PWGSC
Mechanical and Maintenance Engineering
Real Property Branch
and the **National Laboratory Knowledge Network**

MD 15129 - 2006

Perchloric Acid Fume Hoods and Their Exhaust Systems

Guidelines for the use of perchloric acid fume hoods in PWGSC and other government of Canada facilities

Travaux publics et Services gouvernementaux Canada
Public Works and Government Services Canada
Canada

Public Works and Government Services Canada is pleased to present MD15129, *Guidelines for Perchloric Acid Fume Hoods and their Exhaust Systems*, dated March, 2006.	Travaux publics et Services gouvernementaux Canada a le plaisir de vous présenter la dernière édition des IM15129, *Lignes directrices pour les hottes à acide perchlorique et les systèmes d'évacuation connexes*, mars 2006.
Building, operating, and managing laboratory facilities require unique skills and knowledge to protect the health and safety of laboratory workers. One of the most distinctive pieces of equipment found in laboratories is the perchloric acid fume hood. While perchloric acid is useful for certain analytical procedures, it brings with it the potential for some dangerous repercussions due to the formation of perchlorates within the fume hood, ductwork, and/or exhaust fan. Perchlorates are extremely shock sensitive, and can explode with very little disturbance.	La fabrication, l'exploitation et l'entretien des installations de laboratoire exigent des compétences et des connaissances particulières pour protéger la santé et la sécurité des personnes qui y travaillent. L'une des pièces d'équipement les plus courantes dans un laboratoire, c'est la hotte à acide perchlorique. Malgré son utilité dans certains procédés analytiques, l'acide perchlorique peut avoir des effets dangereux, compte tenu de la formation des perchlorates dans la hotte, les conduits et les ventilateurs d'extraction. Les perchlorates sont extrêmement sensibles au choc, et la moindre agitation peut provoquer une explosion.
A previous version of this guideline was published in the late 80's. However, the following have led to the revision of this document: • There have been many recorded instances of fires and explosions associated with improper installations. • Advances in alternative materials for fume hoods, ductwork, and exhaust fans now provide options in the construction of these systems. • There is now better consensus regarding methods of testing for the presence of perchlorates in existing systems, particularly prior to the de-commissioning of a perchloric acid system.	Une première version des lignes directrices a été publiée à la fin des années 80, mais cette version a dû être révisée pour les raisons qui suivent : • de nombreux cas d'incendie et d'explosion ont été enregistrés en raison d'une mauvaise installation; • les progrès réalisés en matière de hottes, de conduits et de ventilateurs d'extraction permettent d'avoir un nouveau choix de matériaux pour la construction de ces systèmes; • il existe maintenant un meilleur consensus concernant les méthodes d'essai et de détection de la présence de perchlorates dans les systèmes existants, surtout avant le démantèlement d'un système de hotte à acide perchlorique.
The formation of PWGSC's National Laboratory Knowledge Network also resulted in a greater focus on sharing the laboratory resource material. This	La création du Réseau national des experts en laboratoire de TPSGC a également permis de mettre plus d'emphase sur le partage des ressources documentaires. Les nouvelles lignes

guideline is part of that effort, and will assist in providing consistency in the procuring, installing, maintaining, and decommissioning of perchloric acid fume hood systems.	directrices font partie de cet effort, et elles aideront à assurer la cohérence dans l'achat, l'installation, l'entretien et le démantèlement des systèmes de hotte à acide perchlorique.
This document is complementary to MD15128, *Minimum Guidelines for Laboratory Fume Hoods*, and requires that the fume hood performance tests contained there-in be applied to new and existing perchloric acid fume hoods, in order to assure effective containment.	Le présent document complète les IM15128, *Lignes directrices minimales pour les hottes de laboratoire*, et exige que les essais de performance, tels qu'ils sont décrits ci-après, soient appliqués aux systèmes de hotte à acide perchlorique nouveaux et existants afin d'assurer un confinement efficace.
The objective of MD15129 dovetails very appropriately with commissioning efforts on our laboratory projects. It is important that commissioning officers be fully familiar with the contents of this document, and prepared to oversee the collection of all the data and tests results required to properly install safely-operating perchloric acid fume hood systems.	L'objectif des IM15129 concorde parfaitement avec les activités de mise en service de nos projets de laboratoire. Il est important que les agents de mise en service se familiarisent avec les présentes lignes directrices, et qu'ils soient prêts à superviser la cueillette des données et les résultats des essais nécessaires à l'installation adéquate et à l'exploitation sécuritaire des systèmes de hotte à acide perchlorique.
We encourage you to use this guideline when perchloric acid systems are encountered or are being planned. Additional copies, as well as an electronic version, can be obtained from the Documentation Centre at: doc.centre@pwgsc.gc.ca.	Nous vous encourageons à utiliser ces lignes directrices lorsque vous rencontrez ou prévoyez de rencontrer des systèmes de hotte à acide perchlorique. Vous pouvez obtenir des exemplaires additionnels, ainsi qu'une version électronique, au Centre de documentation à : doc.centre@pwgsc.gc.ca.
For more information regarding MD15129, please contact: Edward Durand Telephone: (819) 956-2490 E-mail: edward.durand@pwgsc.gc.ca or Tim Lee Telephone: (780) 497-3967 E-mail: Tim.Lee@pwgsc.gc.ca	Pour plus de renseignements sur les IM15129, prière de communiquer avec : Edward Durand Téléphone : (819) 956-2490 Courriel : edward.durand@pwgsc.gc.ca ou Tim Lee Téléphone : (780) 497-3967 Courriel : Tim.Lee@pwgsc.gc.ca

Garnet Strong
A/Director General / Directeur général par intérim
Professional and Technical Program / Programme profesionnel et technique
Real Property Branch / Direction générale des biens immobiliers

MD 15129 - GUIDELINES FOR PERCHLORIC ACID FUME HOODS & THEIR EXHAUST SYTEMS

PREFACE

General

This document has been developed jointly by the Mechanical and Maintenance Engineering Group of Architectural and Engineering Resources Directorate (AER), Asset and Facilities Management Services (AFMS) and the PWGSC National Laboratory Knowledge Network.

Feedback

Corrections, recommendations, suggestions for modifications or additional information and instructions that will improve this document and motivate its use are invited. For this purpose the attached form entitled *"Request for change to these guidelines"* may be used and mailed or FAXED to the address shown. E-mail or other forms of electronic transmission may also be used for this purpose.

MD 15129 - GUIDELINES FOR PERCHLORIC ACID FUME HOODS & THEIR EXHAUST SYSTEMS

Request for changes to these Guidelines
Return this form to either Edward Durand or Patrick Plue

Revision no: _____ Date: _____

Edward Durand, National Manager Mechanical and Maintenance Engineering Professional and Technical Program Architecture and Engineering Resources Real Property Branch PWGSC Tel: (819) 956-2490 FAX: (819) 956-2720 E-mail: edward.durand@pwgsc.gc.ca	Patrick Plue, Mechanical Engineer Mechanical and Maintenance Engineering Professional and Technical Program Architecture and Engineering Resources Real Property Branch PWGSC Tel: (613) 759-6928 FAX: (613) 715-5140 E-mail: patrick.plue@pwgsc.gc.ca

Type of change suggested:

1. Correction of information
2. Deletion of information
3. Addition of information

Details of suggested changes: If necessary, make the suggested changes to a photocopy of the relevant page(s) of these Guidelines and attach to this sheet.

