

### 3.4.11

#### AOAC Official Method 931.01 Phosphorus in Plants

Micro Method  
First Action 1931  
Final Action

##### A. Reagents

(a) *Phosphorus standard solution*.—0.025 mg P/mL. Dissolve 0.4394 g pure dry  $\text{KH}_2\text{PO}_4$  in  $\text{H}_2\text{O}$  and dilute to 1 L. Dilute 50 mL of this solution to 200 mL.

(b) *Ammonium molybdate solution*.—Dissolve 25 g ammonium molybdate in 300 mL  $\text{H}_2\text{O}$ . Dilute 75 mL  $\text{H}_2\text{SO}_4$  to 200 mL and add to ammonium molybdate solution.

(c) *Hydroquinone solution*.—Dissolve 0.5 g hydroquinone in 100 mL  $\text{H}_2\text{O}$ , and add one drop  $\text{H}_2\text{SO}_4$  to retard oxidation.

(d) *Sodium sulfite solution*.—Dissolve 200 g  $\text{Na}_2\text{SO}_3$  in  $\text{H}_2\text{O}$ , dilute to 1 L, and filter. Either keep this solution well stoppered or prepare fresh each time.

##### B. Preparation of Test Solution

To 1 or 2 g test portion in small porcelain crucible, add 1 mL  $\text{Mg}(\text{NO}_3)_2$  solution [dissolve 950 g P-free  $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  in  $\text{H}_2\text{O}$  and dilute to 1 L], and place on steam bath. After few minutes, cautiously add few drops HCl, taking care that gas evolution does not push solids or test solution over edge of crucible. Make 2 or 3 further additions of few drops HCl while test solution is on bath so

that as material approaches dryness it tends to char. If contents become too viscous for further drying on bath, complete drying on hot plate.

Cover crucible, transfer to cold furnace, and ignite 6 h at  $500^\circ\text{C}$ , or until even gray ash is obtained. (If necessary, cool crucible, dissolve ash in a little  $\text{H}_2\text{O}$  or alcohol–glycerol, evaporate to dryness, and return uncovered to furnace 4–5 h longer.) Cool, take up with HCl (1 + 4), and transfer to 100 mL beaker. Add 5 mL HCl and evaporate to dryness on steam bath to dehydrate  $\text{SiO}_2$ . Moisten residue with 2 mL HCl, add ca 50 mL  $\text{H}_2\text{O}$ , and heat few minutes on bath. Transfer to 100 mL volumetric flask, cool immediately, dilute to volume, mix, and filter, discarding first portion of filtrate.

##### C. Determination

To 5 mL aliquot filtrate in 10 mL volumetric flask, add 1 mL ammonium molybdate solution, rotate flask to mix, and let stand few seconds. Add 1 mL hydroquinone solution, again rotate flask, and add 1 mL  $\text{Na}_2\text{SO}_3$  solution. (Last 3 additions may be made with Mohr pipet.) Dilute to volume with  $\text{H}_2\text{O}$ , stopper flask with thumb or forefinger, and shake to mix thoroughly. Let stand 30 min, and measure *A* with spectrophotometer set at 650 nm. Report as percent P.

References: *JAOAC* **14**, 216(1931).

*J. Biol. Chem.* **59**, 255(1924).

CAS-7723-14-0 (phosphorus)

#### 4.8.14

### AOAC Official Method 965.17 Phosphorus in Animal Feed and Pet Food

#### Photometric Method

First Action 1965

Final Action 1966

(Not applicable to mineral–mix feeds. Dry ashing procedure is not applicable to feeds, pet foods, or mineral mixes containing monobasic calcium phosphate.)

#### A. Apparatus

*Spectrophotometer*.—Capable of isolating 400 nm band and accepting 15 mm diameter cells.

#### B. Reagents

(a) *Molybdovanadate reagent*.—Dissolve 40 g ammonium molybdate  $4\text{H}_2\text{O}$  in 400 mL hot  $\text{H}_2\text{O}$  and cool. Dissolve 2 g ammonium metavanadate in 250 mL hot  $\text{H}_2\text{O}$  and cool; add 250 mL 70%  $\text{HClO}_4$ . (*Caution*: See [Appendix B](#), safety notes on perchloric acid.) Gradually add molybdate solution to vanadate solution with stirring, and dilute to 2 L.

(b) *Phosphorus standard solutions*.—(1) *Stock solution*.—2 mg P/mL. Dissolve 8.788 g  $\text{KH}_2\text{PO}_4$  in  $\text{H}_2\text{O}$  and dilute to 1 L. (2) *Working solution*.—0.1 mg P/mL. Dilute 50 mL stock solution to 1 L.

#### C. Preparation of Standard Curve

Transfer aliquots of working standard solution containing 0.5, 0.8, 1.0, and 1.5 mg P to 100 mL volumetric flasks. Treat as in **D**, beginning “Add 20 mL molybdovanadate reagent, . . .”. Prepare standard curve by plotting mg P against %*T* on semilog paper.

