VALPARAISO UNIVERSITY LABORATORY SAFETY MANUAL

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INTRODUCTION

Valparaiso University is dedicated to protecting the health and safety of its laboratory users through compliance with applicable local, state, and federal regulations. The Occupational Safety and Health Administration (OSHA) established 29 CFR 1910.1450, Occupational Exposure to Hazardous Chemicals in Laboratories, on Jan. 31, 1990, to provide guidelines for the safe use of chemicals in laboratories. This standard applies in locations where laboratory use of hazardous chemicals occurs and there is a risk of exposure. Laboratory use of hazardous chemicals entails the handling or use of chemicals in containers that can easily and safely be manipulated by one person or where multiple chemicals or chemical procedures are used.

The purpose of this Laboratory Safety Manual is to provide all laboratory workers and students a plan to avoid unnecessary chemical exposures and to address basic standard operating procedures (emergency procedures, chemical procurement, maintenance, etc.) that are used at Valparaiso University and to provide the necessary framework for compliance with the OSHA Lab Standard.

RESPONSIBILITIES

University President, Vice Presidents, Deans, Department Heads, Managers, Laboratory Supervisors

- Establish laboratory safety as an institutional priority.
- Provide adequate financial and political support for safety compliance at Valparaiso University.
- Include laboratory safety, chemical storage, and disposal considerations in long-range facilities planning.

Campus Health and Safety Committee

- Support departmental implementation of laboratory safety policy and appropriate training resources.
- Facilitate inspections; test and flush eyewash stations, safety showers, and chemical fume hoods; and assess overall cleanliness.
- Prepare, implement, and maintain this written Laboratory Safety Manual, setting forth general procedures, control measures, and information intended to assist supervisors in protecting employees and students from harm arising from chemical exposure.
- Assist supervisors in locating and obtaining safety data sheets (SDSs) upon request.
- Maintain a master chemical inventory.

Facilitate scheduled training and safety events (see Appendix 1).

Lab Supervisor

- Maintain an up-to-date copy of the Valparaiso University Laboratory Safety Manual and ensure that laboratory personnel comply.
- Provide guidance to all laboratory users of chemicals to ensure that the laboratory is a safe and healthy workplace and learning environment.
- Create appropriate laboratory-specific standard operating procedures (SOPs) to supplement this plan and forward to the Campus Health and Safety Committee.
- Train or arrange for training of laboratory workers, including students and visitors, at the time of initial employment and each time new procedures or hazards are introduced.
- Maintain training records.
- Implement and enforce the use of safety procedures including necessary personal protective equipment, engineering controls, or work practices.
- Ensure that the areas where hazardous chemicals are used or stored are secured when not in use and that lab doors are closed and locked when not occupied in accordance with the <u>Academic Building Access Policy</u>.
- Ensure that all chemical containers used or stored in the lab are properly labeled.
- Provide review for hazardous chemical safety issues that may be included in grant work.
- Correct identified deficiencies on lab inspection report and submit a written plan of action with completion date to the Campus Health and Safety Committee.
- Maintain current chemical inventory and emergency contact list and forward updated information to the Campus Health and Safety Committee.
- Maintain employee exposure to hazardous chemicals below permissible exposure limits set forth in OSHA 29 CFR 1910.subpart Z.
- Arrange for appropriate air monitoring when required by a chemical-specific standard or when exposure is anticipated or suspected and notify affected lab users of results in a timely manner.
- Arrange for appropriate routine medical surveillance as required by OSHA regulation for specific hazardous chemicals.
- Provide necessary personal protective clothing and equipment.
- Ensure that engineering controls are functioning properly and arrange for maintenance if required.
- Ensure the availability of safety data sheets and relevant reference materials for each chemical used or stored in the lab.
- Contact the Campus Health and Safety Committee to schedule chemical waste pickup.
- Maintain records of employee exposure determinations and lab related exposure incidents. Forward copies to the Campus Health and Safety Committee and Human Resource department.

Lab Users

Read and follow the guidelines in the Laboratory Safety Manual.

- Participate in initial and refresher training.
- Do not remove or deface labels on incoming chemical containers.
- Immediately label all secondary containers with the chemical constituents, hazard warning, responsible party or manufacturer, and date of preparation.
- Report all exposure incidents or hazardous conditions to your lab supervisor.
- Become familiar with laboratory hazards and use prescribed hazard controls (personal protective equipment, engineering controls, work practice).
- Request information or training when unsure about how to handle a hazardous chemical or procedure.
- Inform supervisor of any identified hazard or potential hazard.
- Perform only authorized work, preparations, and experiments in the laboratory.
- Working alone will not be permitted unless the lab has no significant safety concern and is reviewed by the Campus Health and Safety Committee as a lab with limited risk.

INFORMATION AND TRAINING

TRAINING

Students, faculty, and staff who work with chemicals and/or work in a laboratory setting must undergo training. Supervisors shall apprise all faculty and employees of the hazards of chemicals with which they work. The goal of the training program is to ensure that all workers are adequately informed about the risks associated with laboratory work and what to do when an accident occurs.

This information will be provided:

- At the initial assignment to the work area;
- Prior to assignments involving new exposure situations; and
- At least annually.

The content of initial assignment shall consist of, at a minimum:

- The Laboratory Standard and Hazard Communication Standard and their contents (29 CFR 1910.1450 and 29 CFR 1910.1200);
- The location and availability of the Laboratory Safety Manual;
- The permissible exposure limits (PELs) or, in the absence of a PEL, other recognized recommended exposure limits (RELs) such as the ACGIH TLVs, AIHA WEELs, or NIOSH RELs;
- Signs and symptoms associated with exposures to hazardous chemicals in the laboratory; and
- Location and availability of known reference material on the hazards, safe handling, storage, and disposal of chemicals found in the laboratory. Known reference material shall include SDSs and may also include quality reference material available from reputable sources such as peer-reviewed books,

journals, etc.

Training shall be conducted in the following topics at least annually:

- The methods and observations that employees may use to detect the presence or release of a hazardous chemical. These methods may include workplace monitoring, visual appearance, or odor of hazardous chemicals;
- Physical and health hazards of chemicals in the work area;
- The measures employees may take to protect themselves from these hazards, such as the use of engineering controls, appropriate work practices, emergency procedures, and PPE; and
- The specific work practices of this Laboratory Safety Manual.

The lab supervisor will provide lab-specific training for their department and individuals working in their laboratory. Training will include:

- Location of emergency equipment such as eyewash stations, fire extinguishers, fire pull stations, safety showers, etc.;
- Location and use instructions for personal protective equipment in the lab;
- Emergency evacuation plans, including exits, evacuation routes and designated meeting locations;
- Chemical storage and associated hazards and waste disposal procedures;
- Location of designated areas for use of carcinogens, reproductive toxins, or acutely toxic substances; and
- Location and access instructions for a copy of the laboratory chemical inventory, laboratory safety plan, safety data sheets, and laboratory specific standard operating procedures or methodologies.

All training shall be documented. Documentation shall, at a minimum, consist of a training session sign-in sheet and a copy of the training syllabus. Training documentation shall be maintained with the employee's training record and/or administrative folder. Electronic online training and documentation will also be accepted.

MEDICAL PROGRAM

If a job or position requires medical testing, this testing is provided as described in the Valparaiso University Campus Health and Safety Program and 29 CFR 1910.1450. If an employee exceeds the PEL, medical testing is covered. For further details consult with your supervisor.

All employees who work with hazardous chemicals will have an opportunity to receive medical consultation at no cost under the following circumstances:

 When the employee develops symptoms associated with a hazardous chemical to which the employee may have been exposed in the lab;

- When exposure monitoring reveals an exposure level above an action level or permissible exposure limit for an OSHA regulated substance for which there is a medical surveillance requirement; or
- When an event likely to produce a hazardous exposure occurs while the employee is in the lab (e.g., a spill, leak, or explosion).

