Laboratory Safety Manual

Advancing Life and Regenerating Motherland

(ALARM)

Contents Page No

LABORATORY PROJECT AND EXPERIMNT

Tables Page No

1. Introduction

The laboratory safety manual included the laboratory health and safety and standard operating procedures prepared for use in Environmental Watch Laboratory (EWLab) and in Advancing Life and Regenerating Motherland (ALARM).

It was prepared to ensure safe laboratory practices during the research work and testing procedure. The expected information include in manual was concerned with safe lab-practice, the use of personal protective equipment, emergency procedure used and, storage of chemicals and the proper method of waste disposal. This manual also covers hazard communication incident response. This manual also covers hazard communication incident response. This information is intended to help those in laboratory to minize hazard to laboratory staff. The metals and other substances are test in the test sample. Appendix is concluded with the standard recommended levels of the parameters include in air, water and soil.

In the views of wide variety of chemical products chandelled in laboratory, it is not assumed that the precautions and requirements states here are all inclusive. This information should be updated as needed with supplementary information to better protect the health and safety of anyone working in or visiting the laboratories.

It is also included procedure for sampling and the summary of analytical test that have been reviewed as their ability to be used in the field by a variety of users. In addition, it is also included spectrophotometic method and various methods available for analyzing The metals and other substances are test in the test sample. Appendix is concluded with the standard recommended levels of the parameters include in air, water and soil.. There are many special procedures conducted within our laboratories, which require unique health and safety precautions.

EWLab will have additional procedures that apply to its own situations and work. In all cases the laboratory supervisor is ultimately responsible for training safe work practices and must insist upon the use of proper procedures to eliminate unnecessary hazards.

Objective

Enviromental watch laboratory (EWLab) manual was intended to improve the environmental performance by assisting in the development and and implementation of environmental manangement programs that is to meet regulatory requirements and prevent pollution .The main objectives of this manual is

- To provide a guide for establishing and monitoring healthy and safe working condition in EWLab.
- To promote safe practice by all EW- Lab staff members.
- To define health and safty responsibilities within EW-Lab community.
- To provide information of the standard procedure for the analysis of the sample.
- To provide the general guidelines and basic rules considered the minimum for the safe operation of a laboratory at EWLab;
- To provide accurate and valuable research data to perform the research efforts for the solution of environmental pollution.

1.1 Role of Laboratory

The role of the analytical laboratory is to provide qualitative and quantitative data to be used in decision making. To be valuable, the data must accurately describe the constituent characteristics and concentrations in the sample submitted to the laboratory. Decisions made using water and wastewater data are far reaching.

In wastewater analysis, the laboratory data define the treatment plant influent, the status of the steps in the treatment process, and the final load imposed upon the water resources. Decisions on process changes, plant modifications or even the construction of a new facility may be based upon the results of laboratory analysis. The financial pressures alone are significant reasons for extreme care in analysis.

Research investigations in environmental pollution control rest upon a firm base of laboratory data. The progress of the research and the alternate pathways available is generally evaluated on the basis of laboratory data. The value of the research effort will depend upon the validity of the laboratory results. Thus, learning to perform laboratory tests on water, wastewater, air, and solid wastes plays an important role in the environmental protection.

1.2 Defination in a laboratory

1.2.1 Training Laboratory

A laboratory where a group of trainees simultaneously receive instruction in, and perform, experimental procedures associated with a formally approved EcoDev / ALARM course on environmental management or technical training.

1.2.2 Testing and Research Laboratory

A laboratory set up primarily to conduct research or to test environmental parameters.

1.2.3 Supervisor

A supervisor is a person who has charge of a workplace or authority over a worker. At EWLab, this includes all responsible persons and staff who supervise a laboratory. The Steering Committee of the Environmental Watch Program will appoint a supervisor for EWLab. This supervisor is responsible for all matters of health and safety in the lab and will keep the records pertaining to health and safety for the lab. The steering committee will ensure that on appointment each supervisor attends a Health and Safety for Supervisors training session provided by EWLab.

1.2.4 Technician/User

A laboratory technician or user is anyone, trainee, staff or internee, who works as a user or for pay in a laboratory, including those who have supervisory responsibilities.

1.2.5 Unattended Procedures/Equipment

A procedure or piece of equipment that is left operating when no one is in the lab.

1.2.6 Employees

Employees are expected to follow all applicable practices and procedures contained in the Laboratory Safety Manual, attend designated training sessions, and report hazardous or unsafe conditions to the lab supervisor, Advancing Life and Regenerating Motherland (ALARM).

1.2.7 Trainees

Trainees are expected to observe all applicable safety practices and procedures contained in this Laboratory Safety Manual, attend designated training sessions, and report any unsafe or hazardous conditions to the lab supervisor, Advancing Life and Regenerating Motherland (ALARM).

1.2.8 Visitors

Visitors are considered to be all persons entering a laboratory other than the EW, laboratory staff, enrolled students and authorized. Visitors to EW laboratories will be under the supervision of the host laboratory. The host is responsible for laboratory security during the visitation, visitor training and notification of potential hazards, and oversight of visitor compliance with applicable safety practices and procedures contained in the Laboratory Safety Manual.

1.3 Administrative Research Laboratory

1.3.1 Responsibilities of Supervisors

The supervisor of a laboratory has overall responsibility for safety.

Prior to any work being performed by a new laboratory user it is the supervisor's responsibility to ensure that users are aware of safety rules and follow them and that the following training has been provided:

- An appropriate safety orientation when individuals are first assigned to a laboratory space;
- Generic WHMIS (Workplace Hazardous Materials Information System) training, and specific WHMIS training provided by the supervisor;
- Radiation Safety Training, provided by the Radiation Safety Expert, if applicable;
- Training on special or unusual hazards in the lab;
- Training in the use of laboratory specific emergency equipment and emergency response; Records of the training must be kept on file in the EWLab and a copy sent to EcoDev / ALARM.

In addition the supervisor is responsible for the following:

- That adequate emergency equipment in proper working order is readily available;
- That an incident investigation report is completed for every incident or injury that occurs in his/her lab.

Examples include incidents requiring first aid or other medical attention and incidents resulting in property damage, such as, spills, fires, explosions as well as near misses in either category. Incidents resulting in personal injury to a worker require completion of a Workplace Safety Insurance Procedure of Myanmar Insurance Policy.

- That every two weeks safety and housekeeping inspections of the lab are conducted with a record of the inspection kept on file in the lab.
- That an appropriate alternate is appointed as supervisor when the laboratory supervisor is absent. In a training lab where safety is a concern, the supervisor or alternate will always be present. In a research lab, an alternate will be appointed when the supervisor is away from the EWLab.

1.3.2 Responsibilities of Laboratory Technicians

All laboratory technicians are responsible for:

- Following all applicable safety rules and practices as outlined in this manual and by the supervisor;
- Using and wearing personal protective equipment according to instructions;
- Reporting all incidents to the laboratory supervisor;
- Reporting all unsafe conditions to the laboratory supervisor;
- Completion of recommended occupational health screening programs when applicable; and
- Attending all training courses as directed by the supervisor.

1.3.3 General Health and Safety Principles

Good laboratory practice requires that every laboratory technician and supervisor observe the following:

- Food and beverages are not permitted in the lab. Consume food and beverages only in properly designated areas.
- Use appropriate personal protective equipment at all times.
- Use laboratory equipment for its designed purpose.
- Confine long hair and loose clothing.
- Use a proper pipeting device. Absolutely no pipeting by mouth.
- Avoid exposure to gases, vapours, aerosols and particulates by using a properly functioning laboratory fumehood.
- Wash hands upon completion of laboratory procedures and remove all protective equipment including gloves and lab coats.
- Ensure that the laboratory supervisor is informed of any unsafe condition.
- Know the location and correct use of all available safety equipment.
- Determine potential hazards and appropriate safety precautions before beginning new operations and confirm that existing safety equipment is sufficient for this new procedure.
- Avoid disturbing or distracting other workers while they are performing laboratory tasks.
- Ensure visitors to the laboratory are equipped with appropriate safety equipment.

- Be certain all hazardous agents are stored correctly and labelled correctly according to Workplace Hazardous Materials Information Systems (WHMIS) requirements.
- Consult the material safety data sheet prior to using an unfamiliar chemical and follow the proper procedures when handling or manipulating all hazardous agents.
- Follow proper waste disposal procedures.

1.4 Preparing for Laboratory Work

Before starting to work in a laboratory, familiarize the lab-staff with the following:

- The hazards of the materials in the lab, as well as appropriate safe handling, storage and emergency protocols. Read labels and material safety data sheets (MSDSs) before moving, handling or opening chemicals. Never use a product from an unlabeled container, and report missing labels to your supervisor.
- The agents, processes and equipment in the laboratory. If laboratory staff are unsure of any aspect of a procedure, check with lab supervisor before proceeding.
- The location and operation of safety and emergency equipment such as fire extinguishers, eye wash and shower, first aid and spill response kits, fire alarm pull stations, telephone and emergency exits
- Emergency spill response procedures for the materials lab staff will handle
- Emergency reporting procedures and telephone numbers
- Designated and alternate escape routes

1.4.1 During Laboratory Work

- Restrict laboratory access to authorized persons only. Children are not permitted in labs.
- Smoking; eating; drinking; storing food, beverages or tobacco; applying cosmetics or lip balm and handling contact lenses are not permitted in laboratories.
- Wear lab coats (knee length) and safety glasses in laboratories employing chemicals, biohazards or radioisotopes. Open shoes, such as sandals, should never be worn in the lab.
- Tie back or otherwise restrain long hair when working with chemicals, biohazards, radioisotopes, or moving machinery.
- Keep work places clean and free of unwanted chemicals, biological specimens, radios, and idle equipment. Avoid leaving reagent bottles, empty or full, on the floor.
- Work only with materials once with knowing their flammability, reactivity, toxicity, safe handling and storage and emergency procedures.
- Consult material safety data sheets (MSDS) before working with hazardous chemicals or infectious material. Replace MSDS that are more than 3 years old.
- Prepare and maintain a chemical inventory for the lab.
- Never pipette by mouth; use mechanical transfer devices.
- Walk, do not run, in the lab.
- Keep exits and passageways clear at all times.
- Ensure that access to emergency equipment (eyewashes, safety showers and fire extinguishers) is not blocked.
- Report accidents and dangerous incidents ("near-misses") promptly to your supervisor
- Wash your hands thoroughly before leaving the laboratory.

- Conduct procedures involving the release of volatile toxic or flammable materials in a chemical fume hood.

1.4.2 Cleaning up Before Leaving

Perform a safety check at the end of each experiment and before leaving the lab. Make sure to:

- Turn off gas, water, electricity, vacuum and compression lines and heating apparatus
- Return unused materials, equipment and apparatus to their proper storage locations
- Label, package and dispose of all waste material properly, "Waste Preparation Procedures"
- Remove defective or damaged equipment immediately, and arrange to have it repaired or replaced
- Decontaminate any equipment or work areas that may have been in contact with hazardous materials.
- Leave behind protective clothing (lab coats, gloves, etc.) when leaving the laboratory
- Close and lock the door to the laboratory if lab-staff are the last one to leave

1.4.3 Evaluating Laboratory Hazards, an Ongoing Process

There are many categories of hazards that might be encountered in a laboratory setting, and situations can change frequently. Even after identified and controlled all current risks, it is critical that the possibility of new unexpected dangers can arise. Periodically verify that the Laboratory Information Card (LIC) and other hazard warnings are current carry out weekly inspections on the condition of:

- fire extinguishers
- emergency wash devices such as eyewashes and drench hoses (run these for several minutes and update inspection tags
- first aid kit contents
- fume hood and other ventilation devices
- tubing for circulating water, vacuum, gases
- chemical storage compartments

Also, ensure that fire extinguishers and emergency showers are inspected, tested and tagged annually.

Among potential laboratory hazards, be alert for the following:

Chemical products

- flammable
- toxic
- oxidizing
- reactive
- corrosive

Microbiological disease-producing agents and their toxins

- viruses
- bacteria
- parasites
- rickettsiae
- fungi

Physical or mechanical hazards

- ionizing and non-ionizing radiation
- electrical
- poor equipment design or work organization (ergonomic hazards)
- tripping hazards
- excessive noise or heat

Psychosocial conditions that can cause psychological stress

1.4.4 Working alone policy

Working alone is an unsafe practice at any time. However, if the nature of the work makes it unavoidable, take measures to ensure that others are aware of the location and have someone check time to time, either in person or by telephone.

Before conducting any work alone in a laboratory go through this checklist to determine if it is appropriate to proceed:

- Is the supervisor aware of the plans?
- Are there any hazardous experiments involved?

Examples:

- High temperature
- High vacuum
- Extremely flammable materials (low flash point)
- Poisonous materials

2. Chemical Hazards and Chemical Spill Response

2.1 Toxic chemicals

Chemicals can gain entry into the body by:

- ***Inhalation*** of gases, vapours and particulate material (e.g. mists, dusts, smoke, fumes)
- ***Absorption*** through skin of liquids, solids, gases and vapours
- ***Ingestion*** of chemicals directly or indirectly via contaminated foods and beverages and contact between mouth and contaminated hands (nail-biting, smoking)
- ***Injection*** of chemicals through needles and other contaminated laboratory sharps

2.1.1 Working with Embryotoxins

Embryotoxins are substances that cause adverse effects on a developing fetus. These effects may include embryolethality, malformations, retarded growth, and postnatal function deficits.

A few substances have been demonstrated to be embryotoxic in humans. These include:Acrylic acid, Aniline, Benzene, Cadmium, Carbon, sulfide, N,N-dimethylacetamide, Dimethylformamide, Dimethylsulfoxide, Diphenylamine, Estradiol, Formaldehyde, Formamide, Hexachlorobenzene, Iodoacetic acid, Lead compounds, Mercury compounds, Nitrobenzene, Nitrous oxide, Phenol, Thalidomide, Toluene, Vinyl chloride, Xylene, Polychlorinated and polybrominated biphenyls.

Embryotoxins requiring special controls should be stored in an adequately ventilated area. The container should be labeled in a clear manner such as the following: EMBRYOTOXIN: READ SPECIFIC PROCEDURES FOR USE. If the storage container is breakable, it should be kept in an impermeable, unbreakable secondary container having sufficient capacity to retain the material, should the primary container fail.

2.1.2 Working with High Chronic Toxic Chemical

High chronic toxicity), along with their corresponding chemical class, are: high chronic toxicity), along with their corresponding chemical class, are:

Alkylating Agents:

α-Halo ethers
Aziridines
Diazo, azo,and azoxy compounds
Electrophilic alkenes and alkynes
Epoxides

AcylatingAgents:
Organohalogen Compounds:
Natural Products:
Inorganic Compound:
Aromatic amines:
Other Extremely Hazardous Chemicals:

The above substances (in both lists) must be used and stored in areas with restricted access. Special warning signs must be posted in these areas. Containers should be stored in chemical-resistant trays, and work must be performed within or above these trays. Cover surfaces where these substances are used with absorbent, plastic-backed paper. Performance-certified hood or other containment devices must be used when generation of toxic vapor, gases, dusts, or aerosols might occur.