#### D. Determination

Ash 2 g test portion, in 150 mL beaker, 4 h at 600°C. Cool, add 40 mL  $\text{HCl}$  (1 + 3) and several drops  $\text{HNO}_3$ , and bring to bp. Cool, transfer to 200 mL volumetric flask, and dilute to volume with  $\text{H}_2\text{O}$ . Filter, and place aliquot containing 0.5–1.5 mg P in 100 mL volumetric flask. Add 20 mL molybdovanadate reagent, dilute to volume with  $\text{H}_2\text{O}$ , and mix well. Let stand 10 min; then read %*T* at 400 nm against 0.5 mg standard set at 100% *T*. (Use 15 mm diameter cells.) Determine mg P from standard curve.

$$\text{P, \%} = \frac{\text{mg P in aliquot}}{\text{g test portion in aliquot}} \times 10$$

References: *JAOAC* **48**, 654(1965); **59**, 937(1976).

CAS-7723-14-0 (phosphorus)

Revised: March 1996

# Appendix B: Laboratory Safety

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## Introduction

This chapter is not intended to be an exhaustive treatise on laboratory safety. These precautionary notes serve only as a reminder of possible hazards involved in the use of particular operations or substances. Refer to recommended texts at end of chapter for fuller treatment of subject. Follow safety requirements of those in the country, region, or jurisdiction within which your organization is situated. Consult guidelines issued by professional associations and government agencies.

## Cautionary Statements

Nature and amount of each chemical and its prescribed use were criteria used in determining if cautionary statement for method was indicated. Safety hazard was considered to exist when nature, amount, and use of chemical or equipment specified in method appeared likely to produce any of the following:

(a) Concentration of vapors from flammable liquid exceeding 25% of lower flammability limit of that liquid described by National Fire Protection Association, Boston, MA, USA.

(b) Contact between analyst and amounts of material highly active physiologically or toxic to humans in excess of Threshold Limit Values published by American Conference of Governmental Industrial Hygienists, PO Box 1937, Cincinnati, OH 45201, USA.

(c) Contact between analyst and amounts of highly corrosive material sufficient to produce serious injury.

(d) Contact between analyst and radiations which could be harmful.

(e) Explosion or violent reaction.

(f) Injury to analyst by hazards in equipment or processes which are not readily detectable by analyst.

When in doubt about possible hazards not covered in this chapter, consult references at end of chapter or other sources of information such as hazard warnings on labels and manufacturers' data sheets.

## Potential Hazards of Equipment

### Refrigerators

Refrigerators should be explosion-proof or explosion-resistant when used for storage of ether and other highly volatile, flammable liquids. Ordinary refrigerators can be made explosion resistant by removal of light switch, receptacle, and associated wiring, and by placing thermoregulation controls outside of refrigerator.

### Glass

Dispose of chipped or broken glassware in special containers; minor chips may be fire-polished and glassware retained. If glassware is to be repaired, mark defective area plainly and store in special location until repairs are completed.

Use heat-resistant glassware for preparation of solutions that generate heat (e.g., not bottles or graduated cylinders).

### Fire Extinguishers

Class B and C dry chemical fire extinguishers (for flammable liquid and electric fires) should be conveniently available to each laboratory room. Carbon dioxide fire extinguishers should be used on fires in electronic equipment.

Become familiar with the location of all fire extinguishers and the appropriate methods for their effective use.

### Blenders

Motor on high-speed blenders used to mix flammable solvent with other materials should be explosion-proof. Blend toxic or flammable liquids in effective fume-removal device.

### Centrifuges

Adjust all tubes to equal weight before loading them into centrifuge. Make certain that stoppers of tubes placed in pivot-type head will clear center when tubes swing to horizontal. Do not open centrifuge cover until machine stops completely. Before removing tubes, turn electric switch to "off." Do not rely on zero-set rheostat. Use only tubes specially designed for centrifuging. Do not exceed safe speed for various tube materials (glass, cellulose nitrate, polyethylene, etc.) recommended by tube manufacturer. Cellulose nitrate tubes may explode if autoclaved. Heating cellulose nitrate tubes to 60°C may cause them to produce harmful nitrogen oxide fumes.

### Atomic Absorption Spectrophotometer

Follow all manufacturer's instructions for installation, operation, safety, and maintenance. Use only hose/tubing to conduct gases that are approved by manufacturer and supplier. Use effective fume removal device to remove gaseous effluents from burner. Use only C<sub>2</sub>H<sub>2</sub> which is dissolved in solvent recommended by manufacturer. Open C<sub>2</sub>H<sub>2</sub> tank stem valve only a quarter turn. Change tank when C<sub>2</sub>H<sub>2</sub> pressure shows 75–100 lb. If instrument has a drain trap, ensure that it is filled with H<sub>2</sub>O before igniting burner. Following repair to C<sub>2</sub>H<sub>2</sub> supply line, check for gas tightness at all connections with soap solution or combustible gas detection system. When aspirating solutions containing high concentrations of Cu, Ag, or Hg, spray chamber should be rinsed with 50–100 mL H<sub>2</sub>O before shutting down to clean these metals from chamber. *See* safety notes on compressed gas cylinders.

