**Safe use of Perchloric Acid**

**Department of Materials Engineering**

**University of British Columbia**

**Supervisors in research labs must provide a written safe work procedure for use of perchloric acid as part of necessary safety training. This document may be used or modified by supervisors as needed. Personnel in research labs using perchloric acid must be trained for its use. A record of training must be kept.**

**Hazards**

Perchloric acid is a strong mineral acid. It is one of the strongest acids. It is also an oxidizing acid. Perchloric acid can react vigorously with metals forming HCl and/or hydrogen gas. Hydrogen gas is explosive. Perchloric acid is able to dissolve even gold.

Perchloric acid is strongly corrosive to skin and eyes and may cause severe skin burns and eye damage.

However, the greatest hazard of concentrated perchloric acid is that in contact with organic matter it can cause fires and explosions. Prolonged contact with wood can cause explosive decomposition products to build up which can explode without warning through vibration or mechanical action; the material becomes shock sensitive.

Perchloric acid reaction products formed from contact with metals or organic matter may be shock sensitive and can explode.

72% and less perchloric acid at room temperature is generally not considered to be strongly oxidizing. There are exceptions such as contact with some metals and other chemicals with which it is incompatible. The oxidizing power and threat of fire and explosion increase with increasing temperature and concentration.

Perchloric acid is acutely toxic.

Ordinary clothing (e.g. cotton) is rapidly attached by perchloric acid solutions.

**Handling and Use Requirements**

Perchloric acid forms an azeotrope with water so that the common maximum aqueous solution concentration is 72.5% by weight. Concentrated perchloric acid (70% or higher) is not permitted in the Forward building. Lower concentration perchloric acid may be used in the Forward building under restricted conditions as indicated in this document. At this writing the Brimacombe Building does have a perchloric acid fume hood.

Carefully read an SDS for perchloric acid before using it.

Required personal protective equipment:

Gloves (neoprene, nitrile or PVC)

Labcoat (NOT cotton – rubber or plastic, full length)

Safety goggles (not just safety glasses); preferably a face shield. Impact-resistant eye protection is required.

Use <60% perchloric acid if at all possible. It is less hazardous.

In the Forward building digestion of solid materials with perchloric acid is not permitted.

Evaporation of perchloric acid solutions or perchlorates is not permitted.

Heating perchloric acid with organic matter is not permitted.

Avoid contact with organic matter, except when preparing an organic solution for a documented purpose (such as etching for metallography) using a documented procedure only.

Avoid contact with other reducing agents.

Do not use ordinary rubber stoppers in contact with perchloric acid.

Check incompatibility compilations for contact with any material other than glass.

Do not use perchloric acid over a wood surface; do not contact perchloric acid with wood.

Do not contact perchloric acid with strong dehydrating agents like concentrated sulfuric acid. Highly reactive, very concentrated perchloric acid can form.

Do not contact perchloric acid with metals and metal equipment (except for etching of metal surfaces using a documented procedure).

Heating of aqueous perchloric acid solutions should be done using a temperature-controlled hotplate or a water bath in a glass vessel. Determine in advance if perchloric acid at temperature is compatible with the components of the solution.

Dilute stronger perchloric acid solutions by adding the acid to water, not water to the acid. Otherwise excessive heat can be evolved.

Transfer perchloric acid over a sink with running water or over a containment such as a tray (that is compatible with perchloric acid) to catch minor spills. HDPE is only moderately resistant to damage by 72% perchloric acid at room temperature. PVC is better.

Use good chemical handling practices to avoid contaminating the perchloric acid stock solution. Otherwise it can cause explosion or fire and accumulation of shock-sensitive perchlorate salts due to reaction with accidentally introduced impurities.

Experimental apparatus must be situated over a spill containment such as a tub or tray made of a material that is compatible with perchloric acid at the temperature of the perchloric acid solution. The containment must be able to hold a greater volume than the whole of the perchloric acid solution in use. Again, work may not be done over a wood surface, even if a containment is in place.

Check incompatible materials compilations before contacting perchloric acid with other materials including greases, such as those used for sealing ground glass apparatus. Teflon is compatible with perchloric acid.

**Storage**

Obtain minimum quantities and order in more as needed.

Perchloric acid may not be transferred into smaller bottles and kept at the bench.

Perchloric acid bottles must be stored in a corrosives cabinet and in a secondary containment compatible with the acid. The secondary containment must be big enough to hold more than all of the full bottle of perchloric acid.

The shelf it is stored on must be made of a material that can contain small spills and is compatible with the acid. (If the acid is stored in a corrosives cabinet made of steel the shelves must be lined with a material compatible with the acid.) Storage in cabinets made with wood is not permitted. Storage on wood shelves is not permitted.

Check the perchloric acid regularly for discolouration. Discoloured solutions may be contaminated and must be disposed of at once.

If it is anticipated that the acid will not be used again for several months or so, dispose of it.