2.2 Flammable chemicals

Flammable and combustible liquids, solids or gases will ignite when exposed to heat, sparks or flame. Flammable materials burn readily at room temperature, while combustible materials must be heated before they will burn. Flammable liquids or their vapours are the most

common fire hazards in laboratories. Examples –*Organic Solids:* camphor, cellulose nitrate and napthalene.*Inorganic Solids:* decaborane, lithium amide, phosphorous heptasulfide, phosphorous sesquisulfide, potassium sulfide, anhydrous sodium sulfide and sulfur.Organic liquid: alcohol, ester, ether, ketoe, toluene, acetone.

For specific details on the safe handling of flammable chemicals in the laboratory:

- Keep away from heat,aparks and open flame.
- Post NO SMOKING signs in work or storage area.
- Keep the minium quantity in the work area.
- Store away from oxidizers.Label containers FLAMMABLE.
- Make sure sprinklers and fire extinguishers are available and working.

2.2.1 Health Effects Associated with Flammables

In general, the vapors of many flammables are irritating to mucous membranes of the respiratory system and eyes, and in high concentrations are narcotic. The following symptoms are typical for the respective routes of entry:

Acute Health Effects: Inhalation — headache, fatigue, dizziness, drowsiness, narco , Ingestion - slight gastrointestinal irritation, dizziness, fatigue. Skin Contact — dry, cracked,and chapped skin Eye Contact — stinging, watery eyes, inflammation of the eyelids

Chronic Health Effects: The chronic health effects will vary depending on the specific chemical, the duration of the exposure, and the extent of the exposure. However, damage to the lungs, liver, kidneys, heart, and/or central nervous system may occur. Cancer and reproductive effects are also possible.

Flammable Groups Exhibiting These Health Effects: Hydrocarbons — aliphatic hydrocarbons are narcotic but their systemic toxicity is relatively low. Aromatic hydrocarbons are all potential narcotic agents, and overexposure to the vapors can lead to loss of muscular coordination, collapse, and unconsciousness. Benzene is toxic to bone marrow and can cause leukemia. Alcohols — vapors are only moderately narcotic.Ethers — exhibit strong narcotic properties but for the most part are only moderately toxic. Esters — vapors may result in irritation to the eyes, nose, and upper respiratory tract. Ketones — systemic toxicity is generally not high.

2.2.2 First-Aid Procedures for Exposures to Flammable Materials

Inhalation Exposure — remove person from contaminated area if it is safe to do so. Get medical attention and do not leave person unattended.

Ingestion Exposures — remove the person, if possible, from source of contamination. Get medical attention.

Dermal Exposures — remove person from source of contamination. Remove clothing, jewelry, and shoes from the affected areas. Flush the affected areas with water for at least 15 min and obtain medical attention.

Eye Contact — remove person from source of contamination. Flush the eyes with water for at least 15 min. Obtain medical attention.

2.3 Oxidizing chemicals

Oxidizers provide oxidizing elements such as oxygen or chlorine, and are capable of igniting flammable and combustible material even in an oxygen-deficient atmosphere.Oxidizing chemicals can increase the speed and intensity of a fire by adding to the oxygen supply, causing materials that would normally not burn to ignite and burn rapidly. Oxidizers can also:

- React with other chemicals, resulting in release of toxic gases
- Decompose and liberate toxic gases when heated
- Burn or irritate skin, eyes, breathing passages and other tissues

Examples of Common Oxidizers

Peroxides Nitrites
Nitrates Chlorates
Perchlorates Chlorites
Hypochlorites
Dichromates

Precautions to follow when using and storing oxidizers in the laboratory include the following:

- Keep away from flammable and combustible materials
- Keep containers tightly closed unless otherwise indicated by the supplier
- Mix and dilute according to the supplier's instructions
- To prevent release of corrosive dusts, purchase in liquid instead of dry form
- Reduce reactivity of solutions by diluting with water
- Wear appropriate skin and eye protection
- Ensure that oxidizers are compatible with other oxidizers in the same storage area

2.3.1 Health Effects Associated with Oxidizers

Oxidizers are covered here primarily due to their potential to add to the severity of a fire or to initiate a fire. But there are some generalizations that can be made regarding the health hazards of an oxidizing material. In general, oxidizers are corrosive and many are highly toxic.

Acute Health Effects

Some oxidizers, such as nitric and sulfuric acid vapors, chlorine, and hydrogen peroxide, act as irritant gases. All irritant gases can cause inflammation in the surface layer of tissues when in direct contact. They can also cause irritation of the upper airways, conjunctiva, and throat.

Some oxidizers, such as fluorine, can cause severe burns of the skin and mucous membranes. Chlorine trifluoride is extremely toxic and can cause severe burns to tissue.

Nitrogen trioxide is very damaging to tissue, especially the respiratory tract. The symptoms from an exposure to nitrogen trioxide may be delayed for hours, but fatal pulmonary edema may result.

Osmium tetroxide, another oxidant commonly employed in the laboratory, is also dangerous due to its high degree of acute toxicity. It is a severe irritant of both the eyes and the respiratory tract. Inhalation can cause headache, coughing, dizziness, lung damage, difficulty breathing, and may be fatal.

Chronic Health Effects

Nitrobenzene and chromium compounds can cause hematological and neurological changes. Compounds of chromium and manganese can cause liver and kidney disease. Chromium (VI) compounds have been associated with lung cancer.

2.3.2 First Aid for Oxidizers

In general, if a person has inhaled, ingested, or come into direct contact with these materials, the person must be removed from the source of contamination as quickly as possible when it is safe to do so. Medical help must be summoned. In the case of an exposure directly to the skin or eyes, it is imperative that the exposed person be taken to an emergency shower or eyewash immediately. Flush the affected areas for a minimum of 15 minutes and then get medical attention.

2.4 Reactive chemicals

Product that can undergo dangerous reaction (burn, explode, or produce dangerous gases) when exposed to heat, light, physical movement (such as jarring, compression), water, moisture, or incompatible materials. Examples of dangerously reactive materials are plastic monomers such as butadiene and some cyanides. Water reactive molecules are Alkali metals (lithium, sodium, potassium) Magnesium, Silanes, Alkylaluminums, Zinc, Aluminum. Pyrophoric material can ignite spontaneously in the presence of air. Examples of pyrophoric materials: Diethylzinc Triethylaluminum Many organometallic compounds.

- May be sensitive to jarring, compression, heat or light
- May react dangerously with water or air
- May burn, explode or yield flammable or toxic gases when mixed with incompatible materials
- Can vigorously decompose, polymerize or condense
- Can also be toxic, corrosive, oxidizing or flammable
- Some chemicals may not be dangerous when purchased but may develop hazardous properties over time (e.g. diethyl ether and solutions of picric acid).

Follow these precautions when working with dangerously reactive chemicals:

- Store and handle away from incompatible chemicals
- Keep water-reactive chemicals away from potential contact with water, such as plumbing, fire sprinkler heads and water baths
- Handle in a chemical fume hood
- Wear the appropriate skin and eye protection
- Work with small quantities
- Use up or dispose of these chemicals before they attain their expiry date

Other Shock-Sensitive Materials

These materials are explosive and sensitive to heat and shock. Examples of shock-sensitive materials:Chemicals containing nitro groups Fulminates Hydrogen peroxide (30+%) Ammonium perchlorate Benzoyl peroxide (when dry) Compounds containing the functional groups: acetylide, azide, diazo, halamine, nitroso, and ozonide.

2.4.1 Health Hazards Associated with Reactives

Reactive chemicals are grouped as a category primarily because of the safety hazards associated with their use and storage and not because of similar acute or chronic health effects. For health hazard information on specific reactive materials, consult the MSDS or the manufacturer. However,there are some hazards common to the use of reactive materials. Injuries can occur due to heat or flames, inhalation of fumes, vapors and reaction products, and flying debris.

2.4.2 First Aid for Reactives

If someone is seriously injured, the most important step is to contact emergency responders as quickly as possible. Explain the situation and describe the location clearly and accurately.

If someone is bleeding severely, apply a sterile dressing, clean cloth, or handkerchief to the wound. Then put protective gloves on and place the palm of your hand directly over the wound and apply pressure and keep the person calm. Continue to apply pressure until help arrives.

If a person's clothes are on fire, he or she should drop immediately to the floor and roll. If a fire blanket is available, put it over the individual. An emergency shower, if one is immediately available, can also be used to douse the flames.

If a person goes into shock, have the individual lie down on his/her back, if safe to do so, and raise the feet about 1 ft above the floor.

2.5 Corrosive chemicals

Corrosives are materials, such as acids and bases (caustics, alkalis) which can damage body tissues as a result of splashing, inhalation or ingestion. Also:

- They may damage metals, releasing flammable hydrogen gas
- They may damage some plastics
- Some corrosives, such as sulphuric, nitric and perchloric acids, are also oxidizers; thus they are incompatible with flammable or combustible material
- They may release toxic or explosive products when reacted with other chemicals
- They may liberate heat when mixed with water
- Burns eyes and skin on contact.

Examples of Corrosives

Sulfuric acid, Chromic acid, Stannic chloride, Ammonium bifluoride, Bromine, Ammonium hydroxide

Precautions for handling corrosive materials include:

- Wear appropriate skin and eye protection
- Use in the weakest concentration possible
- Handle in a chemical fume hood
- Use corrosion-resistant containers when transporting and storing corrosives
- Always dilute by adding acids to water, never add water to acid.
- Store acids separately from gases
- In case of skin or eye contact with corrosives,flush area with cool water for 15minutes,remove affected clothing,and get medical help.

2.5.1 Health Effects Associated with Corrosives

All corrosives are severely damaging to living tissues and also attack other materials, such as metal.

Skin contact with alkali metal hydroxides, e.g., sodium hydroxide and potassium hydroxide, is more dangerous than with strong acids. Contact with alkali metal hydroxides normally causes deeper tissue damage because there is less pain than with an acid exposure. The exposed person may not wash it off thoroughly enough or seek prompt medical attention.

All hydrogen halides are acids that are serious respiratory irritants and also cause severe burns. Hydrofluoric acid is particularly dangerous. At low concentrations, hydrofluoric acids do not immediately show any signs or symptoms upon contact with skin. It may take several hours for the hydrofluoric acid to penetrate the skin before you would notice a burning sensation. However, by this time permanent damage, such as second and third degree burns with scarring, can result.

Acute Health Effects

Inhalation — irritation of mucous membranes, difficulty in breathing, fits of coughing, pulmonary edema

Ingestion — irritation and burning sensation of lips, mouth, and throat; pain in swallowing; swelling of the throat; painful abdominal cramps; vomiting; shock; risk of perforation of stomach

Skin Contact — burning, redness and swelling, painful blisters, profound damage to tissues; and with alkalis, a slippery, soapy feeling

Eye Contact — stinging, watery eyes, swelling of eyelids, intense pain, ulceration of eyes, loss of eyes or eyesight

Chronic Health Effects

Symptoms associated with a chronic exposure vary greatly depending on the chemical. The chronic effect of hydrochloric acid is damage to the teeth; the chronic effects of hydrofluoric acid are decreased bone density, fluorosis, and anemia; the chronic effects of sodium hydroxide are unknown.

2.5.2 First Aid for Corrosives

Inhalation — remove person from source of contamination if safe to do so. Get medical attention. Keep person warm and quiet and do not leave unattended. Ingestion — remove person from source of contamination if safe to do so. Get medical attention and inform emergency responders of the name of the chemical swallowed.

Skin Contact — remove person from source of contamination if safe to do so and take immediately to an emergency shower or source of water. Remove clothing, shoes, socks, and jewelry from affected areas as quickly as possible, cutting them off if necessary. Be careful to not get any chemical on your skin or to inhale the vapors. Flush the affected area with water for a minimum of 15 minutes. Get medical attention.

Eye Contact — remove person from source of contamination if safe to do so and take immediately to an eyewash or source of water. Rinse the eyes for a minimum of 15 minutes. Have the person look up and down and from side to side. Get medical attention. Do not let the person rub the eyes or keep them tightly shut.

2.6 Chemical spill Response

2.6.1 Chemical Spills

The user of the hazardous material is responsible for cleaning up a spill. Spill kits should be available in all labs. When a chemical is spilled contact the lab supervisor immediately. If the spill is beyond the resources or abilities of the users to cleanup, contact the Safety Officer at 01 230 5540. This duty must not be delegated to other staff such as caretakers.

In cleaning up a spill the following guide should be followed:

- Determine what was spilled and if the area is safe. If there is any doubt about the safety of an area or the nature of the spilled material evacuate the area using the fire pull station. If the pull station is used, meet emergency personnel to explain the situation.
- Administer first aid where needed.
- Secure the area to prevent others from entering.
- Gather required information such as MSDS's. Consult your supervisor and/or lab technician of EWLab. Carefully evaluate the situation and form an action plan.
- Put on all required personal protective equipment.
- Using appropriate cleanup agents, cleanup spill.
- Dispose of residue according to the Procedure for the Disposal of Hazardous Materials or contact EWLab for advice.
- Fill out an Injury/Incident Report.

2.6.2 Emergency Procedure for Hazardous Materials Spill

All accidents, hazardous materials spills, or other dangerous incidents should be reported. A list of telephone numbers must be posted on the door to each laboratory (and must be kept up to date). Telephone numbers shall also be posted beside every telephone in the laboratories.

Laboratory Supervisor **-** 09 731 749 18

Advancing Life and Regenerating Motherland (ALARM) **-** 01 230 5540 extension *803

Fire station **-** Ahlone ph: 220802, Kyeemyindine ph: 534825, Sanchaung ph: 527099, 536687.

Callers should explain any emergency situation clearly, calmly, and in detail.

2.6.3 Emergency Procedures for Minor Spills

- Trained personnel should use the spill control kit appropriate to the material spilled to clean up the spill.
- If the spill is minor and of known limited danger, clean it up immediately. Determine the appropriate cleaning method by referring to the material's MSDS. During cleanup, wear the appropriate protective gear.
- Cover liquid spills with compatible adsorbent material such as spill pillows or a kitty litter/ vermiculite mix, if it is compatible. If appropriate materials are available, corrosives should be neutralized prior to adsorption. Clean spills from the outer area first, cleaning toward the center.
- Place the spilled material into an appropriate impervious container and seal. Schedule its disposal.
- If appropriate, wash the affected surface with soap and water. Mop up the residues and place them in an appropriate container for disposal.
- If the spilled material is not water soluble, a solvent such as xylene may be necessary to clean the surface(s). Check the solubility of the spilled material in various solvents and use the least toxic effective solvent available. Wear appropriate personal protective equipment.
- Notify the Laboratory Supervisor about the need to replace the used items from the spill control kit.

2.6.4 Hazardous chemical spills

In the event of a spill of a hazardous (volatile, toxic, corrosive, reactive or flammable) chemical, the following procedures should be followed:

- If there is fire, pull the nearest alarm. If you are unable to control or extinguish a fire, follow the fire safety procedures.
- If the spill is in a laboratory or chemical storeroom:
    - Evacuate all personnel from the room
    - Be sure the hood/local exhaust is turned on
    - If flammable liquids are spilled, disconnect the electricity to sources of ignition if possible
    - Call the emergency telephone number to request additional assistance if laboratory staff cannot manage the clean-up by thern.
- If the spill is in a corridor or other public passageway:
    - Evacuate all people from the area and close off the area to keep others out.
    - Call the emergency telephone number, to have the air system in the area shut down (to prevent contamination of other areas) and to request additional assistance.

2.6.5 Corrosive liquids spill

- Alert everyone present. If vapours are being released, clear the area.

