FT-IR Training Notebook: Transmission

Lab Manager: Dr. Perry Cheung

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 2 hour block on Faces for your training

FT-IR Operation

I.   Sample Preparation

II.   Pellet Press

III.   Pellet Retrieval

IV.   Sample Holder

V.   Initiate Software

VI.   Collect Background

VII.   Collect Sample

VIII.   Saving Data

IX.   Peak Identification

X.   Cleanup

XI.   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 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
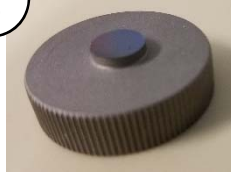
II. Pellet Press – 1/x
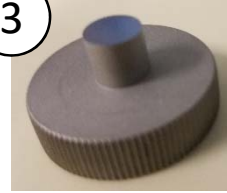
1. Retrieve the following items from the storage box:
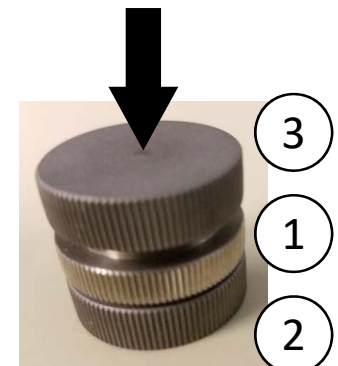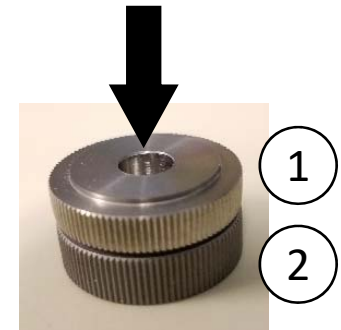
①

Collar

②

Short Anvil

③

Long Anvil

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

①
②

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

③
①
②

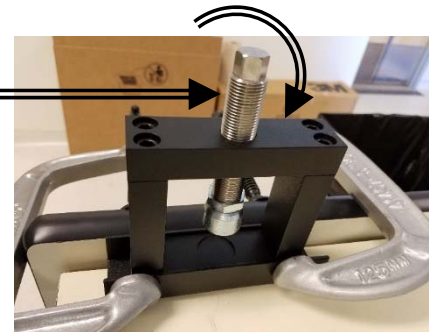5. Insert the **Long Anvil** on top of the **Collar Assembly**

II. Pellet Press – 2/x

6. Tighten the *C-clamps* if loose to prevent *Pellet Press* from moving
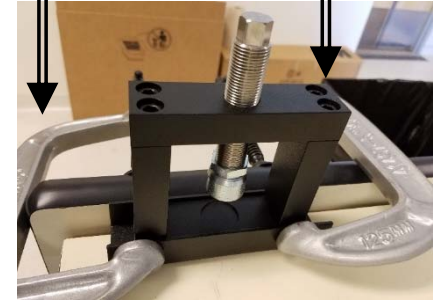
7. Insert the *Collar Assembly* into the *Pellet Press* and align it with the recessed circle
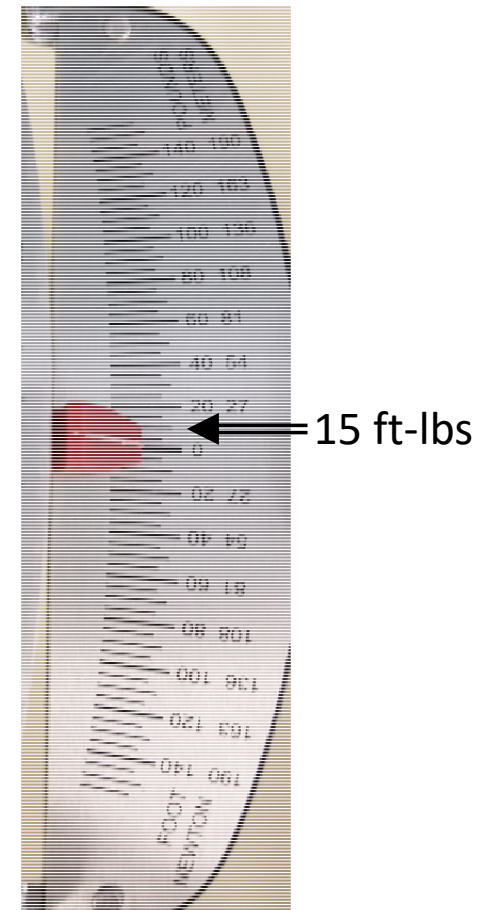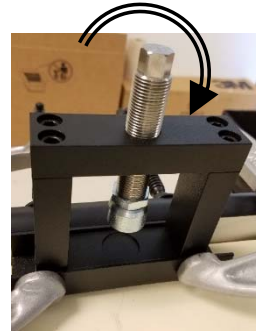
8. Hand-tighten the *Nut* at the top