EMERGENCY PROCEDURES

Planning ahead for emergencies is critical for remaining safe during an emergency and recovering quickly from an emergency. See the Valparaiso University Campus Health and Safety Program Document for more information.

- Know the emergency procedure for your building or area.
- Identify the location of emergency equipment and learn how to use it.
 Commonly used equipment includes fire extinguishers, fire alarm pull stations, telephones, first aid kits, and emergency eyewash stations and safety showers.
- Know the hazards and the proper precautions. Materials and equipment in your building or area pose different hazards. These hazards may require special precautions to avoid or minimize risk.
- Identify at least two exits from your area. One of them may be inaccessible during an emergency. Ensure that the path out is clear of any obstructions and that all doors leading out of the building can be opened.
- If you have a disability that could interfere with an emergency evacuation, work with your supervisor to plan for emergency situations.
- Study and remember the features of buildings, including stairways, exits, emergency phone locations, and elevators.

For general emergency information, refer to the <u>Campus Alert System</u>. This is also known as the Campus Emergency Flip Chart.

Small Chemical Spills

A spill is any chemical out of the control of the person working with the material. Small spills are distinguished from large spills, not only in their size, but also in the toxicity of the chemical and the person's ability to clean it up. Laboratory users may clean up small spills when they have the necessary materials in the lab and have the appropriate training to clean it up safely. A small spill is one that meets all of the following criteria:

- There is a relatively small amount of chemical, usually one liter or less of liquid;
- A single person can clean it up (a contaminated employee requiring the use of an eyewash or safety shower is considered a large spill);
- Proper PPE and additional equipment needed to clean up the spill is available; and
- The clean-up will not exceed any recognized exposure limit (e.g., PEL, TLV-TWA,

TLV-STEL, WEEL, etc.). Each laboratory is equipped with a spill kit. The unit supervisor will designate where each spill kit is located.

All spills involving mercury must be reported immediately to your supervisor or VUPD at ext. 5430.

Spills that the user cannot clean up that occur after normal business hours should be reported to VUPD at ext. 5430. Individuals should evacuate the area and wait to meet VUPD to describe the situation.

When cleaning up the spill, remember the acronym SWIM:

- S Stop the Spill: Secure the source of the material as rapidly as possible to reduce the volume necessary for cleanup.
- W Warn Others: Let others in the area know that there is an emergency in progress. Enlist others for help if necessary.
- I Isolate the Area: Do not allow others to track contamination throughout the facility. Consider securing ventilation for chemicals that evaporate rapidly by ensuring laboratory doors are closed so fumes do not carry to adjacent areas.
- M Minimize Your Own Exposure: Wear proper PPE for emergencies. Goggles and safety glasses, gloves (perhaps double gloves), and a lab coat are necessary. If a respirator is needed to prevent overexposure, the spill is "large" and a HAZMAT team is required.

Start at the edge of the spill and work toward the center with absorbents. Clay materials (such as kitty litter or commercial materials such as Spill-X) may also be used if convenient. Bag all clean-up material as hazardous waste and label for the next waste pickup.

Large Chemical Spills

Large chemical spills require a hazardous materials (HAZMAT) team to clean up. Valparaiso University relies on the Valparaiso Fire Department to coordinate large spill cleanup. In the event of a large spill, evacuate the area, and if necessary the entire laboratory or building, and call VUPD (ext. 5430) or 911.

Chemical Exposure

Eyes: Flush eyes in emergency eyewash for at least 15 minutes. If wearing contacts, flush eyes until the person can reasonably remove the contacts, then continue flushing. Immediately call emergency medical services at 911. Notify supervision.

Minor Skin Contact: Flush affected area with copious amounts of water. If irritation persists after flushing for at least 15 minutes, continue to flush and notify VUPD at ext. 5430. Notify supervision.

Major Skin Contact: Flush using emergency shower. Remove contaminated clothing and continue to flush for at least 15 minutes. Immediately notify emergency medical services at ext. 5430 or 911. Notify supervision.

Fire

Campus personnel are not required to fight fires, but may extinguish small fires (e.g., a trash can fire) with a fire extinguisher if they are properly trained and feel comfortable doing so.

Small fires contained in laboratory hoods should be left to burn out on their own. The hood sash should be shut and the exhaust fan turned on. Everyone in the laboratory should be notified that an emergency is in progress and notify VUPD at ext. 5430.

In the event of a large fire, evacuate the building and immediately use a pull-station and call VUPD at ext. 5430 or 911. Students and staff should assemble in a designated parking lot far enough away from the building. Laboratory supervisors are responsible for accounting for their students and workers.

Fire extinguishers should be visually checked monthly and checked yearly by Facilities Management or their designee. After using a fire extinguisher, inform your supervisor and a work order will be filled out to ensure that it is promptly recharged.

STANDARD OPERATING PROCEDURES

The following standard operating procedures are general safety guidelines applicable to all Valparaiso University laboratories. Individual labs should supplement these with lab specific standard operating procedures.

Personal Safety and Responsibility

The following safe work practices are established for laboratory workers.

- No eating, drinking, smoking, or application of cosmetics is allowed in any laboratory.
- Do not store food or drinks in chemical or sample storage refrigerators.
- Do not take chemical work outside the laboratory workspace.
- Perforated shoes, sandals, or cloth sneakers should not be worn within the laboratory.
- Keep all work areas clean and free of contamination.
- Safety data sheets (SDSs) shall be maintained for all chemicals that are received with incoming shipments of hazardous chemicals. These SDSs shall

- be readily accessible to all laboratory employees.
- Chemical labels on incoming containers shall not be removed or defaced. All
 chemical labels for chemicals or solutions produced in the laboratory must
 contain, at a minimum, the following information: chemical name, date
 prepared, and the person who prepared the solution. The use of formulas or
 symbolic nomenclature (i.e., AgNO₃) alone is not permitted.
- If children or visitors are allowed in the laboratory, ensure they are under constant supervision of qualified people.
- Never pipette by mouth.
- Wash promptly if minor skin contact is made with any chemical, regardless of its corrosivity.
- Avoid contamination of personal clothing by wearing a laboratory coat.
 Laboratory coats should be changed when significant amounts of contamination are detected.
- Avoid inhaling chemicals by performing all work with hazardous chemicals within a properly operating laboratory chemical hood. Refer to the section on the proper operation of laboratory chemical hoods in this laboratory safety manual for more information.
- Confine long hair and loose clothing when working with chemicals. It is best to remove jewelry to avoid possible contamination or destruction of the item by chemical vapors.
- Access to emergency equipment (eyewashes, showers, and fire extinguishers) must be unobstructed at all times.
- Clean work areas regularly.
- Do not use floors, stairwells, or hallways as chemical storage areas.
- Student use of cellular devices should be minimized in laboratory settings to reduce chances of contamination and distraction. Use of cellular devices in laboratories shall be governed by the appropriate supervisor.
- Do not use deionized water or laboratory ice for personal consumption.
- Cap all containers after use and before transporting.
- Transport chemicals within a tightly sealed chemically resistant container inside of a chemically resistant secondary container or pan that can contain any spill or leak.
- Ground all metal containers when dispensing flammable liquids. Only small quantities of flammable liquids should be transferred to glass containers.
- Clean drips and/or spillage off of container exteriors immediately.
- Maintain clear exits and aisles.
- An "unattended operation" that is hazardous in nature is one where the
 laboratory worker is not present while a procedure is underway. Workers
 should ensure that an adequate warning is placed on or near the equipment
 (or laboratory chemical hood) stating that there is a procedure in progress,
 any hazards it might pose, and who to contact in the event of an emergency.

