

Renishaw inVia Reflex Micro-Raman Standard Operating Procedure (2/8/11)

This user guide describes the proper operation of the Micro-Raman for acquisition of a single point spectral acquisition.

System startup

1. Make sure the Standby / Run switch on the Modu-Laser RC-1300 Controller is in the Standby position. If the Modu-Laser power supply is off, turn it on. Turn the key on the RC-1300 controller to On.

2. Turn on the Renishaw inVia Raman Microscope (switch on right side of instrument).

3. Turn on Proscan II stage controller box.

4. Allow instrument to warm up for at least 15 minutes.

5. Flip switch on Modu-Laser controller from standby to run. Adjust laser power with knob.

6. Start Wire 2.0 by clicking “flask” icon on desktop.

When the WIRE motor reference options pop-up appears, make sure “Reference unreferenced motors only” is selected and then click “OK”.

If any other pop-up or error boxes appear, contact NEST Lab personnel immediately.

7. Once the system has loaded, go to Tools ->System Health ->Health Check. When the “Silicon Calibration Reference” box appears, ensure that “Regular” is selected then click “OK”. This tool checks the alignment and calibration of the instrument. When the health check is finished, if any action is recommended, click “OK” to close the dialog and perform any actions as stated in the system health viewer.

The most common action required is a quick calibration. To run the quick calibration, go to Tools -> Calibration ->Quick Calibration. The calibration will run automatically. When the calibration is finished, repeat the Health Check. If no further actions are required, the instrument is ready to be used.

If you run into any issues in the software, running the task manager and ending any process name that begins with “WIRE” will successfully terminate the program, which could then be re-launched without a complete reboot of the PC. It is NOT ideal to restart the software

Sample Analysis

8. Place the sample on the sample stage, underneath the x5 microscope aperture. You may view the sample surface using the monitor video display or through the eyepieces at the top of the Raman. Focus the surface using the coarse adjustment knob (bottom right of microscope) and fine adjustment knob (on stage remote control). Rotate through all lenses in numerical order, using the fine focus in between.

Go only as high as the sample allows. Some samples may be too tall to focus beyond the x5. When you are finished, close the enclosure door.

Be extremely careful not to allow the sample, under any circumstances, to come into contact with the microscope lenses. This could result in serious damage to the instrument.

9. Go to Measurement -> New Spectral Acquisition to open the Spectral Acquisition setup dialog box. The following parameters should be entered:

9.1. Range tab:

Grating Scan Type should be “Extended” and the word “SynchroScan” should appear in the white box beneath.

Spectrum Range should be either Low 100 or High 3200 (see note below) and Raman shift should appear in the white box to the right.

You may select the laser and grating that you wish to use. The possible combinations are the 514 nm laser with the 1800 grating or the 785 nm laser with the 1200 laser. Be sure that the correct set of lens, either A1, A2, A3 or B1, B2, B3 are installed. Lens set A1-A2-A3 is installed for the 514 nm laser

9.2. Acquisition tab:

Exposure time: 10s (see note below)

Accumulations: 1 (see note below)

Laser Power: 10% (see note below)

9.3. File tab:

File name ->Browse ->Desktop ->User Data -> “your folder”. Enter a file name to which the Raman data will automatically be saved.

*If you do not already have a folder on Y: drive, create a new folder in the User Data folder with your first and last name. (i.e. John Doe)

Auto Increment: Selected

This option will automatically save each subsequent run to the entered file name incrementally. (i.e. If your file name you entered is called “Raman” then each subsequent run will be saved as “Raman1”, “Raman2”, “Raman3” etc.)

Note: Acquisition parameter values may be adjusted and may vary from sample to sample. Values listed here are general parameters that will provide a good starting point for most samples.

Spectrum range: Low and High

Values are set to the maximum (3200 cm^{-1}) and minimum wavelengths (100 cm^{-1}) that can be used as default values. These can be narrowed to a smaller range to focus in on key features. If you are working with a known sample, it is acceptable to begin with a narrower scan range. However, scanning the entire range initially does not add much time to the analysis, and ensures that all possible peaks will be observed.

Exposure time: The exposure time can be increased or decreased (to a minimum of 0.02s). An exposure time of less than 10s will only be needed in a very limited number of cases. Increasing exposure time can help to eliminate noise in the measurement. In most cases, an exposure time of 60s seems to be effective; however, this can also vary from one sample to another.

Accumulations: This is the number of times that the sample is excited by the laser for the given exposure time. (i.e. 1 accumulation at 10 s each(default) or 5 accumulations at 10 s each etc) Increasing the number of accumulations can have the same effect as increasing the exposure time and may help to improve the signal-to-noise ratio. The spectrum displayed will be the average of these accumulations. Multiple accumulations will not be necessary for most samples.

Laser Power: This will be the most frequently varied setting. It can be adjusted within the WIRE software and it can also be adjusted with the knob on the Modu-Laser RC-1300 controller. Ideally, the laser power should be high enough to give a strong peak intensity, but low enough such that it does not damage the sample. A value of 10% power should be safe for most samples. The power can be varied by choosing another value from the drop-down box. For many samples using 100% laser power will not be harmful, but it is best to start lower and work up than to risk damaging the sample on the first run. Lowering the laser power may be necessary if the resulting intensity saturates the CCD detector (indicated by the resulting spectrum appearing as a flat line).

9.4. Unless the sample has some pre-determined needs, no adjustments need to be made under any of the other headings. To apply your settings, click either “Apply” then “OK” or simply click “OK”

10. You are now ready to run the acquisition. To run the acquisition, click once on the “Run” icon in the toolbar (Erlenmeyer with blue triangle). The acquisition will begin immediately and the spectra will be displayed after a short time.

11. Steps 9 and 10 may be repeated as often as needed to optimize the settings for your particular sample.

Shutting Down

When you are finished using the instrument, turn off all of the components in this order:

12. Close software program.

13. Turn down power on Modu-laser RC-1300 controller (fully CCW). Flip switch to Standby. Turn key to off position. Fan will continue to run for ~2 minutes, and then will shut off when the laser is cool.

14. While the fan on the laser is running, you can turn off the Proscan II control box and turn off the Raman spectrometer.

15. Leave the Modu-Laser power supply on (black box behind the spectrometer).