Page(s): _____ Chapter: _____ Section: ____

Details of suggested changes

(Use additional sheets if necessary)

Signature: _____ Date: _____

MD 15129 - GUIDELINES FOR PERCHLORIC ACID FUME HOODS & THEIR EXHAUST SYSTEMS

Table of Contents

MD 15129 - GUIDELINES for PERCHLORIC ACID FUME HOODS & THEIR EXHAUST SYSTEMS

Chapter 1 Glossary of terms

Assay work:
Analysis to determine the presence, absence, or quantity of one or more components. In this context, typically involves heating perchloric acid in a fume hood to temperatures approaching its boiling point with the objective of destroying organic matter.

Digestion process:
The process of breaking a substance down into simpler chemical compounds.

Induction blower:
A blower which injects air into a venturi section of ductwork in a manner which induces additional air from the upstream area of the duct.

Organic:
Of, relating to, or containing carbon and chiefly or ultimately of biological origin.

Organic solvents:
Capable of dissolving or dispersing carbon substances.

Oxidize:
To combine or cause to combine with oxygen, ***or***, to dehydrogenate.

Perchlorate:
A salt or ester of perchloric acid. May appear as a powdery, white substance.

Perchloric acid, $HClO_4$:
A fuming, corrosive, strong acid that is the highest oxygen acid of chlorine and a powerful oxidizing agent when heated.

Reducer:
Substance used to change another from a higher to a lower oxidation state, thus losing electrons.

Rinsate:
Solution resulting from a flushing of the ductwork, fan, and/or fume hood with water.

Scrubber:
In this context, a component which exposes the exhaust air stream to water spray intended to cleanse the air stream of perchloric acid vapours.

Venturi:
A narrowed, tapered, circular section, which causes an increase in air velocity, thus creating a decrease in pressure (that is, a suction) which can be used to induce additional airflow.

Wash down ring:
A component placed within a section of ductwork, that disperses water in an effort to rinse the internal duct surfaces.

Wash down:
In the context of this document, to rinse all surfaces that have been exposed to perchloric acid vapours (including fume hood, fan, and ductwork).

Chapter 2 General

2.1 Introduction and purpose

Perchloric acid is a unique chemical used in analytical laboratory work. Unfortunately, once evaporated and condensed it can form an unstable, dangerous product within a fume hood and its associated exhaust system. This document is intended to help clarify the means to mitigate those dangers, to provide a safe installation, and to provide a consistent approach to the use of perchloric acid fume hoods in PWGSC and other Government of Canada facilities.

2.2 Scope of these Guidelines

These guidelines describe only those fume hood and exhaust system requirements that are unique to perchloric acid work. In addition to the specifics discussed herein, the performance criteria for laboratory fume hoods, found in MD 15128 – *Minimum Guidelines for Laboratory Fume Hoods*, will apply to all new installations, and retrofitting of laboratories containing perchloric acid fume hoods. Note that existing and older installations may be incapable of meeting the recommended performance criteria. The Laboratory Director shall take appropriate assessment and corrective measures where perchloric acid fume hood performance is uncertain.

Where physical intervention on an existing perchloric acid fume hood and its exhaust system is required (for maintenance, de-commissioning, or other), strict procedures are required to ensure that fire and/or explosion are prevented. Chapter 5 of this document includes recommendations for safe performance of such work.

References

- MD 15128 – *Minimum Guidelines for Laboratory Fume Hoods*, Public Works and Government Services Canada, 2004
- `National Master Specification, NMS 15805 – *Perchloric Acid Fume Hood and Exhaust Systems – Stainless Steel*
- National Master Specification, NMS 15805 – *Perchloric Acid Fume Hood and Exhaust Systems -- Plastic*

Chapter 3 Perchloric Acid Fume Hood Installations

3.1 Characteristics of Perchloric Acid and its Derivatives

Perchloric acid ($HClO_4$) is an oily, colourless, odourless, corrosive, fuming liquid, with a boiling point of 203^{o}C (see MSDS in Appendix A). It is a strong mineral acid commonly available in 72% concentration. At normal temperatures it is a strong acid only, while at high temperatures (160^{o}C and above) it behaves as a powerful oxidizer, making it particularly useful for digesting organic materials.

In assay work and for other uses, perchloric acid is heated in the fume hood to temperatures approaching its boiling point. The vapours are drawn through the slots in the back baffle, and into the plenum and exhaust system. Unfortunately, a proportion of these vapours will condense on cooler duct surfaces, fan casing, and so on. The condensed perchloric acid, if not removed, will form perchlorates, with the potential for disastrous results when disturbed in the future. It is for this reason that every time a perchloric hood is used, the wash down system must be cycled to place the perchlorates back into solution, thus cleansing the entire network immediately after its use.

Perchlorates are unstable, explosive substances that have been the source of many extraordinary accidents in laboratory facilities using perchloric fume hood systems. It is notable that accidents that do occur are severe[1], and usually involve only small quantities of reactant. Reported incidents normally involve workers that are experienced (see Appendix A2).

3.2 Alternatives to the use of Perchloric Acid

Due to the expense of installing and operating a dedicated perchloric acid system, and due to the inherent danger associated with such activities it is advisable to fully explore alternate diagnostic methods which would preclude the use of perchloric acid. Several departments have successfully eliminated perchloric acid fume hood systems from their facilities, in favour of newer analytical procedures.

3.3 Fume Hoods for Perchloric Acid Work

It is recommended that a bypass type of fume hood be used for perchloric acid work. Once "balanced" for the design sash opening, it will continue to ventilate effectively, even when the sash is moved to a closed position.

A perchloric acid fume hood must be designated solely for perchloric acid work. For reasons indicated above, this work must not be performed in a fume hood previously used for other work, as organic deposits left behind may combine with perchloric acid to form explosive compounds.

Aside from the basic need to provide containment of harmful vapours, the two primary objectives in the installation of a perchloric acid fume hood system are to prevent accumulation of deposits by use of a wash down system, and to avoid corrosive action of perchloric acid on any of the components. To achieve these:

1. A spray bar with nozzles is located behind the rear baffle of the fume hood (Figure 1) in order to wash down that area **after each use**. It is important that there be no crevices or other areas where perchloric acid can accumulate, and the wash water must be allowed to drain freely. The rinsate (a very weak solution of perchloric acid) flows to the drain trough and is normally directed to sanitary sewer. If tests show the concentration of the rinsate to be a concern, the rinsate may be diverted to a holding tank for neutralization prior to release. Solutions of 5% sodium bicarbonate or sodium hydroxide have been traditionally used recommended for safely neutralizing dilute solutions of perchloric acid.
2. A short length of hose, with a trigger nozzle, is connected to the fume hood's water outlet to permit ready wash down of the fume hood's interior surfaces after each use.
3. An integral drain trough with 50mm drain outlet exists toward the rear of the work surface.
4. The interior liner and work surface are integral, continuous, coved, and with seamless welds. The work surface must be depressed by 12mm. Where heat is a concern, use 316 stainless steel. Otherwise a PVC, polypropylene or other compatible, non-metallic liner is a viable alternative (see Appendix A3 for compatible materials).
5. No interior access panels are permitted. Rather, all access to services must be from outside the fume hood, through an outer panel(s).
6. The rear baffle must be readily removable to inspect for chemical build-up and to aid in cleaning procedures if required. (NEVER remove the baffle unless the spray nozzles behind it have been activated for at least10 minutes immediately prior to removal.)
7. Laminated safety glass must be used in the sash.
8. Use only explosion proof light fixtures.
9. Signage must be prominently displayed on the hood, indicating "FOR PERCHLORIC ACID WORK ONLY".

