

# Instrument Standard Operating Procedure

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## Raman Reflex

*Last Updated: 11/27/2023*

You must be a “Qualified Self-User” to operate this instrument independently.  
You must be on the labs “Instrument Reservation Schedule” before touching the instrument for any reason.

Any problems, STOP, Post a note on the instrument and send an email to [mtim@mit.edu](mailto:mtim@mit.edu) immediately.

Do not perform any maintenance.

Do not install any software

Do not adjust any optics.

### **Instrument Hazards:**

Lasers, Cryogenic Liquids Chemicals & Electricity.

### **Laser Safety:**

Lasers: Class 3B, Interlocked system (No exposed laser beams).

Wavelength and Power: 785nm 100mw, 533nm 50mw, 437nm 25mw.

### **Required Apparel:**

Safety Glasses

Laser Safety Glasses when interlocks are disabled.

Wear clean gloves when handling optics.

### **CORAL:**

Engage the instrument using CORAL when you enter the lab.

Disengage CORAL when you are leaving the lab.

Report all problems immediately to [mtim@mit.edu](mailto:mtim@mit.edu).

**Check out the system when you arrive:**

If you encounter any problems: Stop work and send Tim ([mtim@mit.edu](mailto:mtim@mit.edu)) an email immediately.

Lasers, Spectrometer and Microscope Laser Safety Enclosures – Closed.

Computer – On

Software - Closed.

**Operation:**

Turn the Lasers on (Key).

*785nm laser must warmup ~ 30 seconds before performing health check.*

**Logon to computer:**

User name: Admin

Password: 134139

Open the raman software: "Wire".

**Verify Performance:**

On the machine status toolbar:

Choose your desired Laser Wavelength & Grating.

*(1200 l/mm and 2400 l/mm installed, 600 l/mm available.)*

**Run the Health Check**

473nm & 532nm – Regular or Confocal

785nm – Line Focus

*Do not do any alignments unless health check requires it.*

*Do the alignment or quick calibration if prompted by the health check.*

*If the results tell you to do an alignment or quick calibration you will find this under the "Tools" menu.*

**Confocal Note:**

The Raman Microscope is inherently Confocal because It has two pinholes in the beampath.

To optimize the confocality of the sytem the laser beamsize is adjusted to the size of the backplane of the microscope objective you are using.

When you create a new spectral acquisition measurement file you will find two options.

Regular or High Confocal.

Regular – Illuminates 13 rows of pixels on the ccd.

High Confocal – only reads the center row of pixels.

High confocal is a way of collecting data from a thinner slice of your sample.If high confocal is selected anything not in focus won't be read by the ccd.

**Perform your work:**

Focus onto sample.

Determine Power.

Set Measurement Range.

Determine Exposure.

Determine Accumulations.

**Save your data:**

Walk away with a copy of your data if it is important to you.

Use a USB thumb drive to transfer your data.

Do not connect to the Q drive!

**Data Analysis:**

Wire software is available to users.

Available on the desktop of the Raman computer.

Follow the readme instructions.

**Shutdown at end of session:**

Close the Software

Turn off the Lasers (Key)

Cleanup

Disengage CORAL

**Problems?**

Send [mtim@mit.edu](mailto:mtim@mit.edu) an email immediately.

It is OK to try to resolve the problem by restarting the system.

**Restart the system:**

Restart the Computer.

Turn the Main Power Off then back on (Located on the back of the spectrometer).

Open the software (Wire).

Initialize the motors if needed.

Turn on the Lasers (Key).

There is also a power switch to the Raman spectrometer that is not normally turned off.

The power switch is located on the back of the spectrometer (facing the wall).

**Interlock tripped?**

Close the Spectrometer and Sample compartment doors.

Reset the interlock in the software under the Tools menu.

Turn the laser off and back on using the key.

**Utilities:**

Electricity (wall).

**Emergency Shutdown:**

Shutdown the computer.

Turn off the spectrometer power.

Power switch is located on side of the spectrometer facing the wall.

**Restart after an emergency:**

Restart the computer.

Turn on the spectrometer power.

Power switch is located on side of the spectrometer facing the wall.