### Flame Photometer

Use effective fume removal device to remove gaseous burner effluents.

### Photofluorometer

Considerable amounts of O<sub>3</sub> are formed by UV light radiated by quartz lamp. Ozone is toxic even in low concentrations; remove through effective fume removal device placed near quartz lamp.

### Monitoring Equipment

Monitor unattended operations with equipment that will automatically shut down process if unsafe condition develops.

Reference: Furr, A.K. (1995) "CRC Handbook of Laboratory Safety," CRC Press LLC Headquarters, 2000 NW Corporate Blvd, Boca Raton, FL 33431, USA.

### **Compressed Gas Cylinders**

Identify by name(s) contents of compressed gas cylinders on attached decal, stencil, or tag, instead of by color codes. Move cylinders (with protective cap) upright, secured to cart. Secure cylinders in upright position by means of strap, chain, or nontip base. Let contents of C<sub>2</sub>H<sub>2</sub> cylinders settle and let all cylinders come to room temperature prior to opening. Use only correct pressure gages, pressure regulator, flow regulator, and hose/tubing for each size of gas cylinder and type of gas as specified by supplier. Use soap solution or combustible gas detection system to check all connections, especially when system is pressurized and gas is not flowing, to check for slow leak. Use special heater on N<sub>2</sub>O gas line. Close gas tank valve and diaphragm on regulator when gas is not in use. Service regulator at least yearly. Use toxic gases only in effective fume removal device. When burning gas, use flashback prevention device in gas line on output side of regulator to prevent flame being sucked into cylinder.

Reference: "Handbook of Compressed Gases" (1981)  
Compressed Gas Association, Van Nostrand  
Reinhold Co., New York, NY, USA.

### **Distillation, Extraction, and Evaporations**

(a) *Flammable liquids*.—Perform operations behind safety barrier with hot H<sub>2</sub>O, steam, or electric mantle heating. Use effective fume removal device to remove flammable vapors as produced. Set up apparatus on firm supports and secure all connections. Leave ample headroom in flask and add boiling chips *before* heating is begun. All controls, unless vapor sealed, should be located outside vapor area.

(b) *Toxic liquids*.—Use effective fume removal device to remove toxic vapors as produced. Avoid contact with skin. Set up apparatus on firm supports and secure all connections.

### **Electrical Equipment**

Accidents involving electric equipment may result in *mechanical injury*, e.g., fingers being caught in chopping mill knives; *electric shock*, which may be due to lack of or improper grounding, defective equipment, exposed wiring, or inadequate maintenance; and *fire* through ignition of flammable vapors by electrically produced spark. Ground all electric equipment to avoid accidental shock. Installation, maintenance, and repair operations should be performed by qualified electricians.

### **Parr Bomb**

Follow manufacturer's directions closely to avoid explosion.

### **Pressure**

Do not conduct pressure operations with standard glassware. In certain circumstances, glassware specifically designed to withstand pressure may be used. Observe manufacturer's recommended safeguards when using pressure apparatus such as calorimeter bomb, hydrogenator, etc.

### **Vacuum**

Tape or shield with safety barrier containers and apparatus to be used under vacuum to minimize effects of possible implosion. Vacuum pump drive belts must have effective guards.

### **Hazardous Radiations**

UV radiation is encountered in AA spectrophotometry, fluorometry, UV spectrophotometry, germicidal lamps, and both long- and shortwave UV lamps used to monitor chromatographic separations. Never expose unprotected eyes to UV light from any source either direct or reflected (e.g., flames in flame photometer, lamps, electric arcs, etc.). Always wear appropriate eye protection such as goggles with uranium oxide lenses, welder's goggles, etc., when such radiations are present and unshielded. Keep skin exposure to UV radiations to minimum.

## **Safety Techniques and Practices**

### **Spraying Chromatograms**

When strong corrosive and toxic reagents are sprayed on chromatograms, use gloves, face shield, respiratory protection, and appropriate fume removal device to protect skin, eyes, and respiratory tract against mists or fumes generated by spraying device.

### **Pipets**

Do not pipet any liquids by mouth. Use pipet fillers or rubber tubing connected through trap to vacuum line for this purpose.

### **Wet Oxidation**

This technique is among the most hazardous uses of acids but can be performed safely. Observe precautions in this chapter for particular acids used and rigorously follow directions given in specific method.

### **Hazardous or After Hours Work**

Anyone working alone after hours or on hazardous procedures should arrange to be contacted periodically as a safety measure.

### **Glass Tubing**

Protect hands with heavy towel or gloves such as those made of Kevlar when inserting glass tubing into cork or rubber stopper. Fire polish all raw glass cuts.