Working alone in a laboratory is not permitted unless the lab has no significant safety concerns and is approved by the supervisor. Students in undergraduate teaching laboratories shall not work alone under any circumstances.

Personal Protective Equipment (PPE)

Personal protective equipment is the final barrier between the hazardous chemicals and exposure. Though often dismissed as "too uncomfortable" or "too bulky," PPE is a vital element in exposure prevention.

Gloves

In order to prevent skin exposure, chemical protective gloves should be worn when handling chemicals. Chemical protective gloves shall be used at all times when particularly hazardous substances are used. Change gloves frequently throughout the day and wash your hands to prevent trapping hazardous material on your skin by the glove material. Remove gloves before leaving the lab or handling uncontaminated items (e.g., doorknobs, computer keyboards, instruments).

No glove is impervious to all chemicals, and each glove material must be evaluated against the chemicals that will be handled when the glove is on. Be sure to fully understand the capability of the glove to protect you against your procedure. Check the SDS for recommendations.

- Nitrile gloves: Provide excellent dexterity and good general-purpose chemical protection against acids, bases, and some solvents.
- Other chemically resistant gloves: Gloves made of materials such as butyl rubber, neoprene, and laminated or composite materials provide protection against a variety of chemicals.
- Latex gloves: Do not use latex gloves for chemical protection.
- Furnace and hot gloves: Furnace gloves are typically made of spun or woven ceramic material and can withstand temperatures in excess of 300°C. Hot gloves, usually thick cotton, can provide limited protection to about 150 °C.
- Cold gloves: Cold gloves are used in transferring liquid nitrogen to prevent skin exposure.

Protective Eyewear

Protective eyewear is required in all teaching and research laboratories. The minimum level of protection is safety glasses with side shields. All people in laboratories, including visitors, must wear appropriate ANSI Z87.1 approved eye protection when the potential exists for eye injury. Safety goggles or glasses with side shields may be used as appropriate. Standard prescription eyeglasses are not sufficient. Contact lenses may be worn in the lab with appropriate safety glasses or splash goggles.

Chemical splash goggles should be worn in addition to safety glasses whenever there is an increased risk of chemical spill, splash, or fragmentation in the work area. Chemical splash goggles shall be worn when transferring greater than 50 milliliters of corrosive liquids or solvents in an open process, such as pouring. Additionally, chemical splash goggles shall be worn whenever using glassware at pressures above or below atmospheric pressure.

Face shields shall be worn in addition to safety glasses whenever transferring liquid nitrogen from storage Dewar flasks to other containers. Additionally, face shields should be worn in addition to safety glasses or chemical splash goggles when the employee or supervisor believes a process warrants additional facial protection. Such processes may include, but are not limited to: working with highly reactive or explosive material, working with large volumes of highly concentrated acids/bases (e.g., 90% nitric acid), or working with glass under extreme pressure or temperature.

Protective Clothing

Chemical protective clothing in addition to laboratory coats and safety glasses will be provided as necessary.

- Wear closed-toed shoes made of a non-woven material with non-slip soles.
- Wear clothing that covers exposed arms and legs while working in the lab.
- Wear a closed lab coat while in the lab.
- Lab coats must be removed before leaving the lab unless walking to another laboratory.
- Nonflammable, nonporous aprons should be used when corrosive liquid chemicals are used.

Chemical Specific Procedures

Particularly Hazardous Substances (PHSs)

A PHS is a chemical that meets any *one* of the following criteria:

- The substance is a "select carcinogen." A select carcinogen meets one of the following criteria:
 - Regulated by OSHA as a carcinogen; or
 - Listed as "known to be a carcinogen" by the National Toxicology Program (NTP) Annual Report on Carcinogens; or
 - Listed under Group 1 ("carcinogenic to humans") by the International Agency for Research on Cancer (IARC) Monographs; or
 - Classified as certain Group 2A or 2B by the IARC or "reasonably anticipated to be carcinogens" by NTP; or
- The substance is a reproductive toxin, mutagen, or teratogen; or
- The substance is a chemical with a high degree of acute toxicity ($LD_{50} < 50$ mg/kg oral).

There is increased health risk when working with PHSs. All work with PHSs shall be performed in a properly operating laboratory chemical hood or properly functioning glove box unless it can be otherwise quantitatively demonstrated that there is no measurable inhalation exposure during routine work practices. Gloves used in PHS experiments shall be disposed of as hazardous waste. Check the SDS for appropriate handling and disposal procedures.

Toxic Chemicals

Many SDSs and other reliable references often state recommended exposure limits, PELs, or both as exposure guidelines. When these limits are stated in reference materials, they will be used to assist the lab worker, supervisor, and Campus Health and Safety Committee in determining appropriate safety precautions, control measures, and personal protective equipment for working with that chemical. The following are established guidelines for handling toxic chemicals:

- When a TLV (NIOSH) or PEL (OSHA) value for a particular chemical is less than 100 mg/m³, the chemical shall be handled in a properly operating laboratory chemical hood, glove box, or other ventilated enclosure that exhausts to the outdoor atmosphere.
- If the TLV, PEL or other recommended exposure limit is not available for that substance, the animal or human median lethal concentration (LC₅₀) should be reviewed if available. If that value is less than 2,000 mg/m³ (when administered continuously for one hour or less), the chemical shall be handled in a properly operating laboratory chemical hood, glove box, or other ventilated enclosure which is equipped with appropriate traps and/or scrubbers.
- When handling substances with substantial vapor pressure in amounts that are likely to exceed airborne concentration limits (PELs, TLVs, etc.), work with such substances shall be performed in an operating laboratory chemical hood, glove box, or other ventilated enclosure that exhausts to the outdoor atmosphere.

Whenever an overexposure is suspected, contact your supervisor immediately for possible air monitoring.

Flammable Chemicals

A "flammable" chemical is one with a flash point below 100°F (37.8°C). Lab practices shall be in accordance with OSHA and NFPA standards and guidelines on flammable chemicals in the laboratory. Flammable chemicals shall be stored in flammable solvent storage cabinets and used in an operating laboratory chemical hood whenever possible and with a limited risk away from sources of ignition.

Reactive Chemicals

A reactive chemical is one that meets any one of the following criteria:

- Is described as reactive in the SDS;
- Is ranked by the NFPA as 3 or 4 for reactivity;
- Is identified by the US Department of Transportation as a Class 5 oxidizer, an organic peroxide, or DOT Class 1.1–1.6 explosive;
- Is defined as a reactive by the US EPA in 40 CFR 261.23;
- Is defined as unstable by OSHA; or
- Is known or found to be reactive with other substances.

All reactive chemicals will be handled with prudent safety precautions to prevent unwanted reactions. All reactive chemicals shall be segregated in storage. Air reactive chemicals shall be stored under inert gas or under an appropriate vapor barrier (e.g., oil).

Corrosive Chemicals and Contact Hazards

Corrosive, allergens and sensitizing chemicals are often found in chemical laboratories. Many times, the manufacturer will label these chemicals on the SDS, or more information may be found in reliable resources.

A corrosive chemical is one that fits the OSHA definition of corrosive in appendix A of the hazard communication standard (29 CFR 1910.1200) or fits the US EPA definition of corrosive as found in 40 CFR 261.22 (i.e. has a pH greater than 12.5 or less than 2.0) or is known or found to be corrosive to living tissue.

A contact hazard is an allergen or sensitizer that is identified by the manufacturer on the SDS or label, or is identified or described as an allergen or sensitizer in medical or industrial hygiene literature.