- Do not attempt to wipe up a corrosive liquid unless it is very dilute.
- Gloves, boots, apron and eye protection must be used when neutralizing an extensive corrosive spill. Respiratory protection is required if the liquid releases corrosive vapour or gas.
- Pour the required neutralizing or adsorbing material around the perimeter of the spill, then carefully add water and more neutralizing material to the contained area. Carefully agitate to promote neutralization.
- Use pH paper to verify that all contaminated areas are neutralized and safe to wipe up.
- If an adsorbent (eg. spill control pillows) is used instead of a neutralizer, scoop up the absorbed spill, place it in a plastic bag, seal it, and then place in a labeled box. If neutralized material contains no toxic heavy metals (e.g. chromium), flush down the drain with plenty of water.

2.6.6 Corrosive solids spill

Small spills can be cleaned up mechanically with a dustpan and brush. Larger spills should be cleaned up using a HEPA (high-efficiency articulate) filter vacuum. For spills containing fine dusts, an air-purifying respirator with dust filters is recommended, as are gloves, protective goggles, and a lab coat.

2.6.7 Corrosive Gases

In the event of the release of a corrosive gas (e.g. chlorine) or gases that are absorbed through the skin (e.g. hydrogen cyanide), a complete chemical resistant suit and a self-contained breathing apparatus are required. There is no practical means of absorbing or neutralizing a gas - the leak must be corrected at the source.

2.6.8 Mercury spill

If a small amount of mercury is spilled (e.g. broken thermometer), use an aspirator bulb or a mercury sponge to pick up droplets, place the mercury in a container, cover with water, seal it, and label the bottle appropriately. To clean up the residual micro-droplets that may have worked into cracks and other hard-to-clean areas, sprinkle sulphur powder or other commercially available product for mercury decontamination. Leave the material for several hours and sweep up solid into a plastic bag, seal it and label it appropriately.

3. Chemical Storage, Handing and Safety in Laboratory

3.1 General Storage Guidelines

- Do not block access to emergency safety equipment such as fire extinguishers, eyewashes, showers, first aid kits or utility controls such as breaker boxes or gas shut-off valves
- Avoid blocking exits or normal paths of travel: keep hallways, walkways and stairs clear of chemicals, boxes, equipment and shelf projections

- Ensure that the weight of stored material does not exceed the load-bearing capacity of shelves or cabinets
- Ensure that wall-mounted shelving has heavy-duty brackets and supports and is attached to studs or solid blocking. Regularly inspect clamps, supports, shelf brackets and other shelving hardware
- Arrange items so that they do not overhang or project beyond the edges of shelves or counter tops
- Do not stack materials so high that stability is compromised
- Leave a minimum of 18 inches (45.7 cm) of clearance between sprinkler heads and the top of storage
- Use a safety step or stepladder to access higher items; never stand on a stool or a chair

3.2 Ergonomics

- Store frequently used items between knee and shoulder height
- Store heavy objects on lower shelves

3.3 Chemical Storage

- Store hazardous chemicals in an area that is accessible only to authorized laboratory workers
- Minimize quantities and container sizes kept in the lab
- Do not store chemicals in aisles, under sinks or on floors, desks or bench tops
- Store chemicals away from sources of heat (e.g., ovens or steam pipes) and direct sunlight
- Never stack bottles on top of each other
- Do not store chemicals above eye level/shoulder height
- Store larger containers on lower shelves
- Store liquids inside chemically-resistant secondary containers (such as trays or tubs) that are large enough to hold spills
- Store chemicals inside closable cabinets or on sturdy shelving that has 12.7 mm-19 mm (½ - ¾ inch) edge guards to prevent containers from falling
- Ensure that chemicals cannot fall off the rear of shelves
- Store chemicals based on compatibility and not in alphabetical order, If a chemical presents more than one hazard, segregate according to the primary hazard
- Designate specific storage areas for each class of chemical, and return reagents to those locations after each use
- Store volatile toxic and odorous chemicals in a way that prevents release of vapours (e.g., inside closed secondary containers, ventilated cabinets, paraffin sealing)
- Store flammables requiring refrigeration in explosion-safe or lab-safe refrigerators
- Label reactive or unstable chemicals (e.g., ethers) with the date of receipt and the date opened
- Inspect chemicals weekly for signs of deterioration and for label integrity
- Dispose of unwanted chemicals promptly through Hazardous Waste Management
- Keep inventory records of chemicals, and update annually

3.4 Flammable liquid storage cabinets

Flammable chemicals should be stored inside flammable liquid storage cabinets. Only those flammables in use for the day should be outside the cabinet. Guidelines for cabinet use include:

- Use NFPA or UL approved flammable liquid storage cabinets
- Keep cabinet doors of the cabinet closed and latched
- Do not store other materials in these cabinets

3.5 Unstable chemicals

Many chemicals, most notably ethers (e.g., THF, dioxane, diethyl and isopropyl ether), are susceptible to decomposition resulting in explosive products. Ethers, liquid paraffins, and olefins form peroxides on exposure to air and light. Since most of these products have been packaged in an air atmosphere, peroxides can form even if the containers have not been opened.

- Discard unopened containers of ethers after one year
- Discard containers of ethers within six months of opening
- Never handle ethers beyond their expiry dates; contact your local waste disposal coordinator to arrange to have the material stabilized and removed

3.6 Explosive chemicals

Many chemicals are susceptible to rapid decomposition or explosion when subjected to forces such as being struck, vibrated, agitated or heated. Some become increasingly shock sensitive with age. Picric acid becomes shock sensitive and explosive if it dries out.

- Refer to the label and the Material Safety Data Sheet to determine if a chemical is explosive.
- Write the dates received and opened on all containers of explosive or shock-sensitive chemicals
- Inspect all such containers every month
- Keep picric acid solutions wet i.e., 30% or more water
- Discard opened containers after six months, and closed containers after one year, unless the material contains stabilizers
- Wear appropriate personal protective equipment and perform experiments behind face shield.
- Work with small quantities.

3.7 Workplace Hazardous Materials Information System (WHMIS)

Workplace Hazardous Materials Information System (WHMIS) is a Canada-wide system for providing information on the safe use of hazardous materials, referred to as *controlled products*, in the workplace. It is intended to protect the health and safety of workers by promoting access to information on hazardous materials; this information is provided by means of product labels, material safety data sheets (MSDS) and education programs.

WHMIS is governed by federal and provincial laws and regulations (Quebec's *Regulation respecting Information on controlled products (R.Q. c. S-2.1, r.10.1*) and any person supplying or using controlled products must comply with its requirements

Controlled products are products, materials, and substances that are regulated by WHMIS legislation, based on their hazardous properties and characteristics. WHMIS divides hazardous materials into six main categories or classes based on their characteristics.

The main objectives of WHMIS are hazard identification and product classification. WHMIS consists of three main components:

Labelling - Labels alert people to the dangers of the product and basic safety precautions. Any hazardous material, whether in transit, storage, or use, must be labelled. A label may be a mark, sign, stamp, device, sticker, ticket, tag, or wrapper and must be attached to, imprinted, stencilled, or embossed on the container of the controlled product. There are 2 types of labels supplier labels and workplace labels.

A supplier label must contain the following information:

- product identifier (name of product)
- supplier identifier (name of company that sold it)
- hazard symbols (WHMIS classification symbols)
- risk phrases (words that describe the main hazards of the product)
- precautionary statements (how to work with the product safely)
- first aid measures (what to do in an emergency)
- reference to the MSDS
- when the product are control in control condition, work place label must be donecontrolled products are produced, manufactured or prepared (e.g., stock solutions) at the workplace;
- the controlled product is transferred from the original container into another container; and
- the original supplier label becomes illegible or damaged or when it is removed;

A workplace label must contain the following information:

- product identifier (product name)
- information for the safe handling of the product
- reference to the MSDS

The product name must include the full name of the product or solution, as it appears on the material safety data sheet and include its concentration.

***Material Safety Data Sheets (MSDS)* -** Material Safety Data Sheets (MSDS) provide more details than labels. They are technical bulletins that provide chemical, physical, and toxicological information about each controlled product, as well as information on precautionary and emergency procedures. They must be readily accessible to anyone who works with, or who may otherwise be exposed to, controlled products.

Training **-** Training and education provides more detailed instruction on the specific procedures necessary to carry out work safely. WHMIS training is a major component of the WHMIS legislation and therefore is mandatory for all personnel working with controlled products

3.7.1 Understanding Hazard Warning Information

WHMIS Symbols

The classes of controlled chemical products and their corresponding symbols or pictograms, as well as general characteristics and handling precautions are outlined in table 1.

Table -1 Safe handling of Controlled Products. Summary of General Characteristics and Procedures for Handling and Storage of WHMIS-Controlled Products.

Class and Symbol	Characteristics	Precautions
Class A Compressed Gas 	• Gas inside cylinder is under pressure • The cylinder may explode if heated or damaged • Sudden release of high pressure gas streams may puncture skin and cause fatal embolis	• Transport and handle with care • Make sure cylinders are properly secured • Store away from sources of heat or fire • Use proper regulator
Class B Flammable and Combustible Material 	• May burn or explode when exposed to heat, sparks or flames • Flammable: burns readily at room temperature • Combustible: burns when heated	• Store away from Class C (oxidizing materials) • Store away from sources of heat, sparks and flame • Do not smoke near these materials
Class C Oxidizing Material 	• Can cause other materials to burn or explode by providing oxygen • May burn skin and eyes on contact	• Store away from Class B (flammable and combustible) materials • Store away from sources of heat and ignition • Wear the recommended protective equipment and clothing
Class D Poisonous and Infectious Material  **Division 1:** Materials Causing Immediate and Serious Toxic Effects	• May cause immediate death or serious injury if inhaled, swallowed, or absorbed through the skin	• Avoid inhaling gas or vapours • Avoid skin and eye contact • Wear the recommended protective equipment and clothing • Do not eat, drink or smoke near these materials • Wash hands after

		handling
Class D Poisonous and Infectious Material  **Division 2:** Materials Causing Other Toxic Effects	• May cause death or permanent injury following repeated or long-term exposure • May irritate eyes, skin and breathing passages: may lead to chronic lung problems and skin sensitivity • May cause liver or kidney damage, cancer, birth defects or sterility	• Avoid inhaling gas or vapours • Avoid skin and eye contact • Wear the recommended protective equipment and clothing • Do not eat, drink or smoke near these materials • Wash hands after handling
Class D Poisonous and Infectious Material  **Division 3:** Biohazardous Infectious Materials	• Contact with microbiological agents (e.g., bacteria, viruses, fungi and their toxins) may cause illness or death	• Wear the recommended protective equipment and clothing • Work with these materials in designated areas • Disinfect area after handling • Wash hands after handling
Class E Corrosive Material 	• Will burn eyes and skin on contact • Will burn tissues of respiratory tract if inhaled	• Store acids and bases in separate areas • Avoid inhaling these materials • Avoid contact with skin and eyes • Wear the recommended protective equipment and clothing
Class F Dangerously Reactive Material 	• May be unstable, reacting dangerously to jarring, compression, heat or exposure to light • May burn, explode or produce dangerous gases when mixed with incompatible materials	• Store away from heat • Avoid shock and friction • Wear the recommended protective equipment and clothing

3.8 Physical Hazards and Ergonomics

3.8.1 Electrical Safety

- All electrical outlets should carry a grounding connection requiring a three-pronged plug.
- Never remove the ground pin of a three-pronged plug.
- Remove cords by grasping the plug, not the cord.

- All wiring should be done by, or under the approval of, a licensed electrician.
- Electrical equipment that has been wetted should be disconnected at the main switch or breaker before being handled. Familiarize yourself with the location of such devices.
- Know how to cut off the electrical supply to the laboratory in the event of an emergency.
- Ensure that all wires are dry before plugging into circuits.
- Electrical equipment with frayed wires should be repaired before being put into operation.
- Tag and disconnect defective equipment.
- Be sure that all electrical potential has been discharged before commencing repair work on any equipment containing high voltage power supplies or capacitors.
- Use only $C0_2$, halon , or dry chemical fire extinguishers for electrical fires.

3.8.2 Glassware Safety

- Use a dustpan and brush, not your hands, to pick up broken glass.
- Discard broken glass in a rigid container separate from regular garbage and label it appropriately.
- Protect glass that is subject to high pressure or vacuum. Wrapping glass vessels with cloth tape will minimize the possibility of projectiles.
- Glass is weakened by everyday stresses such as heating and bumping. Handle used glassware with extra care.
- Discard or repair all damaged glassware, as chipped, cracked or star-cracked vessels cannot handle the normal stresses.

3.8.3 High Pressure and Vacuum Work

Pressure differences between equipment and the atmosphere result in many lab accidents. Glass vessels under vacuum or pressure can implode or explode, resulting in cuts from projectiles and splashes to the skin and eyes. Glass can rupture even under small pressure differences. Rapid temperature changes, such as those that occur when removing containers from liquid cryogenics, can lead to pressure differences, as can carrying out chemical reactions inside sealed containers.

The hazards associated with pressure work can be reduced by:

- checking for flaws such as scratches and etching marks before using vacuum apparatus
- using vessels specifically designed for vacuum work. Thin-walled or round-bottomed flasks larger than 1 L should never be evacuated
- taping glass vacuum apparatus to minimize projectiles due to implosion
- using adequate shielding when conducting pressure and vacuum operations
- allowing pressure to return to atmospheric before opening vacuum desiccators or after removal of a sample container from cryogenics
- wearing eye and face protection when handling vacuum or pressure apparatus

3.8.4 Equipment Safety

Whenever lab equipment is purchased, preference should be given to equipment that

- limits contact between the operator and hazardous material, and mechanical and electrical energy
- is corrosion-resistant, easy to decontaminate and impermeable to liquids
- has no sharp edges or burrs

Every effort should be made to prevent equipment from becoming contaminated. The following are precautions and procedures to be observed with some commonly used laboratory equipment.

3.8.4.1 Analytical Equipment

The following instructions for safe use of analytical equipment are general guidelines; consult the user's manual for more detailed information on the specific hazards:

- Ensure that installation, modification and repairs of analytical equipment are carried out by authorized service personnel.
- Read and understand the manufacturer's instructions before using this equipment.
- Make sure that preventive maintenance procedures are performed as required.
- Do not attempt to defeat safety interlocks.
- Wear safety glasses and lab coats (and other appropriate personal protective equipment as specified) for all procedures.

3.8.4.2 Ovens and Hot Plates

Laboratory ovens are useful for baking or curing material, off-gassing, dehydrating samples and drying glassware.

- Discontinue use of any oven whose backup thermostat, pilot light or temperature controller has failed
- Avoid heating toxic materials in an oven unless it is vented outdoors (via a canopy hood, for example)
- Never use laboratory ovens for preparation of food for human consumption
- Glassware that has been rinsed with an organic solvent should be rinsed with distilled water before it is placed in a drying oven

3.8.4.3 Atomic Absorption Spectrometers (AAS)

Sample preparation for atomic absorption procedures often require handling of flammable, toxic and corrosive products. Familiarize yourself with the physical, chemical and toxicological properties of these materials and follow the recommended safety precautions. Atomic absorption equipment must be adequately vented, as toxic gases, fumes and vapours are emitted during operation. Other recommendations to follow when carrying out atomic absorption analysis are:

- Wear safety glasses for mechanical protection.
- Check the integrity of the burner, drain and gas systems before use.
- Inspect the drain system regularly; empty the drain bottle frequently when running organic solvents.
- Allow the burner head to cool to room temperature before handling.
- Never leave the flame unattended. A fire extinguisher should be located nearby.
- Avoid viewing the flame or furnace during atomization unless wearing protective eyewear.

