FT-IR Training Notebook: ATR

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MSE Fee-For-Service Facility

Materials Science and Engineering

University of California, Riverside

October 31, 2018 (rev. 1)

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

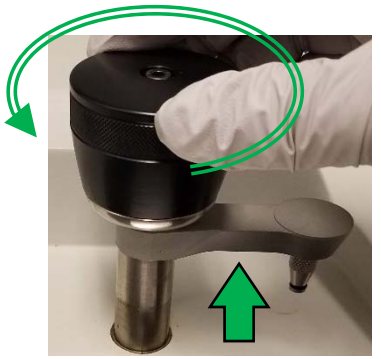
VIII. Peak Identification

IX. Cleanup

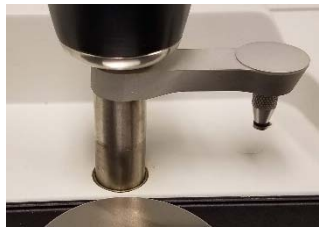
X. Library Search

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***

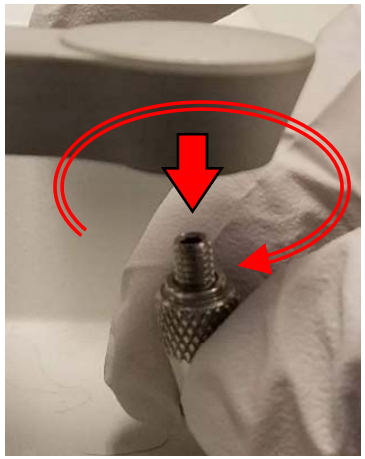


2. Inspect the ***Pressure Tip*** by moving ***Tower Arm*** to ***Cleaning Position***
   - Move ***Tower Arm*** to the right until it stops



Cleaning Position

3. Clean the ***Pressure Tip*** (remove if necessary) with appropriate solvent
   - Recommend ***Water*** then ***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

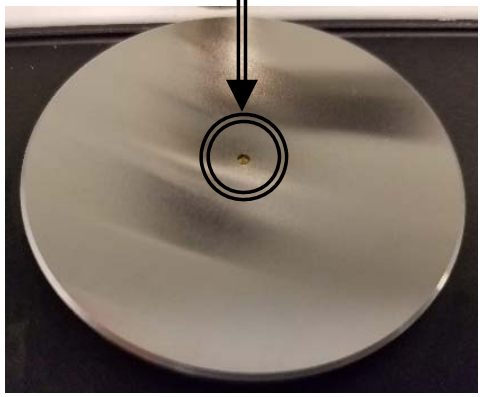
4. Identify appropriate ***Pressure Tip*** for your sample
   - ***Flat*** – for thin samples such as polymer films
   - ***Concave*** – for powders and curved surfaces
   - ***Volatiles Cover*** – for volatile liquids


Flat


Concave


Cover

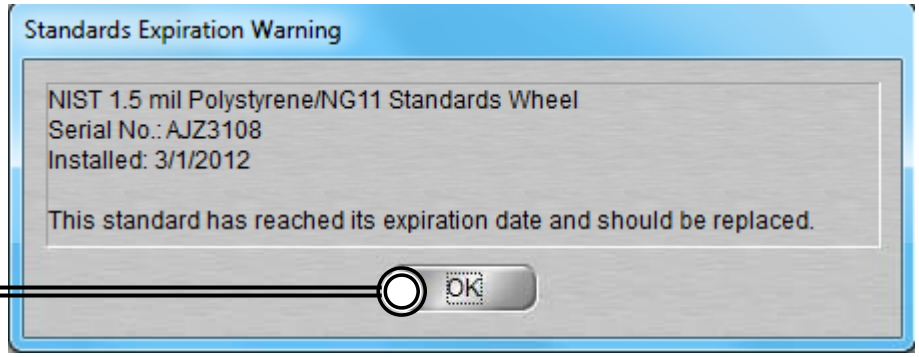
5. Use provided ***Q-Tips*** and appropriate solvent to clean the ***Diamond Crystal***
   - Recommend ***Water*** then ***IPA***
   - DO NOT USE ACETONE!
   - DO NOT USE KIM WIPES!



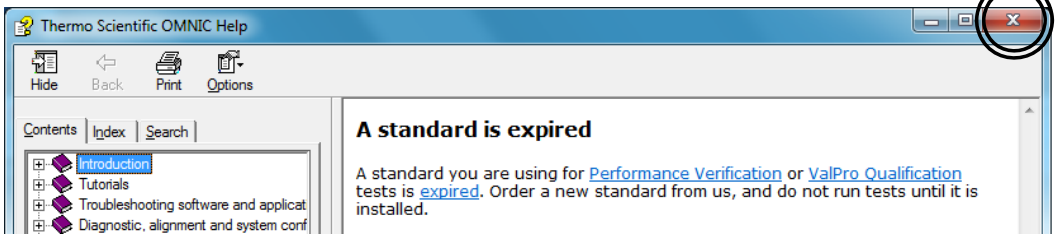
II. Initiate Software – 1/3

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

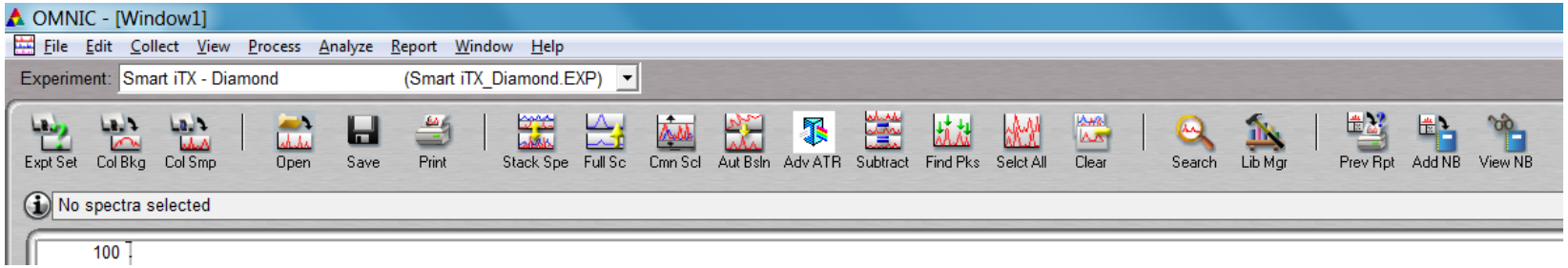
2. Ignore the ***Standards Expiration Warning*** and click ***OK***



