FT-IR Training Notebook: ATR

Lab Manager: Dr. Perry Cheung

MSE Fee-For-Service Facility

Materials Science and Engineering

University of California, Riverside

January 25, 2021 (rev. 2.2)

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

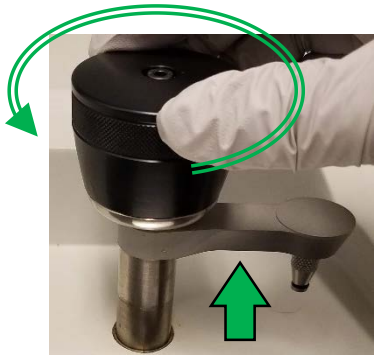
IX. Cleanup

X. Library Search

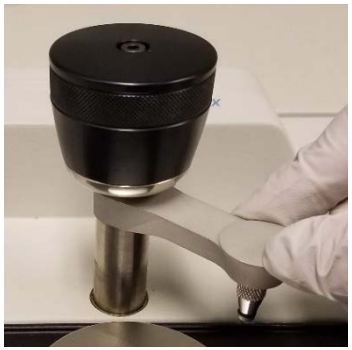
XI. Smart Transmission Accessory

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***

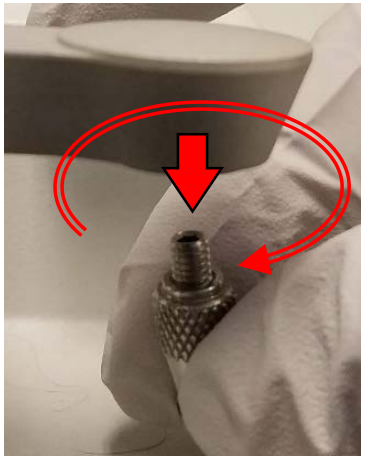


2. Inspect the ***Pressure Tip*** by moving ***Tower Arm*** to ***Cleaning Position***
   - Move ***Tower Arm*** to the right until it stops



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

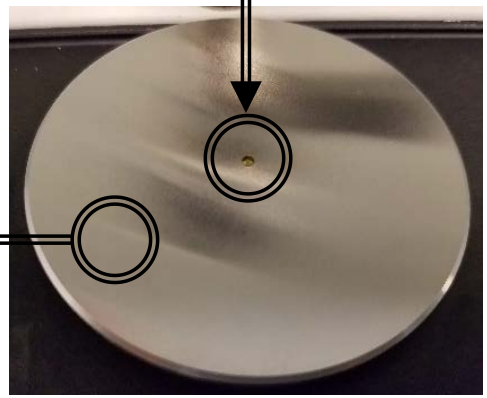
4. Identify appropriate ***Pressure Tip*** for your sample
   - ***Flat*** – for flat samples such as polymer films
   - ***Concave*** – for powders and curved surfaces
   - ***Volatiles Cover*** – for volatile liquids


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!



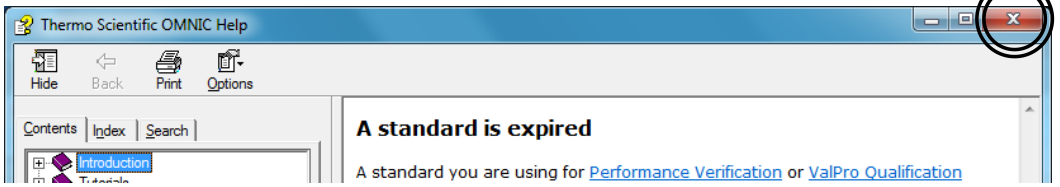
6. You may use ***Kim Wipes*** with ***Water*** and ***IPA*** to clean ***Metal Surface*** afterwards but avoid scratching the ***Diamond Crystal***

II. Initiate Software – 1/10

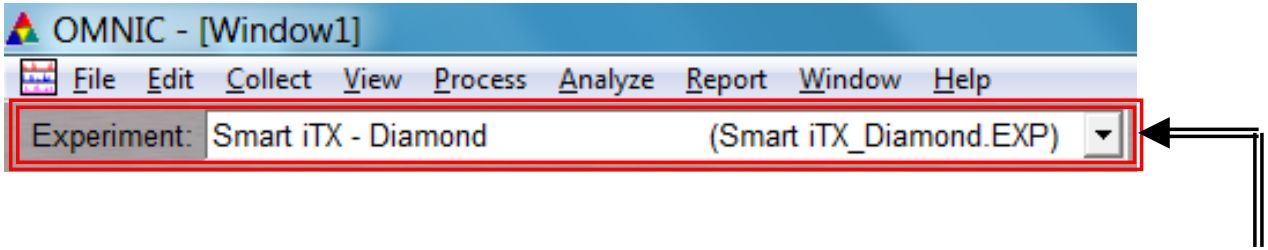
1. Double left-click on the ***OMNIC software icon*** for FT-IR