## Raman Polarization Measurement Configuration Modes

Polarized –  $\frac{1}{2}$  lambda waveplate - Linear

Depolarized –  $\frac{1}{4}$  lambda waveplate - Circular

### Isotropic Samples

Depolarized Raman

Incident – Depolarized

Analyzer – Polarized X or Y

### Anisotropic Samples

Polarized Raman

Incident – Polarized X and Y

Analyzer – Polarized X and Y

Rotatable Sample Stage

Angle Resolved Polarized Raman

Incident – Linear Polarized ( $\frac{1}{2}$  waveplate) X and Y

Analyzer – Linear Polarized ( $\frac{1}{2}$  Waveplate) 0-190 degrees

Rotatable Sample Stage

In the measurement setup under the advanced tab you can choose to put a polarizer into the excitation beam path. Either circular or linear.

Remember sample orientation will usually make a difference.

Measuring a piece of silicon at different orientations is a good example of this.

There is a rotatable stage insert that could be used.

The 532nm laser has a computer controlled polarizer ( $\frac{1}{2}$  Lambda Waveplate).

Note: 60 degrees = 0 degrees, operates from 60 deg to 400 deg.

Pol orientation on sample stage: 60 degrees = X, 150 degrees = Y

To use the computer controlled 532nm polarizer:

Find the “Setup Polarization Measurement” under one of the menu dropdown list.

To run the experiment choose “Run Polarization Measurement” under the same menu dropdown list.

When finished – Reset the polarizer to 60 degrees.

If you have a problem with the polarizer covering your measurement range or you need a polarizer in the collection path

there are wavelength specific polarizers that can be manually placed in the Excitation and Collection paths. Circular or Linear using the software.

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Almost all the lasers we supply on our inVia Raman systems are horizontally polarized, so that is L/R as you face the microscope stage. On the collection side, if you place the Polariser plate after the Rayleigh filter assembly then you'll reject all the signal that is not parallel to the entrance slit of the spectrometer. To see the other polarization state, you'll then also need to insert the half wave plate (still with the Polariser in place).

To rotate the incident polarization direction, you need to insert a half wave plate before the sample. We typically do this immediately in front of the laser or we can do it before the beam hits the first filter and is directed into the microscope. We can also fit quarter wave plates here to circularly polarize (scramble) the light.

Hope that helps

Regards

Matthew



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## **532nm Low Wavenumber Filter (LWNF)**

You must be trained by Tim to specifically use this filter.

Wear Gloves, Laser Safety Glasses

Open Spectrometer Cover.

Remove 532nm Edge Filter.

Install 532nm LWNF.

Setup Experiment for 1 second exposure, 100% Power and 1 Accumulation, 2400 grating, range centered at zero.

Focus microscope onto Silicon or Sulfur Reference sample.

Click the "Cycle" icon.

Adjust the slit until you maximize sample signal and minimize laser signal (Counter Clockwise).

Close the spectrometer.

Measure your sample(s).

When Finished

Put on new gloves.

Return slit to original position (Fully Clockwise).

Remove the LWNF.

Install the 532nm edge filter.

Run a health check (532nm regular).

Vendor Contacts:

<http://www.renishaw.com/en/invia-performance--9452>

Sample info:

Other Instruments:

Harvard CNS

MIT Institute for Soldier Nanotechnology



ACKNOWLEDGMENT OF SUPPORT:

CMSE requires that all publications and patents resulting from work done under this project or with the assistance of the CMSE Facilities, be acknowledged. Please be certain that one (or a combination) of the following statements is included in papers with support from CMSE:

*"This work was supported primarily by the MRSEC Program of the National Science Foundation under award number DMR - 1419807."*