Handle all corrosive or contact hazard chemicals with good laboratory practice and proper personal protective equipment. Per recommendation from the SDS, wear appropriate PPE. Consider the use of a face shield in combination with safety goggles and gloves known to be resistant to permeation or penetration to that chemical. While a laboratory coat and safety glasses are always required, consider the use of a laboratory apron over the lab coat if necessary.

Compressed Gases and Cryogenic Liquids

Compressed gases can pose significant mechanical and chemical hazards within the facility. Mechanical hazards are present simply because of the tremendous amount of stored energy within a compressed gas cylinder. Typical pressures for the cylinders used in the laboratory are about 2,500 psig. Chemical hazards manifest themselves in terms of the substance within the compressed gas cylinder.

Cylinder Contents — Gas cylinder color shall not be used to determine contents.

Gas cylinders must be clearly labeled (or stenciled) with the

contents of the cylinder. Notify the gas distributor if cylinders contents are not clearly identified. Cylinders must be labeled with content status (full, empty, or in-service). Cylinder transfilling is prohibited.

- User Responsibilities The maintenance of a compressed gas cylinder is the responsibility of the supplier, not of the user. The user shall not perform any maintenance on the cylinder, cylinder valve, or relief device, nor shall the user alter any prescribed markings on the cylinder or in any way modify the cylinder. Modification includes painting or defacing the cylinder. If the possibility that a foreign substance has entered the cylinder or valve and contaminated the contents, the user will notify their supervisor. The supervisor shall ensure that the gas supplier is notified and the cylinder is taken out of service.
- Cylinder Placement Gas cylinders shall not be placed where they might become part of an electrical circuit. If a cylinder is used in conjunction with electric welding, it shall not be grounded or used for grounding. Cylinders shall not be exposed to temperatures greater than 125°F or subjected to artificially created low temperatures.
- Cylinder Condition If a container or valve is noticeably corroded or damaged, notify your supervisor.
- Valve Caps Valve protection caps for a cylinder shall always be in place and hand tight except when the cylinder is in use or connected for use. Do not switch valve caps since not all gas suppliers use the same cap threads. If a valve cap is cracked or dented, notify your supervisor. If the container has a valve outlet cap or plug, keep the device on the valve unless the container is connected for dispensing.
- Safe Handling of Containers Always use a hand truck to move compressed gas cylinders more than five feet or outside the tank storage room. Always ensure the protective cap is equipped. Do not use the valve protective caps for lifting.

Cylinder Use and Storage

Cylinder Positions in Storage and Use — All compressed gas cylinders in storage and in use shall be upright and chained in place.

Compressed gas cylinders in use shall be secured to prevent falling or rolling. All cryogenic liquid cylinders shall be stored

upright.

- Connections and Content Withdrawal Container valve connections shall not be forced. Regulator threads must match those on the container valve outlet. Only valve outlet connections and regulators that conform to CGA V-1, American National/Compressed Gas Association Standard for Compressed Gas Cylinder Valve Outlet and Inlet Connection shall be used in laboratories. Teflon pipe thread tape shall not be used when connecting gas cylinders and regulators.
- Regulators A suitable pressure regulator shall always be used when using compressed gases. Before a regulator is removed from the container, relieve the pressure on the regulator by closing the cylinder valve and opening the outlet valve of the regulator.
- Residual Container Pressure When using a non-liquefied compressed gas from a container (except acetylene), the pressure should not be reduced below the operating pressure of the system or less than 20 psig to prevent backflow of atmospheric air or contaminants into the cylinder.
- *Transportation* Gas cylinders shall not be transported in personal vehicles or unsuitable vehicles.

Safe Handling of Gases by Hazard Class

- Asphyxiate Gases/Inert Gases Inert and simple asphyxiate gases (e.g., nitrogen, helium) may cause suffocation by displacing the oxygen in the air. Inert gases are chemically inactive, odorless, tasteless, and colorless. A catastrophic failure of a liquid nitrogen/helium tank may displace oxygen in sufficient quantity to cause asphyxiation. Only emergency personnel trained and qualified to respond to inert gas leak and using proper personal protective equipment shall enter an area of deficient oxygen concentration.
- Oxidizers Regulators and equipment used for oxygen are to be clean and free from oils, greases, and other contaminants. Oxidizing gases shall be stored separately from flammable gases and other combustible material.
- Cryogenic Liquids Cryogenic liquids are gases that are handled in liquid form at relatively low pressures and extremely low temperatures, usually below -130°F. They are handled in double wall-vacuum insulated containers. Cryogenic liquids can cause thermal burns

when the liquid contacts bare skin. When handling cryogenic liquids, wear suitable eye protection (safety glasses and face shield), a lab coat, and loose-fitting insulated gloves.

Handle containers with care and store containers in the upright position in a well-ventilated area. Transfer the liquid slowly to prevent excess spattering and to lower the stress on material caused by rapid cooling. Store and transfer cryogenic liquids under positive pressure to prevent infiltration (and solidification) of moisture or air.

Exposure Determination

It is required by regulation to measure the employee's exposure to any substance regulated by a standard that requires monitoring if there is a reason to believe that exposure levels for that substance routinely exceed the action level of the permissible exposure limit (PEL).

The employer shall conduct and document hazard assessments for all procedures that utilize hazardous chemicals (those with a 3 or 4 in any NFPA rating) within the laboratory facility to determine if there is a reason to believe any PEL or recommended exposure limit is exceeded. Refer to 29 CFR 1910 Subpart Z for a list of toxic and hazardous substances and PEL levels. Monitoring will be performed in accordance with the relevant substance standard and the Laboratory Standard, 29 CFR 1910.1450.

LABORATORY CONTROLS

Control measures to reduce employee exposure utilize a multi-layered control method to reduce employee exposure to hazardous chemicals in the laboratory facility. This approach includes administrative controls (also called standard operating procedures) and the use of personal protective equipment (PPE) which have been discussed in previous sections. Engineered controls provide another layer of protection.

Chemical Fume Hoods

Proper Operation: All chemical fume hoods shall be properly operating at the time of use. "Proper operation" is defined such that the hood shall demonstrate effective capture and containment of chemical vapor emissions and particulate matter during operation. (See also: ANSI Z9.5 (latest edition). It is understood that individual hoods may not maintain 100 ± 20 fpm face velocity, however the hood will be deemed satisfactory if containment is clearly demonstrated using smoke or similar testing. Hood face velocity alone shall not be used to determine sufficient containment of chemical vapor.

Testing and Maintenance: Chemical fume hoods shall be tested semiannually for containment as described in Appendix 2 of this plan. Documentation for proper hood testing and maintenance shall be maintained by Valparaiso University Facilities Management.

Test Failure: In the event a hood fails a routine containment test or if there is any reason to suspect the hood has failed to sufficiently contain chemical vapors, the hood shall be placed out of service until it is repaired. A prominently displayed sign notifying employees and students that the hood is out of service will be posted. A complete containment check will be performed in accordance with Appendix 2 of this laboratory safety manual before the hood is released for use.

Hood Use and Discipline: Use the chemical fume hood for all operations that might result in an odoriferous, volatile, toxic, or harmful release. Toxic or unknown compounds should never be smelled. Finely divided solids (powders) have the potential to become airborne and uncontrolled within the laboratory environment, and as such, dusts are recognized as hazardous. The following general rules apply to the use of laboratory chemical hoods:

- When working with hazardous, unknown, or poorly characterized substances, use only hoods that have been evaluated for proper operation.
- Keep all reactions and work at least six inches behind the plane of the hood sash.
- Hood sash should be 18 inches or lower when in use. For all hoods with vertically rising sashes, work with the sash in the lowest position possible. When the hood is not in use, keep the sash closed.
- Never put your head into a hood to check the progress of a procedure or an experiment. Remember, the sash plane is the barrier between contaminated and uncontaminated air.
- Keep hoods clean and free from bottles. Do not use the hood as a storage cabinet.
- Report any real or suspected malfunction to your supervisor. Do not use the hood until you are sure it is working properly.
- Work that requires hood shutdown requires notification of building users (department chairs and administrative assistants) prior to shutdown.

