Chemical Hygiene Plan

Section I – Optical Characterization Facility Chemical Hygiene Plan

1. **General provisions**

<u>No chemical work is allowed in the optical characterization facility (OCF)</u>

The nature of OCF operation assumes that self-contained, non-hazardous samples are tested by users and/or OCF staff. Handling of such samples is covered by this generic chemical hygiene plan (CHP) on file at OCF. Users are responsible for development of safe operating procedures (SOPs) for samples containing chemicals listed in the UCLA settlement Exhibit 1 and OSHA particularly hazardous substances list. User-developed SOPs must be filed at OCF and can be reused. OCF may have canned SOPs for common hazardous chemicals used in the preparation of samples, such as solvents. If chemical hazards are present during the optical characterization work, appropriate PPE must be worn and adequate engineering safety controls (fume removal hoses, spill containers, etc.) must be established in addition to the experiment-specific laser safety measures.

2. **Safety training requirements**

All OCF users must have documented general laboratory safety training and familiarize themselves with the OCF CHP in its entirety (Sections I, II, and III). Users from Chemistry and Biochemistry department must have documented safety training compliant with terms of UCLA settlement agreement. Current safety training requirements are available from Chemistry Safety Officer Dr. Alex Moretto (amoretto@chem.ucsb.edu) Non-Chemistry department UCSB users must have completed an equivalent general lab safety class. Out of campus users which cannot provide proof of the safety training are not allowed to conduct any work in the lab. However, they can submit samples for characterization by OCF staff given that chemical safety documentation requirements for them are satisfied. Also, out of campus users may need to provide a proof of liability insurance coverage if they intend to work at OCF labs. Please contact OCF manager Dr. Alexander Mikhailovsky regarding the liability insurance requirements.

OCF users operating ANSI safety class IIIa and higher lasers must complete laser safety training program. This includes laser safety class offered through ES&H web-site, reading USCB laser safety manual and OCF-specific addendum, and safety orientation by OCF staff. Please contact Dr. Alexander Mikhailovsky (mikhailovsky@chem.ucsb.edu, x2327) for the current training requirements. No laser safety training is required if the laser is integrated into a research instrument and its beam cannot be accessed readily by the user.

3. Dress code and PPE requirements

Use of chemical personal protection equipment (PPE) is not required in OCF when the work is conducted with optical instruments and lasers. Laser PPE use is regulated by the laser safety guidelines outlined in UCSB laser safety manual and lab-specific laser safety addendum. Chemical PPE is required when handling samples and/or reagents required for experiments or OCF operations and must be chosen according to the hazard level and nature of the work.

Wear clothes that cover the maximum amount of skin. Long-sleeved shirts and long pants are recommended. Open toe shoes are not allowed in the lab. Sunglasses should not be worn in the lab.

4. Handling samples for optical characterization experiments

- Samples for characterization at OCF should be contained or sealed to prevent contamination of the environment and instrumentation and eliminate hazards related to the exposure of researchers to compounds studied. This includes but not limited to the use of sealed cells, encapsulation into chemically inert matrix, or placement into cryostats/vacuum vessels or other closed containers.
- Users are responsible for development of safe operating procedures (SOPs) for samples containing chemicals listed in the UCLA settlement Exhibit 1 and OSHA particularly hazardous substances list. User-developed SOPs must be filed at OCF and can be reused.
- If chemical hazards are present during the optical characterization work, appropriate PPE must be worn and adequate engineering safety controls (fume removal hoses, spill containers, etc.) must be established in addition to the experiment-specific laser safety measures.
- If a sample requires adjustments involving chemical manipulations (e.g., dilution), adequate chemical safety measures must be in effect. These include use of PPE and engineering controls. For non-hazardous and non-vapor emitting chemicals, the work can be done at the OCF lab given that users provide spill prevention equipment and dispose of the chemical waste generated. All other work must be performed at a properly equipped chemical laboratory.

