PERCHLORIC ACID FUME HOODS

Certain laboratory procedures may require the use of perchloric acid. Formation of explosive perchlorate crystals may be a by-product of the experiment. Special fume hoods, commonly known as perchloric acid fume hoods, MUST be used when conducting experiments that require perchloric acid. The fume hoods have self-contained wash-down units to inhibit crystal formation within the fume hood and the exhaust ductwork. Perchloric Acid fume hoods are located in SA 232. The operating instructions for the fume hood in SA 232 are posted in the laboratory and in Appendix I.

Anyone planning to use perchloric acid must read and understand the Material Safety Data Sheet (MSDS). A supervisor or Environment Health and Safety (EH&S) has to be consulted if any portion of the MSDS cannot be understood.

PERCHLORIC ACID / PERCHLORATES

1) Introduction

Fume hoods used for normal chemical operations must not be used for perchloric acid work. Fires and explosions have occurred where organic residues present in the fume hood system were rendered explosive after contact with the perchloric acid fumes and vapours. The acid fumes are usually formed during heating or digestion of materials. The digestion of organic materials may be a highly exothermic reaction and may create acid fumes.

Perchloric acid (60-72 percent) reacts with alcohols and certain other organic compounds to form very unstable perchlorate esters at room temperature. Perchloric acid (68-72 percent) acts as a strong acid at room temperatures. Perchloric acid is an exceedingly strong and active oxidizing agent as well as a strong dehydrating agent at elevated temperatures (approximately 160 °C, or 320 °F). Anhydrous perchloric acid (highly unstable) is usually formed when perchloric acid is mixed with concentrated sulphuric acid or phosphorus pentoxide.

Perchloric acid must be used in laboratories with perchloric acid fume hood facilities. Special fume hoods and ducts for perchloric acid work have been constructed of stainless steel. An exhaust air scrubber system has been installed in the ductwork and behind the baffle to emit a fine water spray to dilute and wash down the acid fumes and vapours when the fume hood is in use.

2) Access

The University of Calgary has a fume hood facility for work with perchloric acid.

- The Perchloric Acid lab is in Science A, Room 232. The key for the room may be signed out from EH&S in MSC260.

3) Storage

Do not keep large quantities of perchloric acid on hand.

Perchloric acid that shows any sign of discoloration should be disposed of at once through Hazardous Materials Services.

Store the perchloric acid in either a glass-stopper glass bottle, or in the original bottle. The containers should be kept in a heavy glass, Pyrex, porcelain, or ceramic tray. The tray and
Perchloric Acid
Fume Hood
Procedure

Perchloric acid should be stored in a metal cabinet separate away from organic chemicals or other combustibles, and away from sources of heat.

If experiments are to continue the next morning, it is permissible to store the materials and apparatus within the approved perchloric acid fume hood.

The bottle and tray must be rinsed with water when procedures are complete.

Persons who have picked up bottles that were contaminated with perchloric acid on the outside have received severe acid burns.

Do not allow the perchloric acid to freeze.

4) Spills and Cleanup Supplies

Spills should be neutralized with a liberal amount of 6 N Sodium Hydroxide solutions or a 1:1 mixture of Sodium Carbonate and sand (EXOTHERMIC reaction) and EH&S must be contacted immediately. The spill should be checked for neutralization, then picked up and transferred into a large mouth glass container or temporary spill tray of heavy glass, Pyrex, porcelain, or ceramic. Repeated washing and mopping of the area is required to remove all residues, making sure the mop is thoroughly rinsed out when the cleanup is completed.

Spill clean up supplies (see Spill Response Procedures for suggested content) must be provided by the investigator and be available prior to starting to work with perchloric acid. Ensure that sufficient neutralizer is present for the amount of perchloric acid being used.

- One litre of 6N sodium hydroxide will neutralize about 500 millilitres of 70% perchloric acid.

5) Personal Protective Equipment (PPE)

Consult the MSDS for appropriate Personal Protective Equipment.