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

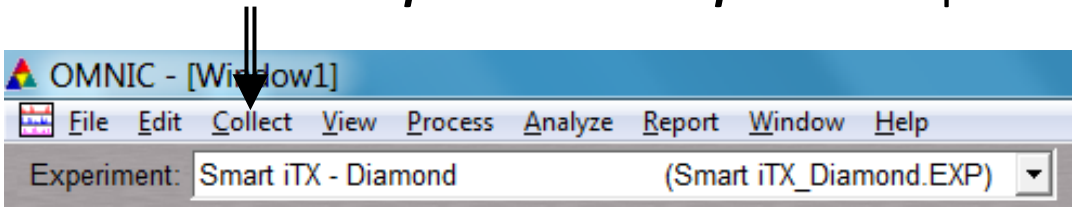


4. The ***OMNIC main window*** will now appear



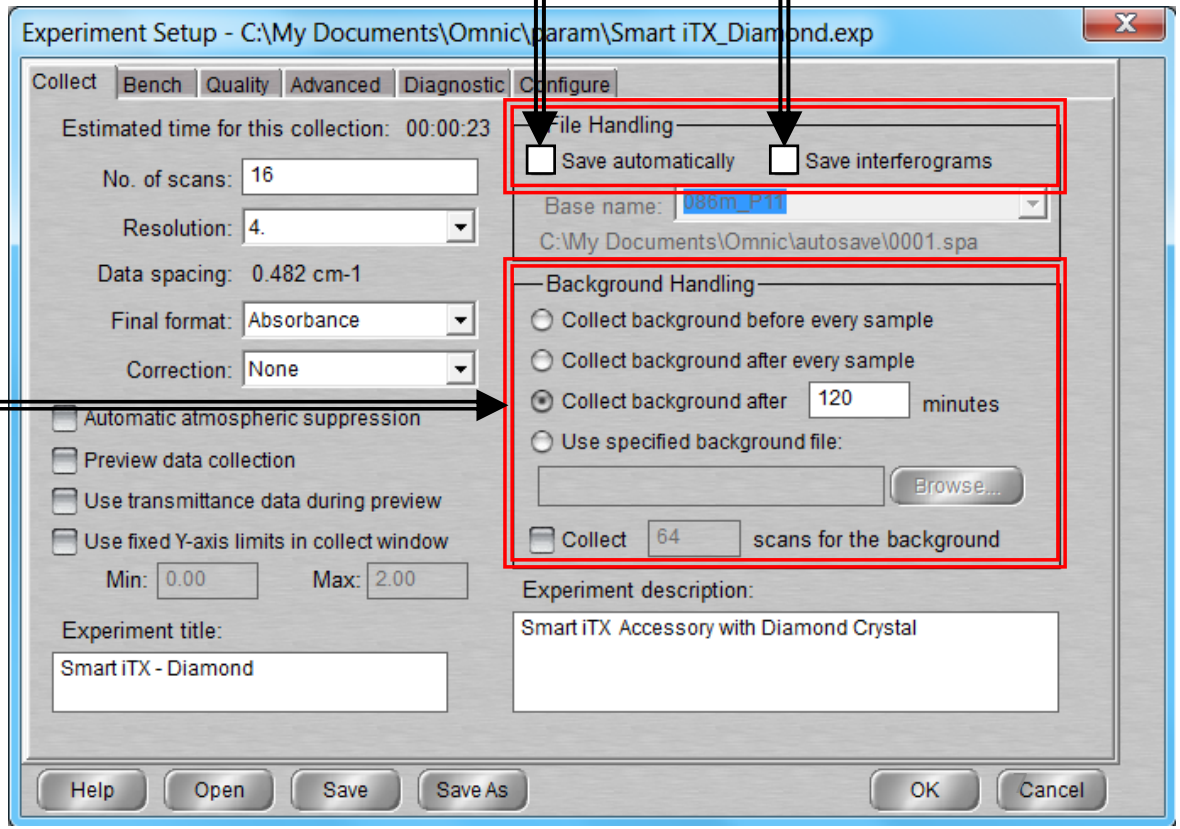
II. Initiate Software – 2/3

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



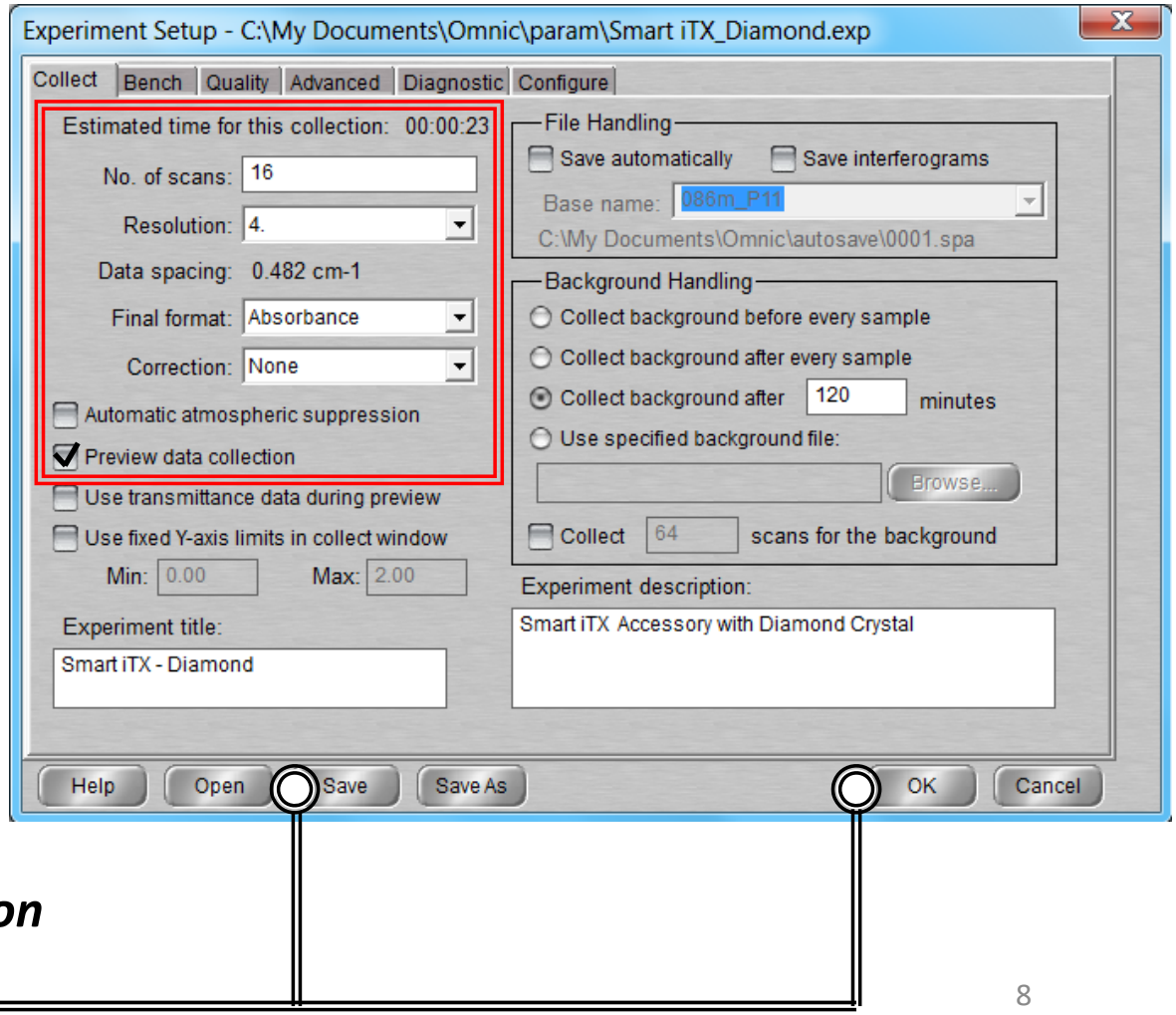
6. **Uncheck** both the ***Save automatically*** and ***Save interferograms*** under ***File Handling***

7. Set preferred ***Background Handling*** settings
   - Before every sample
   - After every sample
   - After 120 minutes (default)
   - Use specific file



II. Initiate Software – 3/3

5. Select desired ***No. of scans*** – recommend powers of 4 (4, 16, 64, 256, 1024,…)
