Nicolet RAMAN Procedure

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Getting Started

1.) Sign in on the Log Sheet. Record your full name, advisor's name, department, start time, and your recharge number.

2.) Log into your personal IR/RAMAN account.

3.) Open OMNIC by double clicking the icon:
   Click "OK" for error messages that
   appear (should be two of them).

Instrument Setup

1.) Select "Default-Transmission" located under the *Experiment* drop down menu. This puts you into IR mode in order to align the beam.

2.) Make sure: (a) that the sample chamber is empty (IMPORTANT: before opening the sample chamber make sure the shutters are closed – levers are up. Failing to do so will cause damage to the source and detector), the sample chamber door and main door are closed, and shutters to the source and detector are open (levers are down).

(b) Next go to the *Bench* tab: **Detector** should be set on DTGS KBr
                                                 Beam Splitter should be set on KBr
                 You should also see a signal in the window:
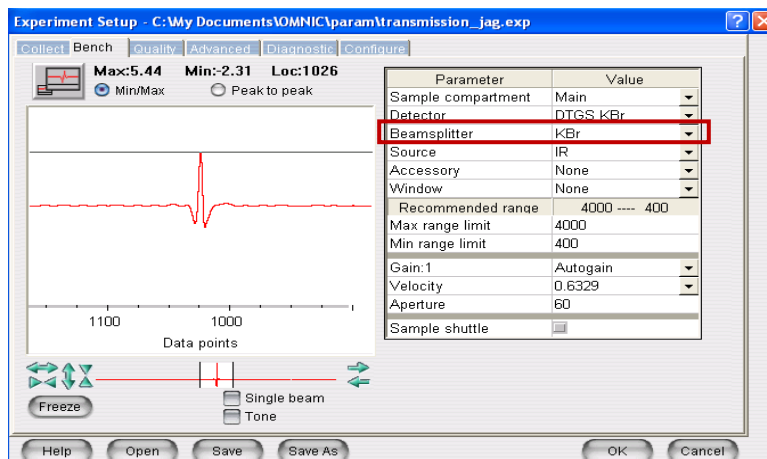
IF no signal is seen like the one above make sure the shutters are
open (levers are down). If they are closed (levers are up), then open
them and wait a few minutes for signal to appear (**Gain** should be at
1 or 2).

 IF still no signal, then do (a) and if still no THEN do (b) the following:

a.) Make sure the beamsplitter is set on KBr (above: red box)

b.) Go to *Diagnostics* tab and hit the "Align…" button to optimize the
intensities. Please wait until the Align Bench comment box has
disappeared from the screen before doing anything else.

Exit from the *Experiment Setup* box by pressing "OK."

3.) **Physically change the beam splitter** from KBr to CaF$_2$: (<u>IMPORTANT</u>: Please
handle these with <u>extreme care</u>).
    (a) Turn the lever inside the chamber *counter-clockwise* until it hits about 4
       o'clock, this releases the KBr splitter
    (b) Place the CaF$_2$ splitter where you removed the KBr splitter, and vice versa.
    (c) Turn the lever inside the chamber clockwise until it's almost at 12 o'clock

