



STANDARD OPERATING PROCEDURES
DEVELOPED TO SUPPLEMENT THE BROWN UNIVERSITY CHEMICAL HYGIENE PLAN &
LABORATORY SAFETY MANUAL

SOP Part 1, Brief Description of Research – Provide a brief description of the type of research being conducted in the laboratory covered by this plan.

SOP PART 1 – BRIEF DESCRIPTION OF RESEARCH

This lab is the Electron Microscope Facility and is used to investigate nontoxic solids (metal alloys, semiconductors, ceramics, and polymers) by scanning and transmission electron microscopes and optical microscopes. The FEI/Philips CM20 TEM (020A), LEO 1530VP SEM (020E), JEOL 2100F STEM(020C), FEI/ThermoScientific FIB (020D) and ThermoScientific Quattro ESEM (020G) are in this facility. Sample conductive coating equipment and plasma cleaning equipment are also located in this lab. Wet chemicals found in the lab (020) are solvents such as methanol, acetone, and isopropyl alcohol, epoxies, water and oil based diamond suspensions, and oil for vacuum pumps. Once used the chemicals are all disposed of in properly labeled waste bottles. Liquid Nitrogen cryogenic liquid and Nitrogen, Oxygen, and Argon compressed gases are also in the lab (020B and 020F). The first aid and spill kits are all kept in room 020, near the main entrance.

The FEI Helios 600 Dualbeam SEM (also referred to as FIB or focused ion beam) equipment is in room 020D. In addition to high voltage electrons to produce scanning electron microscope images this equipment can produce Gallium ion beams of up to 30 keV used to form secondary electron images or to remove material from the sample by sputtering. The Gallium ion beam or electron beam can also be used to do assisted deposition of Platinum metal or do selective removal of Carbon from precursor gases.

Sample preparation for the Quattro SEM using Chromic acid will take place in the fume hood located in ERC 020. The Chromic acid will be stored in an acid cabinet in ERC 020B. There is very limited access to ERC 020B. See SOP Part 8 for additional information.



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SOP Part 2, List of Common Chemicals – Provide a list of the most commonly used chemicals in the laboratory along with hazard information, compatibility, and special handling instructions for each chemical listed.

Assistance and resources for determining hazard information, and compatibility, may be obtained by contacting the University Chemical Hygiene Officer at 863-1737 or by browsing the resources available at the Brown University EHS website:

<http://www.brown.edu/Administration/EHS/lab>

SOP PART 2 - LIST OF COMMON CHEMICALS

<i>Full Chemical Name</i>	<i>Hazards</i>	<i>Incompatibilities</i>	<i>Handling Instructions</i>
Acetone	Flammable Skin, eyes, respiratory tract irritant Affects central nervous system	Concentrated nitric and sulfuric acid mixtures, oxidizing materials, chloroform, alkalis, chlorine compounds, acids, potassium t-butoxide	Keep away from heat, sparks and flame. Keep container closed. Use only with adequate ventilation. Wash thoroughly after handling. Avoid breathing vapor. Avoid contact with eyes, skin and clothing.
Alumina Suspensions	May cause drying of the skin minor eye irritation and create a dust nuisance	Strong acids and oxidizing agents.	Wear appropriate protection and use adequate ventilation. Keep in a closed container when not in use. Store in a cool, dry place.
Argon, Compressed	Difficulty breathing	No data available	Provide ventilation and wear appropriate chemical resistant gloves.
Apiezon L	Transient eye irritation and skin disorders.	Avoid strong oxidizing agents. Avoid temperatures > 120C/248F.	No special precautions.
Colloidal Silica	Mild skin and eye irritation	Do not mix this product with any chemical that will lower the pH.	Keep containers tightly closed. Store at temperatures above 2C (35F).
Diamond suspension	Minor irritation if inhaled or on skin. Mechanical irritation in eyes.	Strong acids, bases, or oxidizing agents; particularly when hot.	Store in a cool dry place. Keep container closed when not in use. Wear suitable