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

II. Pellet Press – 3/x

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**

←——— 15 ft-lbs

II. Pellet Press – 4/x

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

Hold down

18. Repeat this time holding the *Collar* and twist the *Lower Anvil* and out

Hold down

19. The *Collar* should now have a clear and whole *Pellet* for analysis

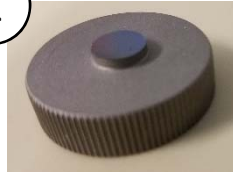20. If the *Pellet* is not uniformly clear, repeat **Steps 2 - 18**
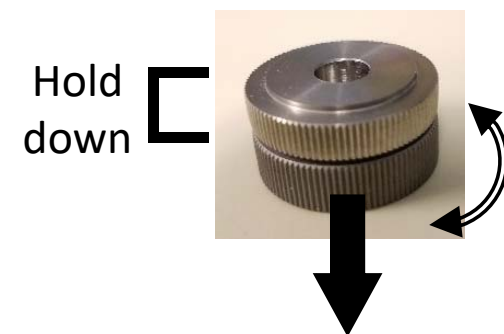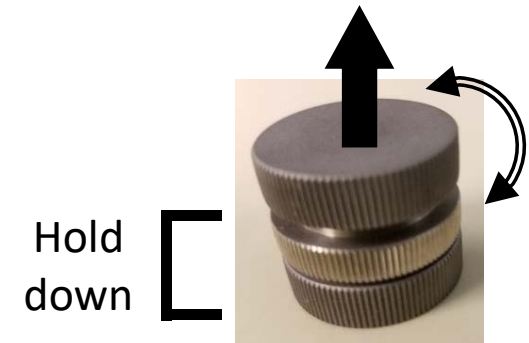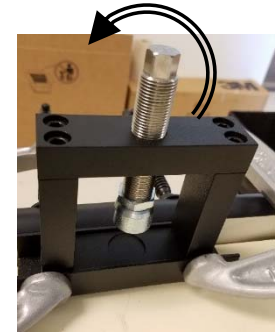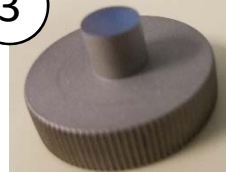
III. Pellet Retrieval – 1/x

① **Collar**

② **Short Anvil**

③ **Long Anvil**

④ **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**

9

IV. Sample Holder – 1/x

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**)

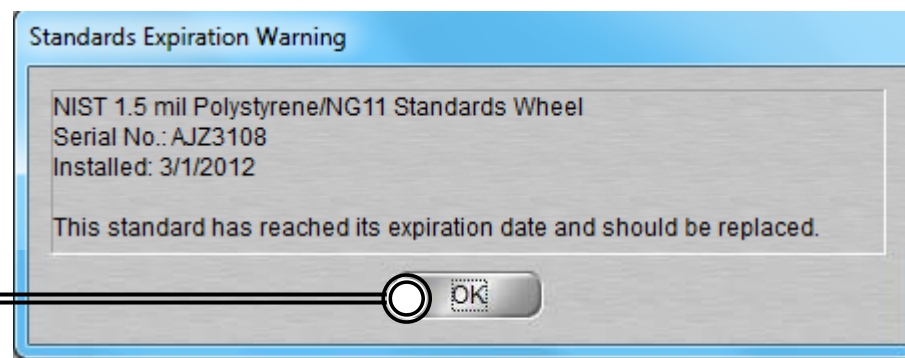5. Sandwich the **Pellet** between the two magnetic strips as shown
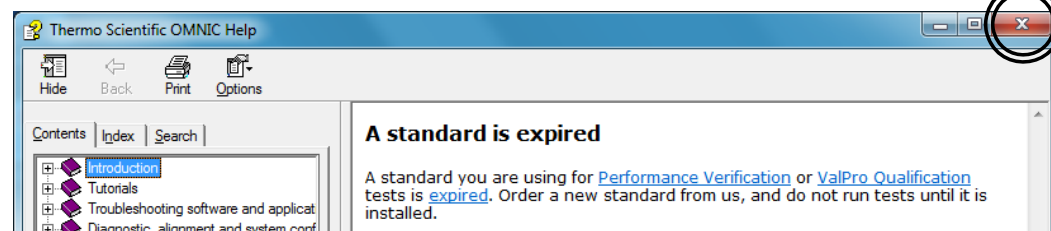
V. Initiate Software – 1/3

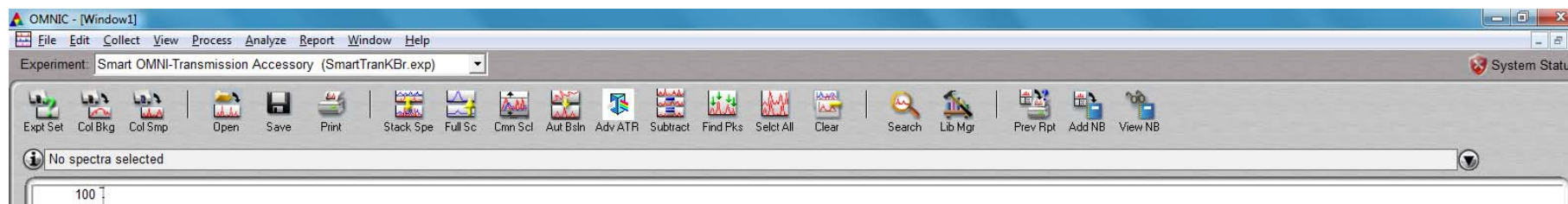1. Double left-click on the **OMNIC software icon** for FT-IR

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

Standards Expiration Warning

NIST 1.5 mil Polystyrene/NG11 Standards Wheel
Serial No.: AJZ3108
Installed: 3/1/2012

This standard has reached its expiration date and should be replaced.

OK

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

Thermo Scientific OMNIC Help

Hide   Back   Print   Options

Contents | Index | Search

Introduction
Tutorials
Troubleshooting software and applicat
Diagnostic, alignment and system conf

A standard is expired

A standard you are using for Performance Verification or ValPro Qualification tests is expired. Order a new standard from us, and do not run tests until it is installed.

