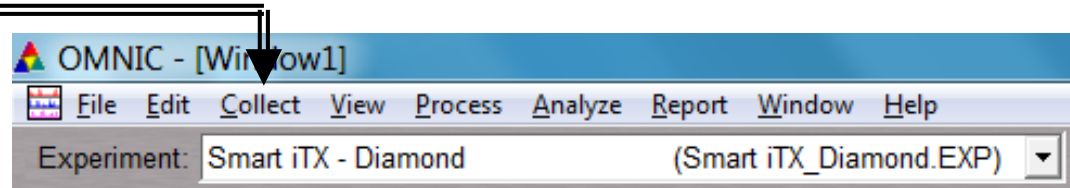


- Install ***Flat Pressure Tip***, move into ***Sampling Position***, and lower the ***Pressure Tower*** until the ***Pressure Tower Knob Clicks*** and ***Freely Rotates*** when the maximum pressure is reached

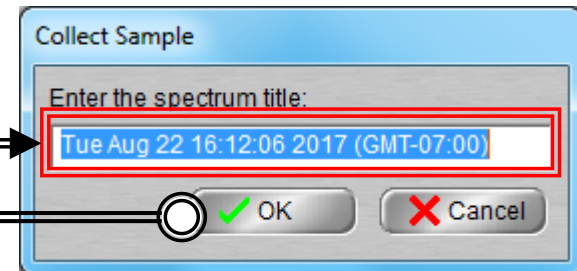


VI. Collect Sample – 1/2

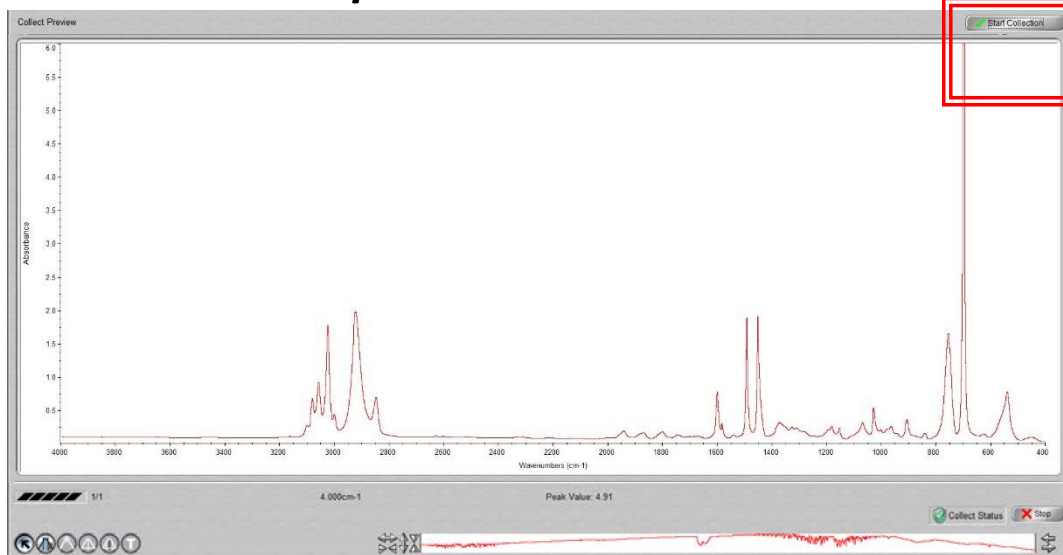
1. Select **Collect** -> **Collect Sample**



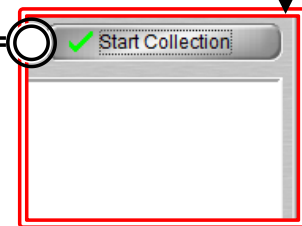
2. Enter **Spectrum Title** and click **OK** to **Collect Sample**