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<i>Full Chemical Name</i>	<i>Hazards</i>	<i>Incompatibilities</i>	<i>Handling Instructions</i>
			protection; avoid unnecessary skin contact.
Dust Off 1,1-Difluoroethane	Inhalation or high concentrations of vapor is harmful and may cause heart irregularities, unconsciousness, or death	Alkali or alkaline earth metal – powdered Al, Zn, Be, etc.	Avoid high temperatures and open flames.
Glycerol	May cause nausea, headache, and diarrhea. Skin and eye irritation. May cause kidney injury.	Strong oxidizers. Can react violently with acetic anhydride, calcium oxchloride, chromium oxides and alkali metal hydrides. Avoid heat, flames, and ignition sources.	Keep in a tightly closed container, store in a cool, dry, ventilated area. Protect against physical damage. Isolate from incompatible substances. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid).
Isocut fluid	Harmful if swallowed. Skin irritant. Mechanical eye irritant. Liquid aspirated into lungs may cause chemical pneumonitis.	Strong bases or oxidants.	Wash hands before eating. Avoid prolonged or repeated contact with skin. DO NOT take internally. FOR INDUSTRIAL USE ONLY. Store away from heat. Keep out of the reach of children.
Isopropyl Alcohol	Flammable liquid and vapor. Harmful if swallowed or inhaled. Causes irritation to the eyes and respiratory tract. Affects central nervous system. May be harmful if absorbed through skin. May cause irritation to skin.	Heat, flame, strong oxidizers, acetaldehyde, acids, chlorine, ethylene oxide, hydrogen-palladium combination, hydrogen peroxide-sulfuric acid combination, potassium tert-butoxide, hypochlorous acid, isocyanates, nitroform, phosgene, aluminum, oleum and perchloric acid.	Protect against physical damage. Store in a cool, dry well-ventilated location, away from any area where the fire hazard may be acute. Outside or detached storage is preferred. Separate from incompatibles. Containers should be bonded and grounded for transfers to avoid static sparks. Storage and use areas should be No Smoking areas. Use non-sparking type tools and equipment, including explosion



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<i>Full Chemical Name</i>	<i>Hazards</i>	<i>Incompatibilities</i>	<i>Handling Instructions</i>
			proof ventilation. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product. Small quantities of peroxides can form on prolonged storage. Exposure to light and/or air significantly increases the rate of peroxide formation. If evaporated to a residue, the mixture of peroxides and isopropanol may explode when exposed to heat or shock.
M-Bond 610 Adhesive	Flammable. May cause respiratory irritation, central nervous system depression, severe irritation and pain on contact of the eye. Vapors may irritate eyes. May cause severe irritation or burns of the skin. Absorption into the skin may cause dermatitis.	Avoid heat, flame, other sources of ignition, light and air. Avoid subjecting resin to temperatures above 90F/32C. Avoid acids, strong oxidizing agents, strong bases, strong reducing agents.	Keep containers tightly closed. Store in a cool, dry well ventilated, flammable liquid storage area. Take precautionary measures against static discharges. Keep away from open flame and spark producing equipment. Keep product out of light. Use caution when opening cap. Avoid prolonged exposure to vapors and skin contact. Avoid breathing vapors.
M-Bond Curing Agent 600/610	Flammable. May cause respiratory sensitization. May cause upper respiratory tract irritation. Eye; may cause pain, severe irritation with corneal injury which may result in permanent	Avoid heat, sources of ignition. Avoid oxidizing agents, acids, bases, alcohol, water.	Store below 80F in a dry, well ventilated, flammable liquid area. Keep containers tightly sealed. Avoid prolonged exposure to vapors and skin contact.



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<i>Full Chemical Name</i>	<i>Hazards</i>	<i>Incompatibilities</i>	<i>Handling Instructions</i>
	impairment of vision, even blindness. Skin; short single exposure may cause severe skin burns, allergic skin reactions. A single prolonged exposure may result in the material being absorbed in harmful amounts.		
Chromic Acid	Corrosive. Strong oxidizer. Causes burns by all exposure routes. May cause allergic respiratory and skin reactions. Causes cancer. May cause heritable genetic damage. May impair fertility.	Metals, reducing agents, bases, acetic acid, acetic anhydride, alcohols, ammonia, halogens, phosphorus, diethyl ether, dimethyl formamide, acetone, diethyl formamide, methanol, glycerol, ethanol, camphor, pyridine, hydrocarbons, ketones, turpentine, combustible materials, sulfides.	Wash thoroughly after handling. Remove contaminated clothing and wash before reuse. Discard contaminated shoes. Use only in a well-ventilated area. Do not get in eyes, on skin, or on clothing. Do not ingest or inhale. Use with adequate ventilation. Do not store near combustible materials. Keep container tightly closed. Store in a cool, well-ventilated location.



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LIST OF COMMON CHEMICALS - CONTINUED