General Laboratory Ventilation

The general laboratory ventilation system shall be operated so as to provide sufficient make-up air for all laboratory chemical hoods. Indications of insufficient general

laboratory ventilation include laboratory chemical hoods that cannot sufficiently contain chemical vapors, lingering odors within the laboratory, insufficient heating or air conditioning, excessive humidity within the laboratory facility, and the like. Issues should be immediately reported to the laboratory supervisor.

- General room ventilation patterns must not be altered. Do not block room air supply grills or return duct grills or remove drop ceiling tiles.
- Laboratory doors should be kept closed to ensure proper containment of fumes.
- Local exhaust (i.e., snorkels) should be used to capture point source discharges of toxic chemicals from apparatus as appropriate.
- Toxic chemicals should not be used outside of a chemical fume hood or other containment systems.

Fire Alarm System

The fire alarm system shall be tested in accordance with the local fire department's requirements.

Emergency Eyewashes and Safety Showers

Access to emergency eyewashes and safety showers shall be unrestricted and unobstructed. Keep caps on eyewashes to prevent dirt build-up on the nozzles. If the eyewash is missing or has a cracked eye cap, order replacement(s). At a minimum, all eyewashes shall be flushed weekly for at least three minutes to ensure the lines are clear and free from microbial growth in compliance with ANSI standards. Eyewashes must provide at least 15 minutes of clear running water. At a minimum, all showers shall be tested monthly to ensure they are operable. Eyewashes and safety showers shall meet ANSI Z358.1-2014 recommendations including the eyewash flow of 3 gpm and shower flow of 20 gpm.

Use of the eyewash or safety shower should be followed by appropriate medical treatment.

CHEMICAL PROCUREMENT AND STORAGE PROCEDURES

Chemical Procurement

Before placing an order, review the current stock and chemical inventory to ensure that the laboratory has indeed used all of the previous stock. When placing an order for any chemical, obtain information on the proper handling, peroxide storage, and disposal of the chemical from the supplier. This information may be in the form of the SDS. This information must be made readily accessible to all potential handlers and users of the chemical. It is the unit supervisor's responsibility to ensure that these conditions are met including conducting any specialized training that must take place. Ensure that all chemicals arrive with appropriate labels.

Purchase orders for chemicals for research and curriculum use shall be reviewed by the department head prior to purchase.

It is the policy that all chemicals shall be ordered using the minimum quantity in order to ensure the freshness of reagent chemicals and to minimize future disposal costs.

Chemical purchases with personal funds for use in Valparaiso University labs are prohibited.

Chemical Storage

Chemicals should be stored in a manner that reduces the chance of incompatible chemicals coming in contact and minimizes exposure to those working in the chemical storage area.

- Minimize the quantity of chemicals stored in the lab. Be particularly aware of materials with a high hazard or shelf life, including peroxide formers.
- Chemicals that have not been used in the past three years and are not anticipated to be used in identified laboratory procedures should be considered for disposal.
- Store chemicals in compatibility groups.
- A maximum total of 10 gallons of flammable liquids may be stored in a lab outside of a flammable storage cabinet.
- Store all chemicals in a manner that minimizes potential spillage onto personnel, equipment, and other chemical containers.
- Chemicals should be stored in closed cabinets. If open shelving must be used, it must be secured to the wall. Each shelf must have a minimum lip of 0.75 inches.
- Store corrosives and liquids below eye level (4–4.5 feet).
- Provide a barrier between compatibility groups in storage (e.g., use shallow tubs capable of containing a container spill).
- Containers will be labeled prior to storage as part of the lab inspection checklist for each department.

In general, chemicals with the following functional groups are prone to instability: peroxides, azides, nitro, nitroso, azo, nitrate ester, amino, and nitro amine. These reagents should be dated, carefully handled using prescribed procedures, and promptly (and properly) disposed after use.

The following is a partial list of chemical incompatibilities. A more complete list may be found in: (1) *Prudent Practices in the Laboratory*, (2) *Bretherick's Handbook of Reactive Chemical Hazards*, and (3) *Hazards in the Chemical Laboratory*.

Chemical	Incompatible with	
Chemical	Incompatible with	

Acetic acid	Nitric acid, peroxides, permanganates, chromic acid, hot perchloric acid
Acetone	Concentrated sulfuric acid, concentrated nitric acid
Alkali metals (group I)	Water, carbon tetrachloride, carbon dioxide, halogens
Alkaline metals (group II)	Carbon tetrachloride, chlorinated hydrocarbons, halogens, carbon dioxide

Ammonia (anhydrous)	Mercury, hydrogen fluoride, halogens, calcium hypochlorite
Ammonium nitrate	Acids, metal powders, flammable liquids, chlorates, nitrites, organics and combustibles
Activated carbon	Calcium hypochlorite, oxidizers
Fluorine	Isolate from everything
Hydrocarbons	All halogens, chromic acids, peroxides
Hydrofluoric acid	Ammonia (aqueous or anhydrous). Keep away from skin!
Hydrogen peroxide (>10%)	Copper, chromium, iron, metals and their salts, flammable liquids, combustible materials, nitromethane
Hydrogen sulfide	Fuming nitric acid, oxidizing gases
Nitric acid (concentrated)	Acetic acid, acetone, alcohols, chromic acid, hydrocyanic acid, flammable liquids and gases
Peroxides (organic)	Acids, avoid friction
Phosphorus pentoxide	Alcohols, water, strong bases
Sulfuric Acid	Chlorates, perchlorates, permanganates

Peroxide Testing

The following chemicals are known to form peroxides and can pose an explosion hazard. This list is not exhaustive; refer to Appendix 3 for a more complete list of peroxide formers. Peroxides derived from compounds labeled with an asterisk (*) may explode without concentration.

- Butadiene
- Chlorobutadiene
- Cyclohexane
- Cyclopentane
- Diethyl ether
- p-Dioxane

- Furan
- Isopropyl ether*
- Potassium amide*
- Potassium metal*
- Sodium amide*
- Styrene
- Tetrahydrofuran
- Vinyl acetylene
- Vinyl acetate
- Vinyl chloride
- Vinyl ethers
- Vinyl pyridine
- Divinyl acetylene*
- Divinyl ether*

All peroxide forming compounds shall be dated upon receipt and again upon opening. These compounds should be tested at least annually for peroxides according to the procedure outlined in Appendix 3.

All severe poisons ($LD_{50} \le 5$ mg/kg) shall be stored separately in a key-controlled, locked poison cabinet.

Hazard Identification

Chemical manufacturers and importers are required by OSHA to review available scientific evidence concerning the hazards of the chemicals they produce or import and to report the information to employers who distribute or use their products.

Valparaiso University relies upon labeling provided by the manufacturer or suppliers. Individual departments are responsible for ensuring that each secondary chemical container in the workplace is properly labeled. Secondary container labels must contain the identity of the chemical and be in compliance with Globally Harmonized System (GHS) labeling standards. Employees shall not remove or deface existing labels on incoming containers of hazardous substances. GHS requires that chemical labels contain a signal word, applicable pictogram(s), and a hazard statement for each hazard class and category.

Fixed containers such as storage tanks must be labeled with the identity of the chemical it contains and the appropriate hazard warning. Alternative written identification systems for fixed containers may be implemented as long as they convey the same hazard warning information.