Many fume hood manufacturers have specific literature available regarding the perchloric acid hoods that they produce.

Variable air volume (VAV) fume hoods are not appropriate for perchloric acid use, as decreased duct velocities due to sash adjustment could increase the potential for condensation within exhaust ductwork. Also, it is equally important to avoid the use of VAV control dampers or any other device within the ductwork as much as possible.

3.4 Ductwork for perchloric acid systems

The same considerations as for the fume hood apply here, except that the ductwork has greater potential for condensation and subsequent deposition of perchlorates. To minimize this potential, the designer shall provide for a target duct velocity of no less than 7.5m/s (1500fpm).

Higher duct velocities will result in a higher percentage of perchloric acid vapour being transported to the exhaust fan and out into the environment, but may result in high noise levels.

It is recommended that the ductwork be conspicuously stenciled or tagged at close intervals as a ready reminder to maintenance personnel who may be working in the vicinity.

These systems must not be manifolded with other fume hoods.

3.4.1 Materials:

Only non-organic material, able to withstand the corrosive properties of perchloric acid is acceptable. Typically welded 316 stainless steel has been used, however the use of unplasticized polyvinyl chloride (PVC) is also acceptable, as long as the duct shafts containing PVC material have automatic fire sprinklers or are located within a 2-hour fire rated chase. (When in doubt, always seek approval from the Fire Commissioner/Authority regarding these installations.) Where required, use only neoprene gaskets to avoid reaction with perchloric acid vapours.

Note that PVC ductwork provides better noise attenuation that does stainless steel.

3.4.2 Installation:

Several basic rules shall be applied to construction of the ductwork:

1. Choose a direct, short route to the exhaust location.
2. Avoid horizontal ductwork, and minimize the number of elbows; if a horizontal section is unavoidable, slope it at 5% back to the fume hood so as not to trap condensate or rinsate.
3. Winterize the ductwork and wash down system on exterior locations (insulate above roof). Note that the solenoid valve for the water line servicing wash down rings above the roofline must have a drain-free feature when not activated, as shown in Figure 2. This will empty all vulnerable lines.
4. Provide a minimum 3m stack height above roof parapet walls, with 15 m/s discharge velocity (3000 fpm) for dispersion effectiveness. Do not install a rain cap, as stack exhaust velocities of greater than 13 m/s will prevent rain from entering the stack[2].
5. For quality assurance purposes, specify a mock-up to establish required quality of fully smooth welded connections (regardless of material types).
6. Never use flex connections or any other connections which would permit the accumulation of perchlorates.

3.4.3 Scrubber:

A scrubber on the exhaust is normally not required. However, if called for in the project brief:

1. Locate it as close as possible to the fume hood, in order to minimize the length of heavily contaminated ductwork.
2. The need for wash down rings located downstream of the scrubber is not eliminated by virtue of having a scrubber present.
3. See MD 15128 for further details on scrubber installations for fume hoods.

3.4.4 Wash down:

Given the importance of this element in the safe use and operation of a perchloric acid fume hood system, it is critical that it functions properly.

Consider that, once sprayed on the interior of a vertical duct, there is the tendency of the wash water to form rivulets, thus not washing all surface areas within the ductwork. For this reason, wash down devices (such as PVC conical spray nozzles, wash down rings) should be located at maximum 2m intervals up the ductwork, and at every elbow, and must be operated under sufficient water pressure to properly establish the spray pattern.

Other considerations:

1. If using PVC solid cone nozzles, mount them at the center axis of the exhaust ductwork.
2. Provide separate valves for the fume hood and for each wash down device in order to ascertain that each is functioning (to facilitate the monthly inspection).
3. A manual switch must initiate the wash down cycle, after which a timer is to control solenoid operation and length of wash down.

3.5 Exhaust fans for perchloric acid systems

It is recommended that the fan be conspicuously stenciled or tagged as a ready reminder of its unique function to maintenance personnel who may be working in the vicinity.

3.5.1 Fan Type:

An induction exhaust system (often called an “air injector” or “venturi” fan) is **preferred** for perchloric acid fume hoods in that the motor and blower are not within the exhaust air stream (see Figure 2). It is only the venturi section that encounters the perchloric acid vapours, and thus the complication of effectively washing down a fan is avoided. The price that is paid for this feature is that such systems have lower air moving efficiency, thus increasing horsepower and operating costs. Also note that venturi fan systems can be very noisy, and with very little sound attenuation present this noise is readily transmitted down to the fume hood location.

Plastic centrifugal blowers (unplasticized type 1 PVC) are also being used successfully for exhausting perchloric acid hoods, since they are inert to the effects of perchloric acid vapours. They are also comparatively quiet. However, as with ductwork, methods for washing down such fans are critical in the design of these systems. Drains must also be provided for these fans. Experience has shown that centrifugal fans are relatively difficult to wash down effectively.

A third fan option is a roof-mounted, high plume, entrainment style of fan. However, these type of fans should be selected with care. Such direct drive fans require provision of a variable frequency drive for the motor in order to achieve the exact airflow which will produces the proper fume hood face velocity. These fan installations have also been known to present noise problems.

Other fan requirements, as noted in NFPA 45[3]:

1. Under no circumstance shall a fan **motor** be located within the ductwork.
2. Drive belts must be conductive, and not within ductwork.
3. Dedicated fans are required, rather than manifolded systems.

Also note that fans located such that their bearings' lubricant may come in contact with perchloric acid vapours should **only be lubricated with fluorocarbon-type grease**.

Two-speed fan installations are acceptable, if monitors, alarms, and commissioning procedures are the same as those described in MD 15128. The low speed condition is not to be used during active procedures, but rather is intended to address those occasions when no work is being performed in the fume hood.

3.5.2 Fan Installation:

1. For ease of maintenance, an induction-type fan can be safely located in the penthouse without fear of leakage (as would be the case for a typical centrifugal blower). However, it should be noted that induction systems require the injector fan to draw in about 5 times the amount of air that is being exhausted from the fume hood. If the fan is located in the penthouse, the induction air is costly, tempered air. In some cases this may be useful in ventilating the mechanical penthouse. Locating an induction system on the penthouse roof should be considered only as a last option because during extreme cold temperatures the stack will be at sub-freezing temperatures, and thus the water line to the upper-most wash down ring would have to be either heat-traced or temporarily disabled for those periods. This concern does not apply to centrifugal fans located on the roof, as all air moving up the stack is warm laboratory air only.

2. Locate the fan to provide the shortest direct route, with minimal elbows and horizontal ducts. (Ideally a laboratory selected to contain a perchloric acid fume hood would be located on an upper floor of the building, if possible.) Again, the penalty in doing so is the potential for fan noise being transmitted back to the laboratory.

