# FT-IR Training Notebook: Transmission

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 2 hour block on Faces for your training

# Transmission FT-IR Operation

I. Sample Preparation

II. Pellet Press

III. Pellet Retrieval

IV. Sample Holder

V. Smart Transmission Accessory

VI. Initiate Software

VII. Collect Background

VIII. Collect Sample

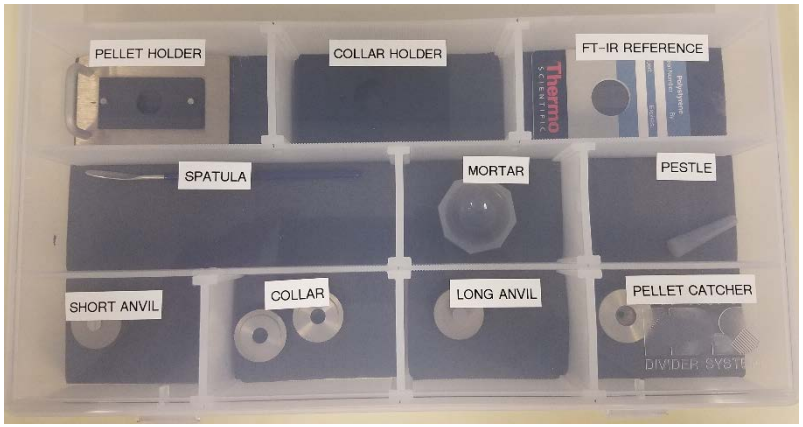
IX. Saving Data

X. Peak Identification

XI. Cleanup

XII. Library Search

# I. Sample Preparation – 1/1



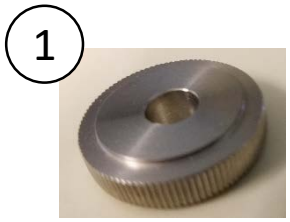

1. Retrieve ***Agate mortar and pestle*** from the storage box in the drawer
2. Recommend **15 mg** of ***Sample Blend*** (sample + KBr) torqued at **15 ft-lbs** for about a minute for a clear pellet
3. A **1:149 sample:KBr *Sample Blend*** is recommended to achieve necessary transparency of KBr
   - Example: 1 mg of sample blended with 149 mg of KBr
4. Weigh out the appropriate amounts of sample and KBr using provided balance
5. Use provided ***Agate mortar and pestle*** to grind and mix the powder blend

NOTE: DO NOT USE ALL 150 MG OF BLEND FOR PELLET, ONLY USE ABOUT **15 MG** FOR EACH PELLET!!!

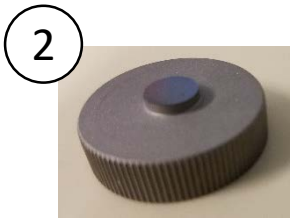
5. Weigh out ~ 15 mg of the ***Sample Blend*** (sample + KBr) using provided balance

# II. Pellet Press – 1/4

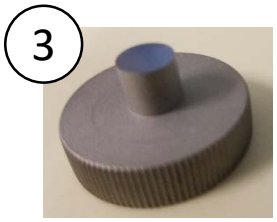
1. Retrieve the following items from the storage box:



Collar

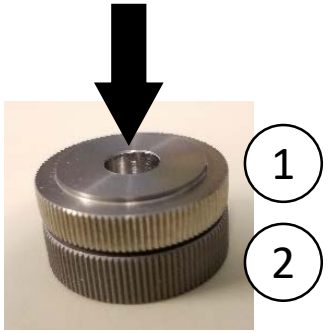


Short Anvil



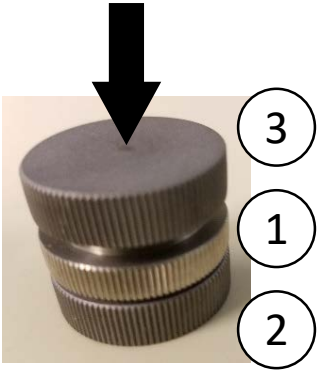
Long Anvil

2. Place the ***Collar*** above the ***Short Anvil*** first




3. Carefully insert the ***Sample Blend*** into the ***Collar Assembly***

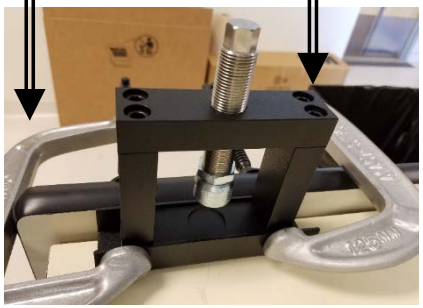
4. Tap the ***Collar*** assembly lightly to spread the powder uniformly across the collar assembly




5. Insert the ***Long Anvil*** on top of the ***Collar Assembly***

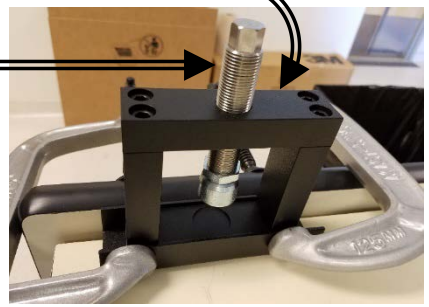
# II. Pellet Press – 2/4

6. Tighten the ***C-clamps*** if loose to prevent ***Pellet Press*** from moving

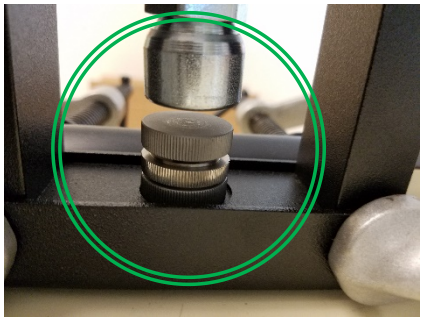


7. Insert the ***Collar Assembly*** into the ***Pellet Press*** and align it with the recessed circle

8. Hand-tighten the ***Nut*** at the top



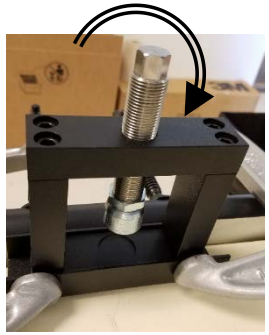
9. Check and adjust the ***Press*** to be parallel with the top of the ***Long Anvil*** face



# II. Pellet Press – 3/4


10. Retrieve the ***Torque Wrench*** from the drawer

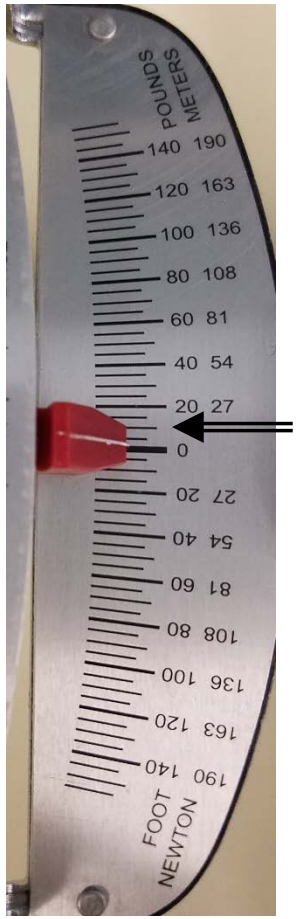
11. Use the ***Torque Wrench*** and tighten **clockwise** until **15 ft-lbs** of torque is applied



