

Standard Operating Procedure

Perchloric acid

This SOP is not complete until it has been signed and dated by the PI and relevant lab personnel.

Print a copy and insert into your
Laboratory Safety Manual and Chemical Hygiene Plan.
Refer to instructions for assistance.

Department:	Chemistry & Biochemistry - Chemical Engineering
Date SOP was written:	December 14, 2012.
Date SOP was approved by PI/lab supervisor:	January 18, 2013
SOP reviewed by:	Alessandro Moretto, Chem. Lab. Safety Officer
Principal Investigator:	Prof. Susannah Scott
Internal Lab Safety Coordinator/Lab Manager:	Stephanie Goubert-Renaudin
Lab Phone:	805-893-8941
Office Phone:	805-893-7403
Emergency Contact:	EH&S 24 hour line: 805-893-3194 (Name and Phone Number)
Location(s) covered by this SOP:	ESB 3324 and 3328. (Building/Room Number)

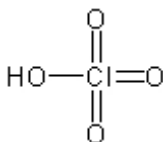
Type of SOP: ☐ Process ☒ Hazardous Chemical ☐ Hazardous Class

Purpose

Perchloric acid is an inorganic compound, usually encountered as an aqueous solution. This colorless compound is a strong acid comparable in strength to sulfuric acid and nitric acid. It is a powerful oxidizer, but its aqueous solutions up to 70% are remarkably inert, only showing strong acid properties and no other oxidizing properties. Above concentrations of 70% the speed of oxidizing reactions rapidly increases with increasing acid concentration. It is useful for preparing perchlorate salts, especially ammonium perchlorate, an important rocket fuel. Perchloric acid is also dangerously corrosive and readily forms explosive mixtures. Perchloric acid is mainly produced as a precursor to ammonium perchlorate, which is used as rocket fuel. The growth in rocketry has led to increased production of perchloric acid. Several million kilograms are produced annually. It is one of the strongest Brønsted-Lowry acids. Despite hazards associated with the explosiveness of its salts, the acid is often preferred in certain syntheses.

Physical & Chemical Properties/Definition of Chemical Group

CAS#:	7601-90-3
Class:	<u>Produces explosive salts, crystals and perchlorate residues</u>
Molecular formula:	HClO ₄
Boiling Point:	203 °C @ 760 mm Hg
Melting Point:	-18 °C
Decomposition Temperature:	Not available



Potential Hazards/Toxicity

EMERGENCY OVERVIEW: Appearance: clear, colorless liquid. Causes digestive and respiratory tract burns. Causes eye and skin burns. Strong oxidizer. Contact with other material may cause a fire. Heating may cause an explosion. Contact with other material may cause explosion. Corrosive to metal.

Highly important danger information!

In addition to being a corrosive liquid, while not combustible, under some circumstances perchloric acid may act as an oxidizer and/or present an explosion hazard. Perchloric crystals, or perchloric acid in crystalline form, is an extremely dangerous inorganic compound. The crystals are sometimes formed due to condensation inside of ventilation hoods in chemical labs or in bottles stored for extended periods in the lab. Among the principle hazards are that the perchlorite crystals are subject to exploding on impact.

Organic materials are especially susceptible to spontaneous combustion if mixed or contacted with perchloric acid. Under some circumstances, perchloric acid vapors form perchlorates (in glove box duct work) which are shock sensitive.

Target Organs: Eyes, thyroid, skin, mucous membranes.

Potential Health Effects:

Eye: Causes eye burns.

Skin: Causes skin burns.

Ingestion: Harmful if swallowed. Causes gastrointestinal tract burns.

Inhalation: Causes severe respiratory tract irritation with possible burns.

Chronic: Prolonged or repeated skin contact may cause dermatitis.

Personal Protective Equipment (PPE)

Eyes: Wear safety goggles. Use chemical splash and impact-rated goggles.

Skin: Natural Rubber, PVC, Nitrile or Viton gloves must be worn while handling concentrated perchloric acid

Clothing: Wear long pants, closed toed shoes and a lab coat

Respirators: A respiratory protection program that meets OSHA's 29 CFR 1910.134 and ANSI Z88.2 requirements or European Standard EN 149 must be followed whenever workplace conditions warrant a respirator's use.

