Perchloric Acid Fume Hood Mechanical Systems

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Forward

Perchloric acid ($HClO_4$) is a clear colorless liquid, useful in the laboratory as a strong oxidant. Desirable in chemical processing, perchloric acid offers the properties of a mineral acid, without introducing ions like chloride, nitrate, or sulfate. This corrosive chemical carries hazards typical of most acids; it is harmful if swallowed, causing digestive and respiratory tract burns, and if exposed to the outside of your body can cause eye and skin burns. Additionally, perchloric acid is also explosively unstable under some conditions. For this reason, perchloric acid applications require special equipment for safe handling.

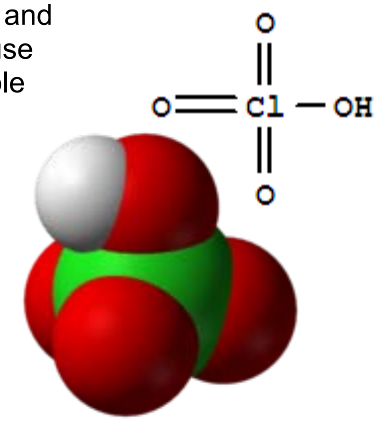

Figure 1. Perchloric Acid (HClO4) Molecule

Scope

The scope of this document will cover appropriate handling, proper ventilation equipment design, and equipment decontamination procedures.

References

The source material for this document comes from a variety of reputable resources listed below. The reader is encouraged to conduct his/her own thorough review of these documents prior to engaging in any work related to the design or use of a perchloric acid system.

- ✓ ACGIH Industrial Ventilation, *A Manual of Recommended Practice*, VS-35-03, 2-91
- ✓ NFPA 45-2011, Section 8.11 Perchloric Acid Hoods
- ✓ ANSI/AIHA Z9.5, 2003, 3.2.4 Perchloric Acid Laboratory Chemical Hoods
- ✓ CRC Handbook of Laboratory Safety, CRC Press, Boca Raton, FL, 1990.
- ✓ Michigan State University, EHS, *Safe Use of Perchloric Acid*, 3/21/2011

Please reference Material Safety Data Sheets (MSDS) for detailed composition, first aid measures, safe handling of an accidental release, and/or firefighting measures. Dismantling procedures can be found in a variety of documents developed by Oak Ridge National Laboratories and the University of Cincinnati, University Health Services, Environmental Health and Safety, Advisory No. 8.1: *Procedures for Dismantling Exhaust Ventilation System Suspected of Contamination with Perchlorates*, 4-08-1997.

Introduction

Perchloric acid is not explosive in solution, only extremely corrosive and harmful to breathe. This is justification for the use of a chemical fume hood. If the acid is evacuated in the same ventilation equipment that captures organics, the perchloric acid vapors saturate, or the salt residue of the perchloric acid is saturated by the organics, and a new molecular structure is formed that is highly unstable. This occurs when the perchloric acid vapor is allowed to condense in the ductwork and then evaporate, leaving behind a salt called perchlorate. Perchlorate crystals are explosive and can be detonated by heat, flame, friction, percussion or chemical reaction.

Though something as small as the vibration of the blower motor can cause a violent reaction, normally, no additional difficulties are encountered until it is time to dismantle the system. The fear here is that a mechanical contractor, unaware of the hazards lying within, may attempt to dismantle or service the mechanical system and in the process dislodge crystals resulting in a catastrophic situation.

Fortunately, perchloric acid can be neutralized with water, and perchlorate salts will dissolve in water. Perchloric acid applications require special equipment that includes specific materials of construction, wash down systems, and dedicated mechanical systems.

There may be exceptions to the above, especially if the acid is dilute, in small amounts, infrequently used, and not heated. Extreme care should be taken to avoid spills in this situation. Examples of this type of work may include perchloric transfers or titrations. The issue should be addressed with the facility's Health and Safety Officer or Chemical Hygienist to determine if the specific application falls into this category.

The information provided in this document is believed to be accurate, and based on the resources cited above. Labconco Corporation accepts no responsibility or liability for accidents that may possibly occur in the use of perchloric acid.

Part I. System Design

1. Fume Hood

Fume hoods for perchloric acid use are designed to prevent the accumulation of perchlorate, and prevent perchloric acid from coming into contact with materials that may cause a vigorous reaction or explosion.

