

Analytical Chemistry Laboratory Guide

Department of Biological and Agricultural Engineering
University of California, Davis

1. LABORATORY SAFETY

1.1 Safety Information

1.1.1 **Safety Net Publication:** UCD Environmental Health and Safety (EH&S) publishes safety information sheets called Safety Nets which cover a variety of general safety and laboratory safety topics. Copies are kept in a file in a binder located in the Chemistry lab 1322 Bainer Hall. Review the table of contents of the SafetyNet binder and read the SafetyNet Sheets which may be applicable to your specific activities. References are made throughout this manual to specific SafetyNet publications which provide additional information on specific topics. Review the following Safety Net publications related to general laboratory safety:

1) #19 - General Safety Guidelines for Chemical Lab

2) #40 - Health and Safety Hazards: A Student's Right-to-know

1.1.2 **Material Safety Data Sheets:** Know the hazard potential of each chemical you are using. Familiarize yourself with the Material Safety Data Sheets (MSDS) for the materials you are using. The MSDS binders are located in the chemistry lab in 1322 Bainer.

1.2 Personal Protection and First Aid

1.2.1 Wear eye protection (safety glasses or goggles) whenever you are working with material that can injure your eyes, especially acids, bases, and oxidizers.

1.2.2 If you wear contact lenses, familiarize yourself with the special precautions listed in **Safety Net #5**.

1.2.3 Know the location of the emergency eye-wash stations, showers and first aid kits. The eye wash stations and showers are located in the chem lab, shop, and room 1340. Familiarize yourself with the operation of the eye wash station and shower. First aid kit is located in the chemistry lab.

1.2.4 Wear hand protection when handling corrosive or hazardous materials. The use of nitrile rubber is recommended when handling concentrated corrosive materials or organic solvents. Disposable PVC examining gloves offer minimal protection and are permeable to many organic substances. Check rubber gloves for holes and cracks before using. Do not touch surfaces with contaminated gloves. (door knobs, equipment bench tops, drawers, etc.) Rinse gloves well after use. Refer to **Safety Net #50** for guidance on selection of chemical-resistant gloves.

- 1.2.5 Open-toed shoes or sandals are not permitted in the chemistry labs.
- 1.2.6 Use of lab coats is recommended to protect your clothing. Protective aprons should be worn when handling highly corrosive materials.

1.3 Chemical Emergencies

In a chemical emergency do not hesitate to follow these procedures because a spill or contact seems too trivial. It is better to overreact!

- 1.3.1 For skin contact, flood the affected area with water immediately and continues flooding for at least 15 minutes. If a substantial portion of the body is involved, use a safety shower. If the chemical is toxic, or if its toxic properties are unknown, call Emergency Medical Services at **(911)** and Environmental Health and Safety (EH&S) at **(2-1493)** in that order.
- 1.3.2 For eye contact, flood eyes with water and continue flooding for at least 15 minutes. Remove contact lenses if possible, or move to corner of the eye. Immediately call Emergency Medical Services **(911)** and EH&S **(2-1493)** in that order.
- 1.3.3 For inhalation or ingestion, follow direction on the product label or MSDS. Call Emergency Medical Services **(911)** and EH&S **(2-1493)** immediately. Refer to **Safety Net #52** for additional information on emergency medical care.
- 1.3.4 In case of spillage of any chemical:
 - 1. If the volume of spill is greater than 500 ml (1 pint), or any amount of extremely toxic substance is spilled, evacuate and seal the area. Call the Fire Department **(911)** and EH&S **(2-1493)** in that order. If you suspect or know the substance is flammable, extinguish or remove all ignition sources before sealing the room.
 - 2. If the volume of spill is less than 500 ml (1 pint) and the substance is not extremely toxic, check the container or MSDS for special instructions. If no instructions are immediately available, encircle and cover the spill with absorbent material until the liquid is adsorbed. Do not flush with water. Neutralize strong acids with sodium bicarbonate, sodium carbonate, or calcium hydroxide after absorbing.
(Note: DO NOT absorb hydrofluoric acid- neutralize immediately).
 - 3. For formaldehyde and mercury spills see **SafetyNets #11 and #16**, respectively.
 - 4. Solid spills are not usually emergencies. If the spilled material is toxic, use damp cloths or paper towels to transfer the material to plastic bags. Sweeping or brushing dry material may cause airborne dust.
 - 5. All absorbed spill material must be transferred into double plastic bags, sealed and disposed of as a hazardous waste (see section 1.4.4). Notify lab supervisor, call, and hold for pickup by EH&S S (2-1493).

