Raman Training Notebook

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

October 28, 2023 (rev. 2.3)

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. 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. Peak Identification

XI. Manual Baseline Correction

XII. Cleanup

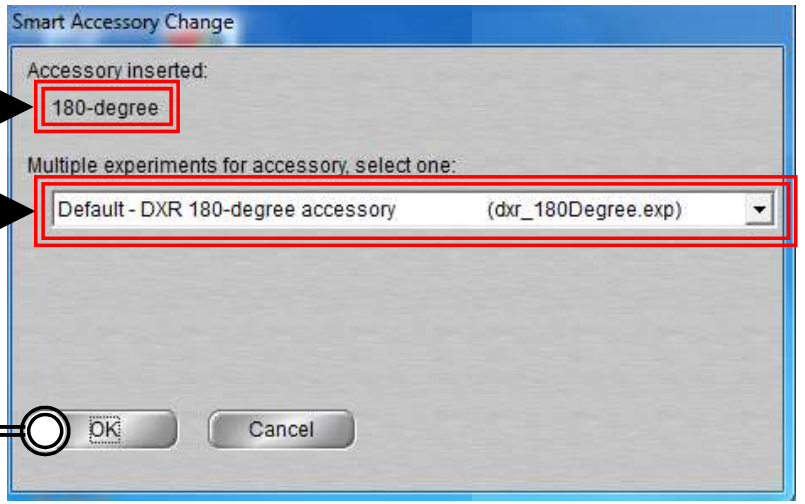
I. Initiate Software – 1/3

1. Double left-click on the ***OMNIC for Dispersive Raman*** icon

2. A dialogue may appear indicating installed accessory and confirm that it is correct: 180-degree

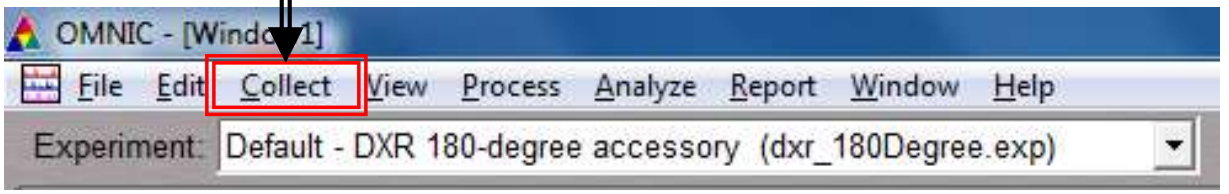
3. Select the "***Default – DXR 180-degree accessory***"