- Hollow cathode lamps are under negative pressure and should be handled with care and disposed of properly to minimize implosion risks.

3.8.4.4 Mass Spectrometers (MS)

Mass spectrometry requires the handling of compressed gases and flammable and toxic chemicals. Consult MSDSs for products before using them. Specific precautions for working with the mass spectrometer include:

- Avoid contact with heated parts while the mass spectrometer is in operation.
- Verify gas, pump, exhaust and drain system tubing and connections before each use.
- Ensure that pumps are vented outside the laboratory, as pump exhaust may contain traces of the samples being analyzed, solvents and reagent gas.
- Used pump oil may also contain traces of analytes and should be handled as hazardous waste.

3.9 Personal Protective Equipment

University's policies regarding eye and face protection (Section 11.1) and protective clothing (Section 11.2) are outlined below.

3.9.1 Eye and Face Protection

All students, staff, faculty and visitors must wear appropriate eye and/or facial protection in the following:

- All areas where hazardous materials, or substances of an unknown nature, are stored, used or handled
- All areas where the possibility of splash, flying objects, moving particles and/or rupture exist
- All areas where there are other eye hazards, e.g. UV or laser light

Instructions for selection and use of protective eyewear are as follows:

- Light-to-moderate work: approved safety glasses with side shields.
- Work with significant risk of splash of chemicals, or projectiles: goggles.
- Work with significant risk of splash on face, or possible explosion: full face shield, plus goggles.

3.9.2 Lab Coats

Appropriate protective clothing (e.g., lab coats, aprons, coveralls) is required in all experimental areas where hazardous materials are handled.

Instructions for selection and use of protective laboratory clothing are as follows:

- select knee-length lab coats with button or snap closures
- wear a solid-front lab coat or gown with back closures and knitted cuffs when working with highly toxic or infectious agents

- wear protective aprons for special procedures such as transferring large volumes of corrosive material
- remove protective clothing when leaving the laboratory
- remove protective clothing in the event of visible or suspected contamination

3.9.3 Hand Protection

In the laboratory, gloves are used for protection from radiation, chemical products, biohazardous material and physical hazards such as abrasion, tearing, puncture and exposure to temperature extremes.

3.9.4 Glove Selection Guidelines

Base selection of glove material on:

- identification of the work procedures requiring hand protection
- type and length of contact (e.g., occasional or splash vs. prolonged or immersion contact)
- whether disposable or reusable gloves are more appropriate

Table-2 Recommended Glove Materials for a Variety of Laboratory Hazards

Hazard	**Degree of Hazard**	**Recommended Material**
Abrasion	Severe	Reinforced heavy rubber, staple-reinforced leather
	Less severe	Rubber, plastic, leather, polyester, nylon, cotton
Sharp edges	Severe	Metal mesh, staple-reinforced heavy leather, Kevlar, aramid-steel
	Less severe	Leather, terry cloth (aramid fibre)
	Mild with delicate work	Lightweight leather, polyester, nylon, cotton
Chemicals and liquids	Varies depending on the concentration, contact time, etc. Consult MSDS, manufacturer or permeation chart	Choice depends on chemical. *Examples*: natural, nitrile or butyl rubber, neoprene, PTFE (polytetrafluoroethylene), polyvinyl chloride, polyvinyl alcohol, Teflon™, Viton™, Saranex™, 4H™, Chemrel™, Barricade™, Responder™
Cold	Leather, insulated plastic or rubber, wool, cotton	
Heat	Over 350°C	Asbestos Zetex™
	Up to 350°C	Neoprene-coated asbestos, heat-resistant leather with linings, Nomex, Kevlar™
	Up to 200°C	Heat-resistant leather, terry cloth (aramid fibre) Nomex, Kevlar™
	Up to 100°C	Chrome-tanned leather, terry cloth

Electricity	Rubber-insulated gloves tested to appropriate voltage (CSA Standard Z259.4-M1979) with leather outer glove	
General duty	Cotton, terry cloth, leather	
Product contamination	Thin-film plastic; lightweight leather, cotton, polyester, nylon	

Chemical Glove Selection

No single glove material is resistant to all chemicals, nor will most gloves remain resistant to a specific chemical for longer than a few hours. Determine which gloves will provide a acceptable degree of resistance by consulting the MSDS for the product, contacting glove manufacturers or by referring to a compatibility chart or table for permeation data. These resources may use the following terms:

- "permeation rate" refers to how quickly the chemical seeps through the intact material: the higher the permeation rate the faster the chemical will permeate the material;
- "breakthrough time" refers to how long it takes the chemical to seep through to the other side of the material, and
- "degradation" is a measure of the physical deterioration (for example, glove material may actually dissolve or become harder, softer or weaker) following contact with the chemical

Selection, Use and Care of Protective Gloves

Guidelines for glove use include the following:

- choose a glove that provides adequate protection from the specific hazard(s)
- be aware that some glove materials may cause adverse skin reactions in some individuals and investigate alternatives
- inspect gloves for leakage before using; test rubber and synthetic gloves by inflating them
- make sure that the gloves fit properly
- ensure that the gloves are long enough to cover the skin between the top of the glove and the sleeve of the lab coat
- discard worn or torn gloves
- discard disposable gloves that are, or may have become, contaminated
- avoid contaminating "clean" equipment: remove gloves and wash hands before carrying out tasks such as using the telephone
- always wash your hands after removing gloves, even if they appear not to be contaminated
- do not reuse disposable gloves
- follow the manufacturer's instructions for cleaning and maintenance of reusable gloves
- before using gloves, learn how to remove them without touching the contaminated outer surface with your hands

3.9.5 Respirators

Respirators should be used only in emergency situations (e.g. hazardous spills or leaks) or when other measures, such as ventilation, cannot adequately control exposures.

There are two classes of respirators: air-purifying and supplied-air. The latter supply clean air from a compressed air tank or through an air line outside the work area, and are used in oxygen-deficient atmospheres or when gases or vapours with poor warning properties are present in dangerous concentration.

Air-purifying respirators are suitable for many laboratory applications and remove particulates (dusts, mists, metal fumes etc.) or gases and vapours from the surrounding air.

3.10 Fire Safety

Laboratory fires can by caused by bunsen burners, runaway chemical reactions, electrical heating units, failure of unattended or defective equipment, or overloaded electrical circuits. Remember the laboratory staff with the operation of the fire extinguishers and the location of pull stations, emergency exits and evacuation routes where there work . In the event that the general alarm is sounded use the evacuation routes established for your area and follow the instructions of the Evacuation Monitors. Once outside of the building, move away from the doors to enable others to exit.

3.10.1 Fire Extinguishers

Fire extinguishers are rated as A, B, C or D (or combinations of A, B, C and D) for use against the different classes of fires. Remember the laboratory staff with the class ratings of the extinguishers in their work area so that they will know what types of fire they can attempt to extinguish with them.

Learn how to use the extinguisher in their lab, as there will be no time to read instructions during an emergency. Attempt to fight small fires only, and only if there is an escape route behind the worker. Remember to have the extinguisher recharged after every use. If encountering with the fire fight, remember the acronym "PASS" when using the extinguisher:

- **P**: Pull and twist the locking pin to break the seal.
- **A**: Aim low, and point the nozzle at the base of the fire.
- **S**: Squeeze the handle to release the extinguishing agent.
- **S**: Sweep from side to side until the fire is out.
- Be prepared to repeat the process if the fire breaks out again

3.10.2 Preventing Fires

Use the following precautions when working with or using flammable chemicals in a laboratory; keep in mind that these precautions also apply to flammable chemical waste.

- Minimize the quantities of flammable liquids kept in the laboratory.
- Use and store flammable liquids and gases only in well-ventilated areas. Use a fume hood when working with products that release flammable vapours.

- Keep flammable solvent containers, including those for collecting waste, well capped. Place open reservoirs or collection vessels for organic procedures like HPLC inside vented chambers.
- Store flammable chemicals that require refrigeration in "explosion-safe" (non-sparking) laboratory refrigerators.
- Keep flammable chemicals away from ignition sources, such as heat, sparks, flames and direct sunlight. Avoid welding or soldering in the vicinity of flammables.
- Use portable safety cans for storing, dispensing and transporting flammable liquids.
- Clean spills of flammable liquids promptly.

3.11 Hazardous Waste Disposal

3.11.1 Hazardous Waste Disposal Guidelines

- Label all waste materials completely and legibly, using labels available from ***Hazardous Waste Management***. Inadequately labeled containers will not be accepted.
- Package waste materials in approved containers, available from HWM.
- Over filled and/or leaking containers cannot be accepted for disposal.
- Never discharge wastes into the sewer unless you have verified that hazardous wastes regulations permit you to do so. For information, contact HWM

3.11.2 Waste Minimization

In order to minimize the amount of hazardous waste presented for disposal, it is important to follow these guidelines:

- *Avoid overstocking*: one of the main sources of laboratory waste is surplus stock - the result of over buying. Recent pricing arrangements with suppliers have greatly reduced the benefits of purchasing chemicals in large volumes. Also, there is little need to store large quantities of chemicals, as orders are generally shipped the day after an order is received.
- *Do not accept donations of materials* that you don't plan to use. Many companies have traditionally unloaded unwanted reagents by donating them to laboratories, which eventually transfers the cost of disposal to the University.
- *Substitute hazardous experimental materials* for non-hazardous ones. For example, use aqueous-based, biodegradable scintillation fluids whenever possible.

3.11.3 Waste Preparation Procedures

Chemical Waste

Chemical or unknown composition cannot be accepted If the material is older than one year, do not attempt to open or move the container.

Organic Solvents and Oils

- Collect in the containers provided by *Hazardous Waste Management (HWM, local 5066)*.
- Indicate the composition of the contents as accurately as possible on the attached label.

Peroxide-forming (e.g. ether) and explosive (e.g. dry picric acid) chemicals

- Do not mix with solvents or other waste.
- If the material is older than one year, do not attempt to open or move the container. Contact HWM consultant for advice.

Corrosives (acids and bases)

- Collect acids (pH<7) and bases (pH>7) separately in the plastic containers provided by HWM. Do not mix acids with bases.
- Indicate the composition of the contents, as accurately as possible, on the attached label.

Broken glassware (uncontaminated)

- Designate a cardboard box for broken glass; label it "BROKEN GLASS", and place glass inside. When the box is full, seal it with tape and place it next to the garbage receptacle for pickup by the cleaning staff.

Empty chemical reagent bottles

- Remove the cap from the empty bottle and allow volatile materials to evaporate into the fume hood.
- Rinse the bottle three times with tap water and let dry.
- Remove or obliterate the label.
- Place the uncapped bottle next to the garbage receptacle.

3.12 Laboratory Ventilation and Fume Hoods

3.12.1 General Ventilation

General ventilation, also called dilution ventilation, involves dilution of inside air with fresh outside air, and is used to:

- maintain comfortable temperature, humidity and air movement for room occupants
- dilute indoor air contaminants
- replace air as it is exhausted to the outside via local ventilation devices such as fume hoods

General ventilation systems comprise an air supply and an air exhaust. The air may be supplied via a central HVAC (Heating, Ventilation and Air Conditioning) system or, especially in older buildings, via open able windows. Laboratory air may be exhausted through either local exhaust devices or air returns connected to the HVAC system.

3.12.2 Local Ventilation Devices

Local exhaust ventilation systems capture and discharge air contaminants (biological, chemical, radioactive) or heat from points of release. Common local exhaust ventilation devices found in laboratories include:

- chemical fume hoods
- biological safety cabinets

- direct connections

3.12.3 Chemical Fume Hoods

Chemical fume hoods are enclosed units with a sliding sash for opening or closing the hood. They are able to capture and exhaust even heavy vapours, and are preferred for all laboratory procedures that require manual handling of hazardous chemical material.

3.12.4 Biological Safety Cabinets

Biological safety cabinets are for use with biological material; depending on the cabinet class, they provide protection of the environment, user and/or product. They are not recommended for use with hazardous chemicals because most models recirculate air into the laboratory, and because the HEPA filter that is integral to the protective function can be damaged by some chemicals.

Direct connections

Direct connections provide direct exhausting of contaminants to the outdoors and are used for venting:

- flammable liquid storage cabinets
- other toxic chemical storage cabinets
- solvent and waste reservoirs, such as for HPLC solvent systems
- reaction vessels, sample analyzers, ovens, dryers and vacuum pump outlets

3.13 Compressed Gases and Cryogenics

3.13.1 Hazards of Compressed Gases

Compressed gases are hazardous due to the high pressure inside cylinders. Knocking over an unsecured, uncapped cylinder of compressed gas can break the cylinder valve; the resulting rapid escape of high pressure gas can turn a cylinder into an uncontrolled rocket or pinwheel, causing serious injury and damage. Poorly controlled release of compressed gas in the laboratory can burst reaction vessels, cause leaks in equipment and hoses or result in runaway chemical reactions. Compressed gases may also have flammable, oxidizing, dangerously reactive, corrosive or toxic properties. Inert gases such as nitrogen, argon, helium and neon can displace air, reducing oxygen levels in poorly ventilated areas and causing asphyxiation.

Safe handling, storage and transport of compressed gas cylinders

- All gas cylinders, full or empty, should be securely supported using suitable racks, straps, chains or stands.
- When cylinders are not in use or are being transported, remove the regulator and attach the protective cap.
- An appropriate cylinder cart should be used for transporting cylinders. Chain or strap the cylinder to the cart.
- Verify that the regulator is appropriate for the gas being used and the pressure being delivered. Do not rely upon the pressure gauge to indicate the maximum pressure ratings; check the regulator's specifications.

- Do not use adaptors or Teflon tape to attach regulators to gas cylinders.
- Never bleed a cylinder completely empty; leave a residual pressure.
- Do not lubricate the high-pressure side of an oxygen regulator.
- Do not expose cylinders to temperature extremes.
- Store incompatible classes of gases separately.

3.13.2 Cryogenic Hazards

Cryogenics are very low temperature materials such as dry ice (solid CO2) and liquefied air or gases like nitrogen, oxygen, helium, argon and neon. The following hazards are associated with the use of cryogenics:

- asphyxiation due to displacement of oxygen (does not apply to liquid air and oxygen)
- embrittlement of materials from extreme cold
- frostbite
- explosion due to pressure build up
- condensation of oxygen and fuel (e.g. hydrogen and hydrocarbons) resulting in explosive mixtures

Cryogenic Handling Precautions

The following are precautions for handling cryogenics:

- Control ice build up
- Use only low-pressure containers equipped with pressure-relief devices.
- Protect skin and eyes from contact; wear eye protection and insulated gloves.
- Use and store in well-ventilated areas.
- Keep away from sparks or flames.
- Use materials resistant to embrittlement (e.g. latex rubber tubing).
- Watches, rings, bracelets or other jewelry that could trap fluids against flesh should not be worn when handling cryogenic liquids
- To prevent thermal expansion of contents and rupture of the vessel, do not fill containers to more than 80% of capacity.
- If cryogens must be transported by elevator, take adequate precautions to prevent possible injury. Send cryogenic liquid tanks in elevators without any passengers and ensure that nobody gets on the elevator while the cryogen is being transported.

