

Guidelines for Using and Storing Perchloric Acid

I. Introduction

Perchloric acid is a strong mineral acid commonly used as a laboratory reagent. It is a clear, colorless liquid with no odor. Most perchloric acid is sold as 60%-62% or 70%-72% acid in water. The OSHA Lab Standard requires a Chemical Hygiene Plan that includes standard operating procedures relevant to safety and health considerations for laboratory work that involves the use of hazardous chemicals. This particular standard operating procedure was created with the goal of complying with 29 CFR 1910.1450 (e)(3)(i) and educating the Baylor community in the safe use of perchloric acid.

II. Scope

These guidelines present information on how to handle and store perchloric acid safely. **All Baylor lab personnel who work in labs containing perchloric acid should familiarize themselves with this document.** A sign off sheet should be kept by the lab safety coordinator/PI that states the person has read this document and is aware of the unique dangers and special precautions that need to be taken when handling this acid.

III. Overview of Hazards

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

Perchloric acid can be a health hazard if inhaled, ingested or splashed on skin or in eyes. To prevent injury, goggles or a face shield over safety glasses, gloves and an apron over lab coat, should be worn when handling perchloric acid. Symptoms of overexposure include irritation and/or burning of the affected area. Inhalation burns are serious and require immediate medical attention. If perchloric acid is ingested, drink approximately 8 oz. of water and seek medical attention. Do not induce vomiting.

Because of its reactivity hazard, perchloric acid digestions of any size must always be performed in a chemical hood. Those that require temperatures above ambient should be conducted in a special perchloric acid hood that is equipped with a wash down system. Hoods used for hot digestions must be labeled "Perchloric Acid Hood Only. Organic Chemicals Prohibited." Solvents must never be used or stored in a designated perchloric acid hood.

IV. Using Perchloric Acid

A. \leq 72% Perchloric Acid at Room Temperature

At room temperature, perchloric acid up to concentrations of 72% has properties similar to other strong mineral acids. It is a highly corrosive substance and causes severe burns on contact with the eyes, skin, and mucous membranes. When used under these conditions, perchloric acid reacts as a strong non-oxidizing acid. The following precautions should be taken when using perchloric acid under these conditions:

- Substitute with less hazardous chemicals when appropriate. Use dilute solutions (<60%) whenever possible.
- Conduct operations involving cold perchloric acid in a properly functioning laboratory (chemical) hood with current certification sticker.

- Always use impact-resistant chemical goggles, a face shield, neoprene gloves, and a rubber apron when handling perchloric acid.
- When using or storing even dilute perchloric acid solutions avoid contact with strong dehydrating agents (concentrated sulfuric acid, anhydrous phosphorous pentoxide, etc.). These chemicals may concentrate the perchloric acid and make it unstable.
- Always transfer perchloric acid over suitable containment in order to catch any spills and afford a ready means of cleanup and disposal.
- Perform all operations on chemically resistant surfaces. Avoid contact with cellulose materials such as wood, paper and cotton., which could result in a fire or explosion.

B. $\leq 72\%$ Perchloric Acid Heated

When heated to temperatures above 150° C perchloric acid becomes a strong oxidizer and eventually becomes unstable. Concentrated solutions are very dangerous and can react violently with many oxidizable substances, such as paper and wood, and can detonate. Vapors may also contaminate work surfaces or ventilation equipment with perchlorate residues, which may form highly unstable compounds, such as metallic perchlorates. These compounds may ignite or detonate under certain conditions. The following additional precautions should be followed when heating perchloric acid:

- Perchloric acid digestions and other procedures performed at elevated temperatures should be done in a specially designed perchloric acid fume hoods. If procedures involving heated perchloric acid are performed only rarely other accepted methods to capture and contain vapors may be used in place of a perchloric acid hood.
- Lower the fume hood sash as much as possible so that it can function as a physical barrier or use a safety shield to provide splash/splinter protection. Perchloric acid fume hoods should have shatterproof glass.
- Never heat perchloric acid in an oil bath or with an open flame. Electric hot plates, electrically or steam-heated sand baths, heating mantles, or steam baths are preferred. Use explosion proof electrical equipment.
- Avoid allowing hot perchloric acid to come into contact with any organic materials, including paper or wood, because a fire or explosion can occur. Avoid storing these materials in perchloric acid work hoods. Avoid using greases or hoses that are incompatible with perchloric acid.
- Be sure you understand the reaction(s) that can occur when using perchloric acid. Perchloric acid may react violently with many chemicals, including acetic anhydride, alcohol, reducing agents, and many metals.
- In wet digestions with perchloric acid, treat the sample first with nitric acid to destroy easily oxidizable matter.
- Do not distill perchloric acid in a vacuum, because the unstable anhydride may be formed and cause a spontaneous explosion. Protect vacuum sources from perchloric

