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## SAFETY IN A CHEMISTRY LABORATORY

R. G. Elsbrock

RELEASED FOR ANNOUNCEMENT  
IN NUCLEAR SCIENCE ABSTRACTS

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### SAFETY IN A CHEMISTRY LABORATORY

by

R. G. Elsbrock

RELEASED FOR ANNOUNCEMENT  
IN NUCLEAR SCIENCE ABSTRACTS

Compliments of The Central New Mexico Section of the American Chemical Society in Cooperation with The United States Atomic Energy Commission, Sandia Corporation, State Board of Education, and Albuquerque Public Schools.

October 1966

## FOREWORD

"Chemicals in any form can be safely stored, handled, and used if their hazardous physical and chemical properties are fully understood and the necessary precautions, including the use of proper safeguards and personal protective equipment, are observed."

By Manufacturing Chemists Association, Inc.

## INTRODUCTION

Briefly, this manual attempts to alert personnel associated with and working in chemistry laboratories to safe practices in handling hazardous chemicals. Information contained in this publication has been gathered from sources believed to be reliable and to represent the best current opinion on the subject. No warranty, guarantee, or representation is made by the Sandia Corporation as to the correctness or sufficiency of any representation contained in this publication. It should not be concluded that all known and acceptable safety measures are contained herein. It is our hope that this work will assist in developing a sincere attitude toward safety-mindedness in the chemistry laboratory and elsewhere.

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## I. RULES FOR LABORATORY SAFETY

A person in charge of a chemistry laboratory should be safety-minded. Attitudes toward safety are reflected in the behavior of the workers because they are prone to emulate supervisors.

### 1.1 Accident Prevention

Accident prevention is based on certain common standards of education, training of personnel, and provision of safeguards against accidents. To meet these standards, the following suggestions are offered:

1. Have a staff composed of safety-minded individuals.
2. Maintain an educational program to promote laboratory safety.
3. Sustain a regular inspection program of all working areas and equipment.
4. Use corrective discipline for unsafe laboratory practices.
5. Provide adequate safeguards against physical exposure to hazardous chemicals and equipment.
6. Establish a policy of using personal protective equipment.
7. Have first-aid facilities and personnel competent to use them.

### 1.2 Review of Hazards

Before any experiment is attempted, a thorough review of possible dangers and hazards should be made. The following are a few suggestions.

1. Laboratory instructions should be read before starting an experiment.
2. If an experiment is not thoroughly understood, use small quantities of the materials involved for a trial. Wear goggles or a face shield.
3. Experiments in progress should never be left unattended.
4. Avoid breakage and possible injury by keeping all equipment as far back from the table edge as possible.



5. Before heating any chemical, find out if it is flammable. If the flash point is low, you should select a safe heating apparatus, e. g., heating mantle or a steam bath -- never a burner.
6. Require, during all hazardous operations, that one or more individuals be within communicating distance of the operator.
7. Use signs, bulletins, or posters to warn others of hazards in a particular operation.

## References

1. "Safety Manual for Chemical Laboratories," by G. N. Quam, Villanova University, Villanova, Pennsylvania, 1961.
2. "Guide for Safety in the Chemical Laboratory," by The General Safety Committee of the Manufacturing Chemists Association, Inc., Washington, D. C.

## II. CHEMICAL HAZARDS

The intent of this discussion is to acquaint the laboratory worker with hazards associated with some widely used laboratory chemicals. The user is advised to seek complete information in publications listed under "Bibliography."

The chemical hazards discussed here relate principally to fire, health, and explosions. They are therefore grouped under these three headings. The hazards of each chemical should be recognized and appropriate safe procedures put into practice to cope with them.

### 2.1 Fire Hazards

Three factors must be present in order for a fire to occur: a source of ignition (heat or a static spark), a fuel, and an oxidizing agent.

The proper combination of these three factors will cause a fire. The fire can be extinguished by breaking any side of the fire triangle: Remove the fuel or separate the fuel from the oxidizing agent, dilute the oxidizing agent, or cool the system.

Three classifications of fires are recognized:

Class A - Ordinary combustibles such as paper, wood, clothing, etc. These are best extinguished with water.

Class B - Flammable liquids (to be discussed in greater detail later).

Class C - Electrical apparatus. These are best extinguished with CO<sub>2</sub> or dry chemicals.

Evaluation of the fire hazard associated with any particular material is usually made in terms of the flash point, explosive limits, and specific gravity of the material.

The flash point is the lowest temperature at which a liquid gives off vapors near its surface in sufficient quantities to form an inflammable mixture with air. The lower the flash point, the more likely is the material to be ignited by a flame, spark, or hot surface.

Flash points are determined either by the "closed-cup" or "open-cup" method. In the closed-cup method, a sample of the material is heated under carefully prescribed conditions in a closed system charged with air. As the temperature is raised, an electric spark or a small flame is applied

to the vapors near the surface of the liquid, and the temperature at which the vapors flash is recorded. The open-cup determination is performed in a similar fashion, but in an open system, and some vapor escapes during the test. The vapor is somewhat cooler than in the closed-cup method, and the open-cup flash point is usually 2° to 50° higher than the closed-cup flash point.

The autoignition temperature of a gas or vapor is the "lowest temperature to which any part of a flammable vapor- or gas-air mixture must be raised...to cause combustion which will propagate itself." Propagation of a flame refers to the spread of a flame from layer to layer of the vapor-air mixture, independent of the source of ignition. The autoignition temperature of a solid is the lowest temperature at which a vapor, on coming in contact with the air, will ignite spontaneously.

Obviously, these two parameters alone do not serve as the sole index of the fire hazard of any material. Combustion is an extremely complicated chemical reaction, the rate of which will be dependent, not only on the temperature of the system and the concentration of the reactants, but also upon the state of subdivision of the reactants and the presence of foreign material which can accelerate or inhibit the reaction. The more finely a combustible solid is subdivided, the more rapid will be the reaction; the ignition temperature is lowered. Foreign material may lower the activation energies of the combustion reactants, accelerate the reaction, and thus lower the ignition temperature; or foreign material may prevent inter-laminar flame propagation or act as a free radical scavenger, inhibit the combustion reaction, and raise the ignition temperature appreciably. Therefore, tables of flash points and ignition temperatures should be taken as guides in determining the fire hazard of a particular material and not as quantitative indications of conditions of safety.

The explosive limits are the maximum and minimum concentrations of vapor in air beyond which flame propagation does not occur when the mixture is in contact with a source of ignition. These maximum and minimum concentrations, usually expressed as percent by volume of vapor in air, are known as the upper and lower explosive or flammable limits. Mixtures below minimum concentrations are too "lean" to explode; those above are too "rich." The region between the explosive limits is termed the explosive range and is usually a rather narrow zone at low concentrations. Explosive limits are not appreciably affected by normal changes in temperature and pressure, but at high temperatures the explosive range is usually at low concentrations, which implies that very small quantities of materials can give rise to very great fire hazards.

The specific gravity of a solid or liquid, expressed relative to water as 1.00, and the vapor density of a gas or vapor, expressed relative to air as 1.00, are also parameters important in evaluating the hazards of a particular material. Solids or liquids with a specific gravity less than 1.00, which are water immiscible or insoluble, will float on the surface of the water. Should these materials be introduced into the sanitary sewer system they may be carried great distances, filling the sewer with flammable or explosive vapors. Application of water to a fire involving these materials will spread the fire over a wide area as the burning liquid floats on the moving water. Materials with a vapor density greater than 1.00 are heavier than air and tend to flow along benches and flow along the floor.

The National Safety Council has set up three classifications of flammable liquids based on their flash points:

Class 1 - Liquids with a flash point at or below 20°F.