The hazard warning label should clearly convey the appropriate GHS precautions, signal words, pictogram, or symbols that assure that the worker is aware of the chemical hazards associated with the chemical. Employees must be trained on the new label elements and the safety data sheet (SDS) format. Widely accepted systems for hazard identification and labeling include:

ANSI Z129.1-2000 as well as the GHS: Hazardous Industrial Chemicals — Precautionary Labeling uses a word hierarchy, or signal word to convey levels of hazard. The two signal words are *DANGER* and *WARNING*, the meaning of each are provided below.

DANGER — If this product gets in or on you, immediate harm will be caused.

WARNING — If this product gets in or on you, in sufficient quantity, you will suffer harm.

The Hazard Communication Standard requires pictograms on labels to alert users of the chemical hazards to which they may be exposed (Figure 1). Each pictogram consists of a symbol on a white background framed within a red border and represents a distinct hazard(s). The pictogram on the label is determined by the chemical hazard classification.

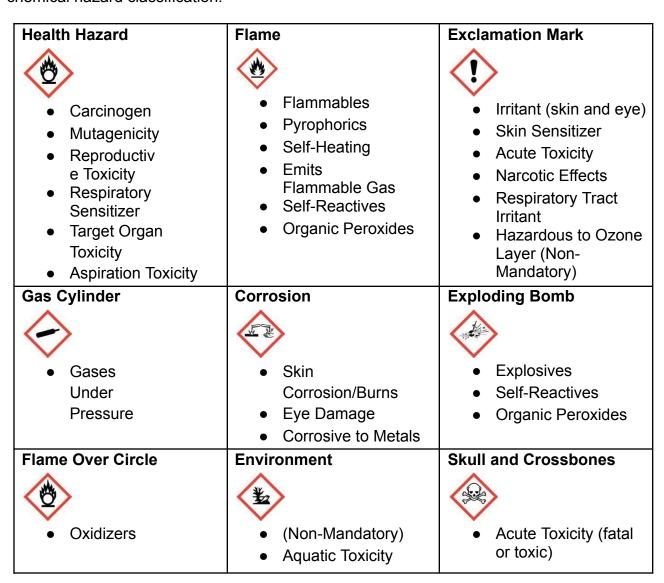


Figure 1. Hazard Communication Standard pictograms.

ENVIRONMENTAL AND WASTE MANAGEMENT

Valparaiso University is committed to responsible environmental stewardship. The purpose of this section is to provide environmental management guidance to faculty and staff as they conduct operations involving hazardous materials within laboratories. This section is designed to:

- Highlight policies which comply with environmental laws and regulations;
- Provide specifications for an environmentally safe workplace by improving overall environmental performance;
- Maximize the investment in environmental affairs; and
- Integrate environmental objectives into all laboratory and University functions.

The following Environmental Management Plan incorporates the best management practices available at the time of the writing. It closely follows the EPA's guidance found in the publication Environmental Management Guide for Small Laboratories (Publication 233-B-00-001, May 2000). It is not to be used as a substitute for environmental management regulations found in 40 CFR, transportation regulations found in 49 CFR, occupational safety regulations found in 29 CFR, or any state environmental, transportation, or occupational safety regulations. Always consult with a professional from environmental regulatory agencies if there is any question regarding regulatory interpretation.

Protection of the Air

Valparaiso University is not considered to be a "major source" of air pollution as defined by the Clean Air Act, nor does the laboratory use ozone depleting substances in routine analysis. Valparaiso University does not store any chemicals in quantities requiring a risk management plan per 40 CFR 68.

In order to reduce air emissions of hazardous chemicals from the laboratory, Valparaiso University has adopted the policy that evaporation of solvents via laboratory chemical hoods *not* be used as a disposal method (evaporation as a waste reduction method is forbidden by RCRA regulations).

Protection of the Water

Disposal of chemicals down the laboratory sink can be a dangerous practice. It may result in unwanted chemical reactions downstream, fire, odor, and damage to the plumbing system of the building. Additionally, drain disposal may cause environmental problems at the wastewater treatment plant. The following policy is adopted for all teaching and research laboratories at the University.

Indiscriminate sink disposal of laboratory chemicals is forbidden. However, if carefully

controlled, some wastes, such as neutralized acid/base wastes, may be safely discharged via the sewer. Additionally, this may be the most desirable disposal method as it minimizes waste sent off site.

The national pretreatment standards are listed below. These are found in 40 CFR 403.5 and contain specific standards prohibiting all sewer users from discharging the following pollutants into a sewer or publicly owned treatment works (POTW):

- Flammable or explosive pollutants (flammable liquids with a flash point of less than 140°F);
- Pollutants that will cause corrosive structural damage to the POTW, and in no cases discharges of pH less than 5.0;
- Solid or viscous pollutants that may cause an obstruction of flow in the POTW;
- Pollutants capable of releasing fumes or vapors in sufficient quantities to detrimentally affect the health and safety of POTW workers;
- Pollutants, including oxygen demanding pollutants (high BOD) at a concentration and flow which may cause interference with the POTW;
- Wastewater with sufficient heat to inhibit biological activity in the POTW (waste must not exceed 104°F at the POTW); or
- Petroleum, oil, non-biodegradable cutting oil, or products of mineral oil origin in amounts that will cause interference of pass through.

Aqueous waste that does not meet sink discharge standards or cannot be made to meet sink discharge standards as part of a written procedure shall be collected in properly labeled containers in the laboratory and disposed of as hazardous waste. No aqueous waste may be sink discharged without first performing a hazard determination per RCRA regulations.

Hazardous Waste Management

The Resource Conservation and Recovery Act (RCRA) of 1976 was written to provide so-called "cradle-to-grave" tracking of hazardous waste. In 1984, Congress expanded the scope of RCRA with the passage of the Hazardous and Solid Waste Amendments (HSWA). These amendments provided the wake-up call to laboratories to start managing hazardous waste.

Most laboratories generate hazardous waste and are therefore subject to RCRA regulations.

The laboratory supervisor or department head is responsible for coordinating the following tasks with each unit's representative:

 Identifying all hazardous waste produced by teaching and research laboratories:

- Preparing an inventory of all hazardous waste produced by teaching and research laboratories;
- Characterizing the waste generated by teaching and research laboratories;
- Maintaining a monthly waste log. Each waste log will contain, at a minimum, a description of the waste, type of waste (i.e., hazardous or acutely hazardous), hazardous waste class, and quantity. This waste log will be updated at least monthly and shall also include the amount of hazardous waste generated that month and the amount accumulated that month;
- Ensure all hazardous waste is properly stored before disposal:
 - All containers are properly labeled with the words "Hazardous Waste" and the contents identified with complete chemical names.
 No abbreviations (i.e., MeOH) or formulae are to be used on the label.
 - All containers are closed except when adding or removing waste
 - Maintain 1–1.5 inches of air space in the top of any container.
 - All containers are maintained in good condition (no leaks, rust, dents, etc.).
 - All containers are compatible with the waste that is stored in them.
 - The storage area is inspected weekly.
 - Ensure that accumulation quantity never exceeds 1,000 kg or 1 kg of acutely hazardous waste.
 - Ensure all applicable storage requirements are met, including those found in 29 CFR 1910.106 (flammable liquid storage) and NFPA 45.
- Ensure laboratory faculty and staff are trained in proper hazardous waste management;
- Maintain records of waste determinations, waste generation, training, and disposal and distribute manifest copies to the proper agencies; and
- Ensure that only RCRA-trained staff sign manifests as the generator.