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

OMNIC - [Window1]

File   Edit   Collect   View   Process   Analyze   Report   Window   Help

Experiment: Smart OMNI-Transmission Accessory (SmartTranKBr.exp)

System Status

Expt Set   Col Bkg   Col Smp   Open   Save   Print   Stack Spe   Full Sc   Cmn Scl   Aut Bsln   Adv ATR   Subtract   Find Pks   Select All   Clear   Search   Lib Mgr   Prev Rpt   Add NB   View NB

No spectra selected
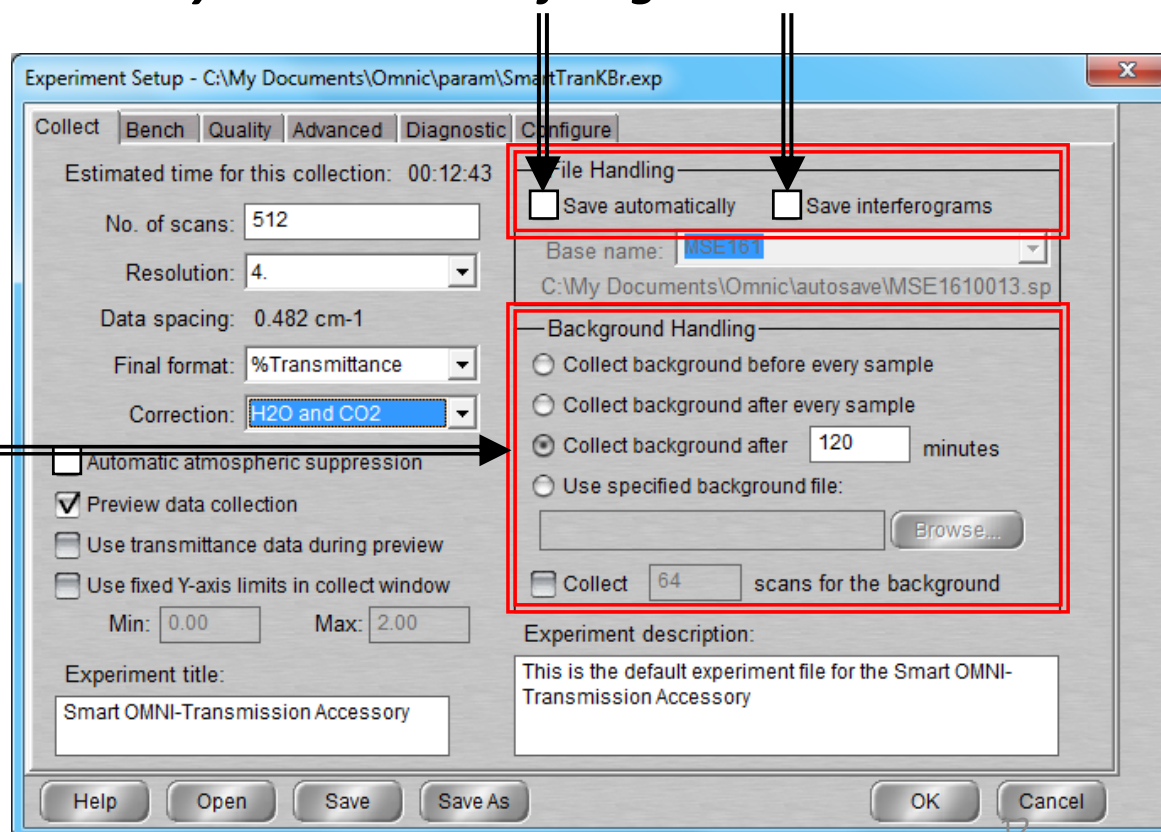
100

V. Initiate Software – 2/3

5. Select **Collect -> Experiment Setup** at the top window
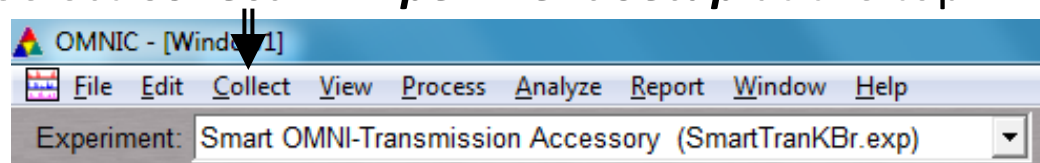
6. **Uncheck** both the **Save automatically** and **Save interferograms** under **File Handling**
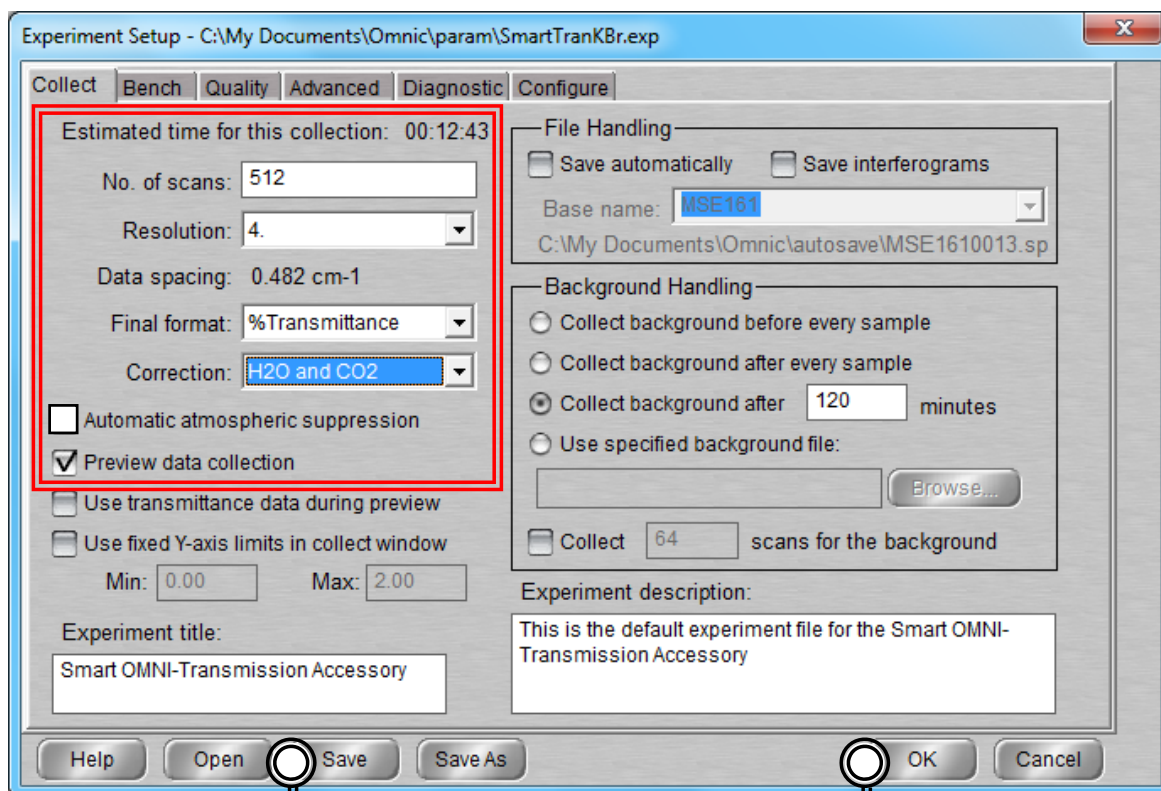
7. Set preferred **Background Handling** settings
   - Before every sample
   - After every sample
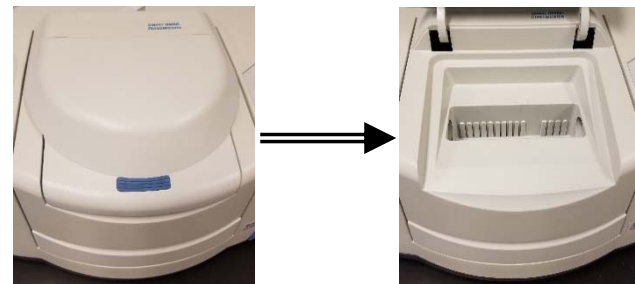   - After 120 minutes (default)
   - Use specific file

V. 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)*
   - *H2O and CO2*
   - *Etc…*

10. Check *Preview data collection*

11. Click "*Save*" and "*OK*"

Experiment Setup - C:\My Documents\Omnic\param\SmartTranKBr.exp

Collect | Bench | Quality | Advanced | Diagnostic | Configure

