Department of
Civil & Environmental
Engineering

University of Massachusetts

Environmental Engineering Research Laboratory

Rules and Protocols

College of Engineering
University of Massachusetts

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CONTENTS

A. LABORATORY SAFETY and SECURITY

1. General Rules

Students should be aware that all research laboratories present health hazards. The University has published a safety manual which can be obtained from the UMass Environmental Health and Safety Office (EH&S) or from their website: http://www.ehs.umass.edu/lhs.html. There are several copies of this manual in the EWRE laboratory. In addition, we would like to stress some of our own protocols and regulations that build upon the EH&S guidelines. Before entering the laboratory, the following rules must be reviewed and understood:

1. Animals are not permitted in the Environmental Engineering Laboratories.

2. Walkways should be kept clear of obstructions (e.g., bicycles). Personal items should only be stored in office areas.

3. Eating, drinking and smoking are not permitted in the laboratory areas. Eating and drinking are only permitted in designated areas.

4. Mouth pipetting is forbidden; always use a pipetting bulb or a mechanical pipeter.

5. Avoid very loosely-fitted clothing. A white lab coat is recommended. Always wear closed-toed shoes. Sandals are not permitted for lab work. Clothing must cover the lower leg. Shorts or short skirts are not permitted. Safety glasses are recommended. Long hair should be pinned back.

6. Never work alone in the laboratory. If you must work after hours, be sure that someone else is in the lab area who can provide emergency assistance.

7. Volatile reagents or chemicals likely to emit hazardous fumes should be kept in a vented chemical cabinet or an explosion-proof refrigerator, and used only in an externally-vented fume hood.

8. Never work with a volatile solvent near an open flame.

9. Never leave flames unattended.

10. Promptly clean up all chemical spills. Elab II cabinetry and equipment have sustained substantial damage due to lack of attention to spills. Please clean your equipment and work spaces after each use/day even if you believe there hasn't been a spill. If a spill involves significant amounts of acids or bases, it should be first neutralized with powdered sodium bicarbonate.

11. Always read precautions on reagent bottles before opening. Refer to material safety data sheets (MSDS) when precautions are unclear. MSDS are located in a file cabinet in room 301 and organized by alphabetical order of chemical name. They can also be accessed from the EH&S website at: http://www.ehs.umass.edu/resource.html .

12. Gas cylinders should always be secured in one of the tank "farms". In a few cases, they may be strapped to a bench. Always remove regulator and replace cap before moving a gas cylinder.

13. All chemicals, solutions, samples, etc. that are to be stored for more than a few hours must bear a stick-on label which includes the following information: identity of sample, solution, etc.; date; name of researcher; special precautions, if any. Only numbered BOD bottles and autoinjector vials are exempt.

14. Wear goggles, lab coat, and gloves when mixing strong acids and bases. Any concentrated acids should be used under a hood. Use only borosilicate (e.g., pyrex, kimax) glass bottles. High sodium glass will crack under the heat of dissolution of strong acids and bases. Always add the acid or base slowly to water; never the reverse.

15. Students who regularly work with raw or treated wastewater (biosolids) must be up-to-date with the appropriate vaccinations. The EWRE Laboratory Safety Coordinator has a list of recommended and required vaccinations offered by University Health Services.

16. Basic hygiene precautions and PPE are important for students who handle biosolids. Best practice includes hand washing with soap & water both before and after contact with biosolids. Wear lab coat, goggles and gloves when working with biosolids.

2. Emergency

You should also become familiar with the laboratory shower and eye-wash units, fire extinguishers and first aid kits. There are combination shower and eye-wash stations in each lab room, and several in the larger rooms with multiple bays. If you believe you may have had limited skin contact with a chemical or solution, immediately wash with soap and water. More extensive contact may require use of the shower. If you splash something in your eyes, please make use of the eye wash station.

Safety-related items are located as follows:

a. Shower & Eye Wash
One to three in each room

b. First Aid Kit

one per lab, centrally located near telephone

c. Material Safety Data Sheets
i. Room 301E in file cabinet

3. Disposal of Chemicals

a. What chemical wastes are hazardous or non-hazardous?

Nearly all chemicals are considered hazardous, and NONE should be disposed as normal trash. Only in a few cases can chemicals be released down the sink or disposed of with special trash (e.g., glass sharps). Students should exercise common sense, and only flush small quantities of non-corrosive (e.g. neutral pH), water-soluble non-hazardous salts (e.g., sodium chloride, potassium sulfate). Anything flushed down the sink drains should be done with copious amounts of water.

b. Disposal of hazardous wastes

Solid or liquid hazardous waste should be placed in an empty, clean compatible glass or plastic container with screw cap. Each bottle or container should be properly identified with a completely filled out “Hazardous Waste” label. You must be specific regarding contents, using the full chemical name NOT the chemical formula. Extra labels are available in the file folder near the telephone in all labs. Containers should be kept closed, however caps should not be tight if there is any possibility of high vapor pressure. Whenever possible, separate non-halogenated from halogenated compounds. Identify using either the "Red" Non-Halogenated Organic Solvent or "Yellow" Halogenated Organic Solvent label also located in the file folder near the telephone.

Each hazardous waste container should be placed in a secondary container (usually a gray bin) labeled with a bright orange "Hazardous waste" label. ***It is essential that hazardous waste labels bear all requested information***. Avoid keeping more than one container with the same waste in the same gray bin or anything that is not hazardous waste (i.e., glassware, water bottles, pipettes, etc.) in these bins.

Any unknown, old or unwanted chemicals found in the laboratory should be reported immediately to the Laboratory Safety Coordinator.

Be aware that many instruments (e.g. ICP, HPLC) use organic solvents, halogenated organic compounds or other chemicals that must be disposed of as hazardous waste. Check with the person in charge of the instrument about what to do and where or how to collect the waste from the instrument.

Pickup of hazardous waste can be arranged on the CEMS website by going to:
www.umass.cems.sr.unh.edu/CEMS/RequestRemoval

Once again, you will need to know all of the chemical constituents in the containers, and estimate approximate concentrations in order to fill out this on-line form.

4. Fume Hoods

Fume hoods are located in all of the laboratories in the 2nd and 3rd floors of Elab II. Their performance specifications are regularly checked by EH&S as indicated by one or more stickers on the sash. Nearly all of the fume hoods are half-height, although there is a full-height hood on Room 208 (bay A). University regulations regarding fume hoods can be found at: http://www.ehs.umass.edu/fume-hood.html .

Below are some of the 3rd floor fume hoods that are largely devoted to a single use:

- 301 B(b) is for SPE extractions
- 301 D(a) is for DBP extractions
- 301 D(b) is for small-scale sample concentration
- 308 B(a) is for ozonation
- 308 B(b) is for chlorine gas dissolution and chlorine dioxide generation

It's very important that you clean your equipment and work spaces after each use/day even if you believe there hasn't been a spill. Cleaning should include the bench, sash, windows & apron and floor area near the fume hood.

5. Security

All laboratory users should have keys to the 2nd and 3rd floor laboratory doors (note that 301 has a unique key). Please consult your advisor if you do not. You may have to unlock these doors in the morning. Please be certain to lock all doors in the evening before you leave. Normally our protocol is for each lab user to lock his/her room at the end of the day, and certainly they should all be locked after 5 PM, if no-one else is using the room. Please be aware of non-EWRE personnel in the lab areas. If you believe they are not authorized to use the facilities, you should notify authorities.

B. LABORATORY MANAGEMENT

The Environmental Engineering laboratory is a complex and potentially dangerous environment. It is essential for reasons of safety and efficiency that there be a formal management structure. Cooperation is also essential for fostering a safe and efficient work environment. This lab organizational structure is outlined below. The various roles within this management structure have been assigned to many of the senior graduate students, post-docs, faculty and staff. These assignments are usually announced in a separate memo at the beginning of each semester. If you are uncertain about your own lab management assignments (if any), or the identity of some of the key management personnel, please contact your advisor or the EWRE Program Coordinator.

1. Faculty Laboratory Coordinators

These are one or more EWRE Faculty members who are assigned by the EWRE Program Coordinator to oversee the program's laboratories. These people work most closely with the Laboratory Management team.

2. Laboratory Managers

There is a group of staff/post-docs who have been assigned responsibility for much of the day-to-day function of the various zones in the EWRE labs[1]. Each is assigned to serve at least one of the seven zones in Elab II (see diagram on following page). The Laboratory Managers are available to answer questions regarding the location and availability of lab resources. These persons also oversee many general utility functions. Specific Laboratory Manager duties include:

a. Maintenance of high-purity water systems
   DI taps: check and report key WQ characteristics (e.g., TOC) in your area
   Other special water

b. Special Disposal
- waste chemicals & hazardous wastes
- maintain chemical inventory
- lab safety issues

c. Gases
- liaison between EWRE and delivery personnel
- inventory of tanks

d. Building Maintenance
- liaison between EWRE and physical plant
- oversee general lab cleanliness
- broken glass disposal