The Campus Health and Safety Committee is responsible for coordinating the following tasks:

- Tracking the generator status. (Currently, Valparaiso University is a conditionally exempt small quantity generator). Ensure proper planning is done to keep hazardous waste generation rate at less than 100 kg/month.
- Arrange for disposal of hazardous waste with a reputable and permitted hazardous waste disposal firm.
- Ensure all paperwork (i.e., manifests) from the hazardous waste disposal firm is correct for each waste pickup.

Specific Waste Instructions

Valparaiso University combines similar and appropriate chemicals for disposal.

Laboratory workers should fill out the waste tag located on the safety can at the time of disposal.

Date	Initial	Waste	Quantity

Any solutions that are nonhazardous waste and may have come in contact with blood or other potentially infectious materials (OPIMs) may be poured down the sink followed by bleach.

Biological Waste Instructions

Separate biological waste from general trash and hazardous chemical waste. Use the universal biohazard symbol on containers containing biological waste.

Select proper packing material for the waste. Ensure the container is leak proof and puncture resistant. Ensure the packing material will maintain its integrity during transportation.

Do not compact biological waste.

Minimize storage time.

Select the most appropriate treatment option for the waste. Use a licensed medical waste hauler to transport biological waste to its disposal.

All cultures, stocks, and specimens of microorganisms or other potentially infectious materials and any lab equipment or waste coming in contact with these materials must be treated prior to disposal;

All regulated waste must be incinerated or decontaminated by autoclaving or steam sterilization and placed in a durable, leak-proof container that is closed before being removed from the work area.

The following table (Table 1) identifies the appropriate treatment methods for specific types of biological waste, which includes any other materials or equipment such as PPE contaminated with the biological material.

Table 1. Treatment methods for biological waste (based on Elizabeth City State University Laboratory Safety Manual 1/08/2018).

Biological Material	Treatment Method
Stocks and cultures of nonpathogenic materials and microorganisms (RG/BSL-1)	Autoclave or chemical treatment
Pathogenic microorganisms (RG/BSL ≥ 2)	Autoclave or chemical treatment
Human blood, blood components and products, and OPIM	Autoclave or chemical treatment
Animal or human primary cells, cell lines, and culture media	Autoclave or chemical treatment
rDNA waste, transgenic flies and plans	Autoclave or chemical treatment
Unfixed tissue, cells (and culture media), and fluids from humans or animals know to have been infected with human pathogens	Autoclave or chemical treatment
Animal carcasses, tissues, organs, or parts	None required; bagged followed by landfill disposal
BSL1 rDNA waste which is not in organisms or viruses	None required; bagged followed by landfill disposal

Glass and Sharps

- Used or contaminated needles, syringes, small bore pipettes, slides, lancets, scalpels, and razor blades are to be placed in a red sharps container.
- No part of a sharp may extend beyond the cap of the sharps container at any time.
- Seal sharps container when 75% full and notify your supervisor who will facilitate disposal.
- Place uncontaminated large bore pipettes, broken laboratory glass, and broken plastic into a small rigid cardboard box. Label the box "Broken Glass."
- Do not place unbroken glass into the broken glass container.
- Seal the box when 75% full and notify your supervisor who will facilitate disposal.

Appendix 1: Schedule of Training and Safety Events

Annually

Review Chemical Hygiene Plan (including SDSs)

Chemical inventory

Waste disposal

Fume hood maintenance

Review BBP Exposure Control Plan

Training for BBP

Review Radiation Safety Plan

Training for radiation safety

Semi Annually

Staff training

Hood monitoring

Monthly

Waste generator status check

Emergency shower flow check

Space inspection

Weekly

Emergency eyewash flush of plumbed eyewashes

As necessary

Waste determination

Appendix 2: Laboratory Chemical Hood Preventative Maintenance

Periodicity: Semiannually

Performed by: Trained Facilities Management person or qualified

contractor as outlined below or in equivalence to

the ASHRAE 110 standard

Materials:

1. Calibrated mechanical anemometer

- 2. Smoke candles (Regin Smoke Emitters, 90 Sec or equivalent)
- Matches/lighter
- 4. Watch glass method

Note: All hood testing will be done "as is" (i.e., during a routine day) and no additional challenges to the HVAC system will be added to perform the test. Operating hoods will be checked as they are operating, secured hoods will be started, checked, and then secured before checking another hood in the same laboratory area.

Laboratory Chemical Hoods

- A. If the hood is in use, request permission of the lab worker to perform testing. If an analysis is in progress, the smoke testing of the hood may be performed at a later date.
- B. Place the sash at a comfortable operating level, normally 6–8 inches from the full-open position.

Smoke Visualization and Face Velocity Test:

- C. Light a smoke candle in a watch glass.
- D. Sweep the candle under the front airfoil to ensure that the direction of airflow is into the hood.
- E. Sweep the candle over the face of the hood to ensure that the direction of flow is "into" the hood at all points.
- F. Sweep the candle over the face area approximately 6 inches inside the hood.
- G. Place the candle in the back third of the hood, approximately in the center. Observe from an angle flush with the face that no smoke is exiting the hood. Ensure any smoke vortexes are not exiting the hood

into the operating space.

NOTE: Any smoke that exits the hood is cause for failure. All work in the hood will be stopped, the hood secured, and the fan motors tagged out. A "Do Not Use" sign will be placed on the hood. Maintenance will be scheduled as soon as possible.

- H. Set up an imaginary grid of 12, approximately equally spaced areas on the face of the hood as noted in Figure 2. Take face velocity measurements in the center of each of these grids. Ensure the probe of the anemometer normal to the face and at the face, not inside the hood.
- I. Record the average velocity measurement for each grid area on the sheet.
- J. Calculate the average velocity. Record the average, highest and lowest measured velocities on the sheet.
- K. Adjust sash height to obtain approximately 100 fpm average. Note this height on the sheet. Return the sash to the original test height of step C above.

NOTE: Average face velocities less than 80 fpm or greater than 120 fpm should be monitored closely, and maintenance performed on the laboratory airflow system to achieve a better and more economical air balance. The hood may still be used if the smoke visualization test was satisfactory.

Figure 2. Hood Form

Face velocity meas	urements, ft/min:			
Hood Identifier:			Date/Time:	
Instrument Make/Mo	odel		Serial #	
Calibration Date: _				
Smoke containment	t Satisfactory Uns	atisfactor	ry (circle one)	
Average Face veloc	sity:	_ft./min	at sash height	ofin.
Highest grid velocity	y	Lowest	grid velocity	
Manometer reading	:in. H20	Monitor	reading	ft.
/min Checked by:(initial)				
Unusual conditions/comments: (Indicate areas on grid that are partially blocked (25–50%) or Blocked (>50%)				

Appendix 3 - Peroxide Testing Process

There is a variety of compounds that may form potentially explosive peroxides over time due to oxidation processes. It is important that these chemicals be tested periodically and be treated to remove any peroxides that are present.

Danger of explosion dramatically increases if solvents are allowed to concentrate or allowed to evaporate entirely. Therefore, compounds that are highly susceptible to peroxide formation should always be tested before distillation.

I. Potential Peroxide-Forming Compounds

A. Peroxide Hazard on Storage (*Test after three months*) — Chemicals that may form explosive levels of peroxides without being concentrated by evaporation or distillation. These materials can be hazardous even if never opened.

Compound Name	Synonyms	CAS#	In Inventory?
1,3-Butadiene		590-19-2	No
Chloroprene	Chlorobutadiene	126-99-8	No
Diethyl ether	Ethyl ether, ether	60-29-7	Yes
Divinylacetylene		821-08-9	No
Isopropyl ether	Diisopropyl ether	108-20-3	No
Potassium amide		17242-52-3	No
Potassium metal (Avoid aqueous test solutions)		7440-09-7	Yes
Sodium amide		7782-92-5	Yes
Tetrafluoroethylene		116-14-3	No
Vinyl ether	Divinyl ether	109-93-3	No
Vinylidene chloride	1,1-dichloroethylene	75-35-4	Yes

B. Peroxide Hazard on Concentration or Storage (*Test after 12 months*) — Chemicals that form explosive levels of peroxides on concentration or storage. They have the potential to become more hazardous after opening.