3.14 Emergency Procedures

First Aid

Know how to handle emergency situations before they occur:

- Become familiar with the properties of the hazardous products used in working area.
- Familiarize laboratory staff with the contents of the first aid kit and learn how to use them. Keep instructions readily available and easy to understand.
- Locate and know how to test and operate emergency equipment, such as showers and eyewashes, in working area.
- Learn about first aid and hot line in working area.

The emergency first aid procedures described below should be followed by a consultation with a physician for medical treatment.

Burns

In the laboratory, thermal burns may be caused by intense heat, flames, molten metal, steam, etc. Corrosive liquids or solids such as bases and acids can cause chemical burns; first aid treatment for chemical burns is described below.

Burns to the Skin

First aid treatment of skin burns encompasses the following:

- If the burn is electrical in origin, ascertain that the victim is not in contact with the power supply before touching him/her. If the victim remains in contact with a power source, unplug the device or shut off the main power switch at the electrical distribution panel.
- Dial hot line- fire station Ahlone ph: 220802, Kyeemyindine ph: 534825, Sanchaung ph: 527099, 536687, if the burn is serious. Seek immediate medical treatment for all electrical burns, even if they don't appear to be serious.
- Remove jewelry, including watches, from the burned area.
- Expose the burnt area, but avoid removing clothes that are stuck to the skin.
- If possible, immerse burnt surfaces in cold water for at least 10 minutes, or apply cold wet packs.
- Avoid applying lotions, ointments or disinfectants to a burn. First and second degree burns can be washed with soap and water after the cool down period.
- Cover first and second degree burns with a moist bandage; apply dry compresses to third degree burns and to entry and exit wounds of electrical burns.
- Do not burst blisters, as they form a natural barrier against infection.

LABORATORY PROJECT AND EXPERIMNT

1. General sampling Procedure

1.1 General Equipment

Use only specified equipment, including sample containers and other sampling equipment. In particular, laboratory supplied containers must be used as specified: the use of alternative sample containers or sampling methods will make the sample unusable and the laboratory may reject incorrect samples.

1.2 Equipment calibrating, cleaning and maintenance

Ensure that sampling equipment is clean and is maintained in good working order before use and at the end of sampling. Generally, it will not need to clean sampling equipment thoroughly, apart from rinsing it at the end of each sampling trip. However, if a site that is particularly contaminated (e.g. if there is an algal bloom, or the site smells strongly of hydrocarbons, sewage or something else) is sampled the equipment must be rinsed prior to sampling at the next site; or ideally leave that site until the end of the sampling run in order to avoid cross contamination with subsequent samples. Keep some spare deionised/distilled/filtered water for this purpose. Equipment must be cleaned periodically to prevent a build-up of dirt. To do this:

- rinse the equipment well in tap water
- clean with De-Con 90 (a phosphate free detergent)
- rinse well with tap water
- rinse three times with de-ionised water
- allow to dry.

Ensure all field measurement instruments are fully calibrated before starting sampling (pre-field) and again once all sampling has been completed (post-field). The results of the calibration should be marked in a calibration information box on the field observations form (FOF).

It is preferable to use new, pre-cleaned sampling containers to store samples, but if existing ones need to be re-used, rinse with detergent (De-Con 90 is recommended), then very thoroughly wash and rinse with deionised or distilled water. De-Con 90 is an antibacterial/microbial reagent and is useful for cleaning and/or decontaminating glassware, ceramics, rubbers, plastics, stainless steel and ferrous metals. De-Con 90 is not suitable for use on non-ferrous metals, notably aluminium and zinc, or on polycarbonate. Other washing solvents include dilute hydrochloric acid (HCl) (0.1 moles/L HCl), which can remove metal contaminants, and dilute ethanol or methanol (5% in distilled water) which can be used to remove organic contaminants (only important if sampling for metals or organic parameters).

Important: It is essential that the containers are washed and rinsed very thoroughly with deionised or distilled water after using any of the above described solvents to remove completely any trace of these solvents before sampling commences. The deionised/distilled/filtered water unit must be checked to ensure it is well cleaned and maintained and serviced regularly. Be aware that when using deionised or distilled or filtered water for blanks and for rinsing equipment, that this water is free of contaminants. Ensure that dispensers of this water are maintained regularly and filters cleaned to ensure that they produce non-contaminated water. A good practice is to purchase deionised water from the analytical laboratory for sample analysis.

1.3 Sampling and Sample Handling

Regardless of the intent, all numbers generated by a water quality laboratory are ultimately represented as the concentration levels in the sample matrix at the time of collection. Such numbers tend, automatically, to endorse the sample collection, preservation, and shipment procedures. Thus, quality assurance programs limited to the care of the sample beginning with its receipt by the laboratory are inadequate. The laboratory must share responsibility for the preservation and shipment of all samples that it will accredit with concentration values.

Two approaches are available that will generally protect the laboratory from generating numbers that may not reflect actual conditions of the sample at the time of collection. The best, but usually least practical solution, is for the laboratory personnel to collect all samples. The alternative is for the laboratory to adopt a policy of sample rejection based on minimum standards of sample identification and age.

Guidelines for establishing these standards are discussed in this section. It is recommended that copies of this material be supplied to all sample collectors along with an understanding of the specific policy of the laboratory toward rejecting samples that do not meet these criteria. In all of the cases to be discussed, it is the responsibility of worker to (a) coordinate his sampling, preservation, and shipment with the laboratory, (b) obtain clean sample containers from the laboratory, (c) provide adequate sample identification and compositing instructions, and (d) provide duplicates and blanks as required by the laboratory. Additional prearrangements should be made with the laboratory if sample splitting is desired, to create separate supernatant and settleable matter samples, or if calculations on a wet-weight basis in addition to the standard dry-weight calculations are desired.

Bottles and caps are to be supplied by the laboratory because rigorous cleaning is required even for new bottles. New and recycled bottles should be washed as described in reference

1. Samples received in bottles of unknown origin or questionable cleanliness should be rejected by the laboratory. For water samples that are to be analyzed by solvent extraction methods, narrow-mouthed, screw-cap bottles such as Boston rounds are preferred because they have less tendency to leak and are easy to handle in the laboratory.

Multiple samples are usually required for purgeable organics analysis because of leakage and because the measurement process is destructive to the sample. All vials should be identified with waterproof labels. The water sample vials are filled to overflowing from a bubble-free source so that a convex meniscus is formed at the top. They are sealed by carefully placing the septum, Teflon (du Pont) side down on the opening of the vial and screwing the cap firmly in place.

All samples for organic analysis should arrive at the laboratory on the same day collected, or should be shipped and maintained at less than 4°C for arrival by the next day. The samples are usually shipped in insulated ice chests. Water samples should be prechilled before packing to reduce the ice requirements during shipment. The bottles should be stabilized in the container with styrofoam and covered with ice. The information needed to identify the samples should be attached to the outside of the ice chest.

Water samples that clearly have not met the preservation criteria during shipment (e.g., in the case of a spill) should be accepted only if resampling is impossible. Results from such samples must be qualified in the laboratory report.

1.4 Sample preservation

Equivocal and unequivocal preservation of samples, either domestic sewage, industrial wastes, or natural waters, is a practical impossibility. Regardless of the nature of the sample, complete stability for every constituent can never be achieved. At best, preservation techniques can only retard the chemical and biological changes that inevitably continue after the sample is removed from the parent source. The changes that take place in a sample are either chemical or biological. In the former case, certain changes occur in the chemical structure of the constituents that are a function of physical conditions. Biological changes taking place in a sample may change the valence of an element or a radical to a different valence. Soluble constituents may be converted to organically bound materials in cell structures, or cell lysis may result in release of cellular material into solution. The well known nitrogen and phosphorus cycles are examples of biological influence on sample composition.

Methods of preservation are relatively limited and are intended generally to (1) retard biological action, (2) retard hydrolysis of chemical compounds and complexes and **(3)** reduce volatility of constituents.

Preservation methods are generally limited to pH control, chemical addition, refrigeration, and freezing. Table I shows the various preservatives that may be used to retard changes in samples.

2. Chemical Parameter to Measure

2.1 Physical Parameter

2.1.1 Electrical Conductivity

Electrical conductivity is the measure of the ability of water to conduct an electric current and depends upon the number of ions or charged particles in the water, and is measured by passing a current between two electrodes (a known distance apart) that are placed into a sample of water. The unit of measurement for electrical conductivity is expressed in either micro Siemens per centimetre (μS/cm) or milli Siemens per centimetre (mS/cm). Electrical conductivity determinations are useful in aquatic studies because they provide a direct measurement of dissolved ionic matter in the water. Low values are characteristic of high-quality, low-nutrient waters. High values of conductance can be indicative of salinity problems but also are observed in eutrophic waterways where plant nutrients (fertiliser) are in greater

abundance. Very high values are good indicators of possible polluted sites. A sudden change in electrical conductivity can indicate a direct discharge or other source of pollution into the water.

However, electrical conductivity readings do not provide information on the specific ionic composition and concentrations in the water.

Table –3 Sampling Procedures for Electrical Conductivity

Collection technique using hand-held meter – *in situ* field measurement	Meter should be kept in gentle motion through the water column while a reading is being taken. Allow several minutes for the meter to stabilise. Ideally, measurements should be made about 10 cm below the water surface (and then about 10 cm above the sediment surface); however, this is not always possible in shallow water bodies. A mid water column reading will be sufficient in these cases
Sample collection technique for laboratory analysis at 25°C	Unfiltered sample
Volume	125 mL
Container	Plastic A Bottle cap must have a teflon liner Use new pre-cleaned bottles
Collection technique	Direct collection into sample bottle or transfer into a sample bottle from collection vessel. Ensure sample bottle is pre-rinsed three times with sample water (3 × 20 mL) before final collection.
Treatment to assist preservation	Refrigerate at 1–4°C, do not freeze
Filling technique	Excessive turbulence should be avoided to minimise presence of air bubbles in the sample. Fill container completely to the top to exclude air. The sample must be free of air bubbles and capped tightly.
Maximum sample holding time and storage conditions	Analyse within 24 hours for samples of low conductivity, i.e. below 20 µS/cm. Other samples can be held for one month if sample is kept refrigerated at 1–4°C and stored in an airtight container.
Units of measurement	µS/cm (or mS/cm).
Analysis method	Conductivity is measured electrometrically with (or without) temperature compensation and is calibrated against a standard solution of potassium chloride. *Measurement of Conductivity Method 2510* (APHA, 1998).
Comments	It is preferable to perform this test in the field.

2.1.2 Salinity

In measuring the salinity of water, we consider the concentration of salt dissolved in the water. Concentrations are usually expressed in parts per thousand (PPT) which can also be donated by the symbol ‰ (per mille). These are the classes of salinity we use for water:

fresh water – less than 5

brackish water– from 5 ‰ to 25 ‰

saline water – from 25 ‰ to 36 ‰

super-saline (or hyper-saline) water – greater than 36 ‰ (more saline than seawater

Open ocean salinities are generally in the range between 32 ‰ and 37 ‰.

Units of Measurement

All water that has not been deionised or distilled contains some salt. Salt concentration is often described in units of parts per thousand (ppt), ppm, milligrams per litre (mg/L) or per cent. The relationship between these units is: 1 ppt = 1,000 ppm = 1000 mg/L = 0.1 per cent. Salinity is also expressed in practical salinity units (psu), a measure of conductivity at a constant pressure and temperature that is about equivalent to ppt.

Conductivity Method

The electrical conductivity of water is proportional to its concentration of electrically conductive salt ions. Conductivity, the amount of electrical current that can pass through the water, is easily measured with a hand-held device called a conductivity probe or meter. Conductivity then can be converted to salinity if temperature and pressure are also known. Some salinity-measuring devices do this conversion but are not accurate at concentrations higher than about 70,000 ppm.

Hydrometer Method

Water's density, or specific gravity, increases in proportion to its salt concentration. Temperature also affects the density of water and is needed to convert specific gravity to salinity. Specific gravity can be measured using a hydrometer, a calibrated glass tube that is designed to float in a sample of water. The depth at which the hydrometer sits at the waterline determines the specific gravity of the sample. Then a "table," such as the one linked in the Resources section, can be used to determine water salinity.

Refractometer Method

Refractometers estimate salinity by measuring the degree to which a water sample refracts light in comparison to a pure water sample. After a few drops of water are placed on the daylight plate, the salinity value can be read through the scope.

2.1.3 Temperature

Temperature can be measured using a thermometer with a range of 0–50°C or a suitable electronic thermometer. The probe (or thermometer) is placed in the water to be measured. The temperature is measured after the reading has stabilised: this may take several minutes.

Since the solubility of dissolved oxygen decreases with increasing water temperature, high water temperatures limit the availability of dissolved oxygen for aquatic life. In addition, water temperature regulates various biochemical reaction rates that influence water quality. Heat sources and sinks to a water body include incident solar radiation, back radiation, evaporative cooling and heat conduction, thermal dischargers (e.g. cooling water from power plants), tributary inflows and groundwater discharge.

2.1.4 Turbidity

Scope and Application

This method is applicable to drinking, surface, and saline waters in the range of turbidity from 0 to 40 nephelometric turbidity units (NTU). NOTE 1 : NTU's are considered comparable to the previously reported Formazin Turbidity Units (FTU) and Jackson Turbidity Units (JTU).

Summary of Method

The method is based upon a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension. The higher the intensity of scattered light, the higher the turbidity. Readings, in NTU's, are made in a nephelometer designed according to specifications outlined in Apparatus *5*. A standard suspension of Formazin, prepared under closely defined conditions, is used to calibrate the instrument.

Formazin polymer is used as the turbidity reference suspension for water because it is more reproducible than other types of standards previously used for turbidity standards.

2.1.5 Oil and Grease

Scope and Application

This method includes the measurement of Freon extractable matter from surface and saline waters, industrial and domestic wastes. It is applicable to the de termination of relatively non-volatile hydrocarbons, vegetable oils, animal fats, waxes, soaps, greases and related matters.

The method is not applicable to measurement of light hydrocarbons that volatilize at temperatures below 70°C. Petroleum fuels from gasoline through #2 fuel oil are completely or substantially lost in the solvent removal operation.

The method covers the range from 5 to 1000 mg/l of extractable material.

Summary of Method

The sample is acidified to a low pH (<2) to remove the oils and greases from solution. After they are isolated by filtration, they are extracted with Freon using a Soxhlet extraction. The solvent **is** evaporated from the extract and the residue weighed.

Definitions

The definition of grease and oil is based on the procedure used. The source of the oil and/or grease, and the presence of extractable non-oily matter will influence the material measured and interpretation of results.

2.2 Chemical Parameter

2.2.1 pH

Scope and Application

This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.

Summary of Method

The pH of a sample is an electrometric measurement, using either a glass electrode in combination with a reference potential (saturated calomel electrode) or a combination electrode (glass and reference).

Comments

The sample must be analyzed as soon as practical; preferably within a few hours. Do not open sample bottle before analyses.Oil and greases, by coating the pH electrode, may interfere by causing sluggish response.At least three buffer solutions must be used to initially standardize the instrument. They should cover the pH range of the samples to be measured. Field pH measurements using comparable instruments are reliable.

pH Electrodes

A wide variety of special- and general-purpose pH electrodes are now available to meet all applications in the general analytical laboratory. A survey through any laboratory supply catalog may confuse more than clarify the selection process. A rugged, full-range, glassor plastic-bodied combination electrode is a good choice for routine use. An added convenience is an electrode that contains solid geltype filling materials not requiring the normal maintenance of an electrode containing liquid filling solutions.

