

Standard Operating Procedures

For the Bruker RFS 100/S, Raman Spectrometer

Introduction

This instrument utilizes a 3 watt Nd:YAG laser which emits light with a wavelength of 1064 nm. The spot size of the laser can be changed between 1mm and 0.1mm. This light is used to excite the Raman effect in the sample. This radiation is then detected by a liquid nitrogen cooled Ge-diode and preamp assembly. The useful data is found at wavenumbers ranging between 110 (cm^{-1}) to ~ 3500 (cm^{-1}).

When the instrument is idle

The raman spectrometer does not require water or gas purges when not in use.

Operation

- A. Sign the log book for the Raman, including your name, date, samples, and laser power settings used
- B. Cool the detector with liquid nitrogen
 1. Grab the small liquid nitrogen container
 2. Fill the small brown reservoir with liq N_2
 3. When the instrument is filled with liq N_2 it starts to spill out the bottom of the brown reservoir. It usually takes about 2.5 times the volume of the brown reservoir to fill the instrument.
 4. Wait about 30 minutes for the detector to cool off. The detector should stay cold for $\sim 5 - 12$ hrs depending on the conditions.
- C. Placing a sample in the raman
 1. Always make sure the laser is off, (key is in the off position and the red interlock LED is lit on the power supply behind the computer) before opening the sample chamber on the front of the instrument
 2. Open the lid to the chamber, DO NOT TOUCH THE LENS
 3. In front of the lens is a black assembly which is the place where the sample is placed.

4. For powder samples in the aluminum holders, simply pull the black knob out and place the holder between the black piece with the U-shaped hole and the spring-loaded piece that was pulled out. Make sure the powder is facing the lens and that the holder is aligned with the black piece directly behind it
5. For other samples such as glass slides, there is a small plastic piece that the small aluminum holders rest, that can be removed to provide more space
6. Close the lid to the chamber

D. Open the OPUS software on the computer next to the raman

1. Hit return twice (there is no password to enter)
2. Go to Measure, then Measurement
3. This brings up a series of menus, the only ones that are normally used are the basic, advanced, and check signal tabs
4. Under the basic tab enter in the sample name and the laser power used (determined later) for that particular spectra
5. Under the advanced tab enter in the filename and path (either e:\Raman\Data\Gom Group\Your Name or e:\Raman\Data\Guest\Your Name)
6. This menu also can adjust the resolution (usually 4 cm^{-1}) and the number of scans (usually 32, although 64, 128, and 256 can also be used to increase the signal to noise ratio)
7. Now click the check signal tab. If the detector is cold the amplitude should be about 20-40 and remaining fairly steady. If the reading is 200-300 the detector will be cold in a few more minutes. A reading of 4 or 5 means that the detector is still warm.

E. Turn on the laser

1. Turn the key to the on position (90° to the right)
2. Open the Simple software shortcut is on the desktop
3. Click on the Open when available (a slight 10 sec pause is normal).
4. Set the power by typing it in or using the sliding bar at the bottom.

5. Set the power of the laser output in mW (values of 5 to 300 are typical) The max that can be used is 1000 but should not be used often. Start by setting a low value such as 20 mW and check the signal strength on the opus program
6. The ideal amplitude, under the check signal tab, is around 2000, but in general try to get the largest value possible while not using too high of a laser power. The higher power settings can produce heating of your sample which will appear as a very large broad hump from about 3500 to 1500 wavenumber. If the power is set at a relatively low value ~10-20 mW and the amplitude is 5 or so, the sample is most likely fluorescing. It will be difficult to get a spectra from that sample.
7. When the signal is good, switch back to the opus software and click on the basic tab. Type in the current power setting and then select Collect Sample

F. After collecting a spectra

1. As soon as the software makes all of the necessary scans (i.e. 32, 64 ...) set the laser power back to zero
2. The spectra that appears in the opus software is not a raman spectra. To obtain the actual raman spectra select the file you just created on the left side of the screen
3. Then go to Manipulate, Convert Spectra, and select the file in the box that you wish to convert. Then check to see that the conversion wavenumber is set at 9398.5 (1064 nm). If that's correct click on convert at the bottom of the window.
4. The new spectra is the actual raman spectra

G. Changing samples

1. Turn off the laser using the reverse order of the steps in E (i.e. set power to zero, close, off, and turn the key to the straight upright position) Do not close the simple program.
2. Open the sample chamber and change the sample
3. Repeat steps E3 - E8

H. Finishing

1. When finished collecting spectra make sure the laser power is off (red LED is lit on the power supply and the key is set to off) and then close the simple program by typing quit or exit and then hitting F4.
2. SIGN THE BILLING SHEET on the wall next to the doors