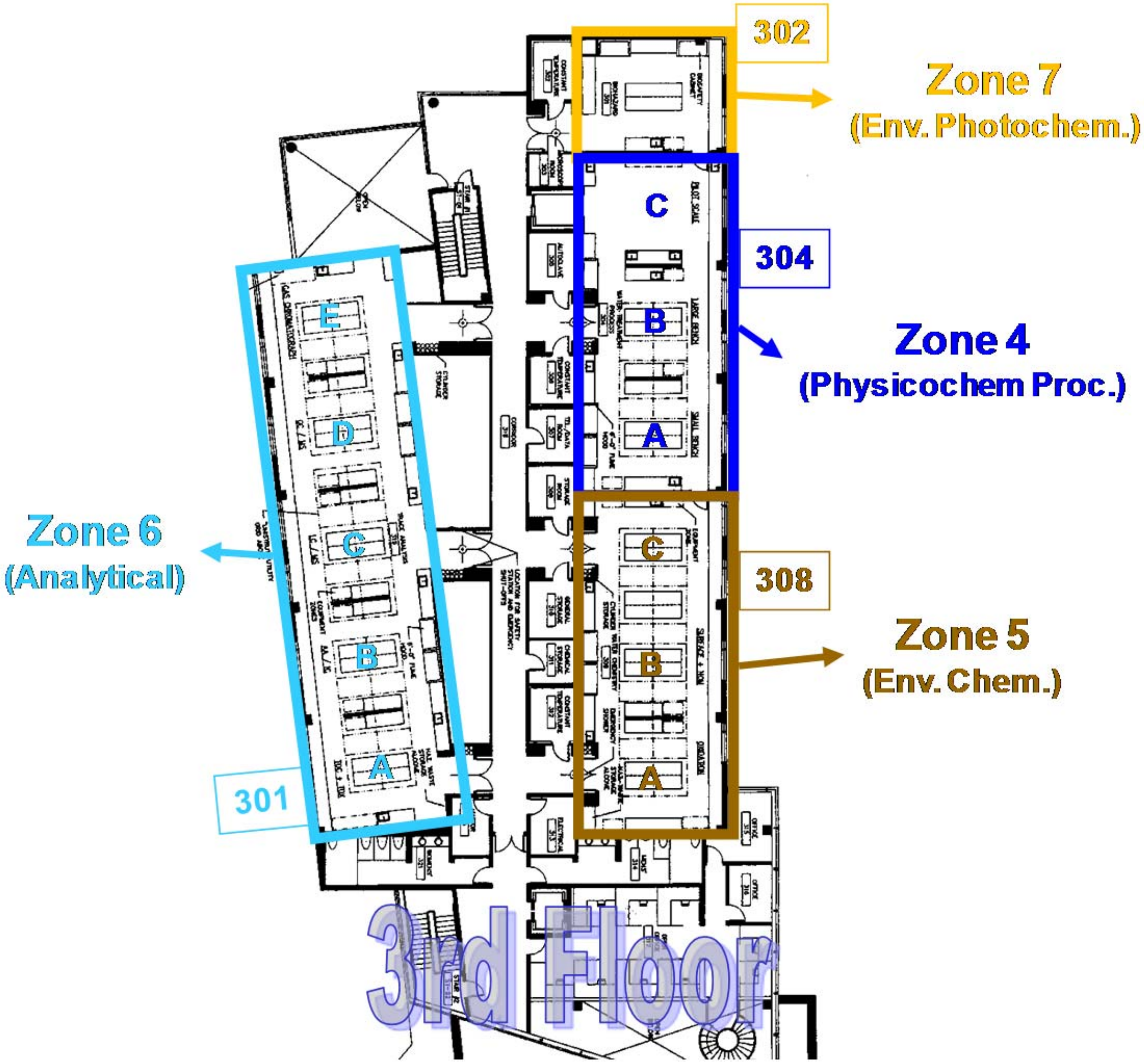
e. Miscellaneous
- facilitate ordering of general use supplies
- backup contact for delivery of packages if the primary recipient isn't available

f. Analytical Equipment
- oversee maintenance
- assurance of training and operation by students
- contact person for all analytical equipment issues

g. Laboratory Safety & Efficiency
- updating lab personnel about problems in laboratory
- updating any changes or scheduled events in the laboratory

[1] -These assignments are usually made on a semester or yearly basis


203
Zone 3
(Env. Micro.)
207
211
Zone 2
(Bioprocesses)
213
C
B
A
208
D
C
B
A
Zone 1
(Groundwater)
2nd floor



3. Equipment Managers[2]

These are EWRE students/staff (Ph.D. students; post-docs, staff; and second year MS students) who have been asked by the Faculty Lab Coordinator to help oversee the operation and maintenance of some specific piece of equipment. The duties are summarized in section E. INSTRUMENTS.

4. QC Officers[3]

These include faculty QC officers, and the QC managers they oversee. The QC Managers are EWRE students/staff (Ph.D. students; post-docs, staff; and second year MS students) who have been asked by the Faculty QC officer to help oversee quality control of a particular analytical method (e.g., THMs, HAAs, TOC). With single-use instruments (e.g., TOC and TOX analyzers), and with

[2] Assignment of equipment manager positions are typically made on a semester or AY basis via a separate memo.
[3] Assignment of QC officer positions are typically made on a semester or AY basis via a separate memo.

some other methods closely assigned to a particular instrument, the same person may serve as both QC manager and equipment manager. QC managers are responsible for the oversight and compilation of analytical quality control data (e.g., calibration methods, analyte recoveries, data precision and accuracy). They also make judgments on data acceptability, and consult with the faculty QC officer on remedial actions when data quality does not meet the stated objectives. The specific duties are summarized in the individual method SOPs that exist as separate documents, most of which can be accessed from:

http://www.ecs.umass.edu/eve/research/sop/index.html

5. Laboratory Users

All laboratory users (i.e., EWRE graduate students, technicians, post-docs, etc.) are expected to help with the laboratory management in a number of ways. For example, the Lab Managers or Faculty Lab Coordinators may ask for help with general lab clean-up and inventory from time to time. Lab users are expected to maintain their own assigned lab areas. They should also report any problems or hazards that are noticed in other areas (e.g., leaky plumbing, a chemical spill, broken glass).

C. PERSONAL LAB SUPPLIES

Students working on laboratory research will need to have in their possession certain lab supplies that should be reserved for their own use (i.e., not shared with other students). These items are generally purchased with research grant funds, and are stored either in the student's office or in his/her personal lab drawers. Some of the items that fall under this category are listed below. In particular, supplies listed under #2 and #3 should be stored apart from the general use supplies. Upon completion of your laboratory work at UMass, these supplies (#2 & #3) should be returned to your research advisor, so they may be re-assigned to an incoming student.

1. Required for all Students Conducting Laboratory Research
    a. Protective Lab Coat
    b. Lab Notebook
    c. Goggles

2. Recommended for all Students Conducting Laboratory Research
    a. Pipet bulb
    -
    -b. volumetric glassware
        i. pipets
        ii. volumetric flasks
        iii. graduated cylinders
        iv. burets
    -c.. chemically-resistant gloves

3. Required for students using certain instruments on a regular basis
    a. for those using pH/ISE Meters
        i. pH electrode, if this is the measurement of interest
        ii. filling solutions for the pH electrode
            & a container for storage of the electrode
    b. for those using UV-Vis Spectrophotometers
        i. a pair of 1-cm spectrophotometric cells with
            windows suitable for the wavelengths of interest
    c. for those using Gas Chromatographs
            (sometimes these are shared; e.g, DBP analysis)

i. one 10 μL syringe (unless you're only using GCs with autosamplers)
ii. additional syringes or volumetric ware for preparing standard solutions
iii. packed GC column or capillary column

Items under category 2, and 3a are available in limited supply for general use. If you find that you are using them on a regular basis, you should arrange to obtain your own.

D. LABWARE

1. Non-volumetric Glassware

Most non-volumetric glassware (beakers, flasks) is for general use. You may, however, wish to store frequently used glassware away from general use. Certain uses require unique cleaning procedures, and the repeated use of the same piece of glassware for the same test can lead to improved results.

2. Volumetric Glassware

Most volumetric glassware (pipets, volumetric flasks, graduated cylinders, burets) is dedicated to specific projects. However, there are some reserved for general use. Avoid using volumetric flasks for storage of solutions. This is an inefficient use of expensive glassware, and caustic solutions can permanently etch the glass.

Volumetric glassware is rated at 20^{o}C and should always be used at or near that temperature. This is necessary, because glass expands and contracts much less than water does, so that changes in temperature change the mass of water contained or delivered by volumetric glassware. Burets and pipets should be stored in drawers. Frequently used pipets may be kept upside down on the bench top in a specially designed pipet rack. Volumetric flasks should be stoppered or sealed with aluminum foil and stored upright in wall cabinets. Volumetric glassware should be dried at temperatures of 50°C or below. One or more low temperature drying ovens are maintained in Elab II for this purpose. In many cases, volumetric glassware need not be perfectly dry before re-use. Additional information on the cleaning of volumetric glassware can be found in part G.

3. Eppendorf Pipets and Automatic Pipeters

These are dedicated either to specific projects, or to specific uses. For example there has traditionally been a set of eppendorf pipets for use with DBP extraction and analysis. Automatic pipeters are used with the DPD-titrimetric method for chlorine, and the indigo method for ozone.

4. Reagent and Sample Bottles

Glass and plastic bottles are stored for general use in several locations in the lab area. These should be used for the storage of samples or reagent solutions. Be careful to choose a bottle that is compatible with the material being stored (e.g., acid-washed plastic for trace metal samples; glass for samples undergoing trace organic analysis). Pay special attention to the cap and liner. The liner should be sufficiently compressible to form a tight seal with the bottle's lip. It should also be made of a material that will not degrade upon contact with the liquid, nor should it contaminate the liquid. In general, cardboard liners should be avoided, and teflon is best. All reagent and sample bottles should be carefully washed after use and returned to their proper drawer.

5. Miscellaneous Laboratory Supplies

Most general lab supplies (e.g., gloves, labels, weighing dishes, parafilm, liquid soaps, kimwipes) are kept in drawers and cabinets in each main laboratory. These are available for use by students conducting laboratory research. However, students are recommended to keep a pair of gloves for their personal use. Less used supplies or extra (overflow) is stored in the locked room labeled “305 Storage”. This room also has a large number of coolers.

E. INSTRUMENTS

1. General

Management: Each year a group of graduate students (most are Ph.D. students and post-docs) will be assigned the duty of overseeing the use and maintenance of the program's major instruments. These "Instrument Managers" will keep the laboratory managers informed of any problems with their instruments and related supplies. Specifically, the Instrument Managers will:

1. Take responsibility for the training of other EWRE graduate students in the use of the instruments. Either the Instrument Manager personally does the instructing, or he/she recommends another student for this purpose.
2. Maintain and periodically review the log book. Assure that the book is being properly used and relevant information is being recorded.
3. Be alert to the need for a formal schedule book (e.g. if scheduling conflicts arise among users). If it is needed, make one available, and assure that the book is being properly used.
4. Verify that the operating and repair manual(s) are kept in their proper places and in good condition.
5. When necessary, and with the advice of the Faculty Laboratory Coordinators, arrange for maintenance and repair of the instrument(s). Some types of maintenance and repair will require the assistance of a commercial repair person, and some can be done by the Instrument Manager.
6. Oversee the proper use of the instrument(s), making sure that they are not abused, and the areas around the instrument(s) are kept clean and free from obstructions.
7. Assure that an adequate stock of instrument-related general-use supplies are on hand, and order additional supplies if they are not. The Instrument Manager should keep track (roughly) of expenditures for general-use supplies, so that he/she can equitably spread the financial burden among the users.