Open ampules in fume removal device over tray large enough to hold contents if ampule should break. If contents are volatile, cool before opening.

## **Safe Handling of Concentrated Corrosive Acids**

Use effective *acid-resistant* fume removal device whenever heating acids or performing reactions which liberate acid fumes. In diluting, always add acid to H<sub>2</sub>O unless otherwise directed in method. Keep acids off skin and protect eyes from spattering. If acids are spilled on skin, wash immediately with large amounts of H<sub>2</sub>O.

### **Acetic Acid and Acetic Anhydride**

Reacts vigorously or explosively with strong oxidizers. Wear face shield and heavy rubber gloves when using.

CAS-108-24-7 (acetic anhydride)

CAS-64-19-7 (acetic acid)

### **Chromic and Perchromic Acids**

Can react explosively with acetic anhydride, acetic acid, ethyl acetate, isoamyl alcohol, and benzaldehyde. Less hazardous with ethylene glycol, furfural, glycerol, and methanol. Conduct reactions behind safety barrier. Wear face shield and heavy rubber gloves.

CAS-1333-82-0 (chromic acid)

#### **Formic and Performic Acids**

Strong reducing agents; react vigorously or explosively with oxidizing agents. Irritating to skin, forming blisters. Performic acid (formyl hydroperoxide) has detonated for no apparent reason while being poured. Wear face shield and heavy rubber gloves when using.

CAS-64-18-6 (formic acid)

#### **Fuming Acids**

Prepare and use with effective fume removal device. Wear acid-resistant gloves and eye protection.

#### **Hydrofluoric Acid**

Very hazardous with  $\text{NH}_3$ . Extremely corrosive to all tissues. It can cause painful sores on skin and can cause decalcification of bone. Use any commercial hydrofluoric acid spill control kit. Wear goggles and acid-resistant gloves. Keep a kit containing calcium gluconate cream for use in case of skin contact and seek medical attention immediately.

CAS-7664-39-3 (hydrofluoric acid)

#### **Nitric Acid**

Reacts vigorously or explosively with aniline,  $\text{H}_2\text{S}$ , flammable solvents, hydrazine, and metal powders (especially Zn, Al, and Mg). Gaseous nitrogen oxides from  $\text{HNO}_3$  can cause severe lung damage. Copious fumes are evolved when concentrated  $\text{HNO}_3$  and concentrated HCl are mixed. Avoid premixing. Use effective fume removal device when fumes are generated. Handle with disposable polyvinyl chloride, not rubber, gloves.

CAS-7697-37-2 (nitric acid)

#### **Oxalic Acid**

Forms explosive compound with Ag and Hg. Oxalates are toxic. Avoid skin contact and ingestion.

CAS-144-62-7 (oxalic acid)

#### **Perchloric Acid**

Contact with oxidizable or combustible materials or with dehydrating or reducing agents may result in fire or explosion. Persons using this acid should be thoroughly familiar with its hazards. Safety practices should include the following:

(a) Remove spilled  $\text{HClO}_4$  by immediate and thorough washing with large amounts of  $\text{H}_2\text{O}$ .

(b) Hoods, ducts, and other devices for removing  $\text{HClO}_4$  vapor should be made of chemically inert materials and so designed that they can be thoroughly washed with  $\text{H}_2\text{O}$ . Exhaust systems should discharge in safe location and fan should be accessible for cleaning.

(c) Avoid use of organic chemicals in hoods or other fume removal devices used for  $\text{HClO}_4$  digestions.

(d) Use goggles, barrier shields, and other devices as necessary for personal protection; use polyvinyl chloride, not rubber, gloves.

(e) In wet combustions with  $\text{HClO}_4$ , treat test portion first with  $\text{HNO}_3$  to destroy easily oxidizable organic matter unless otherwise specified. *Do not evaporate to dryness.*

(f) Contact of  $\text{HClO}_4$  solution with strong dehydrating agents, such as  $\text{P}_2\text{O}_5$  or concentrated  $\text{H}_2\text{SO}_4$ , may result in formation of anhydrous  $\text{HClO}_4$  which reacts explosively with organic matter and with reducing agents. Exercise special care in performing analyses requiring use of  $\text{HClO}_4$  with such agents. Extremely sensitive to shock and heat when concentration is 72%.

(g) Also observe precautions outlined in:

- References: (1) "Perchloric Acid Solution," Chemical Safety Data Sheet SD-11 (1999) Manufacturing Chemists Association of the United States, 1825 Connecticut Ave, NW, Washington, DC 20009, USA.  
(2) "Applied Inorganic Analysis," Hillebrand, W.F., Lundell, G.E.F., Bright, H.A., & Hoffman, J.I. (1953) 2nd Ed., John Wiley & Sons, Inc., New York, NY 10158-0012, USA, pp 39–40.  
(3) "Notes on Perchloric Acid and Its Handling in Analytical Work" (1959) *Analyst* **84**, 214–216.  
(4) "Perchlorates" (1960) ACS Monograph No. 146, J.C. Schumacher (Ed.), Reinhold, American Chemical Society, 1155 16th St, NW, Washington, DC 20036, USA.