6. Check ***Estimated time for collection***
7. Select desired ***Resolution***
   - Recommend ***4***
8. Select desired ***Final format***
   - *% Transmittance*
   - *Absorbance*
   - *Etc…*
9. Select desired ***Correction***
   - *None (default)*
   - *Kramers-Kronig*
   - *H2O*
   - *Etc…*
10. Check ***Preview data collection***
11. Click "***Save***" and "***OK***"

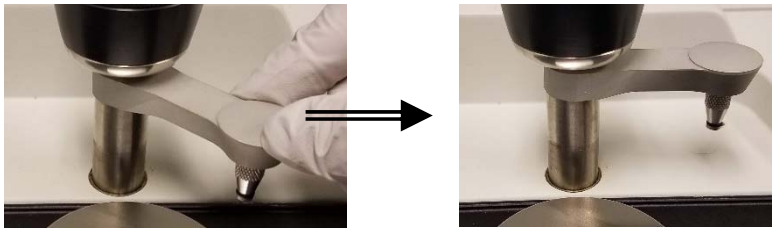


III. Collect Background – 1/2

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

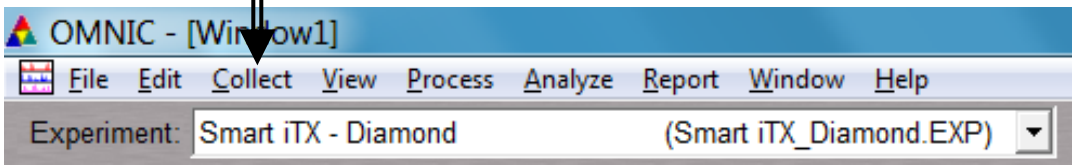
2. A single ***Background*** can be used to analyze multiple samples, but it is recommended to collect new ***Background*** at least every 2 hours