General Use: Students must become familiar with the instruments before attempting to use them without assistance. To gain the necessary familiarity, one or more of the following sources of information must be consulted:

a. The instrument manager
b. Another person who has experience with the instrument
c. Operation manual(s) supplied by the instrument manufacturer or sometimes available on the EVE equipment resource web pages
d. A good general reference such as Principles of Instrumental Analysis, by D.A. Skoog et al. (Brooks/Cole Publ.) located in ELab II, office 312.
e. CD library of Instrument and Methods training located in ELab II, 301 bay E.

For all instruments with an assigned manager, the first step is to consult the instrument manager. He/she can then advise you regarding the necessary reading, and a training session can be arranged on your behalf. Always remember to enter the appropriate information in the instrument log books, and leave the instrument and surrounding benches clean and free from debris.

Locations: Most instruments are located on benchtops in the laboratories. The locations for most major equipment are indicated in the text that follows as each category of equipment is presented. Some older and lesser used equipment is stored in the locked room labeled, "310 Chemical Storage". Equipment that is very rarely used or equipment that is only for field use may be stored in one of the basement cages.

2. Analytical Balances

Available Instruments: The following balances are in the EWRE labs, and most are for general use.

Model #	Type	Location	High use	UMA #
Mettler H31AR		207 Elab II		
Ohaus Galaxy 4000D	Top Loading	304-A Elab II		058620
Denver Inst. XE-3100D	Top Loading	missing[4]		
Mettler H80		24 Marston		
Sartorius 2432		24 Marston		
Denver APX 203	Top Loading	213 Elab II		
AND HM 202		213 Elab II	Yes	
Fisher Accu 2202	Top Loading	208-B Elab II		
Ohaus Adventurer		308-C Elab II	Yes	139212
Mettler Toledo PB 1502	Top Loading	207 Elab II		106616
Ohaus AS3101	Top Loading	304-B Elab II		
Ohaus Explorer Pro		301-D Elab II	Yes	144373

- Students should become familiar with the operation of these balances, before attempting to make a measurement. These instruments are very delicate, and improper use can easily cause malfunctions, leading to costly repairs. Reagents to be weighed out should be transferred with a spatula to a tared weighing dish or weighing paper. The spatula should be cleaned with reagent-grade water and dried with a kimwipe after each use. As always, chemical spills must be cleaned up immediately.

Management. Since measurements with analytical balances are so quick and widely performed, the use of these instruments need not be recorded in log books. However, each user should be alert to problems, and take corrective actions when necessary. If a problem is detected, the instrument manager should be consulted.

A manager is formally designated to oversee the analytical balances. The manager should pay special attention to **cleanliness** in and around the balances. He/she should check the areas around each balance that is in current use on a regular basis. In particular, those designated above as "High use" should be checked on a weekly basis. If there is a recurring problem with cleanliness, the equipment manager should report it to one of the Faculty Lab Coordinators, so that the problem can be corrected. The equipment manager should also coordinate annual maintenance and calibration service. The specific general-use instrument-related supplies that should be maintained include:

a. weighing dishes & paper
b. desiccant
c. spatulas & scoopulas.

[4] Last seen in 302 Elab II in Feb 2007

3. pH/ISE Meters and Electrodes

Available Instruments: The following pH/ISE meters are available for use in the Environmental Engineering labs:

Model	Use	Location	Serial #	UMA #
Orion 520A				
Fisher 600				
Fisher 610A				
Fisher 630				
Orion 940	General	308	058531	
Orion 940	General	308 bay B ELab II	058530	
Orion 940	General	301 bay D ELab II		
Orion 720A		213 Elab II	011513	
Orion 720A		213 Elab II	037089	121758
Corning 320	Portable	207 Elab II	C5688	

Many of these meters are for general use. Those associated with a specific project may often be used by others, however, permission must always be granted prior to use. As a general rule, pH electrodes are kept as personal (or project-specific) equipment. Be careful to maintain pH electrodes. Replace filling solutions and keep the porous junctions clean. Nearly all problems with pH measurements are the fault of the electrode and not the meter. Ion selective electrodes for ammonia, fluoride, calcium and nitrate are available for general use.

In addition, there are two automatic titration systems in the EWRE labs:

a. Orion 960 - Elab II Storage Room
b. Tanager 9501 - Room 308 Elab II

The Orion 960 must be used with an Orion 940 pH/ISE meter, and it is generally available. The Tanager System is a stand-alone set of components that operate with a PC.

Management. Since pH/ISE meters are numerous, easy to use and relatively trouble-free, the use of these instruments need not be recorded in log books. However, each user should be alert to problems, and take corrective actions when necessary. Most problems with pH measurements lie with the electrode. Only on rare occasions does a meter malfunction. If a problem is traced to the meter, the instrument manager should be consulted.

4. UV-Vis Spectrophotometers

Available Instruments: The Visible and Ultraviolet-Visible range Spectrophotometers available for use in the Environmental Engineering labs are summarized in the table below.

Most of the 1-cm UV spectrophotometer cells in the lab are reserved as personal lab supplies by students working as RAs. If there is an intermittent or short-term need for a UV cell, arrangements can be made with other students on an informal basis. However, those students who find themselves continually borrowing cells should probably purchase a set for their own use. At times there has been a general use 1-cm UV spectrophotometer cell in the area of the new Agilent Diode Array Spectrophotometer in Room 308. Longer pathlength cells (5 & 10-cm) are also located here.

Management: A student manager is formally designated for these instruments. The manager should attend to all of the items listed in #1 above. The specific general-use instrument-related supplies that should be maintained include:

a. UV and Vis bulbs
b. paper and ink for printers (if any)
c. General use 1-cm UV cell (if it exists), 5-cm cells, 10-cm cells.

UMass Environmental Engineering: Vis and UV/Vis SPECTROPHOTOMETERS[a]

Characteristic	Bausch & Lomb to Milton Roy to:			ThermoSpectronic; ThermoElectron		Perkin-Elmer		Hewlett-Packard		Hach
	Spec 20	Spec 21D	Spec 70	Genesys 20	Genesys 10UV	Lambda 3A	Lambda 3B	HP 8452	HP 8453	DR/4000U
Location		Basement Cage #4	Basement, Cage #4	Marston 24	304 Elab II 213 Elab II	Basement Cage #4	Elab II storage	301 Elab II	308 Elab II	304 Elab II
Notes	Lowest quality, but rugged	Fair precision	Not functional?	Very good	Very good	Scanning, old & Cranky	Used for parts only	Recently repaired, very good	Excellent, easy to use	Very good
Optical system	Single beam	Single beam	Single beam	Single beam	Split beam	Double Beam	Double Beam	Diode Array	Diode Array	Single Beam
Monochromator	Grating			Grating	Grating	Holographic Grating	Holographic Grating	Holographic Concave Gr.		Seya-Namioka split-beam
Groove Density	600/mm			1200/mm	1200/mm	1440/mm	1440/mm			1200/mm
Detector	Phototube			Solid state		Photo-multiplier	Photo-multiplier	328 Photo-diodes		
Lamp(s)	Tungsten			Tungsten	Xenon	Tungsten-Br Deuterium	Tungsten-Br Deuterium	Deuterium[b]		Tungsten, Deuterium
Readout	Analog	Digital	Analog	Digital	Digital	Digital	Digital	Digital		Digital
Cell Holder	0.5-1" tube			1 cm & tubes		0.1-10cm	0.1-10cm	0.1-10cm		1-10 cm
Wavelength Range	340-625 nm*	Vis	Vis	325-1100nm	190-1100nm	190-900 nm	190-900nm	190-820nm		190-1100nm
Wavelength Accuracy	2.5 nm			2.0 nm	1 nm	0.5 nm	0.3 nm	2 nm		1 nm
Wavelength Precision	1.0 nm			0.5 nm	0.5 nm	0.2 nm	0.1 nm	0.05 nm		0.1 nm
Effective Bandwidth	20 nm			8 nm	5 nm	<2 nm	1 nm	2 nm		4 nm

Diode Array - Simultaneous spectra every 0.6 sec, microprocessor controlled, qualitative and quantitative functions, kinetics functions

[b]Plasma Discharge Deuterium lamp

*Accessory phototubes extend range to higher wavelengths (400-715 or 625-950)

+Requires optional stray light - second order filters (these fit directly in the filter holder), otherwise stray light may be higher

[d]at 1.0 Abs unit

5. Inductively Coupled Plasma (ICP) Emission/MS

Inductively coupled plasma instruments offer a highly sensitive and robust means of analyzing for broad spectrum of metals and in some cases, non-metals in waters and wastewaters. In their most common form, they are used with a simple direct inlet to the nebulizer. They may also be coupled to an autosampler inlet for automated operation. Chromatograph inlet devices have also been used to add an additional dimension to the analysis (e.g., LC-ICP/MS). The most direct chromatographic inlet is an HPLC or IC, however, GC inlets are also feasible. Both types of instruments are capable of multi-element analysis. Sensitivity and MDLs are dependent on the particular element of interest.

Available Instruments: Two Perkin Elmer ICP instruments are available for analysis of metals and some metalloids. The emission instrument (Perkin Elmer Optima 5300 DV) located in 208 Elab II is dedicated to groundwater research. The ICP/MS (Perkin Elmer Elan 9000) is in Bay A of 301 Elab II. While more difficult to use, this instrument is also more versatile. The ICP/MS is commonly used for analysis of drinking waters, surface waters, membrane reject and wastewaters. It includes a dynamic reaction cell, which allows one to eliminate certain types of interfering ions.

