

## Experiment Safety Review Form Review Number: NC-01-2010

PRINCIPAL INVESTIGATOR: Charles Black

GROUP: NC

EXT: 4397

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LIFE NUMBER: 23566

**Project Title:** High-Resolution Scanning Electron Microscopy and x-ray microanalysis

**Location(s):** 0735

**Area(s):** 0735-FIRST-1-1L32

**Proposed Start Date and Duration:** 6/11/2010 - 1 years

### SIGNATURES:

**Principal Investigator:** Charles Black

**Date:** 2/1/2011

**Experiment Review Coordinator:** Robert Sabatini

**Date:** 2/1/2011

**Co-PI or Alternate Contact (s):** Fernando Camino

**Date:** 2/2/2011

**Co-PI or Alternate Contact (s):** Aaron Stein

**Date:** 2/2/2011

**Reviewer:** Lorraine Davis

**Date:** 12/16/2010

**Approval:** Emilio Mendez

**Date:** 7/1/2010

**Review/Approval (ERC) Comments:**

02/01/2011 12:42 AM

**Walkthrough Signature:** Robert Sabatini

**Date:** 2/2/2011

**Expiration Date (max 1 yr.):** 2/1/2012

**FUA Change Required?** No

**Fire Rescue Run Card Changes Required?** No

**Has a NEPA Review been Performed for this Project?** Yes

**Required Approvals (i.e., IACUC, IBC, etc.):**

**Project Termination Acceptance Signature:**

**Date:**

**Comments:**

## I. Define the Scope of the Work

### A. Description

Laboratory 1L-32 hosts two scanning electron microscopes (SEMs). The Hitachi S-4800 is an ultra-high resolution field emission SEM. The JEOL JSM-7600F is a thermal field effect SEM that, besides high resolution imaging, possess x-ray analysis instrumentation, namely, an energy dispersive spectrometer (EDS) and a wavelength dispersive spectrometer (WDS)

Access to and exit from lab 1L32 is done exclusively via the service galley that connects to this lab (see attached lab layout). In case of an emergency, users can exit either via the service galley or via the exit that connects to lab 1L31. Lab 1L31 hosts a low energy transmission electron microscope, and for this reason, transit via 1L31 is forbidden during normal conditions.

The materials to be imaged and analyzed in this laboratory consist of thin film organic, metallic, semiconducting, and insulating material samples. MSDSs are used to evaluate material hazards and special handling requirements.

Sample specimens are prepared and mounted on SEM sample holders on two workbenches located on opposite walls of the lab: one workbench for each SEM. Sample specimens consist of thin material films on rigid substrates, typically silicon or glass. These are mounted onto sample holders using either mechanical spring clips, double-sided carbon tape, or in some cases silver paint. NITRILE GLOVES are worn at ALL TIMES when handling sample specimens, sample holders, or any other materials to be introduced into the vacuum chambers of any of the two SEMs.

Sample specimens may involve nanomaterials and users will consult the Interim Procedure for Nanomaterial Handling found in SBMS. Nanomaterial samples to be imaged or analyzed by SEM will have already been put into a fixed form (i.e., fixed to a surface or otherwise immobilized and prevented from aerosolizing) following the Interim Procedure prior to being brought into the laboratory.

Sample specimens may be blown clean using a stream of compressed dry  $N_2$  gas from a pressure-regulated gas cylinder or using dry air from a small can. **NOTE: Samples that are at risk of aerosolizing must be blown clean in an approved exhaust hood.**

It is occasionally necessary to clean sample holders with organic solvents (ONLY isopropanol or ethanol) in order to remove residual sample mounting tape. If a significant amount of solvent is used (>100ml) then these cleaning procedures are carried out in chemical fume hoods (e.g., a hood in lab 1L-10) using the appropriate PPE (safety glasses with side shields and nitrile gloves). Smaller amounts of isopropanol or ethanol will be used at the lab bench in 1L-32 and dispensed from squeeze bottles.