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



Cleaning Position

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



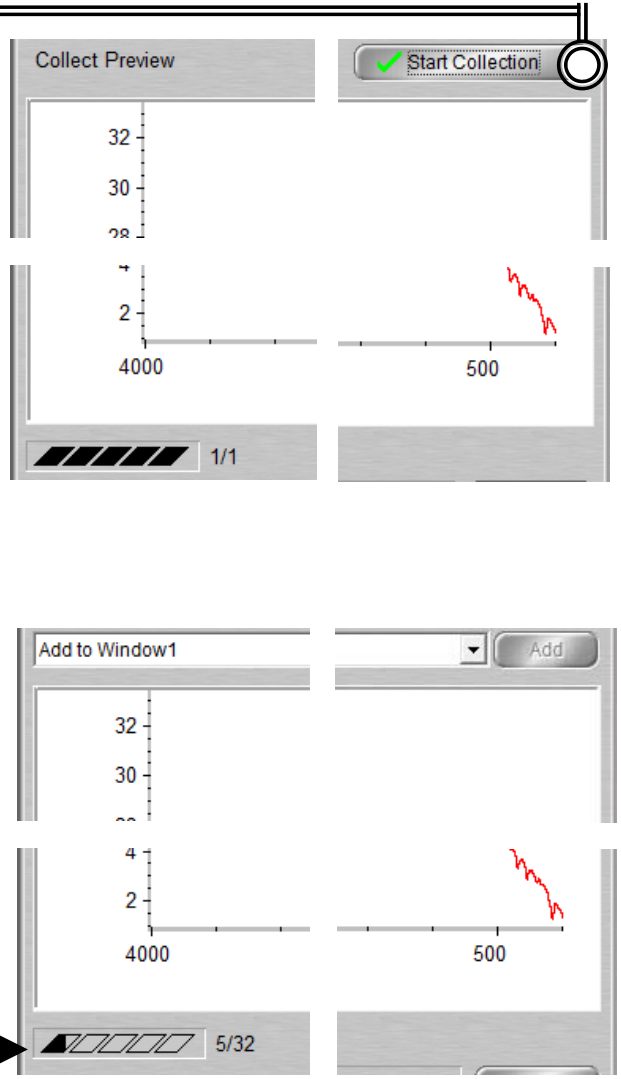
5. Confirm to collect background by clicking ***OK***

III. Collect Background – 2/2

6. Preview ***Background Collection***

7. Click ***Start Collection*** to begin ***Background Collection***

8. The ***Background Collection*** will begin with the progress shown at the bottom

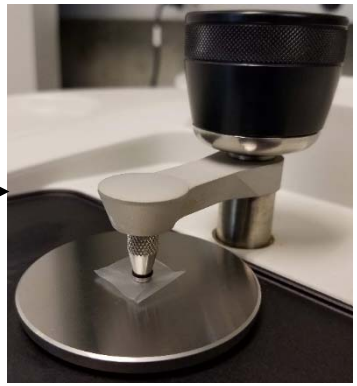


IV. Sample Prep: Solids – 1/1

1. For ***Solid***, ***Powder***, or ***Thin Films***

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

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



Sampling Position

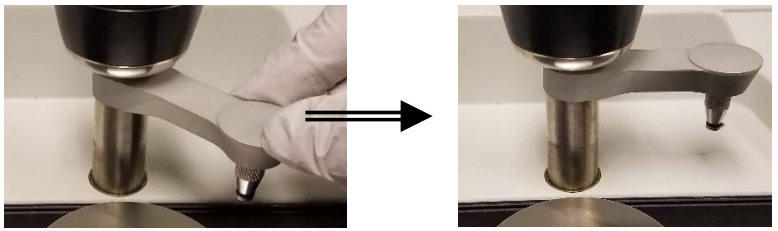
4. Place sample onto ***Crystal***, directly under ***Pressure Tip***

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

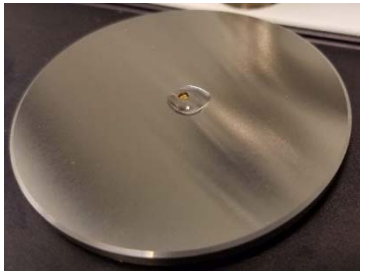
V. Sample Prep: Liquids – 1/1

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

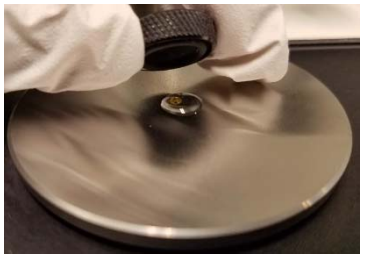


Cleaning Position

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



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

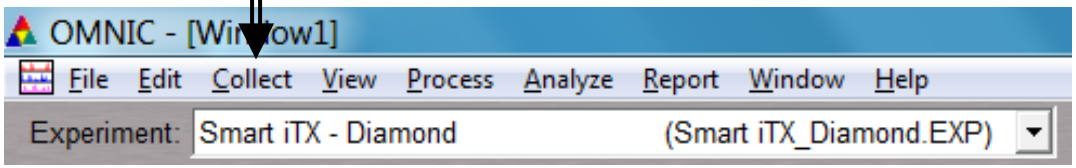


4. To reduce of evaporation place ***Volatiles Cover*** over sample

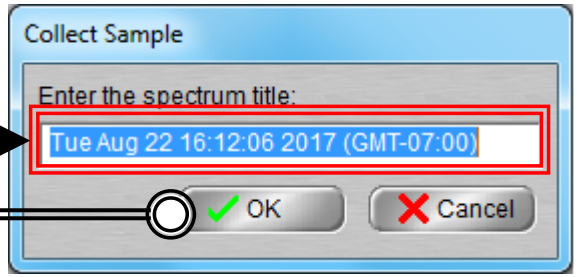
5. Install ***Flat Pressure Tip***, move into ***Sampling Position***, and lower the ***Pressure Tower*** until the ***Pressure Tower Knob Clicks*** and ***Freely Rotates*** when the maximum pressure is reached