Management: A separate student manager is formally designated for each of these instruments. The managers should attend to all of the items listed in #1 above. The specific general-use instrument-related supplies that should be maintained include, but are not limited to:

a. Nebulizer parts
b. Autosampler vials
c. Gases

6. Gas Chromatographs

Gas chromatographs are commonly used in the EWRE program for analysis of trace organic contaminants. These include organic solvents, hydrocarbons, halogenated and non-halogenated disinfection byproducts, pesticides, pharmaceuticals and endocrine disrupters. Nearly all GCs in use are equipped with capillary columns, flash vaporizations injectors and electron capture, flame ionization or thermal conductivity detectors.

Available Instruments: The following gas chromatographs are available for general use in the Environmental Engineering labs:

EWRE Gas Chromatographs

Model	DS#	UMA #	Use	Location	Detectors
Gow Mac			Gases	211 Elab II	TCD
Varian 3300				Elab II Basement Cage #4	FID
Varian 3500				Elab II Basement Cage #4	ECD, FID
Hewlett Packard 5890E II	1	108545	EDB	301 D Elab II	ECD
Hewlett Packard 5890 II	2		Aldehydes etc	301 D Elab II	ECD
Hewlett-Packard 5890A		098230		301 D Elab II	ECD
Hewlett-Packard 5890E II		106408	Air pollutants	211 Elab II	FID
Agilent 6890	3	141331	DBPs	301 D Elab II	ECD
Agilent 6890	4	141332	DBPs	301 D Elab II	ECD

The two 6890s and two of the 5890s are situated in bay D of 301 Elab II. They are networked to an Agilent ChemStation data system (see DS#s above). All four have autosamplers/auto-injectors. The older 5890s are less reliable in auto inject mode, however. These four instruments are equipped with ECDs, and are mostly used for halogenated organic compounds (e.g., disinfection byproducts) and compounds that can be derivatized with electrophilic reagents (e.g., carbonyls).

The Varian 3300 has an ECD and an FID, and can handle both capillary and packed columns. The 3500 also has an ECD and an FID, but is dedicated to capillary columns. It is equipped for subambient operation. These instruments can be linked to Spectra-Physics recording integrators (4270, 4290, and Chromjet). Two of these integrators have been linked to a PC-based data system (Spectra-Physics WINner System) through LABNet.

Management: A student manager is formally designated for these instruments. The manager should attend to all of the items listed in #1 above. The specific general-use instrument-related supplies that should be maintained include, but are not limited to:

a. Syringes and other parts for auto-injectors
b. Graphite and vespul ferrules for capillary columns
c. Extra injector housing
d. Gases

7. Total Organic Carbon (TOC)

These instruments are designed to measure the small concentrations (0-≤ 10 mg/L) of organic compounds typically found in natural water and treated drinking water. Most employ an acidify-purge step to remove inorganic carbon (e.g., carbonates), followed by high temperature combustion and IR detection of the carbon dioxide produced.

Available Instruments: The Environmental Engineering program has five total organic carbon (TOC) analyzers, most of which are available for general use. All but the Dohrmann are based on the high-temperature combustion method. The Shimadzu 5000 in 308 Elab II is most commonly used for drinking water and clean surface water samples. There is a similar instrument in room 208 dedicated to groundwater research. For those interested in total nitrogen analysis, the Shimadzu TOC/V is the appropriate instrument. This is located in room 308 and can measure TN and organic carbon simultaneously. All of these Shimadzu instruments are equipped with PC-based data systems that can be used to store and re-calibrate results. A fourth Shimadzu (model 4000) is occasionally used for on-line analysis. This is a skid-mounted unit located in the corner of room 308.

The Dorhmann instrument, an ambient temperature UV/persulfate analyzer, is not commonly used. It cannot store results, so that it must be run attended at all times. All analyzers are equipped with autosamplers except the Beckman. This last instrument is quite old and now only used for certain specialized needs (e.g., sludges).

EWRE Organic Carbon Analyzers

Model	Use	Location	Comments
Shimadzu 5000	Drinking Water: General Use	308 bay A Elab II	Instrument of choice for most
Shimadzu 5050A	Groundwater	208 Elab II	Just MHD
Shimadzu TOC/V[5]	Clean Water: General Use	308 bay A Elab II	For TN too
Shimadzu 4000	On-line BDOC	308 bay A Elab II	Just on-line
Dohrmann DC-80	General	Basement cage # 4, ELab II	Demo only
Beckman 215	General – Archaic	Basement cage #4, Elab II	Not currently used

Management: A student manager is formally designated for this instrument. The manager should attend to all of the items listed in #1 above. The specific general-use instrument-related supplies that should be maintained include, but are not limited to:

[5] Capable of measuring Total Nitrogen as well

i. Extra catalyst for furnace tubes
ii. Reagent solutions
iii. Oxygen/Air

8. Total Organic Halide (TOX)

These instruments are capable of measuring the total amount of organic chlorine, bromine and iodine in water samples (wastewater, natural water, drinking water). They all employ adsorption to activated carbon for sample pre-concentration and separation of organic halides from inorganic halides. The carbon is then introduced into a high-temperature combustion furnace. The resulting HX is trapped and analyzed for total halogen by precipitation of AgX_2 in a microcoulometer. It is also possible to trap the HX and measure the halide by specific ion analysis (e.g., IC), thereby determining TOCl, TOBr, and TOI.

Available Instruments: A Euroglass PC-controlled TOX analyzer is available for use in 301 Elab II (Bay A). This is the more commonly used of the two. It uses only oxygen as a carrier/reactive gas, and is therefore better suited for subsequent specific halide analysis by IC. A Dohrmann DX-20 total organic halide analyzer is also available for general use. Both analyzers include a carbon adsorption module for pre-concentration and removal of inorganic halide.

Management: A student manager is formally designated for this instrument. The manager should attend to all of the items listed in #1 above. The specific general-use instrument-related supplies that should be maintained include, but are not limited to:

- i. Activated Carbon Glass mini-columns
-. ii Carrier gases (Oxygen & carbon dioxide)
-. iii Acetic acid electrolyte

9. Liquid Chromatographs (HPLC and IC) and LC/MS

Liquid chromatographs (LCs) are better suited for analysis of highly soluble (hydrophilic) organic contaminants than GCs. There are a group of HPLCs designed especially for ion analysis, called ion chromatographs. These use ion exchange columns and conductivity detectors. In general, HPLCs are also better for compounds that would decompose in hot GC injectors. Recent advancements in HPLC technology have helped to overcome some of the drawbacks that had previously limited their use, poor chromatographic resolution, detector insensitivity, incompatibility with MS. HPLC, especially with MS detection has become an essential tool in environmental engineering.

Available Instruments: Three Waters High Performance Liquid Chromatographs (HPLCs) and two Dionex Ion Chromatographs (ICs) are available in the EWRE labs. Two of HPLCs are Alliance units that have autosamplers and dual pumps for binary solvent programming. Each has a diode array detector and one also has a fluorescence detector. The third instrument is a new higher pressure LC, called an Ultra Performance Liquid Chromatograph (UPLC). This is a Waters Acuity model, which promises higher resolution than the standard HPLC.

All three HPLCs can be used as an inlet to a mass spectrometer (LC/MS). The older Alliance HPLC (Model 2690) is situated so that it can feed directly into EWRE's ICP/MS. It is also used with size exclusion columns and may be linked to a carbon-specific detector (Ionics TOC monitor). The newer Alliance (Model 2695) and the Acquity can feed into a triple quadrupole mass spectrometer (Waters Quattro Micro). These LC/MS combinations are frequently used for analysis of perchlorate and pharmaceuticals in water.

The Dionex ICs have a conductivity detector employing chemical suppression technology. For most inorganic analytical applications, the ICs are the appropriate choice, whereas the HPLCs are for use with organic analytes.

EWRE Liquid Chromatographs

Model	DS#	UMA #	Use	Location	Detectors
Waters Alliance 2690			size exclusion, Amino Acids, HPLC-ICP/MS	301 A Elab II	996 PDA, 474 fluorescence
Waters Alliance 2695			Organic chloramines, PPCPs, halobenzoquinones	301 C Elab II	2996 PDA; MS (quattro-micro)
Waters Acquity (UPLC)			EDCs, perchlorate	301 E Elab II	PDA, MS (quattro premier)
Dionex IC			Iodide, bromide	301 A Elab II	Conductivity
Metrohm IC			Cations/anions	211	Conductivity

Management: A student manager is formally designated for the HPLCs and all non-dedicated IC instruments. The manager should attend to all of the items listed in #1 above. The specific general-use instrument-related supplies that should be maintained include, but are not limited to:

a. Priming Syringes
b. Sample vials
c. Analytical Columns
d. Guard Columns
e. Reagents for Mobile Phases
f. Helium for degassing

10. Gas Chromatograph – Mass Spectrometer (GC/MS)

The use of mass spectrometers as detectors for gas chromatography represents a powerful addition to standard GC for identification of unknown contaminants or verification of suspected compounds. These GC/MS instruments are more complex to operate than GCs, but their use has revolutionized environmental engineering. There are several types of mass spectrometers, each with its own set of advantages and capabilities. Single quadrupoles are simple and inexpensive, but their abilities to provide unambiguous identification are limited. Ion traps can be quite sensitive and their design often allows for tandem mass spectrometry (e.g., MS/MS). Time of flight mass spectrometers have much higher resolution, allowing the assignment of unambiguous molecular formulae.

Available Instrument: Four GC/MS instruments are available in the EWRE labs. The newest is a Varian 2200 ion trap instrument. This is used for standard analyses, such as nitrosamines and dioxane. The next newest and most selective is the time-of-flight instrument (Waters GCT, or GC-TOF). This is capable of mass resolution of 10 ppm, and most appropriate for analysis of unknowns. In addition, there is a Hewlett-Packard 5988 Gas Chromatograph - Mass Spectrometer, which is a single quadrupole unit. It is capable of electron impact ionization, as well as both negative and positive chemical ionization. This instrument is equipped with a pyroprobe, and was last used for Pyrolysis GC/MS of NOM samples. Both the GC-TOF and the HP 5988 are located in 301 Elab II. In addition, there is an older Varian Ion Trap GC/MS available in 301 Elab II.