3. Preview **Sample Collection**

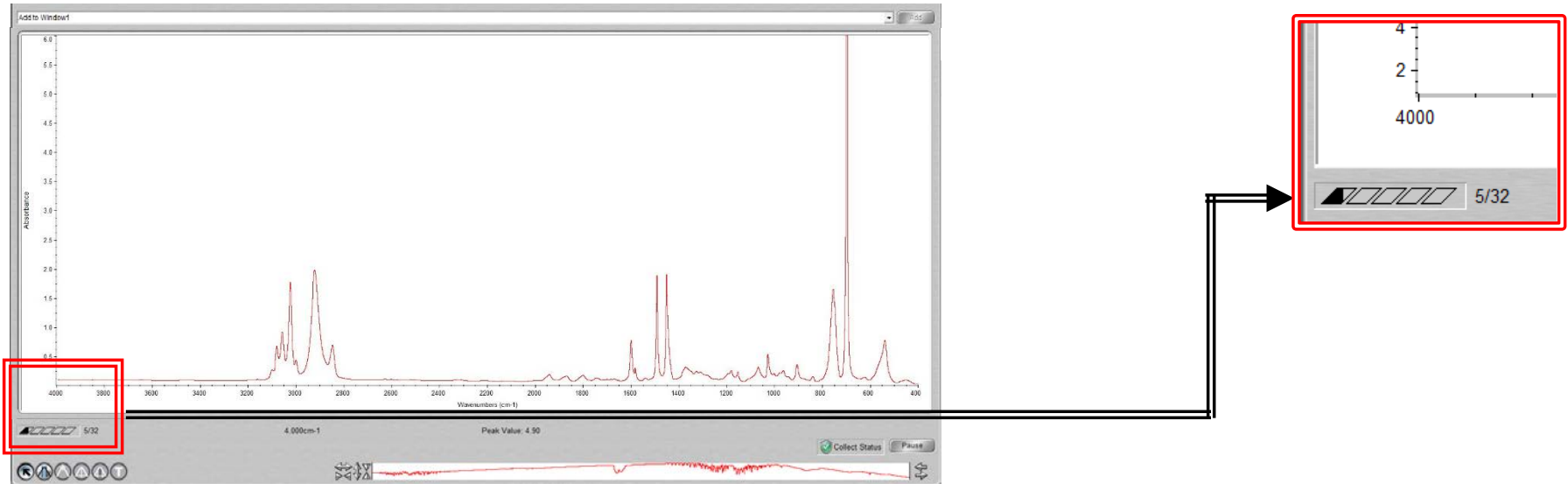


4. Click **Start Collection** to begin **Sample Collection**



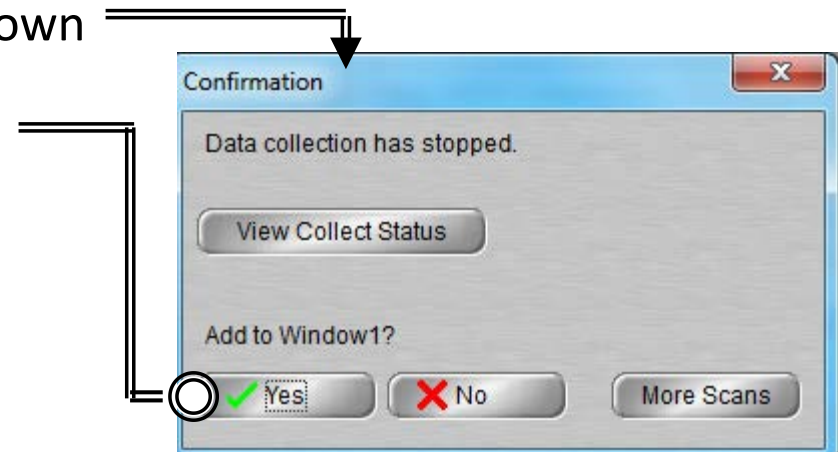
VI. Collect Sample – 2/2

5. The **Sample Collection** will begin with the progress shown at the bottom


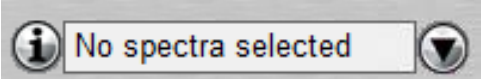



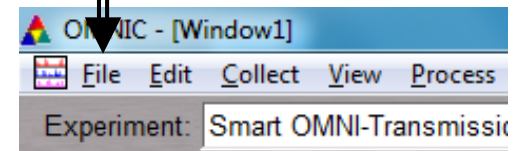
6. Confirmation of **Data Collection** will be shown

7. Click **Yes** to add data to current Window



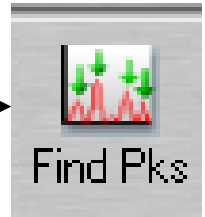
VII. Saving Data – 1/1

1. Specific spectra can be selected using the  selection tool at the bottom of window and clicking on it or selecting from dropdown box 
2. Multiple spectra can be selected/deselected by holding down the **Ctrl** key and clicking spectra
3. Click **File -> Save** to save a spectrum (e.g. default is SPA) using the current filename 
4. Click **File -> Save As** to save a spectrum into another file type (e.g. CSV or TIFF)
5. Click **File -> Save Group** to save more than one spectrum as a group in one file having file extension .SPG to open later



