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X10C GAS HANDLING SYSTEM OPERATIONAL PROCEDURES

Experimental Overview

The fundamental purpose of the X10C Gas Handling System is to provide a safe and effective means to enable transmission and fluorescence XAFS/XANES investigation of short-range structural and functional order of catalytic materials under controlled conditions of reduction, oxidation and poisoning. In-situ experiments routinely involve exposing a 1g sample mounted in a hermetically sealed stainless steel Catalyst Reactor Cell to different gas environments and various temperatures from liquid nitrogen to 500C. The cell is equipped with beryllium windows on Conflat flanges and designed with a static gas volume of 25cc to minimize the amount of flammable/toxic gases in the reaction region. The X10C Gas Handling System, temperature control electronics and mechanical alignment mechanism are functionally independent systems.

System Description

The system consists of a hazardous gas cabinet, gas feed enclosure, and the X10C hutch enclosure. Gases from the inert gas bottle rack are also routed to the gas feed enclosure. All enclosures share a common exhaust header. Photohelic pressure sensors are used to monitor the exhaust flow pressure in the X10C enclosure and hazardous gas cabinet. Exhaust rates below the lower trip setting will terminate gas flows by closing all solenoid valves and activating the sonic alarm.

An annual validation of the condition of the gas handling system will also be preformed and the validation check list (see attachment 1) will be posted on the gas cabinet. Old check lists will be filed when a new one is posted.

Operation

Startup:

1. **Ensure that the main power for the Roof Exhaust Unit is turned on and the Start button has been pressed using PPE consisting of safety glasses, leather palmed gloves, natural fiber long sleeve shirt and long pants. The Exhaust main disconnect switch and Start button are located on a box which is on the aisle concrete wall between the X-10 and X-9 area.**
2. **Ensure that the H2, Methane, Ethylene and UHP Helium or Argon gas cylinders are attached to the appropriate inlet connections of the gas handling system. Open all valves on the gas cylinders that are required (Inert Gas Bottle Rack and Hazardous Gas Cabinet). Adjust regulators to 10psi.**
3. **Check Hazardous Gas Cabinet Photohelic pressure sensor for adequate exhaust flow reading of 0.12 on meter.**
4. **Check H2 and Methane alarms to make sure they have not tripped.**
5. **Check X10C Hutch Photohelic pressure sensor for adequate exhaust flow reading of 0.14 on meter and return pressure trips for gas flow high and low settings. Reset the gas flow trips, if necessary, by toggling switches on indicators at Gas Handling System front control panel.**
6. **Turn Mass Flow Controller unit power on. It becomes illuminated when power is applied.**

7. **Ensure that the black manual valves of the Hutch Mixing Manifold are closed. In the closed state, the handles are orthogonal to the piping. The valves are located on the downstream inside wall of the x10C hutch, below the solenoid valves.**
8. **Manual valves V1 and V2 on the Hutch Mixing Manifold are labeled *" Reactor Cell"* and *"Bypass"*. Select the Bypass mode. When the handles are pointed toward each other the flow will bypass the Reactor Cell.**
9. **Open Helium/Argon and return line manual valves on the Hutch Mixing Manifold.**
10. **Push the green Start button at the top of the Control/Interlock Logic Unit. No PPE is required since the system uses only low voltage for operation. The green light will begin flashing to indicate that the unit is not fully operational, PLU timers are active and the return flow interlock is bypassed. It will not be fully operational for approximately 10 minutes, with the green light transitioning from a flashing to continuously on condition. During this 10 minute time period, only the Helium/Argon flow can be engaged.**
11. **Turn on the Helium/Argon flow with the solenoid valve control switch at the Control/Interlock Logic Unit. The switch will illuminate upon activation. Set the select switch on the Brooks Controller to channel 1. The LED meter displays the flow rate of the selected channel. The gas flow can be adjusted with the rotary knobs on the Brooks Controller.**
12. **Purge the Hutch Mixing Manifold with Helium/Argon for 10 minutes with a Brooks Controller reading of at least 5.0 on the LED meter.**

Note: The H2S scrubber system is not currently certified, therefore the Hydrogen Sulfide gas supply system is not operational and tagged "Out of Service" at this point in time.

Standard Operation:

After the green light transitions from flashing to continuous on, the system is ready to flow experimental gases.