<i>Full Chemical Name</i>	<i>Hazards</i>	<i>Incompatibilities</i>	<i>Handling Instructions</i>
Methyl alcohol	Poison! Danger! Vapor harmful. May be fatal or cause blindness if swallowed. Harmful if inhaled or absorbed through skin. Cannot be made nonpoisonous. Flammable liquid and vapor. Causes irritation to skin, eyes and respiratory tract. Affects central nervous system and liver.	Strong oxidizing agents such as nitrates, perchlorates or sulfuric acid. Will attack some forms of plastics, rubber, and coatings. May react with metallic aluminum and generate hydrogen gas. Avoid heat, flames, ignition sources.	Protect against physical damage. Store in a cool, dry well-ventilated location, away from any area where the fire hazard may be acute. Outside or detached storage is preferred. Separate from incompatibles. Containers should be bonded and grounded for transfers to avoid static sparks. Storage and use areas should be No Smoking areas. Use non-sparking type tools and equipment, including explosion proof ventilation. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product. Do Not attempt to clean empty containers since residue is difficult to remove.
Methylene Chloride	Harmful if swallowed, inhaled or absorbed through skin. Affects central nervous system, liver, cardiovascular system, and blood. Causes irritation to skin, eyes, and respiratory tract. Suspect cancer hazard. May cause cancer.	Strong oxidizers, strong caustics, plastics, rubber, nitric acid, water + heat, and chemically active metals, such as aluminum and magnesium powder, sodium, potassium, and lithium. Avoid contact with open flames and electrical arcs. Liquid methylene chloride will attack some forms of plastics, rubber, and coatings.	Keep in a tightly closed container, stored in a cool, dry, ventilated area. Protect against physical damage. Isolate from any source of heat or ignition. Outside or detached storage is recommended. Containers of this material may be hazardous when empty since they retain product residues



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<i>Full Chemical Name</i>	<i>Hazards</i>	<i>Incompatibilities</i>	<i>Handling Instructions</i>
			(vapors, liquids); observe all warnings and precautions listed for this product. To minimize decomposition, all storage containers should be galvanized or lined with phenolic coating. This material may corrode plastic or rubber.
Micro-90	Eye, skin, inhalation irritation with prolonged exposure.	May etch aluminum and zinc. Do not mix with other cleaners. Mixing with chlorine-based cleaners may produce toxic gases.	Avoid contact with eyes, skin and clothing. Use in a well-ventilated area. Store in a cool place in original container and protect from sunlight. Keep container closed when not in use. Use only stainless steel, polyethylene or plastic-lined containers for handling. Do not store in contact with aluminum, zinc, copper or their alloys.
Nitrogen, Compressed Gas	Difficulty breathing	Metals, oxidizing materials	Provide ventilation
Nitrogen, Cryogenic Liquid	Difficulty breathing, frostbite	Metals, oxidizing materials	Provide ventilation. Wear cold insulating clothing and insulated gloves. Wear splash resistant safety goggles. Provide an emergency eye wash fountain and quick drench shower in the immediate area.
OCTOIL	May cause mild skin irritation. Signs and symptoms of exposure to this material through breathing, and/or passage of the material through the skin may include stomach	Avoid contact with strong oxidizing agents.	Containers of this material may be hazardous when emptied. Since emptied containers retain product residues (vapor, liquid, and/or solid).



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<i>Full Chemical Name</i>	<i>Hazards</i>	<i>Incompatibilities</i>	<i>Handling Instructions</i>
	or intestinal upset (nausea, vomiting, diarrhea) irritation (nose, throat, airways).		
Sulfur Hexafluoride	Difficulty breathing	Combustible materials, metals, oxidizing materials	Provide ventilation, wear appropriate chemical resistant gloves. Store below 49C.
Trichlorotrifluoroethane	May cause eye irritation. Vapor reduces oxygen available for breathing. May cause dermatitis. May cause central nervous system effects. May cause cardiac disturbances. This is a CFC substance which destroys ozone in the upper atmosphere.	Incompatible with alkali or alkaline earth metals such as sodium, potassium, aluminum, barium, lithium, samarium, sodium-potassium alloy, titanium, zinc, magnesium, and beryllium.	Wash thoroughly after handling. Remove contaminated clothing and wash before reuse. Use with adequate ventilation. Avoid contact with skin and eyes. Avoid ingestion and inhalation. Do not vent to atmosphere. To comply with provisions of the U.S. Clean Air Act, any residual must be recovered. Keep from contact with oxidizing materials. Store in a cool, dry, well-ventilated area away from incompatible substances. Keep away from metals. Keep containers tightly closed. Do not store in aluminum containers
Toluene	Poison! Danger! Harmful or fatal if swallowed. Harmful if inhaled or absorbed through skin. Vapor harmful. Flammable liquid and vapor. May affect liver, kidneys, blood system, or central nervous system.	Heat, flame, strong oxidizers, nitric and sulfuric acids, chlorine, nitrogen tetroxide; will attack some forms of plastics, rubber, coatings.	Protect against physical damage. Store in a cool, dry well-ventilated location, away from any area where the fire hazard may be acute. Outside or detached storage is preferred. Separate from incompatibilities. Containers should be bonded and grounded