4. Click ***OK***



5. A dialogue showing "***Resetting Step Motors***" may appear

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



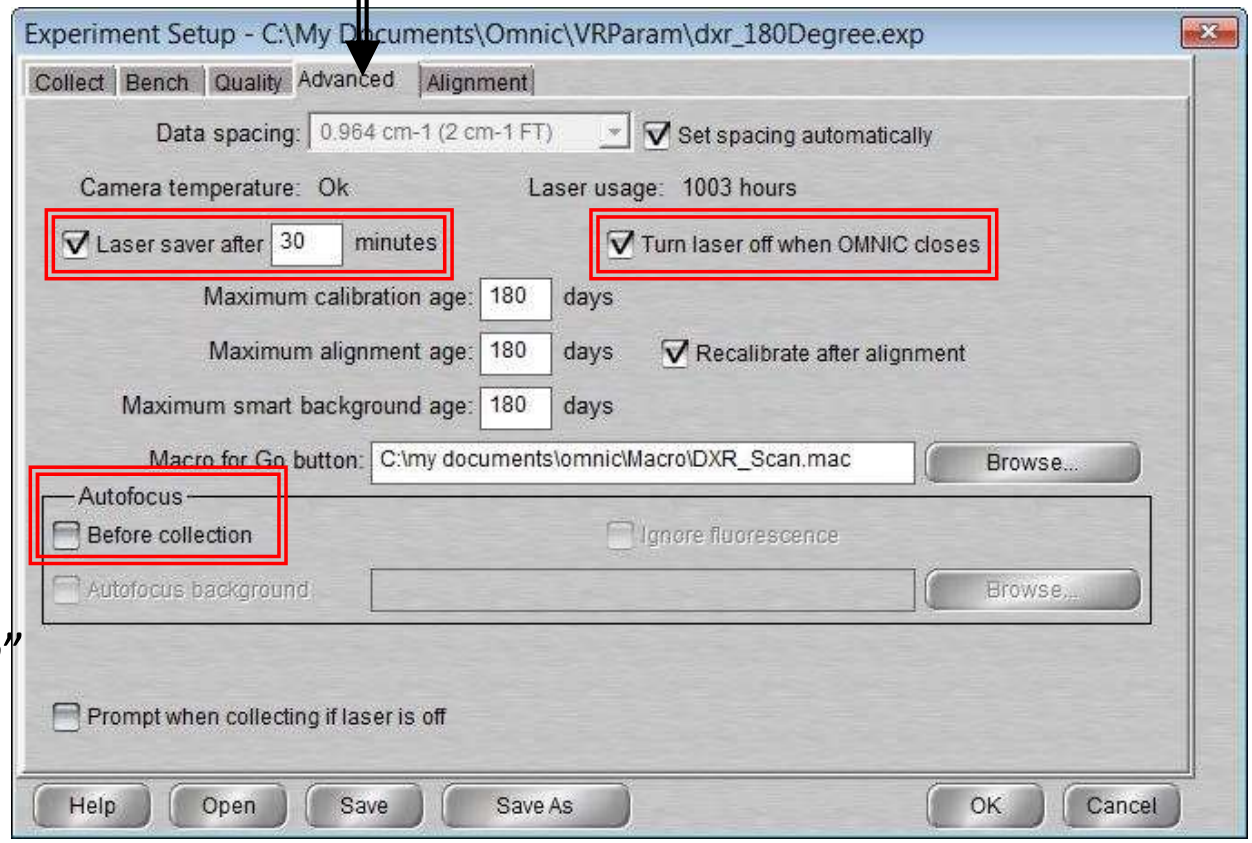
I. Initiate Software – 2/3

7. Select the ***Advanced*** tab

8. Confirm "***Laser saver"*** is checked and set to "***30 minutes***"

9. Confirm "***Turn off laser***" is checked

10. Confirm Autofocus option "***Before Collection***" is unchecked

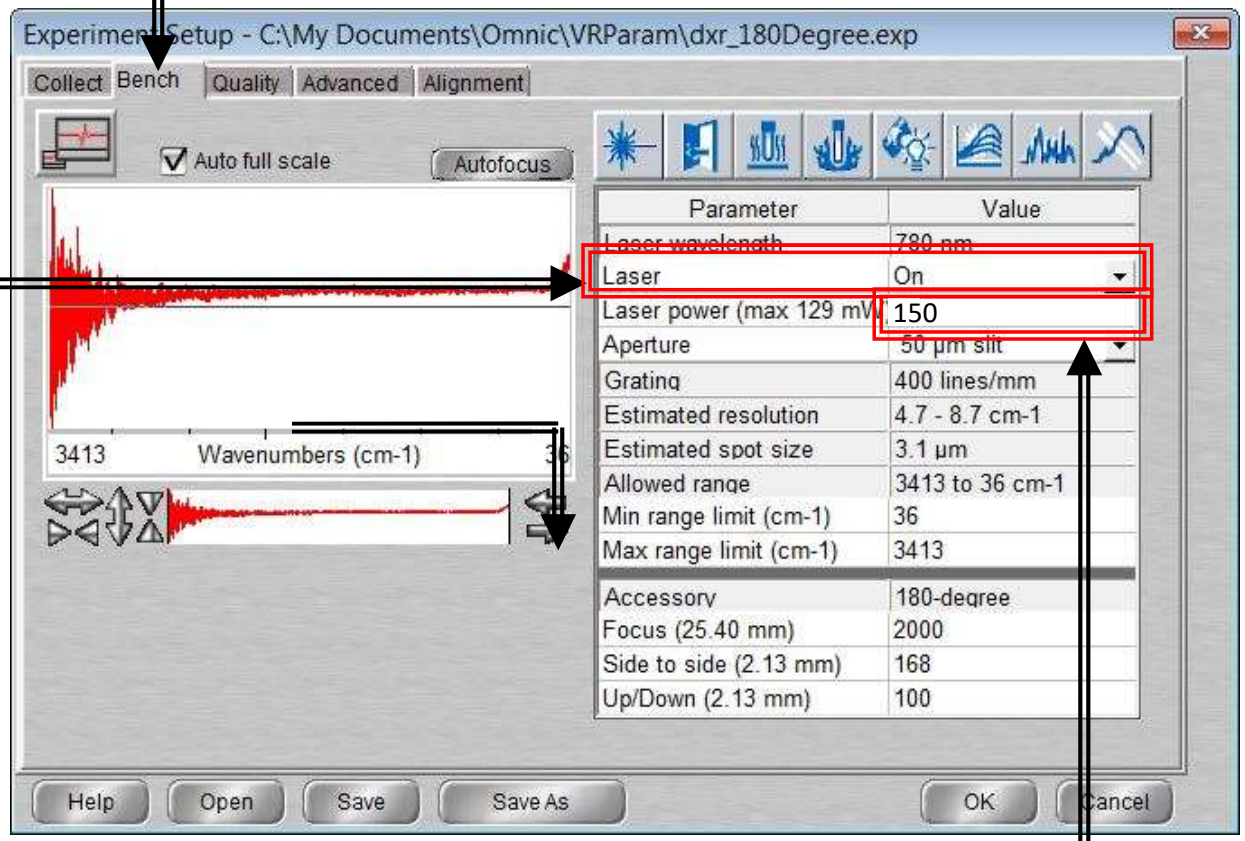


I. Initiate Software – 3/3

11. Select the ***Bench*** tab

12. Turn on ***Laser*** by clicking and selecting "***On***"

13. An info box will appear

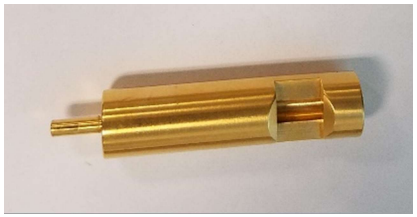


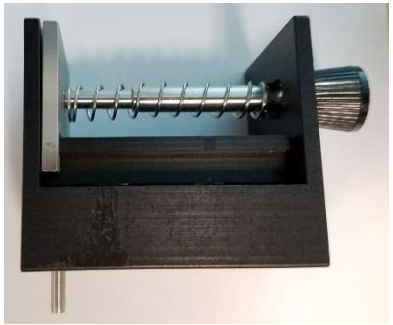
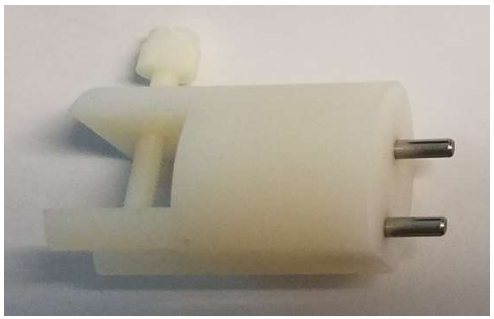
14. Set Laser power to "**150"** as a suitable level by clicking and entering value

15. The laser will only be emitted when the enclosure is closed

II. Sample Holder – 1/2

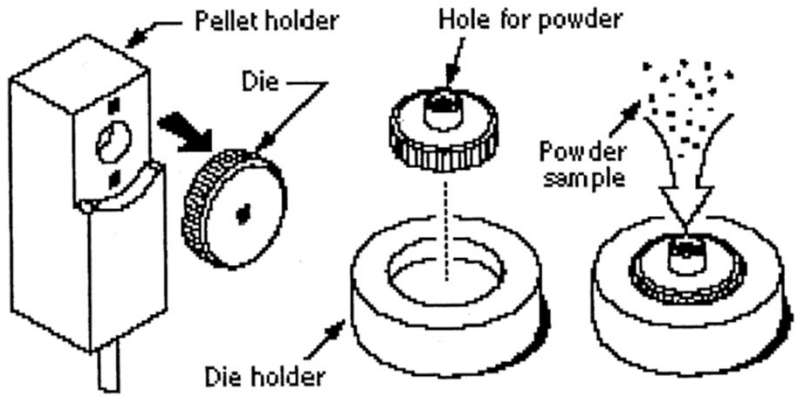
1. Depending on your sample, the sample holder and preparation will vary…
2. Several sample holders are available for use located in the storage container
3. CLEAN UP AFTER EACH USE AND WIPE DOWN!

NMR Tube Holder	Capillary Tube Holder	Thin Film Holder
		
Large quantities of liquid and powder	Small quantities of liquid and powder	Thin film or thin planar samples

Flat Bulk Sample Holder	Bulk Sample Holder
	
Flat bulk samples too large for the Thin Film holder	Irregular shaped bulk samples

II. Sample Holder : Pellet – 2/2

1. Remove the ***Die*** from ***Pellet Holder***

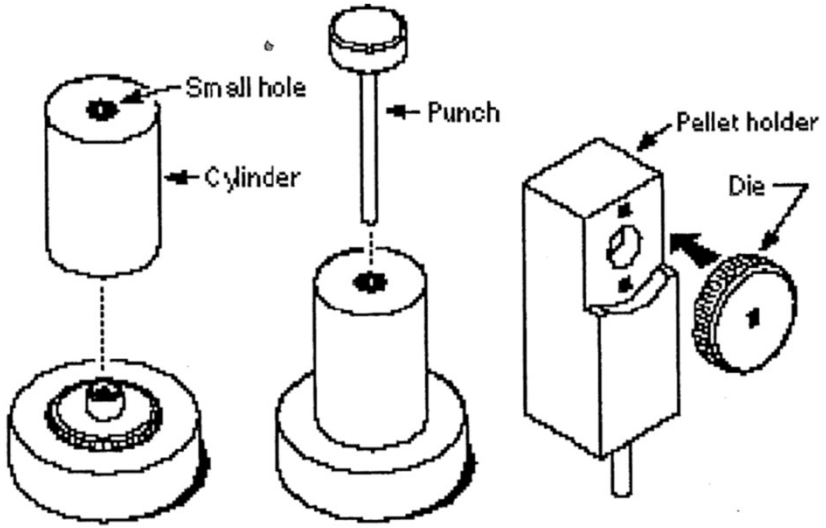


2. Place ***Die*** in ***Die Holder***

3. Fill hole in ***Die*** with the powdered sample

4. Place ***Metal Cylinder*** over ***Die*** with the small hole facing up

5. Insert ***Punch*** into hole at top of ***Cylinder*** and apply force with hand to push ***Punch*** down as far as it will go