Class 2 - Liquids with a flash point above 20°F and below 70°F.

Class 3 - Liquids with a flash point above 70°F, but below 200°F.

There are several sources of ignition. Static electricity can be a bad problem in a dry atmosphere. It may be necessary to ground solvent containers and, in some circumstances, other precautions may be necessary, such as avoiding metal spatulas, etc. Spontaneous combustion, which starts with slow oxidation, generating heat and igniting the material, is quite possible with oil-soaked rags. Prevention consists of total exclusion of air by the use of tight metal refuse containers or good ventilation. Flash fires are spread by oil spills, oil waste deposits, or vapor-laden air.

## 2.2 Health Hazards

Chemicals such as the gases of hydrogen chloride, sulfur dioxide, and ammonia, are primarily irritants to the mucous membranes of the respiratory tract and to the eyes. In the respiratory tract, these gases cause an expansion of the blood vessels, secretion of plasma, and narrowing and blocking of the bronchioles. Irritated mucous membranes are particularly subject to infection. Further, if the very fine air sacs, the alveoli, in the lung fill with plasma, the useful volume of the lung is diminished and oxygen deficiency results.

Chemicals absorbed through the lungs will be distributed rapidly throughout the body. Many materials, in particular alcohols, esters, the

chlorinated hydrocarbons such as carbon tetrachloride, and both the aromatic and aliphatic hydrocarbons, act as narcotics and affect the nervous system and liver. Dizziness, headaches, and symptoms similar to those observed during alcoholic intoxication may appear. Damage to the respiratory center may result in death.

Not only may harmful chemicals be carried by the blood, but they may also directly react with the blood and act as blood poisoners. For example, aromatic amines and nitroso and nitro compounds appear to convert hemoglobin into the much more stable methemoglobin. Methemoglobin will not give up its oxygen to the tissues, and serious oxygen deficiency results. Further, the formation of methemoglobin is followed by destruction of the red blood corpuscles, leading to further oxygen deficiency.

Carbon monoxide is another material which interacts directly with the blood. Since carbon monoxide has about 300 times the affinity for hemoglobin as does oxygen, absorption of carbon monoxide into the blood forms carboxyhemoglobin, thus blocking the release of oxygen to the brain, and serious, often fatal, oxygen deficiency results.

Many commonly encountered chemicals present a health hazard.

Injurious chemicals may enter the body in three ways:

1. Through the lungs.

Toxic chemicals are most frequently encountered as vapors, gases, or dusts contaminating the laboratory air. Inhalation of these chemicals may irritate the mucous membranes of the respiratory system and open the way for infection. Further, the lungs are the main path for the absorption of gases into the blood, and hence poisonous chemicals, when inhaled, are readily able to interact with the blood and/or be dispersed rapidly by the bloodstream to all parts of the body. Inhalation of toxic chemicals can be prevented by the use of properly designed fume hoods and protective devices such as gas masks or respirators. Completely closed systems, under vacuum, may be necessary when handling very toxic gases.

2. Through the gastrointestinal tract.

Usually, a harmful material is introduced by ingestion as a result of carelessness or uncleanness. Eating and drinking in the laboratory is hazardous, particularly if laboratory beakers and bottles are used as cups or for storage of food. The all-too-common practice of pipetting by applying suction to the pipet by mouth has been responsible for numerous cases of poisoning.

### 3. Through the skin.

Although the skin is impermeable to most substances, some penetrate readily; all may be introduced through cuts or forced in by broken glassware. Some chemicals damage the skin and then are absorbed through the damaged area.

The action of hydrogen sulfide and hydrogen cyanide with the body appears to proceed by still a different pathway. These two gases, often encountered in the undergraduate laboratories, block cell metabolism and oxygen uptake by their action on various vital enzyme systems.

Finally, some materials, such as benzene, will cause direct disintegration of the red blood corpuscles through their action in the bone marrow.

Although the absorption of chemicals through the lungs is the prime health hazard in most research laboratories, a second, almost equally important, hazard arises from the interaction of chemicals and the skin.

Organic solvents such as benzene, carbon tetrachloride, and ether dissolve the protective oils and fats in the skin. The skin is then permeable to the solvent and allows the material to enter the body. Further, the skin, being defatted by this contact, may become infected by the entrance of bacteria, and eczema or other forms of dermatitis may result. Amines, phenols, and nitro compounds are particularly hazardous. Many fused ring aromatic systems are potential carcinogenic materials.

Strong acids and bases will cause burns of the skin quite comparable to burns due to heat. Tissue destruction may be extensive; and, if the eyes are involved, temporary or permanent blindness may result.

Aside from direct damage to the body by toxic materials, indirect damage may result by the operation of the body's defense mechanisms. The body will often react to render the toxic material less harmful or to excrete the material rapidly. For example, vomiting and diarrhea may result from the ingestion of toxic materials. Toluene may be oxidized to benzoic acid, which then combines with glycine and is excreted as hippuric acid in the urine. Unfortunately, some of the products of these in vivo reactions are themselves quite toxic, and often the increased load placed upon the excretory system will cause severe and permanent damage.

Very small quantities of toxic material can often be taken into the body without serious damage. The body may be able to cope with this material through its defense mechanisms or may be able to tolerate the low concentration of toxic substances; however, the body can only do so much. If the concentrations of toxic material pass a certain threshold value,

which differs for each individual and for each toxic substance, the body cannot cope with the problem and injury to the system occurs.

Many toxic materials are not excreted rapidly by the body, and repeated exposures to quantities too small to be harmful per se may cause a buildup of concentration in the body until the threshold value is exceeded and poisoning results. Materials of this type, of which mercury is a prime example, are said to be cumulative. Repeated absorption of a toxic substance may even lower the threshold, increasing a particular individual's sensitivity to the material.

The body may or may not give warning of the presence of toxic substances. Some materials are immediately recognized by smell, taste, irritation of the mucous membranes, pain, etc.; others are not, since their reactions are either pleasant or unnoticed, or because the body becomes accustomed to them. The latter materials, gasoline vapors for example, are quite dangerous since olfactory fatigue prevents any warning of toxic concentration.

The first symptoms of poisoning are usually not specific and may be mistaken for some other disease, malnutrition, or simply fatigue.

In order to give some idea of the relative dangers associated with the use of many common chemicals, The Committee on Threshold Limit of the American Conference of Government Industrial Hygienists has established a list of chemicals and the MAC value accepted for each. The MAC value, for gases expressed in parts per million and for dusts in milligrams per cubic meter of air, indicates the maximum allowable concentration of the toxic material to which an individual may be exposed during an 8-hour period. The MAC values are continually being revised, usually downward, and the MAC values must not be considered a quantitative dividing line between safety and danger. Any quantity of toxic material is too much.

### 2.3 Explosive Hazards

Any compound or mixture which may be potentially explosive\* should be regarded with suspicion and handled with more than usual care. When any doubt exists about a system, its potential energy (or potential explosion energy) can often be calculated.

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\* Such a compound or mixture is one whose heat of formation ( $\Delta Q_f = -\Delta H_f$ ) is smaller by more than  $\sim 100$ - $200$  cal/g than the sum of the heats of formation of its products.



The hazards involved in working with a potentially explosive system are directly proportional to the speed with which the energy is released and to the amount of gas evolved. Thus, since reaction rates are more difficult to predict than potential energy content, caution is always necessary. The volume of gas can, of course, be readily calculated if this energy is known. The most accurate values are obtained when all heats of formation involved are known, but if they are not, they can be readily obtained with a good degree of reliability from Pauling's bond-strength values (by Kharasch's method for calculation of heat of combustion, etc.), or estimated by analogy with sufficient precision. In making the calculation when data are not available for the distribution of elements in the products, one should assume a reasonable reaction to yield the most exothermic products.