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<i>Full Chemical Name</i>	<i>Hazards</i>	<i>Incompatibilities</i>	<i>Handling Instructions</i>
	Causes irritation to skin, eyes and respiratory tract.		for transfers to avoid sparks. Storage and use areas should be No Smoking areas. Use non-sparking type tools and equipment, including explosion proof ventilation. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquids); observe all warnings and precautions listed for this product.
VWR-19 TKO W/7 Ultra mechanical pump oil	Exposure to oil mists may cause nausea and eye irritation.	Strong oxidizers. Avoid prolonged temperatures > 200C.	None listed
Xylenes	Harmful or fatal if swallowed. Vapor harmful. Affects central nervous system. Causes severe eye irritation. Causes irritation to skin and respiratory tract. May be harmful if absorbed through skin. Chronic exposure can cause adverse liver, kidney, and blood effects. Flammable liquid and vapor.	Strong oxidizing agents and strong acids.	Protect against physical damage. Store in a cool, dry well-ventilated location, away from any area where the fire hazard may be acute. Outside or detached storage is preferred. Separate from incompatibles. Containers should be bonded and grounded for transfers to avoid static sparks. Storage and use areas should be No Smoking areas. Use non-sparking type tools and equipment, including explosion proof ventilation. Containers of this material may be hazardous when empty since they retain product



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<i>Full Chemical Name</i>	<i>Hazards</i>	<i>Incompatibilities</i>	<i>Handling Instructions</i>
			residues (vapors, liquid); observe all warnings and precautions listed for the product. Do Not attempt to clean empty containers since residue is difficult to remove.
TB-CIDE Quat	Causes serious eye irritation.	Strong oxidizing agents. Strong acids.	Keep containers tightly closed in a dry, cool and well ventilated space. Wear splash goggles. Wear rubber or other chemical resistant gloves. Wash hands and any exposed skin thoroughly after handling.
Oxygen, Compressed gas	Short term exposure: irritation, chest pain, cough, changes in body temperature, nausea, difficulty breathing, irregular heartbeat, dizziness, disorientation, hallucinations, mood swings, pain in extremities.	Combustible materials, halo carbons, metals, bases, reducing agents, amines, metal salts, oxidizing materials.	Protect from physical damage. Avoid heat, flames, sparks and other sources of ignition. Store in a clean, cool, dry place. Store in a well ventilated area. Store below 125 F.

<i>Full Chemical Name</i>	<i>Hazards</i>	<i>Incompatibilities</i>	<i>Handling Instructions</i>
Gallium Metal	Causes skin irritation. May cause gastrointestinal irritation with nausea, vomiting, and diarrhea. May be harmful if swallowed. Causes	Hydrogen peroxides, hydrochloric acid, halogens, phosphorus, sulfur, alkalies, oxygen, metals, oxidizing agents.	Wash thoroughly after handling. Avoid contact with eyes, skin, and clothing. Avoid ingestion and inhalation. Use with adequate ventilation. Wash clothing before reuse. Keep from contact with moist air



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<i>Full Chemical Name</i>	<i>Hazards</i>	<i>Incompatibilities</i>	<i>Handling Instructions</i>
	respiratory tract irritation. May be harmful if inhaled.		and steam. Store in a tightly closed container. Store in a cool, dry, well-ventilated area away from incompatible substances. Store protected from moisture.
Magnesium Sulphate	May cause eye irritation. May cause skin irritation. May be harmful if swallowed. May be harmful if inhaled. No known OSHA hazards.	Strong oxidizing agents.	Provide appropriate exhaust ventilation at places where dust is formed. Keep container tightly closed in a dry and well ventilated place. Use good personal hygiene. Wash thoroughly with soap and water after handling.
Platinum Deposition Trimethyl [(1,2,3,4,5-ETA.)-1 Methyl 2, 4-Cyclopentadien-1-YL] Platinum	May cause eye irritation. May cause mild to moderate skin irritation. If inhaled may cause coughing, shortness of breath, wheezing.	Mixing with oxidizing agents and air.	Avoid contact with eyes, prolonged or repeated contact with skin. Use compatible chemical resistant gloves. Wash hands thoroughly after handling. Store in a dry place. Do not store directly on ground.
Ethyl Alcohol 190 Proof	Hazardous in case of skin (irritant), of eye contact (irritant). Slightly hazardous in case of skin contact (permeator), of ingestion. Non-corrosive for skin, eyes, and lungs.	Reactive with oxidizing agents, acids, alkalais.	Keep away from heat and sources of ignition. Keep container in a cool, well ventilated area. Keep locked up. Do not store above 23C (73.4 F)
70% Isopropyl Alcohol in Water	Highly flammable in liquid and vapor. Causes serious eye irritation. May cause drowsiness or dizziness.	Incompatible materials, strong oxidizing agents, strong acids, metals.	Ensure adequate ventilation. Wear appropriate protective eyeglasses or chemical safety goggles. Wear appropriate protective gloves and clothing. Handle in accordance with good industrial hygiene and safety practice.



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SOP Part 3, Equipment - Provide information relevant to any piece of equipment or instrument that requires special safety considerations to operate. This may include, but not be limited to, instructions for conducting high pressure reactions, using compressed gas cylinder manifolds, distillations, calibration of safety equipment, work with high voltage equipment, power tools, etc.