12. Tightening to **15 ft-lbs** may require repeated turns by lifting up ***Torque Wrench*** and repeating

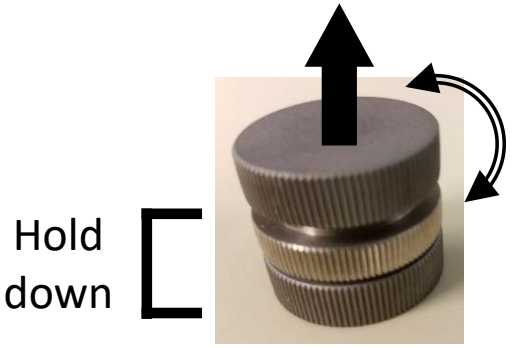
NOTE: Torque-wrench is non-ratcheting, DO NOT turn counter-clockwise to achieve more torque

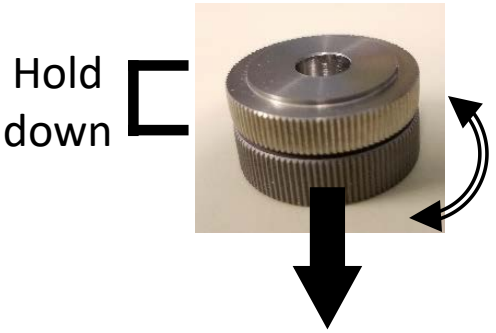
13. Once **15 ft-lbs** of torque is achieved, **HOLD** this position for at least **1 minute**



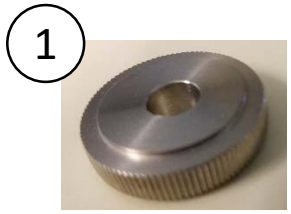
# II. Pellet Press – 4/4

14. Slowly release the torque by untightening **counter-clockwise** using the ***Torque Wrench***
15. Once the ***Press*** is loose, you may continue to loosen and raise the ***Press*** up by hand
16. Carefully take the entire ***Collar Assembly*** out of the ***Pellet Press***
17. Carefully hold both ***Lower Anvil*** and ***Collar*** together and twist the top ***Upper Anvil*** and pull out
18. Repeat this time holding the ***Collar*** and twist the ***Lower Anvil*** and out
19. The ***Collar*** should now have a clear and whole ***Pellet*** for analysis
20. If the ***Pellet*** is not uniformly clear, repeat **Steps 2 - 18**

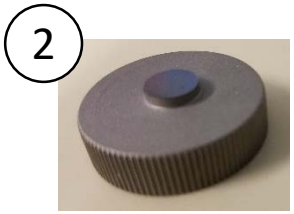


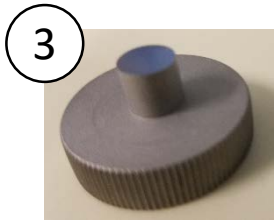
# III. Pellet Retrieval – 1/1




Collar




Short Anvil



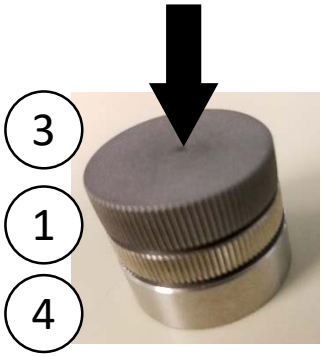

Long Anvil


Pellet Catcher

1. If you wish to keep the pellet for future examination, retrieve the ***Pellet Catcher*** from the storage box


2. Place the collar with the ***Collar*** containing the ***Pellet*** above the ***Pellet Catcher***

3. Center and align the ***Collar*** with the the ***Pellet Catcher***




4. Insert the ***Long Anvil*** into the ***Collar*** and slowly push the ***Pellet*** out of the ***Collar***

5. If done correctly, the ***Pellet*** should still be whole and inside the ***Pellet Catcher***

# IV. Sample Holder – 1/1

Collar Holder

Pellet Holder

1. Retrieve either the ***Collar Holder*** or the ***Pellet Holder*** from the storage box

2. Insert the ***Collar*** with the sample into the ***Collar Holder***

3. If you wish to scan a ***13 mm*** or ***7 mm Pellet***, you will have to use the ***Pellet Holder*** instead

4. Assemble the ***Pellet Holder*** with the magnetic strip that matches your pellet size (***13 mm*** or ***7 mm diameter Pellet***)

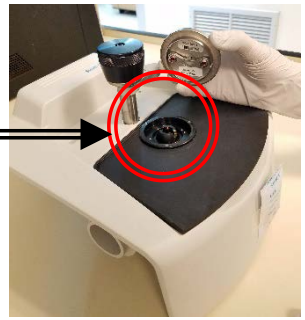
5. Sandwich the ***Pellet*** between the two magnetic strips as shown

# V. 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

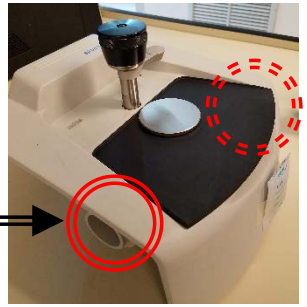
3. The ***Smart ATR Accessory*** contains mirrored optics that need to be carefully taken care to avoid damage and contamination