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

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

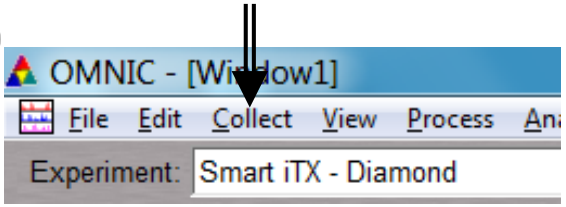


4. The ***OMNIC main window*** will now appear



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

II. Initiate Software – 2/10

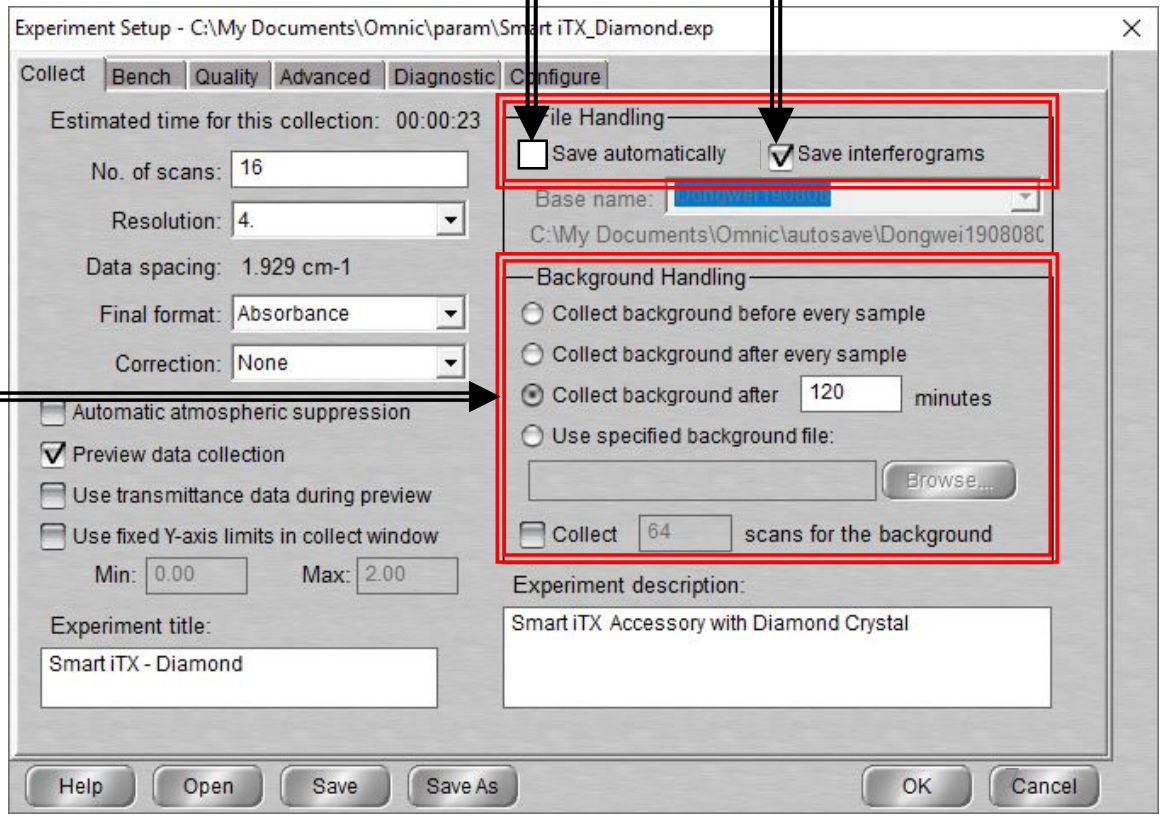


6. Select ***Collect -> Experiment Setup*** at the top window

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



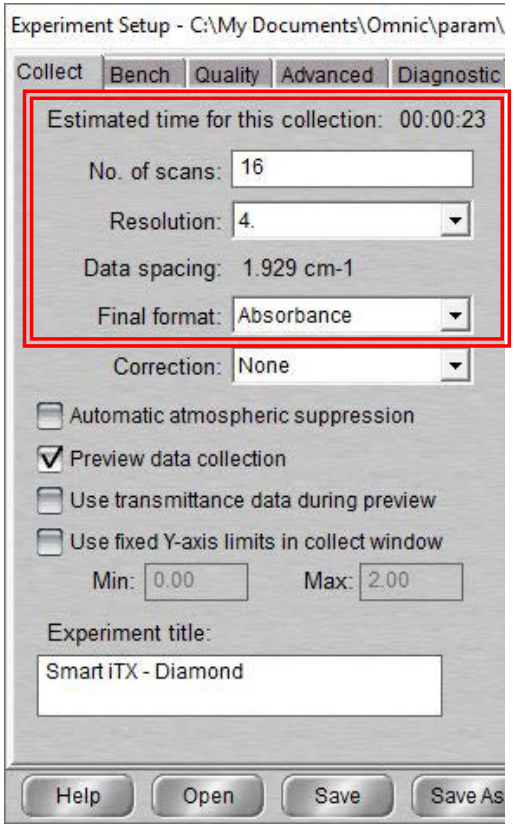
Note: A new background will be requested if there is a change in resolution or data spacing of your sample spectrum!

II. Initiate Software – 3/10

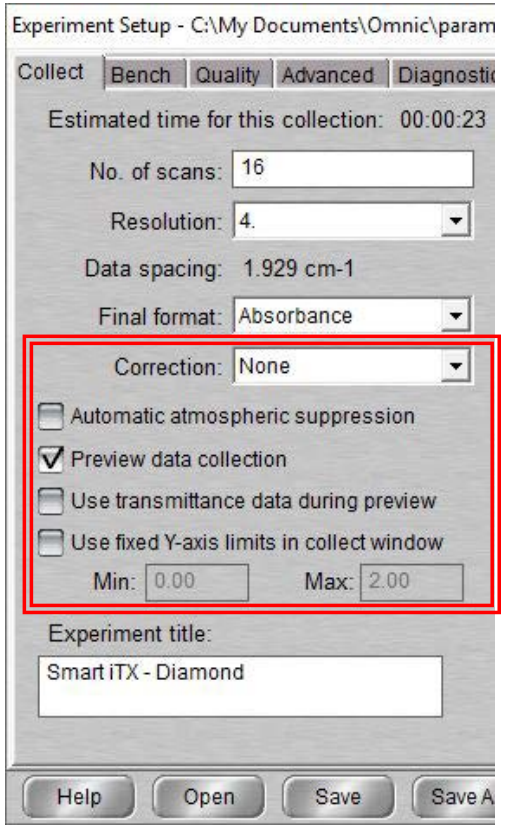
10. Select desired ***No. of scans*** – recommend starting at 16 scans
    - Increase to optimize desired spectrum signal/noise
    - Recommend increments of powers of 4 (e.g. 16, 64, 256, 1024,…)
11. Select desired ***Resolution value –*** recommend 8 or 4
    - Decrease value to increase spectrum resolution
    - 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



II. Initiate Software – 4/10



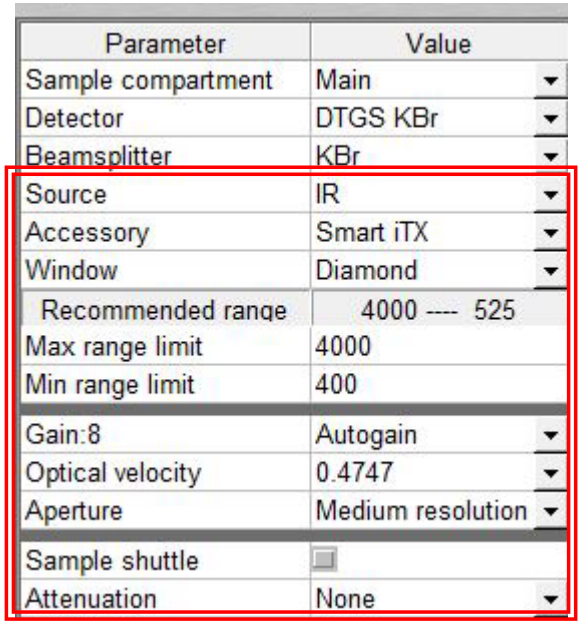
14. Select desired ***Correction type*** to ***None***
15. Decide if ***Automatic atmospheric suppression*** is desired
    - Effects of water vapor and carbon dioxide will be automatically suppressed via quantitative model

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 ***Attenuation*** is set to ***None***

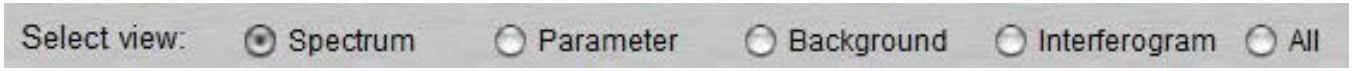


Parameter	Value
Sample compartment	Main
Detector	DTGS KBr
Beamsplitter	KBr
Source	IR
Accessory	Smart iTX
Window	Diamond
Recommended range	4000 ---- 525
Max range limit	4000
Min range limit	400
Gain:8	Autogain
Optical velocity	0.4747
Aperture	Medium resolution
Sample shuttle	
Attenuation	None

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%***
    - ***CO_2 levels*** – checks for CO_2 absorption, recommend ***ON*** at ***50%***
    - ***H_2O levels*** – checks for H_2O 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***
    - ***CO_2 levels*** – checks for CO_2 absorption, recommend ***ON*** at ***50%***
    - ***H_2O levels*** – checks for H_2O 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

