

## Standard Operating Procedure for Sector 16 Glovebox in Bldg. 434 E030

### Changes made in this revision:

- N/A (new procedure)

### Prepared by:

Curtis Kenney-Benson

Jesse Smith

### Reviewed by:

### Approved by:

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## Standard Operating Procedure for Sector 16 Glovebox in Bldg. 434 E030

### 1 INTRODUCTION

#### 1.1 Purpose and Scope

This manual is intended to cover the general guidelines and typical operation procedures for the use of the glovebox (GB) located in Bldg. 434 E030. The GB is intended to be a water and oxygen free environment for the handling of either solid or liquid air-sensitive samples. The procedures below are designed to ensure safe operation of the glovebox, protect the glovebox, and preserve the integrity of the samples.

#### 1.2 General Guidelines

- Only authorized users are permitted to use the glovebox.
- All sample requirements must be met.
- All work must be covered under a posted ESAF specifying glovebox use.
- All procedures associated with safe handling of chemicals are to be strictly followed.
- The points of contact for this equipment are Curtis Kenney-Benson, Jesse Smith and Guoyin Shen.

#### 1.3 Emergency Procedures

- Remain calm, describe in detail the incident in the log book, and inform a GB contact person about the incident.
- If glove is punctured or damaged, inform GB contacts as soon as possible, and place a port cover on the glove, if H<sub>2</sub>O or oxygen levels are high.
- In case of an emergency call 911 and inform GB contacts as soon as possible.

#### 1.4 General Rules for Working in the GB:

- Any work inside the GB or use of antechambers must be recorded in the logbook.
- Jewelry (watches, rings, bracelets, etc), can damage the gloves and must be removed.
- Clean gloves must be worn before inserting hands into GB gloves. DO NOT use any gloves which have come into contact with research materials, solvents, or other hazardous chemicals.
- Aluminum foil must be placed in the work area to collect spilled sample. The foil needs to be removed at the end of the experimental run or between samples if several groups are using the GB simultaneously.
- Avoid sharps (razors, scissors, ...) if possible.

- Authorization from a glovebox contact is required prior to the items being placed in the GB. The sharps should be clearly marked. It is recommended that cut resistant gloves should be worn inside the box while using sharps.
- Use of long sleeve shirts or lab coat while in the GB is encouraged (not required).

## 1.5 Sample Rules

- **No volatile sulfur-containing compounds or volatile phosphines:** These compounds are extremely poisonous to many types of catalysts, including the GB oxygen-scrubber
- All solid materials must be contained under an inert atmosphere and powders must be properly dried to prevent the introduction of adsorbed water.
- In general, liquids should be anhydrous (either by pre-treatment or purchased anhydrous) for use in the glovebox. Containers need to be very well sealed and taped up before bringing into the glovebox antechamber to avoid leakage into the pump and minimize shattered glass in case of a broken container.
- Sample containers should be filled with inert atmosphere, and capable of maintaining low levels of O<sub>2</sub> and H<sub>2</sub>O during transport
- Sample containers that have been stored cold must be allowed to warm to room temperature before being brought into the glovebox to avoid bringing in condensed moisture on the container surface.
- Other necessary materials (Kimwipes, aluminum foil) are available inside the glovebox. Please inform GB contacts if any of these items need to be replenished. Special procedures are needed to introduce these to the glovebox, and only authorized users are permitted to do so.
- All samples/chemicals must be appropriately labeled (chemical name, owner, beamline, and date)
- No samples/materials may be stored in the glovebox beyond the time-frame of the experiment.
- All trash generated as part of work in the glovebox (e.g., used Kimwipes, weighing paper) must be removed upon completion of the work and is responsibility of the user.

## 1.6 Glovebox atmosphere

- The glovebox operating parameters should not to be modified.
- **Recommended O<sub>2</sub> operating limits as indicated on the O<sub>2</sub> meter are normally <5 ppm of O<sub>2</sub>**
- Care must be exercised in using tools (e.g., needles, forceps) which could damage the gloves.
- Watch for and report low pressure or unusual H<sub>2</sub>O and O<sub>2</sub> levels.
- Inform GB contacts if O<sub>2</sub> levels are above 5 ppm

## 2 GLOVEBOX OPERATION

- The usual operating pressure of the glovebox is +2 to +8 mbar overpressure.
- The foot pedal should be used whenever the user's hands are being put in or out of the GB.
- **The normal state of the antechamber (when not in use) is under vacuum.**
- **After completing your work, the antechambers must be left under vacuum.**
- **Do not over tighten the antechamber ports.**
- Inspect gloves for damage prior to use.
- Do not work in GB if levels are above 5 ppm and notify the GB contact immediately.
- Every transfer and Action that happens in the GB must be recorded in the logbook. This includes indicating the chemical names and composition of samples being used.