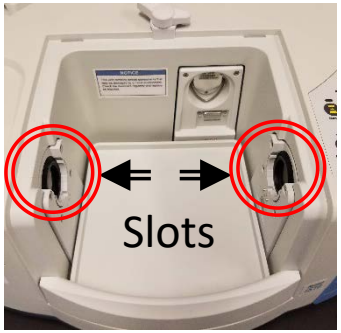


4. Both ***Smart ATR Accessory*** and ***Smart Transmission Accessory*** have nozzles to fit into slots of FT-IR base

Nozzle

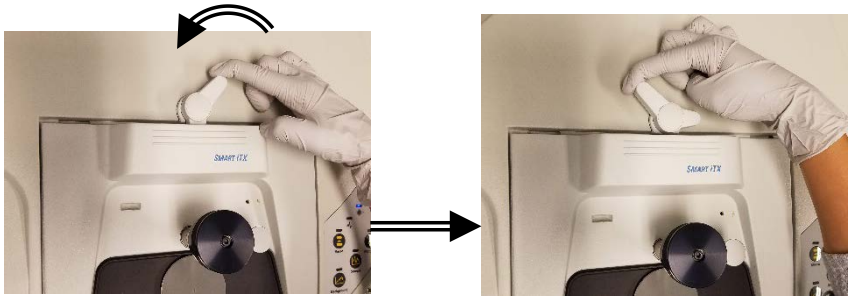
Nozzle



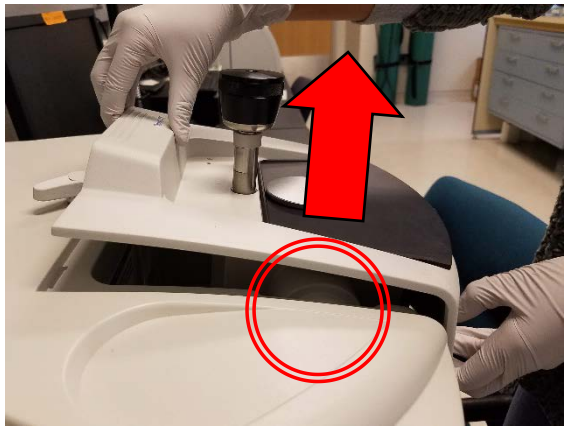


# V. Smart Transmission Accessory – 2/3

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

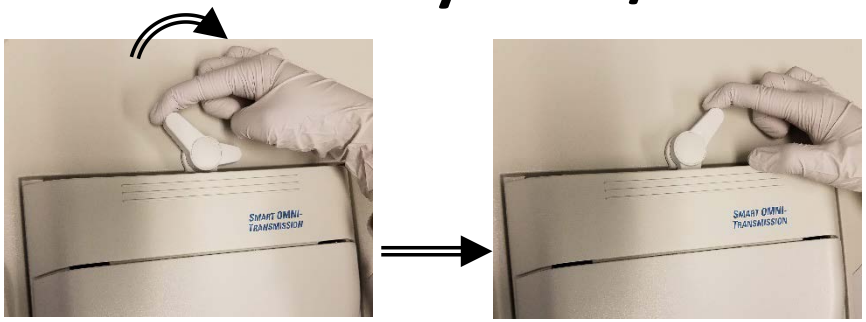


7. Carefully place aside and KEEP AWAY FROM CONTAMINANTS!

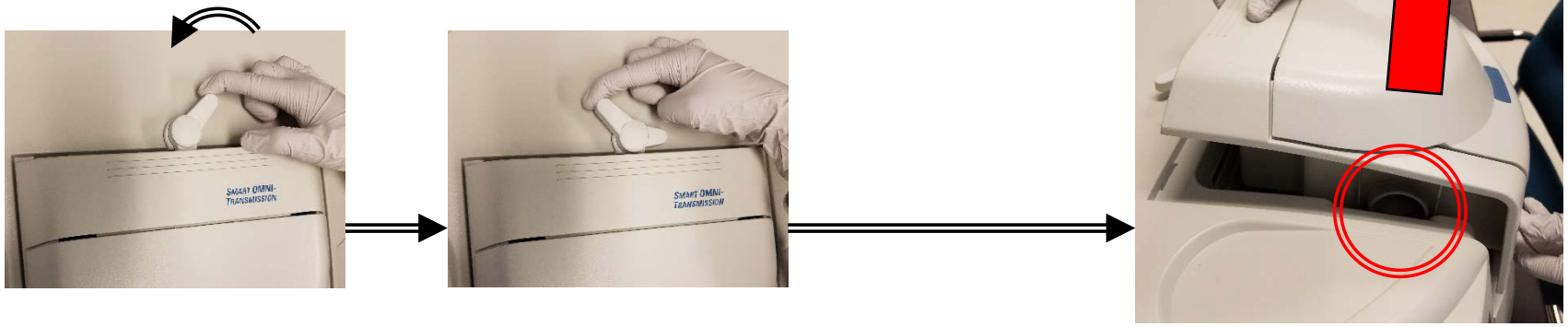
8. Carefully insert the ***Smart Transmission Accessory*** by gently aligning the nozzles into the slots

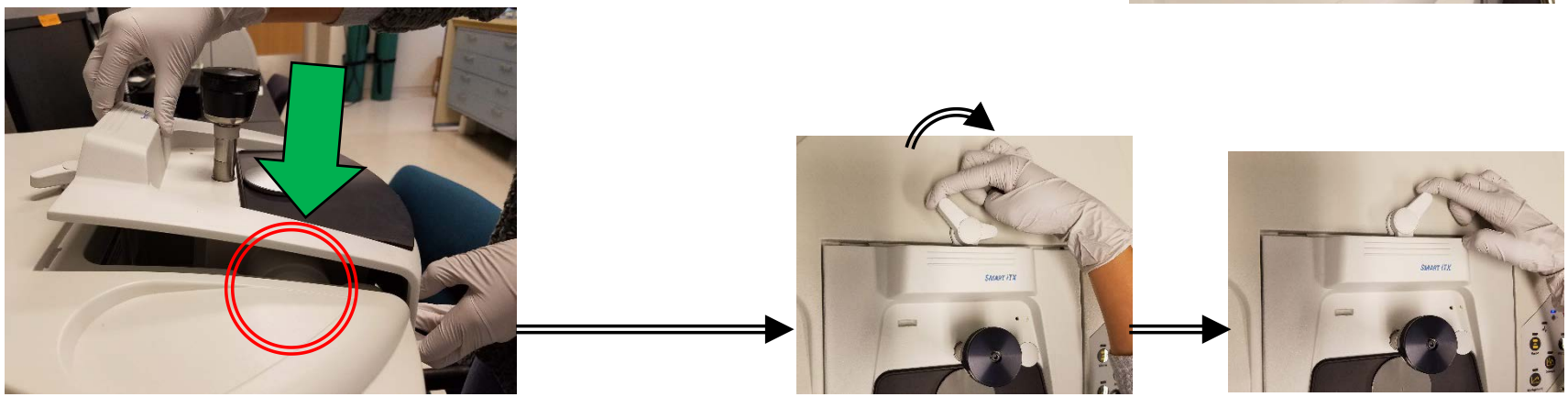
# V. Smart Transmission Accessory – 3/3

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…

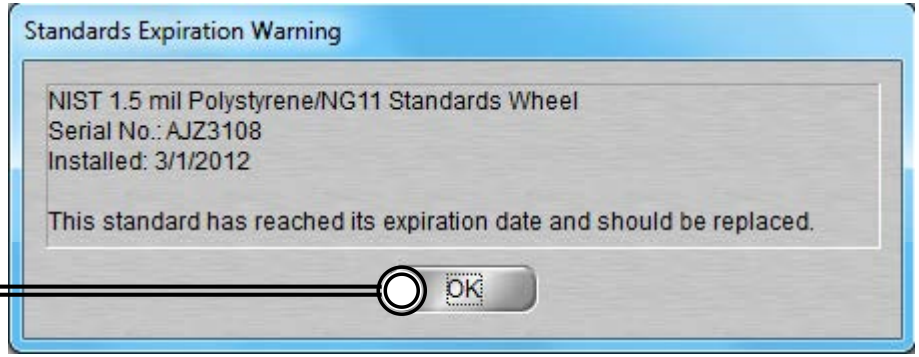


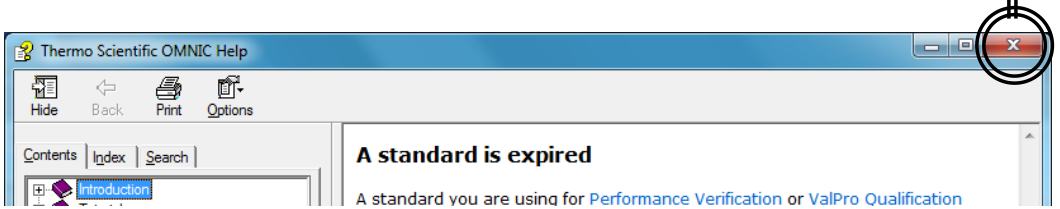
# VI. Initiate Software – 1/10


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

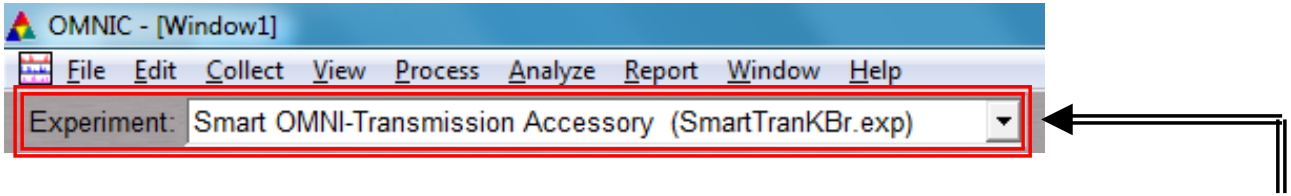
2. Ignore the ***Standards Expiration Warning*** and click ***OK*** if present