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*-or-*

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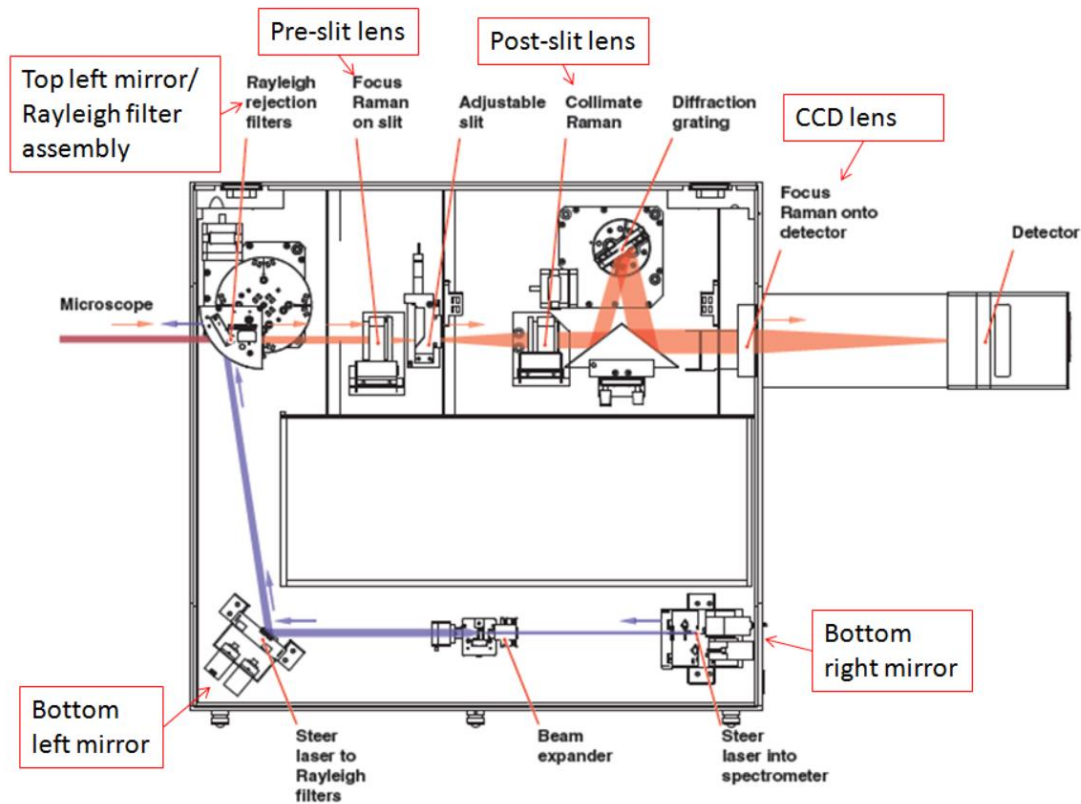
Instrument Specifics:

<https://mtim.mit.edu/asef-otc-web/instruments/raman>

## Renishaw Invia Reflex Raman Confocal Microscope

This is a fully automated confocal Raman microscope system with three laser excitation lines a motorized stage and open sample compartment to accommodate cryostats. A Temperature controlled microscope stage is available for temperature dependent measurements.

### Raman Reflex Beampath



**Data Acquisition Modes:**

Point Mapping  
Imaging - Stream HR fast imaging  
Depth Profiling  
Polarization  
Time Dependent  
Temperature Dependent  
Atmosphere Dependent  
Photoluminescence

**Laser Sources:**

473nm	25mw Linear & Circular
532nm	50mw automated rotate linear
785nm	100mw Linear & Circular

**Detector: 100cm<sup>-1</sup> to 10,000cm<sup>-1</sup>**

1" CCD array, Deep Depletion for enhanced IR Range  
400-1050nm, 1024 X 256 pixel

**Gratings:**

600l/mm, 1200l/mm, 2400l/mm

**Rayleigh Filters:**

473nm edge	10%@150cm <sup>-1</sup> shift, for Photoluminescence measurements
532nm eclipse	<15cm <sup>-1</sup> shift, Stokes & Antistokes
532nm edge	~100cm <sup>-1</sup> ,
785nm edge	10%@50cm <sup>-1</sup> Rectangle w/o aperture

**Microscope:**

Leica Optical Microscope  
Infinity Corrected, 200mm Tube Length  
Free Space with enclosure

**Objectives:**

Semi par-focal

Objective	Opening	WD	NA
10X	4mm	12mm	0.25
LWD50X	2mm	8mm	0.50
50X	4mm	350um	0.75
LWD100X	2mm	~3mm	0.75
100X	2mm	0.1mm	0.90

Reflected Light Polarization

**Microscope Stages:**

Renishaw HSES motorized stage

Step Size: 100nm

Stage Travel: 112mm x 76mm

**Rotating Microscope stage** - polarization experiments

**Temperature controlled microscope stage**

Linkam THMS600 (-196deg C to 600deg C).

Macro Sampling Set:

Cuvettes, Powders , Capillaries, Etc.

Computer & Monitor

HP Computer

Monitor 22"

Reference Spectra:

Organic Polymers, Inorganic materials, Sample substrates

Features:

Data Analysis Software available to users.

\*\*\*\*\*End\*\*\*\*\*