- Proper chemical PPE should be used for handling samples. It should be chosen according to hazards present in the sample. Also, the user should consider the possibility of the sample breakage/spill and select PPE accordingly. If certain PPE items are not available at OCF, the user must provide its own safety equipment.
    - Protective glasses are recommended for handling all samples
    - Gloves and lab coats must be used when there is a possibility of sample leakage and contamination of skin and clothes. The glove must be compatible with the compound studied. To choose the correct glove, go to a Glove Reference Chart. (http://www.ehs.ucsb.edu/units/labsfty/labrsc/lsglove.htm)
    - Appropriate PPE, such as gloves, lab coat, protective glasses or face shield must be used when handling cryogenically cooled samples
    - Laser safety glasses can be used as protective glasses if protection against chemical and laser hazards is required simultaneously, except situations when samples can generate high speed projectile debris for the laser glasses are not rated for such hazards. In this case, laser hazards should be eliminated completely prior the sample handling manipulations by blocking the laser beam completely at the source. Similarly, no manipulations with the sample are allowed during the optical experiment involving samples representing such hazard and appropriate engineering and administrative controls must be established to eliminate chemical hazards to the users.
- Samples must be disposed properly by users and OCF personnel following waste disposal guidelines described in CHP Section II.
- Chemical spills must be handled according by users and OCF personnel according to guidelines described in this CHP Sections I and II.

5. Chemical Spill Procedures

In case of a chemical spill, always remember that your safety and the safety of those around you are most important. Please take steps to insure that everyone is safe before attempting a cleanup. (See Section II) Contacts: EH&S: x3194 or for a serious emergency dial 9-911 from a campus phone.Below is a list of recommended spill control supplies:

Personal Protective Equipment

- 2 pairs chemical splash goggles
- 2 pairs of gloves (recommend Silver Shield or 4H)
- 2 pairs of shoe covers
- 2 plastic or Tyvek aprons and/or Tyvek suits

Absorption Materials

- 4 3M POWERSORB spill pillows (or equivalent)

- 1 3M POWERSORB spill sock
- 2 DOT pails (5 gallon) with polyethylene liners
    - 1 filled with loose absorbent, such as vermiculite or clay
    - 1 with minimum amount of loose absorbent in the bottom

Neutralizing Materials

- Acid Neutralizer
- Caustic Neutralizer
    - commercial neutralizers, such as Neutrasorb (for acids) and Neutracit-2 (for bases) have built in color change to indicate complete neutralization
- Solvent Neutralizer
    - commercial solvent neutralizers, such as Solusorb, act to reduce vapors and raise the flashpoint of the mixture

Mercury Spills

- Small mercury vacuum to pick up large drops (optional)
- Hg Absorb Sponges - amalgamate mercury residue
- Hg Absorb Powder - amalgamates mercury
- Hg Vapor Absorbent - reduces concentration of vapor in hard to reach areas
- Mercury Indicator - powder identifies presence of mercury

Clean-up Tools

- Polypropylene scoop or dust pan
- Broom or brush with polypropylene bristles
- 2 polypropylene bags
- Sealing tape
- pH test papers
- Waste stickers
- Floor sign - DANGER Chemical Spill - Keep Away

UCSB EH&S:
http://ehs.ucsb.edu/units/labsfty/labrsc/emergency/lsemergchemspill.htm

Princeton chemical spill procedures: http://web.princeton.edu/sites/ehs/emergency/spills.htm

University of Deleware chemical spill clean-up procedures (very detailed):
http://www.udel.edu/ehs/chemspillkit/chemspillguide.html

6. Procedures for utilities outage

- All OCF equipment will restart in stand-by mode after power outage of any duration, i.e., laser output will be disabled, high voltage will be removed from the detectors, and vacuum pumps will stop until restarted by the operator.
- If electrical outage takes a long time, turn interlock keys on lasers to OFF position. Put power switches on other equipment to OFF position. Close equipment cooling water supply/return valves unless the equipment manual states otherwise.
- If equipment cooling water supply is interrupted when Ar-ion laser is running and is not restored immediately, turn off valves for water supply and return to/from the laser and do not use the laser for at least 2 hours for cold water entering the hot plasma tube could lead to cracking of the tube.
- If equipment cooling water supply is interrupted when thermoelectrical detector cooler is running and is not restored immediately, turn off the water supply and return valves to/from the detector housing and deenergize the cooler power supply. Operation of the cooler can be resumed immediately when the equipment cooling water supply is restored.
- If Alcatel turbo pump station is deenergized while running it will vent with the ambient air and needs to be restarted when the power returns.

7. Use of compressed gas cylinders

Compressed gas cylinders must be handled carefully by trained individuals. The diffusive nature of gas can result in serious hazards over large areas. Gas cylinders can be hazardous because 1) if they are mishandled, they can become "unguided missiles" with enough explosive force to go through concrete walls due to the high pressure inside the tank. 2) they often contain materials which are inherently toxic or highly flammable. For these reasons, particular care must be exercised with compressed gases.