*See also* references at end of this chapter.

CAS-7601-90-3 (perchloric acid)

#### **Picric Acid**

Highly sensitive to shock when in dry state. In contact with metals and  $\text{NH}_3$ , it produces picrates which are more sensitive to shock than picric acid. Readily absorbed through skin and irritating to eyes. Wear heavy rubber gloves and eye protection.

CAS-88-89-1 (picric acid)

#### **Sulfuric Acid**

Always add  $\text{H}_2\text{SO}_4$  to  $\text{H}_2\text{O}$ . Wear face shield and heavy rubber gloves to protect against splashes. Do not mix with HCl.

CAS-7664-93-9 (sulfuric acid)

### **Safe Handling of Alkalies**

Alkalies can burn skin, eyes, and respiratory tract severely. Wear heavy rubber gloves and face shield to protect against concentrated alkali liquids. Use effective fume removal device or gas mask to protect respiratory tract against alkali dusts or vapors.

#### **Ammonia**

Extremely caustic liquid and gas. Wear skin, eye, and respiratory protection when handling in anhydrous liquid or gaseous state.  $\text{NH}_3$  vapors are flammable. Reacts violently with strong oxidizing agents, halogens, and strong acids.

CAS-7664-41-7 (ammonia)

#### **Ammonium Hydroxide**

Caustic liquid. Forms explosive compounds with many heavy metals such as Ag, Pb, Zn, and their salts, especially halide salts.

CAS-1336-21-6 (ammonium hydroxide)

#### **Calcium Oxide (Burnt Lime)**

Strongly caustic! Reacts violently with  $\text{H}_2\text{O}$ . Protect skin, eyes, and respiratory tract against contact with dust.

CAS-1305-78-8 (calcium oxide)

#### **Sodium Biphenyl, Sodium Methylate, and Sodium Ethylate**

Less caustic than NaOH, but can be injurious. Reacts vigorously with  $\text{H}_2\text{O}$ . Protect skin and eyes when handling.

CAS-132-27-4 (sodium biphenyl)  
CAS-124-41-4 (sodium methylete)  
CAS-141-52-6 (sodium ethylete)

#### **Sodium Peroxide**

Less caustic than sodium and potassium hydroxides but reacts violently with H<sub>2</sub>O, organic matter, charcoal, glycerol, diethyl ether, or P. Wear skin, eye, and respiratory protection when handling multigram amounts.

CAS-1313-60-6 (sodium peroxide)

#### **Sodium and Potassium Hydroxides**

Extremely caustic. Can cause severe burns. Protect skin and eyes when working with these alkalis as solids or concentrated solutions. Add pellets to H<sub>2</sub>O, not vice versa.

CAS-1310-58-3 (potassium hydroxide)

CAS-1310-73-2 (sodium hydroxide)

#### **Sodium, Potassium, Lithium, and Calcium Metals**

Violently reactive with H<sub>2</sub>O or moisture, CO<sub>2</sub>, halogens, strong acids, and chlorinated hydrocarbons. Emits corrosive fumes when burned. Can cause severe burns. Wear skin and eye protection when handling. Use only dry alcohol when preparing sodium alcoholate and add metal directly to alcohol, one small piece at a time. Avoid adding metallic Na to reaction through condenser.

CAS-7440-23-5 (sodium)

### **Safe Handling of Microorganisms**

The following precautions should be taken when working with microorganisms:

- (a) Never pipet any liquid by mouth.
- (b) Wear lab coats or other appropriate protective clothing when working with live microorganisms or media containing hazardous ingredients such as dyes.
- (c) Pregnant women should know and understand the risk involved when working with microorganisms that are potentially abortogenic, such as *Listeria*.
- (d) It is critical to thoroughly wash your hands with a disinfectant hand-washing liquid soap after performing microbiological work.
- (e) Contaminated liquid or solid wastes should be sterilized, preferably by autoclave.
- (f) It is also important that standard laboratory technique be used when working with these hazards.

References: (1) "Laboratory Biosafety Manual" (1983) World Health Organization, WHO Publications Center USA, 49 Sheridan Ave, Albany, NY 12210, USA.  
(2) "Biohazards Reference Manual" (1985) AIHA Biohazards Committee, American Industrial Hygiene Association, 2700 Prosperity Ave, Suite 250, Fairfax, VA 22031, USA.  
(3) "Biosafety in Microbiological and Biomedical Laboratories" (1988) 2nd Ed., Centers for Disease Control and National Institutes of Health, HHS No. (NIH) 88-8395, Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402, USA.  
(4) "EPA Guide for Infectious Waste Management" (1986) Stock No. PB86-199130, National Technical Information Service, Springfield, VA 22161, USA.