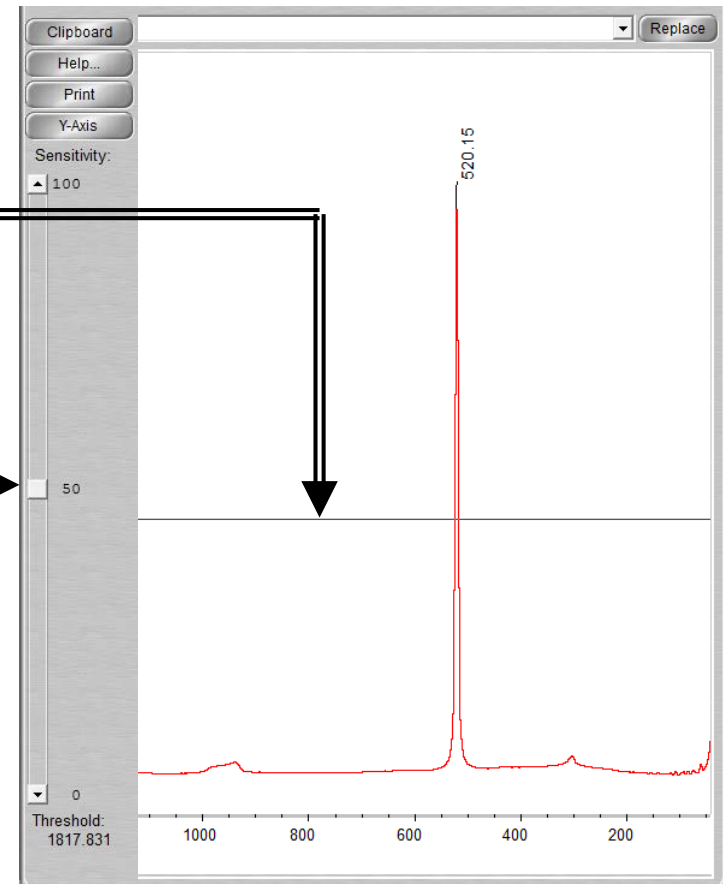
VIII. Peak Identification – 1/1

1. Click on “**Find Pks**” button at the top



2. Click the spectrum window to adjust the **Threshold** position on where peaks are to be considered

3. Adjust the **Sensitivity** button to separate peaks from noise

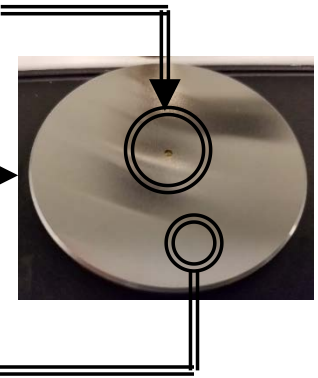


IX. Cleanup – 1/1

1. Remove **Sample** from the **Crystal** without scratching the **Crystal**

2. Use provided **Q-tips** and appropriate solvent to clean the **Crystal**

- Recommend **Water** then **IPA**
- **DO NOT USE ACETONE!**
- **DO NOT USE KIM WIPES!**

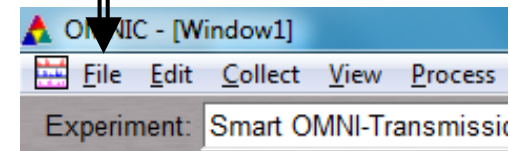


3. Clean the **Pressure Tip** (remove if necessary) and **Metal Surface** with appropriate solvent and **Kim Wipes**

- Recommend **Water** and **IPA**
- **DO NOT USE ACETONE!**



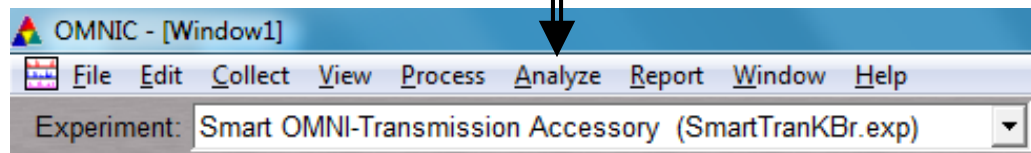
4. Click on **File -> Exit** to shut down the software