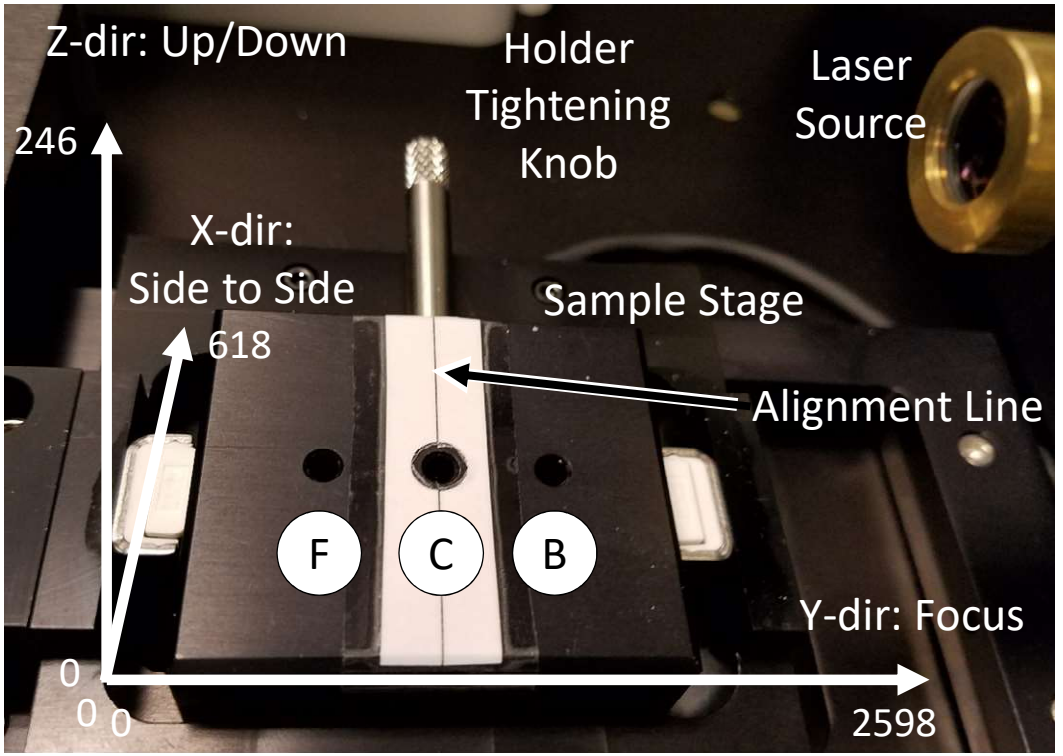
6. Remove ***Cylinder*** from ***Punch Die***

7. Mount ***Die*** on ***Pellet Holder*** so stem of die is inserted into hole in the ***Pellet Holder***

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

Accessory	180-degree
Focus (18.81 mm)	1481
Side to side (2.46 mm)	194
Up/Down (2.46 mm)	130



	NMR Tube	Capillary Tube	Thin Film	Flat Bulk	Bulk	Pellet Holder
Position(s)	**C**	**C**	**C**	**C**	**C + B**	**C**
Focus	2542	2432	2356	2397	1570	2475
Side to Side	168	168	168	168	168	168
Up/Down	150	150	150	150	150	150

III. Sample Holder Alignment – 2/3

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_2SO_4 reference samples

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

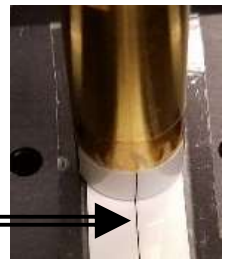
6. Achieve the max signal by optimizing the settings

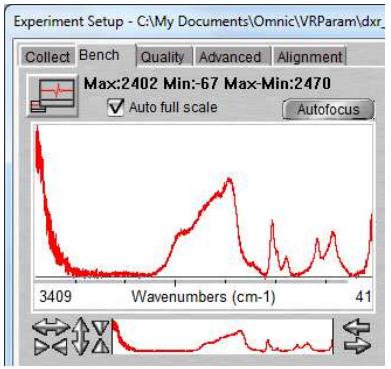
Sensitivity of the settings:

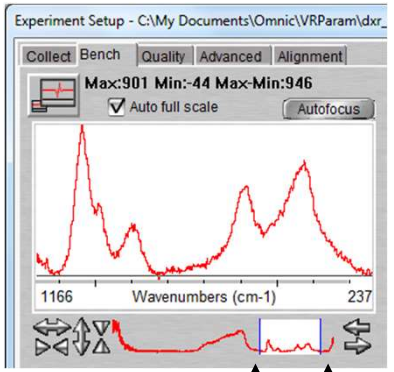
± 1 Focus = ± 10 μm

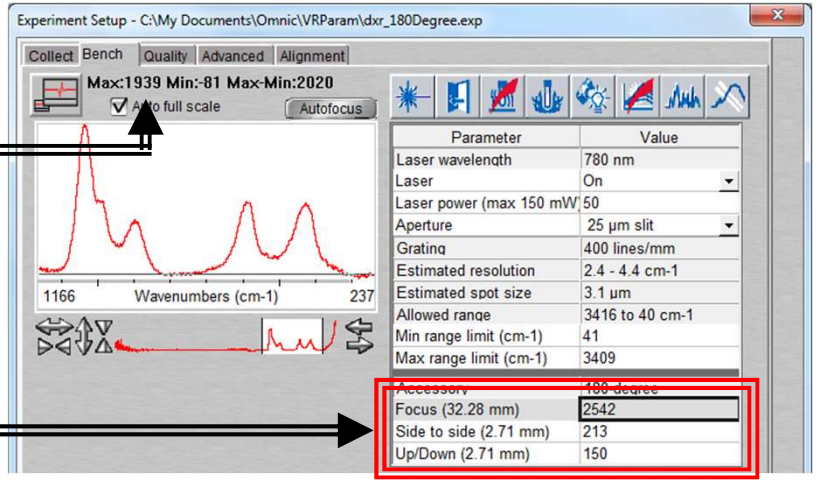
± 1 Side to Side = ± 20 μm

± 1 Up/Down = ± 20 μm









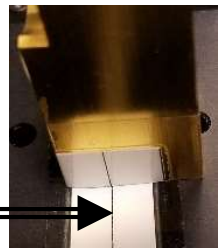
III. Sample Holder Alignment – 3/3

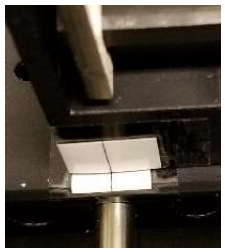
7. Place your sample or Si reference sample into position for the Thin Film, Bulk Flat, and Bulk sample holder