### **Safe Handling of Organic Solvents**

(Do not mix waste solvents.)

#### **Flammable Solvents**

Do not let vapors concentrate to flammable level in work area. It is nearly impossible to eliminate all chance of sparks from static electricity even if electric equipment is grounded. Use effective fume removal device to remove these vapors when released. Metal containers should be grounded using a grounding wire or strap when flammable liquids are poured from them.

#### **Toxic Solvents**

Vapors from some volatile solvents are highly toxic. Several of these solvents are readily absorbed through skin. Use effective fume removal device to remove vapors of these solvents as they are liberated.

See references at end of this chapter.

### **Safe Handling of Special Chemical Hazards**

(a) Do all laboratory sampling, mixing, weighing, etc., under effective fume removal device in area having good forced ventilation of nonrecirculated air, or wear gas mask of proper type. If mask is used, replace cartridges as recommended, since using contaminated mask may be worse than no mask.

(b) Keep off skin. Wear clean protective clothing and nonpermeable gloves (such as polyethylene gloves) as necessary. Wash hands thoroughly with soap and water to avoid contaminating food and smoking materials.

(c) Label all containers with name and approximate content of all pesticides.

(d) Have readily available and study information on symptoms of poisoning and first aid treatment for each type of pesticide being handled.

(e) Consult "Recognition and Management of Pesticide Poisonings" (see reference 19.)

(f) Follow your organization's procedures when disposing of waste pesticides. The manufacturer can be contacted for advice on disposal problems.

(g) Do not enter pesticide *residue* or other laboratories after handling pesticide formulations until protective clothing and gloves have been removed and face and hands have been thoroughly washed with soap and water.

The U.S. Environmental Protection Agency operates a "hotline," called National Pesticide Telecommunications Network (NPTN), staffed to handle pesticide questions. To reach this hotline, dial +1-800-858-7378.

The AOAC Safety Committee recommends the use of nonchlorinated solvents in all methods. Where possible, a study should be conducted to find a substitute solvent for the method.

#### **Acetone**

Highly flammable. Forms explosive peroxides with oxidizing agents. Use effective fume removal device.

CAS-67-64-1 (acetone)

#### **Acetonitrile**

Toxic. Avoid contact with skin and eyes. Use effective fume removal device.

CAS-75-05-8 (acetonitrile)

**Ammoniacal Silver Nitrate**

Use soon after preparation and do not allow to stand for long periods of time.

CAS-1136-21-6 (ammonium hydroxide)  
CAS-7761-88-8 (silver nitrate)

**Aniline**

Toxic. Avoid contact with skin and eyes. Use effective fume removal device. Highly toxic when heated to decomposition. Flammable. May react vigorously with oxidizing agents. Ignites in presence of fuming  $\text{HNO}_3$ . May react violently with  $\text{O}_3$ .

CAS-62-53-3 (aniline)

**Arsenic Trioxide**

Toxic. Forms toxic volatile halides in contact with halide acids. Forms volatile, highly toxic arsine when reduced in acid solution. Protect skin and respiratory tract when handling. Use effective fume removal device when arsine or arsenic trihalide is formed.

CAS-1327-53-3 (arsenic trioxide)

**Asbestos**

Dry asbestos fibers are hazardous when inhaled. Wet fibers form a mat which does not constitute a hazard. Transfer dry fibers in hood to container of distilled  $\text{H}_2\text{O}$  and store under  $\text{H}_2\text{O}$  until needed, e.g., for preparation of mats in Gooch crucibles. Do not dry asbestos in forced draft oven, only in convection oven. Open oven doors slowly to avoid developing convection currents that will make fibers airborne. Reuse of filtering mats is often possible by washing, drying, and ignition, as appropriate. Use alternative if available.

CAS-8012-01-9 (asbestos)

**Benzene**

Toxic. Highly flammable. Avoid contact with skin. Do not breathe vapors. Use effective fume removal device. Decomposes violently in presence of strong oxidizing agents. Reacts violently with  $\text{Cl}_2$ . Considered to be carcinogenic. Use alternative if available.

CAS-71-43-0 (benzene)

**Bromine and Chlorine**

Hazardous with  $\text{NH}_3$ , H, petroleum gases, turpentine, benzene, and metal powders. Extremely corrosive. Use effective fume removal device. Protect skin against exposure.

CAS-7726-96-6 (bromine)  
CAS-7782-50-5 (chlorine)

**Carbon Disulfide**

Extremely flammable with low ignition temperature. Toxic. Use effective fume removal device. Can react vigorously to violently with strong oxidizing agents, azides, and Zn. Avoid static electricity.

CAS-75-15-0 (carbon disulfide)

**Carbon Tetrachloride**

Reacts violently with alkali metals. Toxic. Fumes may decompose to phosgene when heated strongly. Use effective fume removal device.

CAS-56-23-5 (carbon tetrachloride)

**Carcinogens**

Regulations of U.S. Department of Labor require special precautions to avoid exposure of persons to carcinogenic chemicals.