3.6 Commissioning perchloric acid systems

It is essential to verify that the installation is correct, that proper materials (including fasteners, gaskets, lubricants, etc.) have been used, and that the wash down capability is fully functional. See Appendix C for a commissioning check sheet specifically aimed at perchloric acid fume hood installations. These are in addition to the commissioning tasks required for the installation of a typical laboratory fume hood (see MD 15128 – *Minimum Guidelines for Laboratory Fume Hoods*).

It has been recommended in some quarters that a commissioning task to establish the appropriate length of wash down cycle is worthwhile, as opposed to arbitrarily selecting a timeframe of, for instance, 5 minutes. This can be done empirically following an actual representative perchloric

acid activity of significant duration, by taking samples of the rinsate at 1-minute intervals, and having the samples analized for the presence of perchlorates[4]. Using the time frame required to achieve "clean" rinsate, and increasing this figure by 50% or more will provide a conservative estimate of the length of wash down cycle required after every use of the fume hood. *Caution: for abnormally lengthy or unusually intensive perchloric acid activities, the washdown cycle should be increased accordingly.*

3.7 Operating Guidelines

When in use, a perchloric acid fume hood must be operated with the sash at its design opening (as limited by the sash stop, and as indicated by the annual verification sticker).

Prior to each use, ensure that the fume hood monitor is functioning, and is not in an "alarm" condition.

With the exhaust fan continuing to operate, initiate the wash down cycle immediately after each use, before perchlorates are allowed to form.

Monthly inspection/testing of the wash down elements (water supply, valves, wash down rings) must be performed. This exercise is intended to confirm that each individual wash down ring or nozzle is functioning.

When maintenance is required, it must only be performed with the same diligence and safety precautions as described in Chapter 5 – Decommissioning Perchloric Acid Systems.

Annual performance testing of a perchloric acid fume hood is required, as noted in Chapter 4 – Perchloric Acid Fume Hood Performance and Testing Requirements.

By code, each laboratory is required to have a Chemical Safety Plan. Included in such a document are the handling and storage recommendations for all chemicals used (including perchloric acid), plus emergency procedures in case of a spill.

3.8 Fume Hood Log Book

A log book is required at each fume hood. It is to contain pertinent data, information, test results, protocol for perchloric acid fume hood use, a record of each use of the fume hood, and a sign-off area to confirm that the wash down system was cycled for the appropriate length of time. See Appendix B for a sample daily log and sign-off sheet to be located in the log book.

References

Due to the serious consequences of improperly designed and installed perchloric acid fume hood systems, a review of related material found in the following references is suggested for those dealing with such systems:

1. *Laboratory Ventilation Workbook*, 2nd Edition, D. Jeff Burton, 1994
2. *ASHRAE Laboratory Design Guide, RP-969*, 2001

3. NFPA 45 - *Standard on Fire Protection for Laboratories Using Chemicals*
4. *Perchloric Acid Hood Safety: Wash-down System Design and Testing*, R.J. Kelly, Lawrence Livermore National Laboratory, Livermore, CA

Also:

- CRC *Handbook of Laboratory Safety*, 5th Edition, 2000, A. Keith Furr, CRC Press, N.Y.

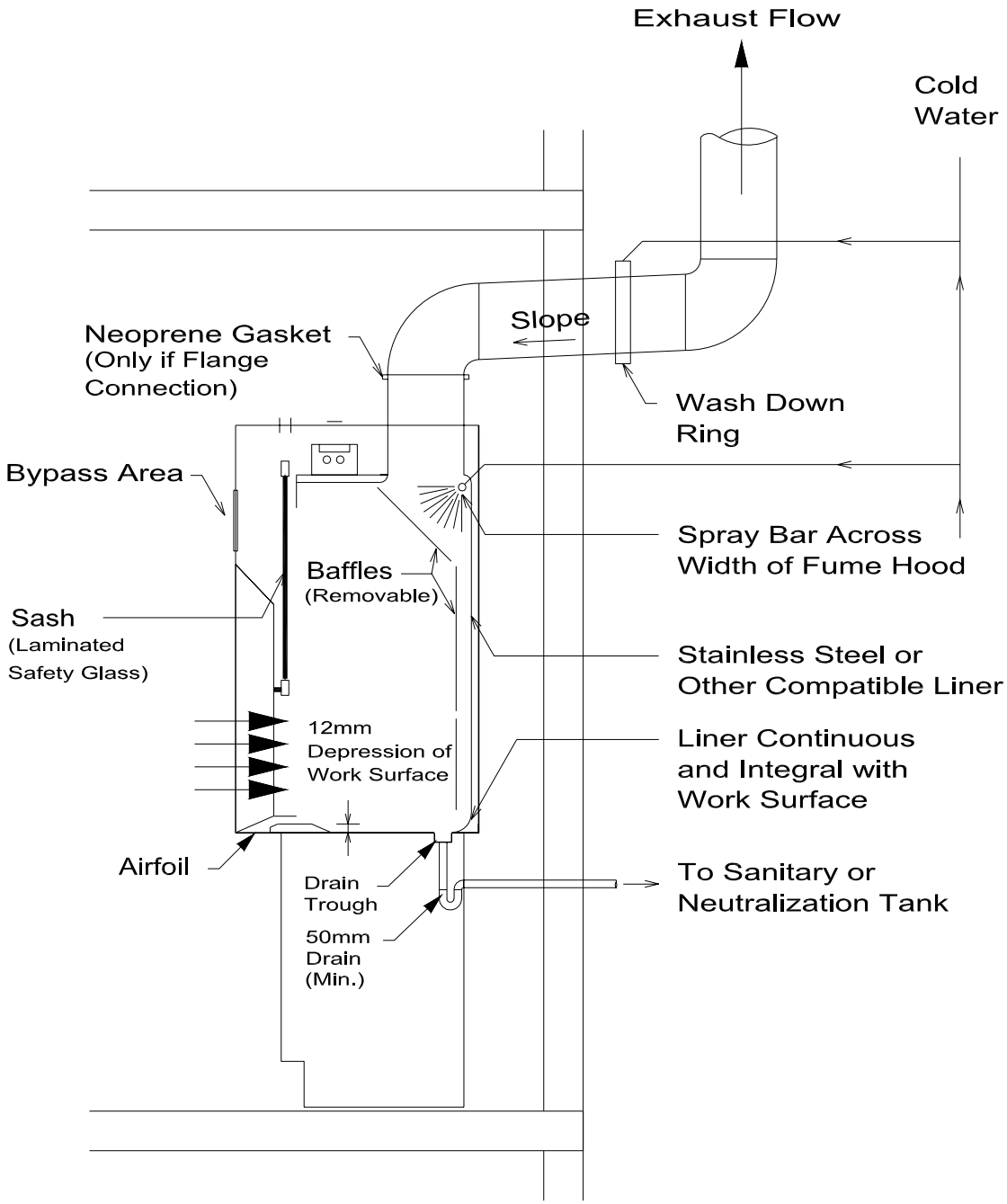
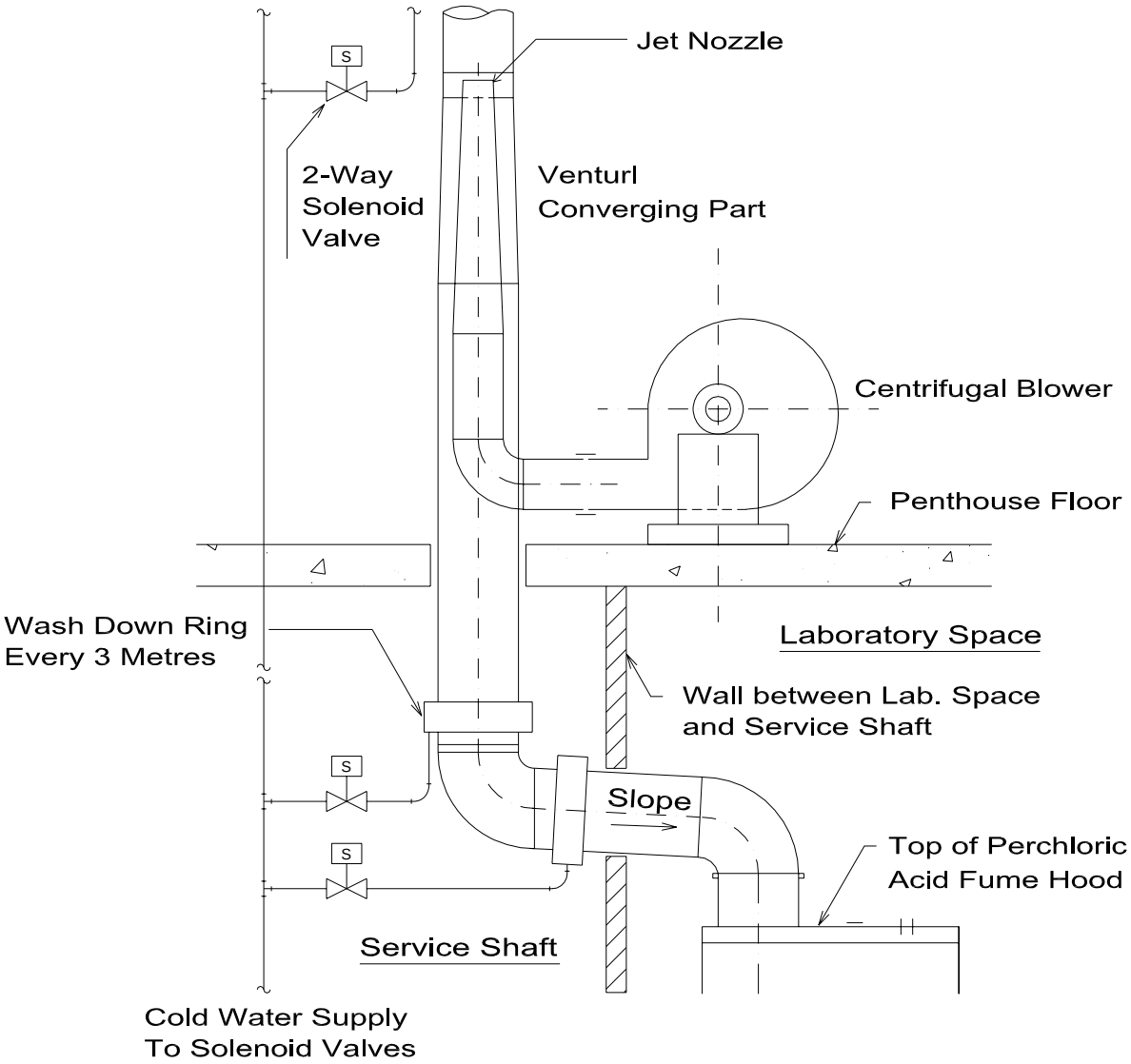