Estimated time for this collection: 00:12:43

No. of scans: 512
Resolution: 4.
Data spacing: 0.482 cm-1
Final format: %Transmittance
Correction: H2O and CO2

☐ 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 OMNI-Transmission Accessory

File Handling
☐ Save automatically   ☐ Save interferograms
Base name: MSE161
C:\My Documents\Omnic\autosave\MSE1610013.sp

Background Handling
○ Collect background before every sample
○ Collect background after every sample
◉ Collect background after 120 minutes
○ Use specified background file:
[   ] Browse...
☐ Collect 64 scans for the background

Experiment description:
This is the default experiment file for the Smart OMNI-Transmission Accessory

Help | Open | Save | Save As   OK | Cancel

VI. Collect Background – 1/2

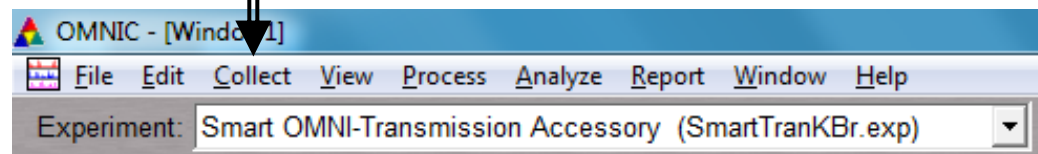1. Open the ***Chamber Cover***

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***

   OMNIC - [Window 1]
   File   Edit   Collect   View   Process   Analyze   Report   Window   Help
   Experiment: Smart OMNI-Transmission Accessory (SmartTranKBr.exp)

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

   Confirmation

   Background
   Please prepare to collect the background spectrum.