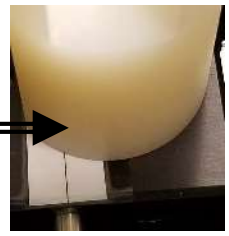
8. Position the Thin Film and Bulk Flat sample holder using the alignment line as a guide and tighten the holder knob

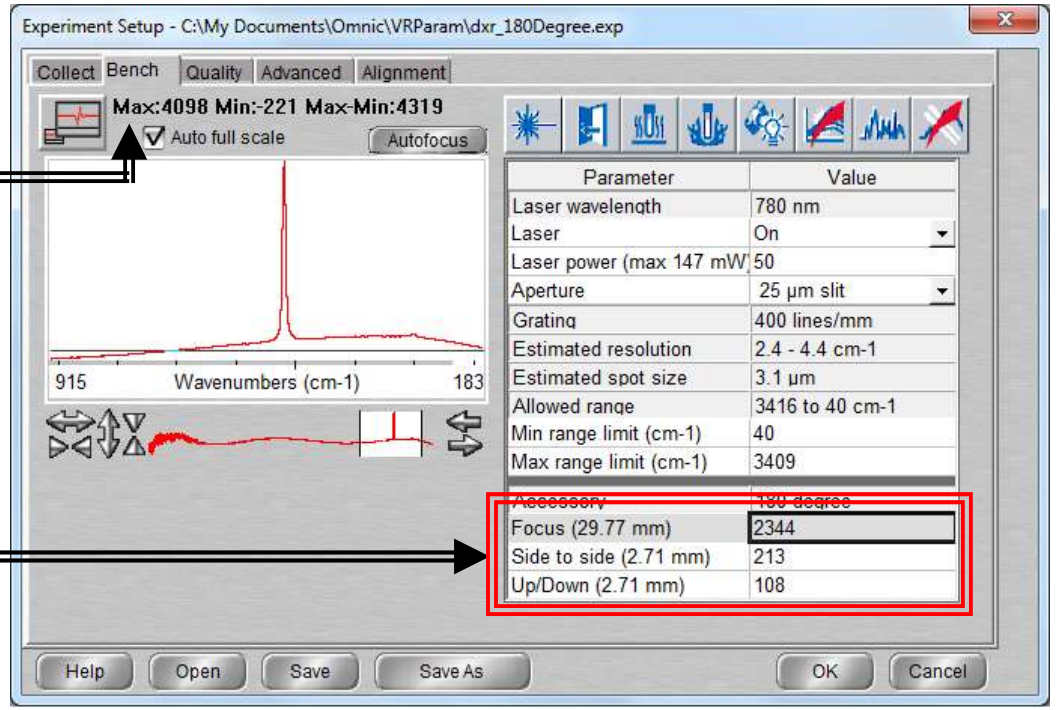
9. The Bulk sample holder does not require any additional alignment

10. Achieve the max signal by optimizing the settings









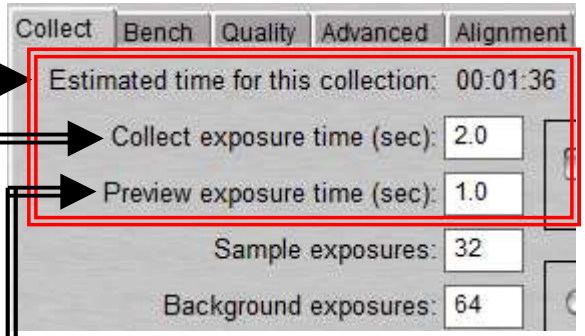
Sensitivity of the settings:

± 1 Focus = ± 10 µm

± 1 Side to Side = ± 20 µm

± 1 Up/Down = ± 20 µm

IV. Collection Parameters – 1/6



1. Select the ***Collect*** tab

2. Estimated time for collection is shown here
   - Remember to check this value after changing collection parameters!

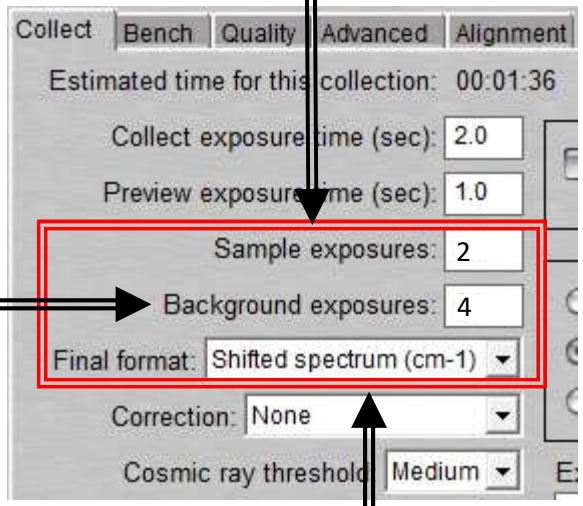
3. Determine the ***Collect exposure time (sec)*** – recommend starting at 2 sec
   - Increase value to improve signal-to-noise ratio until desired result is achieved
   - Note: If CCD overflow occurs, increase # of exposures instead

4. Determine the ***Preview exposure time (sec)*** – recommend 1 sec
   - This value also determines the exposure time for the live display on Bench tab

IV. Collection Parameters – 2/6

5. Determine # of ***Sample exposures*** – recommend starting at 2
   - Increase value to improve signal-to-noise ratio
   - Note: For weak signals, set longer ***Collect exposure times*** instead of increasing # of ***Sample exposures***

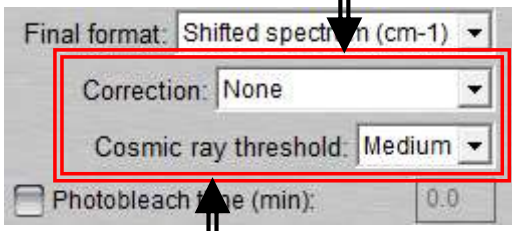
6. Determine # of ***Background exposures*** – recommend greater value than # of ***Sample exposures***
   - Increase value to avoid having background add noise to the spectrum



7. Select the ***Final Format*** – recommend Shifted spectrum (cm^{-1})
   - Raman spectrum (nm): nanometer vs Raman intensity
   - Raman spectrum (cm^{-1}): wavenumber vs Raman intensity
   - Shifted spectrum (cm^{-1}): shifted wavenumber vs Raman intensity
   - Photoluminescence (nm): nanometer vs emission

IV. Collection Parameters – 3/6

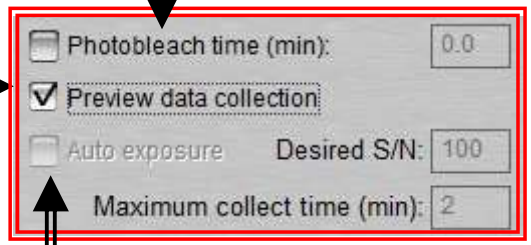
8. Select desired ***Correction*** – recommend None
   - Raman Efficiency – corrects for intensity differences related to frequency
   - Fluorescence – corrects the effect on baseline curvature due to fluorescence
     - Use Polynomial – specify a polynomial of order 1 to 6 for the operation
       - Default is 5th order polynomial
     - Use Reference File – specify a reference spectrum that contains fluorescence artifacts you want removed
       - Reference files and spectra must have the same resolution and final format