FIGURE 1: PERCHLORIC ACID FUME HOOD



Note: All Duct Joints to be Welded

FIGURE 2: DUCTWORK AND FAN

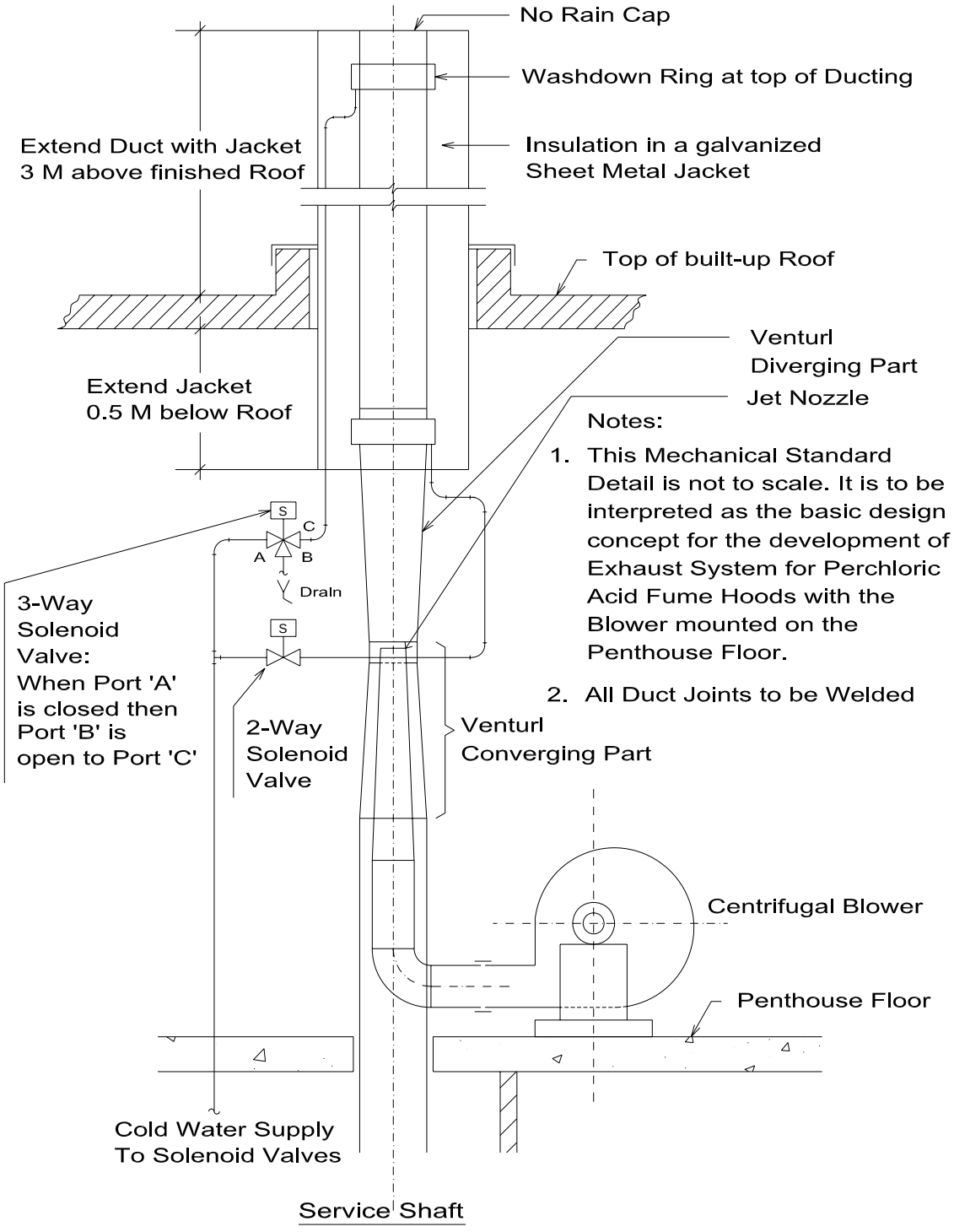


FIGURE 3: FAN AND EXHAUST STACK

Perchloric Acid Fume Hood Venturi Exhaust System
with an integral Washdown System

C.A.P.