### 2.1 Introducing items to the glovebox through the antechamber

1. Check the logbook to ensure that there are no samples in the antechamber. Log in your information if the antechamber is available.
2. Refill the antechamber by closing the vacuum valve and slowly filling the chamber with argon by opening the Ar valve. *IMPORTANT: Do not open the Ar valve fully. Do not open the valve enough that the box pressure falls below 0 mbar on the pressure gauge (e.g. the pressure inside the box goes negative).*
3. Once the chamber is at atmospheric pressure, close the Ar valve.
4. Open the outer antechamber door. Place items into the pull out tray. Close the door.
5. Evacuate the antechamber by opening the vacuum valve.
6. Wait for 10 min.
7. Refill the antechamber half way by opening the argon valve.
8. Repeat steps 5 and 6 two more times.
9. Refill the antechamber completely by opening the argon valve.
10. Once the chamber is full, close the argon valve.
11. Open the inner antechamber door. Move items in to the GB. Close the door.
12. Evacuate the antechamber by opening the vacuum valve.

### 2.2 Removing items from the glovebox through antechamber

1. Check the logbook to ensure that there are no samples coming in the antechamber. Log in your information if the antechamber is available.
2. Ensure that the antechamber is under vacuum and has been for at least one 1 h if the last user came out of the glovebox (if not, perform steps 5 through 8 in Section 2.1 above)
3. Refill the antechamber by closing the vacuum valve and slowly opening the antechamber to argon with the Ar valve. *IMPORTANT: Do not open the Ar valve fully. Do not open the valve enough that the box pressure falls below 0 mbar on the pressure gauge (e.g. the pressure inside the box goes negative).*
4. Open the inner antechamber door. Place items into the pull out tray. **Close the inner door.**

5. Open the outer antechamber door. Move items out. Close the outer door.
6. Evacuate the antechamber by opening the vacuum valve.

### 3 DOCUMENTS/RECORDS CREATED BY THIS PROCEDURE

The documents/records listed below will be created in the execution of this procedure and must be retained as indicated.

<b>Description of Document/Record (include ID number, if applicable)</b>	<b>Custodian</b>	<b>Storage Location and Medium</b>
Training Checklist for Sector 16 Glovebox	Curtis Kenney-Benson	Printed form in a sleeve attached to the Glove Box

### 4 TRAINING REQUIRED

Sector orientation

Sector 16 glovebox training

**Training checklist distribution to the User (See [Appendix A](#))**

# HPCAT Sector 16

**PROCEDURE**

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Procedure #:

Revision #:

## Authorized Users

NAME	Badge #	Date	Signature

## Appendix A

### Training Checklist for Sector 16 Glovebox

- [ ] Samples rules
    - **No volatile sulfur-containing compounds or volatile phosphines.**
    - **Report any volatile samples** (e.g. those that sublime or have solvents or liquids in them): Talk to GB contacts first to obtain approval
    - Sample containers should be filled with an inert atmosphere, and capable of maintaining low levels of O<sub>2</sub> and H<sub>2</sub>O during transport
  - [ ] Before working in the GB
    - Inspect GB gloves before using them to ensure no holes are present
    - *Report immediately the presence of any holes in the gloves to a GB Contact*
    - Check the O<sub>2</sub> level in the glovebox before use (see below) Do not work in the GB if O<sub>2</sub> level is too high (> 5 ppm of H<sub>2</sub>O)
    - Check logbook (other Users might have samples or equipment in the antechambers) **EVERY TRANSFER AND ACTION THAT HAPPENS IN THE GB MUST BE RECORDED IN THE LOG BOOK.** Log O<sub>2</sub> levels for every entry in the logbook.
  - [ ] Parts of the glovebox and their function:
    - The antechambers is for transferring samples in and out of the GB
    - Vacuum pump evacuates the ante chambers,
    - Oxygen sensor detect O<sub>2</sub> levels
    - Pedal is for controlling the pressure of the GB while in use
    - A control panel is on the front of the GB by the antechambers (do *NOT* adjust the glovebox pressure settings)
  - [ ] Glovebox sensors
    - Normal O<sub>2</sub> levels are < 5 ppm
    - *Problems with the sensor must be reported to one of the GB Contacts*
  - [ ] Glovebox pressure:
    - Range: +2 to +8 mbar
    - Do not change the ranges
  - [ ] Large antechamber
    - Normal operation: use 3 10-minute evacuation/refill cycles before transferring anything in to the glovebox.
    - Paper products contain a lot of moisture. Avoid bringing these materials into the glovebox (if necessary talk to the GB contacts)
  - [ ] General Rules for Working in the GB:
    - Any work inside the GB or use of antechambers must be recorded in the logbook
    - Wear clean gloves before inserting hands into GB gloves
    - Preferable to wear long sleeve shirts or lab coat while in the GB
    - Aluminum foil must be placed in the work area to collect spilled samples
- **Avoid sharps (razors, scissors, ...)**, they should not be placed in the GB without authorization.
  - Store sharps out of reach of the gloves, and away from the edges of the GB
  - Any sharps should be clearly marked to distinguish them from the metallic background of the GB
- [ ] Glovebox Contacts:
- |                         |                          |
|-------------------------|--------------------------|
| 1. Jesse Smith          | Bldg. 434 E; Ext. 2-4017 |
| 2. Curtis Kenney-Benson | Bldg. 434 E; Ext. 2-0495 |
| 3. Guoyin Shen          | Bldg. 434 E; Ext. 2-0429 |
- \_\_\_\_\_  
Name of Trainee                      Signature
- \_\_\_\_\_  
Badge Number                      Date
- \_\_\_\_\_  
Name of Trainer                      Signature
- \_\_\_\_\_  
Badge Number                      Date