9. Select desired ***Cosmic ray threshold*** – recommend Medium
   - None – will not reject cosmic ray spikes
   - Low – rejects random cosmic ray spikes with low intensity and higher intensity
   - Medium – rejects random cosmic ray spikes of least moderate intensity
   - High – rejects only random cosmic ray spikes of high intensity

IV. Collection Parameters – 4/6

10. Select desired ***Photobleach time (min)***
    - Fluorescence is an emission process and causes some samples to give off a strong, broad emission when illuminated by the excitation laser
    - Photobleaching can be used to reduce sample fluorescence and may be occurring if baseline offset decreases on successive exposures
        - Estimate the appropriate photobleach time by observing how long it takes the baseline of the spectrum to reach a steady state in the Bench tab

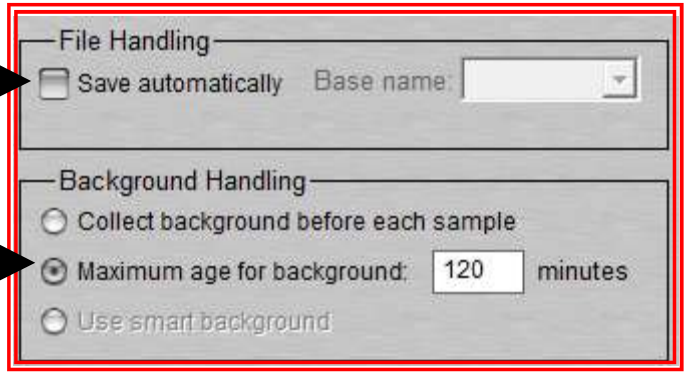


11. Confirm ***Preview data collection*** – is checked ***ON***

12. Confirm ***Auto exposure*** is not checked

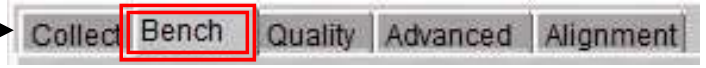
IV. Collection Parameters – 5/6

13. Confirm ***Save automatically*** is not checked



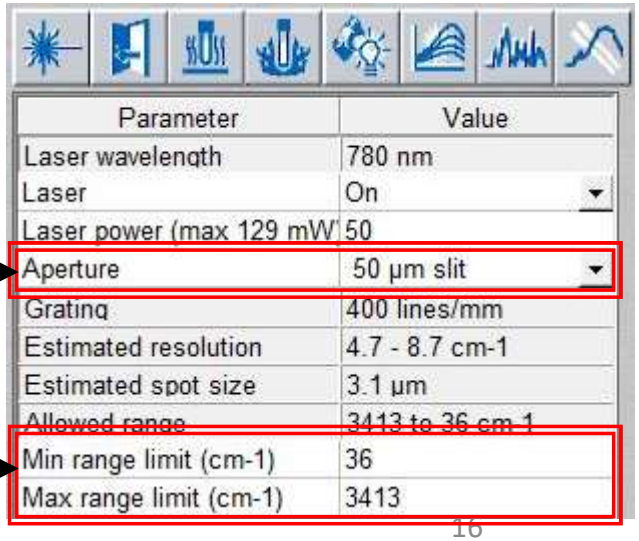
14. Determine ***Background Handling***
    - Collect background before each sample
    - ***Maximum age for background*** – default choice and recommend 120 minutes
    - Use smart background

15. Select the ***Bench*** tab



16. Determine desired ***Aperture***
    - 50 um slit – higher signal – recommended default
    - 25 um slit – higher resolution

17. Adjust the desired scan range limits

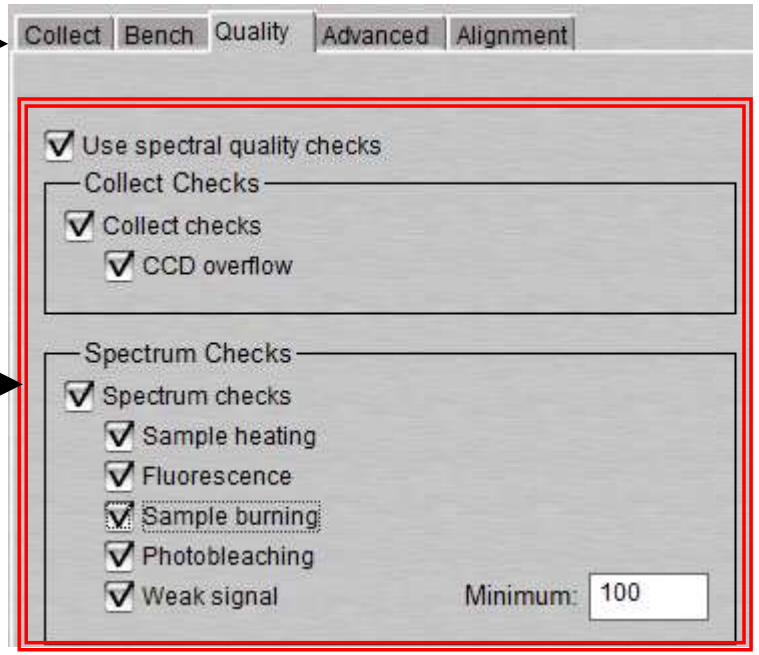


IV. Collection Parameters – 6/6

18. Select the ***Quality*** tab

19. Confirm all checks are selected

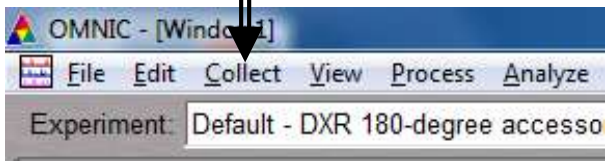
20. Correct the following if issues are found:
    - ***CCD overflow*** – reduce exposure time or decrease aperture size
    - ***Sample heating*** – reduce laser power, exposure time, and # of exposures
    - ***Fluorescence*** – apply correction
    - ***Sample burning*** – reduce laser power
    - ***Photobleaching*** – include ***Photobleaching time*** in ***Collect*** tab
    - ***Weak signal*** – check focus is correct, increase exposure time, increase aperture size, and increase laser power