Materials of Construction

Materials of construction, coatings, and lubricants for all components inside the hood shall be impervious, inorganic, generally acid resistant, and non-reactive with perchloric acid to form corrosive, flammable, or explosive compounds or byproducts.

Figure 2: Grade 316 Stainless Steel Fume Hood for Perchloric Acid Use

One acceptable material of construction for the fume hood liner and baffles is 316 stainless steel. This molybdenum-bearing grade of stainless steel gives Grade 316 better overall corrosion resistant properties than Grade 304 stainless steel, particularly higher resistance to crevice corrosion and pitting. Grade 316 stainless steel fume hood liners typically include a stainless steel work surface, and the corners and seams are welded and ground to ANSI/AWS standards B1.11-2000, D1.6-99, and A5.12. Grade 316 grade stainless steel has excellent moisture and heat resistance, good resistance to a wide range of chemicals, but is not compatible with some inorganic acids and metal scavengers.

The other, and preferred, acceptable liner material for perchloric use is Type 1 Unplasticized Polyvinylchloride (PVC). PVC is one of the most chemical resistant materials used as a fume hood liner. Perchloric acid fume hoods with PVC liners will include a PVC work surface and heat welded corners, yielding a one-piece liner. Though using multiple chemicals inside a perchloric acid fume hood is generally not recommended, PVC is best if other acids are used such as hydrofluoric, nitric, sulfuric, and hydrochloric. PVC has a working temperature around 140° F, and will distort near 160° F. Though heat is rapidly diluted inside a fume hood, a heating apparatus that may elevate liner temperatures beyond this threshold requires a 316 stainless steel liner.

Figure 3: Type 1 Unplasticized PVC Fume Hood for Perchloric Acid Use

Perchloric acid is not compatible with sash material made of organic polycarbonate, which is commonly used in acid digestion hoods. Therefore, sash materials should be tempered or laminated safety glass.

Physical Construction

Regardless of whether stainless steel or PVC is selected for the material of construction, the hood interior shall be designed for easy decontamination. This includes one-piece construction, minimal interior penetrations, and baffles removable for inspection and periodic cleaning. For spill containment, the work area shall be watertight, and dished ½". Like all fume hoods, the interior shall be spark proof, and all service controls shall be remotely located.

An integral washdown system shall be located behind the baffles for decontamination of this area, and an integral trough located at the rear of the work surface, under the baffle, to capture the washdown water. For more detail on these washdown components, or the washdown system as a whole, please reference the Washdown Design section.

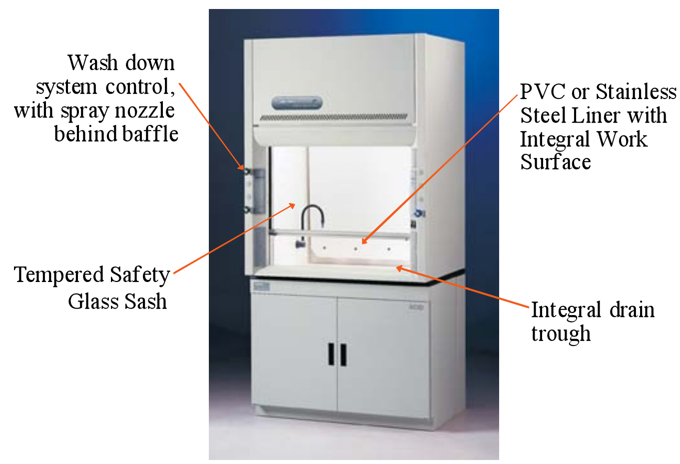

Figure 4: Perchloric Acid Fume Hood critical component detail

Airflow Requirements

Noted acceptable fume hood face velocities for perchloric acid hoods, range between 80 and 100 feet per minute (fpm). However, there is general agreement that the supply and exhaust air shall provide minimum challenge to the fume hood. Face velocity design criteria shall be 100 fpm for the worst case scenario, or 100 fpm with the sash fully open. This represents a reduced hood challenge, compared to lower face velocities, and will consequently increase duct velocity (fpm) and maximize dilution by elevating the volumetric rate (CFM).