Consult 29CFR1910.93c (U.S. Government Printing Office, Washington, DC 20402, USA) and "Guidelines for the Laboratory Use of Chemical Substances Posing a Potential Occupational Carcinogenic Risk," USDHEW (1978).

**Chloroform**

Can be harmful if inhaled. Forms phosgene when heated to decomposition. Use effective fume removal device. Can react explosively with Al, Li, Mg, Na, K, disilane,  $\text{N}_2\text{O}_4$ , and NaOH plus methanol. Considered to be tumor producing agent.

CAS-67-66-3 (chloroform)

**Cyanides**

Reacts with acids to form highly toxic and rapid acting HCN gas. Use only in effective fume removal device. Destroy residues with alkaline NaOCl solution.

CAS-57-12-5 (cyanide gas)

CAS-143-33-9 (NaCN)

**Cyclohexane**

Highly flammable. Use effective fume removal device. Can react vigorously with strong oxidizing agents.

CAS-110-82-7 (cyclohexane)

**Di- and Trichloroacetic and Trifluoroacetic Acids**

Can cause severe burns to skin and respiratory tract. Use rubber gloves, eye protection, and effective fume removal device to remove vapors generated.

CAS-79-43-6 (dichloroacetic acid)  
CAS-76-03-9 (trichloroacetic acid)  
CAS-76-05-1 (trifluoroacetic acid)

**Di- and Triethylamine**

Flammable. Toxic. Corrosive to skin and eyes. Use effective fume removal device. Can react vigorously with oxidizing materials.

CAS-109-89-7 (diethylamine)  
CAS-121-44-8 (triethylamine)

**Dichloromethane**

Use effective fume removal device. Fumes may decompose to phosgene when heated strongly. Can react vigorously with oxidizing materials and alkali metals. Considered to be carcinogenic.

CAS-75-09-2 (dichloromethane)

**Diethyl Ether**

Store protected from light. Extremely flammable. Unstable peroxides can form upon long standing or exposure to sunlight in bottles. Can react explosively when in contact with  $\text{Cl}_2$ ,  $\text{O}_3$ ,  $\text{LiAlH}_4$ , or strong oxidizing agents. Use effective fume removal device. Avoid static electricity. *See also* safety notes on peroxides.

CAS-60-29-7 (ether)

**Dimethylformamide**

Toxic. Flammable. Avoid contact with skin and eyes. Use effective fume removal device. Can react vigorously with oxidizing agents, halogenated hydrocarbons, and inorganic nitrates.

CAS-68-12-2 (dimethylformamide)

### **Enzyme Preparations**

Highly concentrated enzyme preparations can cause allergic reactions after contact with enzyme dust or aerosol. Take the following minimum precautions at all times:

(a) Do all laboratory sampling, mixing, weighing, etc. under effective fume removal in an area having good forced ventilation of nonrecirculated air.

(b) Avoid contact with skin. Wear clean protective clothing and nonpermeable gloves as necessary.

(c) Label all sample containers with the warning: *Caution, highly concentrated enzyme preparation. Avoid inhalation of dust and prolonged contact with unprotected skin.*

### **Ethanol**

Flammable. Use effective fume removal device when heating or evaporating.

CAS-64-17-5 (ethanol)

### **Ethyl Acetate**

Flammable, especially when being evaporated. Irritating to eyes and respiratory tract. Use effective fume removal device.

CAS-141-78-6 (ethyl acetate)

### **Formaldehyde**

A suspected human carcinogen. Exposure to high concentrations may cause skin irritation and inflammation of mucous membranes, eyes, and respiratory tract. Use skin protection and effective fume removal device.

CAS-50-00-0 (formaldehyde)

### **Hexane**

Highly flammable. Use effective fume removal device.

CAS-110-54-3 (hexane)

### **Hydrogen Sulfide**

Hazardous with oxidizing gases, fuming HNO<sub>3</sub>, and Na<sub>2</sub>O<sub>2</sub>. Forms explosive mixtures with air. Toxic. Use effective fume removal device.

CAS-7783-06-4 (hydrogen sulfide)

### **Hypophosphorus Acid**

Reacts violently with oxidizing agents. On decomposition, emits highly toxic fumes (phosphine) and may explode. Use effective fume removal device.

CAS-6303-21-5 (hypophosphorus acid)

### **Isooctane**

Highly flammable. Use effective fume removal device.

CAS-26635-64-3 (isooctane)

### **Magnesium**

When finely divided, liberates H in contact with H<sub>2</sub>O. Burns in air when exposed to flame. Can be explosive in contact with CHCl<sub>3</sub> or CH<sub>3</sub>Cl.

CAS-7439-95-4 (magnesium)

### **Magnesium Perchlorate**

Explodes on contact with acids and reducing materials. Use as drying agent on inorganic gases and materials only.