   ✔ OK   ✗ Cancel

VI. 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
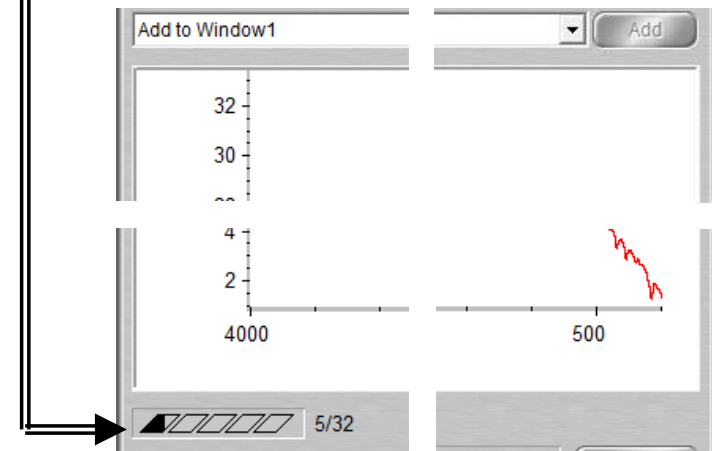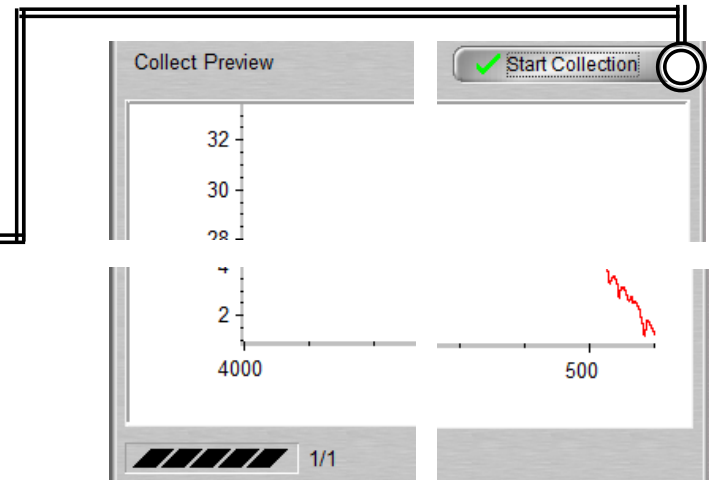
VII. Collect Sample – 1/2

1. Open the *Chamber Cover*
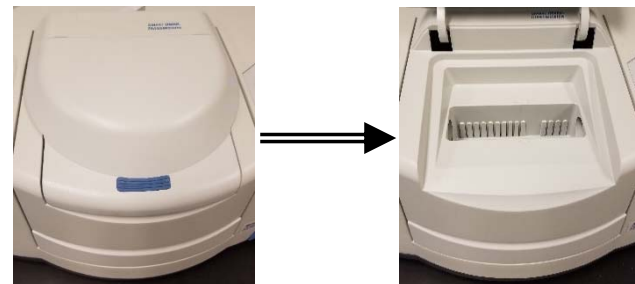
2. Insert your sample into Chamber via:
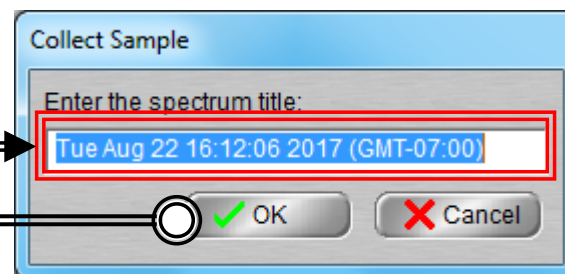
   - Collar Holder

   - Pellet Holder

   *Insert Holders in *Notch* ⧗

3. Select *Collect -> Collect Sample*

   OMNIC - [Window 1]
   File Edit Collect View Process Analyze Report Window Help
   Experiment: Smart OMNI-Transmission Accessory (SmartTranKBr.exp)
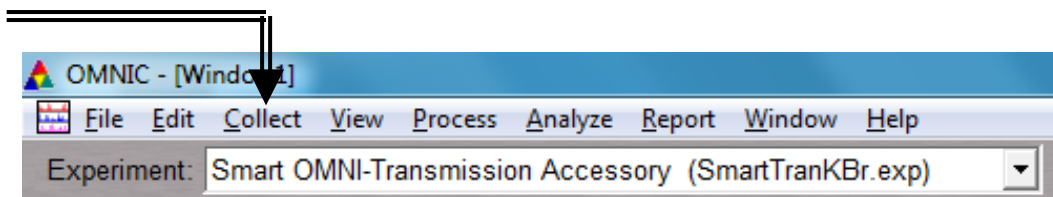
4. Enter *Spectrum Title* and

   click *OK* to *Collect Sample*

   Collect Sample
   Enter the spectrum title:
   Tue Aug 22 16:12:06 2017 (GMT-07:00)
   ✔ OK   ✘ Cancel