Sample holders with specimens are loaded into the microscopes through vacuum loadlocks.

To reduce sample contamination during imaging, both SEMs have the option to cool the sample stage with  $LN_2$ , which is brought into the lab using a conventional  $LN_2$  dewar (4 liters) with loose cap to prevent overpressurization. A procedure for filling the  $LN_2$  dewar is appended to this ESR. Tool operators will wear appropriate PPE and respect the hazards of  $LN_2$  when performing this operation. Required PPE are (1) cryogenic gloves, (2) face shield, and (3) safety glasses with side shields or safety goggles. There is NO  $LN_2$  storage in the laboratory. See appended cryo filling procedure.

#### **Equipment manuals or procedures that are controlled documents:**

Located in lab.

## **B. Human Performance Factors**

The primary safety hazard associated with this laboratory is exposure to cryogenics when filling the small liquid nitrogen container attached to each SEM. We control this hazard by wearing appropriate PPE located in the back of the laboratory (safety glasses with side shields, face shield, cryogenic gloves)

## **C. Waste Minimization/Pollution Prevention**

We introduce samples into the microscopes via small loadlock chambers to minimize the volume of nitrogen gas used in venting the systems.

All images are captured and stored digitally to minimize paper usage.

Minimal quantities of solvents are used during sample preparation and cleaning. We will utilize multi-use double-sided tape and spring-loaded sample clips rather than silver paint for fixing samples to sample holders except in cases where paint is absolutely necessary.

We will use lint-free cloths as work surfaces during sample preparations with each cloth serving multiple uses prior to disposal.

## D. Materials Used /Waste Generated

Materials Used	Disposal Method	Amount per Use	Amount per Year	Comments
see attachment		0.00	0.00	

## II. Identify and Analyze Hazards Associated with the Work

The following hazards were identified:

### Physical Hazards:

- Cryogenics (any substance or device capable of producing temperatures  $\leq 170\text{K}$ )
- Compressed gases (lecture bottles, cylinders, gas lines)
- Flammable liquids

### Chemical Hazards:

- Nano-material bound in a solid matrix or fixed substrate
- Toxic metals (e.g., As, Ba, Be, Cd, Cr, Hg, Pb, Se, Ag)
- [On-site](#) or [off-site](#) transportation of chemicals (see SBMS)

### Ionizing and Non-ionizing Radiation Hazards:

- Radiation generating devices- exempt

### Biological Hazards:

- None

### Offsite Work:

- None

### Other Issues (Security, Notifications, Community, etc.):

- None

### Significant Environmental Aspects

- Any amount of hazardous waste generation
- Any amount of industrial waste generation (e.g., oils, vacuum pump oil)
- spill potential (Other)

## III. Develop and Implement Hazard Controls and Assess Risk

### A. Physical Hazards, Tasks and Controls

Hazard, Default Controls, Task Specific Info	Risk Level
<u>Hazard:</u> Cryogenics (any substance or device capable of producing temperatures $\leq 170\text{K}$ )	Negligible (0-20)

**Default Controls:**

General Requirements:

- Evaluate location oxygen deficiency
- Store/transport only in approved containers (i.e. DOT/ASME or BNL LESHG)
- Never pour from above chest level
- PPE: Long Sleeve Shirt (or Lab Coat), long pants (or skirt covering ankles) and closed shoes

Pressurized transfer to open (vented) container; Or-Pouring &gt; 5 liter volumes of LN2 between open containers:

- Face shield along with either Safety Glasses (w/side shields) or Goggles
- Gloves (Cryo or Heavy Leather)

Pouring small (5 liters or less) volumes of LN2 between open containers:

- Safety Goggles (face shield recommended)
- Gloves (Cryo or Heavy Leather)

Work with samples immersed in LN2 in small (~1 liter) dewars:

- Use Tongs (tools) to manipulate/handle cryogenic samples (do not touch with gloves)• Use insulated non-absorbent gloves with dexterity (cotton/nylon gloves under disposable nitrile gloves)
- Safety Goggles