Management: A student manager is formally designated for this instrument. The manager should attend to all of the items listed in #1 above. The specific general-use instrument-related supplies that should be maintained include, but are not limited to:

a. Septa for injector
b. Graphite and vespul ferrules for capillary columns
c. filaments
d. Gases

11. Equipment for Molecular Biology

Real-time analysis for microbial pathogens and indicator organisms is critical for implementing timely remedial actions and monitoring for unfiltered water sources. A suite of instrumentation exists in the Watershed Molecular Pathogen/Indicator Monitoring Laboratory (Room 211) for this purpose. These include:

- Three Hybrid Auto Incubators (Fisher Isotemp)
- Fotodyne/Analyst Luminary Workstation, including a cooled CCD camera with motorized zoom lens, a light-tight benchtop darkroom, imaging filters and a data system with acquisition software
- Electrophoresis system, including two Fotodyne mini cell chambers, powered by a Foto/Force 250 power supply
- Peltier Thermal Cycler (PTC-200) from MJ Research
- Sterile Chemguard III Advance hood. This is a class II, Type B2 100% exhuast vertical flow cabinet
- Laminar flow hood
- Ultra-low freezers
- Autoclave

The freezers are used to store microbial samples, primers, probes, oligonucleotides enzymes and other required reagents. The PCR workstation is the designated area for preparing reaction mixtures, and is kept separate from any work with cDNA products to minimize contamination of samples. The autoclave is used to supplement two existing autoclaves in the program, the Market Forge and Amerex. The thermocycler and clean-box (MJ Research) are used for amplifying genetic material and working with cDNA to avoid cross contamination of experiments. A rotating/shaking hybridization oven (Amersham Biosciences) and a blotting system (Immunetics) are used for probing and specific amplicon detection. Electrophoresis and gel visualization/documentation instrumentation (FotoDyne, Inc) also allows for the quantitative analysis of isolated and amplified genetic material. This instrumentation can be employed in a number of experimental procedures for amplifying, detecting, and separating different pathogen and indicator organisms in watershed samples.

12. Autoclaves

Available Instruments: Three medium-sized autoclaves (Market Forge Sterilmaster, Hirayama-Amerex, and a Yamato SM510) are available in a dedicated autoclave room on the 2nd floor (204A). A similar room on the 3rd floor (302A) has a small semi-portable autoclave (MDT Harvey Hydroclave MC8).

Room #	Model #	Serial #
204A	Market Forge Sterilmaster	UMA010738
204A	Hirayama-Amerex	WRAIR PBRW 13951
204A	Yamato SM510	UMA137668
302A	MDT Harvey Hydroclave MC8	LA27397C40
Basement cage #4	MDT Harvey Hydroclave MC8	LA27421C40
Basement cage #4	MDT Harvey Hydroclave MC8	LA27383C40

Management: A student manager is formally designated for this instrument. The manager should attend to all of the items listed in #1 above. This is usually a graduate research assistant working on an environmental microbiology project.

13. Ozone Generator

Available Instrument: A large-output Welsbach Model T-408 is available in Room 308, Bay B for general use. It is connected to a gas handling manifold which is housed in the adjacent fume hood. This system also includes gas and liquid phase ozone monitors and a computer-based monitoring and data acquisition system. A much smaller Certizon C25 unit is integrated into the pilot system (constant temperature room 308B).

Management: A student manager is formally designated for this instrument. The manager should attend to all of the items listed in #1 above. The specific general-use instrument-related supplies that should be maintained include, but are not limited to:

i. Desiccant
ii. Spare Orbisphere electrode membranes
iii. Oxygen

14. Turbidimeters

Available Instruments: Two bench-top Hach Turbidimeters are located in Room 304. These instruments require periodic primary calibration with primary standard (formazin) and each use calibration with secondary standards.

15. Other

Generic Type	Model	Location
Microplate AutoReader	Bio-Tek EL 311s	207 Elab II
Viscometer	DV-E	213 Elab II
Centrifuge	Sorvall RC SC Plus	213 Elab II

F. RECORD KEEPING

1. Laboratory Notebook

It is essential that all raw data be recorded in a bound notebook at the time of data collection. Many environmental researchers use paperbound laboratory notebooks containing numbered pages with tear-out carbons. This is an excellent system, because the carbons can be stored separately so that it is unlikely that both the carbons and the original could be inadvertently lost or destroyed.

It is important that the laboratory notebook remain in good physical condition and legible for a long period of time. For this reason use only ball-point pens, do not use felt-tip pens or pencils. Black ink is preferred, but blue is also acceptable. Do not try to erase mistakes. Instead, draw a line through the erroneous entry and record the correct entry nearby. Write legibly, but don't be overly concerned with neatness. Pages must be used consecutively. Leaving a page or pages blank for later use is not good practice. Entries must be presented in chronological sequence and dated. Time of day should be indicated at least every 3 hours. A good practice is to reserve the first page as a periodically-updated table of contents. Avoid excess detail, so that the table of contents can completely fit on one page.

As you plan your experiments you should also think about how you will record data in the lab notebook. This will not only help to produce an organized notebook, but it may lead you to recognize a deficiency in your experimental procedure while you still have a chance to correct it. Data are often best recorded and presented in tabular form. For some repetitive procedures, standardized report forms may be most convenient. These forms should be numbered consecutively and kept in a separate notebook. Reference should be made in the laboratory notebook to the specific report form as appropriate. You should present a complete record of your experiments. All steps in a procedure should be recorded, even if they seem trivial. Very often it is the fine details which take on great importance in retrospect. Record all of your observations. Refer to standard or published procedures whenever possible. However, carefully describe any deviations you may have taken from these procedures. You should also describe any unusual aspects of the laboratory environment. For example, the smell of fresh paint, new asphalt, etc. may indicate possible sources of atmospheric contamination of laboratory solutions. (for more guidance, see Writing the Laboratory Notebook, by Kanare, ACS Publishers, 1985).

2. Log Books

Log books are maintained for the following instruments or systems:

Instruments with Log Books

Instrument
1. Gas Chromatographs
2. ICP Instruments
3. GC/MS
4. HPLCs
5. Organic Carbon Analyzers
6. TOX Analyzers

Users of these instruments or systems should be aware of the specific reporting requirements of the log books. The proper use of log books is critical to the diagnosis of problems, and to the expectation of standard levels of performance.

a. Sign up Calendar
b. Usage Log
c. Daily Performance (ICP-MS
d. Hardware & Software Errors
e. Maintenance Log

G. CLEANING PROCEDURES

1. Glassware Cleaning

For the successful testing and analysis of environmental samples, it is imperative that glassware be scrupulously clean throughout all "crucial" steps of the lab work. Although an experienced analyst will know which steps are crucial, and where a less rigorous cleaning regimen is appropriate, the beginner will not have this advantage. Therefore, unless you have direct experience with alternative cleaning procedures, you are advised to use the following hierarchal set of procedures:

Many of the country's top commercial laboratories have partly abandoned the labor-intensive wet cleaning protocols for high temperature muffling (450ºC or 800ºF) for 5-8 hrs as a general procedure. Many more laboratories used glass muffling for some extremely demanding uses. Analysis of polychlorinated biphenyls (PCBs) is a good example. Some labs routinely use organic solvents for cleaning. However solvent vapors and residues can create problems for certain types of laboratory experiments (e.g., acetone is quite disruptive for labs where disinfection byproduct testing is conducted). For this reason, great care must be taken when using solvents.

Assessing the needs

The first step is to inspect dirty/used glassware and take stock of its recent use history. All internal and external residue (including labeling tape) must be removed (step #1). Visible internal residue must be removed via steps 2, 3, 4 or 5). Glassware that cannot be cleaned of visible residue (external as well as internal) should generally be discarded (e.g., extraction vials with resistant tape residue). Do not initiate final cleaning protocols if the glassware still has obvious residue as this will contaminate soaking baths. Perfectly clean glassware is hydrophilic, and will be uniformly wetted (i.e., no beading of water). If this is not the case, the glassware may need more than the normal cleaning protocol (e.g., including steps 1, 2, 3, 4 or 5). It is important to **wash glassware as quickly as possible** after use.

Glassware is cleaned according to its recent use, its next intended future use, and its appearance. As a guideline, please consult the following table. Recognize that category "C" may cover most glassware used in EWRE. This category calls for pre-rinse, acid soak, post-rinse, drying and clean storage (cleaning steps #6-8).

Category	Description	Cleaning Protocol
A	Glassware that is visibly contaminated, cloudy or discolored	1,2,3, 4 as needed, then 5-8
B	Glassware that was in contact with highly-polluted water (e.g., municipal wastewater, industrial wastewater), but not visibly contaminated	5-8
C	**Glassware that has only been in contact with relatively clean waters (e.g., surface waters, groundwaters, drinking waters)**	**6-8**
D	Glassware that has only been in contact with laboratory stock solutions	Rinse with solvent
E	Glassware that has only been in contact with pure solvents	Rinse with solvent

General Cleaning Protocol.

The following list is a complete generic protocol for cleaning and storing glassware intended for general laboratory use. In several cases soaking solutions are prescribed. These should be prepared and maintained to make sure that they do not become contaminated or otherwise lose their effectiveness. Most laboratory rooms have an acid bath for general use, located adjacent to sink (e.g., NE corner of bay "C" for room 301). Acid baths should be replaced weekly or more often if they become visibly discolored or accumulate sediment. Be very careful to **clean up any spilled acid as soon as it occurs**. We have already experienced some very expensive damage to our wooden casework as a result of spills that were not immediately tended to.

Be aware that most of the cleaning steps below will not be used on a routine basis. Consult the above table for guidelines on which steps to use under a give situation. If the glassware has only been exposed to uncontaminated natural water, steps 6-8 may be sufficient.