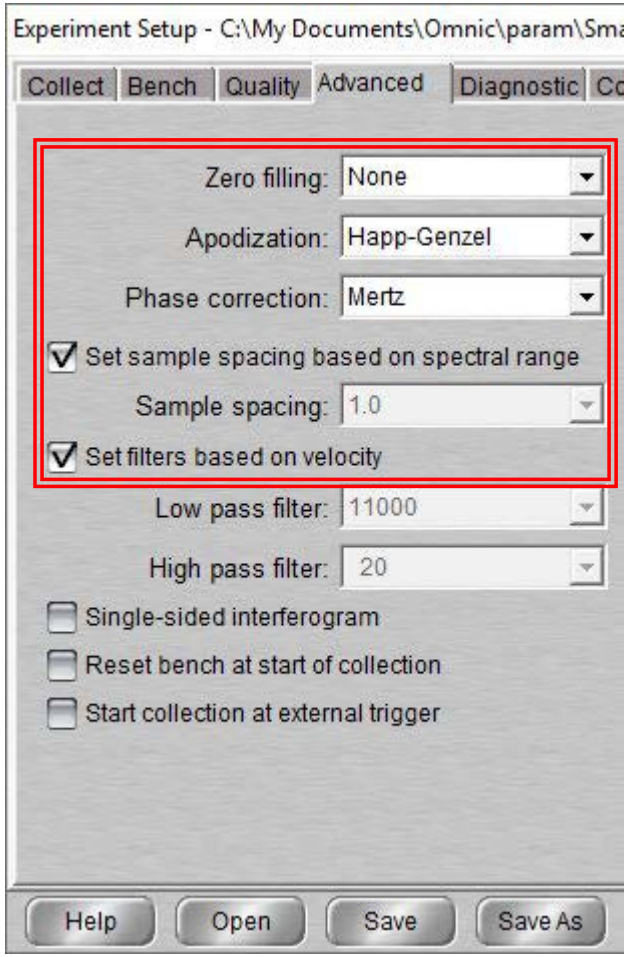
31. Select ***Advanced*** tab

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

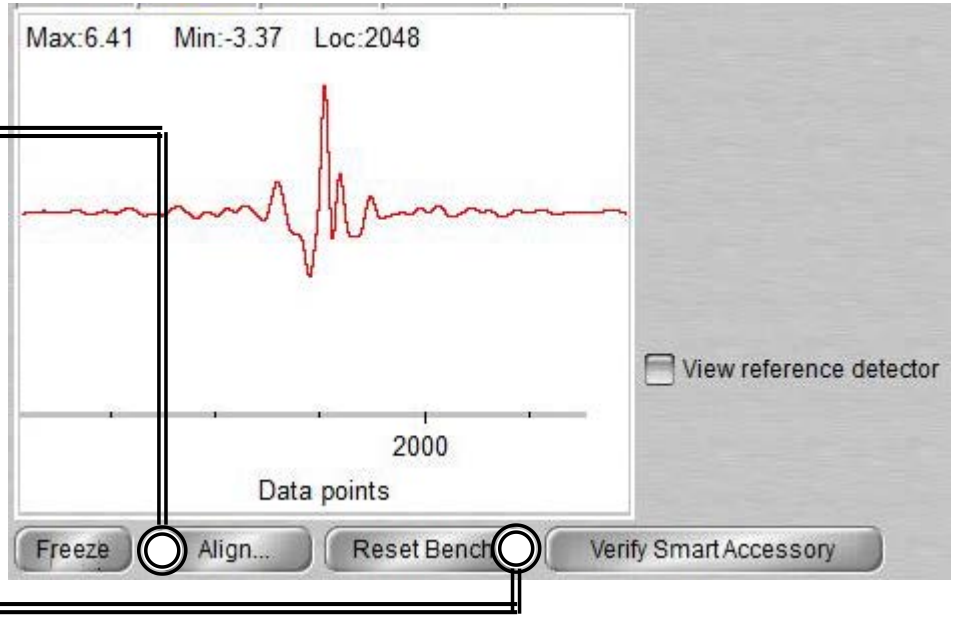
36. Select ***Diagnostic*** tab

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

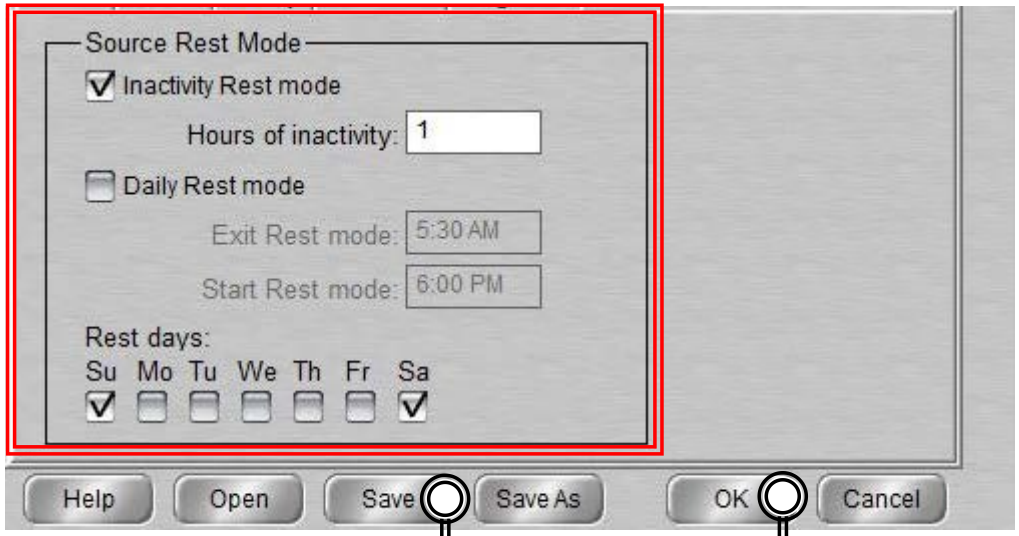


II. Initiate Software – 10/10

40. Select ***Configure*** tab

41. Confirm ***Inactivity Rest mode*** is checked
    - Confirm ***Hours of inactivity*** is set to "***1***" hour

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



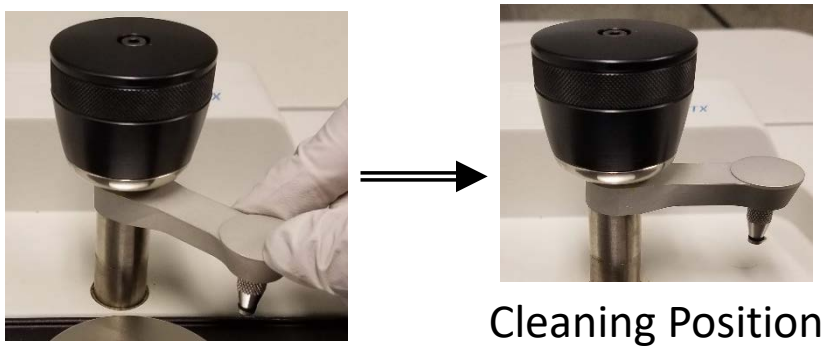
43. Click "***Save***" then "***OK***"

III. Collect Background – 1/2

1. It is critical that the ***Crystal*** is cleaned BEFORE ***Background*** is collected!

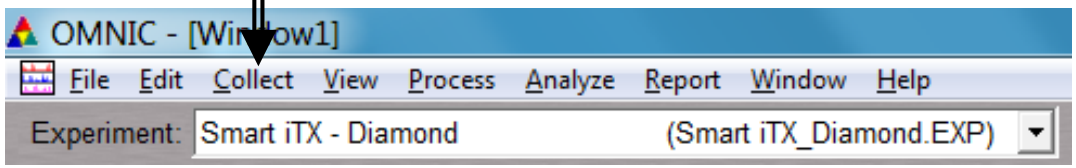
2. A single ***Background*** can be used to analyze multiple samples, but it is recommended to collect new ***Background*** at least every 2 hours