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



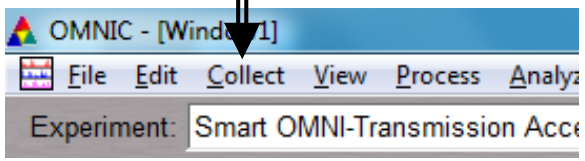

4. The ***OMNIC main window*** will now appear



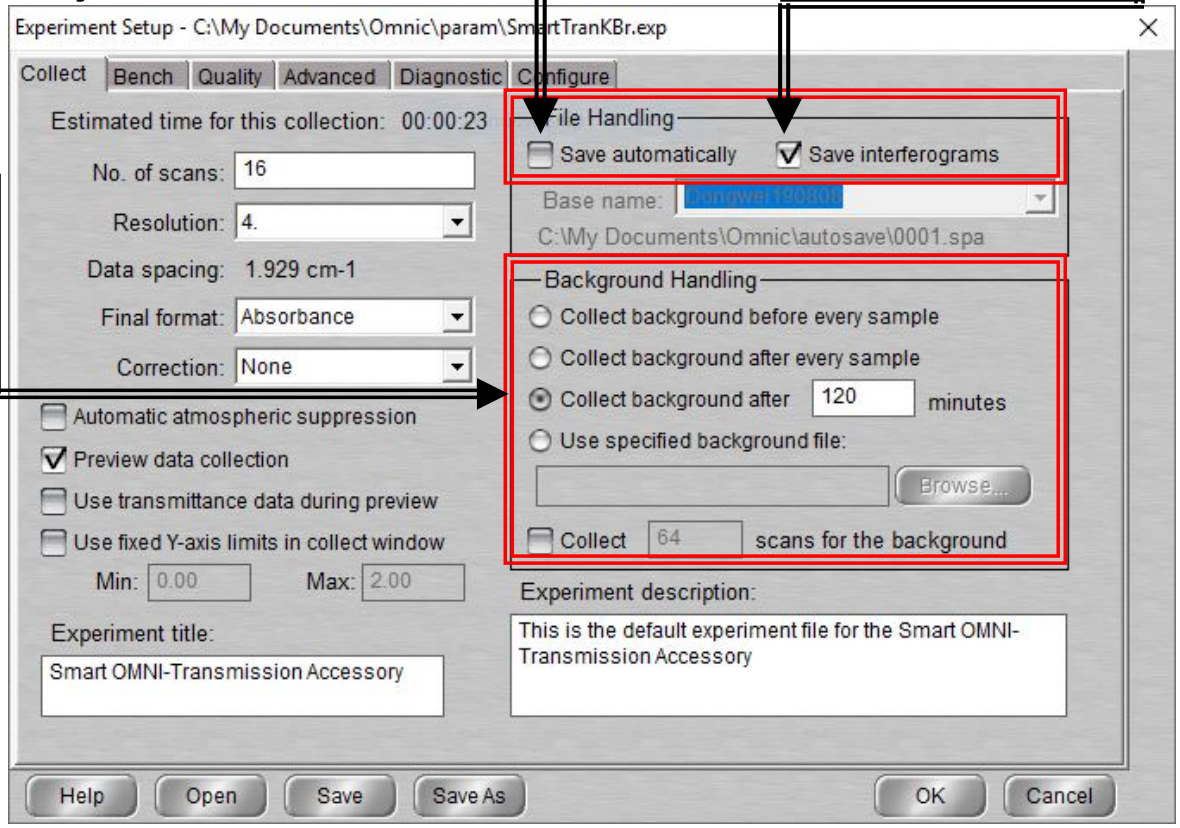

5. Confirm that ***Smart OMNI-Transmission Accessory (SmartTranKBR.exp)*** appears in the Experiment window

# VI. 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



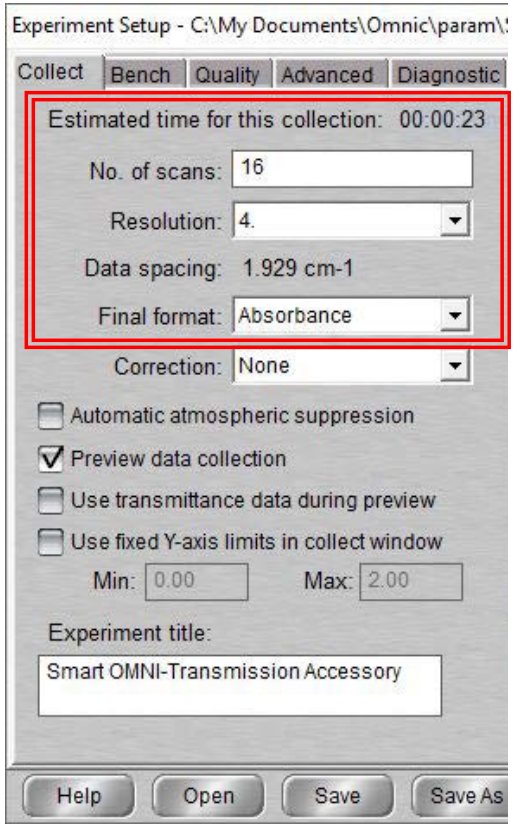

Note: A new background will requested if you there is a change in resolution or data spacing of your sample spectrum!

# VI. 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

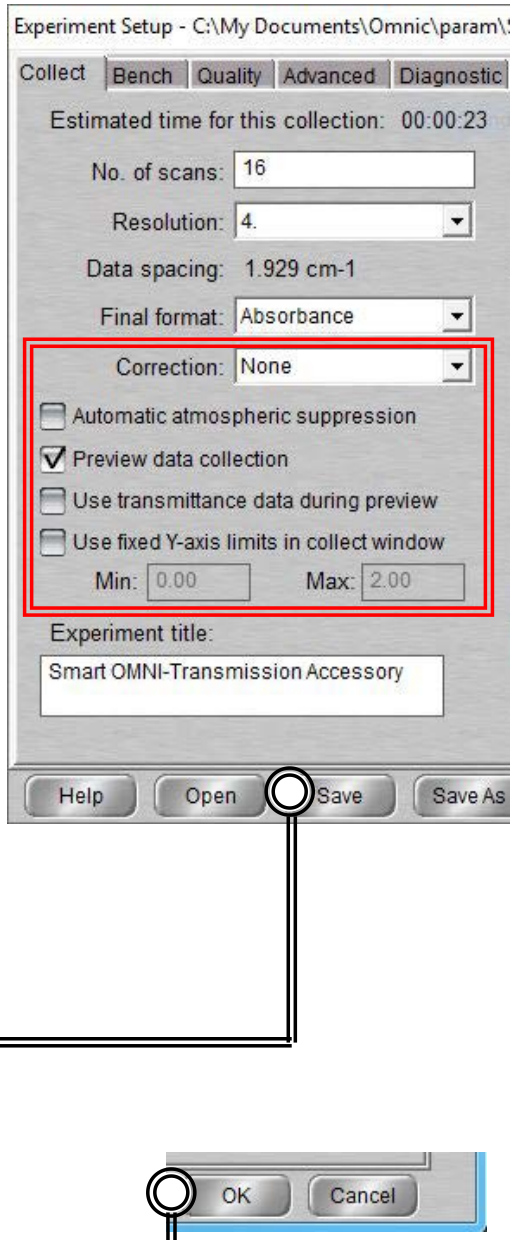


# VI. Initiate Software – 4/10

14. Select desired ***Correction type*** – recommend ***None***
    - *None* (no correction)
    - *H2O and CO2 – requires a reference file!*
    - *Etc…*
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 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%
19. Click "***Save***" then "***OK***"



# VI. Initiate Software – 5/10

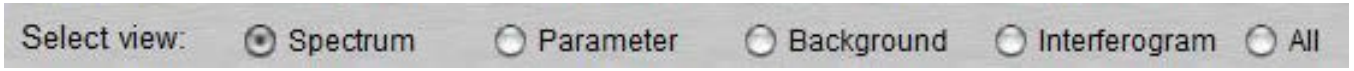
20. Select ***Bench*** and check ***Parameters***