21. Click ***Save*** and ***OK***

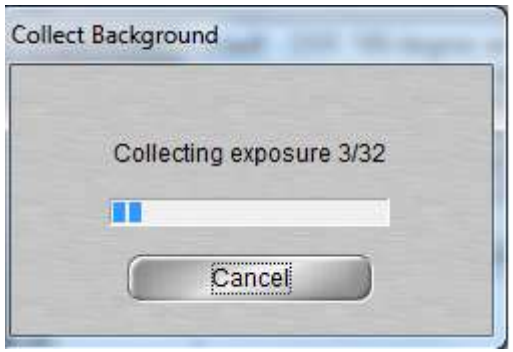
V. Collect Background – 1/1

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



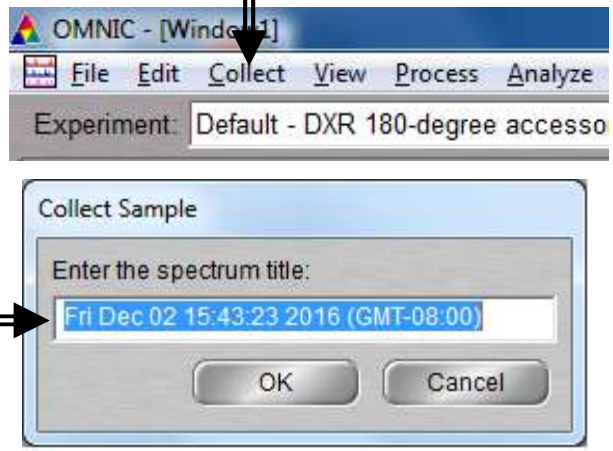
Note: Background measures the response of each pixel in the CCD with camera shutter closed, and does not take into account the sample holder background signal

2. A dialogue box will appear indicating the background exposure progress



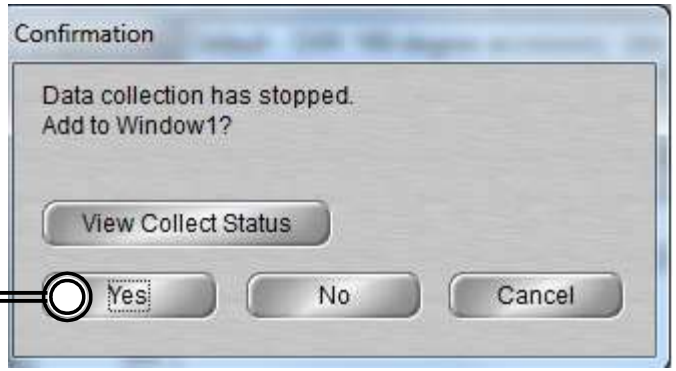
VI. Collect Sample – 1/1

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



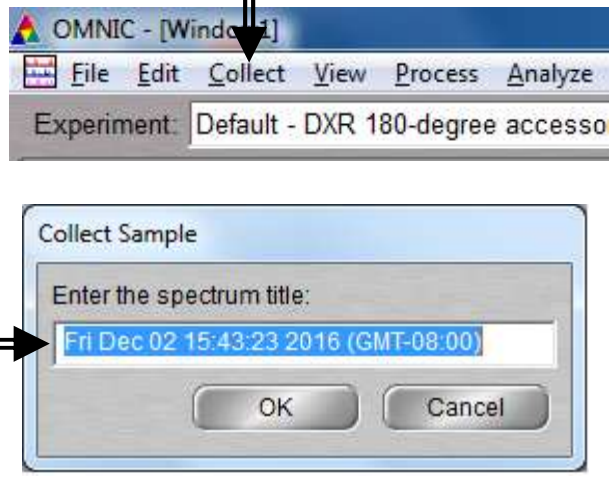
- The spectrum has passed all quality checks
- The spectrum has failed a spectral quality check but not serious
- There is a problem with quality of spectrum, correct problem before collecting the spectrum again

5. The current background will be automatically subtracted from sample data
6. Choose to add the collected spectrum in window specified, click ***Yes***

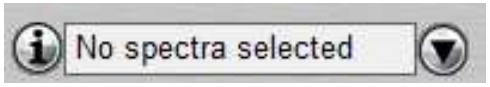


VII. Collect Sample Holder – 1/1

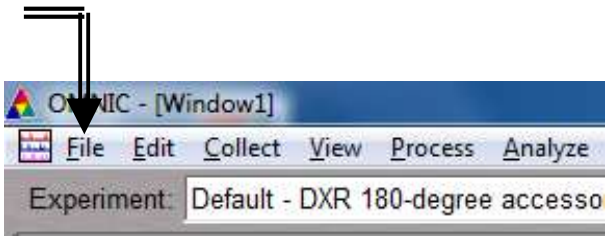
1. If your sample is transparent or are using a secondary sample holder like a glass NMR or capillary tube, you will need to collect background spectrum from primary sample holder
2. Remove sample from the sample holder and insert an empty glass NMR or capillary tube or glass slide if applicable
3. Select ***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 

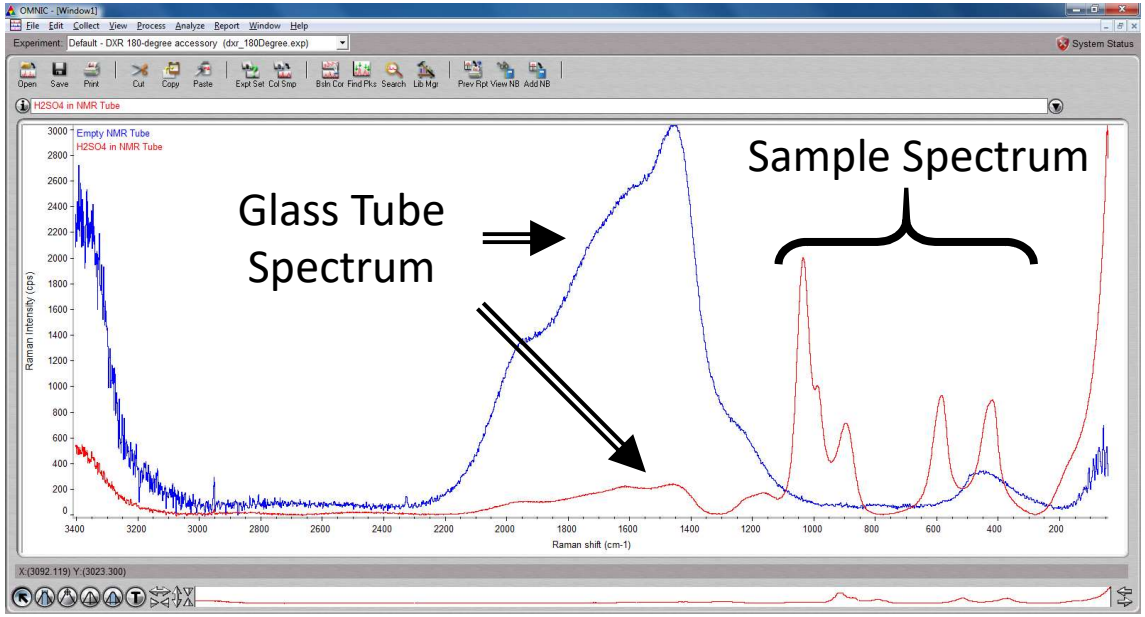
from dropdown
2. Multiple spectra can be selected/deselected by holding down the ***Ctrl*** key and clicking spectra
3. Click ***File -> Save*** to save spectrum (e.g. default is SPA) using the current filename
4. Click ***File -> Save As*** to save a spectrum into another file type (e.g. CSV or TIFF)



5. Click ***File -> Save Group*** to save more than one spectrum as a group in one file having file extension .SPG to open later