Assistance and resources for determining which pieces of equipment may require special instruction within the SOP may be obtained by contacting the University Chemical Hygiene Officer at 863-1737.

SOP PART 3 – EQUIPMENT

X-ray detector on the LEO 1530 VP SEM requires liquid nitrogen for cooling.

Cold fingers on the Philips CM20 TEM and JEOL 2100F STEM require liquid nitrogen for cooling.

The CM20 TEM and JEOL 2100F STEM use Sulfur Hexafluoride gas as a high voltage insulator in the high voltage power supplies and the electron emitters. Sulfur Hexafluoride compressed gas cylinders are stored in 020B.3

All electron microscopes in this facility require compressed gas cylinders for venting to atmosphere and compressed air to actuate valves.

All electron microscopes in this facility can be operated at accelerating voltages high enough to produce x-rays. The equipment is designed so that there is no detectable radiation (x-rays) above background external to the equipment.

Training sessions are required on all electron microscopes covering special safety considerations.

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SOP Part 4, Carcinogens and Reproductive Toxins - List all Select Carcinogens and reproductive Toxins used within the laboratory and identify a designated area for use (i.e., such as one particular chemical exhaust hood) and specific control methods to be used to limit exposure.

Definition of Select Carcinogen - Select Carcinogen means any substance which regulated by OSHA as a carcinogen; or it is listed under the category, "known to be carcinogens," in the Annual Report on Carcinogens published by the National Toxicology Program (NTP - latest edition); or it is listed under Group 1 ("carcinogenic to humans") by the International Agency for research on Cancer Monographs (IARC)(latest editions); or it is listed in either Group 2A or 2B by IARC or under the category, "reasonably anticipated to be carcinogens" by NTP, and causes statistically significant tumor incidence in experimental animals in accordance with any of the following criteria:

- ▶ *After inhalation exposure of 6-7 hours per day, 5 days per week, for a significant portion of a lifetime to dosages of less than 10mg/m(3);*
- ▶ *After repeated skin application of less than 300 (mg/kg of body weight) per week; or*
- ▶ *After oral dosages of less than 50 mg/kg of body weight per day.*

Definition of a Reproductive Toxin – Reproductive Toxin means chemicals which affect the reproductive chemicals which affect the reproductive capabilities including chromosomal damage (mutations) and effects on fetuses (teratogenesis).

SOP PART 4 – CARCINOGENS AND REPRODUCTIVE TOXINS

<i>Full Chemical Name</i>	<i>Hazards</i>	<i>Designated Area</i>	<i>Control Measures</i>
Chromic Acid	Corrosive. Strong oxidizer. Causes burns by all exposure routes. May cause allergic respiratory and skin reactions. Causes cancer. May cause heritable genetic damage. May impair fertility.	ERC 020 fume hood for sample preparation. ERC 020B for storage.	See SOP Part 8. Chromic Acid SOP.



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SOP Part 5, Spill Equipment - List or the location of spill equipment available to your laboratory personnel for small incidental spills.

SOP PART 5 – SPILL EQUIPMENT

Spill equipment is located in room 020 near the entrance to the laboratory.

SOP Part 6, Emergency/safety Equipment - List the location of emergency eyewash stations, drench showers, fire alarm pull stations, fire extinguishers, first aid kits and other safety related devices available to the laboratory staff.

SOP PART 6 – EMERGENCY/SAFETY EQUIPMENT

Location of nearest Eyewash Station: Room 020 near the entrance to the lab.

Location of nearest Emergency Drench Shower: Room 020 near the entrance to the lab.

Location of nearest Fire Alarm Pull Station: Hallway (exiting to the left) near the entrance/exit on the ground floor at 345 Brook St. or upstairs at the exit of the Hazeltine Commons.

Location of nearest Fire Extinguisher: Room 020 near entrance to the lab or in the service chases (020B and 020F).

Location of nearest First Aid Kit: Room 020 near entrance to the lab.

Sink, hand soap, and paper towel dispenser are located in ERC 020 to the right of the entrance.



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SOP Part 7, Housekeeping - Provide information concerning the housekeeping rules specific to this laboratory including, but not limited to, methods for laundering laboratory coats.

SOP PART 7 – HOUSEKEEPING

Facility users are required to take all samples away with them when they have completed their session or store samples in well labeled containers.

Small quantities of solvents are kept in squeeze bottles in the fume hood. Larger quantities (1liter and 4 liter) containers of solvents are kept in the designated solvents cabinet.

In the FIB replenishing Gallium metal for the ion source or precursor materials for Carbon selective etch or Platinum metal deposition must be done in a room with a fume hood and not in this lab.

From Chromic Acid SOP

1. Spills, Cleanup, and Disposal

Spill response should follow the Brown University Chemical Hygiene Plan. Due to the toxic nature of chromic acid, all spills outside of a fume hood should be treated as major spills, and user should evacuate the area and call the Department of Public Safety at 401-863-4111.