Protective rubber gloves, aprons, and eye protection devices should be worn when handling perchloric acid. The use of a full-face shield is recommended for carrying out reactions. Also, a sturdy bench type laboratory shield should be placed between the operator and the reaction.

Work areas should be provided with an emergency shower, and an eye wash station.

6) Use and Handling Precautions

Always use glass, never plastic, labware when using perchloric acid. Perchloric acid should be transferred over a sink into a glass-graduated cylinder and then into the digestion or reaction flask. Another way to measure out the required quantity is with a dispensing burette, which is attached to the storage bottle by means of an adaptor containing a standard ground-glass joint.

Experimental apparatus should utilize glass-to-glass joints. The use of rubber tubing, stoppers, stopcocks, or cork stoppers should be avoided. Polytetrafluoroethylene (Teflon) stopcocks have been used safely. Greases, including silicone types, should not be used. Fluorocarbon lubricants are recommended.

Heat-resistant glassware should be used to minimize breakage due to heat strain if the perchloric acid is to be heated. Vycor and quartz glass has been used successfully to carry out work with 72.5 percent acid, and Teflon and Duriron with hot 70-72 percent acid. Electric hot plates or electrically heated spun-glass mantles are preferred. Sand bath heaters may also be used. Oil baths should never be used to heat flasks or beakers containing hot concentrated perchloric acid. A deep ceramic or glass tray should be built into the apparatus to catch and confine perchloric acid in case of an accident or the beaker breaks.
Perchloric Acid
Fume Hood
Procedure

Thoroughly rinse all labware used in the experiment after each use to remove residues.

- Combustible materials that have been previously wetted with perchloric acid solution and allowed to dry will burn.
- Dilute perchloric acid that was spilled on combustible material such as wood benches or shelving can be ignited by friction, heat, or impact after the perchloric acid dries. Greases, oil, burlap, sawdust, etc. may ignite spontaneously on contact with the acid.
- Clothing that has become contaminated with perchloric acid can be highly flammable and should be removed and rinsed thoroughly with water.

7) Disposal Procedures

- Liquid perchloric acid wastes must be kept separate from other acids.
- Perchloric acid wastes must be placed in a glass bottle.
- Transport all materials and wastes back to your main laboratory. Do not leave any materials in the perchloric acid laboratory. Telephone the waste pickup line to have the wastes removed as soon as possible.
- Do not accumulate large volumes of waste.
- Telephone EH&S at 220-6345 if you have any questions.

This information is provided as a supplement to the information available on the MSDS for Perchloric Acid.
PROCEDURES FOR USING SCIENCE A 232

PERCHLORIC ACID FUME HOOD

1) Sign in and provide the required information in the logbook for every use of the facility.

2) Start the Exhaust fan by pressing the green start button on the fan control box. The red light will indicate that the system is "ON". The fan will start running and there will be movement of the needle on the magnehelic gauge on the front of the fume hood. If there is no noise or movement of the needle on the gauge, DO NOT USE THE FUME HOOD. Contact EH&S immediately at 220-6345.

3) Conduct Experiment.

4) Remove all materials and equipment from the fume hood.

5) The MANUAL SPRAY NOZZLE must be used to WASH down the inner surfaces of the fume hood at the end of the procedure. The manual spray nozzle is located adjacent to the left of the fume hood; the spray nozzle has its own shut off. When done washing down the fume hood, turn manual spray nozzle off.

6) ENSURE THE SASH IS PULLED ALL THE WAY DOWN before the wash down procedure is started. Press the "blue" activation button on the control panel. The light will indicate that the system is "ON". The wash down procedure will be activated and will shut down at the end of the cycle. Ensure that the water is draining away at the back of the hood.

7) Rinse the fume hood down with the manual spray nozzle again.

8) The fume hood is NOT a storage area. If you require the use of the fume hood for more than one day you are required to remove everything from the fume hood and wash the fume hood down. If storing flammable or harmful chemicals in the laboratory restart the Exhaust fan.

9) Please contact EH&S at 220-6345 if you have any questions or concerns.

WASH DOWN THE FUME HOOD
AFTER EVERY USE