

	Environment, Health & Safety Office	SOP #:	EHS-0054
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Perchloric Acid SOP

1. Purpose / Background

The purpose of this document is to provide information and procedures to assure that concentrated perchloric acid is used safely per the MIT Environmental, Health and Safety (EHS) Policy and in accordance with the Guiding Principles in Support of the EHS Policy. To view the EHS Policy and Guiding Principles, go to <http://ehs.mit.edu/site/content/mit-environmental-health-and-safety-policy>.

Quantities of less than 1 gram of concentrated perchloric acid have caused several severe laboratory accidents with the most common cause being contact with organic material or accidental formation of anhydrous perchloric acid (CRC Handbook of Laboratory Safety).

As the temperature and concentration of perchloric acid increases, the oxidizing power increases. Anhydrous perchloric acid or perchloric acid used at elevated temperatures (320^o F, 160^o C) is a very strong oxidizing agent that will ignite in contact with organic material or explode under conditions of shock and heat. At room temperature, 73% or greater perchloric acid is a good oxidizer. When heated, perchloric acid can condense in ventilation ductwork and form very explosive perchlorates.

2. Scope

This SOP is a generic guideline that can be used to aid MIT laboratories and shops to use and store perchloric acid safely. If the Principal Investigator determines that an experiment specific SOP for their work with perchloric acid is warranted, this SOP can be used as a guideline.

3. Prerequisites

Laboratories working with perchloric acid must have a Chemical Hygiene Plan. Shops working with perchloric acid must have a Hazard Communication Program.

4. Procedures

4.1 General Use Procedures

- **Note:** Anhydrous perchloric acid (73% or greater) is inherently unstable and its use requires special precautions. Contact EHS prior to any work involving anhydrous perchloric acid. A lab specific SOP will be required.
- **Information:** Acquire a material safety data sheet (MSDS) for perchloric acid. Due to the severe potential hazards of perchloric acid, carefully review the handling and storage procedures and become familiar with the chemical and physical properties before beginning work. Always review the section on incompatibility and the conditions to which perchloric acid is sensitive. Read the manufacturers' recommendations contained in supplementary documents, such as technical bulletins and contact the EHS Office to review new uses of perchloric acid.
- **Training:** Anyone using perchloric acid should have adequate training and knowledge of the hazards and of the practices and procedures for working with it safely. A lab specific SOP can help with such training. See section 6 for detailed training requirements.
- **Storage Quantities:** See 4.2 General Storage Procedures



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- **SOP:** An experiment specific SOP may be warranted for procedures that involve heating or generating aerosols of perchloric acid or if using concentrations greater than 72%.
- **Personal Protective Equipment:** Due to the corrosivity of perchloric acid, lab coat, safety glasses, and goggles or a full face shield must be worn at all times. If heating perchloric acid or using concentrations greater than 72%, chemical splash and impact-rated goggles, faceshield, rubber gloves and a rubber or plastic apron must be worn.
- **Work practice and engineering controls for procedures that involve heating perchloric acid, or using greater than 72% perchloric acid:**
 - All experiments should be conducted in a water-wash down fume hood that has wash down capabilities in the fan, ductwork, and hood; an individual exhaust system that is discharged outdoors and is equipped with straight runs without sharp turns; and a hood and ductwork that are constructed of noncombustible, non-reactive and acid resistant materials, such as stainless steel or PVC. The EHS laboratory has a water-wash down fume hood that is available for use. For infrequent use of small quantities (milliliters) of concentrated perchloric acid, a local scrubbing system that is approved by the EHS Office can be constructed inside the fume hood in place of a water-wash down hood. Contact the EHS Office for more information.
 - The wash down function on the fume hood should be activated after each use or at least at the end of the day. In addition, the interior of the hood should be washed down by hand daily if the hood is not equipped with spray nozzles. Do not begin heated perchloric acid experiments until the hood has been washed down.
 - Restrict the use of the fume hood by posting a warning sign stating “Perchloric Acid Use Only. Organic chemicals prohibited.”
 - Organic solvents should never be used or stored in a designated perchloric acid hood. Avoid having extra equipment in the hood and work with the sash in the lowest feasible position.
 - The fume hood should be inspected frequently for the accumulation of perchlorates, and any deposits should be saturated with water and removed. If a fume hood is suspected of having perchloric acid or perchlorate contamination, contact the EHS Office to perform a survey before any maintenance work is done.
 - Only experienced laboratory workers using the smallest quantities necessary for the experiment should perform this work. There should be at least two people in the laboratory at all times during the experiments.
 - Do not use direct flame or oil baths to heat perchloric acid. Electric hot plates, water or steam baths should be used. Quartz apparatus is recommended since it is less likely than glass to develop cracks and break.
 - Tongs with a modified jaw design should be used for handling hot flasks and beakers. Crucible tongs do not provide a safe grip and should not be used.
 - Use a safety shield to protect against a possible explosion that may result if perchloric acid contacts organic material.
 - Use freshly prepared perchloric acid in quantities to be used up in the same day; at the end of the day neutralize any remaining acid.
 - Do not use in laboratories with wood floors or wood benchtops. If a spill occurred on wood floors and dries, friction could ignite the wood.