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

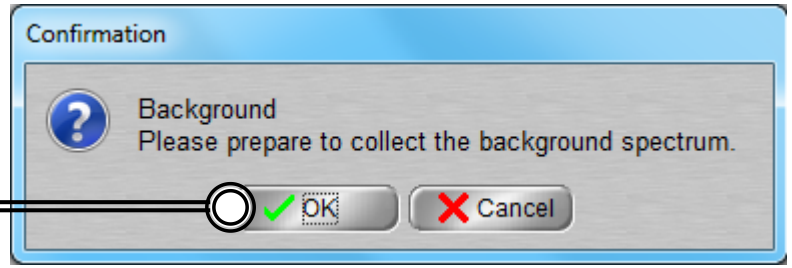


Cleaning Position

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

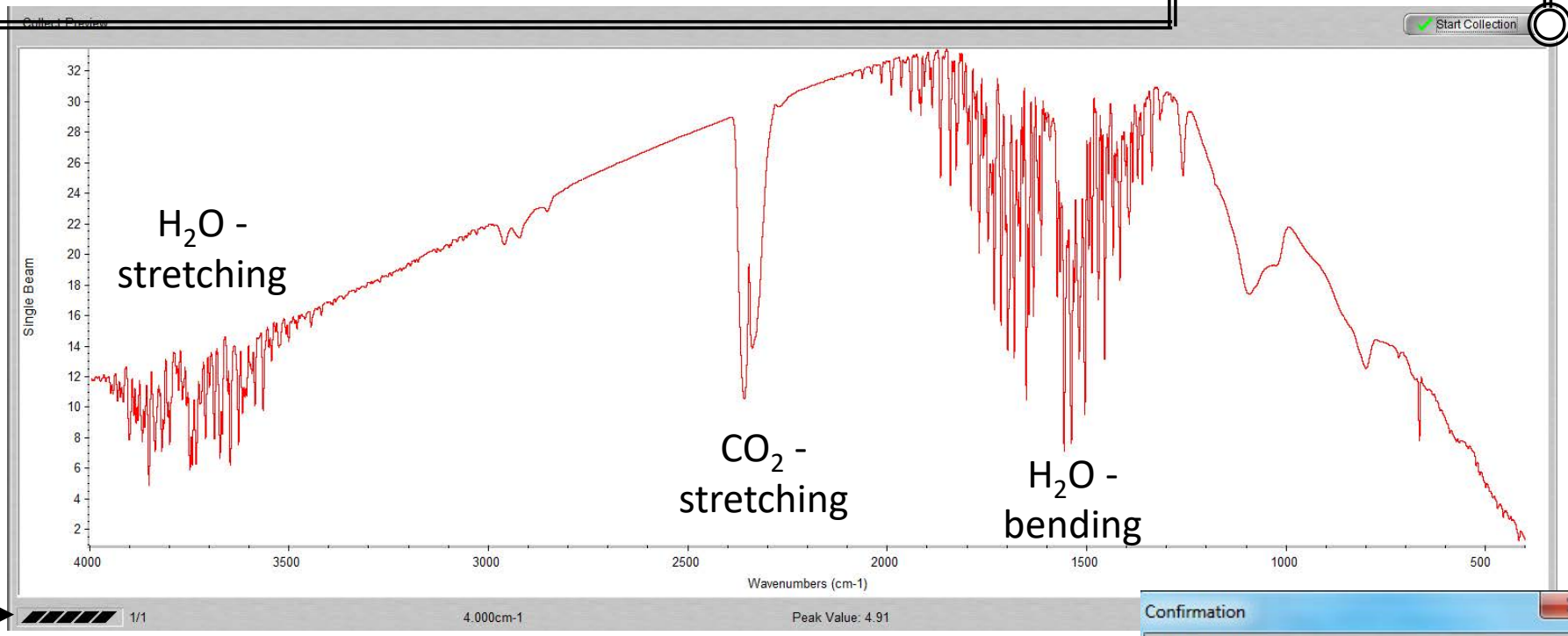


5. Confirm to collect background by clicking ***OK***

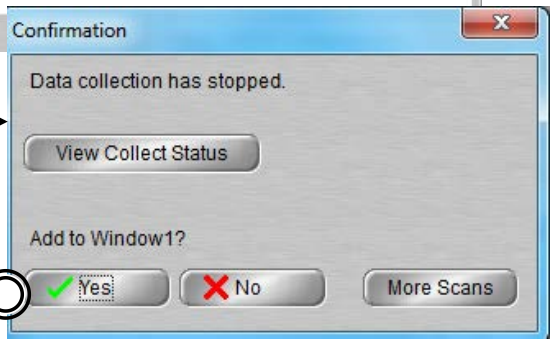


III. Collect Background – 2/2

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



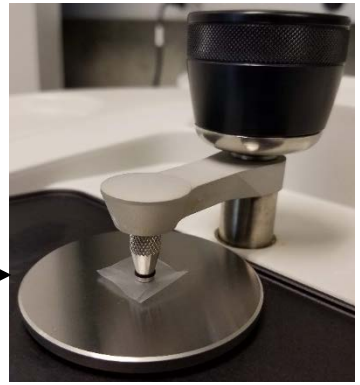
IV. Sample Prep: Solids – 1/1

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***

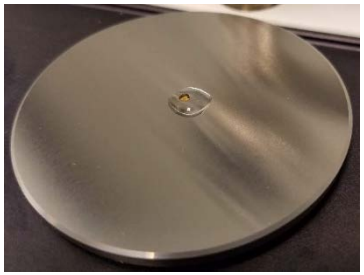
6. The ***Pressure Tower Knob*** will ***Click*** and ***Freely Rotate*** when the maximum pressure is reached

V. Sample Prep: Liquids – 1/1

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

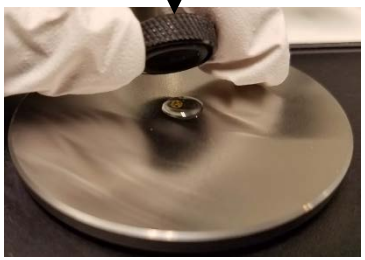
Cleaning Position

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



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

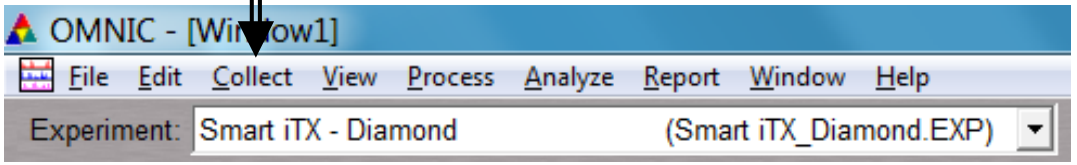
4. For ***highly volatile samples***, place ***Volatiles Cover*** over sample to reduce of evaporation



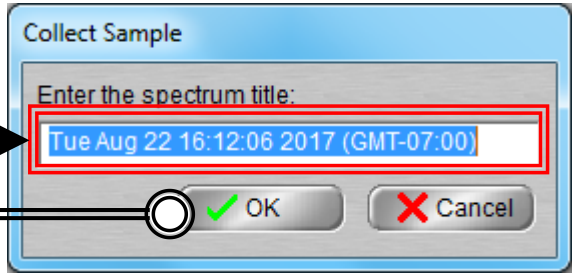
- Install ***Flat Pressure Tip***, move into ***Sampling Position***, and lower the ***Pressure Tower*** until the ***Pressure Tower Knob Clicks*** and ***Freely Rotates*** when the maximum pressure is reached