VI. Collect Sample – 1/2

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

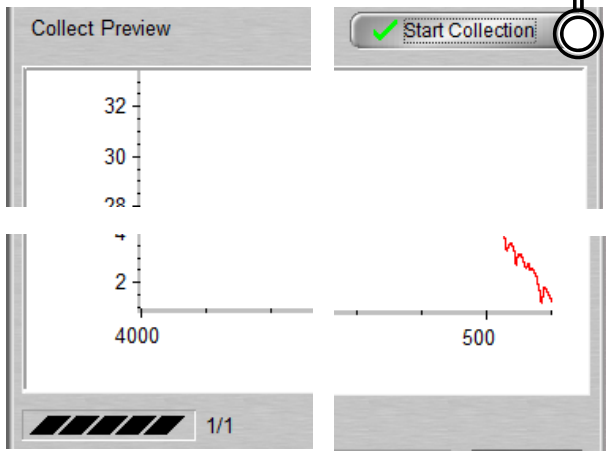


2. Enter ***Spectrum Title*** and click ***OK*** to ***Collect Sample***



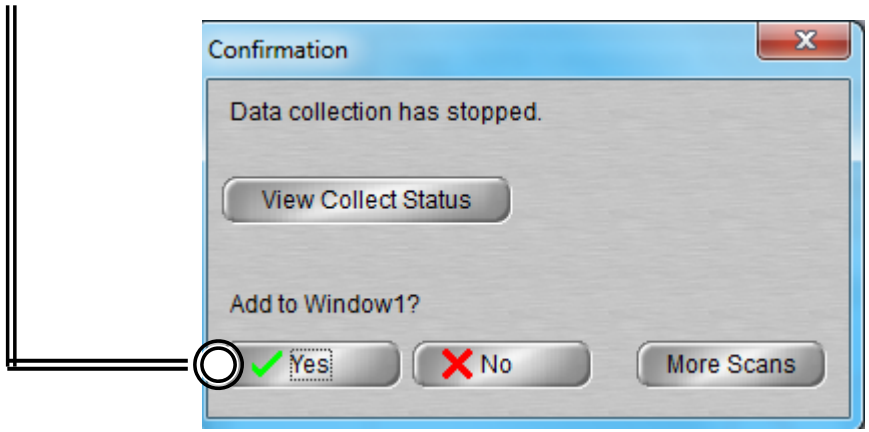
3. Preview ***Sample Collection***

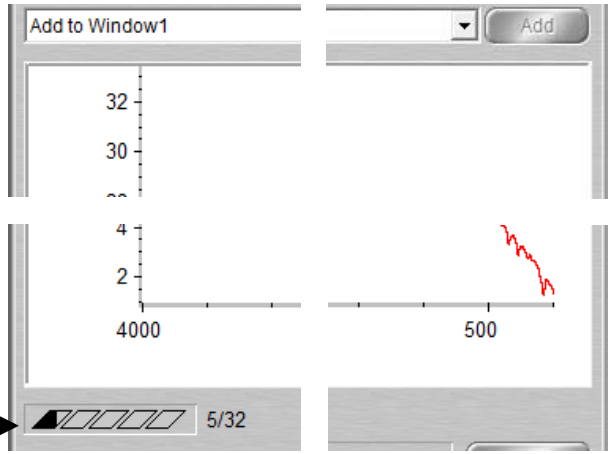
4. Click ***Start Collection*** to begin ***Sample Collection***



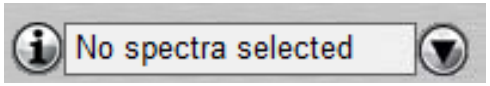
VI. Collect Sample – 2/2

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 to data to current Window

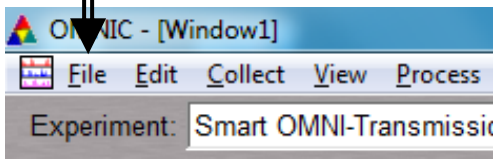




VII. Saving Data – 1/1

1. Specific spectra can be selected using the  selection tool at the bottom of window and clicking on it or selecting 

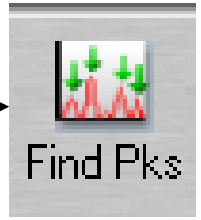
it from the dropdown box
2. Multiple spectra can be selected/deselected by holding down the ***Ctrl*** key and clicking spectra
3. Click ***File -> Save*** to save a spectrum (e.g. default is SPA) using the current filename
4. Click ***File -> Save As*** to save a spectrum into another file type (e.g. 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
6. Click ***File -> Save Current Background*** to a named file if desired for later referencing or processing (optional)

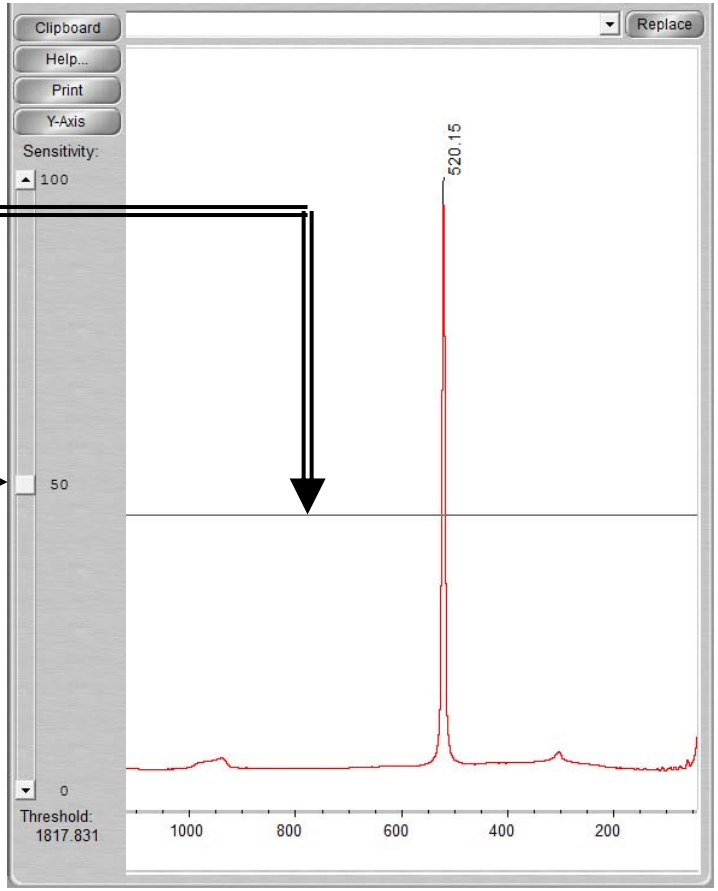
VIII. Peak Identification – 1/1

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

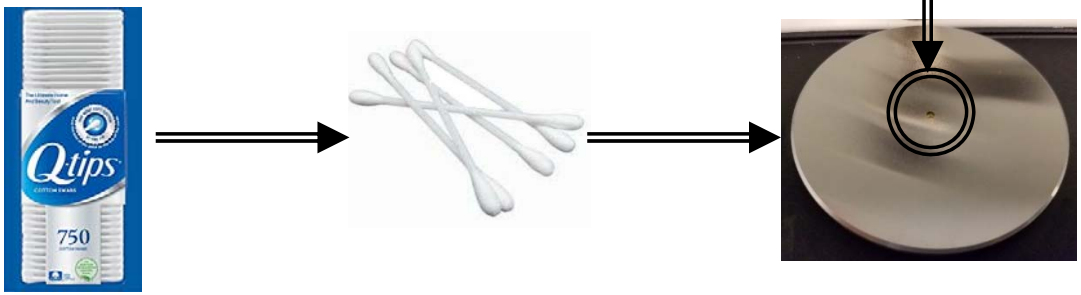
3. Adjust the ***Sensitivity*** button to separate peaks from noise