Engineering Controls

Any procedure involving heating of perchloric acid must be conducted in a perchloric acid fume hood, with the sash down. Without heating, it should be handled in fume hood and avoid contact with organic materials. No organic materials should be stored in the perchloric acid hood.

First Aid Procedures

Eyes: In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Get medical aid immediately.

Skin: Throw away contaminated shoes as the hazardous waste. In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Get medical aid immediately. Wash clothing before reuse.

Ingestion: If swallowed, do NOT induce vomiting. Get medical aid immediately. If victim is fully conscious, give a cupful of water. Never give anything by mouth to an unconscious person.

Inhalation: If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical aid.

Special Handling and Storage Requirements

Handling: Avoid contact with clothing and other combustible materials. Do not get on skin or in eyes. Do not ingest or inhale. Use only with adequate ventilation. Do not allow perchloric acid to come into contact with strong dehydrating agents (for example; concentrated sulfuric acid, anhydrous phosphorus pentoxide). Keep the quantities of perchloric acid handled at the bare minimum for safety. Perchloric acid mist and vapor can condense in ventilation systems to form metallic perchlorates, which can be explosive.

Storage: Do not store near combustible materials. Store in a tightly closed container. Store in a cool, dry, well-ventilated area away from incompatible substances. Avoid storage on wood floors. Perchloric acid should be stored in a secondary containment (such as pyrex baking dish) in the "Acid" cabinet. It must not be stored near organic acids, near bases, or near other organic or flammable material. Shelves and floor material should be non-combustible and acid-resistant. Protect from freezing.

Spill and Accident Procedure

Chemical Spill Dial 9-911 and EH&S (805-893-3194)

Spill – Clean up perchloric acid spills immediately, observing precautions in the Protective Equipment section. Remove all sources of ignition. Absorb spill using an absorbent, non-combustible material such as earth, sand, or vermiculite. Do not use combustible materials such as sawdust. Flush spill area with water. Wet area to prevent drying out. Provide ventilation. Keep combustibles (wood, paper, oil, etc.,) away from spilled material. Keep unnecessary and

unprotected personnel away. Use only non-sparking tools and equipment. Spill may be carefully neutralized with soda ash (sodium carbonate).

Small (<1 L) – If you have training, you may assist in the clean-up effort. Use appropriate personal protective equipment and clean-up material for chemical spilled. Double bag spill waste in clear plastic bags, label and take to the next chemical waste pick-up.

Large (>1 L) – Dial **9-911** from campus phones (and 805-893-3446 from a cell phone) and EH&S for assistance.

Chemical Spill on Body or Clothes – Remove clothing and rinse body thoroughly in emergency shower for at least 15 minutes. Seek medical attention. *Notify supervisor and EH&S immediately.*

Chemical Splash Into Eyes – Immediately rinse eyeball and inner surface of eyelid with water for 15 minutes by forcibly holding the eye open. Seek medical attention. *Notify supervisor and EH&S immediately.*

Medical Emergency Dial 9-911

Life Threatening Emergency, After Hours, Weekends and Holidays – Dial **9-911** (or 805-893-3446 from a cell phone) or go to the Emergency Room of Goleta Valley Cottage Hospital at 351 South Patterson Avenue, Goleta (Phone number: 805-967-3411) Note: All Serious injuries must be reported to EH&S within 8 hours.

Non-Life Threatening Emergency – Go to the Student Health Building, Building 588 (phone number: 893-5361, hours: M, T, R, F 8am-4.30pm, W 9am - 4.30pm, R 5pm to 7pm by appointment). After hours go to the Emergency Room of Goleta Valley Cottage Hospital at 351 South Patterson Avenue, Goleta (Phone number: 805-967-3411) Note: All serious injuries must be reported to EH&S within 8 hours.

Needle stick/puncture exposure (as applicable to chemical handling procedure) – Wash the affected area with antiseptic soap and warm water for 15 minutes. For mucous membrane exposure, flush the affected area for 15 minutes using an eyewash station. Page the needle stick nurse \ and then enter your extension. After hours go to the nearest emergency room: the Emergency Room of Goleta Valley Cottage Hospital at 351 South Patterson Avenue, Goleta (Phone number: 805-967-3411). Note: All needle stick/puncture exposures must be reported to EH&S within 8 hours.