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- Whenever possible, work over a plastic tray that will contain spills and allow a quick clean up.
- If a vacuum source is necessary, contact the EHS Office for recommendations.
- **Work practice controls for handling cold 72% or weaker perchloric acid and perchloric acid hazardous waste**
 - Do not mix perchloric acid waste with other chemical waste. Collect perchloric acid waste separately in an acid resistant glass bottle and place in a plastic secondary container.
 - Do not allow perchloric acid to come into contact with sulfuric acid or other strong dehydrating agents, such as anhydrous phosphorous pentoxide.
 - Rubber stoppers, tubes or stopcocks are incompatible with perchloric acid and should not be used with it.
 - Transfer over a compatible plastic spill tray to catch small spills that water can be added to quickly.
 - Notify the EHS Office immediately if a bottle of perchloric acid appears discolored. The EHS Office will arrange for immediate pickup and proper disposal

4.2 General Storage Procedures

Storing concentrated perchloric acid should be part of a comprehensive chemical storage plan that is outlined in the Chemical Storage SOP. Read the MSDS to determine specific storage recommendations or special storage conditions.

- **Storage Quantities:** The Massachusetts State Building Code 780 CMR Table 307.8(1) limits the amount of Class 3 oxidizers per control area to 10 pounds and the amount of Class 4 oxidizers to 1 pound (see Appendix A). In addition, Class 4 oxidizers are permitted only in buildings equipped throughout with an automatic sprinkler system in accordance with 780 CMR 906.2.1. Perchloric acid 60%-72% by weight is a Class 3 oxidizer. Perchloric acid greater than 72% is a Class 4 oxidizer.
- Do not store on wooden shelves without a plastic secondary container that is large enough to hold the contents of the bottle.
- Do not store with organic materials, flammables, acetic acid, sulfuric acid, other strong dehydrating agents, or bases.
- Store in glass bottles and periodically inspect the plastic cap on the perchloric acid bottle for deterioration and replace when necessary.
- Notify the EHS Office immediately if a bottle of perchloric acid appears discolored. The EHS Office will arrange for immediate pickup and proper disposal.

4.3 General Emergency Procedures

Plan ahead for possible emergencies involving perchloric acid. All personnel who work in areas where perchloric acid is used should be trained in how to respond to potential emergencies.

- Prior to using perchloric acid, consult the MSDS for the appropriate clean-up supplies and ensure that they are readily available.
- Notify people in the area that a spill has occurred.
- For major spills, turn off sources of ignition and vacate the lab immediately. Do not attempt to clean up a large spill since respiratory protection would be required.
- If a minor spill occurs in a plastic tray, dilute the perchloric acid with water to a concentration of <5%, absorb with sand or vermiculite, place in a plastic container, and dispose of as hazardous waste.