1.4 Chemical use and storage

- 1.4.1 General
- 1.4.1.1 A list of chemical stocked in the labs is located in the chem lab MSDS shelf
 - 1.4.1.2 Dry chemicals are stored alphabetically in room 1322
 - 1.4.1.3 Solvents are stored in room 1324 in storage cabinet.
 - 1.4.1.4 Pesticides are stored in room 1324 in pesticide cabinet.
 - 1.4.1.5 When taking chemicals from reagent bottles, pour out slightly more than the amount of chemical needed into a clean beaker or weighing boat, then transfer chemical to the balance. Measure the amount of chemical required and flush the rest down the sink with plenty of water, if it is not a hazardous material that requires storage and pickup. NEVER POUR A CHEMICAL BACK INTO A REAGENT BOTTLE, AND ALWAYS USE A CLEAN SPOON/SPATULA. Secure lids tightly and return reagent bottles to proper storage area.
 - 1.4.1.6 Chemical solutions should be stored in reagent bottles that are properly sealed and labeled. A proper lab is one made with water proof label tape and marking pen and marked with the first initial and last name of the owner, the exact contents, the date of preparation, and expiration date, if applicable.
 - 1.4.1.7 Chemical solutions should not be stored in volumetric flasks.
 - 1.4.1.8 Do not store chemicals on the lab floor. Return chemicals to designated storage areas.
 - 1.4.1.9 Do not mouth-pipette chemical solutions.
 - 1.4.1.10 Never place food or drinks, including coffee Cups, on chemical storage shelves. Never store food for human consumption in a refrigerator with any samples or chemicals.
 - 1.4.1.11 Refer to **Safety Net #42** - Guideline for Chemical Storage
- 1.4.2 Corrosive Chemical - Acids, Bases, Oxidizers
- 1.4.2.1 Prepare acid and base solutions by adding concentrated acids or bases to water. Water added to strong acids or bases can result in explosions.
 - 1.4.2.2 Always wear eye and hand protection and protective clothing when working with corrosive liquids.
 - 1.4.2.3 When working with strong acid solutions or base on lab benches use protective sheeting. Clean up spills immediately.
 - 1.4.2.4 Work with and store corrosive liquid solutions away from laboratory instruments.
 - 1.4.2.5 When working with strong acids that emit vapor, (e.g. HCl, HClO₄, HNO₃) always work under the fume hood.
 - 1.4.2.6 Store acid and base solutions separately.

- 1.4.2.7 Store corrosives and strong oxidizers such as nitric acid, chromic acid, peroxides separately from solvents, organics, and organic acids.
- 1.4.2.8 "Look before you handle". Bottle of corrosive liquids with crusts, powder deposits, crystallization around the cap, discoloration, etc. are indications of contamination. Handle such bottles with extreme caution and notify lab supervisor that these conditions exist.
- 1.4.2.9 Keep containers or corrosive liquids well sealed.
- 1.4.2.10 Do not use ground glass stoppers or auto dispensers with basic solutions. Use only rubber or teflon stoppers. Ground glass surfaces will be fused together when exposed to bases over long periods.
- 1.4.2.11 Store solutions of inorganic hydroxides in polyethylene containers.
- 1.4.2.12 Do not use metal plunger digital or transfer pipetters with HCl or HClO₄ solutions. Acid fumes will corrode stainless steel plungers. Use only glass or teflon plunger pipettes.
- 1.4.3 Organic Chemicals -Flammables, Volatiles, Toxics
- 1.4.3.1 Store flammable liquids in labeled "flammable" storage cabinets.
- 1.4.3.2 If it is not possible to store flammable liquids in approved flammable storage cabinets, not more than 10 gallons of flammable liquids in original containers can be stored in any single laboratory. Note: Calculation of quantity is based on volume of container, not quantity of flammable liquid in container (if there is only 1 drop in a one gallon jug, it is counted as a full gallon).
- 1.4.3.3 Always work under the fume hood with volatile organic chemicals.
- 1.4.3.4 Wear proper hand, eye and clothing protection when working with toxic substances. Certain volatile substances may require the use of respirators. Read labels for protection requirements. Check with EH&S if you are uncertain.
- 1.4.4 Waste Chemicals
- 1.4.4.1 The following categories of waste chemicals are hazardous and should be stored for pickup by EH&S:
- Flammable
 - Corrosive pH<4; pH>10
 - Explosive
 - Oxidizer
 - Toxic
 - Solutions of heavy metals > 100 ppm
 - Cyanides or sulfides > 1000 ppm
 - Chlorinated solvent > 1000 ppm
 - Air or water reactive
 - Dye solutions