VII. 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

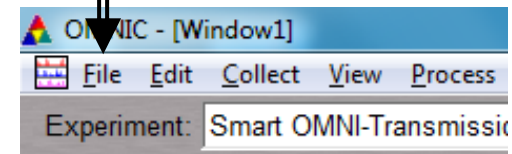9. Click **Yes** to add to data to current Window
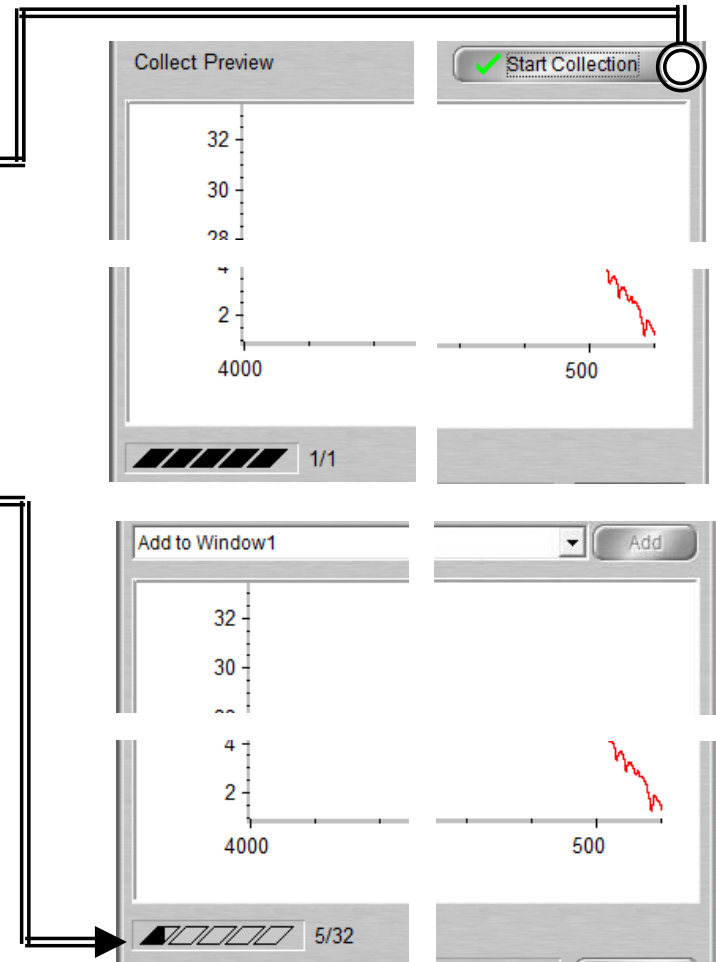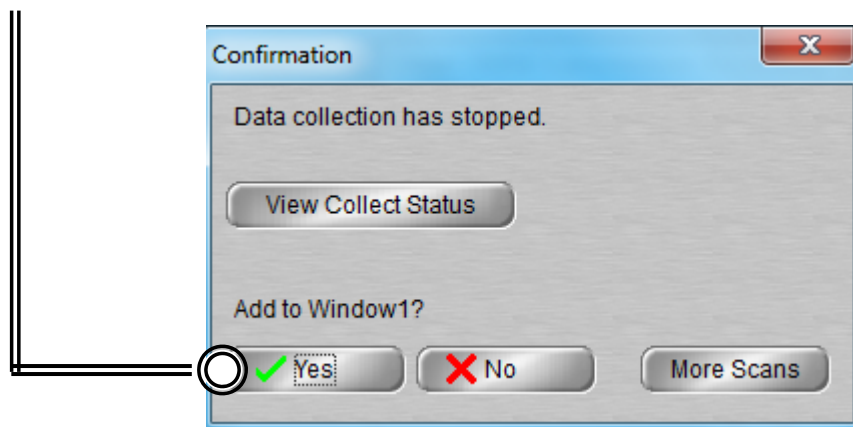
VIII. Saving Data – 1/1

1. Specific spectra can be selected using the [icon] selection tool at the bottom of window and clicking on it or selecting [No spectra selected] it from the dropdown box
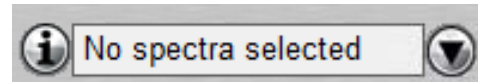
2. Multiple spectra can be selected/deselected by holding down the **Ctrl** key and clicking spectra

3. Click **File -> Save** to save a spectrum (e.g. default is SPA) using the current filename

    OMNIC - [Window1]
    File Edit Collect View Process
    Experiment: Smart OMNI-Transmissic

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)
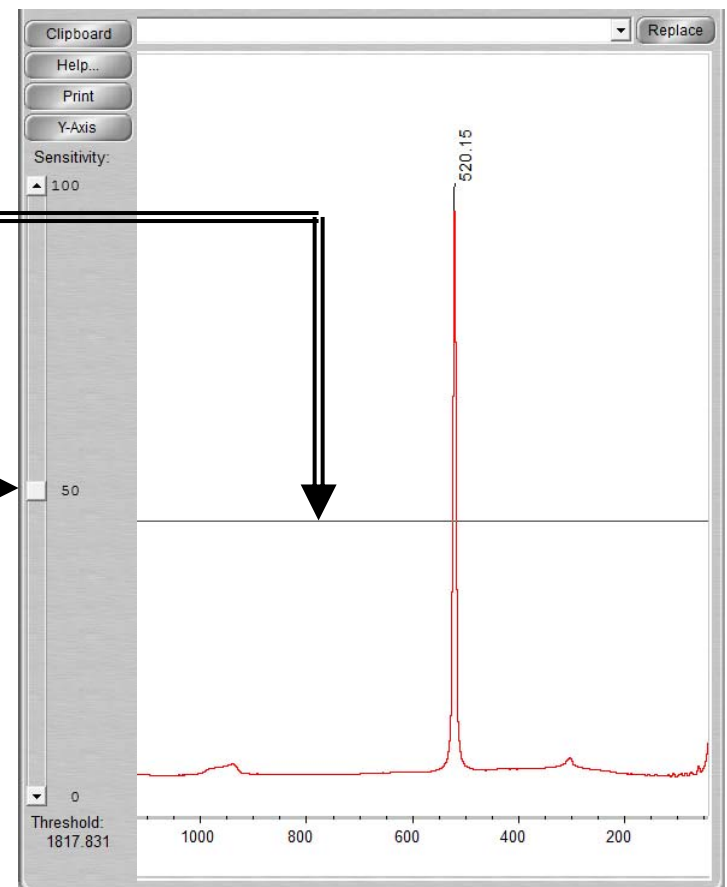
IX. Peak Identification – 1/1
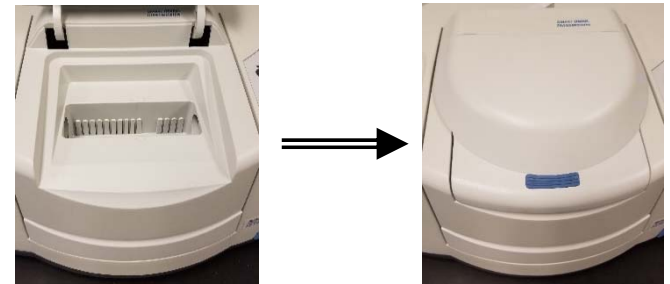
1. Click on "*Find Pks*" button at the top