Chapter 4 Perchloric Acid Fume Hood Performance and Testing Requirements

4.1 Fume hood performance

Fume hood performance criteria shall be as found in MD 15128 – *Minimum Guidelines for Laboratory Fume Hoods*. All tests shall meet the criteria established in the PWGSC Performance Criteria Table 1 of that document, with the exception of fume hood face velocity, which shall have a target velocity of 0.625 m/s (125 fpm)[1]

4.2 As Manufactured Tests

To be performed as required in MD 15128.

4.3 On Arrival Acceptance

As described in MD 15128, with the added verification of perchloric fume hood specific requirements. See Appendix C for Perchloric Acid System Commissioning Check Sheet.

4.4 As Installed Tests

As described in MD 15128.

4.5 As Used Tests

As described in MD 15128.

4.6 Existing Perchloric Acid Fume Hood Systems Tests

As described in MD 15128, plus a monthly verification of wash down system function.

References

1. ACGIH: *Industrial Ventilation: A Manual of Recommended Practice,* 24th Edition. Cincinnati, Ohio: American Conference of Governmental Industrial Hygienists, 2001

Also:

- ANSI/ASHRAE 110 – 1995: *Method of Testing Performance of Laboratory Fume Hoods*
- ANSI/AIHA Z9.5 – 2003: *American National Standard for Laboratory Ventilation*
- CAN/CSA Standard Z316.5: *Fume Hoods and Associated Exhaust Systems*

Chapter 5 De-commissioning Perchloric Acid Systems

5.1 An Approach to De-commissioning

A properly designed, constructed, and operated perchloric acid system is likely to have minimal perchlorates present in any of the components. However, it is important to note that in some laboratories, perchloric acid fume hoods may have been in use even when certain of the components of the system were not functioning properly, or proper wash down was not happening. If this is the case, shock sensitive perchlorates may be present.

Many accidents have taken place during maintenance or removal of perchloric acid systems. Although the stringency required in preventing such occurrences might seem unduly rigorous, the uncertainty of the degree of danger requires that precautions be observed. When in doubt, one must assume that the fume hood, ductwork, and exhaust fan contain unstable, explosive residues.

Phillips et al[1] and Bader et al[2] recommended a sequence of 1) wetting the entire system, 2) testing for perchlorates, 3) removal of the system (while continuing to wet), 4) de-contaminating the components, and 5) final testing to confirm that the materials are "clean". The following sections are based on the extensive experience reflected by their and other's[4,5,7] efforts.

Expert assistance and guidance should be sought and fully involved in de-commissioning of perchloric acid systems.

5.2 Procedures – Maintenance and De-commissioning

Maintenance and de-commissioning procedures *must not be initiated until the perchloric acid fume hood and exhaust system have been tested for the presence of perchlorates.* Such testing can commence after all surfaces have been wetted.

Even when proper safety procedures have been implemented, the use of ballistic gear is recommended if aggressive manipulation of the system is conducted. The gear should consist of fire-retardant coveralls, ballistic vest/faceshield/helmet, and personal protective equipment such as gloves and shoe covers.

Refer to Appendix B for further information regarding the use and maintenance of perchloric acid fume hoods.

5.2.1 Wetting of Surfaces

A well designed, properly constructed/commissioned, consistently used washdown system has the ability to maintain a perchloric acid fume hood and its ductwork in a safe condition. If such a system does not exist or if there is any doubt regarding its effectiveness, then a supplemental method of wetting internal surfaces must be provided.

The primary reason for initial wetting of the system is for safety rather than for decontamination. (Note that extensive wetting or steaming is not necessarily successful in removing all perchlorates.) Optional wetting methods include:

1. As noted in the Appendix of NFPA 45[3], steaming the system for 24 hours in order to condense moisture in every possible location, or,
2. Introduce a fine mist of water within the fume hood while the exhaust fan is running. The high humidity air stream will wet all surfaces in the system. Such wetting should be continued for a period of at least 12 hours, or,
3. Any other wetting method which will assure prolonged water contact with all internal surfaces.

For all of these options, if the ductwork is welded (rather than flanged and gasketted), and if the system is in relatively sound condition, the persistent washing down and testing/re-testing of the rinsate is thought to be a judicious approach[4] in rendering the system less hazardous to work on. (If tests after 12 hours show perchlorates in the final wash water, the misting should be continued for another 12 hours or until the test is negative.)

For older, deteriorated systems (possibly with flanged duct connections) which are to be removed, initial wetting is also required, but there should be no expectation that persistent washing will render the system safe. In this instance, after testing for perchlorates, the system is continuously wetted while it is removed piece-by-piece, submerged in a de-contamination water bath, and held there until all perchlorates have been dissolved. Use only clean cold water, without detergents or other chemicals for wetting and washing procedures.

5.2.2 Testing for perchlorates

Tests shall be conducted on the fume hood and exhaust system for explosive perchlorates prior to any inspection, cleaning, maintenance, demolition or other physical intervention. Only competent laboratory personnel or laboratory testing companies shall perform these tests.

There are several test methods that have been used to establish the presence or absence of perchlorates. Two of these require that a water mist is sprayed into the hood, fan, and ductwork, and the rinsate collected:

1. Methylene blue[3]: – the rinsate is introduced into a 0.3% methylene blue solution (25ml of rinsate per several drops of indicator solution). If perchlorates are present, a violet precipitate will be formed. Proceed to Ion Chromatography to analize subsequent samples. Note: False positives and false negatives have occasionally been reported using this method[1], and are thought to be related to the concentration of perchlorates falling outside the optimum range for this test. Use this test with caution, and only under the supervision of an individual who is expert in analizing the results. It may be used as an indicator, but its results should not be used as an absolute determinant, particularly for decommissioning work.

2. Ion chromatography[5]: -- the rinsate is collected then sent to a lab for analysis by ion chromatography to determine the concentration of perchlorates. This test is

considered the ultimate, definitive test for concentration of perchlorates, but it involves delays associated with lab testing.

Two other tests require that the surface(s), joints, etc. to be tested are accessible:

3. Ion Selective Electrodes/Swab test: -- swab potentially contaminated surfaces with damp gauze, and place these pads into water. A perchloric acid standard curve is prepared from a stock solution, and a specific ion (perchlorate) electrode is used to compare the swab unknowns against the concentrations in the standard curve. This test is the preferred, most practical, field test.