All newly installed fume hoods should be field tested via the ASHRAE-110 test method and meet containment criteria listed in the current version of ANSI/AIHA Z9.5. Hoods should thereafter annually undergo the same test procedure, or anytime a change is made to the building mechanical system.

2. Mechanical System

Materials of Construction

Materials of construction, coatings, and lubricants for all components inside the mechanical system shall be impervious, inorganic, generally acid resistant, and shall not react with perchloric acid to form corrosive, flammable, or explosive compounds or byproducts. This limits the list of acceptable materials to Type 1 Unplasticized PVC and 316 stainless steel (See *Part I: System Design, 1. Fume Hoods, Materials of Construction,* for a brief analysis of these materials). Coated ducting is not recommended with the exception of porcelain on steel. However, because of the unpopularity and difficulties in assembly, porcelain on steel ducting is not covered here.

Grade 316 stainless steel ducting requires heliarc welding, which is expensive. Fluorinated hydrocarbon materials can be used as a sealant if heliarc welding is not possible. PVC ducting should be solvent bonded using the standard three part process (cleaner, primer, solvent cement adhesive). PVC ducting is often preferred after reviewing assembly cost considerations. However, PVC will not hold up as well in the event of an explosion or fire as would stainless steel, and some codes require that PVC ducting is contained within a two hour rated chase, or in an area with fire sprinkler protection.

Physical Construction

As perchloric acid hoods are typically only used for perchloric acid, please consider this in the building's architectural layout and mechanical system design. Each perchloric acid fume hood should have a dedicated exhaust run (no manifolding), dedicated blower, and be of a constant volume design.

The duct run shall be as short and straight as possible. Horizontal sections should be avoided, except for exhaust blower entry, and sloped downward toward the fume hood. The assembly should be smooth without corners or crevices, and constructed for easy visual inspection. Do not use flexible duct connections.

The exhaust stack shall terminate vertically, above the roof eddy zone. Stack height recommendations range between 10' (NFPA 45) and two times the height of the building (ACGIH). Weather caps, including zero pressure weather caps, should not be used.

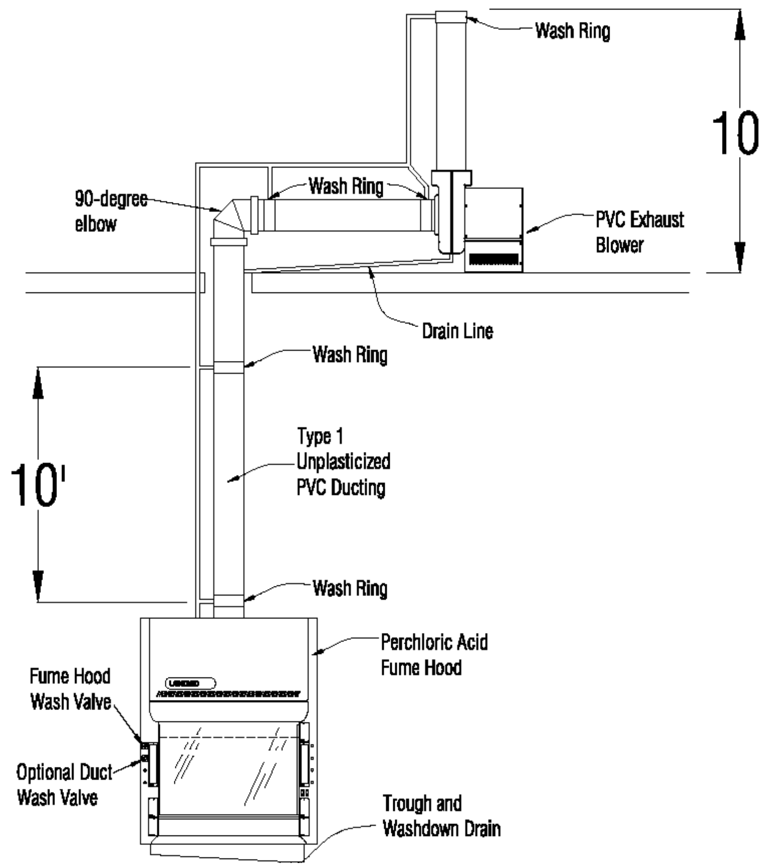

Figure 5: Mechanical and Plumbing systems detail

Airflow Requirements

Duct diameter shall be selected for minimum static pressure losses while providing the correct duct transport velocity between 1000 and 2500 fpm when the hoods are operated at 100 fpm face velocity with the sash fully open. Discharge velocity should be between 2000 and 3000 fpm per ANSI Z9.5.