1. Removal of external residue (e.g., from labeling tape)
    a. Pull off all tape; remove and glue with single edged razor blade if necessary
2. Removal of grease (e.g., stopcock grease) or other stubborn deposits
    a. Using a disposable wipe (e.g., Kimwipes) or rigid cleaner (pipe cleaner), physically remove as much as the grease as possible
    b. Chemical treatment; use one or more of the following
        i. Boil glassware in a weak solution of sodium carbonate
        ii. Soak and rinse in acetone

    iii. Soak in decahydronaphthalene for 2 hours (especially for removing silicone grease)
    iv. Soak in warm (65ºC) alcoholic hydroxide solution for no more than 10 min (doesn't work for Apiezon)
  c. Use multiple acid/base rinses to get inaccessible grease or if trace levels may cause problems (e.g., high silicone contamination in GC/MS or GC/FID.
3. Ultrasound Treatment
  a. Place glassware in an ultrasound bath with either distilled water or detergent solution and
4. Oxidant Soak
  a. Pre-clean glassware with detergent and water
  b. Soak glassware the Nochromix bath for at least 20 minutes and up to 24 hours
    i. Nochromix is a product of Godax Laboratories. It comes as a dried chemical mix (mostly persulfate) with instructions on final preparation (requires addition to concentrated H_2SO_4.
    ii. If Nochromix does not solve the problem, we can use Chromic Acid as an alternative. This solution requires extensive rinsing to remove excess chromium. It is also a carcinogen. Care must be exercised in disposing of chromic acid
  c. Rinse glassware 5 times with distilled water
5. Detergent Wash
  a. Soak for 10-15 minutes in warm detergent solution. Use a soft brush as necessary. Avoid soaking for very long periods of time, as this may tend to roughen the glass surface (A variety of commercially available laboratory detergents may be dissolved in tap water for this purpose; e.g., 1% Alconox, 2% RBS-35; Extran).
  b. Rinse with hot tap water, followed by 3 rinses with distilled water.
6. Acid Soak.
  a. Soak pre-rinsed or pre-washed glassware in an acid bath
    i. In the UMass Environmental Engineering laboratories, we most often use a 5% sulfuric acid bath. In most cases, students have their own acid baths, or they may share baths with others working on the same research project. All glassware must be at least pre-rinsed with Distilled water prior to soaking.
    ii. Some laboratories use 5% or 10% HCl, but this is a volatile acid, and partly for this reason is it less commonly used
    iii. Acid baths need to be changed monthly or more frequently if obvious contamination occurs. Neutralize with Sodium Hydroxide or Sodium Bicarbonate before disposing with copious amounts of water down the sink. Label new bath with content, % concentration, date and your name.
  b. 3 rinses with distilled water.
7. Drying.
  a. Place clean glassware upside down in a clean drying oven set at 110-140°C. Non-volumetric glassware can be dried at higher temperatures. Note that plastic ware should be dried at lower temperatures (50°C or room temperature).
  b. Use of near-sink pegboards is only recommended for short term drying needs or for temperature-sensitive materials (non-glassware)
8. Final Storage
  a. Clean glassware should be stored in a way that avoids atmospheric deposition of contaminants.
    i. <u>Wide mouth glassware</u>: invert glassware in a clean, lined drawer (e.g., for beakers)
    ii. <u>Narrow mouth glassware</u>: cover tops with appropriate stopper, aluminum foil or parafilm
    iii. <u>Other</u>: experience has shown that storing extraction vials (20-60 mL) on their side in a clean constrained drawer space does not cause degradation over short time periods

b. Locations for cleaned glassware should include drawers, and cabinets, but not soaking baths, pegboards, or bench tops.

Trace Organic Analysis. As previously mentioned, some analysis requires high-temperature muffling (e.g., ultra-low level PCB analysis). Some labs use multiple solvent rinses for glassware used in the analysis of pharmaceuticals and personal care products (PCPPs). A popular regimen is hexane, followed by acetone, and then methanol. High purity solvents must be used (e.g., Burdick & Jackson, GC^2 for "Trace analysis below ppb levels"). Rinsing with organic solvents may also be needed to remove accumulated contaminants in glassware used for holding aqueous mobile phases in LC/MS

Trace Metal Analysis. Start with clean glassware (steps 1-5 in general cleaning protocol, as needed). Rinse first with distilled water. Then soak in re-distilled nitric acid (20%) overnight. Alternatively, one can use a mix of 8% nitric acid and 17% hydrochloric acid. This procedure is commonly used for heavy metal analysis by atomic absorption spectrophotometry. Some soak glassware in a hot dilute (0.004M, pH 12) solution of EDTA for 20 minutes to remove metal ions.

Residual Oxidant Analysis (chlorine, ozone). Start with clean glassware (steps 1-5 in general cleaning protocol, as needed). Rinse first with distilled water. Then soak with the appropriate oxidant solution. Use a solution that is slightly more concentrated than the highest level the glassware is likely to be exposed to during testing. In some cases it may be most convenient to leave the glassware in a long-term oxidant soak (e.g., chlorine demand free glassware; for 24 hours). Other oxidants may be too transient, and glassware must then be prepared immediately before use. For example, ozone-demand-free glassware should be treated by filling with a high concentration ozone solution (e.g., 5 mg/L) and letting it sit for 2 hours.

Category & Product		Notes:
Detergent		
	Liquinox	dodecylbenzenesulfonate
	Extran	Ethanolamine, EDTA and NaOH
	RBS-35	
	Decon	
	Pyroneg	
Oxidative Reagent		
	Nochromix	90-95% ammonium persulfate, surfactants
	Persulfate solution	19 g $(NH_4)_2S_2O_8$ in 2 L of 98% H_2SO_4
Alcoholic hydroxide		
	Alcoholic NaOH	Add 120 g NaOH to 2 liters of 95% ethanol
	Alcoholic KOH	Add 100 g KOH to 2 liters of 95% ethanol

2. Lab Benches

Individuals are responsible for cleaning the bench tops in their work areas after each use. A clean sponge and warm tap water are recommended for this purpose. Drawers and cabinets should be kept reasonably clean. Common benches, not assigned to any one person (e.g. benches serving general-use instruments), are the responsibility of everyone. Please clean these areas after each use!

3. Floors

Floors in Elab II will be mopped and polished twice a year by the building janitorial staff. More frequent cleaning will be handled by the laboratory coordinator with the assistance of the EVE graduate students. Aside from the planned cleaning, individuals should mop or otherwise clean the floor whenever a spill occurs.

H. LABORATORY REAGENTS

1. Water

This is the most frequently used "chemical" in the environmental laboratory. A building-wide system exists in Elab II for purification of municipal water to general laboratory water standards. There are high purity water (HPW) taps located in all of the lab rooms. When collecting a sample of HPW for use, it is important to recognize that the quality of this water degrades upon sitting. For this reason, it is important to get a sample of freshly-purified water. This requires that the water be allowed to run for at least 60 seconds so that the line from the ceiling mounted building loop to the tap is completely flushed.

The Elab II system was provided by Fluid Solutions Inc of Lowell. It is fed with city water, which is passed through the following treatment steps

- Multi-media filter
- GAC Filter
- RO cartridge pre- filter (5 µm)
- Reverse Osmosis
- 400 gallon storage tank
- UV reactor
- Deionizer (mixed bed)
- 0.2 µm multi-element filter

The system recirculates water in a loop from the 1st, 2nd and 3rd floors back to the storage tank, and out through the UV reactor, deionizer and final filter.

The effectiveness of deionization should be verified with a resistivity/conductivity meter. These provide a non-specific measure of the concentration of ionic species in the product water. Theoretically pure water has a resistivity of about 18 megohms, and as the concentration of ionic species increases the resistivity decreases.

System water quality should also be monitored by regular analysis of TOC from several of the building taps. Water should be allowed to run for at least 60 seconds so that the line from the ceiling mounted building loop to the tap is completely flushed. A sample should then be collected and analyzed immediately for total organic carbon; results should be recorded in the TOC log book.

Also available to the EWRE program is the Milli-Q Gradient located in 301 bay A, ELab II. The Millipore water system is designed to produce ultrapure (Type 1) water operating directly from the building–wide high purity water (HPW) system. The Milli-Q Gradient is designed for Chromatography applications such as HPLC, UPLC or GC and provides up to 200L/day.

2. Chemical Reagents

Most stable, non-volatile chemicals for general use are stored in the chemical supply room on the 3rd floor of Elab II (labeled “308A Storage”). Volatile solvents are stored in solvent cabinets under most of the hoods in Elab II. Some volatile or unstable compounds are stored in two explosion-proof refrigerators in 308 Elab II. These are grouped and most are stored in plastic secondary containment vessels within the refrigerators. All of these bottles should be recorded in the Environmental Health & Safety chemical inventory (CEMS), so please refer to this on-line resource when looking for a specific volatile reagent. This avoids unnecessary opening of the refrigerators and secondary containment vessels.

Mark every new chemical bottle with the date and your initials (e.g., Rec’d 10 May 2006, DAR) as it is received. At the same time, check for a UMass bar code (normally done upon receipt by LHSS). If there is not a bar code, please notify the Laboratory Safety Coordinator. .

Chemicals that are purchased for a specific research project, and are to be used exclusively for that project, should be stored in the project work area (unless it is a volatile or hazardous substance).

Lab users are encouraged to purchase personal supplies of chemicals whenever the risk from contamination is great. In this case the chemicals should be stored somewhere in the laboratory area assigned to you unless they are volatile or unstable. Laboratory reagents may be purchased at a variety of chemical purities. Grades labeled "Technical" and "USP" are usually not appropriate for environmental work. Purities such as "Analytical Reagent Grade" and "ACS Grade" are acceptable. Information on the maximum concentrations of chemical impurities is often listed on the bottles. Certain types of chemically stable reagents that are commonly used as primary standards (e.g., potassium acid phthalate, arsenic trioxide) may be available as "Primary Standard Grade". These will have a very accurate assay of near 100%. High purities for specialized analysis are sometimes available, such as "Pesticide Grade", "HPLC Grade", "Spectrophotometric Grade", etc. These are more important for trace organic and inorganic analysis. Also, Aldrich Chemical Company (one of the major US supplies of specialized organic chemicals) offers a general top-of-the-line purity called "Gold Label Grade".

Reagents can become easily contaminated when used by a large number of people. Therefore, it is important that great care be exercised in handling high-purity reagents, even if some of the reagent must be wasted following each use. Solid reagents should be transferred directly from their bottle to a weighing bottle or weighing paper by carefully tilting and rotating the bottle so that a fine flow of crystals or powder is created. If a spatula must be used it should be scrupulously clean and dry. Excess reagent must be discarded. Never return excess reagent to the original bottle!