Use of Toxic Gases on Campus

Per *the CA Fire Code*, if the volume of toxic gas within a given building exceeds a particular amount, then expensive and elaborate gas monitoring and alarm systems are required. Therefore, EH&S works with departments and labs to reduce the volumes of these hazardous materials whenever possible.

Below is a list of common toxic gases that researchers could use. If you plan to use any of these, or other toxic gases, inform the EH&S Lab Safety Specialist at x4899 or x8243 before you proceed. In most cases the requirements of the Fire Code can be avoided by going to smaller gas cylinders and/or gas mixtures with inert gases.

- Arsine

- Boron Trichloride
- Boron Trifluoride
- Carbonyl Disulfide
- Chlorine
- Cyanogen
- Fluorine
- Germane
- Hydrogen Cyanide
- Hydrogen Fluoride
- Hydrogen Selenide
- Hydrogen Sulfide
- Methyl Bromide
- Methyl Mercaptan
- Nickel Carbonyl
- Nitric Oxide
- Nitrogen Dioxide
- Nitrous Oxide
- Phosgene
- Phosphine
- Silane

Transport

- To transport or move a cylinder, strap it to a **hand truck** in an upright position.
- Never drop cylinders or bang them against each other or another object.
- Do not move a cylinder by rolling, dragging or walking it across the floor. Never leave a cylinder free-standing.
- Make sure the **valve protection cap** and outlet plug are in place. Leave the valve protection cap on at all times, unless the cylinder is in use.

Storage

- All cylinders must be secured upright with **chains and brackets** bolted to a solid structural member. Chains should be 3/16 inch welded link or equivalent. At least one chain must be used to secure each cylinder at a point two-thirds up the cylinders height. C-clamp bench attachments and fiber/web straps are not acceptable because they are not seismically sound. Any variations of these requirements must be approved by EH&S. (Campus Policy 5445)
- Keep cylinders away from heat and sources of ignition. Do not place cylinder where contact with any electrical circuit can occur. Protect cylinders from weather extremes, dampness and direct sunlight.
- Inspect cylinders and delivery equipment routinely for signs of wear, corrosion, or damage.

- All cylinders must be clearly labeled as to their contents — do not use unlabeled cylinders and do not rely on color coding for identification.
- Understand that “Empty” implies “end of service” and as such, the cylinder may still have greater than 25 psig of pressure remaining.

Leaks

- If the material in the tank is **toxic or flammable** and you suspect a leak, get everyone out of the area and report it to EH&S at x3194 and Dispatch at 9-911.

Use

- Gas delivery systems involving toxic gases must be authorized by EH&S prior to installation and operation.
- Use **regulators** designed for a specific gas. (Consult your gas vendor or catalog for proper regulator **compressed gas association (CGA) number** (on nut) for use with corresponding compressed gas cylinder. Do not use any adapter between cylinders and regulators.
- Post **signs** in laboratory area when using corrosive, toxic or flammable gases. The door placard system maintained by EH&S on the campus may be used for this.
- Never modify, adapt, force or lubricate safety devices, cylinder valve or regulator.
- Do not allow grease or oil to come into contact with **oxygen** cylinder valves, regulators, gauges or fittings. An explosion or fire can result. Oxygen cylinders and apparatus must be handled with clean hands and tools. Remember that oxygen supports and greatly accelerates combustion.
- Never force a gas cylinder valve — if it cannot be opened by the wheel or small wrench provided, the cylinder should be returned.
- When opening cylinder valve, do not hold regulator. Stand with valve between you and regulator. Open cylinder valves slowly, directed away from your face.
- Release a compressed gas gently to avoid build-up of static charge which could ignite a combustible gas.
- Special precautions are necessary for acetylene usage. Note that **acetylene** can form explosive compounds in contact with copper or brass. Consult the vendor or manufacturer for proper operating equipment and procedures.
- **Do not extinguish a flame** involving a highly combustible gas until source of gas has been shut off. Re-ignition can cause an explosion.

Disposal

- Empty cylinders should be labeled “EMPTY” or “MT. Always leave at least 25 psi minimum pressure in all “EMPTY” cylinders to prevent contamination and the formation of explosive mixtures.

- Return damaged or corroded cylinders and cylinders with a test date **more than five years** old stamped on the shoulder to the vendor. Some gas cylinders should be disposed or returned at shorter intervals (e.g., **corrosives** should be disposed or returned every six months since they readily attack the cylinder fittings).