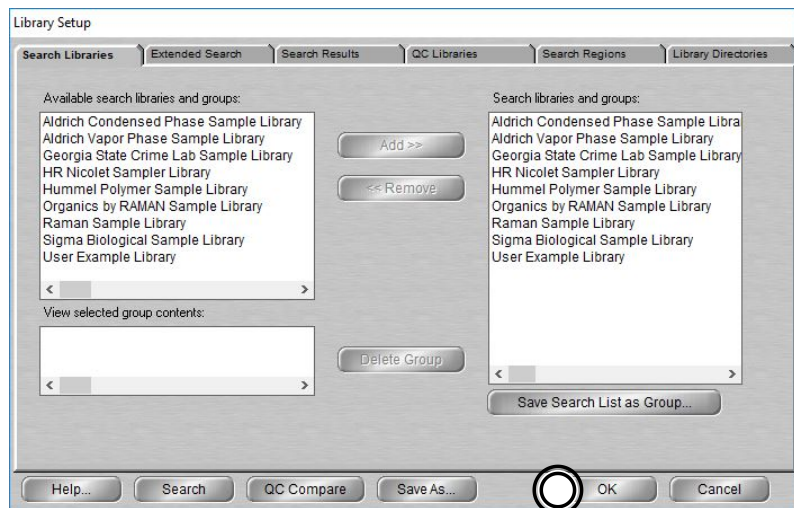
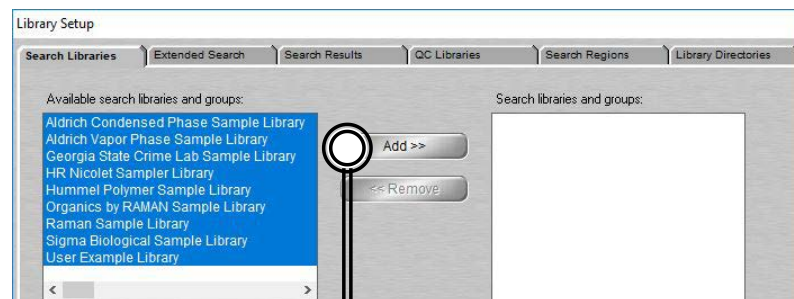
5. Log off of your ENGR account