Beam Splitter
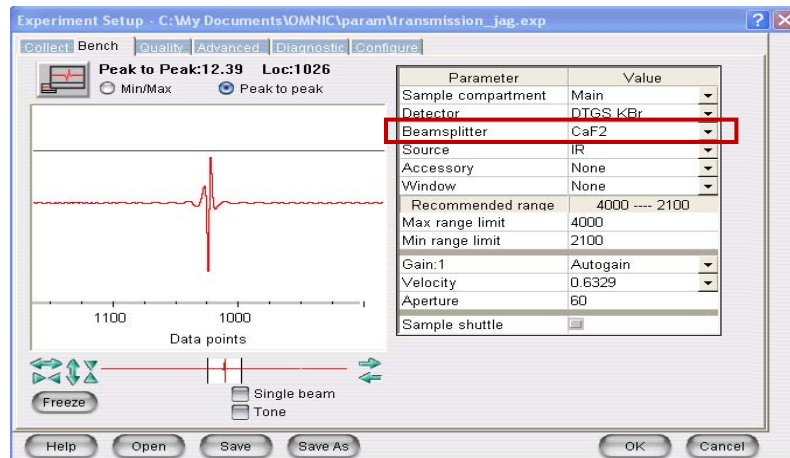Storage Area

          Unlock/Release Position   Lock/Hold Position

4.) Now **change the beam splitter to CaF$_2$ on the software**:
    (a) Go to *Collect => Experiment Setup => Bench* tab
    (b) Choose **CaF$_2$** from the *Beam Splitter* drop down menu
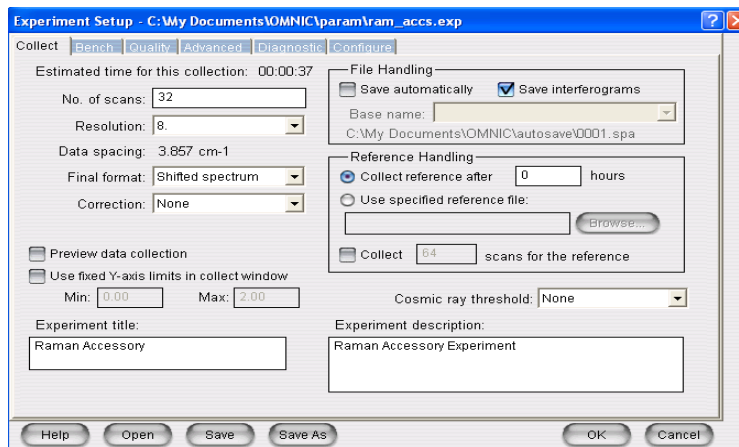   (c) Click *OK* to exit

5.) Go the menu at the top and choose
- (a) *Raman => Use Raman Accessory*
- (b) *Collect => Experiment Setup => Collect* tab and set
  - ● Number of scans = 32
  - ● Resolution = 8
  - ● Final Format = Shifted spectrum
  - ● Cosmic Ray Threshold = None

- (c) Next go to the *Advanced* tab
  - Zero Filling = None
  - Apodization = Happ-Genzel
 - Click *OK* to exit

Collecting a Spectrum

1.) Place the **sulfur sample** (located in "Raman Alignment" box in the 2nd drawer down to the right) in the *Raman sample chamber*.

2.) Go to *Collect => Experiment Setup => Bench* tab and check the following parameters:
- ● Laser = OFF
- ● White Light = OFF
- ● Sample Compartment = Main
- ● Detector = InGaAs
- ● Focus = 198
- ● Beam Splitter = CaF_2
- ● Gain = 1
- ● Velocity = 0.3165
- ● Aperture = 100
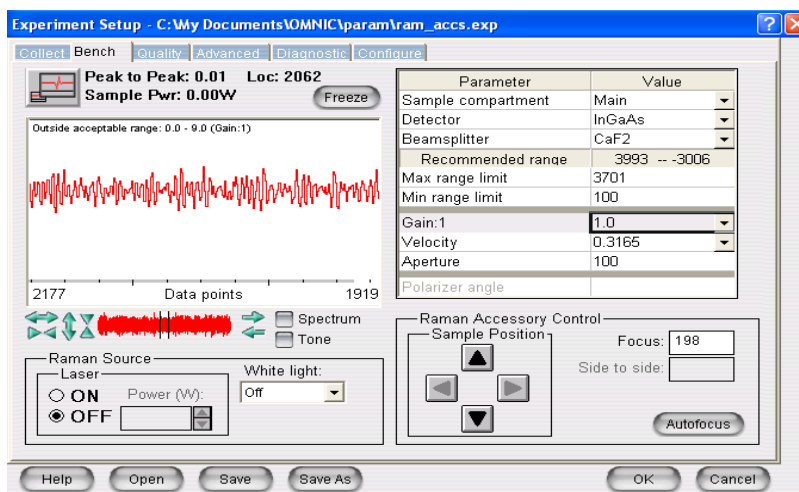
3.) Turn the Laser **ON** and set the Power to **0.804W**

4.) Go to *Diagnostic* tab and click *Align...* Wait for alignment to complete (~ 4 minutes)

5.) Go to the *Bench* tab and click/check the *Spectrum* box just below the spectrum
display; you should now see a Raman spectrum of sulfur with a Max. intensity at
~ 1500 if aligned.
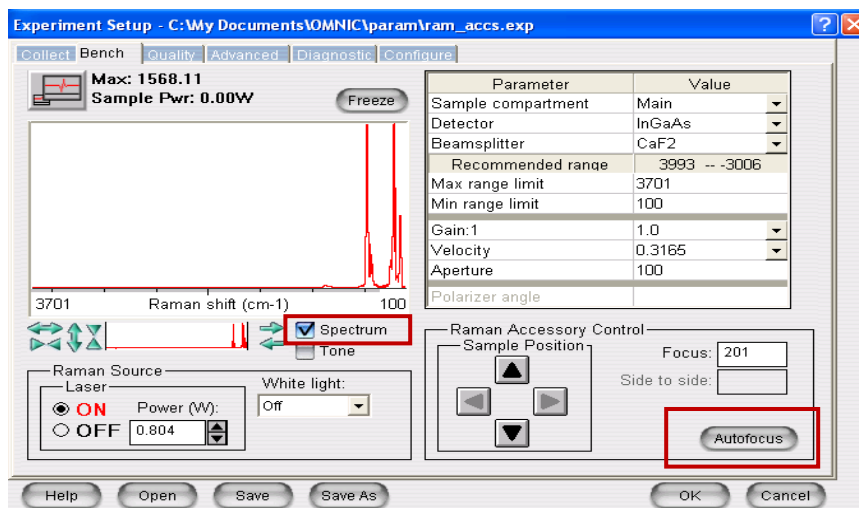
IF the intensity value <u>is much lower</u> than **1500** then do all of the following:

    a.) Click *Autofocus*; wait for it to finish
    b.) Manually maximize the intensity by using the allen wrench (labeled and
        located in the "Tools" drawer) through the right-hand side of the *Raman*
        sample chamber
    c.) Go to back to *Diagnostic* tab and click *Align...* Wait for alignment to
        complete (~ 4 minutes)
    d.) The intensity should now be ~1500, if not higher