IX. Cleanup – 1/1

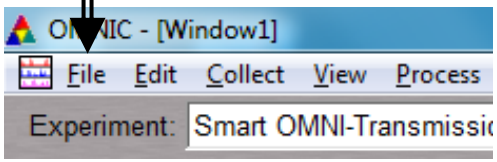
1. Remove ***Sample*** from the ***Crystal*** without scratching the ***Crystal***

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) with appropriate solvent
   - Recommend ***Water*** then ***IPA***
   - DO NOT USE ACETONE!

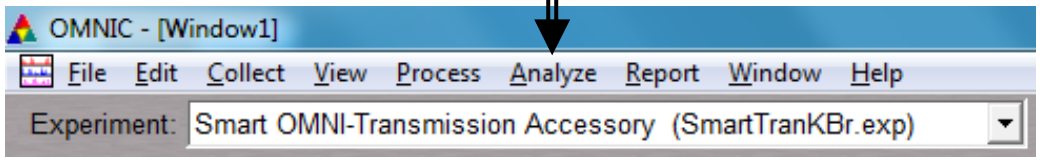
4. Click on ***File -> Exit*** to shut down the software



5. Log off of your ENGR account

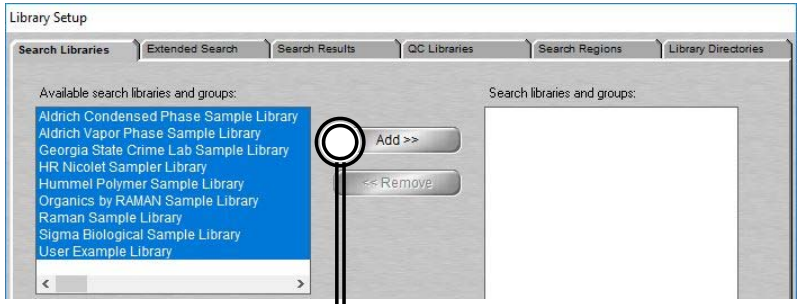
X. Library Search – 1/5

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

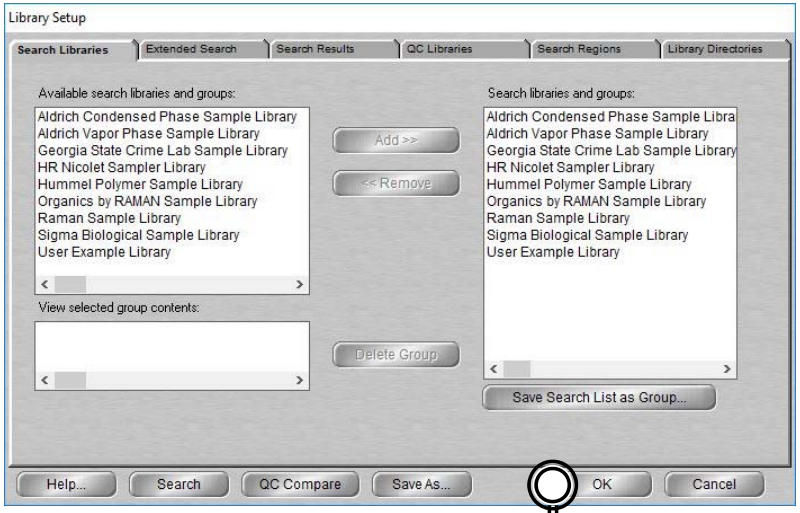


