Working with Perchloric Acid Guideline

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1 Introduction

This document is an addendum to "**HSE-RST-Chem001G_working with Corrosives**" and is offered to provide a short and concise overview of a widely used and unique hazardous corrosive material. Understanding of the parent document is a crucial prerequisite in understanding basic safety fundamentals such as 1) hazard awareness, 2) engineering controls, 3) work practices, 4) PPE and 5) emergency response for working with corrosive materials.

2 Scope

The guideline applies to lab personnel, and it has been developed to assist them in the preparation of lab specific SOPs.

3 Procedure

3.1 Introduction to Perchloric Acid

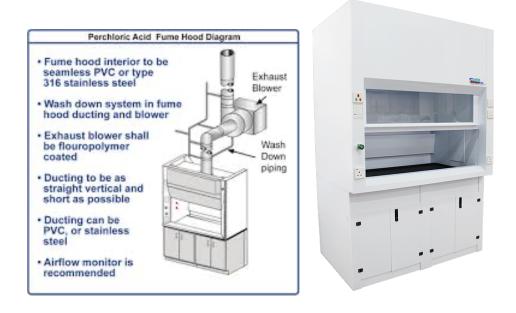




Perchloric acid is not only a highly corrosive material it is a strong oxidizer as well. Therefore not only will one need to follow the basic precautions of working with corrosive materials as detailed and outlined in the preceding pages but one will need to follow basic guidelines of working with oxidizers as well.

Because perchloric acid becomes increasingly unstable (volatile) with increasing concentration, it is not marketed at concentrations greater than 72% v/v in water. It may explode by shock or concussion as an anhydrous material (e.g., if a spill of 70% perchloric acid is allowed to dehydrate).

Perchloric acid does readily react with metals to form potentially explosive metal perchlorate salts. This is why if perchloric acid is to be used on a regular basis in fume hoods, these fume hoods need to be specially designed and have a wash down feature. Perchloric acid fume hoods are specially designed with this wash down feature as well as other features such as a collection basin and non-reactive furniture (e.g. plastic, ceramic, etc.) to reduce metal-perchloric acid interaction. Perchloric acid fume hoods are rare and mostly found in institutions where Geology type work is done – Perchloric acid is heated and used for rock and mineral digestions.



When working with perchloric acid, be sure to remove all organic materials, such as solvents, from the immediate work area. Due to the potential danger of perchloric acid, if possible, try to use alternate techniques that do not involve the use of perchloric acid. If you must use perchloric acid in your experiments, only purchase the smallest size container necessary.

Key take away points for perchloric acid (in addition to the already stated for Corrosive Materials)

- Never clean up spills of perchloric acid with spill pads or paper towels. Neutralize perchloric acid first.
- Store perchloric acid away from metals and organic materials (organic acids). Use secondary
 containers if you must store perchloric acid with organic acids. Anhydrous perchloric acid is a
 powerful oxidizer that may explode if it comes in contact with organic materials. Anhydrous
 perchloric acid can be produced when heating perchloric acid, during the evaporation of
 perchloric acid, or during reactions involving dehydrating agents.
- Be extremely cognizant of the material (organic, metals, etc) and manner (heating, diluted or concentrated) you use perchloric acid with.
- Never heat perchloric acid, unless you have special training, as this helps to dehydrate it and
 makes it more reactive and potentially explosive. Procedures involving heated perchloric acid,
 reactions involving dehydrating agents, or routine use of perchloric acid must be conducted in a
 perchloric acid fume hood equipped with a water wash-down system. The wash down system
 should be turned on immediately after perchloric acid has been heated in the hood or after
 general use of the fume hood. Step by step instructions should be written on how to operate the
 wash-down for perchloric acid hoods.
- Chemicals that are incompatible with anhydrous or hot concentrated perchloric acid include acetic anhydride, acetic acid, aniline, carbon (wood charcoal & carbon black), paper, wood, fiber, or sawdust.
- Tests shall be conducted for explosive perchlorates before any inspection, cleaning, maintenance, or other work performed on the exhaust system or hood interior.

• All containers of perchloric acid should be inspected regularly for container integrity and the acid should be checked for discoloration. Discolored perchloric acid should be discarded as hazardous waste.