VI. Collect Sample – 1/2

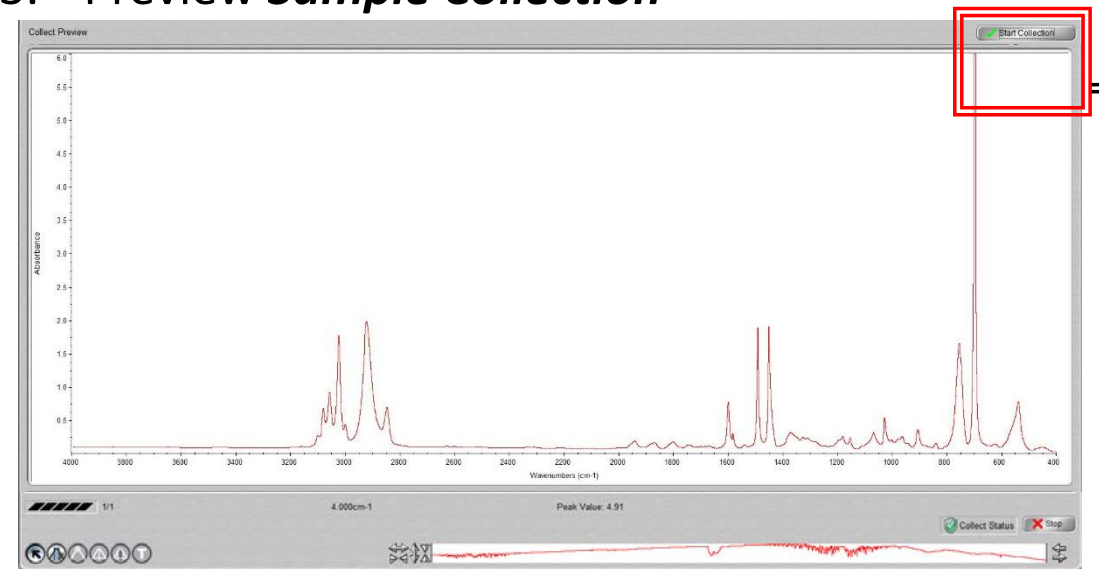
1. Select ***Collect -> Collect Sample***



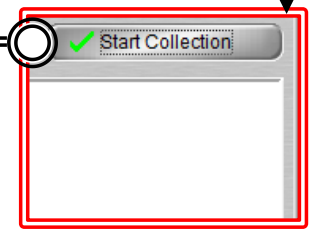
2. Enter ***Spectrum Title*** and click ***OK*** to ***Collect Sample***



3. Preview ***Sample Collection***

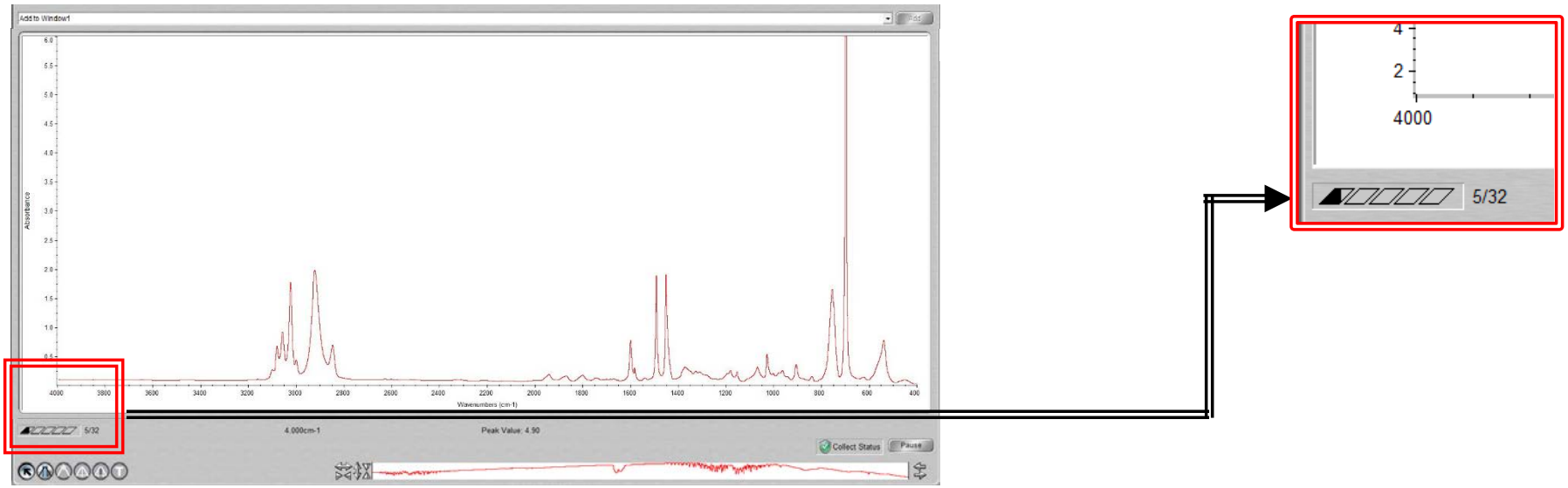


4. Click ***Start Collection*** to begin ***Sample Collection***



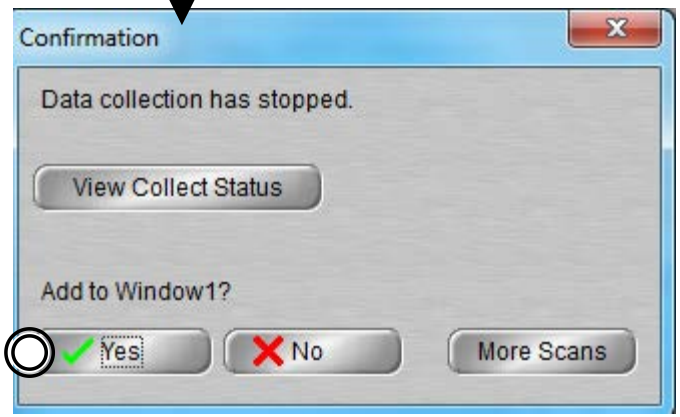
VI. Collect Sample – 2/2

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

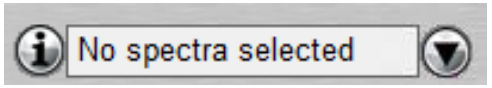


6. Confirmation of ***Data Collection*** will be shown

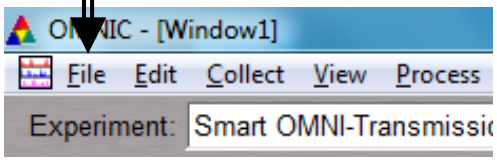
7. Click ***Yes*** to add data to current Window



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)



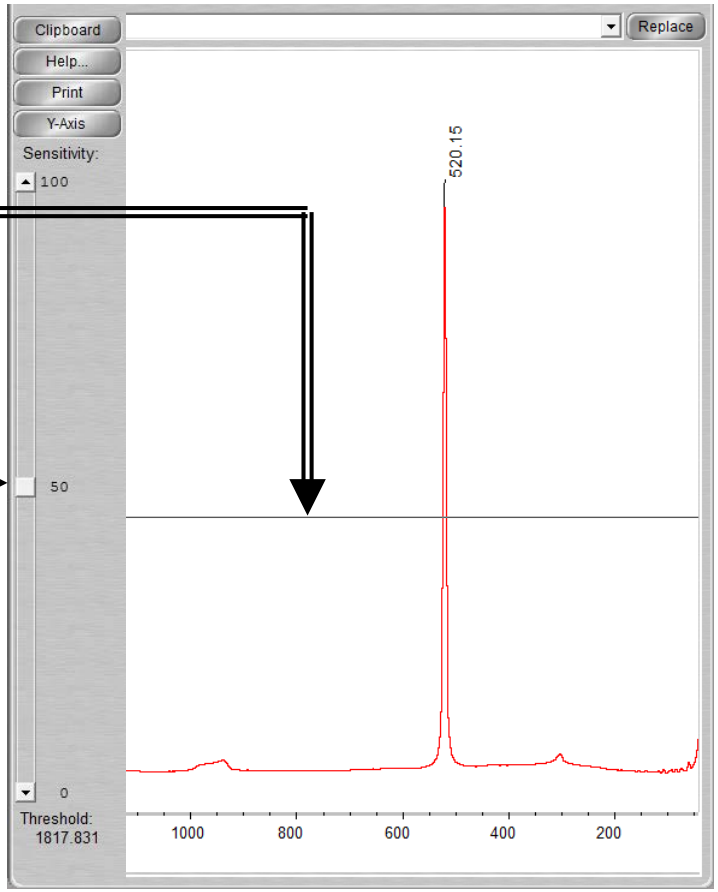
5. Click ***File -> Save Group*** to save more than one spectrum as a group in one file having file extension .SPG to open later