8. Handling cryogens

Examples: Liquid oxygen, liquid nitrogen, liquid helium, dry ice

Hazard Properties

- These materials are extremely cold (-100°C to -270°C) and, upon contact, can instantly freeze other materials. Serious tissue damage may occur upon exposure.
- Evaporating liquid nitrogen will displace the air within a non-ventilated space possibly leading to **suffocation**. Generally, labs have adequate ventilation to prevent this.
- Be aware of **ice that can plug or disable pressure-relief devices**. Ensure adequate pressure relief mechanisms are functional, i.e., never use tight-fitting stoppers or closures without pressure-relief devices.

Practices

- Do not move an **over-pressurized container**. Evacuate and seal area, call EH&S (x3194) or dial 9-911.
- Avoid trapping cryogenic liquids between closed sections of an apparatus.
- **Dewar flasks** or other glassware devices should be taped on the outside or provided with shatterproof protection to minimize flying glass particles in case of implosion. Dewar flasks should be vented with a bored or notched stopper.
- Cool cryogenic containers slowly to reduce thermal shock and flashing of the material.
- Cryogen handlers should be protected by a **face shield or safety goggles, lab coat or apron and gloves or mitts.**
- When utilizing cold baths with solvents, use in a hood with a catch pan. Be aware of increased fire hazard. Be prepared for **vigorous solvent boiling** upon initial addition of solvent.
- Avoid **condensing oxygen** (blue in color) and/or contact with organic material when using liquid nitrogen. Flush cold traps with nitrogen or keep under vacuum to avoid condensation of oxygen from air within the trap. Condensed oxygen when contacted with organic materials can cause a powerful explosion.
- **Liquid helium** requires approved handling techniques and equipment due to over-pressurization hazards and icing.

Check the glassware and valves for cracks and other defects before beginning experimental work. Verify that systems assumed to be under vacuum are so, particularly when using liquid nitrogen. You should be on the lookout for the possibility of condensed air within the apparatus.

Skin contact with liquid nitrogen may least to a frostbite burn. An occasional droplet of nitrogen, such as is encountered when filling a Dewar, often does not freeze the skin because of insulting

film gaseous nitrogen, which forms immediately. However skin is readily frozen if the liquid nitrogen is held on a spot by clothing which is saturated with the refrigerant, or by any other means which leads to extended contact.
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Storage: Storage of liquid nitrogen: use only approved low temperature containers. Make sure liquid nitrogen containers are vented to prevent pressure buildup. You must use extreme care when working with liquid nitrogen. Liquid nitrogen should not be stored in sealed containers, as tremendous pressure could result and an explosion is possible.

Spill and accident procedures: Flood the area (skin and eyes) immediately with large quantities of cool water, apply cold compresses. See a doctor immediately if the skin is blistered or if the liquid nitrogen came in contact with your eyes.

9. Using vacuum systems

Vacuum systems have a variety of hazards associated with their operation. There are risks associated with implosion, as well as the release of toxic materials. The systems are typically complicated and require extensive training prior to use.

General Safety

- Understand the type of vacuum pumps being used and their limitations. Always check with the manufacturer for the appropriate application.
- Prepare for **power outages** whether you are present or not. Some valves close upon loss of power, some open. Understand the effects that a series of valve openings and closings will have upon the system's integrity.
- Always replace the pump **belt guard** to prevent catching fingers or clothing in the mechanism.
- Be aware of the hot surface in **oil diffusion pumps**
- If a glass vacuum line is ever used **above ambient pressure**, it should be shielded from personnel to prevent glass shards from flying if the line were to shatter.
- Glass vessels that are evacuated should be round-bottomed and/or thick-walled and designed for low-pressure work. They should be regularly checked for star cracks and scratches.
- The use of safety glasses is mandatory.

Traps and Venting

- Use of house vacuum systems must employ appropriate **traps** to prevent chemical, radioactive or biohazardous material from contaminating the building lines. Likewise, use of an aspirator should also employ a suitable trap to avoid contaminating the water stream.
- Mechanical vacuum pumps should be protected by **cold traps** – generally liquid nitrogen based.
- If hazardous materials are used with the vacuum system they should be located in, and **vented** to, a fume hood.

- Pump oil from vacuum system exhaust has been known to accumulate in building ductwork systems increasing the likelihood of fire spread. Pump exhaust should only be done **into the fume hood** proper, or if exhausted directly into building ductwork, an oil trap must be installed.
- Operation of low temperature traps must be thoroughly understood. Both the cooling and warming phases deserve undivided attention. For example, when using liquid nitrogen, the **condensation of air** due to an open valve may cause a serious explosion when the air vaporizes upon warming.
- **Dewar flasks are under high vacuum and are therefore subject to implosion.** They should be wrapped in tape or plastic sheathing.

