Renishaw Raman Microscope System

Standard Operating Procedure

Lab: 270 Davenport

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Section 1: Overview

Type of SOP: ☒Process ☐Hazardous Material ☐Hazardous Class of Materials ☒Equipment

Synopsis:

This SOP is designed to provide directions for using the Raman system

Section 2: Risk Assessment Summary (Hazards and control measures)

Information obtained from performing a risk assessment should be entered into this section.

Materials:

Material (name, CAS #, other ID)	Hazards
H_2	Flammable
CH_4	Flammable
O_2	Flammable
Ethylene	Flammable
He	Non- Flammable

Relevant References for Material Hazards:

H_2: https://www.airgas.com/msds/001026.pdf
CH_4: https://www.airgas.com/msds/001033.pdf
O_2: https://www.airgas.com/msds/001043.pdf
Ethylene: https://www.airgas.com/msds/001022.pdf

Equipment Hazards:

Possible class IIIb laser exposure if interlocks are overridden – do not override without explicit permission

Hazardous Conditions:

None.

Technique Hazards:

None.

Personal Protective Equipment

Laser Safety Glasses – required when UV (325 nm) laser is in use, optional but recommended for 445 nm and 532 nm laser operation

Engineering Controls

Raman system is housed in a walk-in fume hood (Dec. 2017). Enclosure is present so that the user does not come in contact with the laser directly.

Section 3: Procedures

Starting up the System

1) If starting the system from complete shutdown turn on the components in the following order: (1) Raman Microscopy, (2) WiRE Software, (3) Laser(s)
2) If using the 442 or 325 nm laser, turn on ~30 min before expected use for it to warm up and come to a stable voltage.

Taking a first measurement

1) Under the "Measurements" tab, select new measurement
2) Pick desired laser (e.g., 532 nm) and acquisition type (e.g., edge versus streamline)
3) View sample through camera to focus the objective on your sample (this is very important)
4) I recommend 0.1% power with a 1 s acquisition time to start and increment up the time to 10 s before turning up the power by half-order of magnitudes (e.g., 0.1 to 0.5 to 1 to 5%)
5) Try taking multiple scans at the desired power and acquisition time to see any change in the spectra (a significant change is indicative that your surface is changing due to the laser)
6) Set up file name and directory to save the spectra (using your folder in "USERS")
7) Turn off lasers when done – unless you discussed with the person after you who is going to use the same laser

Loading the Linkam cell for *in situ* gas-phase spectra

1) Carefully unscrew the holder, remove the quartz window (note: it's fragile), and unload the crucible (sample holder)
2) Evacuate the contents into a scintillation vial (or waste), and blow out the inside of the crucible with compressed air
3) Place crucible in the two-piece teflon holder
4) Add sample (~60-100 mg depending on density) and tap to flatten the surface

5) Reload the cell, carefully place the quartz window on top, and tighten the holder (not *too* tight, just until it's snug)
6) Carefully place Linkam cell in the recess holder
7) The 50x objective has too short of a working distance, so use the L50x, 20x, 5x, or 15x (if using 325 nm laser).
8) Unload the cell and clean everything for the next user
9) All of this is explained in detail in the CCR1000 manual located on the desktop of the Raman computer.

Loading the custom liquid cell for *in situ* liquid-phase spectra

1) Load liquid cell with pellets, O-ring, glass slide. Screw on top metal sheet, careful not to shatter glass slide. Attach liquid inlet and outlet tubing. Place cell in blue/grey Teflon boat (designed to catch any leaks) and start flowing liquid at the rate that will be used during the experiment AT MINIMUM for 5 min. This leak check should be done OUTSIDE of the Raman compartment.
2) Once leak check is complete, turn off liquid flow and lower Raman stage all the way before carefully placing the cell in the Teflon boat on the stage. Make sure it fits in the designated area and does not slide off.
3) After focusing on sample, place interlock override and close the sliding door as much as possible while leaving room for any tubing, thermocouple/heating cables. Make sure there is tubing attached to the Teflon boat and that it is directed OUTSIDE of the Raman compartment into a waste container at all times.
4) Cover well the slit that is left open due to the interlock override with aluminum foil. This ensures laser safety and a darker compartment for the laser and sample. Might need to refocus on sample after these steps.
5) Begin experiment as usual.
6) Unload the cell and clean everything after experiment for the next user.

Section 4: Waste Disposal/Cleanup

Gas waste should be removed via the exhaust. Liquid waste should be collected in an appropriate container and discarded via DRS standards

Section 5: Emergency Response

In case of an emergency (e.g., a fire alarm or power outage), turn off all lasers and Raman system so that they may be powered on in a controlled manner. Shut off any gas lines that go to the system (e.g., H_2, CH_4, etc.)

Training Documentation

Signing this document means that you have read and understand all aspects of this Standard Operating Procedure. The supervisor is the person that acknowledges you took the training and understand the procedure. They can be a lab manager or researcher assigned by the PI to oversee this particular SOP.

Name (Printed)	Name (Signed)	Supervisor	Date