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

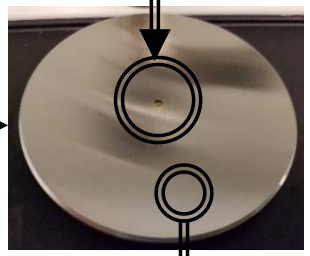
3. Adjust the ***Sensitivity*** button to separate peaks from noise



IX. Cleanup – 1/1

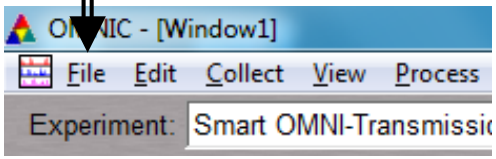
1. Remove ***Sample*** from the ***Crystal*** without scratching the ***Crystal***

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

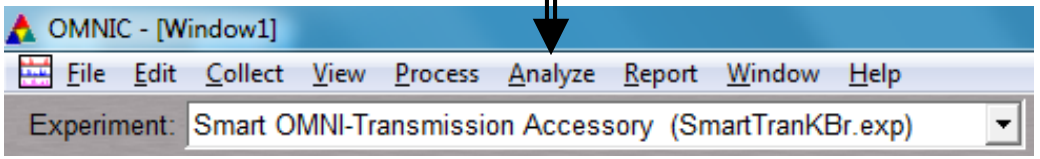
4. Click on ***File -> Exit*** to shut down the software



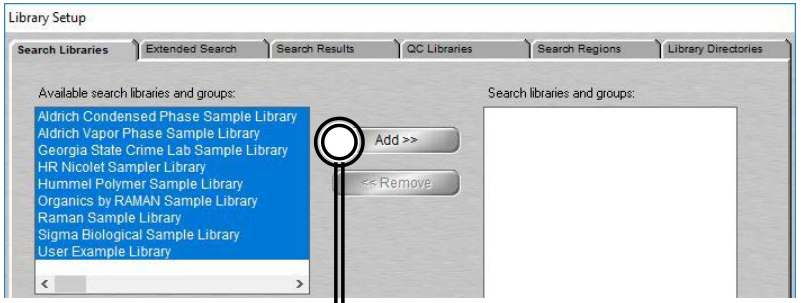
5. Log off of your ENGR account

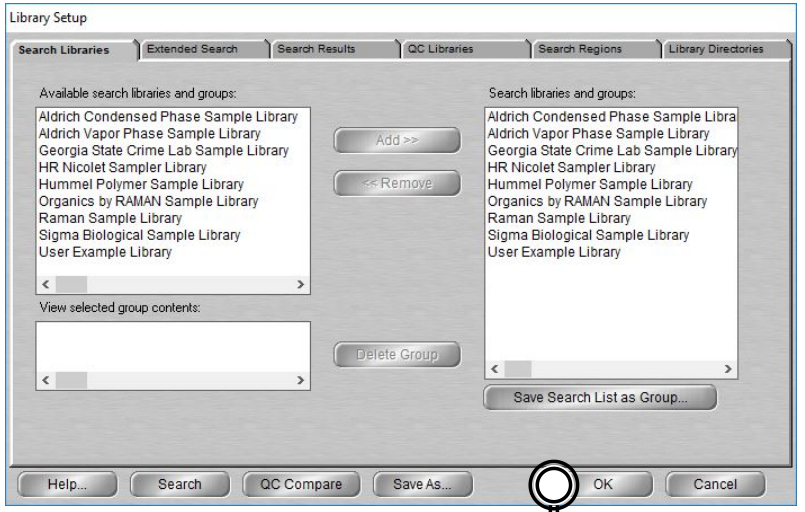
X. Library Search – 1/5

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



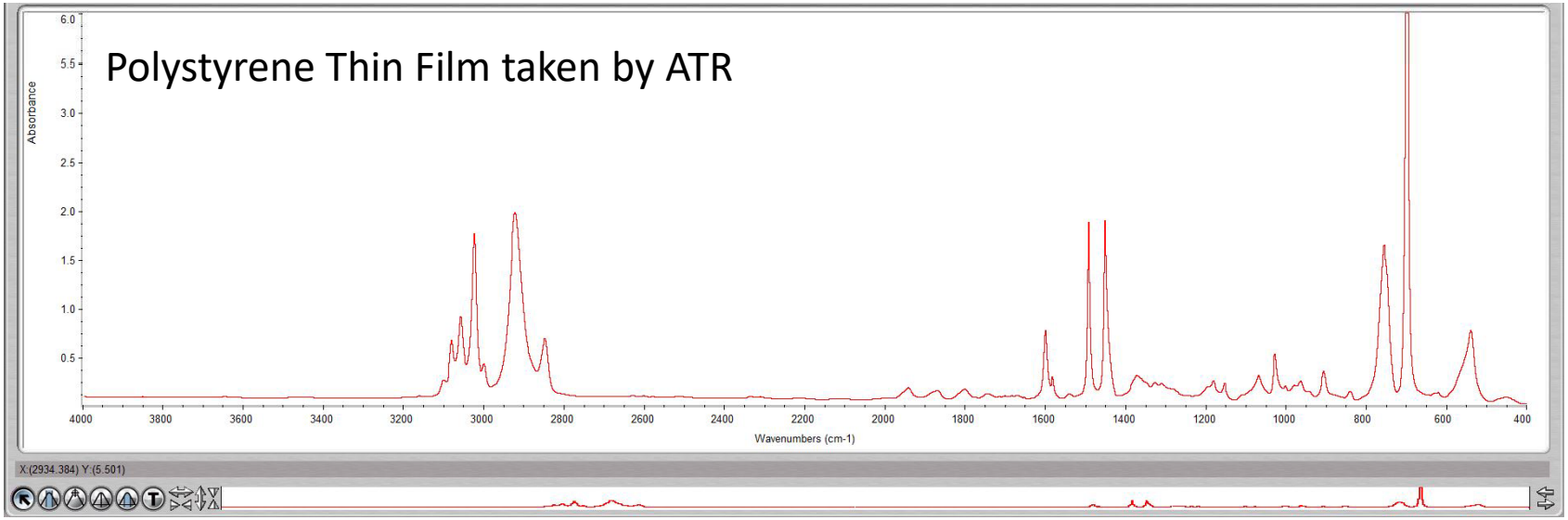
2. Select desired ***Libraries*** or select all
3. Click ***Add >>***
4. Click ***OK***