2.2.2 Dissolved oxygen (DO) (The Winker method)

The Winkler Method is a technique used to measure dissolved oxygen in freshwater systems. Dissolved oxygen is used as an indicator of the health of a water body, where higher dissolved oxygen concentrations are correlated with high productivity and little pollution. This test is performed on-site, as delays between sample collection and testing may result in an alteration in oxygen content.The Winkler Method uses titration to determine dissolved oxygen in the water sample. A sample bottle is filled completely with water (no air is left to skew the results). The dissolved oxygen in the sample is then "fixed" by adding a series of reagents that form an acid compound that is then titrated with a neutralizing compound that results in a color change. The point of color change is called the "endpoint," which coincides with the dissolved oxygen concentration in the sample. Dissolved oxygen analysis is best done in the field, as the sample will be less altered by atmospheric equilibration.

Applications of DO analysis

Dissolved oxygen analysis can be used to determine:

- the health or cleanliness of a lake or stream,
- the amount and type of biomass a freshwater system can support,
- the amount of decomposition occurring in the lake or stream.

Sampling

Dissolved oxygen concentrations may change drastically in lakes depending upon depth and distance from shore. Sampling stations and depths should be selected according to whether or not you are trying to measure these differences or not. If just one surface station is being measured, pick a station near the middle of the lake and collect the sample at arm’s length below the water surface. When collecting stream DO samples at several stations for comparison, it is important to select stations with similar flow conditions. Do not select one station in a slow-moving pool and another in a riffle area (unless of course one of your objectives is to measure these differences). The best sites are smooth-flowing – like the "glide" area between riffles and pools.

DO samples should represent average conditions in the stream reach being measured. A sample collected in the middle of the stream at least a few inches below the water surface is a safe bet. If the sample must be collected from the shore, be sure to pick a site where there is enough current to ensure adequate mixing – don't sample from stagnant, slow-moving water if it is not representative of the stream segment.

Assuming your objective is to compare measurements between stations or between seasons, DO samples should be collected at nearly the same time of day each time you sample. Otherwise, the daily variations in DO concentration that were described in Chapters Two and Three may mask changes due to other factors. The time of sampling and water temperature should be recorded. This problem with daily variations in DO (and other parameters) also comes into play if you sample more than one station. For example, if it takes a full day to accomplish the entire monitoring effort, then by default some stations will be sampled in mid-morning, while others will be sampled in mid-afternoon. To retain as much consistency as possible in the data collected, always sample your stations in the same order.

Reagent List:

- 2ml Manganese sulfate
- 2ml alkali-iodide-azide
- 2ml concentrated sulfuric acid
- 2ml starch solution
- Sodium thiosulfate

These reagents are available in dissolved oxygen field kits, such as those made by the Hach Company. Please use caution when using these reagents, as they can be hazardous to one's health.

Procedure:

- Carefully fill a 300-mL glass Biological Oxygen Demand (BOD) stoppered bottle brim-full with sample water.
- Immediately add 2mL of manganese sulfate to the collection bottle by inserting the calibrated pipette just below the surface of the liquid. (If the reagent is added above the sample surface, you will introduce oxygen into the sample.) Squeeze the pipette slowly so no bubbles are introduced via the pipette.
- Add 2 mL of alkali-iodide-azide reagent in the same manner.
- Stopper the bottle with care to be sure no air is introduced. Mix the sample by inverting several times. Check for air bubbles; discard the sample and start over if any are seen. If oxygen is present, a brownish-orange cloud of precipitate or floc will appear. When this floc has settle to the bottom, mix the sample by turning it upside down several times and let it settle again.
- Add 2 mL of concentrated sulfuric acid via a pipette held just above the surface of the sample. Carefully stopper and invert several times to dissolve the floc. At this point, the sample is "fixed" and can be stored for up to 8 hours if kept in a cool, dark place. As an added precaution, squirt distilled water along the stopper, and cap the bottle with aluminum foil and a rubber band during the storage period.

- In a glass flask, titrate 201 mL of the sample with sodium thiosulfate to a pale straw color. Titrate by slowly dropping titrant solution from a calibrated pipette into the flask and continually stirring or swirling the sample water.
- Add 2 mL of starch solution so a blue color forms.
- Continue slowly titrating until the sample turns clear. As this experiment reaches the endpoint, it will take only one drop of the titrant to eliminate the blue color. Be especially careful that each drop is fully mixed into the sample before adding the next. It is sometimes helpful to hold the flask up to a white sheet of paper to check for absence of the blue color.
- The concentration of dissolved oxygen in the sample is equivalent to the number of milliliters of titrant used. Each mL of sodium thiosulfate added in steps 6 and 8 equals 1 mg/L dissolved oxygen.

2.3 Laboratory Analyzed Chemical Parameter

2.3.1 Chloride

Scope and Application

This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes. The method is suitable for all concentration ranges of chloride content; however,in order to avoid large titration volumes, use a sample aliquot containing not more than 10 to 20 mg C1 per 50 ml. Automated titration may be used.

Summary of Method

Dilute mercuric nitrate solution is added to an acidified sample in the presence of mixed diphenyl carbazone-bromophenol blue indicator. The end point of the titration is the formation of the blue violet mercury diphenyl carbazone complex.

Comments

Anions and cations at concentrations normally found in surface waters do not interfere. Sulfites interfere. If presence is suspected, oxidize by treating 50 ml of sample with 0.5 to 1 ml of H2O2**.**

2.3.2 Total water hardness (as CaCO3)

Historically water hardness was a measure of the capacity of water to precipitate soap, chiefly due to the presence of calcium and magnesium ions in the water. More recently other species such as polyvalent cations have been implicated in the precipitation of soap. Total hardness is therefore now defined as the sum of calcium and magnesium concentrations water, expressed as calcium carbonate equivalents in milligrams per litre according to the following formula.

Hardness equivalent CaCO3/L = 2.497 [Ca, mg/L] + 4.118 Mg, mg/L].

Total acidity and total alkalinity (as CaCO3)

The total *alkalinity* of water is a measure of its acid-neutralising capacity to adesignated pH. It is the sum of all titratable bases, including carbonates,bicarbonates, and hydroxides, and also borates, phosphates, silicates and other bases if they are present.

Total *acidity* is a quantitative measure of the capacity of water to react with a strongbase to a designated pH.For analysis of total alkalinity APHA, 1998 requires titration with a standard hydrochloric acid solution to an end-point pH of 3.7 (i.e. the methyl orange endpoint).To determine total acidity APHA, 1998 requires titration with a standard sodium hydroxide solution to an end-point pH 8.3 (i.e. the phenolphthalein end-point).

2.3.3 Hardness Total

Scope and Application

This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.The method is suitable for all concentration ranges of hardness; however, in order to avoid large titration volumes, use a sample aliquot containing not more than 25mg CaCO,. Automated titration may be used.

Summary of Method

Calcium and magnesium ions in the sample are sequestered upon the addition of disodium ethylenediamine tetraacetate (Naz EDTA). The end point of the reaction ***is*** detected by means of Calmagite Indicator, which has a red color in the presence of calcium and magnesium and a blue color when the cations are sequestered.

Comment

Excessive amounts of heavy metals can interfere. This is usually overcome by complexing the metals with cyanide. Routine addition of sodium cyanide solution ***(Caution:*** deadly poison) to prevent potential metallic interference **is** recommended.

Refferences

The procedure to be used for this determination is found in: Standard Methods for the Examination of Water and Wastewater, 13th Edition, p179, Method 122B (1971).ASTM Standards, Part 23, Water; Atmospheric Analysis, p 169, Method D1126-67 (1 973).

2.3.4 Total suspended solids (TSS)

Total suspended solids (TSS) are defined as the portion of total solids in a water sample retained by a glass fibre (GF/C) filter of pore size >2 μm. This pore size can vary so please check with your analytical lab, however please note that the WIN database has nominated a pore size of 0.45 μm. Once the filter has been dried at 103–105°C and weighed, the amount of total suspended solids is recorded in units of mg/L.

2.3.5 Heavy Metals

2.3.5.1 Aluminum (Standard Condition)

Optimum Concentration Range: 5-100 mg/l using a wavelength of 309.2 nm
Sensitivity: 1.0 mg/l
Detection Limit: 0.1 mg/l

Preparation of Standard Solution

Stock Solution: Carefully weigh 1.000 gram of aluminum metal (analytical reagent grade). Add 15 ml of conc. HC1 to the metal in a covered beaker and warm gently.When solution is complete, transfer quantitatively to a 1 liter volumetric flask and make up to volume with deionized distilled water. 1 ml = 1 mg Al (lOO0 mg/l).

Potassium Chloride Solution: Dissolve 95 **g** potassium chloride (KC1) in deionized distilled water and make up to 1 liter.

Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. They should be prepared calbritation standard using the same type of acid (HC1 or HNO,) and at the same concentration as the samples for analysis. To each 100 ml of standard and sample alike add 2.0 ml potassium chloride solution.

Sample Preparation

The procedure for the determination of total metals given in part of the Atomic Absorption Methods section of this manual has been found to be satisfactory.

Instrumental Parameters (General)

- Aluminum hollow cathode lamp
- Wavelength: 309.2 nm
- Fuel: Acetylene
- Oxidant: Nitrous oxide
- Type of flame: Fuel rich

Interferences

Aluminum is partially ionized in the nitrous oxide-acetylene flame. This problem may be controlled by the addition of an alkali metal (potassium, 1000 pg/ml) to both sample and standard solutions.

2.3.5.2 Chromium

Optimum Concentration Range: 0.2-10 mg/l using a wavelength of 357.9 nm
Semitivity: 0.1 mg/l
Detection Limit: 0.02 mg/l

Preparation of Standard Solution

Stock Solution: Dissolve 1.923 **g** of chromium trioxide (CrO,, reagent grade) in deionized distilled water. When solution is complete, acidify with redistilled HN03 and dilute to 1 liter with deionized distilled water. 1 ml = 1 mg Cr (1000mdl).

Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. The *calibration standards* should be prepared using the same type of acid (HClorHNO,) and at the same concentration as the samples for analysis.

Sample Preparation

The procedure for the determination of total metals as given in part of the Atomic Absorption Methods section of this manual has been found to be satisfactory.

Instrumental Parameters (General)

- Chromium hollow cathode lamp
- Wavelength: 357.9 nm
- Fuel: Acetylene
- Oxidant: Air
- Type of flame: Slightly fuel rich

2.3.5.3 Copper (Standard Condition)

Optimum Concentration Range: 0.2-1 0 mg/l using a wavelength of324.7 nm
Sensitivity: 0.1 mg/l
Detection Limit: 0.0 1 mg/l

Preparation of Standard Solution

Stock soluction: Carefully weight 1.00 g of electrolyte copper(analytical reagent grade).t Dissolve in 5ml redistilled HNO3 and make up to 1 liter with deionized distilled water. Final concentration is 1 mg Cu per ml (1 000 mg/ 1).

Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. The calibration standard should be prepared using the same type of acid (HC1 orHN03) and at the same concentration as the samples for analysis.

Sample Preparation

The procedure for the determination of total metals as given in part of the Atomic Absorption Methods section of this manual has been found to be satisfactory.

Instrumental Parameters (General)

- Copper hollow cathode lamp
- Wavelength: 324.7 nm
- Fuel: Acetylene
- Oxidant: Air
- Type of flame: Oxidizing

2.3.5.4 Lead (Standard Condition)

Optimum Concentration Range: 1-20 mg/l using a wavelength of 283.3 nm
Sensitivity: 0.5 mg/l
Detection Limit: 0.05 mg/l

Preparation of Standard Solution

Stock Solution: Carefully weigh 1.599 g of lead nitrate, Pb(NO3)? (analytical reagent grade), and dissolve in deionized distilled water. When solution is complete acidify with 10 ml redistilled HN03 and dilute to 1 liter with deionized distilled water. 1 ml = I mg Pb (1 000 mg/l).

Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. The *calibration standards* should be prepared using the same type of acid (HC1 or HN03) and at the same concentration as the samples for analysis.

Sample Preparation

The procedure for the determination of total metals as given in part of the Atomic Absorption Methods section of this manual has been found to be satisfactory.

Instrumental Parameters

- Lead hollow cathode lamp
- Wavelength: 283.3 nm
- Fuel: Acetylene
- Oxidant: Air
- Type of flame: Slightly oxidizing

2.3.5.5 Zinc (Standard Condition)

Optimum Concentration Range: 0.05-2 mg/l using a wavelength of 213.9 nm
Sensitivity: 0.02 mg/l
Detection Limit: 0.005 mg/l

Preparation of Standard Solution

Stock Solution: Carefully weigh 1.00 g of zinc metal (analytical reagent grade) and dissolve cautiously in 10 ml HNO3**.**When solution is complete make up to 1 liter with deionized distilled water. 1 ml = 1 mg Zn (1000 mg/l).

Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. *The calibration standards* should be prepared using the same type of acid (HCI or HN03) and at the same concentration as the samples for analysis.

Sample Preparation

The procedure for the determination of total metals as given in part of the Atomic Absorption Methods section of this manual has been found to be satisfactory.

Instrumental Parameters

- Zinc hollow cathode lamp
- Wavelength: 21 3.9 nm
- Fuel: Acetylene
- Oxidant: ***Air***
- Type of flame: Oxidizing

2.3.6 Nitrogen / Nitrate

Scope and application

This method is applicable to the analysis of drinking, surface, and saline waters,domestic and industrial wastes. Modification can be made to remove or correct for turbidity, color,salinity, or dissolved organic compounds in the sample.The applicable range of concentration is0.1to**2**mg NO3 -N/liter.

Summary of Method

This method is based upon the reaction of the nitrate ion with brucine sulfate in a 13 N H2S04 solution at a temperature of 100°C. The color of the resulting complex is measured at 410 nm.Temperature control of the color reaction isextremely critical.

Sample handling and Preservation

Samples may be preserved by addition of 2 ml conc. H2S04/liter or by addition of 40 mg HgC12 per liter and storage at 4°C.

Apparatus

Spectrophotometer or filter photometer suitable for measuring optical densities at 410 nm.Sufficient number of 40-50 ml glass sample tubes for reagent blanks, standards, and samples.Neoprene coated wire racks to hold sample tubes.Water bath suitable for use at 100°C. This bath should contain a stirring mechanism so that all tubes are at the same temperature and should be of sufficient capacity to accept the required number of tubes without significant drop in temperature when the tubes are immersed. Water bath suitable for use at 10-1 5°C

Reagent

Distilled water free of nitrite and nitrate is to be used in preparation of all reagents and standards.Sodium chloride solution (300 g/l): Dissolve 300 g NaCl in distilled water and dilute to 1 liter.Sulfuric acid solution: Carefully add 500 ml conc. H2SO4 to 125 ml distilled water. Cool and keep tightly stoppered to prevent absorption of atmospheric moisture. Brucine-sulfanilic acid reagent: Dissolve 1 g brucine sulfate [(C23Hz6N2 O4*)2* OH, SO4 *7H2 01 and 0.1 g sulfanilic acid (NH2 c6 H4 *SO3* H*H2 0) in 70 ml hot distilled water. Add 3 ml conc. HCl, cool, mix and dilute to 100 ml with distilled water. Store in a dark bottle at 5'C. This solution is stable for several months; the pink color that develops slowly does not effect its usefulness.