4. Diphenylamine[6]: – dissolve one gram of diphenylamine in 10ml of 1-to-1 (18 normal) sulfuric acid to form a diphenylamine sulfate solution. Use a medicine dropper to apply this solution to the test surface. The liquid turns black upon contact with perchlorate. (The solution also reacts with nitrates, but turns blue in that instance.)

The results of these tests will act as a guide to the anticipated extent of perchlorate contamination of the system. With this, informed decisions can be made throughout the de-commissioning process. For instance, heavily contaminated systems will require a thorough work plan and extreme caution in all activities during the de-commissioning process.

5.2.3 Intervention and Dismantling Precautions

The following procedures are recommended:

1. Provide training to the participants by a qualified individual. Instruction will alert workers to the danger, indicate methods of mitigation, and describe activities, equipment, protective gear, and procedures to be followed.
2. Insist on a planning exercise that clearly delineates the steps required, roles and responsibilities specific to the task at hand.
3. Perform maintenance or de-commissioning on weekends or silent hours only, when the laboratory facility is empty of personnel.
4. Provide suitable isolation, barricades, and protective clothing for personnel.
5. Avoid friction between components, heating, sparks or shock (impact) from any source. Even the simple act of loosening nuts and bolts has the potential for explosion.
6. Rather than dismantling joints, cut into ductwork away from joints, elbows, or any other area where higher concentrations of perchlorates would be expected.
7. Prior to any dismantling, extensive wetting (see section 5.2.1) and rinsate testing to confirm the absence of perchlorates in the rinse water is required. Dismantling activities should immediately follow such wetting of all system surfaces.
8. Using non-sparking tools (shears), dismantle fan and manageable lengths of ductwork. Invasive activities such as cutting or drilling should be done under a continuous flow of water.
9. Wash all disassembled parts.

10. Only when tests confirm that no perchlorate residue exists in the rinsate from the disassembled parts, may they be sent to landfill or metal recycling. The objective is to dispose of all ductwork as a non-hazardous waste.
11. Although the rinsate will normally not have an acidity level of concern, the use of small quantities of bicarbonate may be used as a precautionary neutralizing procedure.

5.2.4 Liability

When these procedures are to be carried out by an outside contractor, ensure that he is carrying adequate general liability insurance. It is also advantageous to select contractors based on proof of previous experience in de-commissioning perchloric acid systems.

References and Recommended Reading

The following references from this chapter provide invaluable experience and insight into decommissioning activities that have been proven **safe** over time.

1. Returning Perchlorate-Contaminated Fume Hood Systems to Service, Part I: Survey, Sampling, and Analysis, Phillips et al., *Applied Occupational and Environmental Hygiene*, 9(7):503-509, July, 1994
2. Returning Perchlorate-Contaminated Fume Hood Systems to Service, Part II: Disassembly, Decontamination, Disposal, and Analytical Procedures", Bader et al, *Applied Occupational and Environmental Hygiene*, Volume 14:369-375, 1999
3. NFPA 45 – *Standard on Fire Protection for Laboratories Using Chemicals*
4. Peter A. Breysse, Occupational Health Newsletter, Feb/Mar, 1966, University of Washington
5. www.safety.deas.harvard.edu/advise/PerchloricAcid.htm, Guidelines for Using Perchloric Acid, Harvard University
6. CRC Handbook of Laboratory Safety, 5th Edition, 2000, A. Keith Furr, CRC Press, N.Y.
7. Disassembling a Perchlorate-Contaminated Ventilation System, R. J. Kelly, American Chemical Society – *Chemical Health and Safety Journal*, May/June, 2000

APPENDIX A – Additional Perchloric Acid Information

APPENDIX A1 –

Safety (MSDS) data for concentrated perchloric acid, ca. 70%

General

Synonyms: hydronium perchlorate, dioxonium perchlorate
Molecular formula: $HClO_4$

Physical data

Appearance: colourless odourless liquid
Melting point: -17 ^{o}C
Boiling point: 203 ^{o}C
Vapour density: 3.5
Vapour pressure: 6.8 mm Hg at 25 ^{o}C
Specific gravity: 1.664
Flash point: >230 ^{o}C

Stability

Stable. Avoid heat. **May form explosive peroxides. Incompatible with a wide variety of substances, including organic materials, alcohols, amines, strong acids, strong bases, acid anhydrides, finely powdered metals, strong reducing agents. Contact with wood, paper and other celullose products may lead to explosion. This material must only be used after a full MSDS datasheet has been consulted and a COSHH assessment made.**

Toxicology

Corrosive - causes severe burns. Very harmful by inhalation, ingestion and through skin contact.

Personal protection

Safety glasses, chemical resistant (nitrile) gloves. Face shield for all but well-tried and predictable procedures.

APPENDIX A2 - Incident Reports (from the Chemical Rubber Company *Handbook of Laboratory Safety 4th Edition*, 1995 by CRC Press)

1. An explosion took place in the exhaust duct from a lab hood in which a perchloric acid solution was being fumed on a gas plate. The explosion blew out the windows, lifted the roof, and caused considerable damage to lab equipment. The hood had also been used for general chemical analysis. It was concluded that the explosion originated in deposits of perchlorates and organic materials in the duct.
2. An explosion occurred in the ductwork serving a hood reserved for perchloric acid. A lab technician had been drying alcohol over a Bunsen burner in the hood. The explosion bent the ductwork near the fan, separated the duct from the hood, and blew out several windows.
3. A bottle of perchloric acid was accidentally dropped on the floor. Sawdust was used in the cleanup and deposited in a metal waste can. Several hours later an explosion blew the can open and started a fire. The heat activated the sprinkler system, which put out the fire.
4. The stone bench of a lab fume hood was patched with glycerin cement. Several years later, during remodeling, a workman struck the stone with a chisel. An explosion occurred. The hood had been used for digestion with perchloric acid.
5. A conventional hood used for distillation and ashing of organic materials also used perchloric acid for digestion. During a routine ashing procedure, hot gases went up the duct, setting off a series of explosions. The duct was torn apart at several locations.
6. During routine maintenance involving the dismantling of a fan on a perchloric acid exhaust system, an explosion occurred following a blow with a chisel on some part of the fan housing. The explosion was heard four miles away. Two employees standing nearby were injured. The man holding the chisel was killed after the chisel entered his left nostril and was embedded in his brain.

APPENDIX A3 - Substance Compatibility

Given the violent reactions of perchloric acid with many organic compounds, particular diligence is important in eliminating any contact not otherwise required of the experimental process. For instance, the potential for perchloric acid to contact wood or paper must be minimized. Avoid the use of wood lab benching. Instead, provide epoxy bench tops, and shelves and cabinets of epoxy-painted steel. The following table provides an indication of some materials which are or are not compatible with perchloric acid.

Compatible	Incompatible
Elastomers	
Gum rubber (each batch must be tested to determine compatibility) Vitons (slight swelling only)	**many "manufactured" rubbers**
Metals and Alloys:	
Tantalum Titanium (chemically pure grade) Zirconium Niobium Hastelloy C	Copper Copper alloys (brass, bronze) Aluminum (dissolves at room temperature) High nickel alloys (dissolves)
Plastics	
Polyvinyl chloride Polyethylene Polypropylene Teflona Kel-F Vinylidine fluoride Saran Epoxies	Polyamide (nylon) Modacrylic ester, Dynel acrylonitrile Polyester (dacron) Bakelite Lucite Micarta Cellulose-based lacquers Fibreglass
Others	
Glass Glass-lined steel Alumina Fluorolube	Cotton Wool Wood Glycerin-lead oxide (letharge)

END OF APPENDIX A

APPENDIX B USE AND MAINTENANCE OF PERCHLORIC ACID FUME HOODS

Proper operation of perchloric acid fume hoods --- users

Proper operation of perchloric acid fume hoods is just as important as proper design and installation.