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- If perchloric acid is spilled on the bench top or floor, dilute the acid immediately with some water and neutralize it with soda ash, bicarbonate, or lime. Use an inorganic based absorbent or a plastic foam type absorbent to soak up the solution. Do not use rags, paper towels or sawdust to wipe up the spill since these can dry out and become a fire hazard.
- If the spill is on wood, the wood should be soaked thoroughly with water and the contact area should be removed and replaced.
- Contaminated clothing is considered flammable and must be removed and washed thoroughly with water. Do not dry with heat.

5. Roles & Responsibilities

5.1 The EHS Office is responsible for:

- Providing General Chemical Hygiene Training (web-based or classroom) and HAZCOM training that includes information on chemical hazards.
- Maintaining up to date guidance for the use and storage of perchloric acid.
- Reviewing SOP's and new uses of perchloric acid.
- Addressing questions or concerns regarding perchloric acid.
- Assisting with inspections of use and storage areas for perchloric acid.

5.2 PI's/Supervisors are responsible for:

- Evaluating the need for experiment specific SOPs for perchloric acid use in their laboratory.
- Ensuring that those individuals that they supervise who work with perchloric acid receive adequate training (see Section 6.0 for training requirements.)
- Ensuring that perchloric acid is used and stored safely in the laboratory or work areas that they supervise.
- Ensuring perchloric acid is used and stored in the smallest quantities necessary in the work areas that they supervise.

5.3 The DLC EHS Coordinator or Chemical Hygiene Officer is responsible for:

- Addressing questions or concerns regarding the use or storage of perchloric acid, and consulting with the EHS Office if necessary.
- Inspecting chemical storage areas twice a year during Level II inspections and notifying the laboratory personnel and the PI/Supervisor of problems found that must be corrected or prevented.

5.4 The EHS Representatives are responsible for:

- Assisting the PI/Supervisors with the safe use and storage of perchloric acid in the work area. Specific duties may include periodically inspecting use and storage areas.

5.5 Individuals using perchloric acid are responsible for:

- Knowing and following the perchloric acid SOPs established in their laboratory or work area.
- Assuring that they have adequate training and using perchloric acid in accordance with the training guidance provided.



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- Reporting any incidents, problems or concerns with handling materials to the PI, EHS Coordinator, or EHS Office.
- Wearing the PPE that is specified for perchloric acid use.

6. Training

All laboratory personnel working with perchloric acid must have completed General Chemical Hygiene Training (web-based or classroom) and Laboratory Specific Chemical Hygiene Training.

The Laboratory Specific Chemical Hygiene Training should include the following information if perchloric acid is used in the laboratory or work area:

- The hazards and safe use of perchloric acid.
- The location and function of specialized equipment needed for the safe use of perchloric acid.
- The proper procedure for safe storage of perchloric acid.
- Procedures to be used in case of an emergency with perchloric acid.
- The location of MSDSs and SOPs for perchloric acid.

For non-laboratory personnel that use chemicals in their work area General HAZCOM Training is provided through the EHS Office.

Awareness level training should be given to others who work in areas where greater than 72% perchloric acid is present.

Laboratories or DLCs desiring additional training for certain applications of perchloric acid may contact the EHS Office for help in developing and implementing training specific to their needs.

7. Monitoring Requirements

Work areas where perchloric acid is used and stored should be inspected at least twice a year during the Level II inspections to assure that it is being used and stored safely and in accordance with the rules established for the area.

8. Record Management

The DLC EHS Coordinator and the EHS Office shall maintain records of Level II inspections of storage areas containing perchloric acid. All records related to the use and storage of perchloric acid should be maintained per the Records Retention SOP.

9. References

The following references are available through the EHS Office:

9.1. Standards

- OSHA 1910.1450 Occupational Exposure to Hazardous Chemicals in Laboratories
- Massachusetts State Building Code 780 CMR Table 307.8(1)

9.2 Other SOP/SOGs

- Chemical Storage SOP
- Records Retention SOP



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9.3 Supplementary Documents

- MIT Environment, Health and Safety Policy
- MIT HAZCOM Program
- CRC Handbook of Laboratory Safety

