Raman Training Notebook

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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)
    - SID
    - email
- Coordinate a time with the lab manager for training
- Schedule a 1 hour block on Faces for your training
Raman Operation

I. Initiate Software
II. Selecting Sample Holder
III. Sample Holder Alignment
IV. Collection Parameters
V. Collect Background
VI. Collect Sample
VII. Collect Sample Holder
VIII. Saving Data
IX. Background Subtraction
X. Manual Baseline Correction
XI. Cleanup
I. Initiate Software – 1/3

1. Enter your ENGR Username and Password at the Windows log-in screen
   a. NOTE: This is **NOT** your R’SPACE Username and Password
   b. ENGR Passwords are 14 characters with at least 3 of the following: Uppercase Letter, Lowercase Letter, Number, and Special Character

2. Double left-click on the OMNIC for Dispersive Raman

3. A dialogue will appear indicating the installed accessory

4. Confirm that it is correct: 180-degree

5. Select the "**Default – DXR 180-degree accessory**"

6. Click **OK**
I. Initiate Software – 2/3

7. A dialogue showing “Resetting Step Motors” may appear

8. Select **Collect -> Experiment Setup** at the top window

9. Select the **Advanced** tab

10. Uncheck Autofocus option “Before Collection”

11. Click **Save**
I. Initiate Software – 3/3

12. Select the **Bench** tab

13. Turn on Laser by clicking on the “**Off**” box and select “**On**”

14. A dialogue box will appear

15. Set the Laser power to “**50**” as a suitable level by clicking on the Value field

16. The laser will only be emitted when the enclosure is closed
II. Selecting Sample Holder – 1/1

1. Depending on your sample, the sample holder and preparation will vary...
2. Several sample holders are available for use located in the storage container
3. **CLEAN UP AFTER EACH USE AND WIPE DOWN!**

<table>
<thead>
<tr>
<th>Sample Holder</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>NMR Tube Holder</td>
<td>Large quantities of liquid and powder</td>
</tr>
<tr>
<td>Capillary Tube Holder</td>
<td>Small quantities of liquid and powder</td>
</tr>
<tr>
<td>Thin Film Holder</td>
<td>Thin film or thin planar samples</td>
</tr>
<tr>
<td>Flat Bulk Sample Holder</td>
<td>Flat bulk samples too large for the Thin Film holder</td>
</tr>
<tr>
<td>Bulk Sample Holder</td>
<td>Irregular shaped bulk samples</td>
</tr>
</tbody>
</table>
III. Sample Holder Alignment – 1/3

1. Depending on your Sample Holder, the appropriate Focus position will be different

2. Double-Click and enter the following *preliminary* settings for your Sample Holder

<table>
<thead>
<tr>
<th>Accessory</th>
<th>180-degree</th>
<th>1481</th>
<th>194</th>
<th>130</th>
</tr>
</thead>
<tbody>
<tr>
<td>Focus (18.81 mm)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Side to side (2.46 mm)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Up/Down (2.46 mm)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>NMR Tube</th>
<th>Capillary Tube</th>
<th>Thin Film</th>
<th>Flat Bulk</th>
<th>Bulk</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Focus</strong></td>
<td>2542</td>
<td>2432</td>
<td>2356</td>
<td>2397</td>
<td>2571</td>
</tr>
<tr>
<td><strong>Side to Side</strong></td>
<td>168</td>
<td>168</td>
<td>168</td>
<td>168</td>
<td>168</td>
</tr>
<tr>
<td><strong>Up/Down</strong></td>
<td>150</td>
<td>160</td>
<td>180</td>
<td>151</td>
<td>108</td>
</tr>
</tbody>
</table>
3. Position the NMR and Capillary Tube sample holders using the alignment line as a guide and tighten the holder knob

4. Insert the 50 wt% H$_2$SO$_4$ reference samples

5. Focus on any relevant peak(s) by moving the cursors

6. Achieve the max signal by optimizing the settings

Sensitivity of the settings:
- ± 1 Focus = ± 10 µm
- ± 1 Side to Side = ± 20 µm
- ± 1 Up/Down = ± 20 µm
III. Sample Holder Alignment – 3/3

7. Place your sample into position for the Thin Film, Bulk Flat, and Bulk sample holder

8. Position the Thin Film and Bulk Flat sample holder using the alignment line as a guide and tighten the holder knob

9. The Bulk sample holder does not require any additional alignment

10. Achieve the max signal by optimizing the settings

Sensitivity of the settings:

± 1 Focus = ± 10 μm
± 1 Side to Side = ± 20 μm
± 1 Up/Down = ± 20 μm
IV. Collection Parameters – 1/2