Before any laboratory personnel are allowed to operate a perchloric hood they shall be required to receive training in its uses, limitations and safety features. These instructions may include written instructions, live demonstrations and videotapes prepared by manufacturers, experienced researchers, technical institutes, etc.

Before any new perchloric fume hood is used, a notice listing its uses and limitations shall be prominently displayed at each fume hood. The Laboratory Director should produce this notice.

Work involving the use of perchloric acid must always be carried out in fume hoods specifically designed and designated for perchloric acid. No other procedures shall be allowed in these hoods.

Although it is very difficult to be specific in a manual of this nature, proper use of the fume hood should always include at least the following procedures:

.1 Check fume hood monitor warning lights. Proceed only if green indicator light is illuminated.

.2 Keep working surface uncluttered. This will assist in containment and also reduce disruption of airflow patterns. Where possible, equipment used within the hood should be elevated at least 25 mm above the work surface.

.3 Do not block exhaust slots at the back of the hood.

.4 Do not use the fume hood as a storage facility. Shelves should not be installed inside laboratory fume hoods. Keep storage of perchloric acid in the fume hood to a minimum.

.5 Sash should be maintained at normal operating position or closed (if bypass is available). The *normal operating position* is the sash setting at which the fume hood verification tests take place, and is indicated by a sticker located on the fume hood side post.

.6 Apparatus and materials should be positioned towards the centre and at least 150 mm from the face of the fume hood so as to minimize disturbance of air flow into the fume hood through the sash opening.

.7 Clean-up and spill procedures must be set up to suit processes used and laboratory protocol.

.8 Fill-out and sign the fume hood log book to indicate that proper wash down has occurred following every perchloric acid procedure. A sample sign-off sheet, is as follows:

Daily Log --- Perchloric Acid Fume Hood Use

Fume Hood number ______ Room number ______

Date of use	Approximate Period of Active use	Wash down procedure initiated?	Apparent normal water flow?	Length of wash down? (minutes)	User Sign-off
	___a.m. to ___a.m. ___p.m. to ___p.m.	❑ Yes ❑ No time of day____	❑ Yes ❑ No		
	___a.m. to ___a.m. ___p.m. to ___p.m.	❑ Yes ❑ No time of day____	❑ Yes ❑ No		
	___a.m. to ___a.m. ___p.m. to ___p.m.	❑ Yes ❑ No time of day____	❑ Yes ❑ No		
	___a.m. to ___a.m. ___p.m. to ___p.m.	❑ Yes ❑ No time of day____	❑ Yes ❑ No		
	___a.m. to ___a.m. ___p.m. to ___p.m.	❑ Yes ❑ No time of day____	❑ Yes ❑ No		
	___a.m. to ___a.m. ___p.m. to ___p.m.	❑ Yes ❑ No time of day____	❑ Yes ❑ No		
	___a.m. to ___a.m. ___p.m. to ___p.m.	❑ Yes ❑ No time of day____	❑ Yes ❑ No		
	___a.m. to ___a.m. ___p.m. to ___p.m.	❑ Yes ❑ No time of day____	❑ Yes ❑ No		
	___a.m. to ___a.m. ___p.m. to ___p.m.	❑ Yes ❑ No time of day____	❑ Yes ❑ No		
	___a.m. to ___a.m. ___p.m. to ___p.m.	❑ Yes ❑ No time of day____	❑ Yes ❑ No		
	___a.m. to ___a.m. ___p.m. to ___p.m.	❑ Yes ❑ No time of day____	❑ Yes ❑ No		

Proper maintenance of perchloric acid fume hoods --- O & M personnel

1. Preventive maintenance programs:

Preventive maintenance programs for perchloric acid fume hoods should include, but not be limited to, the following:

.1 Lubricating fan bearings and adjusting fan belts.
.2 Checking sash operation and counterweight pulleys and cables for wear or deterioration.
.3 Checking that sash limit stops are still in place and operate properly.
.4 Checking the integrity of seals around lighting fixtures.
.5 Inspection of all exhaust ducts for leaks and for unauthorized connections.
.6 Checking that the fume hood is being used only for which it was designed.
.7 Checking all surfaces in contact with fumes for damage, abrasion and rough surfaces.
.8 Ensure that a fume hood log book remains available at each perchloric hood (for recording each use of the hood, indication that wash down has occurred, etc.)
.9 Fume hood alarm test.

2. Fully detailed operating and maintenance manuals:

These are essential and shall be SPECIFIC TO THE PROJECT. The Operating Manual and the Maintenance Manual form an integral part of the Building Management Manual.

3. Operating Instructions:

These shall be complete, concise and clear, and shall be located in plain view of the fume hood user as part of the logbook (see 1.8, above). This shall also include all warning notices and alarms.

4. Performance tests:

Performance tests, described in detail in Chapter 4 of MD15128 – *Minimum Guidelines for Laboratory Fume Hoods*, shall be performed at intervals shown therein. Target face velocity for perchloric acid hoods should be 0.625 m/s (125 fpm).

5. Wash Down System tests:

On a monthly basis, testing shall be done to confirm that ***all*** wash down rings, nozzles, and any other components associated with the wash down system are fully functional.

6. HVAC systems:

Maintenance programs shall include verification of HVAC and general exhaust systems including confirmation of pressure relationships.

7. Laboratory use:

The Laboratory Director shall organize regular reviews of programs within labs, and operation of equipment. He shall also set in place procedures for reporting and correcting

defective equipment, and enabling improvements in operating and maintenance procedures.

END OF APPENDIX B

APPENDIX C COMPONENT CHECK SHEET – PERCHLORIC ACID SYSTEM

Room Number:		**Hood Number:**	
	Y/N		**Y/N**
Perchloric Fume Hood			
Stainless steel material		Integral work surface and liner	
Non-plasticized PVC material			
No interior access panels		All welds smooth	
Fasteners		Work surface recessed 12 mm	
Removable baffle		Integral drain trough	
Washdown nozzles behind baffle		50 mm drain outlet	
Washdown valve		Laminated safety glass	
Short length of hose with trigger nozzle for washing interior of fume hood		Sign indicating “WARNING: Perchloric acid work done in this hood”	
Perchloric System Ductwork			
Stainless steel material		Horizontal sections sloped to fume hood	
Non-plasticized PVC material			
Washdown device – max. 2m spacing		Non-reactive sealant/gasket material	
- at every elbow		Duct velocity approximately 7.5 m/s	
- separate valves for each device			
Perchloric Fume Hood Fan			
Make, model, type			
Motor not in airstream		Exhaust stack height >3m	
Drive belt conductive, and not in airstream		Outlet velocity >15m/s	
Non-reactive lubricant			

Completed by: _____ Date:_____

END OF APPENDIX C