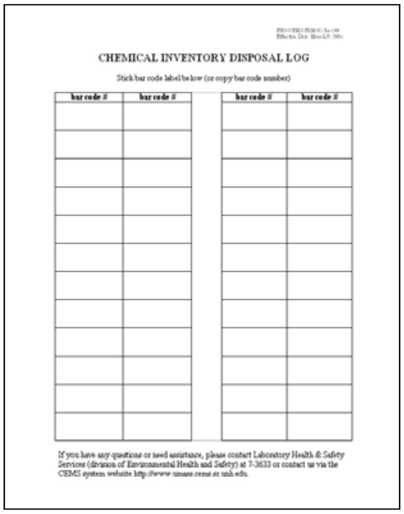
Liquid reagents are best used by pouring a slight excess into a small beaker that has been previously rinsed with the reagent. The required amount can then be pipetted from the beaker. The excess reagent should be discarded. Never return excess to the original reagent bottle, and never pipet directly from a communal liquid reagent bottle.

Volatile organic reagents (e.g., solvents) and unstable compounds should never be stored in individual work areas (e.g., drawers and benchtops). These should be returned to their appropriate storage areas after each use. Most aqueous samples and standard solutions should be stored in a refrigerator or freezer. One of the explosion-proof refrigerators in room 301 is dedicated to the storage of volatile organic liquids. Care should be taken to ensure that tops to bottles and associated septa are tight-fitting so that leaks can be kept to a minimum. Bottles should be organized into compatible groups and placed in rectangular plastic (tupperware, rubbermaid) containers to help prevent cross-contamination.

Elements of the UMass Inventory Management program are summarized below:

1. Empty Containers

- Remove bar code from bottle and place on the “Chemical Inventory Disposal Log” EHS-CEMS-FRM-01 (LHSS will pick up sheets on a periodic basis, usually every other week)

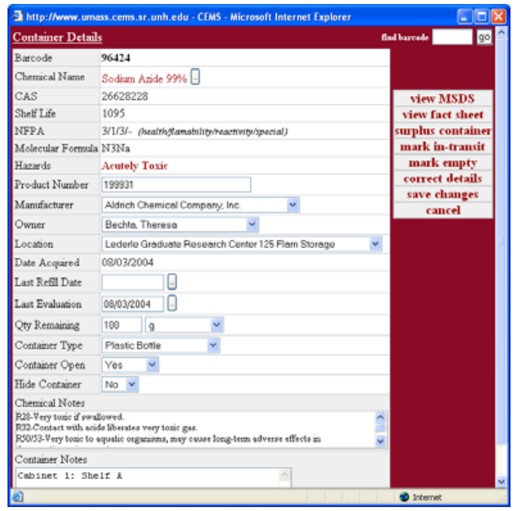


CHEMICAL INVENTORY DISPOSAL LOG

Stick bar code label below (or copy bar code number)

bar code #	bar code #	bar code #	bar code #

If you have any questions or need assistance, please contact Laboratory Health & Safety Services (division of Environmental Health and Safety) at 7-3633 or contact us via the CEMS system website http://www.umass.cems.sr.unh.edu



Note: Special procedures may be required for the disposal of empty containers

2. Hazardous Waste
   - Via CEMS database,
     - o Click <update inventory> and enter bar codes
     - o Click < auto generate waste request>
     - o Fill in the PI, Department and Pick-up Location and add any additional wastes that may not be bar coded
     - o Remember to mark containers as empty

     This form can also be found on the Environmental Health and Safety Homepage http://www.ehs.umass.edu or directly at http;//www.umass.cems.sr.unh.edu/CEMS/RequestRemoval
   - “Hazardous Waste Pick-up Request”. You do not need a CEMs account to use this form

3. For Questions or Concerns contact
   - Laboratory Health and Safety Services 413-577-3633
   - Environmental Health and Safety 413-545-2682
   - EWRE Laboratory Safety Coordinator 413-577-3231

3. Gases

All gas cylinders must be securely anchored at all times. They should be moved with a cylinder cart, only after replacing the protective cap. Gases associated with instruments will be purchased under the direction of the Instrument Manager or the principal user. The laboratory coordinator will manage demurrage charges.

Most new gas tanks will be delivered to tank farm areas in the hallway just outside of, or directly inside specific lab rooms in Elab II. In general, tanks will remain in the tank farms. In a few special cases, they may be located in the labs and securely anchored to a lab bench. Large dewars (liquid N_2, He, Ar) are normally free-standing in the lab rooms. Empty tanks should have the regulator removed, replacement of protective cap , placed in the same secure tank farm area, and clearly marked as “empty” or “MT”. All tanks should always be secured with the locking chain (tank farms) or strap (in labs).

I. HEATING, COOLING AND DRYING

1. Refrigerators & Freezers & Ice

We have numerous refrigerators and freezers in the Environmental Engineering research labs. Explosion-proof refrigerators should be used for storing volatile organic liquids that must be kept under refrigeration. Most of the standard refrigerators have small freezer compartments. These refrigerators/freezers are only used for storage of aqueous samples and standard solutions.

EWRE Refrigerators (some with freezer compartments)

#	Type	Location	Purpose	Model	UMA#
3	Explosion Proof	301 Elab II (d)	DBP's & Pure chemicals	Lab-Line	
10	Half ht	211 Elab II		Fisher Sci	
12	Half ht	302a Elab II		Fisher Sci	
15	Half ht	213 Elab II (b)		Fisher Sci	
28	Half ht	213 Elab II(c)	Samples	Kenmore	
29	Mini	213 Elab II (c)	Samples	GE	
25	Mini	301 Elab II (e)		Haier	
11	Narrow	308 Elab II (b)	Chlorine experiments & TN/TOC solutions	VWR-Revco	
2	Explosion proof	301 Elab II (b)	DBP stds, stocks	Precision Sci.	
20	With Freezer	208 Elab II	Samples	Westinghouse	
19	With Freezer	24 Marston		Sears Coldspot	
13	With Freezer	308 Elab II (a)	DBP/TOC samples; Ice Packs	Gibson	
5	Full size with Freezer	213 Elab II (a)	Samples	Frigidaire	
4	Full size with Freezer	211 Elab II	Stocks	VWR Revco	
16	With Freezer	301 Elab II (d)	DBP extracts, GC & HPLC	GE	
23	Half ht	213 Elab II (c)		Fisher Sci	141339
14	Freezer	302a Elab II		Fisher Sci	
30	Full size with Freezer	207 Elab II		Fisher Isotemp	141338
9	Half ht	213 Elab II (c)		Fisher Sci	

EWRE Freezers

#	Type	Location	Purpose	Model	
27	Half height	213 Elab II (c)	Stock Solutions	VWR-Revco	
26	Ultra-low temp	Elab II Basement cage #4	Ice	Revco	
8	Full size with freezer	213 Elab II (a)		Fisher	103032
21	Ultra-low temp	2nd floor hallway Elab II		Revco	137666

We commonly use sealed plastic Ice packs for keeping samples cool during transit. These are stored in the freezer compartments of several of the Refrigerator/Freezers listed above. In addition, we have some access to Professor Lianhong Sun's ice maker (room 110, Elab II). Please consult Prof. Chul Park first, if you wish to use this resource.

2. Constant Temperature Chambers

The EWRE group has 7 walk-in controlled environment chambers on the 2nd and 3rd floors of Elab II. These are capable of controlling both temperature and humidity. They are generally kept at the designated temperatures and reserved for certain uses as indicated in the table below. **Please do not change the settings on these rooms.** Occasionally, as needed, the temperatures and uses of these rooms are changed, but this is a process that requires broad consensus. These changes are done only after careful consideration of the EWRE faculty and the lab managers. Such requests should be sent to the overall faculty lab coordinator, and to the staff coordinator. They will take the request to the entire EWRE faculty and full group of Lab Managers for consideration.

Please remember that smaller volume storage is available in one of several refrigerating incubators as listed below. These are alternatives to the walk-in chambers, and can often be more easily adjusted to the temperatures needed without inconveniencing others.

EWRE Walk-in Constant Temperature Chambers

Room #	Nearby Lab	Function	Temp	Special restrictions
204B	204	Micro "Add Mix"		
208A	208	Cold Storage	4°C	
208B		Groundwater Research	12°C	
208C		WW Treatment Research	20°C	
304A	304	Drinking Water Research	-4°C	
304B		Cold Storage	4°C	
308B	308	Natural Water Research; O3 pilot	5-25°C	No VOCs

The refrigerating incubators are refrigerator-like chambers where a pre-set constant temperature is maintained. This temperature may be either above or below ambient. These units are most commonly used to store living organisms or to provide a constant temperature environment for chemical or biological reactions (e.g., BOD, THMFP).

EWRE Refrigerating Incubators

Number	Type	Location	Purpose	Model
17	Full size			Percival
6	Full size	301 Elab II (e)	20°C	Fisher low temp 300
7	Full size	308 Elab II (b)	20°C	Fisher low temp 307
14	Full size			Percival
8	Full size	211 Elab		Precision 815
21	Glass-fronted	208 A Elab II		Fisher Isotemp
18	Glass-fronted	213 Elab II (a)		Fisher Isotemp
9	Half-ht			Labline Ambi-hi-lo chamber
23	Half-size			Fisher Model 146A
22	Half-size			Fisher Model 146A

3. Ovens & Incubators

The program has two muffle furnaces (550°C), numerous ovens (usually ~110°C), water baths and incubators (ambient - 65°C). The muffle furnace is located in-308 bay C. The ovens are located at various points in the lab area, and are either designated for glassware drying, plasticware drying or for chemical/sample drying. Water baths are typically used for sample incubation.