Despite the ability to calculate a hazard index, it is most helpful to have structural and other indications to suggest properties and possible hazards. Certain groups, when present in a molecule, can either confer or enhance explosive properties and may increase sensitivity. For the radicals listed below, the ability to directly produce explosive power diminishes in passing from Group 1 to Group 2.

Group 1 - Radicals which confer explosive properties directly (contain phosphores or phosphoric groups)

a. Result in high potential energy

-ONO <sub>2</sub>	Nitrate
-NH-NO <sub>2</sub>	Primary nitramine
=N-NO <sub>2</sub>	Secondary nitramine
R-NO <sub>2</sub>	Aliphatic nitro compound
AR-NO <sub>2</sub>	Aromatic nitro compound
Salts, organic	Perchlorates, chlorates, nitrates, bromates, chlorites, and iodates.

b. Result in relatively low potential energy

-N <sub>3</sub>	Azide
-NO	Nitroso
-N=N-	Diazo
-N=N-S-N=N-	Diazo sulfide

-O-O-	Peroxide
=N-X	Halamines (X=F, Cl, Br, I)
-C≡C-	Acetylenes
-O-O-O-	Ozonides

Group 2 - Radicals which enhance potential energy

-CN	Nitrile
=N-OH	Oxime
-O-	Ether

In considering the above, it might be noted that, other factors remaining constant, as the oxygen balance approaches zero, energy content increases while sensitivity and stability decrease.

In Group 1a, the mixing of radicals in a molecule may have effects other than those expected from additivity alone. For example, nitrates are acid sensitive and are also sensitive to strongly basic groups. Thus, a molecule is immediately suspect if it contains a nitrate group and either (1) a primary nitramine, primary aliphatic nitro, phenolic OH, carboxyl, or other acid group; or (2) an amino, hydrazine, basic ammonia, or similar basic group. In general, the potential hazard of a molecule increases with the number of radicals and especially with their variety. This is also true when an electronegative element such as Cl is present on the same atom as a Group 1 radical; thus, the combination -N(Cl)NO<sub>2</sub> has been found to be particularly unstable and sensitive. In the case of organic salts, the hazard increases with the acid radical, as follows: Nitrates, perchlorates, iodates, bromates, and chlorates (most hazardous). At elevated temperatures, however, bromates are sometimes much less stable than chlorates. Chlorites are difficult to classify, but the lower oxy-acids of chlorine are quite reactive, and chlorites should be considered dangerous, especially when in contact with organic matter. In any case, extreme caution should be observed when mixing any of these salts with organic matter and/or acids (even dilute acids) because halogen oxides may be released.

In Group 1b are listed radicals which will make a molecule both unstable and exceedingly sensitive to impact. This is especially true because even as little as a milligram of such a material (e.g., lead azide) will detonate and release energy and gaseous products with amazing rapidity. Many of the compounds containing such groups are too dangerous to handle (e.g., phenyl diazosulfide has an explosion temperature of 25°C). The nitroso radical is the least hazardous to those in Group 1b, but its presence cannot be ignored.

The presence of groups with specific acid (-NH-NO<sub>2</sub>, -COOH), base (-NH-NH-), reactive (-CHO), oxidizing (-ClO<sub>3</sub>), reducing (-NO), or similar group characteristics cannot be ignored. In general, these usually increase sensitivity and decrease stability. The presence of phenolic groups may lead to a hazard through metal salt formation (especially heavy metals), while under certain conditions the presence of ether and carbonyl groups may lead to the formation of very dangerous peroxides.

Mixtures of organic materials with inorganic salts of the Group 1b acids are quite hazardous, as would be expected from the discussion of organic salts, and some are extremely sensitive to friction and impact. Besides the trends noted above, unsaturated or reactive organics present the greatest hazards, e.g., acetylenic derivatives, sulfur compounds, etc. The acids themselves are infinitely more reactive than the salts, and their liberation, even from salt mixtures, often leads to spontaneous explosion. Where it is possible that acidity may develop in such mixtures, fine sodium bicarbonate or chalk should be incorporated in the mixture.

The following list of specific chemicals is modified from the Explosives Research Section Safety Handbook of Picatinny Arsenal, Dover, New Jersey, dated December, 1959. It is not complete, and is intended as a rough guide only. A thorough literature search should be made on new compounds.

Acetic acid (glacial) will ignite at elevated temperatures (flash point, 140°C). It will also irritate the skin and the mucous membranes of the mouth and throat. MAC, 10 ppm in air. Incompatibles: chromic acid, nitric acid, hydroxyl containing compounds, ethylene glycol, perchloric acid, peroxides, and permanganates.

Acetic anhydride, same as above. Reacts violently with some hydroxyl compounds; it is known to explode with ethylene glycol and HClO<sub>4</sub>.

Acetone decomposes violently when brought into contact with H<sub>2</sub>SO<sub>4</sub>/HNO<sub>3</sub> mixtures such as are used in nitration reactions. This is especially true when the reaction is partially confined by dropping the acetone into a narrow-mouthed bottle containing the mixed acids. Acetone water solutions are flammable over a very wide range. A 5% solution will flash at 60°C if an ignition source is present. The higher the percentage, the lower the flash point, until pure acetone is reached, with its flash point of -17.8°C. MAC, 1000 ppm in air. Narcotic. Explosive range, 2.55-12.8%.

Acetylene, under certain conditions, forms highly explosive compounds with cuprous iron, silver, and mercury. These compounds are easily detonated by heat or shock. Flash point, 0°F (C.C.). Explosive range, 2.5-80%. Usually acts as a simple asphyxiant. Incompatibles: chlorine, bromine, copper, silver, mercury, and fluorine.

Alkali and alkaline earth metals such as sodium, potassium, cesium, lithium, magnesium, calcium, and aluminum are hazardous in these ways: (1) Explosions or fire resulting from contact with water, chlorinated hydrocarbons, or other reactive agents; (2) fires resulting from exposure to air; (3) personnel injury from direct contact with hot metals; and (4) caustic burns from the residue of reaction with water. Storage areas must be dry. Protective clothing must be worn. Both alkali metals and metallic hydrides produce hydrogen on contact with water and sufficient heat to ignite the gas with explosive rapidity. Waste scraps of these materials should be added in small portions to a high-boiling alcohol such as propanol or butanol and disposed of after all reaction ceases. Do not use CO<sub>2</sub> or sodium bicarbonate dry-chemical extinguishers.

Aluminum chloride added to nitrobenzene containing 5% phenol rises in temperature and explodes violently. Heating nitrobenzene with either AlCl<sub>3</sub> or phenol causes no explosion, but at 120°C the three together react violently and form a tar. This shows the danger of working with AlCl<sub>3</sub>, nitrobenzene, and an oxidizable material. AlCl<sub>3</sub> will react with water or steam to form heat and fumes of HCl.

Ammonia (anhydrous) - MAC, 100 ppm in air. Irritating to eyes and mucous membranes. Incompatibles: mercury, chlorine, calcium, hypochlorite, iodine, bromine, and hydrogen fluoride.

Ammonium nitrate decomposes exothermally above 160°C, producing gaseous decomposition products. With large quantities or in a confined space, this reaction develops into a violent explosion. Mixtures with oxidizable substances, such as metal powders and organic materials, are liable to a spontaneous exothermic reaction and can explode.

Aniline is a blood poison and can enter the body through the skin, the lungs, and the digestive tract. It has little appreciable local irritating action, but illness can result from inhalation of vapors or from absorption through the skin. As a result, aniline should only be used in a hood. Many derivatives of aniline (such as methyl violet, nitraniline, dimethylaniline, and diaminobenzene) have similar poisonous properties. MAC, 5 ppm in air. Incompatibles: nitric acid and hydrogen peroxide.

