FT-IR Training Notebook: ATR

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 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

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
   - *Volatile Cover* – for volatile liquids

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
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*
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%**
- **CO₂ levels** – checks for CO₂ absorption, recommend **ON** at **50%**
- **H₂O levels** – checks for H₂O 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
   - *CO₂ levels* – checks for CO₂ absorption, recommend ON at 50%
   - *H₂O levels* – checks for H₂O 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**
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.

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**

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**...

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

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