21. Confirm that the following are correct:
    - ***Source = IR***
    - ***Accessory = OMNI-Transmission***
    - ***Window = KBr***
22. Select desired ***Max*** and ***Min*** range limit for your scans
    - Recommend using ***Recommended range***
23. Select the ***Gain*** parameter
    - Electronically amplifies signal – recommend ***Autogain***
    - DO NOT set to ***Autogain*** if performing quantitative analysis
24. Select the desired Aperture
    - ***High resolution*** – used with resolution at 2 or less
    - ***Medium resolution*** – recommended with resolution 4 and above
25. Confirm the ***Screen wheel*** is set to ***Open***

| Parameter | Value |
|---|---|
| Sample compartment | Main |
| Detector | DTGS KBr |
| Beamsplitter | KBr |
| Source | IR |
| Accessory | OMNI-Transmissio |
| Window | KBr |
| Recommended range | 4000 ---- 400 |
| Max range limit | 4000 |
| Min range limit | 400 |
| Gain:1 | Autogain |
| Optical velocity | 0.4747 |
| Aperture | Medium resolution |
| Sample shuttle | |
| Screen wheel | Open |

# VI. Initiate Software – 6/10

26. Select ***Quality*** 


27. 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

28. 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 ***NO***
    - ***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%***

# VI. Initiate Software – 7/10

29. 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***
30. If you chose to check ***Background***…
    - ***Background correct for accessory*** – checks background spectrum, recommend ***ON*** at ***50%***
    - ***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%***
31. 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***

# VI. Initiate Software – 8/10

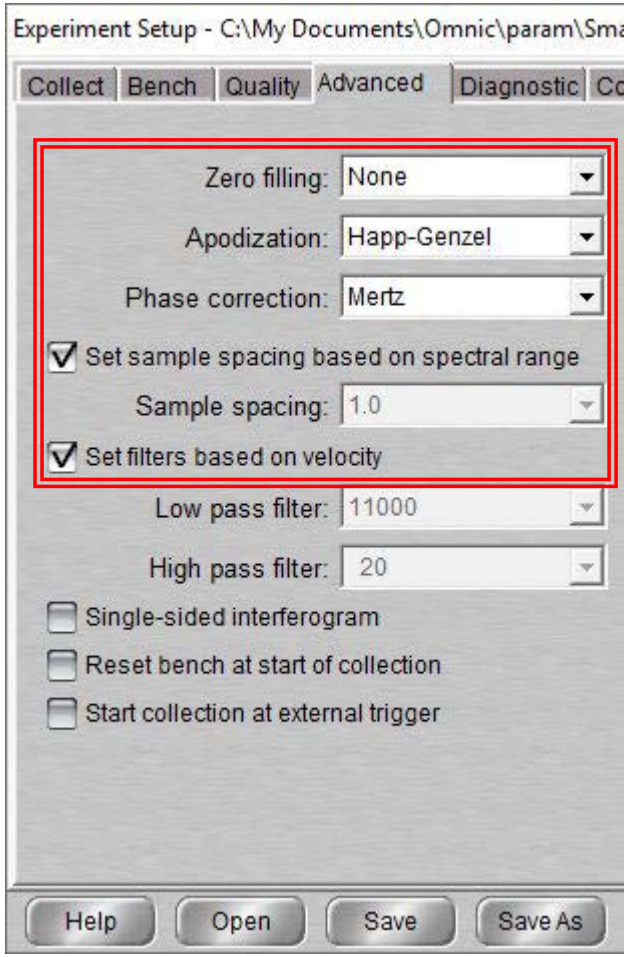
32. Select ***Advanced*** 


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

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

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

36. Confirm that the following are checked:
    - ***Set sample spacing based on spectral range***
    - ***Set filters based on velocity***



# VI. Initiate Software – 9/10

37. Select ***Diagnostic***


38. Click on indicators to check spectrometer components


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

39. 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

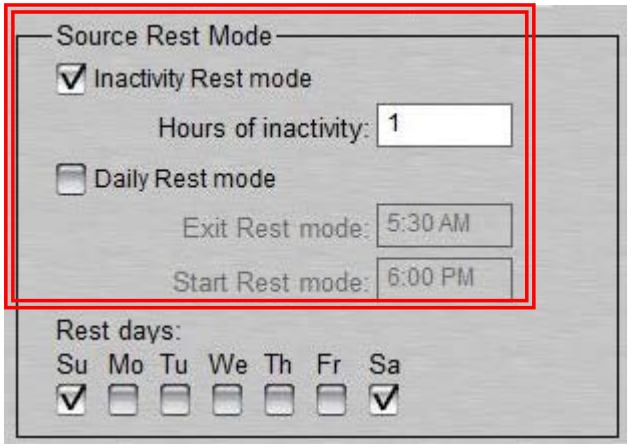
40. Click on ***Rest Bench*** button to reposition the peak if drift occurs

# VI. Initiate Software – 10/10

41. Select ***Configure***

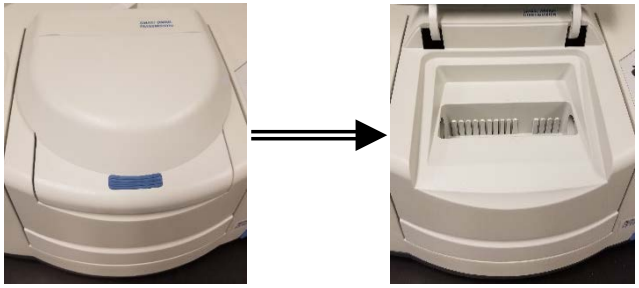

42. Confirm ***Inactivity Rest mode*** is checked
    - Confirm ***Hours of inactivity*** is set to "***1***" hour

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



# VII. Collect Background – 1/2

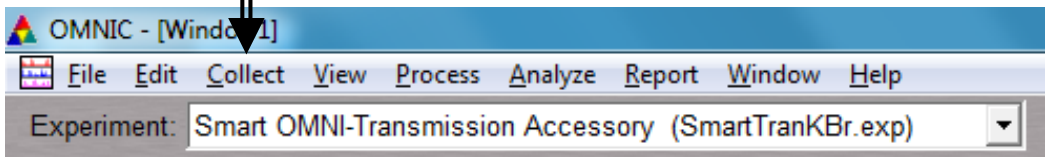
1. Open the ***Chamber Cover***



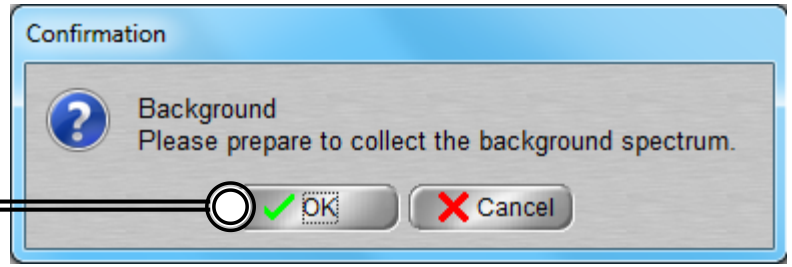
2. Choose one of the following:
   - Empty chamber
   - Collar Holder and a KBr sample
   - Pellet Holder and a KBr sample