Odorous materials
Unknown chemical or mixtures

Before generating any chemical waste, see the lab supervisor to verify proper labeling and storage procedures. Place liquid wastes in a 1 gallon glass or plastic bottles with caps that can be tightly sealed. If you reuse reagent bottles for waste storage, be sure to remove original labels completely from containers. Label each bottle with a Hazardous Waste label obtained from the lab supervisor. Fill-out the label completely indicating exact contents. Do not mix unknown or incompatible chemicals.

If waste storage bottle are full or you will no longer be generating the waste, coordinate waste pickup by EH&S through the lab supervisor.

1.4.4.2 Waste acid or base solutions not contaminated with metals or organics may be disposed of by carefully diluting small amounts with water, neutralizing, and flushing down the drain with large quantities of water.

1.4.4.3 Refer to **Safety Net #43** - Identification and Segregation of Chemical Waste.

1.4.5 Fume Hoods

Keep bench surfaces under hoods clear of chemicals and glassware. Do not store chemical solutions in hoods unless advised to do so by the lab supervisor.

1.4.6 Gas Cylinders

1.4.6.1 Gas cylinders whether in storage or in use and whether full or empty are to be restrained in an upright position using approved holders or harness. When cylinders are not in use cylinder caps must be in place.

1.4.6.2 Cylinders are ordered through the University Storehouse and delivered to the shop.

1.4.6.3 Empty cylinders are to be labeled "Empty" with labeling tape and ordered to be picked up as soon as possible.

1.4.6.4 Move cylinders with a cylinder with a hand truck with restraining chain. Remove regulators and replace valve cap before moving cylinders.

1.4.6.5 All gas cylinder orders should be placed through the lab supervisor or the P.I. Order the minimum necessary quantities of gases to minimize demurrage fees.

1.4.6.6 If leaks are suspected, use soap solutions are similar commercial products to locate the problem.

1.4.6.7 Never use oil or grease on any hose or fitting carrying compressed gas. Explosion danger exists. Never use a gas regulator marked "use no oil" to regulate any gas except those known to be water pumped. Explosion danger exists.

1.4.6.8 Never use a gas regulator that has been used to regulate an oil pumped gas (e.g. compressed air) to regulate oxygen. Explosion danger exists.

- 1.4.6.9 Never expose copper to acetylene gas. Acetylene gas and copper form copper acetylide (Cu_2C_2) - a powerful, erratic explosive.
- 1.4.6.10 Never turn a gas regulator valve clockwise as far as it will go because the diaphragm can be punctured. Pressure regulating valves are closed when the handle is turned counterclockwise until it appears to be free. In general, leave the gas regulator valves set in its operating position at all times, even when the gas is not in use. When finished with the gas, close the main cylinder valve by turning the round handle clockwise until it stops.
- 1.4.7.11 Refer to **Safety Net #60** Compressed Gas Safety

2. **LABORATORY MAINTENANCE**

A messy laboratory is unsafe and indicative of poor laboratory practice and suspect experimental results. Maintenance of the lab is both a collective and an individual responsibility. To encourage each individual to maintain the lab, the following policies and procedures have been instituted.

- 2.1 Glassware
 - 2.1.1 Each person is responsible for washing and rinsing their own glassware. Glassware should be rinsed with tap water followed deionized & distilled water (dd water) and placed in drying racks. Conserve dd water by rinsing with a swiftly flowing small stream from the dd water jug. Please refill the dd water jug if you are the last person to use it. Then return clean glassware to storage areas.
 - 2.1.2 If glassware required acid washing follow the guideline for acid washing in **Safety Net #44** and/or see the lab supervisor.
 - 2.1.3 Glassware to be used on a continuing or repetitive basis my be reserved by labeling with last name and first initial using labeling tape and waterproof marker. Such dedicated glassware must be stored in a designated work/ storage area, not in drying racks.
 - 2.1.4 Dirty reusable glass pipettes should be rinsed with tap water and placed tip up in the pipette wash container. The lab manager will wash dirty pipettes.
 - 2.1.5 A selection of clean, reusable glass pipettes are in the chem lab. Do not place dirty pipettes back in drawers.
 - 2.1.6 Broken glassware must be discarded in the "sharps" container/bucket.
- 2.2 Benches and Counters
 - 2.2.1 Keep bench tops clear of waste paper, dirty glassware, sample bottles, gloves, lab coats, stock chemicals, tools, etc. Return all items to proper storage immediately after use.