Chemical Hazards

- Mechanical pump oil can become contaminated with hazardous materials that were being pumped on. Upon maintenance, proper protective equipment must be employed. A ventilated area should be used for changing pump oil, as harmful vapors may be released. Clean or contaminated pump oil must be disposed of as hazardous waste via EH&S.
- Mechanical pump exhaust may require suitable scrubbing for volatile highly toxic materials. This may involve a relatively simple filter or liquid bubbler.

10. High voltage and electrical safety

As a general rule, no electrical work on OCF equipment is allowed by users. If repairs or modifications are needed, please contact OCF personnel. Report all electrical malfunctions to OCF manager. In case of emergency, deenergize the equipment if this can be done safely, notify OCF personnel, campus facilities, and call 9-91, if needed.

Certain equipment at OCF, such as photomultiplier tube detectors utilizes high voltages up to 5000 V for operation. Normally, high voltage power supplies are connected to detectors by specialized, well-insulated cables and no hazard is present to the users. Nevertheless, the following rules must be followed:

- Use only cables with high voltage rated connectors provided at OCF. Do not use other cables or force the connectors if they do not plug with minimal force.
- If unusual sparking, noises, or burning insulation odor is noticed, deenergize the equipment immediately and report the problem to OCF personnel. (This is a general electrical safety rule, not only for high voltage applications).
- Use only power cables with 3 prong plugs for high voltage power supplies and other equipment unless a different plug is specified by the manufacturer.

11. Laser safety

Laser safety rules and procedures at OCF are regulated by UCSB laser safety manual (on file at OCF or online http://www.ehs.ucsb.edu/units/rad/radrsc/radsaftypdfs/lasersaftyman.pdf) and OCF lab specific addendum (on file at OCF or online).

12. Equipment specific safety

Janis Research VPF-100 Liquid Nitrogen Cryostat

- After placing a sample inside and reassembling the cryostat, connect the temperature controller and verify operation of the temperature sensor and the heater prior to the evacuation of the cryostat.
- Connect the cryostat to the Alcatel turbopump station, evacuate it to less than 10^{-4} mBar, and leave pumping for at least 15 minutes. Close the cryostat valve and turn off the pump station. Disconnect the pump manifold from the cryostat only after the turbo pump rotor rpms drop to 0.
- To cool down the cryostat bring 1 L of liquid nitrogen in a proper Dewar vessel (available at OCF). Wear appropriate PPE to protect yourself from hazards associated with liquid nitrogen (Thermal gloves, lab coat, safety goggles or face shield). Securely place cryostat on the optical table or the floor away from optics and instruments, connect it to the temperature controller. Using thermally insulated funnel, pour a small amount (~100 mL) of the liquid nitrogen into the cryostat. Wait until violent boiling of the liquid nitrogen stops (watch the intensity of the water vapor plume expelled from the cryostat). Repeat the addition of the liquid nitrogen 1-2 times. This will cool the cryostat to near liquid nitrogen temperature and prevent violent boiling and expulsion of the cryogen when the cryostat is filled. Then slowly fill the cryostat with the liquid nitrogen through the funnel until the drops of liquid nitrogen start being expelled back from the cryostat opening. Do not overfill the cryostat. Slowly remove the funnel and discard the LN_2 remaining in it back into the Dewar vessel. Let the cryostat to cool down to minimum temperature (~78 K). During this time thermal impedance dipstick can be cooled by inserting it into the Dewar vessel. When the cryostat reaches the LN_2 temperature, it can be refilled with the cryogen and the cooled dipstick can be inserted into it. The latter should be done slowly to avoid LN_2 splashes and the exhaust port on the dipstick top assembly should be turned away from people and equipment. After the dipstick has been inserted and secured the cryostat can be moved carefully to the instrument and is ready for the use.
- If moisture is collecting on the cryostat or its windows, the vacuum inside the cryostat is bad and it needs to be reevacuated and possible leaks need be sealed.
- After the use, carefully remove the cryostat from the setup. Slowly pull out the dipstick and drain the remaining LN_2 into the Dewar vessel. Before opening, the cryostat should be warmed up to the room temperature overnight or by blowing house air through it at the

rate ~1-3 CFM for at least 1 hour while monitoring the internal temperature. Release the vacuum by opening the cryostat valve before removing the sample.