Gas-phase Epoxidation System + IR

Standard Operating Procedure

Lab: 270 Davenport

Department: Chemical and Biomolecular Engineering

PI/Manager of Space: David W. Flaherty

Written By: Zeynep Ayla

**Section 1: Overview**

Type of SOP: [x] Process [ ] Hazardous Material [ ] Hazardous Class of Materials [x] Equipment

Synopsis:

*This SOP is designed to provide directions for the gas-phase epoxidation system and associated Invenio IR*

**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** |
| Air | Non-Flammable |
| He | Non-Flammable |
| H2O2  | Toxic |
| 1-hexene | Toxic |
| 1,2-epoxyhexane | Toxic |
| Acetonitrile | Toxic |

Relevant References for Material Hazards:

|  |
| --- |
| *H2: http://www.praxair.com/-/media/documents/sds/hydrogen/hydrogen-gas-h2-safety-data-sheet-sds-p4604.pdf?la=en**CH4: https://www.pge.com/includes/docs/pdfs/shared/environment/pge/cleanair/methane1033.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

*Nitrile gloves, safety glasses, thermal gloves.*

Engineering Controls

*The system is housed in a walk-in fume hood.*

**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.

**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., H2, CH4, 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.

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| **Name (Printed)** | **Name (Signed)** | **Supervisor** | **Date** |
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