FT-IR Training Notebook: ATR

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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
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 \( \leq 2 \)

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
II. Initiate Software – 4/10

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 **Screen wheel** is set to **Open**
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
II. Initiate Software – 9/10

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**

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 **Volatile Cover** over sample to reduce 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 then 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

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

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
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
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...