2. Select desired ***Libraries*** or select all

3. Click ***Add >>***

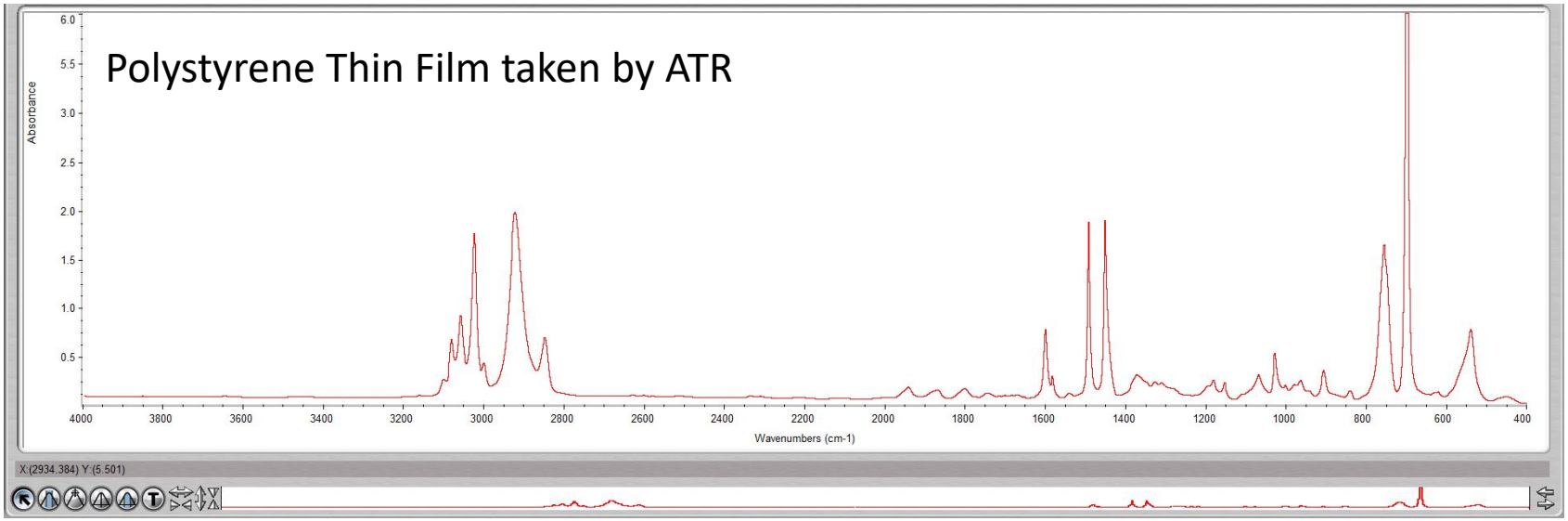


4. Click ***OK***

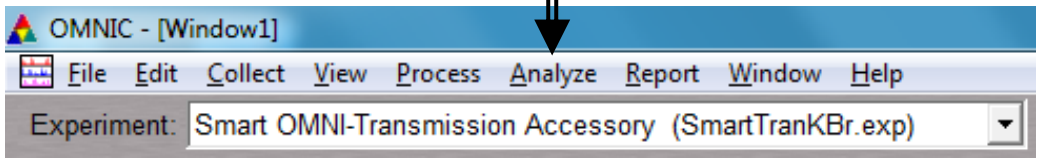


X. Library Search – 2/5

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



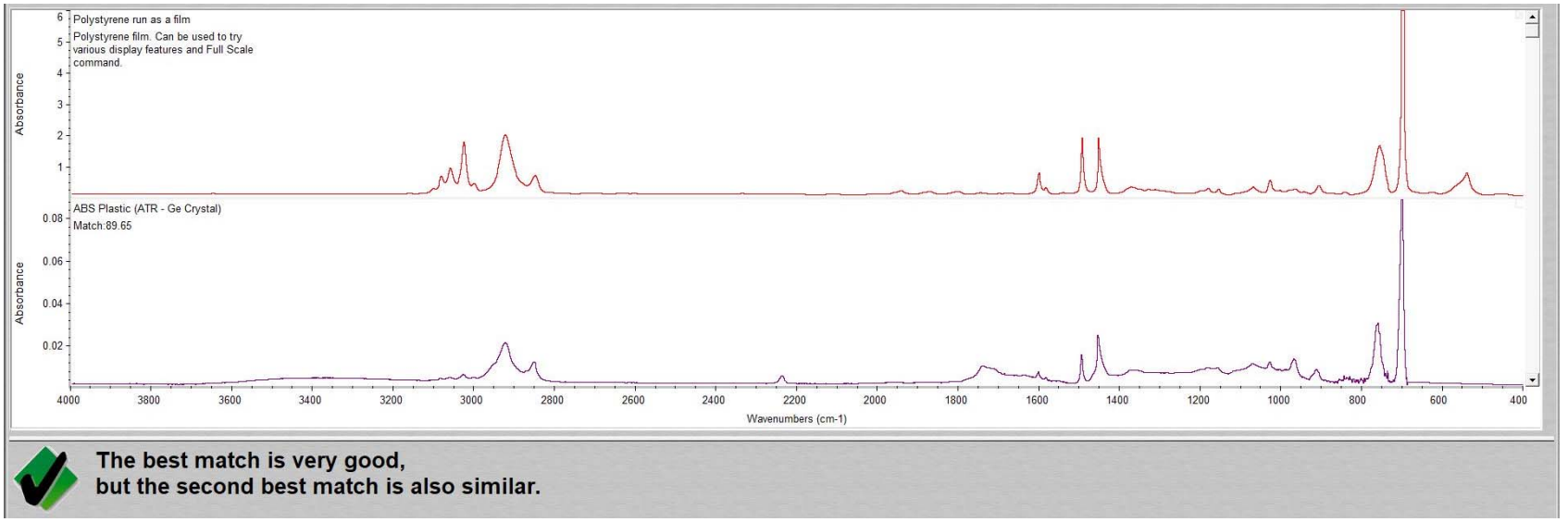
6. Click ***Analyze*** and
   select ***Search…***
   or click ***Search*** icon



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

X. Library Search – 3/5

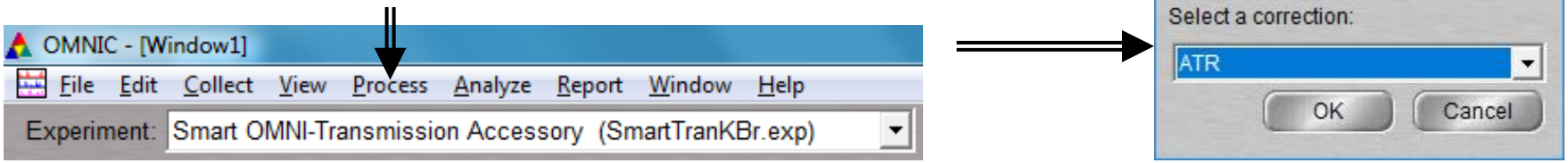
8. The top matches will be shown (below) your acquired spectra (top)