X. Library Search – 1/5

1. Click **Analyze** and select **Library Setup**

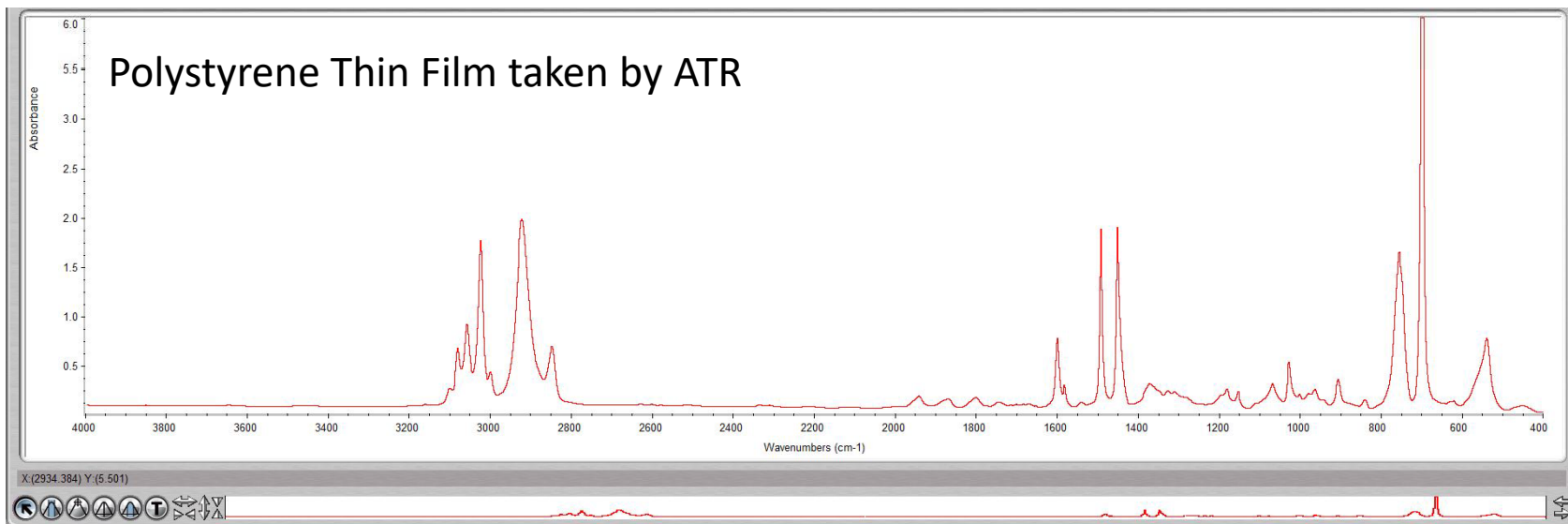


2. Select desired **Libraries** or select all
3. Click **Add >>**
4. Click **OK**

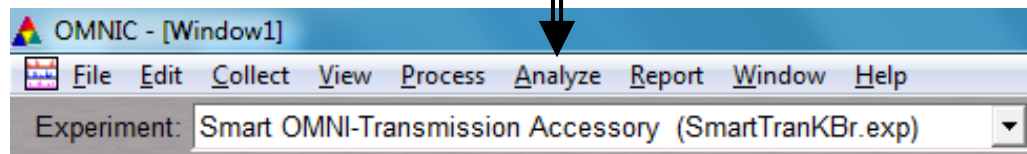


X. Library Search – 2/5

5. Select the desired spectra you wish to search for a library match



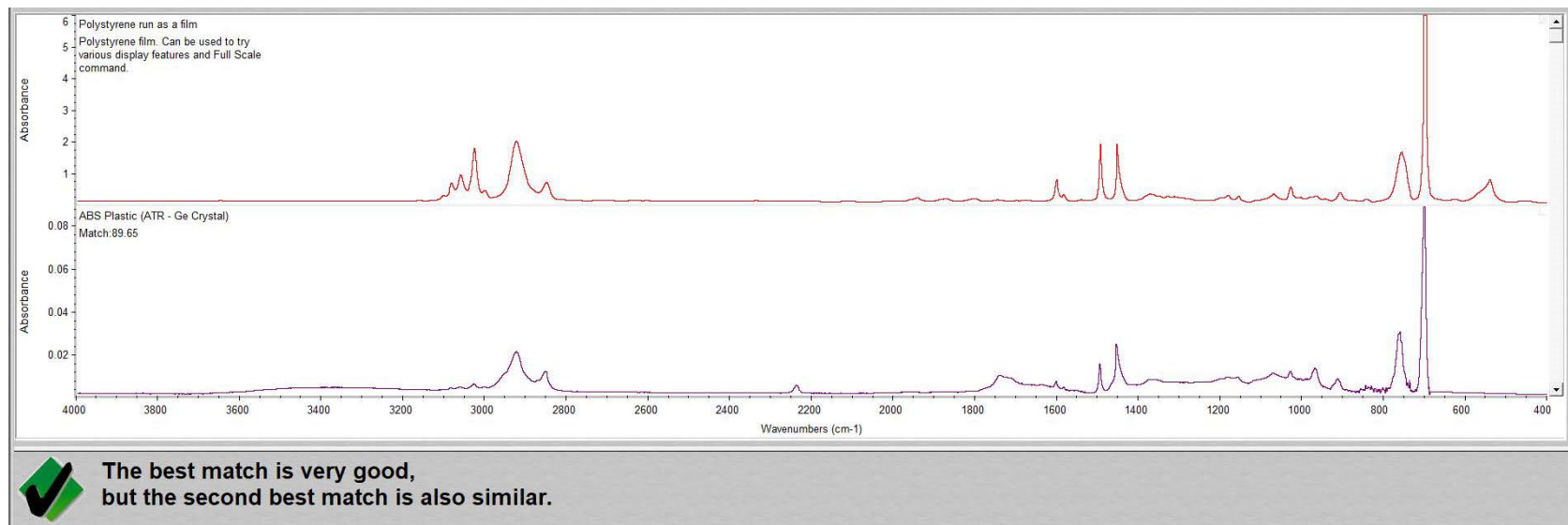
6. Click **Analyze** and
select **Search...**
or click **Search** icon