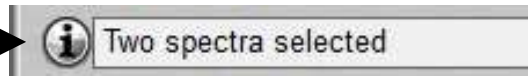
IX. Background Subtraction – 1/3

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

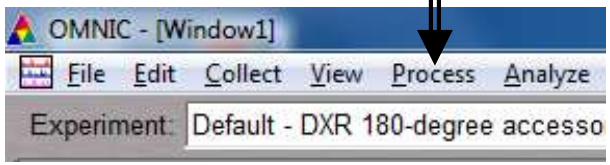


2. Select the sample spectrum (A) first, then hold ***Ctrl*** key and select the reference spectrum (B)

3. "***Two spectra selected***" appears at top

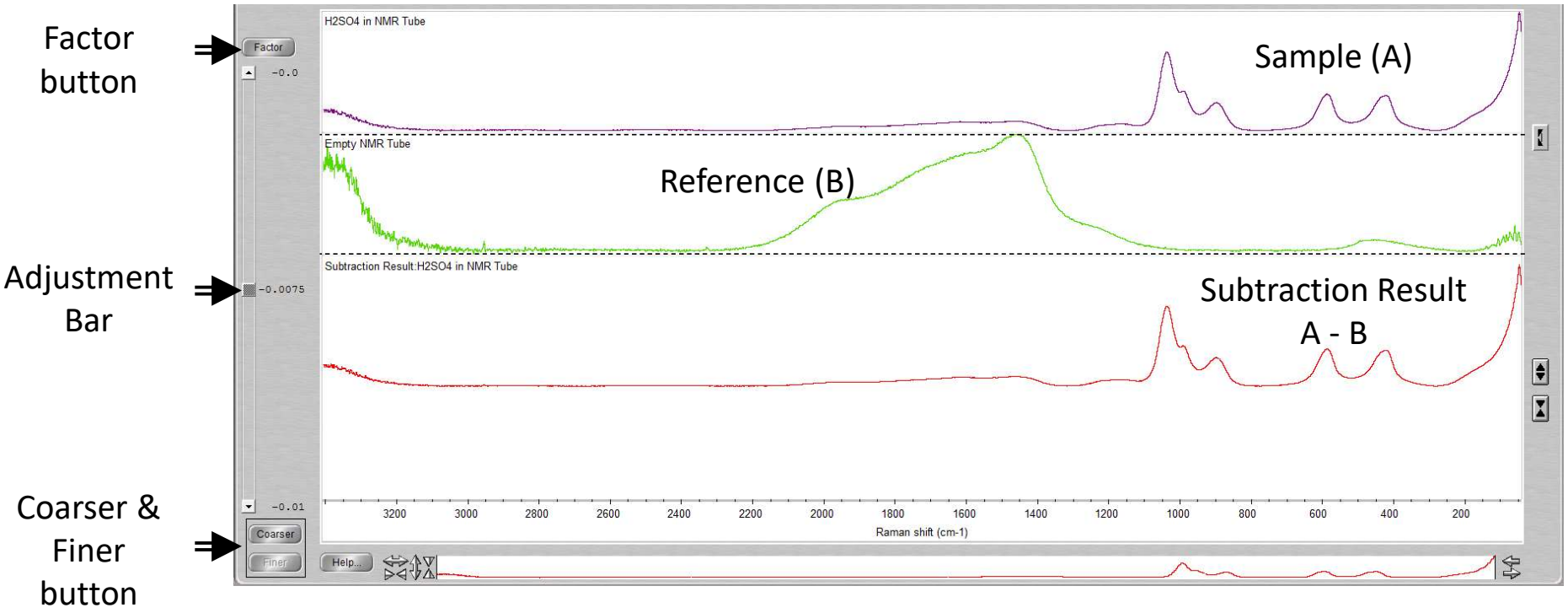


4. Click ***Process -> Subtract***



IX. Background Subtraction – 2/3

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



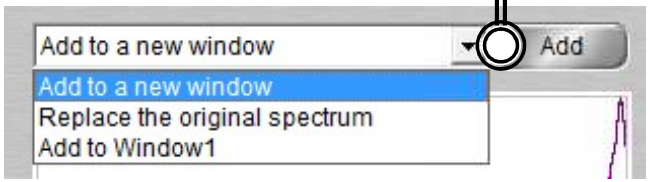
6. Click and move ***Adjustment Bar*** to achieve desired subtraction

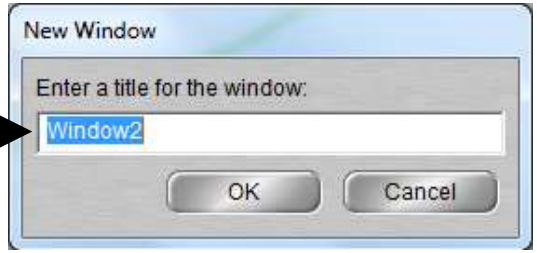
7. Click ***Coarser*** or ***Finer*** to increase or decrease the sensitivity of adjustments

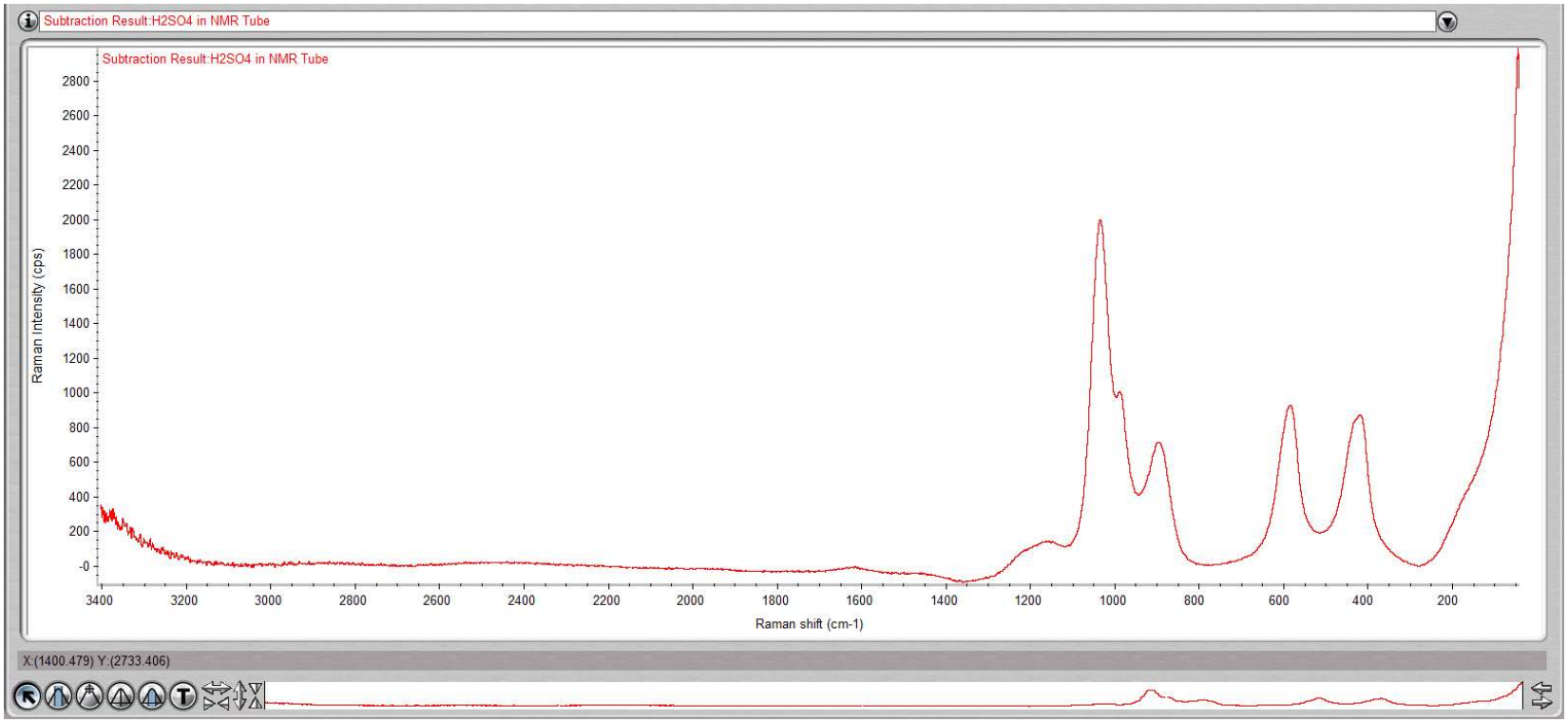
8. Click ***Factor*** button to enter in a specific factor value for subtraction