Benzene is both flammable and highly toxic, and it should never be used where another solvent can be substituted. Its vapor is considerably heavier than air, so it can travel along floors and benches. Benzene and its homologs should never be poured down a sink. Flash point, 12°F (C.C.); MAC 35 ppm in air. Incompatibles: nitric acid and other oxidizing agents.

Bromine (liquid) causes immediate burns on the skin and mucous membrane. Bromine should be handled only in a hood, using goggles and rubber gloves. MAC, 1 ppm in air. Incompatibles: ammonia, acetylene, butadiene, butane and other petroleum gases, hydrogen, sodium carbide, turpentine, benzene, and finely divided metals.

Carbon disulfide is poisonous, endothermic, and can be detonated by shock. Flash point, 22°F (C.C.); autoignition temperature, 257°F; MAC, 20 ppm. Mixed with air or oxygen, it forms a violently explosive mixture which can be fired by flame or shock, or by catalytic agents such as rust.

Carbon monoxide is highly toxic and especially treacherous because it is both odorless and tasteless. It is formed by incomplete combustion and reacting carbonyls. MAC, 100 ppm in air. Explosive range, 12.5-74.2%.

Carbon tetrachloride has a strong narcotic action and can cause serious kidney and liver damage. MAC, 25 ppm in air. When heated to decomposition, it emits highly toxic fumes of phosgene.

Chlorine and hydrogen or various hydrocarbons can ignite spontaneously. Chlorine and gaseous formaldehyde can react explosively under certain conditions at temperatures as low as 100°C. MAC, 1 ppm in air. Extremely irritant to mucous membranes of eyes and respiratory tract. Incompatibles: turpentine, ether, ammonia gas, illuminating gas, hydrocarbons, hydrogen, powdered metals, sodium carbide, benzene, sodium, and potassium metal.

Diazonium compounds react vigorously with sodium sulfide or hydrogen sulfide and are known to explode. They are a severe explosion hazard when exposed to heat, shock, or chemical reaction.

Dimethyl sulfoxide decomposes violently when in contact with a wide range of acyl halides and related compounds, such as cyanuric, acetyl, benzoyl, benzenesulfonyl, thionyl, phosphoryl chlorides, and PCl<sub>3</sub>. DMS should be used with caution as a solvent in exploratory reactions. Unknown toxicity. Flash point, 203°F (O.C.).

Ethers - Diethyl ether is very flammable at room temperature. It has a high vapor pressure, low flash point, and low ignition temperature. Mixtures of ether vapor with air are explosives over a wide range. The following ethers form peroxides on storage: isopropyl ether, diethyl ether, diethylene ether, tetrahydrofuran, ethylene glycol dimethyl ether, and diethylene glycol dimethyl ether. Sunlight hastens the reaction. These materials should be discarded after 1 year if unopened, and within 3 months after opening. Another group including all other ethers, all liquid paraffins, all olefins, tetralin (tetrahydronaphthalene), and aromatics

(excluding benzene and toluene) form peroxides on concentration by distillation or evaporation. In any case, at least 10% undistilled residue should be left in still pots. These light materials should not be poured into sinks. Waste can should be grounded when pouring.

Fluorine is extremely active and hazardous at 20°C; mixtures of F and ClO<sub>2</sub> react explosively to form chlorine and oxygen. Handling procedure is given in sheets published by The Matheson Company.

Hydrazine vapor, when sparked at 100°C, explodes with a yellow flame, producing ammonia, hydrogen, and nitrogen. It is a very sensitive explosive. Very toxic. MAC, 1 ppm in air.

Hydrogen is a very dangerous explosive hazard. Explosive range, 4.1-74.2%. Waste gas is best burned with a quiet flame.

Hydrogen chloride gas is a primary irritant with a destructive action on mucous membranes. MAC, 5 ppm in air.

Hydrogen cyanide and other cyanides and cyanogens are well-known highly poisonous compounds. Poisoning proceeds very rapidly, causing a reflex cessation of the heart action so that first aid generally comes too late. Flash point of HCN, 0°F (C.C.). MAC, 10 ppm. If in contact with alkaline materials, HCN can polymerize or decompose explosively. Incompatible with nitric acid.

Hydrogen fluoride is a poison with a strongly irritant action on the skin and the mucous membranes. The concentrated acid produces open wounds which heal slowly. Soluble fluorides are toxic. Particular care should be taken that no HF gets under the fingernails, as this causes painful and prolonged inflammation.

Hydrogen peroxide detonates rapidly in the pure state, but will not detonate in aqueous solutions containing less than 94% H<sub>2</sub>O<sub>2</sub>, although aqueous solutions will decompose explosively under catalytic influence. Such catalysts are copper, chromium, iron, and most metals and their salts. H<sub>2</sub>O<sub>2</sub> causes burns which should be treated with hot water or with a solution of sodium thiosulfate. In concentrations above 50%, H<sub>2</sub>O<sub>2</sub> can be a fire hazard in the presence of easily oxidizable material. Mixed with organic material such as glycerine, aniline, or nitromethane, it is unusually explosive, especially when contaminated. Storage containers even for dilute solutions should be vented because of gradual decomposition to water and oxygen.

Hydrogen sulfide is very poisonous; MAC, 20 ppm in air. Low concentrations have a very disagreeable odor, but higher concentrations and even

relatively short exposure cause the sense of smell to become temporarily atrophied. Explosive range 4.3-46%. Excess gas is easily disposed of by burning in a hood.

Lithium aluminum hydride is not safe for drying methyl ethers. Tetrahydrofuran can cause fire when used as a solvent for  $\text{LiAlH}_4$ . Extreme caution should be used in reducing any fluorinated compound with  $\text{LiAlH}_4$ . Violent reactions have been observed with two different fluor compounds. Excess trimethyl amine,  $\text{NMe}_3$ , reacts with an ethereal solution of  $\text{LiAlH}_4$  in vacuo at  $-50^\circ\text{C}$  to form a white addition compound which is slightly soluble in ether, insoluble in benzene, and spontaneously flammable in air. For particulars in handling and using this material, see the publications of Metal Hydrides Incorporated.

Magnesium perchlorate may be used as a very effective drying agent and as a quantitative absorbent of vapors of some polar compounds. Caution must be taken to exclude flammable materials, mineral acids, or substances liable to hydrolyze them. Serious explosions have resulted from using magnesium perchlorate for drying an organic liquid which had been in contact with sulfuric acid and had been inadequately washed. The compound used in drying unsaturated hydrocarbons exploded on heating to  $220^\circ\text{C}$ . See the bulletin of the G. Frederick Smith Chemical Company.

Mercaptans of low molecular weight generally have a very strong odor which may cause nausea and headache. High concentrations can produce unconsciousness. When heated to decomposition, they emit highly toxic oxides of sulfur. They will react with water, steam, or acids to produce toxic and flammable vapors, and can react with oxidizing materials.

Mercuric nitrate and organic compounds react vigorously and exothermally at room temperature without exploding during attempts to remove sulfides.

Mercury and ammonia gas are known to explode. The MAC for Hg is very low: 0.01 ppm in air. Clean up spills immediately.

Nitric acid (concentrated) attacks the skin, forming a yellow stain which remains for a considerable period of time. The vapor is a serious respiratory poison. Use of a face shield, protective clothing, and adequate ventilation are imperative.  $\text{HNO}_3$  is a powerful oxidizing agent, causing fire and explosion hazards when mixed with combustible materials. Mixtures of the concentrated acid and an organic substance such as acetone, acetic anhydride, alcohol, or aniline may detonate. Traces of an organic substance with  $\text{HNO}_3$  and mercury may form a highly explosive fulminate.