2. Aluminum oxide etchant procedure

This section describes the workflow when performing the aluminum oxide etching process which makes use of chromic acid.

1. Setup
 - a. Wearing appropriate PPE, make space in the fume hood for the hotplate, double boiler and glassware, and glassware for rinsing.
 - b.
2. Prepare Chemicals
 - a. If pre-mixed etchant is used, it can be carefully poured into a suitably sized dedicated beaker.
 - b. If etchant needs to be prepared, calculate the mass required of water, phosphoric acid, and chromic acid constituents.
 - c. Using a lab balance, first weigh out the required water into a beaker. Next, add the requisite phosphoric acid, followed by the chromic acid. Rinse any disposable pipettes, storing waste in a waste bottle and dispose of the pipette in a suitable solid waste container.
 - d. Bring double boiler up to temperature slowly.
3. Etch Samples
 - a. Hang samples through rack holes with aluminum hanging wire, supported from laboratory stands
 - b. Lower the lab stand until samples are submerged. Start
 - c. sample.
 - d. Final rinse of samples with small volume of acetone and isopropyl alcohol.



- e. Air dry in fume hood before storing samples.
4. Clean up
- a. Store rinsewater in a properly marked waste bottle.
 - b. Store etchant back in its container. Use a funnel to avoid spills.
 - c. Rinse funnel and all contaminated glassware, storing initial rinse water in appropriate waste container.
 - d. Store clean, dry, glassware in sealed container.



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SOP Part 8, Additional Information - Provide additional safety information relevant to the work being conducted in this laboratory that is not already covered in the Brown University Chemical Hygiene Plan or in Parts 1-7 of this SOP. Include a complete list of chemicals used in this laboratory as an attachment (if not already provided).

SOP PART 8 – ADDITIONAL INFORMATION

The door to the microscope facility (ERC 020) and all the doors to the individual microscopes are accessed by Brown card. Access is granted by submission and acceptance of a user agreement to the IMNI office.

Chromic Acid Standard Operating Procedure

1. Purpose

This standard operating procedure (SOP) is intended to provide guidance on how to safely store, handle, use, and dispose of Chromic acid (H_2CrO_4). This SOP does not supplant the material safety data sheet (SDS), and laboratory users should read and consult the SDS before engaging in work with chromic acid.

2. Scope

This SOP was developed by Glint Photonics to describe safe operating procedures when working with a chromic acid containing etch as an external user in the Brown University Electron Microscopy Facility (EMF).

3. Hazard Description

Chromic acid is corrosive to human tissue and metal, as well as acutely toxic, carcinogenic, mutagenic, and teratogenic. Chromic acid and dichromate salts are poisonous and potentially fatal when ingested, inhaled, or absorbed through the skin. They also cause severe skin, eye, and mucous membrane damage including skin ulcers.

4. Process and Hazard Controls

a. Elimination/substitution

- No known alternative to the proposed wet etch process exists for the required purposes.

b. Engineering Controls

- All work with chromic acid must be performed within a laboratory fume hood in good working order, and according to the safe working practices of that hood. This includes keeping the sash as low as possible and ensuring that equipment and chemicals are at least 6 inches inside the lip of the hood.

c. Work Practices

- When diluting, acid should always be slowly added to cool water, and in small amounts. A small amount would be approximately <20% the volume of the diluting water. Chromic acid is a strong oxidizer, and adding to hot water or adding water into the acid can cause uncontrolled boiling and splashing.



d. Personal Protective Equipment

- **Body and arm protection:** A traditional white lab coat should be worn.
- **Hand Protection:** Nitrile gloves should be worn at all times. Additional heavy duty chemical gloves with wrist protection should be worn when pouring or manipulating chemicals that may pose a splash hazard.
- **Face and Eye protection:** Safety goggles are required at all times, even while working in a fume hood with the sash lowered.

e. Transportation and Storage

- Store in a cool, dry place.
- Keep away from reducing agents.
- Store on low shelves and in acid cabinet.
- Segregate from organic acids as well as flammable or combustible materials.
- Use bottle carrier for transporting bottles

5. Spills, Cleanup, and Disposal

Spill response should follow the Brown University Chemical Hygiene Plan. Due to the toxic nature of chromic acid, all spills outside of a fume hood should be treated as major spills, and user should evacuate the area and call the Department of Public Safety at 401-863-4111.

6. Aluminum oxide etchant procedure

This section describes the workflow when performing the aluminum oxide etching process which makes use of chromic acid.