3.2 Storage and Handling

- Perchloric acid should be used only in standard analytical procedures from well recognized analytical texts. Work with > 85% perchloric acid requires special precautions and should be carried out only by specially trained personnel.
- As a minimum, splash goggles, nitrile gloves, and a lab coat should be worn when handling perchloric acid.
- Always transfer perchloric acid over a chemical resistant catch tray in order to catch any spills and afford a ready means of disposal.
- Precautions should be taken to prevent the buildup of explosive perchlorates. Light, mechanical shock, heat and certain catalysts can be initiators of explosive reactions with the perchlorates that may be formed from perchloric acid. Anhydrous acid which may be formed with strong dehydrating agents decomposes at ordinary temperatures and explodes on contact with most organic materials. Perchloric acid containers should be kept open no longer than 15 minutes per experiment.
- Perchloric acid should be stored in well-ventilated locations separated from organic substances and other combustible materials. Do not store perchloric acid in a refrigerator or other dehydrating atmosphere.
- Keep incompatible chemicals away from perchloric acid and the area in which perchloric acid will be used. Those chemicals that are incompatible with perchloric acid include oxidizable organic compounds such as alcohols, ketones, aldehydes, ethers, and dialkyl sulfoxides; strong acids such as sulfuric acid; dehydrating agents; anhydrous phosphorous pentoxide; formaldehyde; antimony or bismuth; and reducing agents. Seventy percent perchloric acid may react with cellulose materials such as wood, paper, and cotton. Preventing contact with incompatible chemicals during storage may be accomplished by placing perchloric acid containers in nonbreakable, chemical resistant containers which are capable of holding the entire contents of the container.

3.3 Fume Hoods

Heating of perchloric acid or perchloric acid reactions that involve heat shall *NOT* be conducted in a general purpose fume hood. A special perchloric acid hood is needed for these experiments.

Use of perchloric acid (<72%) at ambient temperature may be conducted in a general purpose fume hood if the following procedures are followed:

- Only small quantities are used on an infrequent basis.
- Easily accessible areas in the fume hood, which are exposed to perchloric acid, are immediately wet wiped or rinsed with a squirt bottle of distilled water after use. This procedure prevents the buildup of explosive perchlorates. Periodic methylene blue tests should be conducted after each perchloric acid use for the presence of any perchlorates.

3.3.1 Perchloric acid fume hoods

Perchloric acid hoods are specifically designed for the use of perchloric acid and other material that can deposit shock sensitive crystalline materials in the hood and exhaust system. Only those chemicals for which the hood is specifically designed should be used in a perchloric acid hood.

3.4 In the event of exposure

In the event of skin contact, immediately wash with soap and water and remove contaminated clothing. Seek medical attention immediately. In case of eye contact, promptly wash with copious amounts of water for a minimum of 15 minutes (lifting upper and lower lids occasionally) and obtain medical attention. If perchloric acid is ingested, obtain medical attention immediately. If large amounts of this compound are inhaled, move the person to fresh air and seek medical attention attention at once.

3.5 Spills

- Spill control materials should be available to control the release of perchloric acid. Appropriate protective equipment for clean-up should be worn (i.e., lab coats, protective gloves, protective rubber boots).
- Perchloric acid spilled on the floor or bench top represents a hazard since the evaporation of the spill may lead to the formation of more dangerous concentrations of the acid. It should **not** be mopped up, **nor** should it be soaked up with dry combustibles.
- Remove all combustibles from the surrounding area (i.e., wood, paper, oils). A water spray may be used to help reduce vapors and keep the area wet. Measures should be taken to keep the material or spill area

from drying. Neutralize the spill with a dilute solution of sodium bicarbonate and then use absorbent material such as universal pads or absorbent clay to absorb it. Place the material in closed flammable waste disposal can.

• The area of the spill should be thoroughly rinsed once again and tested for the presence of perchlorates. You may want to neutralize this area also.

3.6 Hazardous waste disposal

Excess perchloric acid and waste material containing perchloric acid should be placed in a glass reagent container and labeled as Hazardous Waste.

4 References

- SHA 3404-11R (2011) Laboratory Safety Guidance
- KAUST Laboratory Safety Manual
- HSE-RST-Chem001M Chemical Safety Program
- University of Georgia

5 Help

Questions about this guideline? Contact: hse@kaust.edu.sa