Nitro compounds are very toxic. They may enter the body via the lungs, through the skin, or with food. Recovery from poisoning with nitro compounds is slow. Protection against such poisons is based on cleanliness, ventilation, protective clothing, goggles, and respirators. These statements apply as well to nitrobenzenes, nitromethanes, and nitrochlorobenzene.

Nitrogen oxide fumes are most hazardous, as they can develop unexpectedly (e.g., when a bottle of  $\text{HNO}_3$  breaks, or when  $\text{HNO}_3$  comes into contact with organic substances such as sawdust). Any atmosphere in which nitrogen oxides can be smelled must be considered dangerous. Reactions in which nitrogen oxides may be produced must be performed in a hood. MAC for nitric oxide is 25 ppm in air.

Nitroparaffins are sensitive to alkali, and many sensitive combinations ranging from addition complexes, through metal salts, to the very dangerous fulminates, can be formed, depending on the base involved. All these compounds are usually more sensitive than the original nitroparaffin.

Oxygen - Incompatibles: oils, greases, hydrogen, and flammable liquids and solids. Do not oil reducing gauges or fittings.

Perchlorates in mixtures with reducing materials are more stable than the corresponding chlorates and are not as subject to dangers from the presence of free acids. It should not be assumed that all perchlorate mixtures can be initiated by friction, impact, or spark.

Perchloric acid is an extremely powerful and dangerous oxidizing agent which may react explosively with reducing agents and organic compounds. It should be used with extreme caution and in the smallest quantities possible.

Perchloric acid should always be used in a noncombustible hood, preferably with flushing facilities. Before the acid is used, the exhaust fan should be run for a few minutes to remove all loose particles of dust, etc., from the hood.

Solutions of perchloric acid should be heated with an electric hot plate or a heating mantle, and never in an oil bath.

Organic matter in samples should first be oxidized with concentrated  $\text{HNO}_3$  before adding the  $\text{HClO}_4$ . Never heat  $\text{HClO}_4$  with sulfuric acid or any other high-boiling acid, as dehydration will produce the explosive anhydrous  $\text{HClO}_4$ .



Combustible materials (filter paper, rags, etc.) which have been soaked with aqueous  $\text{HClO}_4$  and dried, should be treated with extreme care as they may be ignited by impact, friction, or heat. The condensed vapor will form explosive mixtures with ammonia or with  $\text{HNO}_3$ .

Never use rubber or cork with apparatus when working with  $\text{HClO}_4$ . Use an all-glass apparatus, and do not use stopcock grease.

The 60-70% commercial acid is stable and can be stored safely. At concentrations above 85%, the acid is liable to spontaneous explosion. Discolored (i.e., contaminated) acid should be poured carefully into cold water and flushed. Perchloric acid reacts with bismuth to form a spontaneously explosive compound.

Perchloric acid esters are violently explosive. An alcoholic solution of  $\text{HClO}_4$  should never be heated unless considerable water is present, as it may explode.

Phosphorous trichloride - An explosion occurred in the preparation of acetyl chloride from acetic acid and  $\text{PCl}_3$ , the  $\text{AcCl}$  being distilled with a free flame.  $\text{PH}_3$  is believed to have been the cause.  $\text{PCl}_3$  reacts with water to give phosphine, which ignites spontaneously. MAC, 0.5 ppm in air.

Sodium covered with sodium methylate or butylate, formed in the presence of toluene, explodes when thrown into water. It should be noted that metallic sodium reacts exothermally with the moisture of body or tissue surfaces, causing thermal and chemical burns due to the reaction with sodium and the sodium hydroxide formed. Only dry sodium chloride, sodium carbonate, or graphite should be used for fighting fires.

Sodium borohydride is a very useful reducing agent like lithium aluminum hydride. See the Metal Hydrides manual.

Concentrated sulfuric acid and oleum cause burns on the skin and have a destructive action on all organic matter. Concentrated  $\text{H}_2\text{SO}_4$  sometimes reacts violently with many organic substances, nitrates, sulfides, chlorates, perchlorates, halogenides, carbides, and many metals. Concentrated  $\text{H}_2\text{SO}_4$  should never be used in a desiccator unless it is certain there will be no reaction with the material to be dried. Accidental spills have caused explosions.

Toluene is less toxic than benzene (MAC, 200 ppm), but more dangerous than xylene. Flash point,  $40^\circ\text{F}$  (C.C.); explosive range, 1.27-7.0%.

Trichlorethylene, often recommended for use in place of  $\text{CCl}_4$ ; is about "half" as toxic, and is often used instead of it. Some persons even grow addicted to it. MAC, 200 ppm in air.

Xylene, although toxic, is less dangerous than toluene or benzene. It is more irritating to the skin than is benzene. Flash point, about  $77^\circ\text{F}$ ; MAC, 200 ppm in air.

#### 2.4 Partial List of Incompatible Chemicals\*

Substances in the right-hand column should be stored and handled so they cannot possibly contact corresponding substances in the left-hand column.

Alkaline and alkaline earth metals, such as sodium, potassium, cesium, lithium, magnesium, calcium, aluminum	Carbon dioxide, carbon tetrachloride, and other chlorinated hydrocarbons. (Also prohibit water, foam, and dry chemical on fires involving these metals.)
Acetic acid	Chromic acid, nitric acid, hydroxyl-containing compounds, ethylene glycol, perchloric acid, peroxides, and permanganates.
Acetone	Concentrated nitric and sulfuric acid mixtures.
Acetylene	Chlorine, bromine, copper, silver, fluorine, and mercury.
Ammonia (anhyd.)	Mercury, chlorine, calcium hypochlorite, iodine, bromine, and hydrogen fluoride.
Ammonium nitrate	Acids, metal powders, flammable liquids, chlorates, nitrites, sulfur, finely divided organics or combustibles.
Aniline	Nitric acid, hydrogen peroxide.
Bromine	Ammonium, acetylene, butadiene, butane and other petroleum gases, sodium carbide, turpentine, benzene, and finely divided metals.
Calcium carbide	Water (see also acetylene).

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\* Compiled by R. H. Sullivan, Burnside Laboratory, ca. 1956.

Calcium oxide	Water.
Carbon, activated	Calcium hypochlorite.
Copper	Acetylene, hydrogen peroxide
Chlorates	Ammonium salts, acids, metal powders, sulfur, finely divided organics or combustibles.
Chromic acid	Acetic acid, naphthalene, camphor, glycerine, turpentine, alcohol, and other flammable liquids.
Chlorine	Ammonia, acetylene, butadiene, butane and other petroleum gases, hydrogen, sodium carbide, turpentine, benzene, and finely divided metals.
Chlorine dioxide	Ammonia, methane, phosphine, and hydrogen sulfide.
Fluorine	Isolate from everything.
Hydrocyanic acid	Nitric acid, alkalies.
Hydrogen peroxide	Copper, chromium, iron, most metals or their salts, any flammable liquid, combustible materials, aniline, nitromethane, caustic soda and other strong alkalies.
Hydrofluoric acid, anhyd. (hydrogen fluoride)	Ammonia, aqueous or anhydrous.
Hydrogen sulfide	Fuming nitric acid, oxidizing gases.
Hydrocarbons (benzene, butane, propane, gasoline, turpentine, etc.)	Fluorine, chlorine, bromine, chromic acid, sodium peroxide.
Iodine	Acetylene, ammonia (anhyd. or aqueous).
Mercury	Acetylene, fulminic acid, ammonia.