3. Blower Specifics

The blower for this system should be located outside of the building, which typically means roof mounted. The blower should be specifically designed for use with perchloric acid. This includes appropriate materials of construction for the housing (See *Part I: System Design, 1. Fume Hoods, Materials of Construction,* for a brief analysis of these materials). The blower wheel, or impeller, should be PVC, polytetrafluoroethylene (PTFE/Teflon), polyvinylidene fluoride (PVDF), or other compatible material. Drive belts and motors should be outside of the airstream, and components within the airstream shall be non-sparking. The part of the blower within the air stream should be equipped with a lower drain to evacuate the washdown water (See *Part I: System Design, 4. Washdown Design, Drain, for more on this).*

Figure 6: Type 1 Unplasticized PVC Blower with drain fitting

4. Washdown Design

Supply within Ducting
The washdown system in the ductwork should be designed to thoroughly decontaminate the ductwork and blower. Washdown nozzles are placed in the ductwork by using a wash ring. The ring has a fitting for connecting fresh water, and locates a water nozzle at the center of the ring.

Figure 7: Wash Ring with water nozzle

Wash ring placement is critical to ensure proper cleaning. Because the nozzles are directional, the rings should be oriented so that the water is directed down, or back toward the fume hood. A washdown ring should be located at the termination of the exhaust stack, before and after each elbow, every 8-10 feet of vertical duct, every 2.5 to 5 feet of horizontal duct, before and after the blower, and just above the fume hood (See Figure 5 on Page 6 for more detail).

The water supply is often routed from the top nozzle down to even pressurization on systems with multiple wash rings. The design should lend itself to easy visual inspection. Additionally, the system should drain following each run to avoid rupturing supply lines due to freezing in cold climate. Heat tape and insulation can also help prevent pipe rupture.

Supply within Hood
Perchloric acid fume hoods should have an integral washdown system to decontaminate the area behind the baffles. The system can be either manually controlled at the face of the hood via a valve, or part of the electronically controlled washdown system. Manual systems often contain an auxiliary port, plugged at the factory, intended to provide water to the wash rings in the ductwork. A flow rate analysis should be conducted to ensure that the water volume at the available pressure is sufficient to supply the additional washdown nozzles. If there are more than 3-4 rings in the ducting, it is improbable that there will be enough water available to feed the top rings.

Control
For systems with multiple wash rings, the rings should be set up in stages. An assessment should be conducted to ensure that each stage has adequate flow rate and pressure to supply the allotted number of wash rings. Each stage will be run independently to avoid flooding the work surface (See *Part 3: Decontamination, 2. Developing the Washdown SOP, for more on this).* Control of the washdown system can be either manual or automatic, though automatic with manual override is preferred (CRC).

Drain
The washdown water will make its way, naturally via gravity, to the trough at the back of the fume hood. The blower should be drained, and collected water routed back to the nearest vertical section of ducting via plumbing and tee.

The trough at the back of the fume hood should be sized for a 2” nominal (2.375” OD) drain. The material of construction shall be the same as the fume hood liner, type 316 stainless steel or PVC. This should not be necked down to 1.5” as is commonly done, nor should a trap be installed. This can result in flooding the fume hood work surface. Connection to a dissimilar drain (i.e. glass) can be done with a heavy rubber reducing boot available at larger hardware or plumbing supply outlets.

Rinse water may be directed to a sanitary system where it will be quickly diluted, however, it is not uncommon to hold this water and measure pH and concentration before releasing to the sanitary system.

Part II. Safe Practices

1. Storage

Never store perchloric acid with organics. Store perchloric acid inside its original container with labeling intact. It should be segregated from all other chemicals and inside secondary containment such as glass or porcelain. It must not be stored near organic acids such as acetic acid, near bases, or near other organic or flammable material.