**Hazard: Compressed gases (lecture bottles, cylinders, gas lines)****Negligible (0-20)****Default Controls:**

- Any systems >15psi must be SME Approved
- Transport cylinders using a cylinder cart
- Secure cylinders to a fixed object/wall
- Use regulator, hoses, and components compatible with gas
- Use hoses and clamps rated for maximum regulator output or use pressure relief device
- Wear safety glasses with side shields when installing/removing/or adjusting regulator
- Label piping/tubing

**Hazard: Flammable liquids****Negligible (0-20)****Default Controls:**

As for chemicals, plus  
Store large quantities in Flam. cabinets as required

**B. Chemical Hazards, Tasks and Controls****Hazard, Default Controls, Task Specific Info****Risk Level****Hazard: Nano-material bound in a solid matrix or fixed substrate****Negligible (0-20)****Default Controls:**

PPE Requirements for Handling: Standard PPE required for the work area. No additional requirements.

Handling Requirements

- No Mechanical abrasion.
- No thermal stresses.
- No etching

Laboratory Posting Requirements - No Posting Requirements

Waste Handling:

- Solids containing nanomaterials which have a potential to be released must NOT be disposed in the regular trash. See SBMS Hazardous Waste Management for more information.
- Collect solid UNP waste in a bag (6 mil thick minimum zip-lock type or J-sealed) or other sealing container (i.e. jar with threaded lid).

- Spell out the chemical name (do not use formulas or trade names) on the RED Hazardous Waste Label.

- The contents line on the label must contain the chemical composition and the word "NANO"
- A second label, in addition to the Red Hazardous Waste Label, is required on the outside

other container/bag stating "CONTAINS NANOMATERIALS" see ES&H Coord. or 90-Day area manager for labels.	
<b>Hazard:</b> Toxic metals (e.g., As, Ba, Be, Cd, Cr, Hg, Pb, Se, Ag) <b>Default Controls:</b> As for chemicals, plus need for SHSD and OMC monitoring and surveillance must be evaluated BURF for Beryllium operation	Negligible (0-20)
<b>Hazard:</b> <u>On-site</u> or <u>off-site</u> transportation of chemicals (see SBMS) <b>Default Controls:</b>	Negligible (0-20)

**C. Environmental Hazards, Tasks and Controls (include on/off site transportation and products/services)**

Hazard, Default Controls, Task Specific Info	Risk Level
<b>Hazard:</b> Any amount of hazardous waste generation <b>Default Controls:</b> Engineering Controls • Waste will be accumulated in chemically compatible containers that appropriately contain/protect the waste. • Waste containers will be closed in a tray (secondary containment) in the Satellite Accumulation Area (SAA).  Administrative Controls • All hazardous waste containers will have a (red) "Hazardous Waste Label" that has the generator's name and the chemical contents (trade name/formula not acceptable). • All waste will be accumulated in closed containers and kept in an established and posted SAA until ready for transfer to the 90-Day Haz Waste Area for pick-up by Waste Management. • For pick-up by Waste management, complete the Nonradioactive Haz Waste Control Form and consult the 90-Day Area Manager to gain access/transfer the waste to the 90Day Area.  Training: Hazardous Waste Gen. (HP-RCRIGEN3).  PPE: When handling waste materials follow PPE requirements specified for the specific materials.  Comply with the SBMS Subject Area: "Hazardous Waste Management".	Negligible (0-20)
<b>Hazard:</b> Any amount of industrial waste generation (e.g., oils, vacuum pump oil) <b>Default Controls:</b> Engineering Controls • Store only compatible wastes together, in suitable containers. • Provide secondary containment for liquid wastes if potential for environmental release exists. • Keep containers closed and secured unless adding waste to container.  Administrative Controls • Use a green industrial waste label, with generator's name and chemical contents (trade name/formula NOT acceptable). Label oils "Used Oil". • When full, complete and submit a WCF for pick up. The waste may be stored in the 90-day area.  Training: Hazardous Waste Generator (HP-RCRIGEN3)  Comply with the SBMS Subject Area: "Industrial Waste".	Negligible (0-20)