Nitric acid (conc.)	Acetic acid, aniline, chromic acid, hydrocyanic acid, hydrogen sulfide, flammable liquids, flammable gases, and nitritable substances.
Nitroparaffins	Inorganic bases.
Oxygen	Oils, grease, hydrogen, flammable liquids, solids or gases.
Oxalic acid	Silver, mercury.
Perchloric acid	Acetic anhydride, bismuth and its alloys, alcohol, paper, wood, grease, oils.
Peroxides, organic	Acids (organic or mineral); avoid friction.
Phosphorus (white)	Air, oxygen.
Potassium Chlorate.	Acids (see also chlorate).
Potassium perchlorates	Acids (see also perchloric acid).
Potassium permanganate	Glycerine, ethylene glycol, benzaldehyde, sulfuric acid.
Silver	Acetylene, oxalic acid, tartaric acid, fulminic acid, ammonium compounds.
Sodium	See alkaline metals (above).
Sodium nitrate	Ammonium nitrate and other ammonium salts.
Sodium oxide	Water.
Sodium peroxide	Any oxidizable substance, such as ethanol, methanol, glacial acetic acid, acetic anhydride, benzaldehyde, carbon disulfide, glycerine, ethylene glycol, ethyl acetate, methyl acetate, and furfural.
Sulfuric acid	Chlorates, perchlorates, permanganates.

Zirconium

Prohibit water, carbon tetrachloride foam, and dry chemical on zirconium fires.

Reference: Based partly on Dangerous Chemicals Code, 1951 Edition, pp. 19-20, Bureau of Fire Prevention, City of Los Angeles, Fire Department, published by Parker & Company, Los Angeles 13, California.

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### III. HOUSEKEEPING

Proper care of equipment and working areas is an important part of a safety program. Good housekeeping implies absence of debris and excess materials. Such conditions obviously promote safety by removal of the source of possible accidents.

#### 3.1 Arrangements

- a. A definite storage place shall be provided for each item and the item placed therein when not in use. Such storage areas shall be always neat, clean, and orderly.
- b. Aisles and walkways shall be kept clean, dry, and free of obstructions or tripping hazards.
- c. Arrangement of laboratory equipment shall be planned so that transportation of hot objects or chemicals in glassware is kept to a minimum. When necessary, use tongs and/or asbestos gloves.
- d. When choosing a site for an operation or the storage of materials, consideration shall be given to the toxicity, flammability, fragility, and/or the other hazardous properties of the materials being handled.

#### 3.2 Equipment

Maintenance:

- a. Equipment shall not be placed near the edge of the lab bench where there is a possibility of its being struck.
- b. Dirty glassware should be washed promptly and returned to proper storage.
- c. Equipment and working surfaces shall be cleaned frequently to prevent accumulation of dust.
- d. Cracked or chipped glassware shall be disposed of in the proper manner and then replaced. If the item is very expensive or cannot be easily replaced, it should be repaired and all the sharp edges fire-polished.
- e. Drawers and cabinet doors shall be kept closed when not in use.
- f. Functional equipment of all types shall be kept in safe working condition.

## Operation:

- a. Apparatus and reagents, after use, should be returned promptly to their place of storage.
- b. Items for which there may be further need shall be arranged in an orderly manner about the working area. Keep bench tops clean.

### 3.3 Spills and Leaks

- a. Spilled materials, whether liquid or solid, corrosive or inert, shall be cleaned up promptly and completely. Spilled corrosive liquid, assumed to be water, has been the cause of numerous chemical burns to laboratory workers; consequently, all liquid spills shall be handled as corrosive unless the material is definitely known to be noncorrosive.
- b. Should the spilled material be flammable, toxic, or corrosive, the person involved shall take all precautionary steps necessary to guard against injury to personnel during the cleaning process. If flammable, all flames in the vicinity shall be extinguished.
- c. Do not place corrosive or toxic materials in the waste cans. Dilute any acidic and basic wastes with water. Laboratory supervision shall be consulted when disposing of toxic materials.
- d. Care shall be exercised so that spills are cleaned up in the approved manner. Leather gloves shall be worn when disposing of pieces of broken glass. Glass fragments shall be swept up immediately and discarded in the prescribed manner.

### 3.4 Waste Disposal

- a. Adequate facilities are supplied for the disposal of wastepaper. Paper should not accumulate on the tables or desks.
- b. Packing materials shall be disposed of immediately.
- c. Flammable materials such as ether, methyl ethyl ketone, etc., shall not be discarded in the sink. Wastes from these and other organic materials may be air evaporated in a flame-free hood or disposed of in a specially provided container for waste solvents.
- d. Acidic or alkaline solutions shall be diluted to less than one molar prior to being discarded in the sinks.



#### IV. SAFETY EQUIPMENT

In a sense, any properly designed tool may be termed an article of safety equipment since, among other considerations, it is designed to do a job safely. Safety equipment may be divided into two general classes: First, that used for protection of life or property in case of emergency or unusual happenings; and second, that used daily as protection against known and anticipated hazards.

##### 4.1 Emergency Equipment

No obstruction, even temporary, should prevent access to this equipment.

- a. A fire alarm shall be turned in immediately if there is a remote possibility that the fire cannot be extinguished by means of the portable extinguishers at hand.
- b. A fire extinguisher is a device or substance which will cool a flaming material below its ignition temperature, or smother it by exclusion of oxygen, or both. In case of fire, serious damage may be avoided through prompt use of an extinguisher.
- c. Water should not be used on an electrical fire until all electrical power to the burning equipment has been turned off. The best way to remove power, if the plug cannot be reached, is to turn off the appropriate circuit breaker(s).
- d. The drinking fountain serves admirably for the irrigation of the eyes, should corrosive liquids be splashed therein. If the faucet over the sink has a piece of tygon tubing attached, this also provides a convenient method for flushing the eyes.
- e. The safety shower should be used without hesitation in the event acid, etc., is spilled on the clothing or skin. The shower should be checked each month for proper operation.

##### 4.2 Protective Equipment

This classification covers items which are used to guard against injury in case of mishap, or for the protection of health. Depending on their frequency of use, they may be assigned to the individual, be located at the points of probable need, or be part of a permanent installation.

- a. Laboratory coats or aprons shall be worn by all persons handling chemicals in the laboratory and by such other persons whose activities subject them to hazards due to spills or splashes.

- b. Eye protection is required when working in the laboratory area. Safety glasses should be worn by personnel.

Splashproof chemical goggles or a faceshield shall be used when handling large quantities of corrosive liquids or in carrying out operations in which there is a possibility of splash of corrosive liquids.

- c. Gloves can protect the wearer from many injuries, but unless judiciously used, they may increase rather than diminish the net hazard.
  - (1) Asbestos gloves should be used when handling hot or cold objects.
  - (2) Leather gloves should be used for handling broken glassware, for inserting glass tubes into rubber stoppers, and for like operations where a firm grasp of a small object is required, but where protection from corrosive chemicals is not necessary.
  - (3) Heavy rubber gauntlet gloves should be used in handling large volumes of corrosive liquids. It shall be remembered that rubber is prone to become slippery when wet, and care shall therefore be exercised when gloves become wet.
  - (4) Surgical gloves or other lightweight rubber gloves should be used for protection against toxic, irritating, corrosive, or radioactive materials in operations requiring dexterous use of the fingers.
- d. Hood windows should be lowered for control of fumes and to provide physical protection in operations involving the heating (and, where possible, other handling) of flammable, corrosive, or potentially explosive materials. A lowered window should not be raised until it is established that no hazardous operation is being carried on behind it.
- e. Safety shields are to be used in operations involving possible explosions or flying particles. Some type of shield should be used for distillations in glass which involve appreciable quantities of corrosive materials or flammable solvents, and/or use of vacuum technique. It should also be used for reactions of chemicals whose characteristics are unknown or those which

may conceivably become explosive or otherwise uncontrollable. The hood window is made of safety glass and is an adequate shield in many cases.