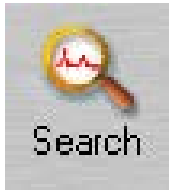


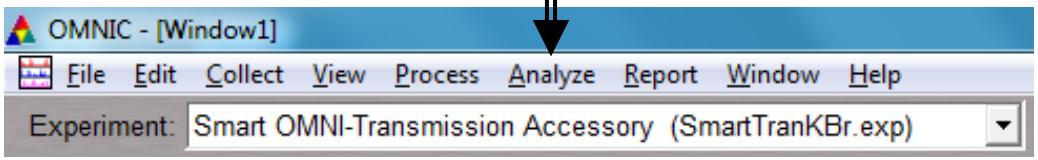
X. Library Search – 2/5

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



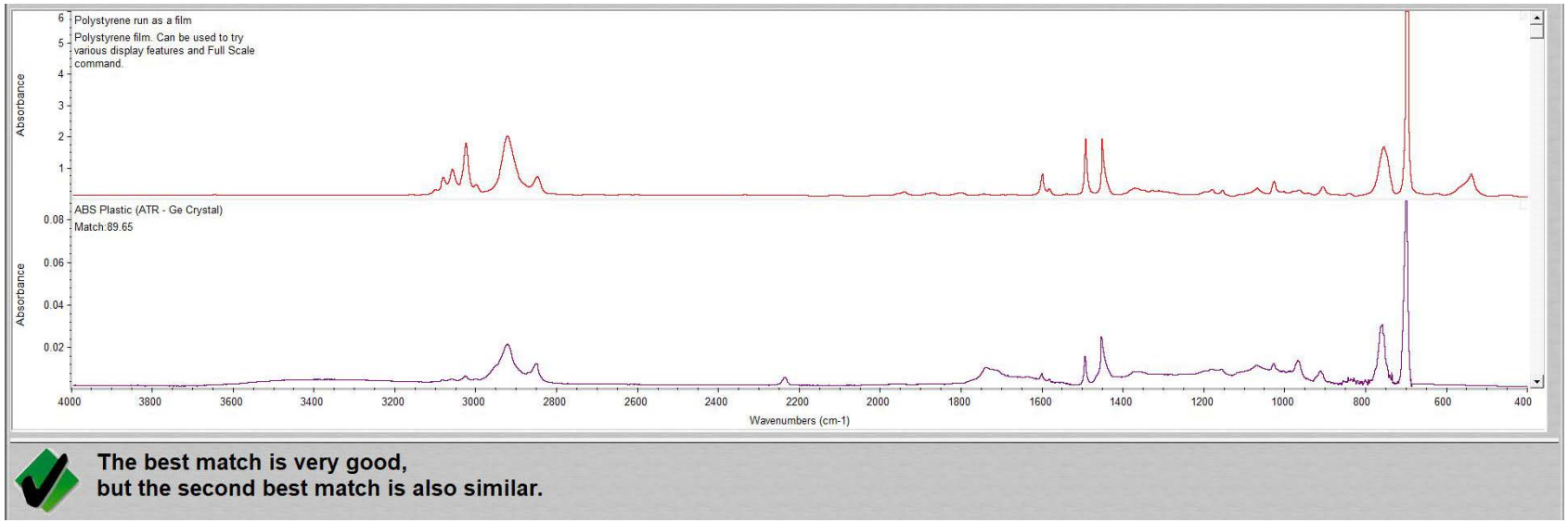
6. Click ***Analyze*** and select ***Search…***
   or click ***Search*** icon



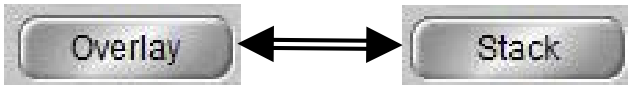


X. Library Search – 3/5

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

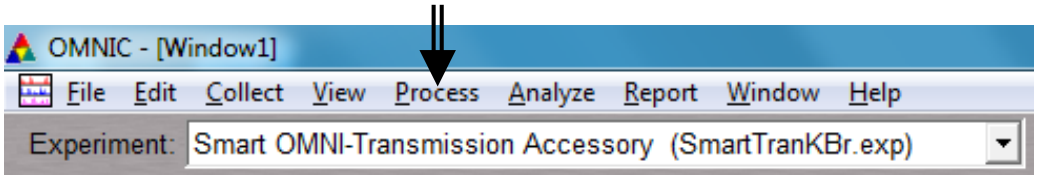


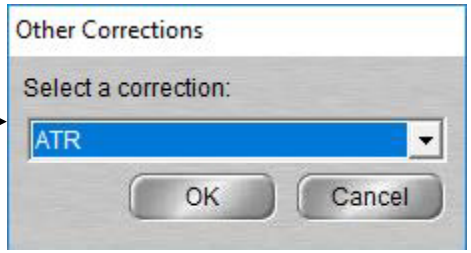
8. Click ***View Match List*** and select either ***Overlay*** or ***Stack*** view



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

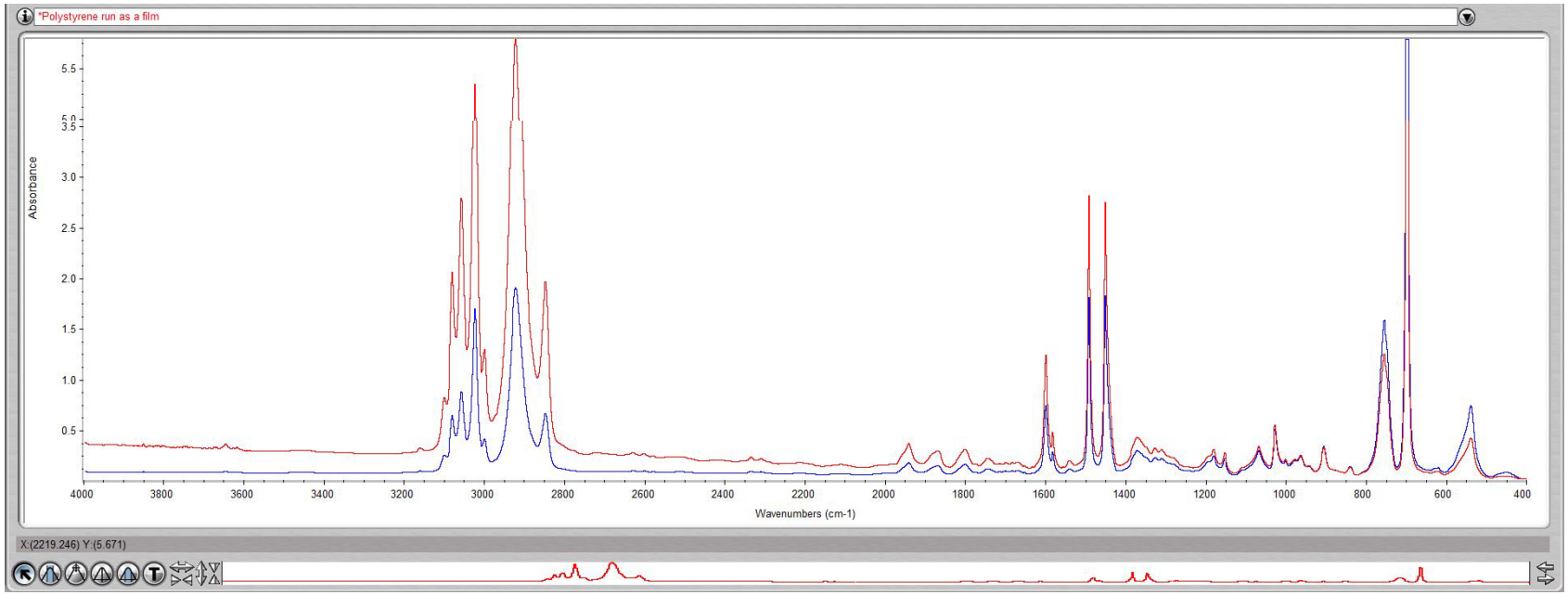
10. Click ***Process > Other Corrections…*** and select ***ATR***