1. **Ensure Reactor Cell inlet and outlet ports are connected to Hutch Mixing Manifold.**
2. **Ensure Reactor Cell inlet and outlet valves are open.**
3. **If flow through the H2O sparger is required, set the appropriate valves on the assembly.**
4. **If flow through the O2 impurity trap is required, set the appropriate valves on the assembly.**
5. **Manual valves V1 and V2 on the Hutch Mixing Manifold are labeled *"Reactor Cell"* and *"Bypass"*. Select either the Bypass or Reactor Cell mode. If the handles are pointed toward each other the flow will bypass the Reactor Cell. Conversely, if they are pointed away from each other, the flow will be through the Reactor Cell. It is generally good practice to initiate the flow in the Bypass mode and then switch to Reactor Cell mode to moderate any startup pressure bursts on the Reactor Cell. Purge the Reactor Cell with Helium/Argon when required.**
6. **Open the appropriate manual gas valves on the Hutch Mixing Manifold.**
7. **Turn on the appropriate gas flow with the solenoid valve control switch on the Control /Interlock Logic Unit. The switch will illuminate upon activation. Interlocks prevent the H2 and O2 solenoid valves from being opened simultaneously. Once the H2 switch has been opened, the interlock logic does not allow the O2 switch to operate for 1 minute after closing the H2 switch. The Green light will flash during this delay and allows the user to purge the manifold with Helium/Argon.**
8. **Set the selector switch on the Brooks Controller to the appropriate gas flow; channel 1: Helium/Argon, channel 2: Hydrogen, channel 3: Methane/ Ethylene, channel 4: not in service. The LED meter displays the flow rate of the selected**

channel. Each gas flow can be adjusted with the rotary knobs on the Brooks Controller.

Should a safety fault occur during operation, the interlock system will trip, causing the Hutch Mixing Manifold solenoid valves to close, except for the Helium/Argon valve. During hazardous gas operation the solenoid valves in the Hazardous Gas Cabinet will also close. At the same time a sonic alarm will sound to advise the user that a fault has occurred. Reset the alarm by pushing the Silence button located near the top of the Control /Interlock Logic Unit cabinet. This does NOT reset the system and the unit will remain in a shutdown state until the fault condition is cleared. To reset the unit, the user should investigate the problem, clear the condition, and push the Green Start button. The system will then be ready to restart the gas flows.

Shutdown:

1) **Purge the Reactor Cell with Helium/Argon for 10 minutes with a Brooks Controller reading of at least 5.0.**
2) **Set flow to Bypass.**
3) **Turn all rotary dials on the Brooks Controller to zero. Turn unit power switch off.**
4) **Close all Hutch Mixing Manifold solenoid valves using switches at top of Control/Interlock Logic System cabinet.**
5) **Close all Hutch Mixing Manifold black handle valves (below solenoids inside X10C hutch).**
6) **Push Red Stop button on Control/Interlock Logic System unit.**
7) **Close all gas cylinder valves.**

Note:

An annual Flow Testing of the X10C Gas Handling System Exhaust Ventilation Cabinets and ductwork will be performed (see Attachment 1). The test will be done with the gas cylinder valves closed. The test will be performed with a clean gas cabinet air intake filter and will have a required volume flow rate of 240 cubic feet/minute measured at the duct port above the cabinet. The Brooks controller cabinet, next to the gas cylinder cabinet, will have a required flow rate of 220 cubic feet/minute measured at the duct port above the cabinet.

Specific Allowed Gases:

1) **UHP Helium**
2) **UHP Argon**
3) **Hydrogen (100cu.ft.)**
4) **Methane (100cu.ft.)**
5) **Ethylene (100cu.ft.)**
6) **Oxygen (100cu.ft)**

Attachment 1

Annual X10C Ventilation Validation Checklist

(Post this form each year on the Gas Cabinet)

Exhaust system air flow:

- Clean gas cabinet air intake filter.
- Required volume flow rate = 240 cubic ft/min at gas cabinet
  = 220 cubic ft/min at Brooks Controller cabinet
- Linear flow rate is measured in the duct above the cabinet. The radius of duct is 0.25 feet.

The area of that duct = $\pi r^2 = \pi(0.25)^2 = 0.196$ square feet.

Gas Cabinet ft/min	Gas Cabinet ft^3/min	Brooks Controller Cabinet ft/min	Brooks Controller Cabinet ft^3/min

SHSD/RCD support _____ _____
(print name) (signature)

_____ _____
(date) (Life #)

Local contact _____ Date_____
(signature)