- f. Soap and water, if frequently used on the hands and arms, will remove unsuspected bits of irritating and/or radioactive materials.
- g. Stepladders or footstools should be used for reaching high objects. Never climb on handrails, swivel chairs, piled boxes, or other makeshift devices. Ladders and stools, when not in use, shall be stored in such a way that they represent no tripping hazard.
- h. Good health and, consequently, good morale are great aids to safety. When feeling a "little off," the laboratory worker should be particularly careful to avoid accidents.
- i. Protective skin cream, if applied to the hands, may be useful in preventing drying and cracking of the skin.

## V. LABORATORY MANIPULATIONS

While there may be more than one way of doing a job, repetition and experience soon teach that one particular method has more desirable qualities and fewer objectionable ones than other methods. If proper completion of the job is one of the desirable qualities, this method then becomes a standard procedure for doing that operation. So it is with laboratory techniques. There are many ways of actually doing the various jobs, but experience has taught us certain "do's" and don't's" which must be observed if we are to obtain the desired results and, at the same time, protect ourselves and our fellow workers from injury.

### 5.1 Glassware

- a. Broken or chipped glassware shall not be used, nor returned to storage. If it is beyond repair, it is to be placed in the broken-glass container. When in doubt regarding proper disposal, consult your instructor.
- b. Broken glass in sinks presents a serious hazard, since it may not be visible in the presence of water. When broken in the sink, it shall be removed promptly. Furthermore, it is well to consider the possibility of its presence when reaching into the sink for any purpose.
- c. Leather gloves shall be worn to clean up the larger pieces of broken glassware. Sweep up the remaining fragments as soon as possible. Place all broken glassware in an ice-cream carton prior to disposal in the waste container.
- d. Remove sharp or jagged edges from glassware before using it. The edges on all Pyrex tubing and/or similar apparatus shall be fire-polished.
- e. Combustible or heat-sensitive materials of any nature shall be thoroughly cleaned from glass containers before any heating for repairs is attempted. After removal of all visible materials, the container shall be thoroughly purged with air to assure absence of flammable vapors.
- f. Inserting glass tubing into stoppers or rubber tubing is hazardous unless the following precaution is observed: Glass or metal tubing shall be inserted entirely through stoppers to assure that the end may not be closed over by heat or solvent action.

- g. To remove glass tubing or thermometers from rubber, it is generally desirable to cut away the rubber. This is particularly true if the tubing or stopper has been in place over an extended period of time, or if it has been subjected to heat.
- h. In handling beakers, support them by grasping around the sides, never over the top. If the hand will not reach more than halfway around the beaker, both hands may be used. Large beakers (one liter or more) should be supported from the bottom when in use.
- i. When heating materials in glass by means of a gas flame, the glass shall be protected from direct contact with the flame through use of a wire gauze or asbestos centered wire gauze.
- j. When placing liquids in bottles which have a positive closure, more than 5 percent of the volume shall be reserved as air space to allow for expansion due to temperature changes.
- k. Frozen stopcocks or ground glass stoppers shall not be forced. Either (1) use a stopcock puller; or (2) if the nature of the contents of the vessel permits, cool the shell until the plug is cold, then heat the shell of the joint momentarily. Withdraw the plug quickly before it has been expanded by the heat.

## 5.2 Vacuum

- a. Glassware under vacuum shall be protected from physical shock which might cause cracks, resulting in collapse with explosive violence.
- b. Flat-bottom flasks shall not be subjected to vacuum unless constructed with heavy walls specifically for such service.
- c. Standard-wall (spherical) flasks larger than one liter, when used in vacuum service, shall be guarded by a shield that is adequate to stop all flying glass, should collapse occur.
- d. Desiccators in vacuum service shall be completely enclosed in a shell which will guard against flying glass, should the vessel collapse.
- e. Cork or rubber stoppers used in vacuum service shall be carefully chosen and shall be of such size that they will not be drawn into the opening which they are intended to close.

- f. Vacuum in any system shall be relieved before any attempt is made to disassemble the equipment.
- g. Hot flasks containing uncondensed vapors or steam shall not be stoppered; to do so will subject them to vacuum on cooling.
- h. Vacuum service lines shall be protected from entry of corrosive or oxidizing materials. The former would corrode the lines, while the latter might lead to explosion in combination with combustible vapors that might be present.

### 5.3 Acids

- a. Rubber gloves shall be worn when handling concentrated acids.
- b. When handling concentrated acids, acid goggles, a rubber apron, and rubber gloves shall be worn.
- c. When handling concentrated acids generally:
  - (1) Splashproof goggles shall be worn when pouring concentrated hydrofluoric acid from the reagent bottles.
  - (2) Pour acids into water when diluting. An exception to this rule may be permitted in the case of hydrochloric or nitric acid, as directed by supervision, but never in the case of sulfuric or phosphoric acid.
  - (3) Store acid containers in inert trays if the acid concentration is greater than 2N. Lead or polyethylene trays are suitable.
  - (4) When emptying acids and cleaning solutions into the drains, first fill the sink with water, pour the acid or cleaning solution into the sink, and then let the water run out. Allow the tap to remain on full for a few additional minutes. Dilutions should always be sufficient to reduce the acid concentration to less than one normal.
  - (5) If the safety of any operation is doubtful, do not guess, contact the instructor.

### 5.4 Bases

Concentrated ammonium hydroxide and solutions of potassium and sodium hydroxide which are 10N or above shall be treated as if they were concentrated acids and the above rules followed. However, they shall not be stored in the same trays with acids. Concentrated bases should not be stored in glass-stoppered bottles.

## 5.5 Volatile Solvents

- a. Volatile solvents not in use shall be stored in an approved safety can.
- b. No open flames are permitted in the areas where volatile solvents are used or stored.
- c. Volatile solvents that are immiscible with water shall be retained in approved safety cans labeled "waste solvent" and periodically disposed of by dumping in a controlled area (e.g., waste-disposal site) and allowed to evaporate. If the solvent contains traces of a corrosive acid (e.g., HCl), the solvent shall be washed with dilute (1N) NaOH solution to neutralize the acid prior to disposal in the safety can. Small quantities of volatile solvents may be allowed to evaporate in a flame-free hood.

## 5.6 Miscellaneous

- a. Air-driven agitators shall be used for stirring any flammable liquid which has a low flash point.
- b. The cover of a centrifuge shall not be lifted until rotation has stopped. The cover shall be closed when the centrifuge is not in use.
- c. When using a cork borer, the stopper shall not be held in the palm of the hand. Rather, it should be placed large end down on a soft, flat surface and held in place with the thumb and forefinger. The cork borer shall be kept sharp and should be lubricated with water or glycerine to prevent binding. It is not to be forced through the stopper, but rather twisted, to cut the material. The use of leather gloves gives added protection in case of slippage.
- d. Rigidity of equipment assemblies shall be assured by the use of clamping devices.
- e. Permanent descriptive labels shall be affixed to all bottles, flasks, or other containers of chemicals, even though the contents are harmless.
- f. Furnace and oven doors shall be provided with vents or safety latches which will release readily in case of internal pressure. Volatile, flammable materials shall not be placed in ovens or furnaces.

- g. Services (air, water, gas, etc.) shall be turned off at the service cock when not in use; otherwise changes in pressure may suddenly dislodge the rubber tubing and lead to accident and possible injury.
- h. Stoppers should not be set down while liquids are being poured, but they should be held in the approved manner.
- i. Hot plates, etc., although not in evident use, may be hot. Never assume such equipment to be cold.
- j. When transporting hot equipment, tongs or asbestos gloves shall be used.
- k. Materials having toxic fumes shall be kept in hoods.