acid/perchlorate contamination. Vacuum pumps should be thoroughly flushed and refilled with Kel-F or Fluorolube.

- Wash down perchloric acid hoods after each use, following operating instructions provided by the manufacturer of the perchloric acid hood.
- All apparatus should have glass-to-glass joints. Use silicon based lubricants for glass-to-glass joints. Do not use rubber stoppers, tubes or stopcocks.
- If an apparatus cracks or breaks due to thermal or mechanical shock, the hazards are sufficient to make it desirable to consider using quartz apparatus since it is necessary in many experiments to chill perchloric acid rapidly from the boiling point.
- When handling beakers of hot acid use properly designed tongs or other remote-handling devices.

C. Anhydrous Perchloric Acid

Anhydrous perchloric acid (> 85% concentration) is very unstable and will usually explode when it comes in contact with organic materials. Follow these additional precautions when working with anhydrous perchloric acid.

- Allow only experienced research workers to handle anhydrous perchloric acid. These workers shall be thoroughly familiar with the literature on the acid. Assure that a second worker is informed of the intended use of the anhydrous perchloric acid. This second worker should be in sound or sight contact with the worker using anhydrous perchloric acid.
- Use a safety shield to protect oneself against the effects of a possible explosion.
- Use the acid in a designated, properly designed perchloric acid hood with a minimum of equipment present. No extraneous chemicals should be present in the hood.
- Use thick gauntlets in addition to PPE previously recommended.
- Use only freshly prepared acid. Do not make any more anhydrous perchloric acid than is required for a day/shift.

V. Perchloric Acid Storage

The quantities of perchloric acid kept in storage should be kept to a minimum. Perchloric acid should be stored in its original container within compatible secondary containment, preferably glass or porcelain. Glass trays should be wiped periodically.

Perchloric acid should be separate from other chemicals, but may be stored with other inorganic acids, preferably in a metal cabinet designed for acid/corrosive storage. Small quantities may also be stored in a perchloric acid hood. Perchloric acid must be stored away from organic chemicals, flammable or combustible materials and strong dehydrating agents such as sulfuric acid and anhydrous phosphorus pentoxide.

If a bottle containing perchloric acid has turned dark and has crystals forming around the bottom of the bottle, there is a potential explosion hazard. Do NOT move the bottle. Immediately contact Environmental Health & Safety.

VI. Perchloric Acid Spills

CLEAN UP SPILLS OF PERCHLORIC ACID ONLY IF YOU HAVE BEEN TRAINED TO DO SO AND THE APPROPRIATE EQUIPMENT IS AVAILABLE!

Contact Environmental Health & Safety for assistance.

To clean a spill, neutralize it with soda ash (sodium carbonate) or other appropriate neutralizing agent. Soak up the neutralized spill with an inorganic based absorbent, if possible. Do not use organic materials, such as kim-wipes or toweling as they may spontaneously ignite upon contact with perchloric acid. If rags or paper towels are inadvertently used, wet them with water and place them in a tightly sealed plastic bag. Do NOT use rags, paper towels, or sawdust and then put them aside to dry out, as such materials may spontaneously ignite. A second neutralization and rinsing of the wetted area is recommended.

Perchloric acid waste must not be mixed with other wastes. It should be placed into acid resistant containers that are clearly labeled and held for disposal.

VII. References

Common Questions About Perchloric Acid, GFS Chemicals:

<http://www.gfschemicals.com/TechnicalLibrary/Questions-About-Perchloric-Acid.asp>

CRC Handbook of Laboratory Safety 4th Edition, 1995 by CRC Press LLC

NFPA 45 *Standard on Fire Protection for Laboratories using Chemicals*