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

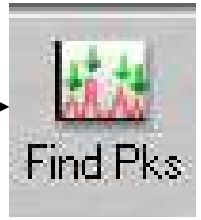






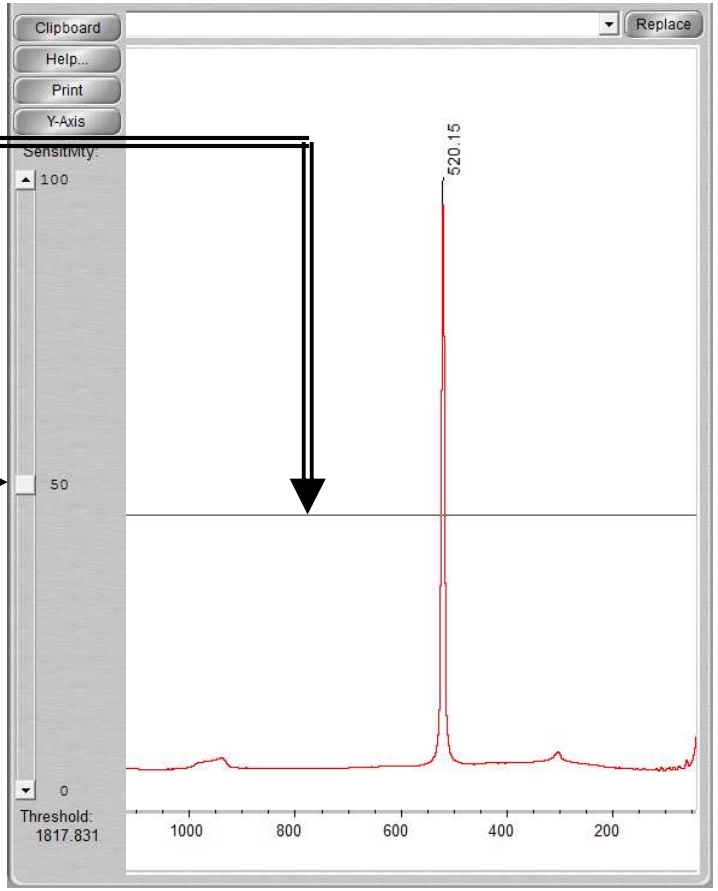
X. Peak Identification – 1/1

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

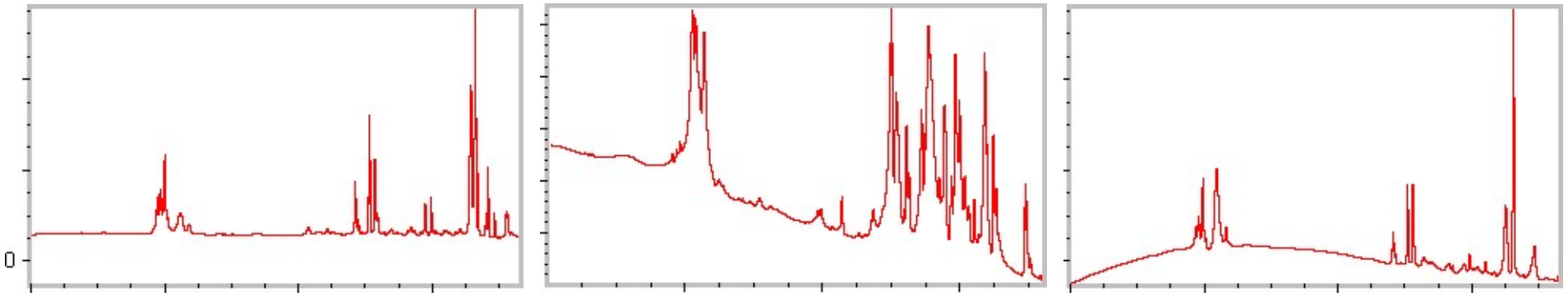
3. Adjust the ***Sensitivity*** button to separate peaks from noise



XI. Manual Baseline Correction – 1/3

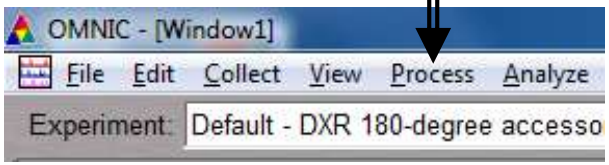
1. If your spectra has a shifted, tilted, or curving baseline, you can choose to correct it manually using the software

Shifted (above zero) | Tilted | Curved



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

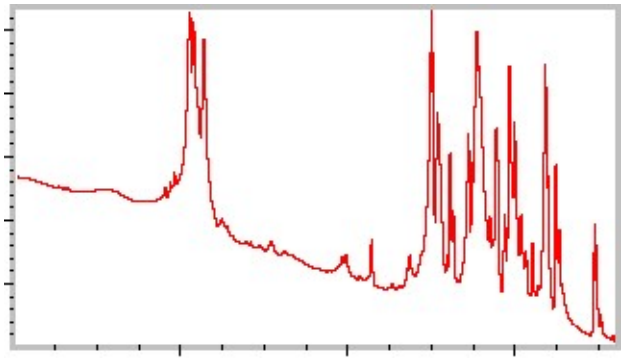
6. Click as few as necessary to straighten pronounced curves or slopes in upper pane

7. Add corrected spectrum to new spectral window

XI. Manual Baseline Correction – 3/3

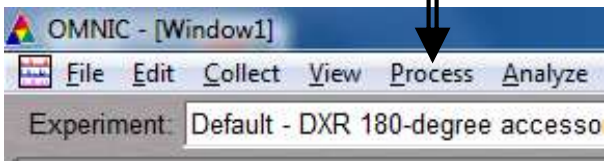
8. You may choose to let the software automatically correct a tilted baseline

Tilted



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

XII. 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