<b>Hazard:</b> spill potential (Other)	<b>Negligible (0-20)</b>
<b>Default Controls:</b>	

#### D. Radiation Hazards, Tasks and Controls

Hazard, Default Controls, Task Specific Info	Risk Level
<b>Hazard:</b> Radiation generating devices- exempt	<b>Negligible (0-20)</b>
<b>Default Controls:</b> If shielding is altered contact FSS for surveys prior to restart.	

#### E. Biological Hazards, Tasks and Controls

None

#### F. Offsite Work Hazards, Tasks and Controls

None

#### G. Other Issues (Security, Notifications to Other Organizations, Community Involvement, etc.)

None

#### H. Recommended Exposure Monitoring

- None

Description or comments:

#### I. EPHA Determination

Chemical Name	Quantity (lbs, gal)	Location (Bldg/Room#)

### IV. Perform Work Within Controls

#### A. Recommended Training and Medical Surveillance Summary

- Laboratory Standard (HP-IND-220)
- Cryogen Safety (HP-OSH-025)
- Hazardous Waste Generator (HP-RCRIGEN3)
- Nanoparticles Protocol (OM-MEDSURV-NANO)
- Compressed Gas Safety (TQ-COMPGAS1)
- Nanotechnology for Nano-workers (TQ-NC-HS2)

#### B. Personnel Training, Qualification, and Authorization List

Employee/Guest Name	Life/Guest#	Dept	Required Training Course(s)	Signed
Chuck Black	23566	NC	Cryogen Safety (HP-OSH-025) [ EXPIRES: NEVER ] Compressed Gas Safety (TQ-COMPGAS1) [ EXPIRES: 2/12/2013 ] Laboratory Standard (HP-IND-220) [ EXPIRES: 11/15/2012 ] Nanotechnology for Nano-workers (TQ-NC-HS2) [ EXPIRES: 6/4/2012 ] Nanoparticles Protocol (OM-MEDSURV-NANO) [ UNASSIGNED: INCOMPLETE ] Hazardous Waste Generator (HP-RCRIGEN3) [ EXPIRES: 11/15/2011 ]	1/11/2011 8:27:05 AM
			Cryogen Safety (HP-OSH-025) [ EXPIRES: NEVER ] Compressed Gas Safety (TQ-COMPGAS1) [ EXPIRES: 3/2/2013 ] Laboratory Standard (HP-IND-220) [ EXPIRES:	