**Spills**

Spills must be cleaned up right away.

Do not attempt to mop up perchloric acid with absorbent materials that are incompatible (e.g. paper).

A laboratory spill kit must be available in the lab for dealing with small spills.

Cleaned up spills and adsorbent material must be collected into a plastic pail, labeled and disposed of through ESF.

Spills of substantial volumes of higher concentration per chloric acid (> 1 L) will require emergency assistance. Call 911 for hazardous materials response.

Clean-up of a small spill:

1. Small spills must be neutralized with an acid neutrlaizing adsorbent such as Spill-X A or a base, such as sodium hydroxide or sodium carbonate. If the volume is more substantial and/or the perchloric acid is more concentrated then too much heat may be evolved upon contact with a strong base. Then a dilute base solution should be added slowly.

2. Soak up the neutralized spill with an inorganic based absorbent if a base other than Spill-X A was used.

3. If rags or paper towels are in advertently used, thoroughly wet them with water and place them in a tightly sealed plastic bag for disposal.

4. A second neutralization and rinsing of the wetted area is recommended.

5. Perchloric acid waste must not be mixed with other wastes. It should be placed into acid

resistant containers that are clearly labeled “Perchloric Acid Wastes” on them.

6. Enter the wastes into the ESF waste inventory system and await instructions for pick-up by ESF staff.

Tests for the presence of perchloric acid: (1) Diphenylamine Test. Dissolve one gram of diphenylamine in 10 ml of "1 to 1" (18 normal; 9 M) sulfuric acid to form a diphenylamine sulfate solution. Using a disposable pipette apply this solution to the test surface. The liquid turns black upon contact with perchlorate. The solution also reacts with nitrates, but turns blue. (2) Methylene Blue Test. Use 0.4% solution of methylene blue in water. Add a few drops of indicator solution to about 25 ml of trial solution, such as water used to test rinse from a length of potentially contaminated duct. Perchlorates will produce a violet precipitate.

**Preparation and use of organic solutions**

It may sometimes be necessary to use dilute organic solutions of perchloric acid. This should be done using only a documented procedure from a reputable source. Such solutions must be stored and used as per documented procedures. Check chemical compatibility compilations. Advice should be should be sought from experienced persons.

The supervisor should be consulted to approve the work in advance.

**Disposal**

Small volumes of dilute perchloric acid may be neutralized with dilute sodium hydroxide solution. The solution should be cooled with a cold water bath or an ice bath to keep the temperature down. Neutralized solutions must be disposed of through the Risk Management Environmental Services Facility (ESF).

In general perchloric acid and perchloric acid solutions containing other salts etc. should be disposed of through ESF.

**Emergency procedures**

Perchloric acid is dangerous through inhalation, skin exposure, eye exposure and ingestion. For emergency assistance in the event of contact with perchloric acid call 911. Next call UBC first aid: (604) 822-2662 or APSC first aid (778) 918-6970, Mon.-Fri., 8:30 AM-4:30 PM. Someone will need to meet emergency personnel at the front door. You must know the building address (6350 Stores Road, V6T 1Z4). Obtain a copy of the SDS for emergency personnel.

Skin exposure: Remove contaminated clothing. Use a safety shower or rinse affected area with water for at least 15 minutes. Obtain emergency medical help.

Eye exposure: Rinse with water using an eyewash station for at least 15 minutes. If applicable, remove contact lenses if at all possible. Obtain emergency medical help.

Inhalation: Remove person from the area if safe to do so. Have victim sit in semi-upright position (back at 45° to floor) if possible. Obtain emergency medical help.

Ingestion: Rinse mouth thoroughly with water. Do not induce vomiting. Obtain emergency medical help.

**Partial list of compatible and incompatible materials**

For compatibility with chemicals check incompatibility guidelines. If you notice that a material you are using is attached by perchloric acid then immediately cease using it, rinse it well and arrange for disposal. The list below is NOT comprehensive. The following materials are not recommended for use with 72% perchloric acid:

Nylon/polyamides

Dynel/modacrylic ester

Dacron/polyester

Bakelite

Lucite (PMMA or polymethylmethacrylate)

Vegetable-based Micarta

Cellulose-based lacquers

Copper/brass/bronze (which form shock sensitive salts)

Aluminum (dissolves)

High nickel alloys (dissolve)

Cotton, wool

Wood

The following are suitable for use with 72% perchloric acid:

Viton

Tantalum

Chemically pure titanium

Zirconium

Niobium

Hastelloy C (slight corrosion rate)

PVC

Teflon

Polyethylene (room temperature; attacked at 50°C)

Polypropylene (room temperature; attacked at 50°C)

Kel-F

Vinylidene fluoride

Saran

Epoxy resins

Glass

Glass-lined steel

Alumina

Fluorolube

Indicate by signature below that you have reviewed the Safe use of perchloric acid document. Return to the lab manager or your supervisor.

Name (Print): \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

Signature: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

Date: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_