Perchloric acid is hygroscopic, meaning that through adsorption or absorption, the substance attracts and holds water molecules from the surrounding environment. Therefore, perchloric acid should always be properly contained when stored. f a bottle containing perchloric acid has turned dark, this is likely crystallization inside the bottle and poses a potential explosion hazard.

2. Proper Equipment Use

Perchloric acid should only be used in a hood and mechanical system designed for its use (See *Part I: System Design)*. Hoods and duct systems constructed other than as described should not be used for perchloric work.

Perchloric acid hoods should not be used for other purposes, especially if the acid is being boiled. Miscellaneous chemistry and radioactive material are not recommended for use in perchloric acid fume hoods. Organics should not be used in the same hood as perchloric acid. For this reason, all apparatus used inside the hood should have inorganic coatings and lubricants.

If capturing the perchloric acid is required, attempts should be considered to use a point of use wet collector. Capturing low volume, high concentrations of hazardous chemicals is easier than low concentration, high volumes.

Hot plates are commonly used in acid digestion applications with perchloric acid. The use of heat without exhaust running can damage the hood, especially PVC hoods. The exhaust system should remain running until all apparatus are completely cooled.

The hood should be certified by a qualified certification technician via the ASHRAE-110 test method before it is initially used. The hood should be recertified whenever it is relocated, serviced, or at least annually thereafter.

3. Handling

For complete handling details, please reference MSDS for your specific chemical combination. Anyone using perchloric acid should be trained in its proper handling. As with other acids, always add acid to water, not the reverse. Use the lowest amount of perchloric acid to fit procedural requirements. Follow basic fume hood safety practices when handling perchloric acid.

- Don’t store containers or supplies at the rear of the hood as it will negatively affect the airflow through the hood.
- Avoid placing your head inside the hood.
- Keep hands out of the hood as much as possible.
- Work as far back in the hood as possible. It is best to keep all chemicals and apparatus at least 6” inside the hood.
- Do not store chemicals inside a hood.
- Keep sash closed when unoccupied, and as low as possible when occupied.

Part III. Decontamination

1. Interior Preparations

After all work is complete, operate the hood for two to three minutes undisturbed. This will purge any remaining airborne contaminants. All objects should be surface decontaminated and removed from the fume hood. Cover open trays and containers before removing them from the hood. Properly store any remaining perchloric acid. Close the fume hood sash prior to initiating washdown procedure.

2. Developing the Washdown SOP

The fume removal system must be washed down sufficiently to ensure that there is no build up of perchloric acid salts. Commonly cited standards disagree in the duration of wash time, with figures ranging from 10 minutes to 30 minutes after each use. Therefore, regardless of whether your washdown system is manually operated with a valve (or series of valves) or an electronically controlled wash system with sequencing valves, a washdown process should be developed. This process will be specifically tailored to each perchloric acid mechanical system. If the system is manually operated, the washdown process should be developed into a standard operating procedure (SOP), while in the case of an electronically operated system, develop a computer program. Though the duration and frequency of your washdown cycle should be determined based on your specific conditions, there are some guidelines for development of the SOP or program.

The site safety officer (or other responsible party) will need to initially run the washdown system after every use. If the washdown system contains multiple stages, each stage should be run

independently for a set duration, starting with the upper most system, which typically includes the blower. Run the upper most washdown system with the blower on for approximately one minute, and then with the blower off for approximately 30 minutes to thoroughly clean this portion of the exhaust system. If additional stages exist, continue moving down the duct run, running each stage separately, with the blower off, for approximately 30 minutes each.

Care should be taken when operating any washdown system with the blower on. This will suspend water in the ductwork until the blower is off. Multiple runs of short duration are better than a long continuous run.

Following the duct washing, the internal hood spray nozzles should be activated to clean behind the baffles in the hood. Work areas in front of the baffle in the hood, including the sash glass, will need to be cleaned directly by the operator (see previous section). Following the initial washdown, take a swab and test the duct and hood for residue. The wash cycle times can be adjusted based on the results of the test. The system should be inspected regularly, and SOP or program adjusted as required.

3. Dismantling

Dismantling of a perchloric acid fume hood or mechanical system should be done by a professional in this field with appropriate liability insurance. This is especially true when dismantling a system that has not been utilized correctly, such as a non-perchloric acid hood without washdown capability. Please see the References section of this document for resources.