Mark bottle with warning; *CAUTION: Brucine Sulfate is toxic; take care to avoid ingestion*. Potassium nitrate stock solution: 1.0 ml = 0.1 mg NO3 -N. Dissolve 0.7218 g anhydrous potassium nitrate (KNO,) in distilled water and dilute to 1 liter in a volumetric flask. Preserve with 2 ml chloroform per liter. This solution is stable for at least 6 months. Potassium nitrate standard solution: 1.0 ml = 0.001 mg NO3-N. Dilute 10.0 ml of the stock solution (6.5) to 1 liter in a volumetric flask. This standard solution should be prepared fresh weekly. Acetic acid (1 + 3): Dilute 1 volume glacial acetic acid (CH3COOH) with 3 volumes of distilled water. Sodium hydroxide: Dissolve 40 g of NaOH in distilled water. Cool and dilute to 1 liter.

Procedure

Adjust the pH of the samples to approximately 7 with acetic acid or sodium hydroxide. If necessary, filter to remove turbidity. Set up the requ-ired number of sample tubes in the rack to handle reagent blank, standards and samples. Space tubes evenly throughout the rack to allow for even flow of bath water between the tubes. This should assist in achieving uniform heating of all tubes.If it is necessary to correct for color or dissolved organic matter which will cause color on heating, a set of duplicate samples must be run to which all reagents except the brucine-sulfanilic acid have been added. Pipette 10.0 ml of standards and samples or an aliquot of the samples diluted to 10.0 ml into the sample tubes. If the samples are saline, add 2 ml of the 30% sodium chloride solution to the reagent blank, standards and samples. For fresh water samples, sodium chloride solution may be omitted. Mix contents of tubes by swirling and place rack in cold water bath (@lO°C). Pipette 10.0 ml of sulfuric acid solution into each tube and mix by swirling. Allow tubes to come to thermal equilibrium in the cold bath. Be sure that temperatures have

equilibrated in all tubes before continuing. Add 0.5 ml brucine sulfanilic acid reagent to each tube (except the interference control tubes) and carefully mix by swirling, then place the rack of tubes in the 100°C water bath for exactly 25 minutes.

Caution: Immersion of the tube rack into the bath should not decrease the temperature of the bath more than 1 to 2OC. In order to keep this temperature decrease to an absolute minimum, flow of bath water between the tubes should not be restricted by crowding too many tubes into the rack. If color development in the standards reveals discrepancies in the procedure, the operator should repeat the procedure after reviewing the temperature control steps. Remove rack of tubes from the hot water bath and immerse in the cold water bath and allow ***to*** reach thermal equilibrium (20-25'C). Read absorbance against the reagent blank at 410 nm using a 1 cm or longer cell.

2.3.7 Nitrogen, Ammonia (Selective Ion Electrode Method)

Scope and Application

This method is applicable to the measurement of ammonia-nitrogen in drinking, surface, and saline waters, domestic and industrial wastes. **This** method covers the range from 0.03 to 1400 mg NH3-N/1. Color and turbidity have no effect on the measurements and distillation is not necessary.

Summary of Method

The ammonia is determined potentiometrically using a selective ion ammonia electrode and a pH meter having an expanded millivolt scale or a specific ion meter. The ammonia electrode uses a hydrophobic gas-permeable membrane to separate the sample solution from an ammonium chloride internal solution. Ammonia in the sample diffuses through the membrane and alters the pH of the intemal solution, which is sensed by a pH electrode. The constant level of chloride in the intemal solution is sensed by a chloride selective ion electrode which acts as the reference electrode.

Sample Handling and Preservation

Preserve by refrigeration at 4^0C; analyze within 24 hours. If longer holding times are desired, preserve with 2 ml conc. H_2SO_4 per liter (pH<2).

Apparatus

- Electrometer (pH meter) with expanded mV scale or a specific ion meter.
- Ammonia selective electrode, such as Orion Model 95-10 or EIL Model 8002-2.
- Magnetic stirrer, thermally insulated, and Teflon-coated stirring bar.

Reagents

Distilled water: Special precautions must be taken to insure that the distilled water is free of ammonia. This is accomplished by passing distilled water through an ion exchange column containing a strongly acidic cation exchange resin mixed with a strongly basic anion exchange resin. Sodium hydroxide, 10N: Dissolve 400 g of sodium hydroxide in 800 ml of distilled water. Cool and dilute to 1 liter with distilled water (6.1). Ammonium chloride, stock solution: 1.0 ml = 1.0 mg NH,-N. Dissolve 3.819 g NH,C1 in water and bring to volume in a 1 liter volumetric

flask using distilled water (6.1). Ammonium chloride, standard solution: 1.0 ml = 0.01 mg NH,-N. Dilute 10.0 ml of the stock solution to 1 liter with distilled water in a volumetric flask.

Procedure

Preparation of standards: Prepare a series of standard solutions covering the concentration range of the samples by diluting either the stock or standard solutions of ammonium chloride. ***Calibration of electrometer***: Place 100 ml of each standard solution in clean 150 ml beakers. Immerse electrode into standard of lowest concentration and add 1 ml of 10N sodium hydroxide solution while mixing. Keep electrode in the solution until a stable reading is obtained. Repeat this procedure with the remaining standards, going from lowest to highest concentration. Using semi logarithmic graph plot t the concentration of ammonia in mg NH3 -N/1 on the log axis vs. the electrode potential developed in the standard on the linear axis, starting with the lowest concentration at the bottom of the scale. ***Calibration of a specific ion meter***: Follow the direction of the manufacturer for the operation of the instrument. ***Sample measurement***: Follow the procedure in calibration of sample in 150 ml beakers. Record the stabilized potential of each know sample and convert the potential reading to the ammonia concentration of the standard curve. If a specific ion meter is used, read the ammonia level directly in mg NH_3 -N/L

2.3.8 Biochemical Oxygen Demand (BOD)

BOD Test Procedures

- To ensure proper biological activity during the BOD test, a wastewater sample:
- Must be free of chlorine. If chlorine is present in the sample, a dechlorination chemical (e.g, sodium sulfite) must be added prior to testing.
- Needs to be in the pH range of 6.5 - 7.5 S.U. If the sample is outside this range, then acid or base must be added as needed.
- Needs to have an existing adequate microbiological population. If the microbial population is inadequate or unknown, a "seed" solution of bacteria is added along with an essential nutrient buffer solution that ensures bacteria population vitality.
- Specialized 300 mL BOD bottles designed to allow full filling with no air space and provide an airtight seal are used. The bottles are filled with the sample to be tested or dilution (distilled or deionized) water and various amounts of the wastewater sample are added to reflect different dilutions. At least one bottle is filled only with dilution water as a control or "blank."
- A DO meter is used to measure the initial dissolved oxygen concentration (mg/L) in each bottle, which should be a least 8.0 mg/L. Each bottle in then placed into a dark incubator at 20°C for five days.
- After five days (± 3 hours) the DO meter is used again to measure a final dissolved oxygen concentration (mg/L), which ideally will be a reduction of at least 4.0 mg/L.
- The final DO reading is then subtracted from the initial DO reading and the result is the BOD concentration (mg/L). If the wastewater sample required dilution, the BOD concentration reading is multiplied by the dilution factor.

2.3.9 Chemical Oxygen Demand (COD)

COD Test Procedures

- Prior to completing the COD test, a series of known standards are prepared using KHP (potassium hydrogen phthalate). Most wastewater samples will fall in the high range, so standards of 100, 250, 500 and 1000 mg/L are typically prepared. COD standards can also be purchased.
- A COD reactor/heating (150°C) block and a colorimeter are turned on so that both instruments are allowed to stabilize.
- Pre-prepared low-range (3 - 150 ppm) or high-range (20 - 1500 ppm) vials are selected for the COD test based on expected results. Both ranges can be used if expected results are unknown.
- One vial is marked as a "blank," and three or four vials are marked with known standard levels. Two vials are then marked for the wastewater sample to make a duplicate run. **Note**: If multiple wastewater samples are being run, at least 10% of samples are duplicated.
- 2 mL of liquid are added to each vial. In the case of the "blank," 2 mL of DI water are added. 2 mL of each standard are added to the corresponding vials. If the wastewater sample is tested at full strength, then 2 mL is added to the corresponding vial. If dilution is required, then serial dilutions are performed and 2 mL of the diluted sample are added to the corresponding vial.
- Each vial is mixed well and placed into the reactor block for two hours. After two hours, the vials are removed from the block to a cooling rack for about 15 minutes.
- The colorimeter is set and calibrated per the specific instructions for that unit (i.e., proper wavelength, blank and standards) and each vial is placed in the unit and the COD concentration read.
- If the sample was diluted, the corresponding multiplication is made.

2.3.10 Cyanides, Amenable to Chlorination

Scope and Application

This method is applicable to the determination of cyanides amenable to chlorination in drinking, surface, and saline waters, and domestic and industrial wastes. The titration procedure is used for measuring concentrations of cyanide exceeding 1 mg/l after removal of the cyanides amenable to chlorination. Below this level the colorimetric determination is used.

Summary of Method

A portion of the sample is chlorinated at a pH>11 to decompose the cyanide.Cyanide levels in the chlorinated sample are then determined by the method for Cyanide, Total, in this manual. Cyanides amenable to chlorination are then calculated by difference.

Reagents

Calcium Hypochlorite solution: Dissolve 5 g of calcium hypochlorite ($Ca(OCl)_2$) in 100 ml of distilled water. Sodium Hydroxide solution: Dissolve 50 g of sodium hydroxide (NaOH) in distilled water and dilute to 1 liter. Ascorbic acid: crystals. Potassium Iodide - starch test paper.

Procedure

Two sample aliquots are required to determine cyanides amenable to chlorination. To one 500 ml aliquot or a volume diluted to 500 ml, add calcium hypochlorite solution (3.1) dropwise while agitating and maintaining the pH between 11 and 12 with sodium hydroxide (3.2).

Caution: The initial reaction product of alkaline chlorination is the very toxic gas cyanogen chloride; therefore, it is recommended that this reaction be performed in a hood. For convenience, the sample may be agitated in a 1 liter beaker by means of a magnetic stirring device.

Test for residual chlorine with KI-starch paper (3.4) and maintain this excess for one hour, continuing agitation. A distinct blue color on the test paper indicates a sufficient chlorine level. If necessary, add additional hypochlorite solution.

After one hour, add 0.5 g portions of ascorbic acid until KI-starch paper shows no residual chlorine. Add an additional 0.5 g of ascorbic acid to insure the presence of excess reducing agent.

Test for total cyanide in both the chlorinated and unchlorinated aliquots as in the method Cyanide, Total, in this manual.

Calculation

Calculate the cyanide amenable to chlorination as follows:

CN, mg/l = A-B

where:

A = mg/ 1 total cyanide in unchlorinated aliquot

B = mg/1 total in chlorinated aliquot

2.3.11 Total Organic Carbon

Total organic carbon (TOC) is the amount of carbon bound in an organic compound and is often used as a non-specific indicator of water quality or cleanliness of pharmaceutical manufacturing equipment. TOC may also refer the amount of organic carbon in a geological formation, particularly the source rock for a petroleum play; 2% is a rough minimum.

A typical analysis for TOC measures both the total carbon present and the so-called "inorganic carbon" (IC), the latter representing the content of dissolved carbon dioxide and carbonic acid salts. Subtracting the inorganic carbon from the total carbon yields TOC. Another common variant of TOC analysis involves removing the IC portion first and then measuring the leftover carbon. This method involves purging an acidified sample with carbon-free air or nitrogen prior to measurement, and so is more accurately called non-purgeable organic carbon (NPOC).

Total Organic Carbon is a broadly useful measurement. TOC is a required measurement in municipal water and wastewater systems, and is also a valuable measurement in a host of industries that rely on TOC analysis for process control and for reporting of regulated organic discharge levels. Industry often turns to TOC analysis to protect vital systems by monitoring raw water feedstock and process water quality.

High temperature combustion

Prepared samples are combusted at 1,350 °C in an oxygen-rich atmosphere. All carbon present converts to carbon dioxide, flows through scrubber tubes to remove interferences such as chlorine gas, and water vapor, and the carbon dioxide is measured either by absorption into a strong base then weighed, or using an Infrared Detector. Most modern analyzers use non-dispersive infrared (NDIR) for detection of the carbon dioxide.

Analytical Methods for TOC Determination

In soils and sediments:

$$\text{Total Carbon} = \text{Inorganic Carbon} + \text{Organic Carbon n} \qquad (1)$$

TOC content can be measured directly or can be determined by difference if the total carbon content and inorganic carbon contents are measured. For soils and sediments where no inorganic carbon forms are present, Equation 1 becomes:

$$\text{Total Carbon} = \text{Organic Carbon} \qquad (2)$$

Typically this is the case so methods described as quantifying total or organic carbon should produce the same result. However, in geographic areas where the parent material/geology is limestone, dolomite, or another carbonate-bearing mineral, inorganic forms of carbon may be present in the samples. In arid regions, soils and sediments may have greater concentrations of carbon being derived from inorganic carbonates than from organic carbon sources.

Qualitative Methods for the Determination of Organic

There are two methods in the literature for the structural characterization of organic carbon forms in soils and sediments. One of these qualitative methods is based on nuclear magnetic resonance (NMR) spectroscopy and the other on diffuse reflectance infrared Fourier transform (DRIFT) spectroscopy. A brief synopsis of these qualitative techniques is presented here because research is ongoing to improve the techniques* qualitative identification capabilities and to advance the science into the area of quantitative determinations of the carbon forms in soils and sediments.

NMR is a valuable tool for the characterization of soil organic matter and humification processes in soils (Kogel-Knaber, 1997). NMR spectroscopy works on the principle of measuring the characteristic energy absorbed and re-emitted or dispersed by atomic nuclei that are placed in a static magnetic field and subjected to an oscillatory magnetic field of known radio-frequency. One specialized form of this technique is cross-polarization magic angle spinning (CPMAS) ^{13}C NMR (Rumpel et. al, 1998). CPMAS ^{13}C NMR is capable of distinguishing chemical structures that are characteristic of recently formed organic matter as well as those organic carbon forms derived from the soil*s parent material/geology, elemental carbon forms derived from ash, and even, carbonaceous particles from airborne lignite-derived contamination in soils. The advantage of NMR techniques is that no extraction of organic matter is needed. However, the NMR methods are expensive and time-consuming (Rumpel et al., 2001).

DRIFT spectroscopy, when used in conjunction with multivariate data analysis (i.e., partial least squares), provides a rapid and inexpensive means of differentiating carbon forms in soils and sediments (Rumpel et al., 2001). Carbon compounds are identified by assignment of the main infrared absorption bands to the bonds being stretched or deformed at that particular frequency. Both inorganic and organic forms of organic compounds may be identified using this technique (Nguyen et al., 1991). In initial experiments, Rumpel et al., (2001) were able to identify and quantify the lignite contribution to the TOC content of soil samples; however, TOC determination was performed by a dry combustion technique (to be discussed).

Wet Chemistry Techniques for the Determination of Total Organic Carbon.

Wet chemistry techniques can be divided into two phases, namely, sample extraction and sample quantitation. The extraction technique employed is essentially the same for all methods in the literature with variations existing only in the strength and combination of reagents used during extraction. Quantitation techniques associated with the wet chemistry determination of TOC either rely on titration (manual or automated), calorimetric, gravimetric, or manometric techniques.