Once the intensity is maximized click *OK* to exit

6.) Turn the Laser *OFF* by going to *Collect => Experiment Setup => Bench* tab. Replace
the sulfur sample with your own sample. Close and lock the chamber door.
**IMPORTANT!!! DO NOT OPEN THE RAMAN SAMPLE CHAMBER DOOR
UNLESS THE LASER IS <u>OFF</u>!!!**

7.) Turn the laser back *ON* and set the *Power* to 0.3W

See if the spectrum changes with time. If so, the sample may be experiencing
decomposition or it may be being burnt by the laser. This can often be seen by
looking at the sample and seeing discoloration (Do not forget to <u>turn the laser off</u>
before viewing your sample!!!)

8.) If no degradation of the sample is visible, then turn *the power up gradually to 0.9W*. Although this is a typical power level, any power level between 0.3 and 2.0W can be used. However, the higher the power level, the more chance of the sample burning along with decreasing the lifetime of the laser, so try to use a lower power level if possible.

9.) To **collect a spectrum**, go to *Collect => Collect Raman...*

10.) You will be prompted for a title and then asked if the sample is ready for Raman

11.) The collect Raman window will appear, and the number of scans taken is shown in the lower left corner in the OMNIC window.

12.) Once all the scans have been recorded, you will be asked whether you wish to add the spectrum to the window.
   (a) If the signal-to-noise ratio of the spectrum is satisfactory, then press the *YES* button.
   (b) If you wish to collect more scans, then press *More Scans*, in which you will then be asked how many additional scans you would like to collect.
   (c) If you do not want to keep the spectrum at all then just click *NO*.

13.) Repeat steps 6 – 12 for the rest of your samples. <u>Again, DO NOT forget to turn the laser off before opening the Raman Sample Chamber!!!</u>

14.) When you're finished with all your samples **THE INSTRUMENT MUST BE PUT BACK INTO DEFAULT FTIR TRANSMISSION MODE!!!** Please refer to the section "When you are Finished" on the following page.

<u>Saving Data</u>

There are two ways to save data; you can do one or the other or both.

1.) To save data as an **OMNIC file**:
   (a) Highlight the data by clicking on the spectrum that you want to save with the left mouse button (the desired spectrum will turn red).
   (b) Go to *File => Save* (change to your own directory; if you do not have one then create one by clicking the right mouse button *=> New => Folder*)
   (c) Type in the name of file in *File Name* box, keeping the **Spectra (*.spa)** extension in the *Save as Type* box, press "OK."

2.) To save data as an **Excel/Text file**:
   (a) Highlight data desired to be saved
   (b) Go to *File => Save as...* (change to your own directory)
   (c) Type in the name of file in *File Name* box, <u>change</u> the **Spectra (*.spa)** extension to **CSV Text (*.CSV)** in the *Save as Type* box, press "OK."

If you wish to **print a spectrum** go to *File => Print* and make sure that the
  HP Laserjet 6L is highlighted

When you are Finished

1.) Under the bench tab in the top left corner there is a Max IR intensity number. Record
  that number on the log sheet.

2.) Make sure the Laser is **OFF**.

3.) Go the menu at the top and choose *Raman => Use Raman Accessory* (check mark
  should disappear). The *Bench* should switch back to IR transmission mode.

4.) Physically change the beam splitter back to KBr and also change it in the software: go
  to *Collect => Experiment Setup => Bench* tab *=> Beam Splitter* = KBr

  Check to see that there is a red interferogram in the window as seen on the first page.

  If nothing appears then Go to *Diagnostics* tab and hit the "Align…" button to
  optimize the intensities. Please wait until the Align Bench comment box has
  disappeared from the screen before doing anything else. If still no signal then please
  contact Jerry.

  Press *OK* to exit.

5.) Exit OMNIC by going to *File => Exit*

6.) Log off your account by going to *Start => Log off* (lower right hand corner). Failing
  to do this may cause erroneous charges to be made to your account as stated in the **IR
  Policy** section.

7.) Leave the spectrometer with all sample chambers closed and the shutters open (levers
  down). Please return any equipment that you have used to its designated area. Do
  not leave your samples in the lab; we are not responsible for them.

8.) Complete the log sheet with your stop time and the actual time of usage. Please
  include any problems that you had with the instrument, if any. If you were able to
  solve the problem yourself, please include your explanation of how you did it; we
  greatly appreciate it.

The manual for the Raman Accessory and for the OMNIC software are very clear in details and can be found on the counter on the opposite wall of the instrument. Please DO NOT REMOVE ANY MANUALS or EQUIPMENT FROM THE ROOM!