     *Insert Holders in ***Notch***


3. Select ***Collect -> Collect Background***

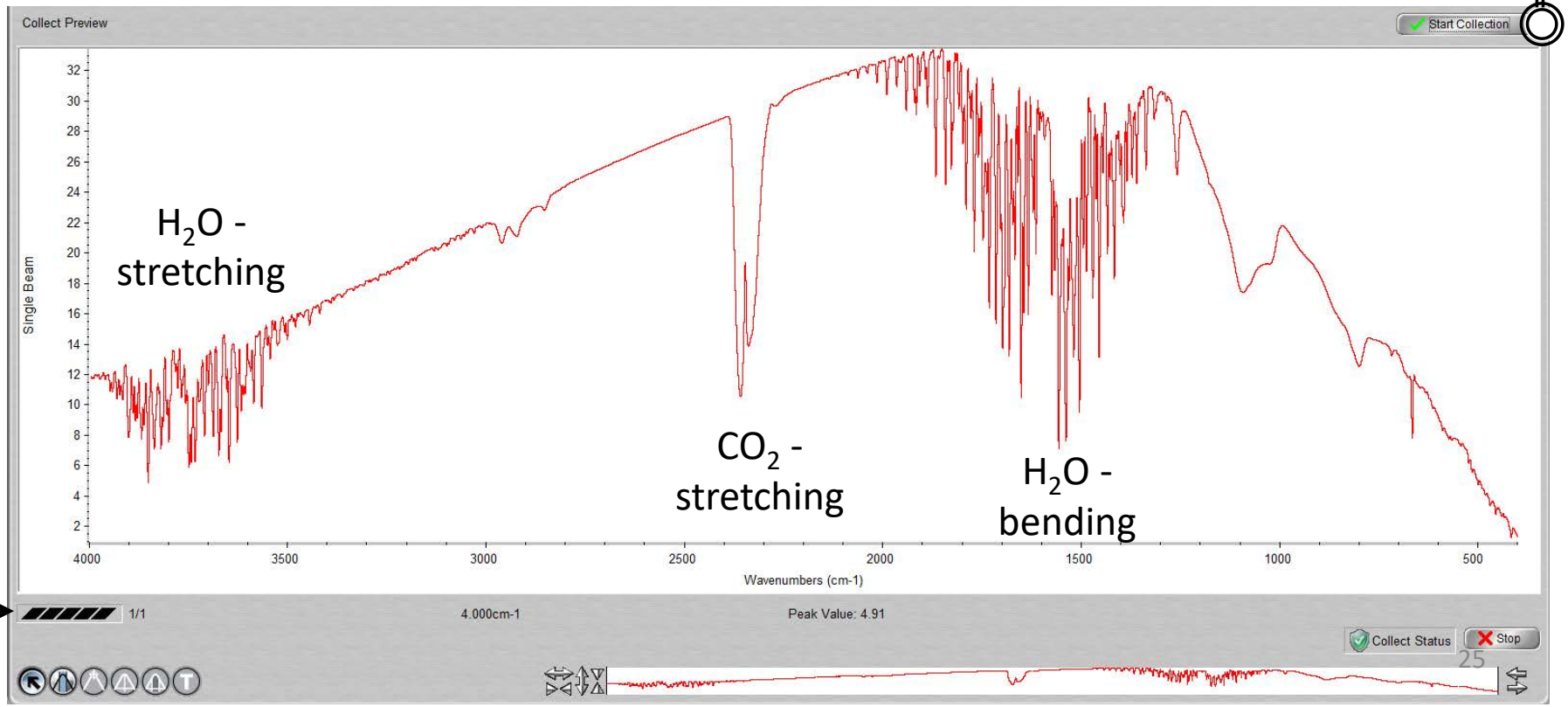



4. Confirm to collect background by clicking ***OK***



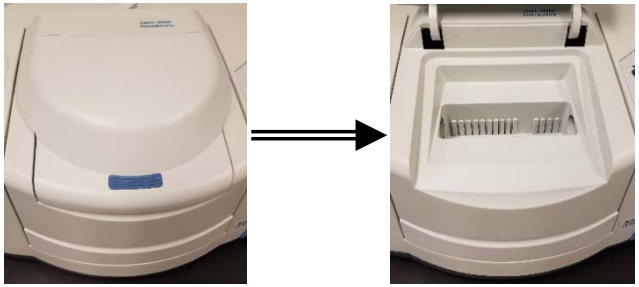
# VII. Collect Background – 2/2

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



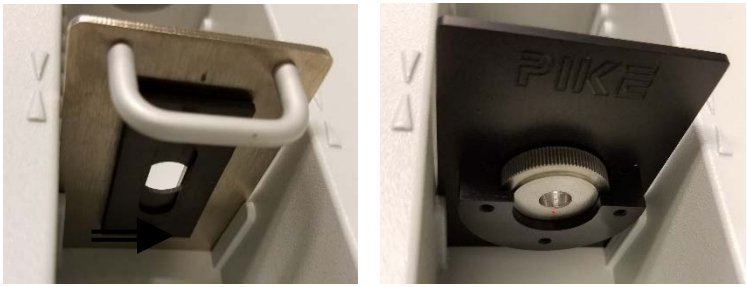
# VIII. Collect Sample – 1/2

1. Open the ***Chamber Cover***

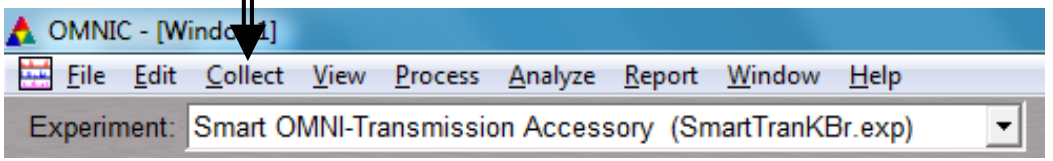


2. Insert your sample into Chamber via:
   - Collar Holder
   - Pellet Holder

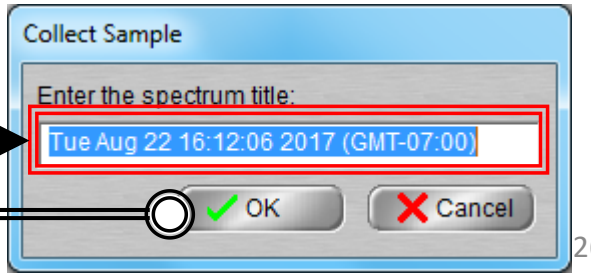
   *Insert Holders in ***Notch***




3. Select ***Collect -> Collect Sample***

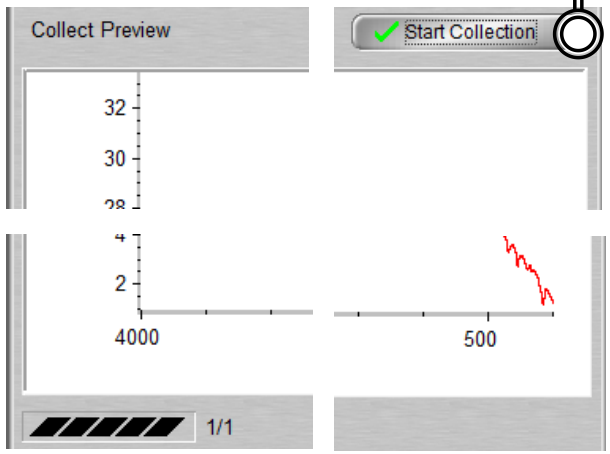


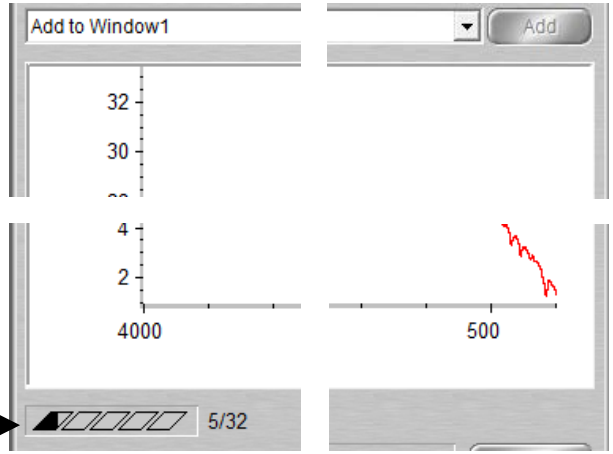

4. Enter ***Spectrum Title*** and click ***OK*** to ***Collect Sample***



# VIII. Collect Sample – 2/2

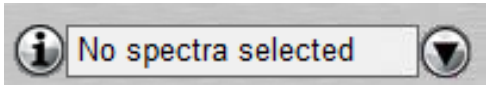
5. Preview ***Sample Collection***
6. Click ***Start Collection*** to begin ***Sample Collection***
7. The ***Sample Collection*** will begin with the progress shown at the bottom
8. Confirmation of ***Data Collection*** will be shown
9. Click ***Yes*** to add to data to current Window