1. Select the **Collect** tab

2. Estimated time for collection is shown here

3. Change the **Collect exposure time (sec)** to improve signal-to-noise (start at 2 sec)
   
   Note: If CCD overflow occurs, increase # of exposures instead

4. Keep the **Preview exposure time (sec)** to low value (e.g. 1 sec)

5. Change the # of **Sample exposures** to desired value and check **Estimated time** (e.g. 32)
   
   Note: For weak signals, set longer **Collect exposure times** instead of increasing # of **Sample exposures**
IV. Collection Parameters – 2/2

6. Change the **Background exposures** to desired value (e.g. 32)

7. Select **Maximum age for background** as **1000** minutes under **Background Handling**

8. Select **Shifted spectrum (cm-1)** as **Final format**

9. Select **Fluorescence** as the **Correction** to correct for baseline curvature due to fluorescence
   - Default is 5\textsuperscript{th} order polynomial
   - Select **None** if you prefer to perform corrections yourself

10. Select **OK** when done selecting options
V. Collect Background – 1/1

1. Select **Collect -> Collect Background**

   *Note:* Background measures the response of each pixel in the CCD with camera shutter closed, and does not take into account the sample holder background signal

2. A dialogue box will appear indicating the background exposure progress
VI. Collect Sample – 1/1

1. Select **Collect -> Collect Sample**

2. Enter **title** for collected spectrum, click **OK**

3. A live display of the collection will appear

4. The following shows the collect status indicator during your collection
   - The spectrum has passed all quality checks
   - The spectrum has failed a spectral quality check but not serious
   - There is a problem with quality of spectrum, correct problem before collecting the spectrum again

5. The current background will be automatically subtracted from sample data (and any correction applied)

6. Choose to add the collected spectrum in window specified, click **Yes**
1. If your sample is transparent or are using a secondary sample holder like a glass NMR or capillary tube, you will need to collect background spectrum from primary sample holder.

2. Remove sample from the sample holder and insert an empty glass NMR or capillary tube or glass slide if applicable.

3. Select Collect -> Collect Sample

4. Enter title for collected spectrum, click OK.

5. A live display of the collection will appear.

6. The following shows the collect status indicator during and after your collection:
   - The spectrum has passed all quality checks.
   - The spectrum has failed a spectral quality check but not serious.
   - There is a problem with quality of spectrum, correct problem before collecting the spectrum again.

7. The current background will be automatically subtracted from sample data.

8. Choose to add the collected spectrum in window specified.
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. Background Subtraction – 1/3

1. Perform a background subtraction to remove effects of a sample holder.

2. Select the sample spectrum (A) first, then hold Ctrl key and select the reference spectrum (B).

3. “Two spectra selected” appears at top.

4. Click Process -> Subtract.
IX. Background Subtraction – 2/3

5. The subtract window appears with the sample spectrum (A) in top pane and reference spectrum (B) below it

6. Click and move \textit{Adjustment Bar} to achieve desired subtraction

7. Click \textit{Coarser} or \textit{Finer} to increase or decrease the sensitivity of adjustments

8. Click \textit{Factor} button to enter in a specific factor value for subtraction
IX. Background Subtraction – 3/3

9. Click on top dropdown to determine where the new subtracted spectra will appear.
10. Click Add to add to desired window.
11. If new window is selected, you will need to name it.
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. Manual Baseline Correction – 1/3

1. If your spectra has a shifted, tilted, or curving baseline, you can choose to correct it manually using the software.

2. Select the spectrum you wish to correct.

3. To correct a baseline, click **Process -> Baseline Correct**.
XI. Manual Baseline Correction – 2/3

4. Select an algorithm from the drop-down list box near upper-left corner of window
   - **Linear**: For tilted or elevated baselines
   - **Spline**: For curved baselines
   - **Polynomial**: Suitable for all, with max order of 6

5. Select **Auto Y** to have baseline points coincide with points on spectrum

6. Click as few as necessary to straighten pronounced curves or slopes in upper pane

7. Add corrected spectrum to new spectral window
XII. Manual Baseline Correction – 3/3

8. You may choose to let the software automatically correct a tilted baseline.

9. Select the spectrum you wish to correct.

10. To correct a baseline, click **Process -> Automatic Baseline Correct**.

11. Click **Edit -> Options**.

12. Set the **Polynomial Order and Number of Iterations** in the **Process** options.
X. Cleanup – 1/1

1. Remove the sample and holder from the stage

2. Clean up the sample holder and return back to cabinet

3. Select `Collect -> Experiment Setup` and click `Bench` tab

4. Click on Laser and turn to “Off”

5. Reset the position of stage to:
   - Focus = 2000
   - Side to side = 168
   - Up/Down = 100

6. Click on `File -> Exit` to shut down the software

7. Log off of your ENGR account