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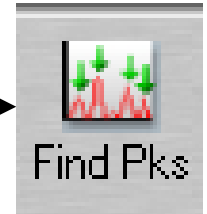
2. Click the spectrum window to adjust the *Threshold* position on where peaks are to be considered

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

X. Cleanup – 1/1

1. Remove *Sample Holders* from the *Chamber*
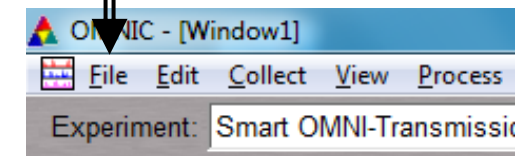
2. Close the *Chamber Cover*

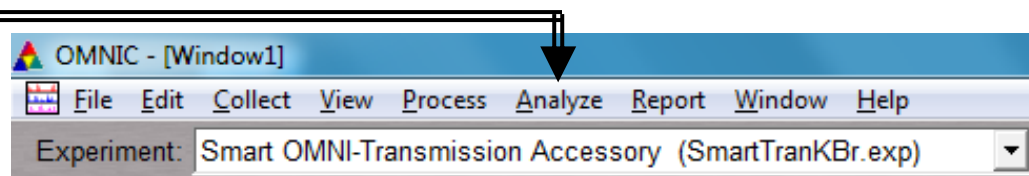3. Clean up *Sample Holders* and any tools used and return back to storage box

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

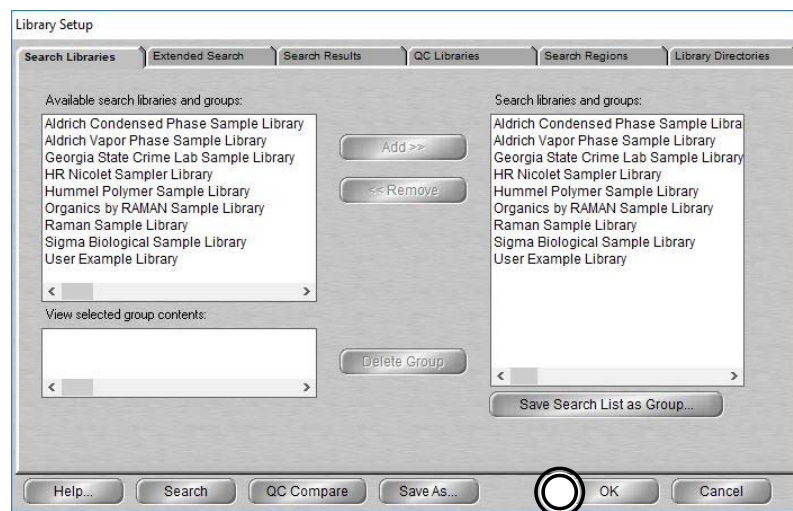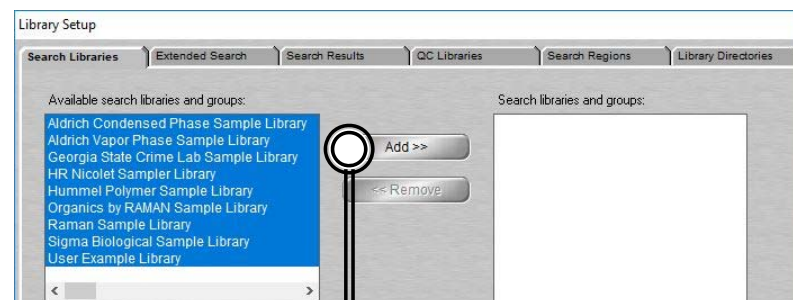5. Log off of your ENGR account