7. Select desired **Libraries** or select all

X. Library Search – 3/5

8. The top matches will be shown (below) your acquired spectra (top)

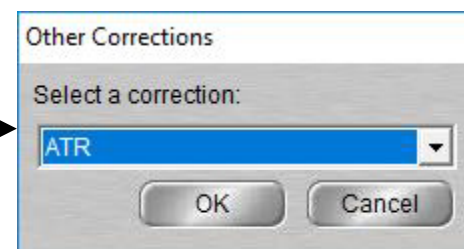
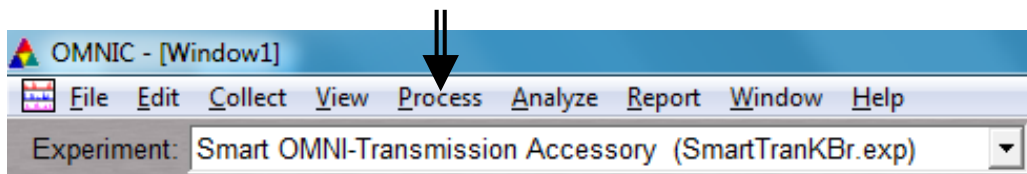


9. Click **View Match List** and select either **Overlay** or **Stack** view



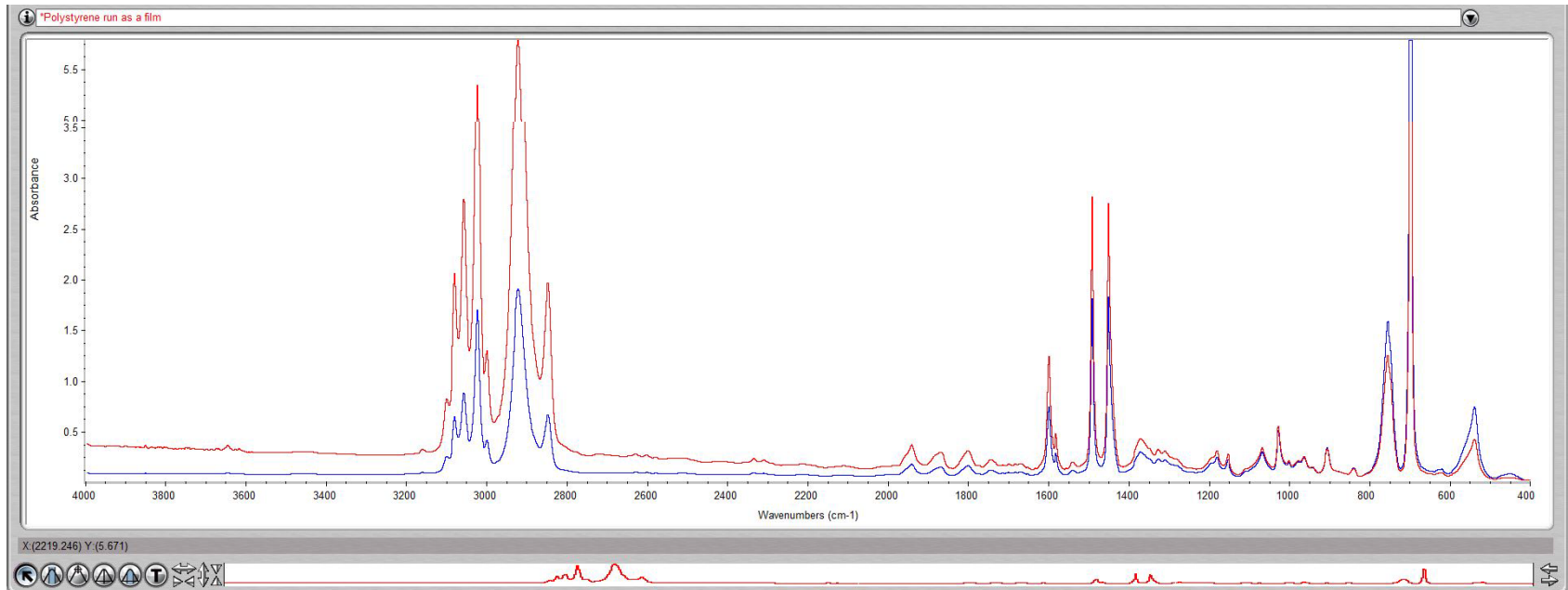
10. Perform **ATR Correction** to achieve better match results