9.4 Helpful Websites

OSHA Regulations and Technical Manuals: <http://www.osha.gov>

10 Definitions

- 10.1 Material Safety Data Sheet (MSDS)** is a written document that outlines health and safety information for a hazardous chemical. An MSDS is prepared in accordance with requirements of OSHA 29 CFR 1910.1200 Hazard Communication.
- 10.2 Major Spill** -- A Major Spill is defined by the EHS Industrial Hygiene Program as one in which ANY of the following conditions apply: the responsible party is unknown (it's an "orphan" spill); the material spilled is unknown; the material spilled is highly toxic; a large (or undetermined) quantity was spilled; a significant fire hazard may be present; the material has the potential to reach the environment (e.g., via a floor drain); the spill is in a common area (e.g., hallway) or other area accessible to the general public; advanced personnel protective equipment (more than gloves and a half-face respirator) is required to respond to the spill; or a responder is unsure whether the spill should be considered "Minor" or "Major".
- 10.2 Minor Spill** -- A Minor Spill is defined by the EHS Industrial Hygiene Program as one in which ALL of the following conditions are met: the responsible party is at the scene; the material spilled is known; the material spilled is not highly toxic; the quantity spilled is small; there is no fire hazard present; spill is completely contained inside a building; the material has little or no potential to reach the environment (e.g., via a floor drain); the spill is not in a common area (e.g., a hallway) or other area accessible to the general public; and advanced personnel protective equipment (i.e., more than gloves and a half-face respirator) is not needed to respond to the spill
- 10.3 Oxidizer** means a chemical other than a blasting agent or explosive, as defined in 1910.109(a) that initiates or promotes combustion in other materials, thereby causing fire either of itself or through the release of oxygen or other gases.
- 10.4 Unstable (reactive)** refers to a chemical which in the pure state, or as produced or transported will vigorously polymerize, decompose, condense, or will become self-reactive under conditions of shock, pressure, or temperature.
- 10.5 Use** refers to packaging, handling, reacting, emitting, generating as a byproduct, or transferring.

Appendix A

780 CMR: State Board of Building Regulations and Standards

The Massachusetts State Building Code

Table 307.8(1) (Reproduced in part)
Exempt Amounts of Hazardous Materials, Liquids and Chemicals Presenting a Physical Hazard
Maximum Quantities per Control Area^{a,k}

Material	Class	Use Groups	Storage ^b			Closed Systems ^b			Open Systems ^b	
			Solid pounds (cubic feet)	Liquid gallons (pounds)	Gas (cubic feet)	Solid pounds (cubic feet)	Liquid gallons (pounds)	Gas cubic feet	Solid pounds (cubic feet)	Liquid gallons (pounds)
Oxidizer	4	H-1	1 ^{e,h}	(1) ^{e,h}	NA	1/4 ^h	(1/4) ^h	NA	1/4 ^h	(1/4) ^h
	3	H-2	10 ^{d,e}	(10) ^{d,e}		2 ^d	(2) ^d		2 ^d	(2) ^d
	2	H-3	250 ^{d,e}	(250) ^{d,e}		250 ^d	(250) ^d		50 ^d	(50) ^d
	1	H-3	1,000 ^{d,e}	(1,000) ^{d,e}		1,000 ^d	(1,000) ^d		200 ^d	(200) ^d

a. For use of control areas, see 780 CMR 417.2

b. The aggregate quantity in utilization and storage shall not exceed the quantity listed for storage.

d. Maximum quantities shall be increased 100% in buildings equipped throughout with an automatic sprinkler system in accordance with 780 CMR 906.2.1. Where note e. also applies, the increase for both notes shall be applied accumulatively.

e. Quantities shall be increased 100% when stored in approved cabinets, gas cabinets, fume hoods, exhausted enclosures, or safety cans as specified in the fire prevention code listed in Appendix A. Where note d. also applies, the increase for both notes shall be applied accumulatively.

h. Permitted only in buildings equipped throughout with an automatic sprinkler system in accordance with 780 CMR 906.2.1.

k. Quantities in parenthesis indicate quantity units in parenthesis at the head of each column. 1 cubic feet = 0.028 m³; 1 pound = 0.45 kg; 1 gallon = .00379 m³