3. Summary of Field Test Kits

Field test kits can be important analytical tools during receiving water investigations. Among others, described how they can be used to obtain rapid and cost-effective data. However, the careful selection of the test kits to be used is critical. It is important to consider several factors, specifically the sensitivity of the procedure, safety hazards associated with the method, the cost (both capital and expendables) to conduct the analyses, and the time and expertise needed to conduct the test. The useful range is the minimum detection limit found during our tests to the upper limit that does not require dilution. The precision is the coefficient of variation based on replicate analyses, and the recovery is the slope of the regression line comparing analyses of spiked samples using these procedures and standard methods. The recovery tests were conducted using both ultra-clean water prepared using reverse osmosis (RO) and stormwater to identify any matrix interference problems. Any problems noted during the tests are also indicated, especially safety concerns, unusual amounts of expertise needed, and storage requirements.

These tests represent several classes of analytical procedures. Simple color indicator paper strips for alkalinity, vacuum vials are also used in several tests.

Many of other types of test kits are more complex and require several steps for the analyses. Some of the most complex procedures may require as many as 10 steps and more than 30 min for analyses.

In general, we found that the field test kits were more accurate than we had originally expected. However, the sensitivities of many of the field test kits were much poorer than expected, making them much less useful. In addition, numerous safety hazards can exist with these kits, sharps and hazardous reagents and wastes being the most serious.

3.1 Speceal Comments Pertaining to Heavy Metal Analyses

There are a number of methods available for heavy metals, with the traditional methods restricted to the laboratory. The following discussion summarizes these available methods, especially their sensitivities.

Environmental Protection Agency (in the *Code of Federal Regulations*, especially 40 CFR, 136 "Guidelines Establishing Test Procedures for the Analysis of Pollutants"). Methods listed in these references are generally taken as approved for many purposes. Most all of the metals can be analyzed using atomic absorption spectrometry (AAS) and inductively coupled plasma emission spectrometry (ICP). In addition, many of the metals have specific chemical tests that use spectrophotometric or titration methods. For most stormwater investigations, only a relatively few of these metals are routinely evaluated, including arsenic, cadmium, chromium, copper, lead, mercury, nickel, selenium, and zinc.

Table –4 Optimal Concentration Ranges of Metals in Samples

Parameters	Flames AAS (mg/l)	Electrothermal AAS (mg/l)	Inductively Coupled Plasma AES (mg/l)
Alumium	5-100	0.02-0.2	0.6-100
Antimony	1-40	0.02-0.3	0.45-100
Arsenic		0.005-0.1	0.75-100
Barium	1-20	0.01-0.2	0.030-50
Beryllium	0.05-2	0.001-0.03	0.005-10
Bismuth	1-5		
Cadmium	0.05-2	0.0005-0.01	0.06-50
Calcium	0.2-20		0.15-100
Casium	0.5-15		
Chromium	0.2-10	0.005-0.1	0.1-50
Cobalt	0.5-10	0.005-0.1	0.1-50
Copper	0.2-10	0.005-0.1	0.1-50
Gold	0.5-20		
Iron	0.3-10	0.005-0.1	0.1-100
Lead	1-20	0.005-0.1	0.6-100
Lithium	0.1-2		0.06-100
Magnesium	0.02-2		0.45-100
Manganese	0.1-10	0.001-0.03	0.06-50
Molybenum	1-20	0.003-0.06	0.12-100
Nickel	0.3-10	0.005-0.1	0.2-50
Platinum	5-75		
Potassium	0.1-2		1.5-100
Selenium		0.005-0.1	1.0-100
Silver	0.1-4	0.001-0.025	0.1-50
Sodium	0.03-1		
Strontium	0.3-5		0.03-50
Thallium			0.6-100
Tin	10-200	0.02-0.3	
Titanium	5-100		
Vanadium	2-100		0.1-50
Zinc	0.05-2		0.03-100

Table compares the optimal metal concentration ranges for AAS and ICP, the most commonly used instrumentation (*Standard Methods* 1995). Instrument detection limits are about 15 times less than the lower values shown on this table, which represent the lower limits of quantification. The lower limits of the flame AAS optimal concentration ranges are generally

about the same as for the plasma AES, while the electrothermal AAS lower limits are 10 to 1000 times lower. However, the plasma AES instrument has a much greater dynamic range than either AAS instrument. The plasma AES also has fewer interferences and can analyze many elements simultaneously. Because of these differences, many laboratories use a plasma AES for general analytical work and an electrothermal AAS for individual samples for single elements at very low concentrations.

X-ray fluorescence can also be used to detect heavy metals in solid samples, such as sediments and soils, including particulates trapped on filters (from water or air samples).

Almost all of the stormwater heavy metals can be released from the particulates using just nitric acid, especially considering metal losses from using a hydrofluoric acid digestion. A nitric acid and perchloric acid mixture may be needed to digest organic material in the samples. Microwave-assisted digestion has become more common recently because of improved metal recovery, much faster digestion, and better repeatability.

Table - 5. Ground Water Quality Standards (Utah Department of Environmental Quality)

Parameter	CASRN	GWQS	Unit
Physical Characteristics			
Color		15.0	
Corrosivity		noncorrosive	
Odor		3.0	
pH		6.5 - 8.5	
Inorganic Chemicals			
Bromate	7789-38-0	0.01	mg/l
Chloramine (as Cl2)	10599-90-3	4.0	mg/l
Chlorine (as Cl2)	7782-50-5	4.0	mg/l
Chlorine Dioxide	10049-04-4	0.8	mg/l
Chlorite	7758-19-2	1.0	mg/l
Cyanide (free)	143-33-9	0.2	mg/l
Fluoride	7681-49-4	4.0	mg/l
Nitrate (as N)	14797-55-8	10.0	mg/l
Nitrite (as N)	14797-65-0	1.0	mg/l
Total Nitrate + Nitrite (both as N)		10.0	mg/l
Metals			
Antimony	7440-36-0	0.006	mg/l
Arsenic	7440-38-2	0.05	mg/l
Asbestos (> 10 microns in length)	1332-21-4	7E+06	fibers/l
Barium	7440-39-3	2.0	mg/l
Beryllium	7440-41-7	0.004	mg/l
Cadmium	7440-43-9	0.005	mg/l
Chromium (total)	7440-47-3	0.1	mg/l

Copper	7440-50-8	1.3	mg/l
Lead	7439-92-1	0.015	mg/l
Mercury (inorganic)	7487-94-7	0.002	mg/l
Selenium	7782-49-2	0.05	mg/l
Silver	7440-22-4	0.1	mg/l
Thallium	7440-28-0	0.002	mg/l
Zinc	7440-66-6	5.0	mg/l
Organic Chemicals			
Pesticides and PCBs			
Alachlor	15972-60-8	0.002	mg/l
Aldicarb	116-06-3	0.003	mg/l
Aldicarb sulfone	1646-88-4	0.003	mg/l
Aldicarb sulfoxide	1646-87-3	0.004	mg/l
Atrazine	1912-24-9	0.003	mg/l
Carbofuran	1563-66-2	0.04	mg/l
Chlordane	57-74-9	0.002	mg/l
Dalapon (sodium salt)	75-99-0	0.2	mg/l
Dichlorophenoxyacetic acid (2, 4 -) (2, 4 - D)	94-75-7	0.07	mg/l
Dinoseb	88-85-7	0.007	mg/l
Diquat	85-00-7	0.02	mg/l
Endothall	145-73-3	0.1	mg/l
Endrin	72-20-8	0.002	mg/l
Ethylene dibromide (EDB)	106-93-4	0.00005	mg/l
Glyphosate	1071-83-6	0.7	mg/l
Heptachlor	76-4-8	0.0004	mg/l
Heptachlor epoxide	1024-57-3	0.0002	mg/l
Lindane	58-89-9	0.0002	mg/l
Methoxychlor	72-43-5	0.04	mg/l
Oxamyl (Vydate)	23135-22-0	0.2	mg/l
Pentachlorophenol	87-86-5	0.001	mg/l
Picloram	1918-02-1	0.5	mg/l
Polychlorinated biphenyls (PCBs)	1336-36-3	0.0005	mg/l
Simazine	122-34-9	0.004	mg/l
Toxaphene	8001-35-2	0.003	mg/l
2, 4, 5 - TP (Silvex)	93-72-1	0.05	mg/l
Volatile Organic Chemicals			
Benzene	71-43-2	0.005	mg/l
Carbon tetrachloride	56-23-5	0.005	mg/l

Dichloroethane (1,2 -)	107-06-2	0.005	mg/l
Dichloroethylene (1,1 -)	75-35-4	0.007	mg/l
Dichloromethane	75-09-2	0.005	mg/l
Di (2-ethylhexyl) adipate	103-23-1	0.4	mg/l
Di (2-ethylhexyl) phthalate (PAE)	117-81-7	0.006	mg/l
Dichlorobenzene (para -)	106-46-7	0.075	mg/l
Dichlorobenzene (o -)	95-50-1	0.6	mg/l
Dichloroethylene (cis - 1,2)	156-59-2	0.07	mg/l
Dichloroethylene (trans - 1,2)	156-60-5	0.1	mg/l

Table - 6. Water Quality Parameters and Drinking Water Standards
(Office of the Chief Engineer, State Ground and Surface Water Resourses Data Centre, THARAMANI, CHENNAI - 600 113.)

SL. NO.	PARAMETERS	UNITS	DRINKING WATER (IS: 10500 – 1991)	
			DESIRABLE	MAXIMUM
1.	Colour	Hazen units	**5**	**25**
2.	Odour	-	**Unobjectionable**	**-**
3.	Taste	-	**Agreeable**	**-**
4.	Turbidity	NTU	**5**	**10**
5.	pH value	-	**6.5 to 8.5**	**No relaxation**
6.	Total hardness (as $CaCO_3$)	mg/l	**300**	**600**
7.	Iron	mg/l	**0.3**	**1.0**
8.	Chlorides	mg/l	**250**	**1000**
9.	Residual, free Chlorine	mg/l	**0.2**	**-**
10.	Dissolved Solids	mg/l	**500**	**2000**
11.	Calcium	mg/l	**75**	**200**
12.	Copper	mg/l	**0.05**	**1.5**
13.	Manganese	mg/l	**0.1**	**0.3**
14.	Sulphate	mg/l	**200**	**400**
15.	Nitrate	mg/l	**50**	**No relaxation**
16.	Fluoride	mg/l	**1.0**	**1.5**
17.	Phenolic compounds	mg/l	**0.001**	**0.002**
18.	Mercury	mg/l	**0.001**	**No relaxation**
19.	Cadmium	mg/l	**0.01**	**No relaxation**
20.	Selenium	mg/l	**0.01**	**No relaxation**
21.	Arsenic	mg/l	**0.05**	**No relaxation**
22.	Cyanide	mg/l	**0.05**	**No relaxation**
23.	Lead	mg/l	**0.05**	**No relaxation**
24.	Zinc	mg/l	**5**	**15**
25.	Anionic detergents	mg/l	**0.2**	**1.0**

26.	Chromium	mg/l	**0.05**	**No relaxation**
27.	Polynuclear aromatic Hydrocarbons	mg/l	**-**	**-**
28.	Mineral oil	mg/l	**0.01**	**0.03**
29.	Pesticides	mg/l	**Absent**	**0.001**
30.	Radioactive materials (a) Alpha emitters (b) Beta emitters	Bq/l Pci/l	**-** **-**	**0.1** **0.037**
31.	Alkalinity	mg/l	**200**	**600**
32.	Aluminlum	mg/l	**0.03**	**0.2**
33.	Boron	mg/l	**1**	**5**

Table – 7, The Quality of Water Intented for Human Consumption (EU's Drinking Water Standards)

Parameter	**Symbol/formula**	**Parametric value (mg/l)**
Arsenic	As	0.01
Benzene	C_6H_6	0.001
Boron	B	1.00
Bromate	Br	0.01
Cadmium	Cd	0.005
Chromium	Cr	0.05
Copper	Cu	2.0
Cyanide	CN =	0.05
1,2-dichloroethane	$Cl\,CH_2\,CH_2\,Cl$	0.003
Fluoride	F	1.5
Lead	Pb	0.01
Mercury	Hg	0.001
Nickel	Ni	0.02
Nitrate	NO_3	50
Nitrite	NO_2	0.50
Pesticides		0.0001
Pesticides - Total		0.0005
PAHs	$C_2\,H_3\,N_1\,O_5\,P_{1\,3}$	0.0001
Selenium	Se	0.01
Trihalomethanes - Total		0.1
Vinyl chloride	C_2H_3Cl	0.0005
Aluminium	Al	0.2 mg/l
Ammonium	NH4	0.50 mg/l
Chloride	Cl	250 mg/l
Colour		Acceptable to consumers and no abnormal change
Conductivity		2500 µS/cm @ 20oC
Hydrogen ion concentration	[H+]	≥ 6.5 and ≤ 9.5
Iron	Fe	0.2 mg/l
Manganese	Mn	0.05 mg/l
Odour		Acceptable to consumers and no

		abnormal change
Oxidisability		5.0 mg/l O2
Sulfate	SO4	250 mg/l
Sodium	Na	200 mg/l
Taste		Acceptable to consumers and no abnormal change
Colony count 22o		No abnormal change
Coliform bacteria		0/100 ml
Total organic carbon (TOC)		No abnormal change
Turbidity		Acceptable to consumers and no abnormal change
Total indicative dose		0.10 Sv/year

Table - 4.WHO's Drinking Water Standards (1993)

Element/ substance	Symbol/ formula	Normally found in freshwater/surfacewater/ground water	Health based guideline by the WHO
Aluminium	Al		0,2 mg/l
Ammonia	NH_4	< 0,2 mg/l (up to 0,3 mg/l in anaerobic waters)	No guideline
Antimony	Sb	< 4 µg/l	0.005 mg/l
Arsenic	As		0,01 mg/l
Asbestos			No guideline
Barium	Ba		0,3 mg/l
Berillium	Be	< 1 µg/l	No guideline
Boron	B	< 1 mg/l	0,3 mg/l
Cadmium	Cd	< 1 µg/l	0,003 mg/l
Chloride	Cl		250 mg/l
Chromium	Cr^{+3}, Cr^{+6}	< 2 µg/l	0,05 mg/l
Colour			Not mentioned
Copper	Cu		2 mg/l
Cyanide	CN^-		0,07 mg/l
Dissolved oxygen	O_2		No guideline
Fluoride	F	< 1,5 mg/l (up to 10)	1,5 mg/l
Hardness	mg/l $CaCO_3$		No guideline
Hydrogen sulfide	H_2S		No guideline
Iron	Fe	0,5 - 50 mg/l	No guideline
Lead	Pb		0,01 mg/l
Manganese	Mn		0,5 mg/l
Mercury	Hg	< 0,5 µg/l	0,001 mg/l
Molybdenum	Mb	< 0,01 mg/l	0,07 mg/l
Nickel	Ni	< 0,02 mg/l	0,02 mg/l
Nitrate and nitrite	NO_3, NO_2		50 mg/l total nitrogen
Turbidity			Not mentioned
pH			No guideline
Selenium	Se	< < 0,01 mg/l	0,01 mg/l
Silver	Ag	5 – 50 µg/l	No guideline
Sodium	Na	< 20 mg/l	200 mg/l
Sulfate	SO_4		500 mg/l

Inorganic tin	Sn		No guideline
TDS			No guideline
Uranium	U		1,4 mg/l
Zinc	Zn		3 mg/l