Compound Name	Synonyms	CAS#	In Inventory?
Acetaldehyde diethyl acetal	Acetal	105-57-7	
Acetaldehyde		75-07-0	Yes
Amyl alcohol (iso)	3-methyl-1-butanol	123-51-3	Yes
Benzyl alcohol		100-51-6	Yes
2-butanol	Sec-butanol	15892-23-6	Yes
Chlorofluoroethylene	chlorotrifluoroethylene	79-38-9	No
Collodion, flexible		None	No
Cumene	isopropylbenzene	98-82-8	Yes
Cyclohexanol		108-93-0	Yes
Cyclohexanone		108-94-1	Yes
Cyclohexene		110-83-8	Yes
2-cyclohexen-1-ol		822-67-3	No
Cyclopentene		142-29-0	No
Decahydronaphthalene	Decalin	91-17-8	Yes
Diacetylene	1,3-butadiyne	460-12-8	No
Dicyclopentadiene		77-73-6	Yes
Diethyl ether	Ethyl ether, ether	60-29-7	Yes
1,4-Dioxane		123-91-1	Yes
Ethylene glycol dimethyl ether	Diglyme	110-71-4	Yes
Ethyl benzene		100-41-4	Yes

Diethylene Glycol Mono Ethyl Ether Acetate		112-15-2	Yes
Ethylene Glycol Diacetate		111-55-7	Yes
Ethylene glycol ether acetates			
Ethylene Glycol Dimethacrylate		97-90-5	Yes
Ethylene Glycol Ethyl Ether Acetate		111-15-9	Yes
Furan		110-00-9	Yes
3-Heptanol		589-82-2	No
4-Heptanol		589-55-9	No
Hexanal		66-25-1	No
2-hexanol		626-93-7	Yes
Methyl acetylene	propyne	74-99-7	No
3-Methyl-2-butanone	Methyl isopropyl ketone	563-80-4	Yes
Methylcyclopentane		96-37-7	No
Methyl isobutyl ketone	4-Methylpentan-2-one	108-10-1	Yes
4-Methyl-2-pentanol		108-11-2	Yes
2-Methyl-2-propanol	t-butanol	75-65-0	Yes
2-Octanol		4128-31-8	Yes
2-pentanone		107-87-9	Yes
3-pentanone		96-22-0	Yes
2-pentanol		6032-29-7	Yes
1-pentene		109-67-1	No
4-penten-1-ol		821-09-0	No

1-Phenylethanol		98-85-1	No
2-Phenylethanol	Phenethyl Alcohol	60-12-8	Yes
2-Propanol		67-63-0	Yes
1-octene		111-66-0	Yes
Tetrahydrofuran		109-99-9	Yes
1,2,3,4-Tetrahydronaphthalen e		119-64-2	Yes
Vinyl ethers			No
Vinyl acetate		108-05-4	Yes
Vinyl chloride		75-01-4	No
2-Vinylpyridine		100-69-6	No
4-vinylpyridine		100-43-6	No
Other Secondary Alcohols			

C. Hazardous to Peroxide Initiation of Polymerization (*Test after 12 Months*) — Chemicals that may autopolymerize as a result of peroxide accumulation. These materials are typically stored with polymerization inhibitors to prevent these reactions.

Compound Name	Synonyms	CAS#	In Inventory?
Acrylic acid		79-10-7	No
Acrylonitrile		107-13-1	Yes
1,3-Butadiene		106-99-0	No
Buten-3-yne	Vinyl acetylene	689-97-4	No

Chlorobutadiene Chloroprene 126-99-8 No	
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Chlorotrifluoroethylene		79-38-9	No
Fluorene	Dibenzocyclopentadiene	86-73-7	Yes
9,10-Dihydroanthracene		613-31-0	No
Indene		95-13-6	Yes
Methyl methacrylate		80-62-6	Yes
Styrene		100-42-5	Yes
Tetrafluoroethylene		116-14-3	No
Vinyl acetate		108-05-4	Yes
Vinylidene chloride		75-35-4	Yes
Vinyl chloride		75-01-4	No
2-Vinylpyridine		100-69-6	No
4-Vinylpyridine		100-43-6	No

Peroxide Detection Methods

Method 1 — Commercially available test strips

These strips can be purchased from most safety or laboratory suppliers and they are very easy to use. Simply dip one of the strips in the suspect material, remove it, and compare the color on the strip to the calibration chart that comes with the test kit. This gives a quantitative peroxide <u>concentration</u>, usually in ppm. **Caution**: these strips have finite ranges. You may need to buy several different test kits to cover all possible ranges, or dilute samples over-range. **Quantofix® Peroxide Test Strips (Sigma-Aldrich Part # 37206)**

It is possible to test for organic and inorganic hydroperoxides with some test strips. To test for hydroperoxides in organic solvents the test zone is wetted with one drop of water after evaporation of the solvent.

Method 2 - Potassium iodide indicator

Prepare a **fresh** solution of 10% (by weight) potassium iodide, (KI), in distilled water (for some chemicals, acetic acid is a better choice). Add 1 ml of this solution to approx. 10 ml of the test material in a clear vial or test tube, shake well and wait about 30 seconds for color changes to occur. Peroxides will oxidize the colorless iodide anion, I⁻, to elemental iodine, I₂, which gives purple or brownish solutions depending on the solvent. Purple, brown or purple-brownish colors indicate relatively high concentrations of peroxides and yellowish colors indicate low concentrations.

Method 3 - Starch-iodide indicator.

This procedure is identical to the KI test shown above, but shows color changes more easily because any iodine that is generated combines with added starch to form an intensely deep blue <u>solution</u>. Perform the test as indicated above, but add 1 drop of a <u>saturated solution</u> of starch in water. A strong blue color indicates peroxides.

II. Treatment of Peroxide-Containing Solvents and Solutions

Method 1 — Activated Alumina

Peroxides can be removed by passing the solvent through a short column of activated alumina. This method is effective for both water-insoluble and water-soluble solvents (except low molecular weight alcohols). Since this method does not destroy peroxides the alumina should be flushed with a dilute acid solution of potassium iodide or ferrous sulfate following treatment to remove peroxides from the alumina.

This method appears to be rather inefficient and costly. A test case done at Valparaiso University with 4-year-old THF showed that only 400 mL of THF could be treated per 100 grams of alumina.

Method 2 — Ferrous Salt

Peroxide impurities in water-soluble solvents are easily removed by gently shaking with a concentrated solution of a ferrous salt. A frequently used ferrous salt solution can be prepared either from 60 g of ferrous sulfate + 6 ml concentrated sulfuric acid + 110 ml water; or from 100 g of ferrous sulfate + 42 ml of concentrated hydrochloric acid + 85 ml of water.

Method 3 — Indicating activated Type 4A molecular sieves

In a 1982 article (*J. Org. Chem.* **1982**, *47*, 3821-3824), David Burfield outlines a method of peroxide removal using indicating activated molecular sieves. The article reports this method is effective with ether solvents. The solvent should first be deoxygenated by bubbling with nitrogen for about five minutes. Then, 5% w/v of indicating molecular sieves are added. The solvent is then capped tightly and stored as normal. The peroxides are gradually destroyed within a day or so. This

procedure does not destroy unreactive peroxide species such as dialkyl peroxides. *Indicating activated Type 4A molecular sieves J.T. Baker Product #2707-01 500g, \$105.*