11. Click **Process > Other Corrections...** and select **ATR**

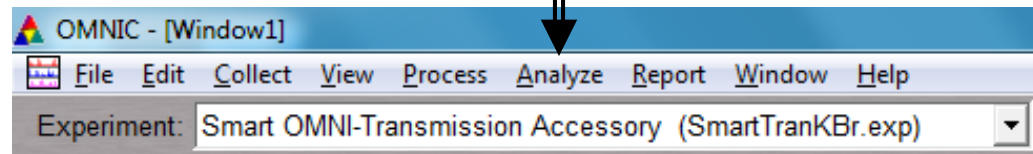


X. Library Search – 4/5

12. The ATR Corrected spectra will be created and marked with a *

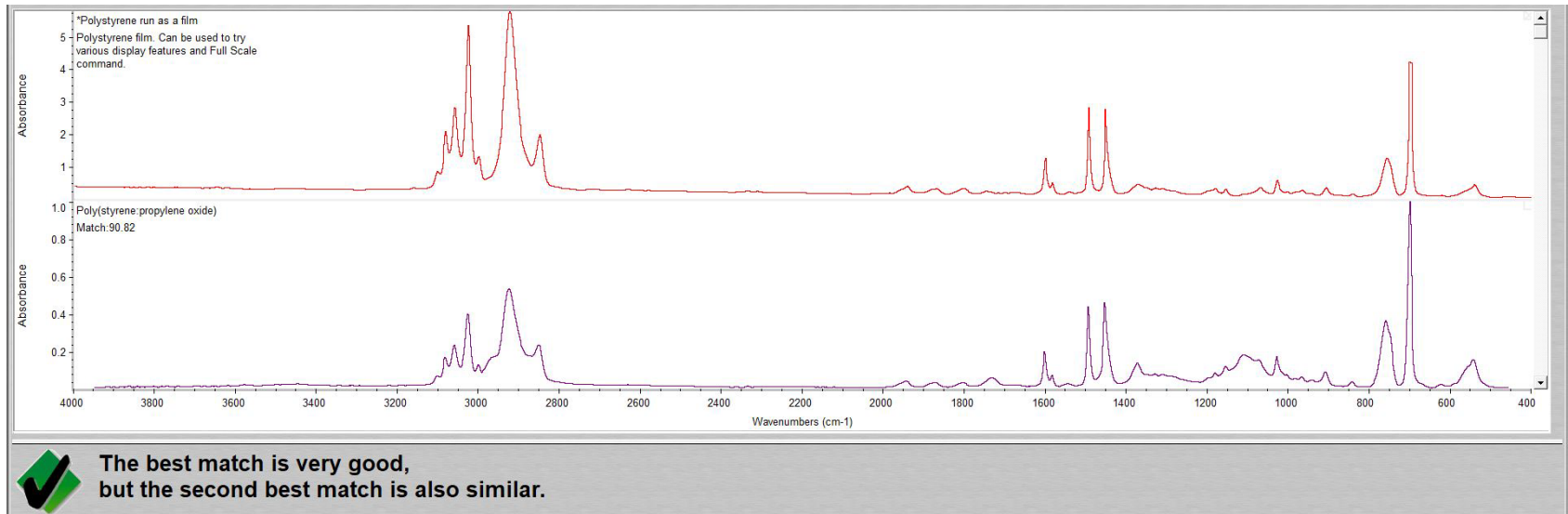


13. Click **Analyze** and
select **Search...**
or click **Search** icon



X. Library Search – 5/5

14. The ATR Correction may result in better matches



15. If a Match does not result, you will have to find matching spectra online instead

XI. Smart Transmission Accessory – 1/3

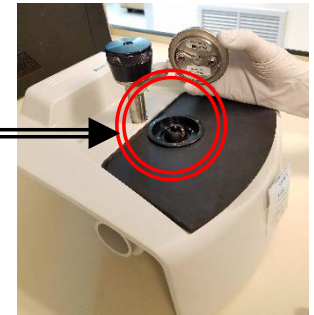
1. The **Smart ATR Accessory** is the default accessory installed