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#### C. Films

Safety Films may be rented or purchased from the following institutions:

1. Indiana University, Audio-Visual Aids Dept., Bloomington, Ind.
2. Manufacturing Chemists' Assoc., Inc., 1825 Conn. Ave. NW, Washington 9, D. C.
3. National Board of Fire Underwriters, New York, N. Y.
4. National Safety Council, 425 N. Michigan Ave., Chicago 11, Ill.
5. U. S. Public Health Service, Division of Occupational Health, Washington 25, D. C.
6. Visual Sciences, Suffern, New York.

D. Posters and Charts

1. Alpha Chi Sigma Fraternity, 5503 E. Washington Street, Indianapolis 19, Indiana (Eight different safety posters, mailed postpaid in any quantity for ten cents each.)
2. The Chemical Poster Company, 619 Templin Road, Iowa City, Iowa. (Posters on Chemical Safety.)
3. U. S. Dept. of Labor, Washington 25, D. C. (Chemical Safety Chart Series.)



## CHEMICAL SAFETY

### Eye-Wash Fountains and Safety Showers

#### Introduction

The following illustrates the importance of having an eye-wash fountain wherever chemicals are used. In a large chemical plant too many serious eye injuries from chemical burns occurred despite the well-established routine of thorough irrigation with neutralizing solutions. It was noted that in several cases in which men had used plain water, the eyes were not injured. In a study, 2000 cases were treated with the standard irrigation, using neutralizing solutions; 2000 cases were treated by the new method of 5 minutes of immediate flushing with water at an eye fountain at the scene of the accident, followed by 15 to 30 minutes of irrigation with water at the plant medical treatment room. The water-washing method saved 98 percent of the eyes from any damage. Of those treated with neutralizing solutions, nearly half required continued treatment and about one fourth suffered eye damage.

#### Required and Approved Types

Eye-wash fountains are required in all laboratories and work areas where chemicals, acids, caustics, and solvents are handled.

To be effective, an eye-wash fountain must supply an adequate flow to the eyes at a pressure which permits the lids to be held open.

The approved eye fountain which meets these requirements is the Aerated Eye Wash Fountain available with large vitreous or stainless-steel bowl and safety positioned fountain heads.

Safety showers must be capable of washing off large spills of chemicals, acids, and caustics, and to put out fire in a person's clothing or hair. Obviously, any shower head that can become clogged from mineral deposits may not function when it is needed. Therefore, such shower heads should be examined periodically for deposits.

Modern safety shower heads of the nonclogging, self-cleaning, nonspraying deluge type are now available.

#### Testing Fountains and Showers

Each area supervisor shall delegate some person to test eye fountains in his area once each week. Individual employees should inspect eye-wash fountains daily for their own protection.

Testing merely requires a simple push on the eye-wash fountain lever to ascertain if two properly separated streams of clear water flow to the proper height (6 to 8 inches above the bowl drain cover) to wash both eyes. The two water-wash streams should merge slightly above the washing height. If they fail to rise and merge at least 5 inches above the bowl drain cover, notify Plant Maintenance.

1. Installation standards

- a. Every overhead shower should be capable of delivering at least 30 gallons of water per minute.
- b. The eye wash fountains must have a sufficient flow rate to operate as indicated above.

Any hand-held eye wash hose type must produce a soft spray pattern of sufficient diameter to flood both eyes simultaneously. (About 4" diameter at 4" from nozzle.)

- c. The control valves which turn on the wash water to the showers and fountains should be a standard positive action type, which requires no more than one motion to activate. The valves should remain on automatically until turned off by a separate motion. The control handles should be conveniently located to the shower to allow them to be turned on quickly even if the victim has been blinded.

2. Maintenance surveillance

- a. At intervals of no more than one year, every emergency wash facility should be inspected and certified to be operating satisfactorily. A certification card similar to those used by the Fire Marshal on fire extinguishers should be attached to the facility on which the inspector can sign, date, and state the condition of the facility.

3. Emergency wash facility identification

- a. On the floor under each shower, a sign should be painted denoting the area as a "Safety Shower."

Safety showers are tested and inspected annually by Plant Engineering. The supervisor of each area where safety showers are required shall promptly report any damage or malfunction.

### Perchloric Acid

The use of perchloric acid in the laboratory presents a constant danger of explosion. The majority of serious accidents in the laboratory have involved chemists with years of experience with perchloric acid. While it is obvious that one should use only the best materials and construction features, this will not guarantee that an explosion will not occur. The improper use or lack of consistent and thorough washing of hood, duct, and blower can also cause explosions.

It is, therefore, imperative that persons using or contemplating the use of perchloric acid familiarize themselves with its use, characteristics, and the many explosions which have occurred.\*

#### Exhaust System

Because many extremely serious and some fatal injuries have occurred from the violent explosions which have taken place in maintenance work on exhaust systems from the reaction or reaction products of perchloric acid on organic materials, only hoods with smooth surfaces, specifically designed for perchloric acid, should be used for perchloric acid. The operating precautions (usually mounted on the manufacturer's

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\* Handbook of Laboratory Safety, Chemical Rubber Company, and AEC Safety Bulletins.

plate on side of hood) should be moved to a conspicuous location and followed closely. The exhaust duct should be a straight, vertical exhaust of approved materials.

Organic materials such as rubber, plastics, various sealing compounds, gaskets, paint, tape, etc., must not be used in any part of the exhaust system.

Use the hood water wash during fuming operations and wash down for 1 hour with both the hood wash and the duct water spray following each use.

### Chemistry Laboratory

Over the years many serious eye and facial injuries have occurred to chemists when flasks, containing an organic material and perchloric acid, exploded.

Several explosions reported as having occurred during the determination of potassium as the perchlorate are probably attributable to heating in the presence of concentrated perchloric acid and traces of alcohol.

A violent explosion took place in an exhaust duct from a laboratory hood in which perchloric acid solution was being fumed over a gas plate. It blew out windows, bulged the exterior walls, lifted the roof, and extensively damaged equipment and supplies. Some time prior to the explosion, the hood had been used for the analysis of miscellaneous materials.

Since perchloric acid is one of the more hazardous materials presently used in the Laboratory, the following basic precautions are listed for your guidance:

1. All procedures using perchloric acid are to be considered hazardous until established otherwise. Do not use organics in a perchloric acid hood at any time.
2. Maintain as small a perchloric acid stock as is practical and store in a noncombustible area away from other combustible materials and chemicals.
3. Perchloric acid reactions should be carried out in hoods specifically designed for that purpose. Commercially available perchloric acid fume hoods are equipped with wash-down or water spray features and are constructed of appropriate materials. For those fuming operations which are temporary in nature, invert a glass funnel, large enough to enclose the fuming apparatus, over the perchloric acid work; attach a *Teflon* hose and run the hose to a water aspirator which is connected to the sink faucet. During the fuming operation, turn the water on—the draft thus furnished will carry the fumes to the water stream where it is diluted and permitted to drain. After each operation, the *Teflon* hose must be flushed thoroughly.
4. Gas flames or oil baths used in heating reaction vessels shall not be used for perchloric acid reactions nor in perchloric acid hoods. Electric heating with controls located outside the hood should be used.
5. All spills should be flushed with quantities of water immediately.
6. Personnel protective equipment should consist of rubber gloves, rubber sleeves, rubber apron, and face shields. If possible the perchloric reactions should also be performed behind safety shields, or with the hood sash closed, to protect any possible passers-by.
7. Anhydrous perchloric acid will explode on contact with wood, paper, carbon, and other organics; and spontaneously, if stored for more than 10 days.

Note: Special hoods are not required for metallurgical sample preparation or for cold solutions of perchloric.