ATR-FTIR Modulation Excitation 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 ATR-FTIR 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
Methanol	High toxicity upon ingestion.

Relevant References for Material Hazards:

H_2: http://avogadro.chem.iastate.edu/MSDS/hydrogen.pdf
Methanol: http://avogadro.chem.iastate.edu/MSDS/methanol.htm

Equipment Hazards:

None

Hazardous Conditions:

Pure Hydrogen and pure oxygen is fed to the liquid in the saturator. Care should be taken that these gases never come into contact with each other

Technique Hazards:

None

Personal Protective Equipment

Safety glasses, nitrile gloves

Engineering Controls

List the necessary engineering controls required for the procedure (e.g., chemical fume hood, biological safety cabinet, etc.).

Section 3: Procedures

Removing the ATR crystal

1) Remove the cell from the mounting assembly
2) Remove the end cap, gold cone, and spider from each end of cell
3) Place a piece of lens paper or soft tissue over one end of the ATR rod. By using the plastic rod insertion tool, gently press the rod out of the cell. As the rod is removed, the emerging end should be supported so as to avoid contact between the rod and cell body.
4) If the rod has been installed for a long time, it may not press out easily. In this case, it often helps to flood O-rings with a lubricating solvent such as acetone

Inserting the ATR crystal into the cell

1) Place an O-ring on the ATR rod near one end.
2) While holding or clamping the cell body in a vertical orientation slide the insertion tool partway through the body from the bottom.
3) Carefully insert the ATR rod from the top using the insertion tool to keep it centered in the cell body.
4) Insert the second O-ring around the lower end of the ATR rod and use the O-ring seating tool to fully seat it. In doing this, you can press the rod from the top using a soft tissue to keep it approximately centered in the cell.
5) By alternatively using the rod insertion tool and the O-ring seating tool make sure that the rod is centered in the cell body and that the O-rings are fully seated in their pockets.
6) Reinstall the end caps, gold cones and spiders.
7) To check the centering of the ATR rod, compare the appearance of the rod as viewed from both ends. If a dark circle appears around either the inner or outer periphery of the optical aperture, the rod is not properly centered. A dark circle around the inner periphery indicates that the rod is too close to this end. In this case, remove the near end cap, spider and cone and loosen the far end cap slightly to allow the rod to move through the O-ring. Use the insertion tool (covered with a piece of tissue) to better center the rod.

Preparing the FTIR and Checking the ATR crystal

1) Make sure that the FTIR is switched on and the purge gas is connected. The three indicators on the FTIR (Laser, humidity, status) light should be green.
2) Fill liquid nitrogen in the FTIR to cool the detector. Fill till liquid nitrogen is seen coming out of the detector.
3) Open OPUS and go to advance measurements. In advance measurements, open the check signal tab to see if there is signal being detected. The value of amplitude should be > 30000
4) Take a background with empty cell compartment.
5) Install the ATR accessory into the sample compartment carefully.
6) Again check the signal. The amplitude would have dropped to < 10000 (possibly even lesser). Adjust the value of aperture (Advance measurements>>optics) and sample signal gain (and background signal gain) to get a value between 15000 and 30000.
7) Take a spectra of the empty cell. Check it's spectrum against a standard.
8) Take a background of the empty cell.
9) Connect the pumps and flow pure methanol in the system and take a spectrum of the same.
10) Do this procedure atleast once every week and check the signal levels from the previous weeks. If there is an increase in the amount of noise (especially in the low wave number region) and decrease in the amount of signal, it indicates that the ATR crystal is getting attacked.
11) Follow the procedure to remove the crystal from the cell and clean it using appropriate solvent.

Evacuating and re-pumping the MCT detector

1) **If the MCT detector lasts for less than 4 hours after filling liquid nitrogen, it is an indication of existence of water and contaminants inside the detector which needs to be evacuated.**
2) **Open the top panel of the FTIR and remove the detector by unscrewing one screw using the screw driver in Fig. Take out the detector vertically to avoid any damange to the mirror inside the chamber and the KRS crystal on the detector.**
3) **Screw the vacuum adapter carefully on the back of the detector and carefully take out the o-rings.**
4) **Put Krytox grease on all o-rings and fittings to clean and grease it.**
5) **Connect the pump tube to the turbo pump.**
6) **Put the vacuum adapter back on the detector by carefully screwing it to the groove and put the nut and tighten it with fingers (additional tightening is not required) on top and pull the shaft to the open position.**

Section 4: Waste Disposal/Cleanup

None

Section 5: Emergency Response

Emergency Response should be a component of the Laboratory Safety Plan. If there are particular response measures that are required by this procedure, include them here.

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