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Standard Operating Procedure for Sector 10 Glovebox in Bldg 433 B030

Changes made in this revision:

- N/A (new procedure)

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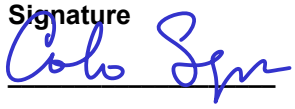
Beamline	Carlo Segre		December 13, 2011

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Standard Operating Procedure for Sector 10 Glovebox in Bldg 433 B030

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 433 B030. 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. The hazards analysis for this SOP are outlined in an Argonne Non-experimental Work Planning and Control Document (NWCD-CSE-CRG04-2011).

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 John Katsoudas, Jeff Miller, and Elizabeth Mader.

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_2O 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 and inert atmosphere and powders must be properly dried to prevent the introduction of adsorbed water.
Non-protic solvents and volatile solids are allowed only after approval from the GB contacts. The use of carbonyl-containing solvents (e.g. 2-propanone) will be evaluated on a case-by-case basis and is ***STRONGLY DISCOURAGED***. In general, liquids should be anhydrous (either by pre-treatment or purchased anhydrous) for use in the glovebox. Solvent containers need to be very well sealed and taped up before bringing into the glovebox antechamber to avoid solvent leakage into the pump and minimize shattered glass in case of a broken container. Solvent bottles must remain closed when the glovebox is not in use. Any solvents brought into the glovebox need to be removed and properly disposed of at the end of the experimental run.
Sample containers should be filled with inert atmosphere, and capable of maintaining low levels of O_2 and H_2O 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, Boron nitride) 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 users

1.6 Glovebox atmosphere

The glovebox operating parameters should not to be modified.

Recommended O_2 operating limits as indicated on the O_2 meter are normally <5 ppm of O_2
Care must be exercised in using tools (e.g., scissors, forceps) which could damage the gloves.
Watch for and report low pressure or unusual H_2O and O_2 levels.
Inform GB contacts if O_2 levels are above 5 ppm

2 GLOVEBOX OPERATION

The usual operating pressure of the glovebox is +1 to +4 inches of water 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 dynamic 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 (A) and slowly opening the antechamber to nitrogen with the N_2 valve (B). *IMPORTANT: Do not open the N2 valve (B) fully. Do not open the valve enough that the box pressure falls below 0" water on the pressure gauge (e.g. the pressure inside the box goes negative).*
3. Once the chamber is at atmospheric pressure, close the N2 valve (B).
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 (A).
6. Wait for 10 min.
7. Refill the antechamber half way by opening the nitrogen valve (B).
8. Repeat steps 5 and 6 two more times.
9. Refill the antechamber completely by opening the nitrogen valve (B).
10. Once the chamber is full, close the nitrogen valve (B).
11. Open the inner antechamber door. Move items in to the GB. Close the door.
12. Evacuate the antechamber by opening the vacuum valve (A).

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 (A) and slowly opening the antechamber to nitrogen with the N_2 valve (B). *IMPORTANT: Do not open the N2 valve (B) fully. Do not open the valve enough that the box pressure falls below 0" water 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 (A).

2.3 Introducing solvents into the glovebox (REQUIRES APPROVAL AND SPECIAL TRAINING)

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 (A) and slowly opening the antechamber to nitrogen with the N_2 valve (B). *IMPORTANT: Do not open the N2 valve (B) fully. Do not open the valve enough that the box pressure falls below 0" water on the pressure gauge (e.g. the pressure inside the box goes negative).*
3. Once the chamber is at atmospheric pressure, close the N_2 valve (B).
4. Open the outer antechamber door. Place the solvent containers into the pull out tray. Close the door. (Solvent containers must have tightly sealed to avoid solvent leakage and taped up to contain shattered glass in case of a broken container).
5. Evacuate the antechamber *by no more than half way* by opening the vacuum valve (A).
6. Refill the antechamber *to just under atmospheric pressure* by opening the nitrogen valve (B).
7. Repeat steps 5 and 6 fifteen more times.
8. Refill the antechamber completely by opening the nitrogen valve (B).
9. Once the chamber is full, close the nitrogen valve (B).
10. Open the inner antechamber door. Move solvents into to the GB. Close the door.
11. Evacuate the antechamber by opening the vacuum valve (A).

2.4 Using solvents in the glovebox (REQUIRES APPROVAL AND SPECIAL TRAINING)

1. Check that no other samples are open inside the glovebox.
2. Make a note of the solvent usage in the glovebox log.
3. Turn off the circulation fans by unplugging the unit (C).
4. Close both circulation valves (D).

5. Open solvents and perform work. Keep a close watch on the O_2 level. If this rises above 5 ppm, open the glovebox vent valve (E).
6. Upon completion of the solvent work (or after 30 minutes, whichever comes first), purge the glovebox for at least 10 minutes by opening the glovebox purge valve (E).
7. Close the purge valve (E) and open the circulation valves (D). Plug in the circulation fans (C).

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.

Description of Document/Record (include ID number, if applicable)	Custodian	Storage Location and Medium	Retention Requirement
Training Checklist for Sector 10 Glovebox	John Katsoudas	In a sleeve attached to the Glove Box	Destroy 75 years after employee termination (DOE ADM 1.29.1.b)

4 TRAINING REQUIRED

Sector orientation
Sector 10 glovebox training
Sector 10 glovebox solvent training (optional)

Training checklist distribution to the User (See Appendix A)

5 FEEDBACK AND IMPROVEMENT

If you are using this procedure and have comments or suggested improvements for it, please go to the APS Policies and Procedures Comment Form* to submit your input to a Procedure Administrator. If you are reviewing this procedure in workflow, your input must be entered in the comment box when you approve or reject the procedure.

Instructions for execution-time modifications to a policy/procedure can be found in the following document: Field Modification of APS Policy/Procedure (APS_1408152).

* http://centraldocs.aps.anl.gov/comment_form.php

Appendix A

Training Checklist for Sector 10 Glovebox

[ ] Samples rules
- **No volatile sulfur-containing compounds or volatile phosphines**.
- **Only approved solvents** are allowed in the GB. When solvents are opened to the GB atmosphere, turn off the circulation system (no more than 30 minutes max). Purge the box for at least 10 minutes upon completion of solvent work. Turn on the circulation system.
- **Report any volatile samples** (e.g. those that sublimate 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 O2 and H2O 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_2 level in the glovebox before use (see below) Do not work in the GB if O_2 level is too high (> 5 ppm of H2O)
- 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_2 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_2 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 O2 levels are < 5 ppm
- *Problems with the sensor must be reported to one of the GB Contacts*

[ ] Glovebox pressure:
- Range: +1 to +4 inches water
- 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 brining 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.
- Keep 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. John Katsoudas Bldg. 433 B001; Ext. 2-0351
2. Jeff Miller Bldg. 200 E185; Ext. 2-1928
3. Elizabeth Mader Bldg. 200 E165; Ext. 2-3014

I have read and understood the SOP for glovebox use, including the specific areas of knowledge listed above.

_____ _____
Name of Trainee Signature

_____ _____
Badge Number Date

_____ _____
Name of Trainer Signature

_____ _____
Badge Number Date