EWRE Ovens, water baths and Incubators

Number	Type	Location	Purpose	Model
	Oven	213 Elab II	Glassware Drying	Fisher isotemp
	Oven	308 Elab II	Glassware Drying	Precision Thelco
	Oven	308 Elab II	Plasticware drying	Blue M
	Oven		Chemical/sample drying	Blue M Stabil Therm
	Oven	208 Elab II	Chemical/sample drying	Fisher Isotemp
	Oven	304 Elab II	Chemical/sample drying	Blue M
	Muffle Furnace/Kiln	308 Elab II	Glassware drying	Paragon
	Incubator	Elab II Basement cage #4	30°C incubator	Precision Thelco Model 7
	Incubator	Elab II Basement cage #4		Precision Thelco Model 4
	Incubator	308 Elab II	20°C incubator	Fisher Scientific
	Incubator			Millipore
	Incubator	211 Elab II		Labline “science teaching”
	Water Bath	211 Elab II		Fisher Isotemp 211
	Water Bath	Elab II Basement cage #4		Fisher Isotemp 202
	Water Bath	211 Elab II		Fisher (20 Liter)
	Water Bath	Elab II Basement cage #4		Isotemp 120

4. Desiccators

Single desiccators are located in various parts of the lab. -

J. TESTING AND ANALYSIS PROTOCOLS

There are many types of repetitive laboratory procedures that EWRE graduate students may need to learn for the purposes of conducting their research. Many of these procedures are documented in standard reference books (e.g., Standard Methods for the Examination of Water and Wastewater), or in journal articles. However, it is quite common that small modifications are made based on the peculiarities of our laboratory facilities. In other cases specific options from published methods have been formally adopted by EWRE researchers. These modifications and clarifications are codified in formal documents, called Standard Operating Procedures (SOPs). If you are conducting or intend to conduct tests from the list below, please obtain a copy of the appropriate SOP. Printed copies can be found in the lab or may be retrieved from: http://www.ecs.umass.edu/eve/research/sop/index.html It is important that these detailed procedures be followed to ensure consistency and data quality. In some cases, research contracts require that they be followed to the letter.

Protocols for which EWRE Laboratory SOPs have been developed

General Type	Protocol
Process	Ozonation
	Chlorination
Analytical	Trihalomethanes (THMs)
	Haloacetic Acids (HAAs)
	Total Organic Halide (TOX)
	Organic Carbon (TOC, DOC)
	Major Anions by IC
	pH
	Chlorine Residual
	Organic Nitrogen
	Haloacetamides

Despite the formal nature of these SOPs, there are mechanisms by which they may be updated. Please feel free to suggest changes to either the faculty QC officer or the graduate QC officer for the particular procedure. Both of these individuals are identified in a separate memo that is regularly updated.

K. PROCUREMENT

One of the normal activities of graduate students working on funded laboratory research is the procurement of supplies. The ordering of laboratory chemicals and supplies is - done by -the laboratory manager or other designated person, with the approval of the appropriate faculty PI. Please plan your lab supply needs ahead of time, so most or all of your purchase orders can be processed through regular channels without having to pay for expedited delivery.

While ordering from vendors is not usually done by the students, it is sometimes helpful if the students are aware of appropriate shipping addresses. With the new chemical management system, orders for chemicals and non-chemical supplies must be separated. Different delivery addresses must be listed for the two types of orders:

For Chemical Orders

Tell the Vendor to use the following Shipping Address when placing your order:

University of Massachusetts
Room 125 Lederle Graduate Research Tower
710 North Pleasant Street
Amherst, MA 01003
Attn: “Responsible Owner”, “Storage Location” (Building,Room# and Bay)

Example: Attn: Dave Reckhow, Elab II 308, bay E

OR

Select "**CHEMS**" as the "Ship To" default in the Panel Header

List the "***Responsible Owner", "Storage Location" (Building & Room#)*** in the "Comments" Panel. (Remember to click the <send to vendor> checkbox

For Supplies (anything but chemicals and major equipment)

Tell the Vendor to use the following Shipping Address when placing your order:

University of Massachusetts
Room 18 Marston Hall
Department of Civil & Environmental Engineering
Amherst, MA 01003
Attn: "Non-Responsible Owner", "Responsible Owner"
Example: Attn: Boning Liu, Dave Reckhow

If you are expecting package deliveries, please check in 18 Marston. CEE staff will often send email to graduate students (i.e., non-responsible owners) asking them to pick up packages once they have been delivered to Marston Hall. Space in 18 Marston is extremely limited, so if you receive such an email please respond quickly or arrange for another student to pick up the package. All packing slips must be returned to the bookkeeper.

Most faculty and staff use the University's ProCard system for purchase of common, low-cost supplies. ProCards are issued directly to faculty/staff. The card facilitates quick purchasing with less paper work than the standard requisition/purchase order procedure. However, the card may only be used for official University business, and only in accordance with University policies and procedures. If your advisor has a ProCard, you might be able to use this method of procurement (usually the ProCard "owner" places the order directly). The maximum allowable purchase on a ProCard is typically $500. As with all purchases, you will need to get a receipt with transaction amount on it, and turn this in to the EWRE grants manager or bookkeeper.

For more information on use of ProCards, please consult the UMass web page: http://www.umass.edu/procurement/Procard/procard_index.html

-1. On-campus Purchases (including chem stockroom)

Purchase from on-campus suppliers or services (e.g., glass shop, ESIO office; statistical consulting) are done as "recharges". Of these, only the chemical stockroom in LGRT requires a purchase order. All others are direct recharges. Students can get approval for the purchase from the PI and the PI will give them the account number to use. The bookkeeper still requires all receipts.

To purchase from the chemical stock room, you must be registered in their system. The CEE bookkeepers have the necessary forms to do this. They can also get ProCard information to the stockroom is that is to be the means of payment. The Chemical stockroom is located in the basement of the Lederle high rise (5 LGRT), and the phone numbers are 7-2600 and 5-2852.

-2 Receiving Non-chemical Orders

a. Packages are delivered to Marston Hall daily. You may check for packages in room 18 Marston. If called to retrieve packages, please respond quickly.

b. Open packages and inspect for damage. Check-off items on the packing slip, date and initial, and give the slip to the EWRE grants manager or bookkeeper.
c. Fill out and return warranty cards for equipment. Write serial number, model number, date received, and project name on the equipment manual or instruction sheet.
d. Follow-up on any problems with the order (broken or missing pieces, incorrect items, long overdue back orders) by calling the vendor.
e. Save instrument boxes in case instrument must be returned for repair. Packaging boxes should be disposed of in the recyclable cardboard container located outside Elab II.

-3. Receiving Chemical Orders

Mark every new chemical bottle with the date and your initials (e.g., Rec'd 10 May 2006, DAR) as it is received. At the same time, check for a UMass bar code (normally done upon receipt by LHSS; if it is shipped through LGRT 125). If there is not a bar code, please contact the Laboratory Safety Coordinator.

EHS-CEMS-FRM-01 Rev00
Effective Date: March 9, 2006

CHEMICAL INVENTORY RECEIPT LOG
(For all substances received that require bar coding)

Name of Responsible Owner (e.g. Principal Investigator, Lab or Studio Technician):______________________________

Complete All of the below information for the received chemical
Apply the respective CEMS barcode to the right of the label

Chemical Name	Product # or Catalog #	Manufacturer	Amount (list unit of measure)	Container Type (e.g. plastic, glass)	Storage Location (Building and Room#)	Bar Code Number(s)

If you have any questions or need assistance, please contact Laboratory Health & Safety Services (division of Environmental Health and Safety) 7-3633 or contact us via the CEMS system website http://www.umass.cems.sr.unh.edu <contact EH&S>

CEMS Database

CEMS home page (on-campus only): http://www.umass.cems.sr.unh.edu/

New Account
Click on "*sign in*" link
Click on "Name not on List? Request Account" link
Fill out form. Make sure you include all required fields in red.
Please provide:

- *"home phone"* (cell phone, home phone, pager#) for emergency purposes only
- *"Non Responsible Owners (e.g. grad students)* the name, phone number and email of the responsible owner *"supervisor",* whose inventory you wish to access.
- The CEMS data manager will contact the responsible owner for authorization. Please allow time for the CEMS administrators to process your request.

You will receive a link via email to enter your password.
Your name should now appear in the drop down list.

-4. Special Concerns Regarding Gas Cylinders

Most gas cylinders will be ordered by the instrument managers. Many cylinders are assigned to a particular account number. Empty tanks must be credited to the correct account and replacement ordered on the appropriate account. Place a drop down tag on the tank when it is received. Mark date received, date empty and account number.

-5. Vendors

Many general laboratory supplies can be conveniently purchased from Fisher Scientific at competitive prices. Fisher, along with a handful of other vendors, offers the University a substantial discount by virtue of the Massachusetts Higher Education Consortium Contract. Below is a list of commonly used vendors. Catalogs for most of these companies and many more can be found in the ELab II Graduate Student Office area (210). Those that are members of the consortium are followed by an asterisk.

a. General		
Fisher Scientific *	800-766-7000	Acc# 508514-01
VWR Scientific *	800-225-4290	Acc# 87465-524
Cole-Parmer Instr. Co	800-323-4340	Acc# 017626-29
b. Chromatography		
Supelco	800-247-6628	Acc# 49450561
Varian	800-538-1735	Acc# 309961
J&W	916-985-7888	Acc# 104287
c. Chemicals		
Sigma-Aldrich	800-558-9160	Acc# 49450561
d. Gases		
AirGas *	800-649-1639	Acc# P1HY1
Aero-All Gas *	800-255-4277	

Eliminate this section

a. University Store
b. Chemical Store Room (basement of Lederle GRC)
c. Glass Shop (basement of Lederle GRC)
d. Engineering Computer Service (ECS)
e. Rocky's Hardware

Buy only small items on these purchase orders. Get authorization from the faculty PI. Keep the receipt and return it to the EWRE grants manager or bookkeeper.

L. EXIT PROTOCOLS

It is the responsibility of students to clean out and wipe down all of their work areas (lab & office) before completing their work at UMass. Students supported by research grants should return their personal lab supplies to their advisor or, on his/her instruction, hand the lab supplies over to another student. All samples or standard solutions used exclusively by the student must be removed from storage areas and properly disposed of. Before completing final clean-up, students must consult with the Laboratory Manager. All keys must be turned in to either the Laboratory Manager or Faculty PI.