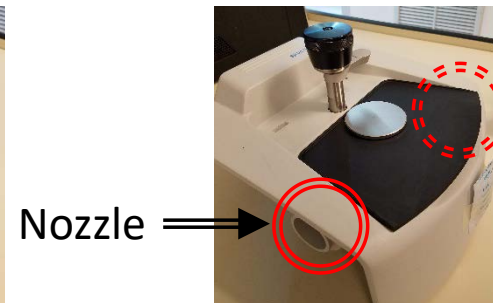
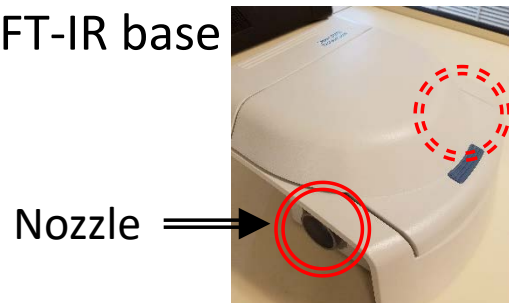
2. Please contact the Lab Manager if you need to use the **Smart Transmission Accessory** for Transmission FT-IR measurements



3. The **Smart ATR Accessory** contains mirrored optics that need to be carefully taken care to avoid damage and contamination

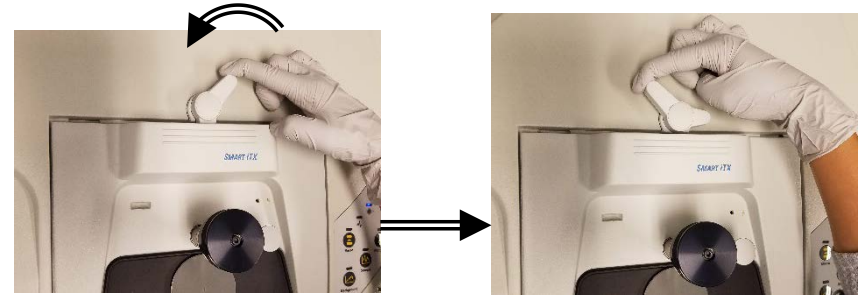


4. Both **Smart ATR Accessory** and **Smart Transmission Accessory** have nozzles to fit into slots of FT-IR base

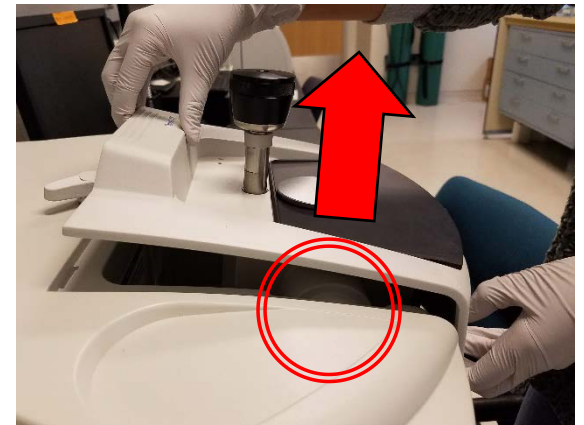


XI. Smart Transmission Accessory – 2/3

5. To remove the **Smart ATR Accessory**, move the lock to the **Unlocked** position



6. Carefully remove **Smart ATR Accessory** by gently pulling upward and position nozzles out of slots



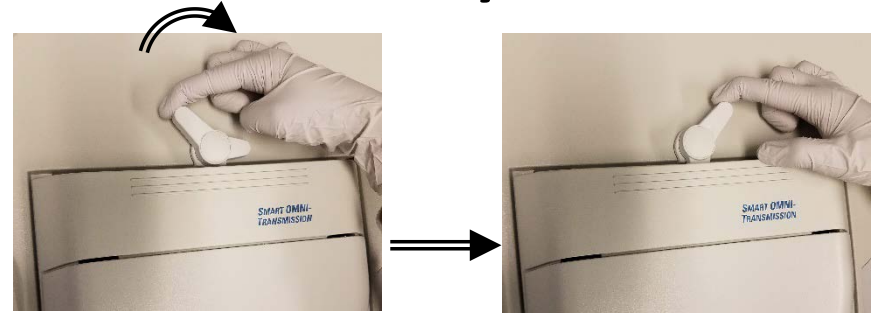
7. Carefully place aside and
KEEP AWAY FROM CONTAMINANTS!

8. Carefully insert the **Smart Transmission Accessory** by gently aligning the nozzles into the slots



XI. Smart Transmission Accessory – 3/3

9. Once firmly seated into the FT-IR base, move the lock to **Locked** position



10. Remember to remove **Smart Transmission Accessory** and reinsert the **Smart ATR Accessory** before leaving...