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

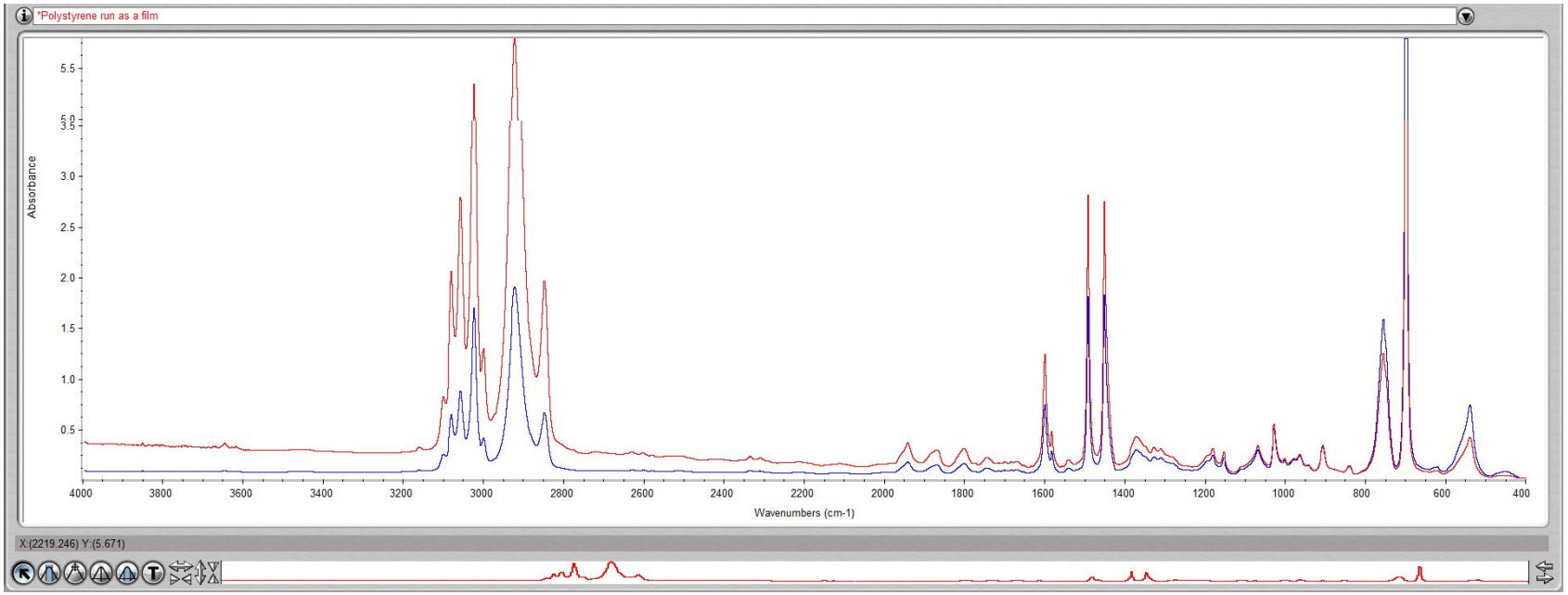
10. Perform ***ATR Correction*** to achieve better match results

11. Click ***Process > Other Corrections…***

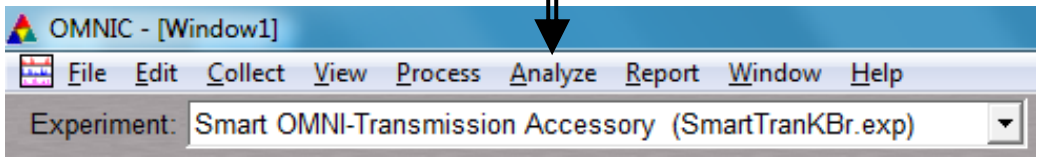


X. Library Search – 4/5

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

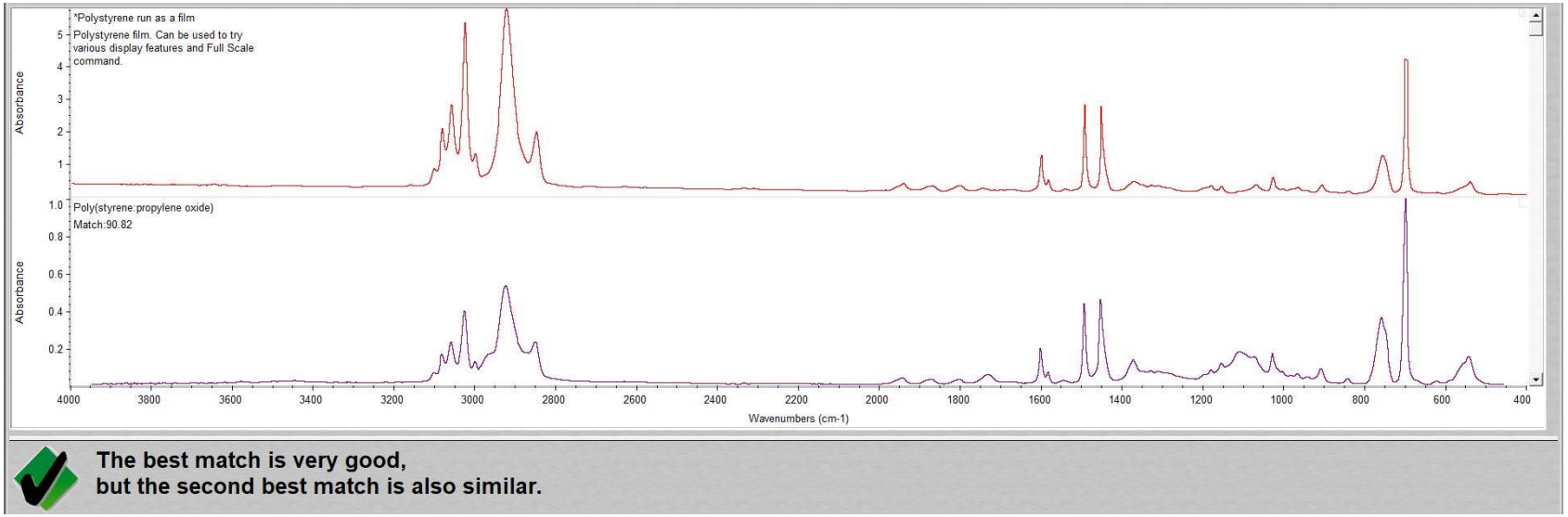


13. Click ***Analyze*** and select ***Search…*** or click ***Search*** icon



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