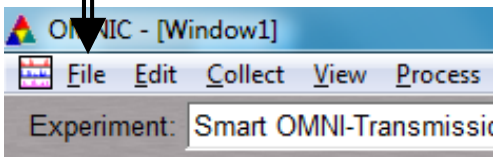




# IX. Saving Data – 1/1

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)



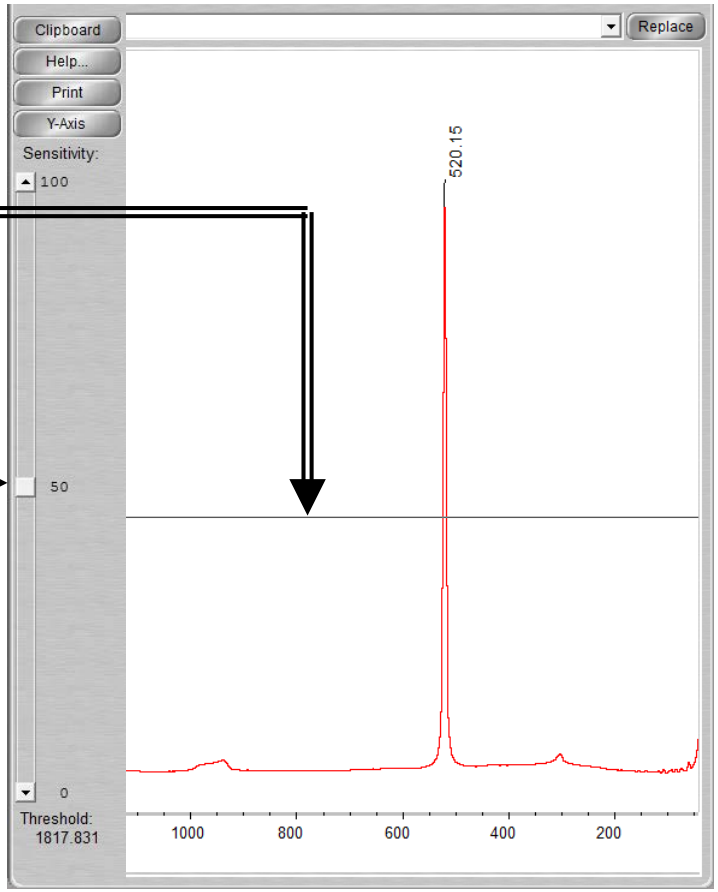

5. Click ***File -> Save Group*** to save more than one spectrum as a group in one file having file extension .SPG to open later

# X. Peak Identification – 1/1

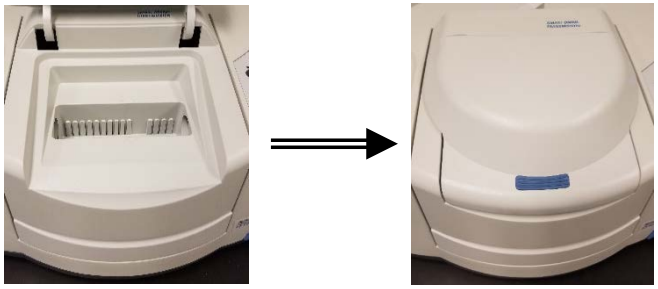
1. Click on "***Find Pks***" button at the top


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

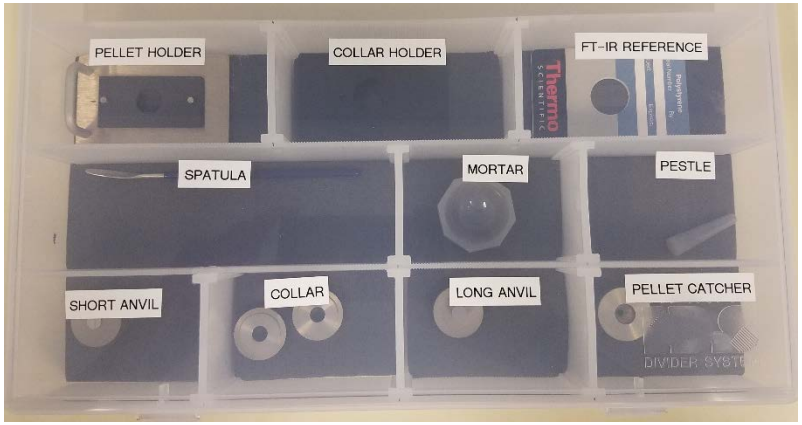


# XI. Cleanup – 1/1



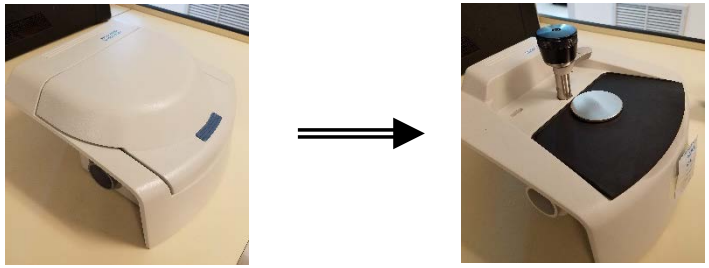
1. Remove ***Sample Holders*** from the ***Chamber***

2. Close the ***Chamber Cover***

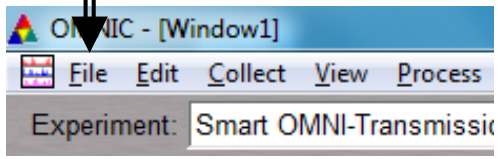



3. Clean up ***Sample Holders*** and any tools used and return back to storage box

4. Remove the ***Smart Transmission Accessory*** and replace back with ***Smart ATR Accessory*** (see V. Smart Transmission Accessory)



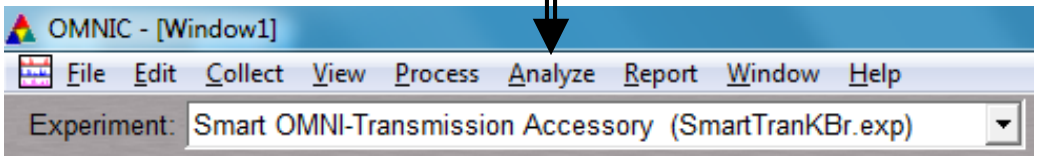
5. Click on ***File -> Exit*** to shut down the software