XI. Library Search – 1/3

1. Click *Analyze* and select *Library Setup*
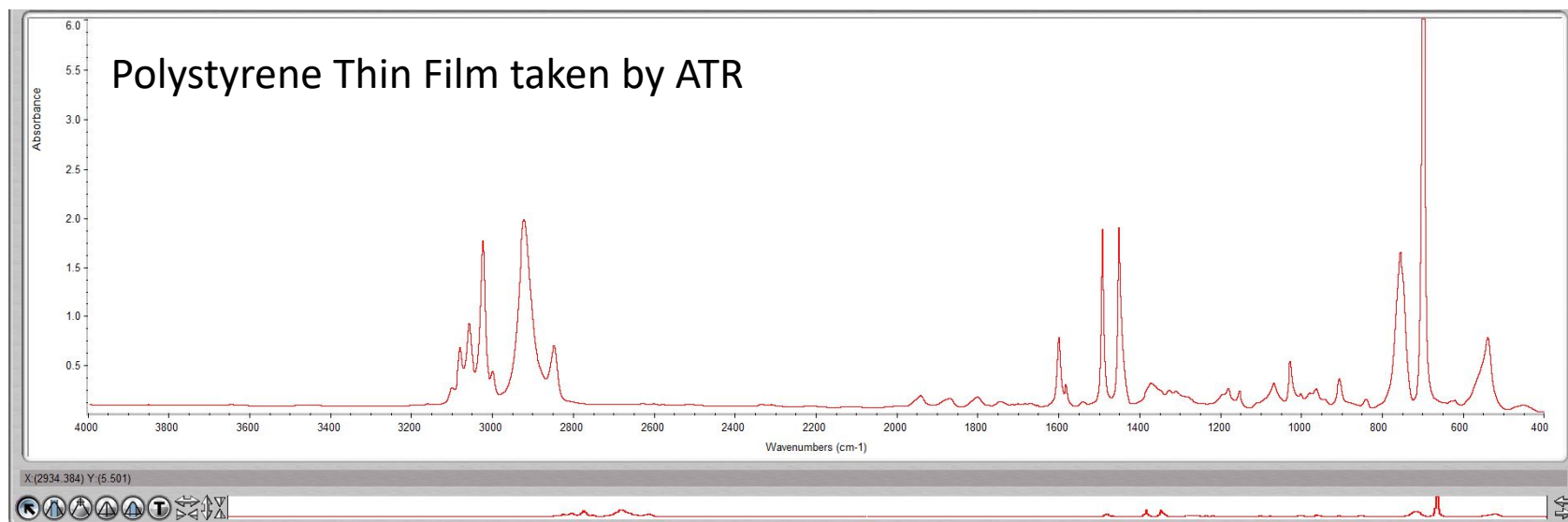
2. Select desired *Libraries* or select all

3. Click *Add >>*

4. Click *OK*

XI. Library Search – 2/3
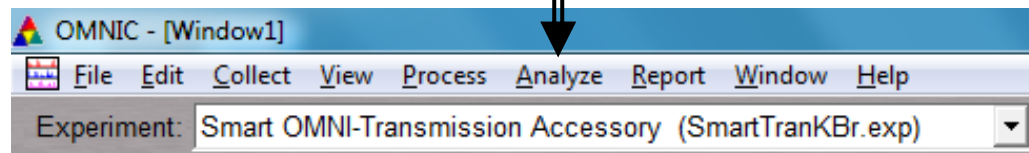
5. Select the desired spectra you wish to search for a library match

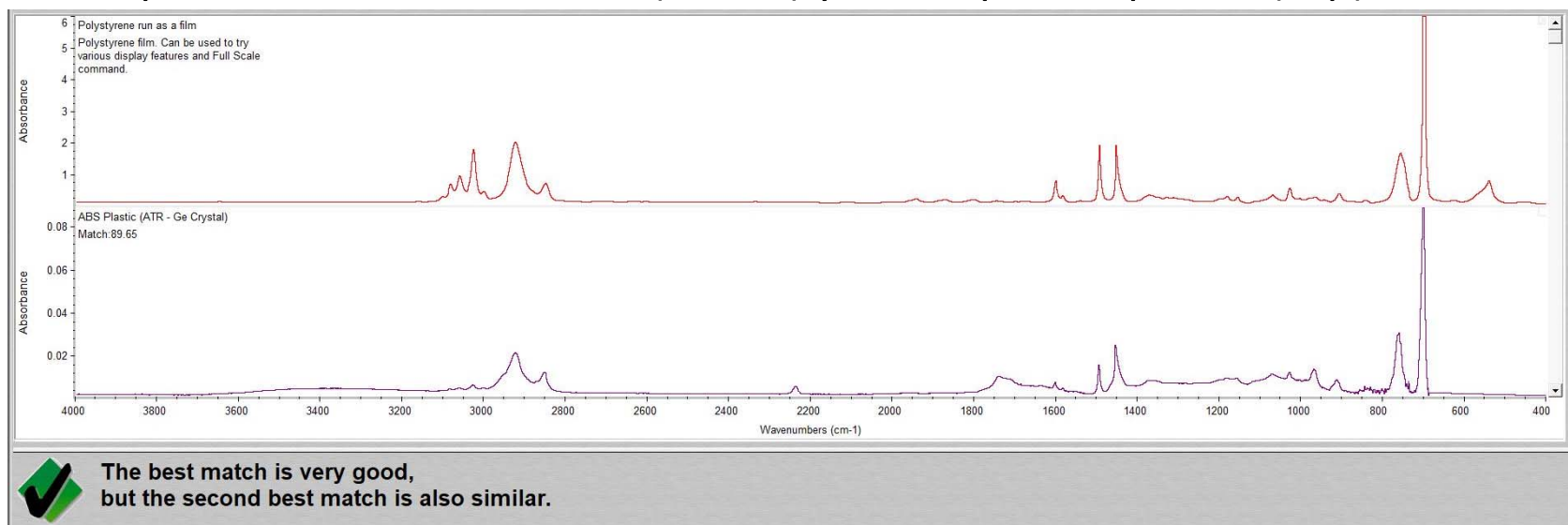Polystyrene Thin Film taken by ATR

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