5. Setup

- Wearing appropriate PPE, make space in the fume hood for the hotplate, double boiler and glassware, and glassware for rinsing.
-

6. Prepare Chemicals

- If pre-mixed etchant is used, it can be carefully poured into a suitably sized dedicated beaker.
- If etchant needs to be prepared, calculate the mass required of water, phosphoric acid, and chromic acid constituents.
- Using a lab balance, first weigh out the required water into a beaker. Next, add the requisite phosphoric acid, followed by the chromic acid. Rinse any disposable pipettes, storing waste in a waste bottle and dispose of the pipette in a suitable solid waste container.
- Bring double boiler up to temperature slowly.

7. Etch Samples

- Hang samples through rack holes with aluminum hanging wire, supported from laboratory stands
- Lower the lab stand until samples are submerged. Start timer.

8. Remove Samples

- After determined time, raise lab stands to remove samples from the etchant.
- Use two 100 mL beakers of rinse water for a serial rinse via submersion of each sample.
- Final rinse of samples with small volume of acetone and isopropyl alcohol.
- Air dry in fume hood before storing samples.

9. Clean up

- Store rinsewater in a properly marked waste bottle.
- Store etchant back in its container. Use a funnel to avoid spills.
- Rinse funnel and all contaminated glassware, storing initial rinse water in appropriate waste container.
- Store clean, dry, glassware in sealed container.



INFORMATION AND INSTRUCTION

Signatures - All persons working (paid or unpaid) in the laboratory must sign the cover page of Section 3. By signing Section 3 the individual is acknowledging that he or she has read and understood the entire Brown University Chemical Hygiene Plan and Standard Operating Procedures.

Assistance in Developing the Standard Operating Procedures – The University Chemical Hygiene Officer 863-1737 is available to assist in the development of SOP's. In addition, the Brown University Office of Environmental Health & Safety offers several resources to aid in the development of the laboratory specific SOP at the following URL:

<http://www.brown.edu/Administration/EHS/lab>

Renewal Instructions - Laboratory specific SOP's are required to be reviewed and if necessary, revised annually. If procedural changes have not occurred or if new chemicals have not been introduced to the research the renewal process may be as simple as completing a new cover page along with the required signatures, and resubmitting the entire package to the EHS office to the attention of the University Chemical Hygiene Officer, Box 1914. Notifications for annual SOP renewal will be sent automatically to each Laboratory Supervisor by the Office of Environmental Health & Safety.

Distribution and Availability – All laboratory employees shall have available the contents of this manual, the laboratory specific SOP, a copy of the Laboratory Safety Standard (29 CFR 1910,1450), and all appendices.



STANDARD OPERATING PROCEDURES
DEVELOPED TO SUPPLEMENT THE BROWN UNIVERSITY CHEMICAL HYGIENE PLAN &
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SOP Part 1, Brief Description of Research – Provide a brief description of the type of research being conducted in the laboratory covered by this plan.

SOP PART 1 – BRIEF DESCRIPTION OF RESEARCH

The Thermo/Electron K-Alpha equipment is in this room. This lab is part of the Electron Microscope Facility and is used to investigate nontoxic solids (metal alloys, semiconductors, ceramics, and polymers) by x-ray photoelectron spectroscopy (XPS). The K-Alpha generates 30 keV electrons to make Al K monochromated x-rays (1.4866 KeV) to produce low energy electrons (XPS). The K-Alpha is designed so that there is no detectable radiation external to the equipment above background.

In addition there is an Olympus optical microscope (Model AH-3) with scanning, printing, and video recording capability in this room.



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SOP Part 2, List of Common Chemicals – Provide a list of the most commonly used chemicals in the laboratory along with hazard information, compatibility, and special handling instructions for each chemical listed.

Assistance and resources for determining hazard information, and compatibility, may be obtained by contacting the University Chemical Hygiene Officer at 863-1737 or by browsing the resources available at the Brown University EHS website:

<http://www.brown.edu/Administration/EHS/lab>

SOP PART 2 - LIST OF COMMON CHEMICALS

<i>Full Chemical Name</i>	<i>Hazards</i>	<i>Incompatibilities</i>	<i>Handling Instructions</i>
Nitrogen, compressed gas	Difficulty breathing	Metals, oxidizing materials	Provide ventilation
Argon, Compressed	Difficulty breathing	No data available	Provide ventilation and wear appropriate chemical resistant gloves.
Dust Off 1,1-Difluoroethane	Inhalation or high concentrations of vapor is harmful and may cause heart irregularities, unconsciousness, or death	Alkali or alkaline earth metal – powdered Al, Zn, Be, etc.	Avoid high temperatures and open flames.
Jun-Air Compressor Oil	Aspiration hazard if swallowed. Do not taste or swallow. Spills may create a slipping hazard.	Avoid contact with oxidizing agents.	Keep containers closed and do not handle or store near heat sparks or other potential ignition sources.