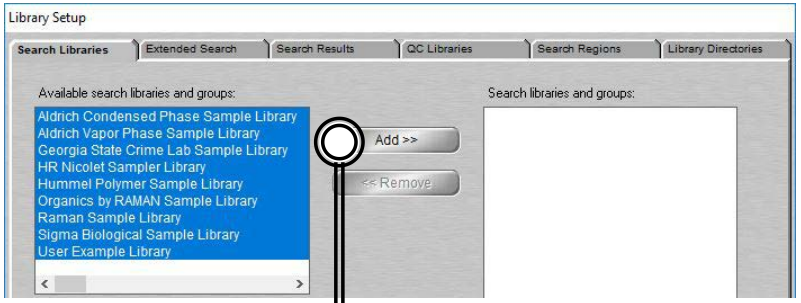

6. Log off of your ENGR account

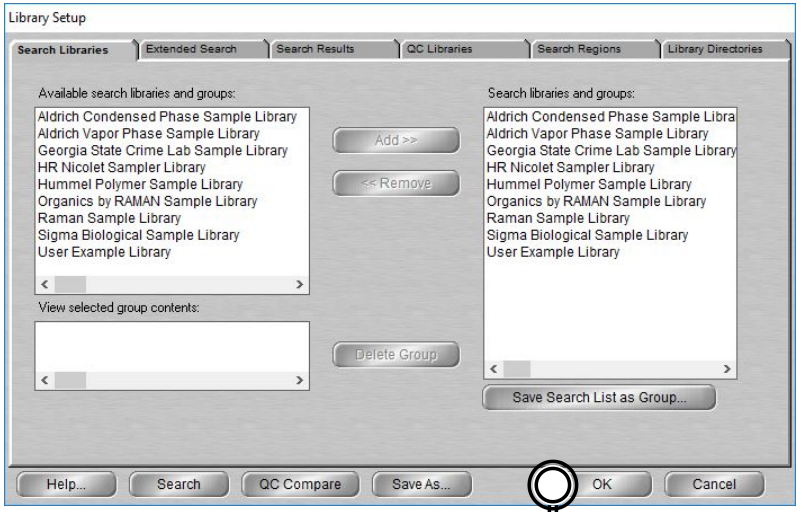
# XII. Library Search – 1/3

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



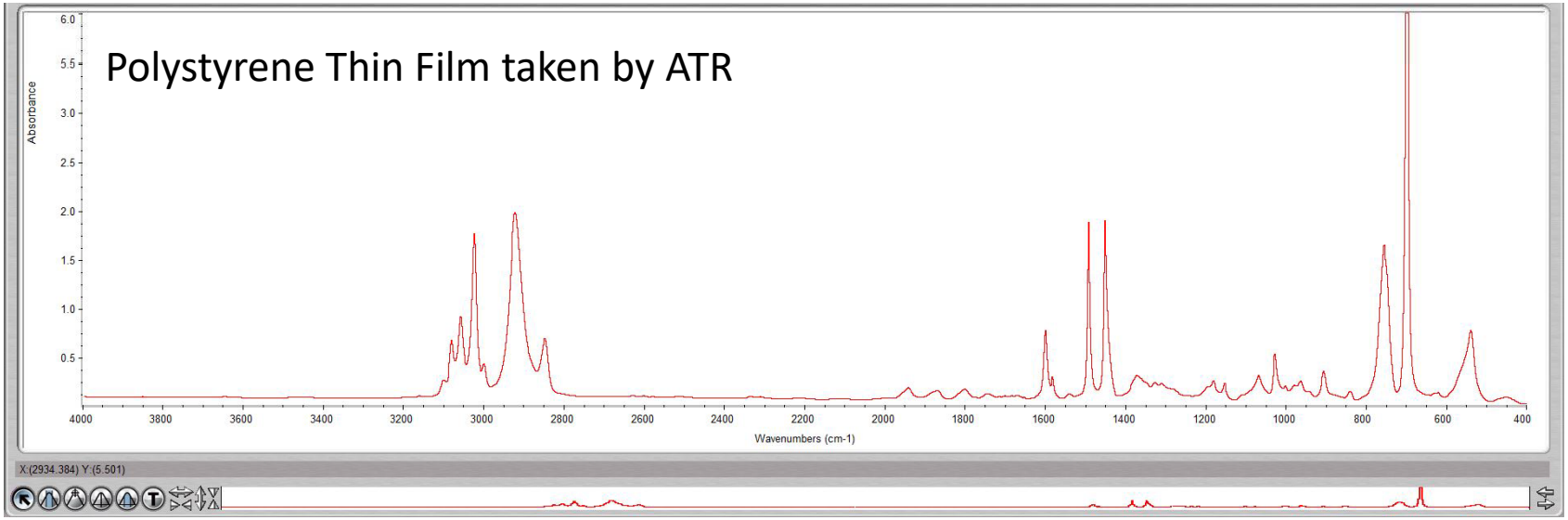

2. Select desired ***Libraries*** or select all
3. Click ***Add >>***
4. Click ***OK***



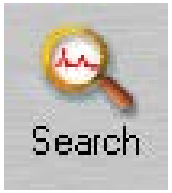



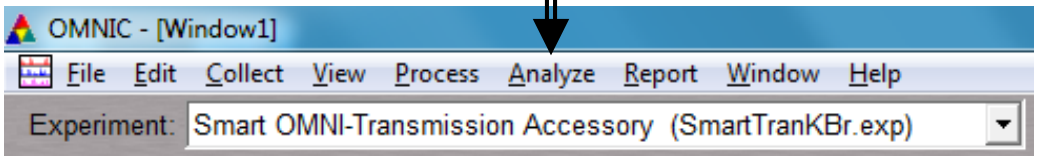
# XII. Library Search – 2/3

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




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

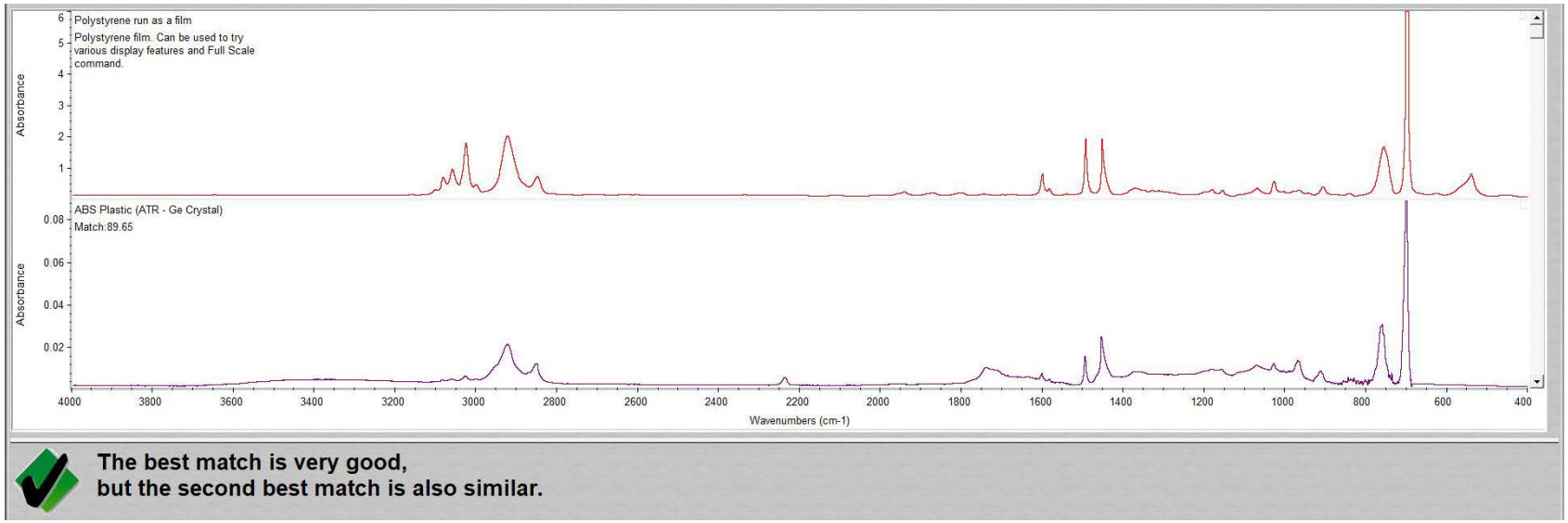


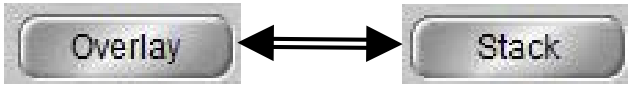

7. Select desired ***Libraries*** or select all

# XI. Library Search – 3/3

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