Aaron Stein	22947	NC	<p>2/28/2013 ]</p> <p>Nanotechnology for Nano-workers (TQ-NC-HS2) [ EXPIRES: 6/4/2012 ]</p> <p>Nanoparticles Protocol (OM-MEDSURV-NANO) [ UNASSIGNED: EXPIRES: NEVER ]</p> <p>Hazardous Waste Generator (HP-RCRIGEN3) [ EXPIRES: 4/11/2012 ]</p>	12/1/2010 11:58:37 AM
Chang-Yong Nam	23659	NC	<p>Cryogen Safety (HP-OSH-025) [ EXPIRES: NEVER ]</p> <p>Compressed Gas Safety (TQ-COMPGAS1) [ EXPIRES: 3/4/2013 ]</p> <p>Laboratory Standard (HP-IND-220) [ EXPIRES: 5/27/2013 ]</p> <p>Nanotechnology for Nano-workers (TQ-NC-HS2) [ EXPIRES: 4/27/2012 ]</p> <p>Nanoparticles Protocol (OM-MEDSURV-NANO) [ UNASSIGNED: EXPIRES: NEVER ]</p> <p>Hazardous Waste Generator (HP-RCRIGEN3) [ EXPIRES: 3/8/2012 ]</p>	11/24/2010 4:45:11 PM
Weiqiang Han	23205	NC	<p>Cryogen Safety (HP-OSH-025) [ EXPIRES: NEVER ]</p> <p>Compressed Gas Safety (TQ-COMPGAS1) [ EXPIRES: 2/23/2013 ]</p> <p>Laboratory Standard (HP-IND-220) [ EXPIRES: 6/9/2012 ]</p> <p>Nanotechnology for Nano-workers (TQ-NC-HS2) [ EXPIRES: 5/24/2012 ]</p> <p>Nanoparticles Protocol (OM-MEDSURV-NANO) [ UNASSIGNED: EXPIRES: NEVER ]</p> <p>Hazardous Waste Generator (HP-RCRIGEN3) [ EXPIRES: 3/3/2012 ]</p>	9/7/2010 9:58:24 AM
Dmytro Nykypanchuk	23299	NC	<p>Cryogen Safety (HP-OSH-025) [ EXPIRES: NEVER ]</p> <p>Compressed Gas Safety (TQ-COMPGAS1) [ EXPIRES: 2/8/2013 ]</p> <p>Laboratory Standard (HP-IND-220) [ EXPIRES: 11/23/2012 ]</p> <p>Nanotechnology for Nano-workers (TQ-NC-HS2) [ EXPIRES: 4/27/2012 ]</p> <p>Nanoparticles Protocol (OM-MEDSURV-NANO) [ UNASSIGNED: EXPIRES: NEVER ]</p> <p>Hazardous Waste Generator (HP-RCRIGEN3) [ EXPIRES: 11/29/2011 ]</p>	6/27/2011 1:47:37 PM
Eli Sutter	23099	NC	<p>Cryogen Safety (HP-OSH-025) [ EXPIRES: NEVER ]</p> <p>Compressed Gas Safety (TQ-COMPGAS1) [ EXPIRES: 3/8/2013 ]</p> <p>Laboratory Standard (HP-IND-220) [ EXPIRES: 1/4/2012 ]</p> <p>Nanotechnology for Nano-workers (TQ-NC-HS2) [ EXPIRES: 5/5/2012 ]</p> <p>Nanoparticles Protocol (OM-MEDSURV-NANO) [ UNASSIGNED: EXPIRES: NEVER ]</p> <p>Hazardous Waste Generator (HP-RCRIGEN3) [ EXPIRES: 12/29/2011 ]</p>	
Fernando Camino	23799	NC	<p>Cryogen Safety (HP-OSH-025) [ EXPIRES: NEVER ]</p> <p>Compressed Gas Safety (TQ-COMPGAS1) [ EXPIRES: 3/4/2013 ]</p> <p>Laboratory Standard (HP-IND-220) [ EXPIRES: 9/2/2011 ]</p> <p>Nanotechnology for Nano-workers (TQ-NC-HS2) [ EXPIRES: 4/29/2012 ]</p> <p>Nanoparticles Protocol (OM-MEDSURV-NANO) [ UNASSIGNED: EXPIRES: NEVER ]</p> <p>Hazardous Waste Generator (HP-RCRIGEN3) [ EXPIRES: 8/23/2011 ]</p>	4/13/2011 3:09:24 PM
			Cryogen Safety (HP-OSH-025) [ EXPIRES: NEVER ]	