- 2.2.2 Sponge off any excess water on bench tops. Do not allow water to drip off bench tops to drawers or cabinets below.
- 2.2.3 Immediately clean up any minor spills of dry or liquid chemicals on bench tops. If the spilled material is hazardous, follow the procedures described in Sec 1.3.4
- 2.2.4 When working with corrosive chemicals on bench tops use absorbent sheets to protect the bench top. Dispose of sheets or wash off clean sheets for reuse.
- 2.2.5 Do not tape off sections of bench top to reserve work space. If it is necessary to tape off bench tops, do not leave tape on for long periods as tapes becomes difficult to remove.
- 2.3 Floors
 - 2.3.1 Immediately clean up any spill, including water, and any broken glass. If spilled material is hazardous follow procedures in Sect 1.3.4 A broom and dust pan is available in the chem lab.
 - 2.3.2 Do not store chemical or instruments on floors.
 - 2.3.3 Do not leave empty boxes or packaging materials on the floor. Throw away item in dumpsters. If you want to save the boxes, contact the lab supervisor/ P.I. for storage location.
- 2.4 Sinks and Drains
 - 2.4.1 Do not flush any hazardous waste materials down the sink drains. (see sect. 1.4.4.)
 - 2.4.2 Do not pour any waste solids in the sinks that may clog drains.
 - 2.4.3 Do not remove strainers from sink drains. Clean the strainers as necessary to maintain good drainage.
- 2.5 Balances
 - 2.5.1 Immediately clean up any spills in or around the balances. (See Sect. 1.3.4) Sponge or sweep off any residual chemical dust from around balances.
 - 2.5.2 Return all chemical bottle to proper storage area after weighing out chemicals
 - 2.5.3 Clean spatulas before returning them to storage bins. Discard any waste materials (weigh boats, papers, etc.)
 - 2.5.4 Turn balances off after use.
 - 2.5.5 Keep storage area for weighing boats closed to prevent contamination.
- 2.6 Refrigerators
 - 2.6.1 Immediately clean up any spills in refrigerators.

3. **LABORATORY EQUIPMENT**

- 3.1 All major and most minor equipment items have been assigned a BAE property number. The locations of all such equipment item are indicated in the lab directory
- 3.2 Equipment items with property numbers removed from normal storage location for use must be signed out on the equipment sign out sheet in the chem lab.
- 3.3 Operating and instruction manuals for equipment are filed in room 1322A in the chem lab. See lab supervisor if you want to copy or borrow.
- 3.4 Do not operate equipment or instruments if you are not familiar with their operation. Read instruction manuals carefully or consult lab supervisor before using equipment.
- 3.5 Routine maintenance of equipment is the responsibility of the user. Perform maintenance at required intervals to maintain proper operation.
- 3.6 If you experience operating problems and are unable to repair the equipment following manual instructions, notify the lab supervisor.
- 3.7 When using macro or micro pipettors care must be taken to maintain the integrity of the piston seal. Never allow liquid to enter the shaft where it can come into contact with the piston or seal. Whenever liquid is present in the tip, the instrument should be held vertically. Never invert the instrument or lay it on its side with liquid in the tip. Do not use pipettors with chrome plated plunger to pipette vaporous acids (HCl, HClO₄). If metal plunger pipettors are inadvertently used with acids, they should be dismantled and cleaned immediately after use.

4. **TOOLS**

Tools can be checked out from the shop Return all tools immediately after use. Failure to comply with proper use and return of tools will result in mental and physical abuse by Jim and his crew!

5. **SUPPLIES AND ORDERING**

- 5.1 The location of general lab supplies are indicated in the lab directory.
- 5.2 Notify lab supervisor when stock of lab supplies runs low.
- 5.3 Place all orders for lab supplies, chemicals, and gas cylinders at least 1-2 weeks in advance.

6. **LAB ACCESS**

Doors to all chem lab rooms should be kept closed at all times. Do not prop open door. Lock doors after normal working hours or when leaving a vacant lab.

Acknowledgments: This user guide and chemical hygiene plan was originally developed for the Department of Civil and Environmental Engineering, Environmental Engineering Laboratory. 10/9/95 It has been revised 4/15/97 for the department of Biological and Agricultural Engineering, Analytical Chemistry Laboratory.