FT-IR Training Notebook: Transmission

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

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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

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**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

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/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**
II. Pellet Press – 4/x

14. Slowly release the torque by unthreading **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/x

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 **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/x

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**

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

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

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

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

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