Don Elliott	14624	IO	Compressed Gas Safety (TQ-COMPGAS1) [ EXPIRES: 3/6/2013 ] Laboratory Standard (HP-IND-220) [ EXPIRES: 7/20/2012 ] Nanotechnology for Nano-workers (TQ-NC-HS2) [ EXPIRES: 5/8/2012 ] Nanoparticles Protocol (OM-MEDSURV-NANO) [ UNASSIGNED: EXPIRES: NEVER ] Hazardous Waste Generator (HP-RCRIGEN3) [ EXPIRES: 3/18/2012 ]	
Jon Allen	24101	NC	Cryogen Safety (HP-OSH-025) [ EXPIRES: NEVER ] Compressed Gas Safety (TQ-COMPGAS1) [ EXPIRES: 2/9/2013 ] Laboratory Standard (HP-IND-220) [ EXPIRES: 12/7/2012 ] Nanotechnology for Nano-workers (TQ-NC-HS2) [ EXPIRES: 4/27/2012 ] Nanoparticles Protocol (OM-MEDSURV-NANO) [ UNASSIGNED: EXPIRES: NEVER ] Hazardous Waste Generator (HP-RCRIGEN3) [ EXPIRES: 12/7/2011 ]	
Ming Lu	24188	NC	Cryogen Safety (HP-OSH-025) [ EXPIRES: NEVER ] Compressed Gas Safety (TQ-COMPGAS1) [ EXPIRES: 4/8/2012 ] Laboratory Standard (HP-IND-220) [ EXPIRES: 4/11/2013 ] Nanotechnology for Nano-workers (TQ-NC-HS2) [ EXPIRES: 4/30/2012 ] Nanoparticles Protocol (OM-MEDSURV-NANO) [ UNASSIGNED: INCOMPLETE ] Hazardous Waste Generator (HP-RCRIGEN3) [ EXPIRES: 4/7/2012 ]	5/11/2011 9:22:31 AM
Matt Sfeir	23401	NC	Cryogen Safety (HP-OSH-025) [ EXPIRES: NEVER ] Compressed Gas Safety (TQ-COMPGAS1) [ EXPIRES: 2/8/2013 ] Laboratory Standard (HP-IND-220) [ EXPIRES: 10/27/2011 ] Nanotechnology for Nano-workers (TQ-NC-HS2) [ EXPIRES: 4/21/2012 ] Nanoparticles Protocol (OM-MEDSURV-NANO) [ UNASSIGNED: EXPIRES: NEVER ] Hazardous Waste Generator (HP-RCRIGEN3) [ EXPIRES: 3/25/2012 ]	5/17/2011 4:28:40 PM
Ranjith Kirshna Pai	n8020		Cryogen Safety (HP-OSH-025) [ UNASSIGNED: INCOMPLETE ] Compressed Gas Safety (TQ-COMPGAS1) [ UNASSIGNED: INCOMPLETE ] Laboratory Standard (HP-IND-220) [ UNASSIGNED: INCOMPLETE ] Nanotechnology for Nano-workers (TQ-NC-HS2) [ UNASSIGNED: INCOMPLETE ] Nanoparticles Protocol (OM-MEDSURV-NANO) [ UNASSIGNED: INCOMPLETE ] Hazardous Waste Generator (HP-RCRIGEN3) [ UNASSIGNED: INCOMPLETE ]	

### C. Emergency Procedures

Follow the Bldg. 735 Local Emergency Plan. Spill kits located in rear lobby and in 90-day area. Eyewash and shower in Service Galley outside of 1L-32 and in Lab 1L-10

### D. Transportation

Laboratory users will bring samples for SEM imaging and/or x-ray analysis. Users should be aware of the guidelines for transporting hazardous materials. Samples arrive in the laboratory affixed to a solid substrate (e.g., silicon wafer or glass



slide) for mounting on the SEM specimen sample holder. Sample preparation other than that described in this document must be done in an environment appropriate for chemical handling.

## **E. Notifications**

ESH Coordinator and Building Manager

## **F. Termination/Decommissioning**

Project termination will go through an ERE

## **V. Provide Feedback**

The controls in place have adequately anticipated and mitigated hazards associated with working in this laboratory.

## **VI. Attachments**

### **Attached Files:**

[Bldg 735 SMF 1L32 SEM microscope.pdf](#)

[Materials Used NC01 1L32.doc](#)

[Operating procedure for Hitachi S-4800 SEM.doc](#)

[1L32-layout.JPG](#)

[cryo filling procedure.pdf](#)

[Operating procedure for JEOL7600F.pdf](#)