Decontamination/Waste Disposal Procedure

Wearing proper PPE, please decontaminate equipment and bench tops. Call EH&S for assistance if needed. Please dispose of the spent perchloric acid and disposables contaminated with it as hazardous waste.

General hazardous waste disposal guidelines:

Label Waste

- Affix an on-line hazardous waste tag on all waste containers as soon as the first drop of waste is added to the container

Store Waste

- Store hazardous waste in closed containers, in secondary containment and in a designated location
- Waste must be under the control of the person generating & disposing of it

Dispose of Waste

- Dispose of regularly generated chemical waste within 90 days
- Call EH&S for questions
- Empty Containers

- Dispose as hazardous waste if it once held extremely hazardous waste (irrespective of the container size)
- Consult waste pick-up schedule
- Prepare for transport to pick-up location
 - Check on-line waste tag
 - Write date of pick-up on the waste tag
 - Use secondary containment

Material Safety Data Sheet (MSDS) Location

MSDS can be accessed online:

<http://www.sigmaaldrich.com/MSDS/MSDS/DisplayMSDSPage.do?country=US&language=en&productNumber=311421&brand=ALDRICH&PageToGoToURL=http%3A%2F%2Fwww.sigmaaldrich.com%2Fcatalog%2Fsearch%3Finterface%3DAll%26term%3Dperchloric%2Bacid%26lang%3Den%26region%3DUS%26focus%3Dproduct%26N%3D0%2B220003048%2B219853269%2B219853286%26mode%3Dmatch%2520partialmax>

Protocol/Procedure

In the lab, concentrated perchloric acid (70% assay) is used in the preparation of diluted solutions (for example water and acetonitrile). When working with organic solvents, perchloric acid is diluted at a low concentration (e.g. 100mM) and is used to keep the sample acidic.

Perchloric acid is stored in the ventilated “Acid” storage cabinet, in a secondary container. Prior to each use, make sure no crystals are observed on the container. If so, do not move the bottle and immediately call EHS for disposal.

When handling perchloric acid, the appropriate PPE is required, including natural rubber, PVC, nitrile or Viton gloves, safety goggles and a lab coat.

Concentrated perchloric acid is always handled within the fume hood and on a cleared space. HClO₄ solutions are always contained in cap-sealed/closed containers.

Preparation of diluted perchloric acid solutions is done by slowly adding the acid to the solvent, at room temperature and 1 atm. Perchloric acid solutions cannot be boiled in the fume hood: the vapors can condense in the ducts, and if they dry out, the residue can react violently with organic materials, and become explosive.

Before being disposed, the small amount of unused 70% HClO₄ is diluted with large excess of water. HClO₄ solution is then discarded in the appropriate waste container kept closed at all times.

Note that heating of perchloric acid, or use in concentrations >72% drastically alter the hazard profile of this chemical. Any deviations in procedure involving these two variables must be fully researched as to the hazards involved, and the new procedure approved by the PI!

Note: Any deviation from this SOP requires written approval from PI.

Documentation of Training (signature of all users is required)

- Prior to conducting any work with perchloric acid, designated personnel, i.e. approved users listed below, must provide training to his/her laboratory personnel specific to the hazards involved in working with this substance, work area decontamination, and emergency procedures.

- The Principal Investigator must provide his/her laboratory personnel with a copy of this SOP and a copy of the SDS provided by the manufacturer.
- The Principal Investigator must ensure that his/her laboratory personnel have attended appropriate laboratory safety training or refresher training as required by EH&S.

I have read and understand the content of this SOP:

Name	Signature	Trainer	Date
Prof. Susannah Scott			
Stephanie Goubert-Renaudin			
Gary Kwanyi Ng			
Alessandro Gallo			
Anthony Crisci			
Haibo Yu			
Taeho Hwang			
Bethany Wigington			
Daniel Coller			
Zachary Jones			
Youhong Wang			
Jinghong Zhou			
Jason Fendi			