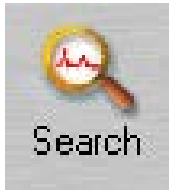


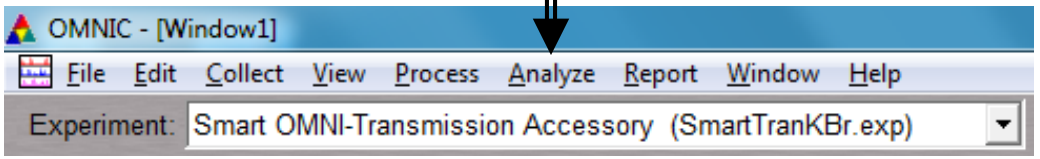
X. Library Search – 4/5

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



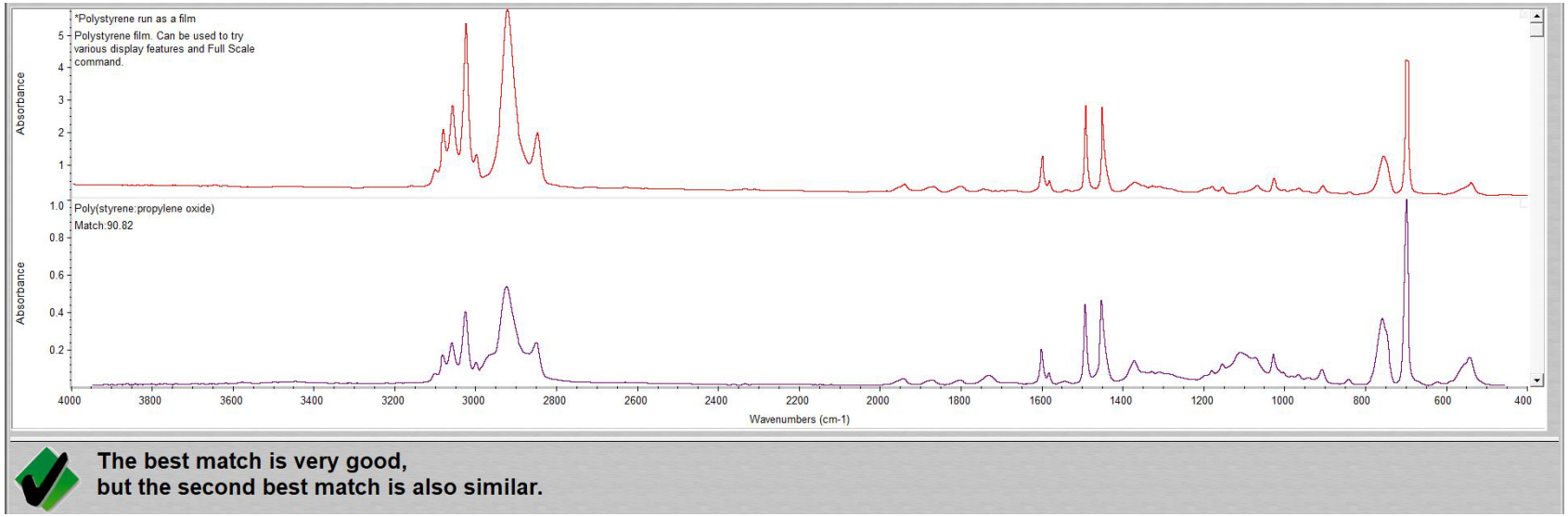
12. Click ***Analyze*** and select ***Search…*** or click ***Search*** icon





X. Library Search – 5/5

13. The ATR Correction may result in better matches



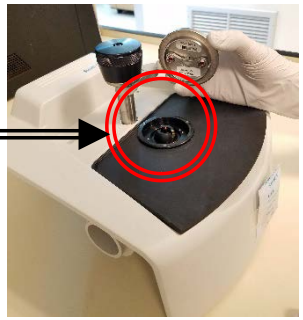
14. If a Match does not result, you will have to find matching spectra online instead

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

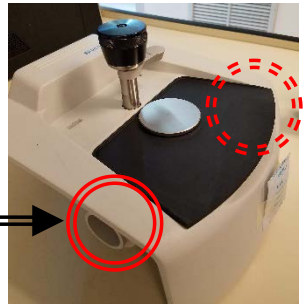
3. The ***Smart ATR Accessory*** contains mirrored optics that need to be carefully taken care to avoid damage and contamination

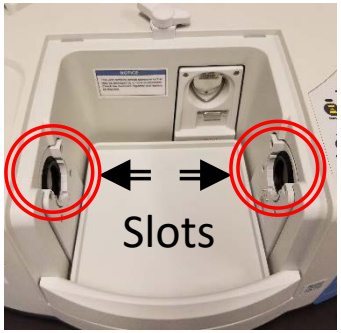


4. Both ***Smart ATR Accessory*** and ***Smart Transmission Accessory*** have nozzles to fit into slots of FT-IR base

Nozzle

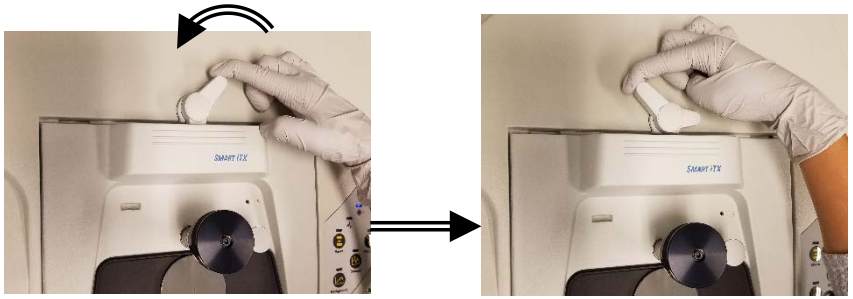
Nozzle



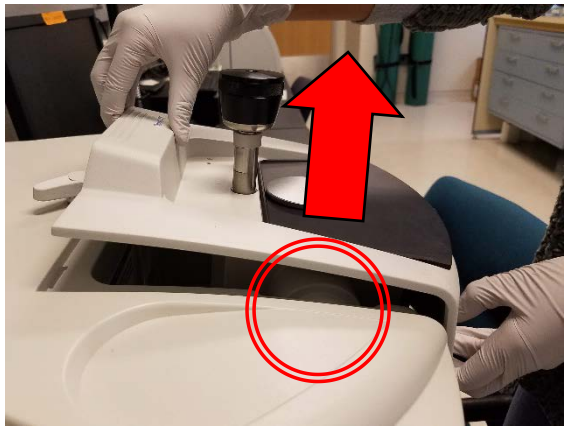


XI. Smart Transmission Accessory – 2/3

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

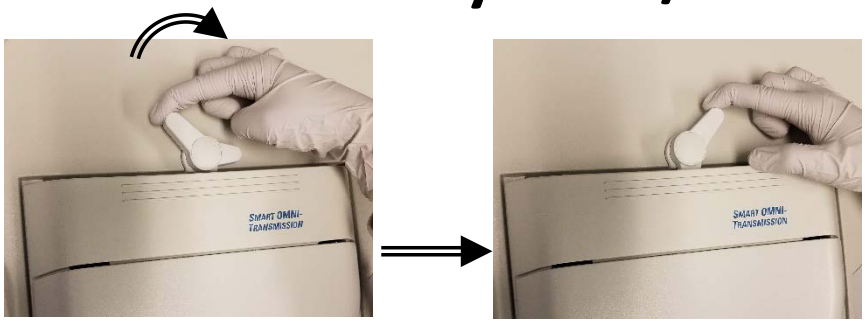


7. Carefully place aside and KEEP AWAY FROM CONTAMINANTS!

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…