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<i>Full Chemical Name</i>	<i>Hazards</i>	<i>Incompatibilities</i>	<i>Handling Instructions</i>



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SOP Part 3, Equipment - Provide information relevant to any piece of equipment or instrument that requires special safety considerations to operate. This may include, but not be limited to, instructions for conducting high pressure reactions, using compressed gas cylinder manifolds, distillations, calibration of safety equipment, work with high voltage equipment, power tools, etc.

Assistance and resources for determining which pieces of equipment may require special instruction within the SOP may be obtained by contacting the University Chemical Hygiene Officer at 863-1737.

SOP PART 3 – EQUIPMENT

The K-Alpha uses compressed Argon for the sputter cleaning source.

The K-Alpha uses compressed Nitrogen to vent parts of the vacuum system to exchange samples.

The K-Alpha has an associated mechanical air compressor and water chiller.

The K-Alpha can be operated at electron accelerating voltages high enough to produce x-rays.

Training sessions are required on all equipment covering special safety considerations.



SOP Part 4, Carcinogens and Reproductive Toxins - List all Select Carcinogens and Reproductive Toxins used within the laboratory and identify a designated area for use (i.e., such as one particular chemical exhaust hood) and specific control methods to be used to limit exposure.

Definition of Select Carcinogen - Select Carcinogen means any substance which regulated by OSHA as a carcinogen; or it is listed under the category, "known to be carcinogens," in the Annual Report on Carcinogens published by the National Toxicology Program (NTP - latest edition); or it is listed under Group 1 ("carcinogenic to humans") by the International Agency for research on Cancer Monographs (IARC)(latest editions); or it is listed in either Group 2A or 2B by IARC or under the category, "reasonably anticipated to be carcinogens" by NTP, and causes statistically significant tumor incidence in experimental animals in accordance with any of the following criteria:

- *After inhalation exposure of 6-7 hours per day, 5 days per week, for a significant portion of a lifetime to dosages of less than 10mg/m³;*
- *After repeated skin application of less than 300 (mg/kg of body weight) per week; or*
- *After oral dosages of less than 50 mg/kg of body weight per day.*

Definition of a Reproductive Toxin – Reproductive Toxin means chemicals which affect the reproductive capabilities including chromosomal damage (mutations) and effects on fetuses (teratogenesis).

SOP PART 4 – CARCINOGENS AND REPRODUCTIVE TOXINS

<i>Full Chemical Name</i>	<i>Hazards</i>	<i>Designated Area</i>	<i>Control Measures</i>



SOP Part 5, Spill Equipment - List or the location of spill equipment available to your laboratory personnel for small incidental spills.

SOP PART 5 – SPILL EQUIPMENT

There should not be any chemicals used in this lab that would require spill equipment.

The oil filled mechanical pump is in a spill containment tray.

SOP Part 6, Emergency/safety Equipment - List the location of emergency eyewash stations, drench showers, fire alarm pull stations, fire extinguishers, first aid kits and other safety related devices available to the laboratory staff.

SOP PART 6 – EMERGENCY/SAFETY EQUIPMENT

Location of nearest Eyewash Station: Hallway (exiting to the left) approximately 50 feet from the entrance to the lab.

Location of nearest Emergency Drench Shower: Hallway (exiting to the left) approximately 50 feet from the entrance to the lab.

Location of nearest Fire Alarm Pull Station: Hallway (exiting to the right) near stair and elevator.

Location of nearest Fire Extinguisher: **Immediately to the left upon entering the door to the lab.**

Location of nearest First Aid Kit: To the right inside the door on the coat rack.

Location of sink for washing hands is in the restroom (exiting to the left) at the end of the hall.

Other Emergency/Safety Equipment Locations:



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SOP Part 7, Housekeeping - Provide information concerning the housekeeping rules specific to this laboratory including, but not limited to, methods for laundering laboratory coats.

SOP PART 7 – HOUSEKEEPING

Users of this equipment are requested to take their samples with them when they leave this area or store samples in well labeled containers.

Other equipment is temporarily stored in BH009.



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SOP Part 8, Additional Information - Provide additional safety information relevant to the work being conducted in this laboratory that is not already covered in the Brown University Chemical Hygiene Plan or in Parts 1-7 of this SOP. Include a complete list of chemicals used in this laboratory as an attachment (if not already provided).

SOP PART 8 – ADDITIONAL INFORMATION

Access to BH 009 is controlled by Brown card. Access is granted by submission and acceptance of a user agreement to the IMNI office.



INFORMATION AND INSTRUCTION

Signatures - All persons working (paid or unpaid) in the laboratory must sign the cover page of Section 3. By signing Section 3 the individual is acknowledging that he or she has read and understood the entire Brown University Chemical Hygiene Plan and Standard Operating Procedures.

Assistance in Developing the Standard Operating Procedures – The University Chemical Hygiene Officer 863-1737 is available to assist in the development of SOP's. In addition, the Brown University Office of Environmental Health & Safety offers several resources to aid in the development of the laboratory specific SOP at the following URL:

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