CAS-10034-81-8 (magnesium perchlorate)

### **Mercury**

Hazardous in contact with NH<sub>3</sub>, halogens, and alkali. Vapors are extremely toxic and cumulative. Regard spills on heated surfaces as extremely hazardous and clean up promptly. High degree of personal cleanliness is necessary for persons who use Hg. Handle only in locations where any spill can be readily and thoroughly cleaned up. Powdered S sprinkled over spilled Hg can assist in cleaning up spills. When Hg evaporation is necessary, use effective fume removal device.

To avoid environmental contamination, dilute liquid remaining in Kjeldahl distillation flask to ca 300 mL with H<sub>2</sub>O, cool to room temperature, and add 50 mL 30% H<sub>2</sub>O<sub>2</sub>. (If Raney powder method is used, 6 mL is enough.) Warm gently to initiate reaction, let reaction go to completion in warm flask, and separate precipitated HgS. Reserve precipitate in closed labeled container for recovery of Hg or disposal appropriate for Hg.

*See also* safety notes on mercury salts.

CAS-74-39-97-6 (mercury)

### **Mercury Salts**

Mercuric salts are highly toxic and mostly H<sub>2</sub>O-soluble. Use skin and respiratory protection when dry mercuric salts are to be used. Use skin protection when concentrated aqueous solutions of mercuric salts are used. Mercurous salts are generally less toxic than mercuric salts. Use of personal protection is advisable when handling these salts and their concentrated solutions.

*See also* safety notes on mercury.

### **Methanol**

Flammable. Toxic. Avoid contact with eyes. Avoid breathing vapors. Use effective fume removal device. Can react vigorously with NaOH plus CHCl<sub>3</sub>, and KOH plus CHCl<sub>3</sub> or KOH plus HClO<sub>4</sub>.

CAS-67-56-1 (methanol)

### **Methyl Cellosolve**

Vapors can be harmful. Use effective fume removal device.

CAS-109-86-4 (methyl cellosolve)

### **Natural Toxins**

Mycotoxins should be handled as very toxic substances. Perform manipulations under hood whenever possible, and take particular precautions, such as use of glove box, when toxins are in dry form because of electrostatic nature and resulting tendency to disperse in working areas. Swab accidental spills of toxin with 1% NaOCl bleach, leave 10 min, and then add 5% aqueous acetone. Rinse all glassware exposed to aflatoxins with methanol, add 1% NaOCl solution, and after 2 h add acetone to 5% of total volume. Let react 30 min and then wash thoroughly. *See* Chapter 49, Natural Toxins, for additional references and for more details on decontamination.

### **Nitrobenzene and Other Nitroaromatics**

Readily absorbed through skin. Symptoms of intoxication can include euphoria and bluish tint of tongue, lips, and fingernails. Wear resistant rubber gloves when handling. Heat or evaporate in effective fume removal device.

CAS-98-95-3 (nitrobenzene)

### **Oxidizers**

(Perchlorates, peroxides, permanganates, persulfates, perborates, nitrates, chlorates, chlorites, bromates, iodates, concentrated H<sub>2</sub>SO<sub>4</sub>, concentrated HNO<sub>3</sub>, CrO<sub>3</sub>.)

Can react violently with most metal powders,  $\text{NH}_3$ , ammonium salts, P, many finely divided organic compounds, flammable liquids, acids, and S. Use exactly as specified in method. Handle in effective fume removal device from behind explosion-resistant barrier. Use face shield.

#### **Pentane**

Extremely flammable. Use effective fume removal device. Avoid static electricity.

CAS-109-66-0 (pentane)

#### **Permanganates**

Moderately toxic. Readily soluble in  $\text{H}_2\text{O}$ . Strong oxidizing agent. May form explosive mixture with  $\text{H}_2\text{SO}_4$  or  $\text{HClO}_4$ . When using with strong acids to destroy organic matter, perform reaction behind safety barrier.

CAS-7722-64-7 (potassium permanganate)

#### **Peroxides**

(a) *Hydrogen peroxide*.—30% strength is hazardous; can cause severe burns. Drying  $\text{H}_2\text{O}_2$  on organic material such as paper or cloth can lead to spontaneous combustion. Cu, Fe, Cr, other metals, and their salts cause rapid catalytic decomposition of  $\text{H}_2\text{O}_2$ . Hazardous with flammable liquids, aniline, and nitrobenzene. Since it slowly decomposes with evolution of  $\text{O}_2$ , provide stored  $\text{H}_2\text{O}_2$  with vent caps. Wear gloves and eye protection when handling.

(b) *Ether peroxides*.—These peroxides form in diethyl ether, dioxane, and other ethers during storage. They are explosive and must be destroyed chemically before distillation or evaporation. Exposure to light influences peroxide formation in ethers. Filtration through activated alumina is reported to be effective in removing peroxides. Store over sodium ribbon to retard peroxide formation.

CAS-7722-84-1 (hydrogen peroxide)

#### **Pesticides**

Many pesticide chemicals are extremely toxic by various routes of exposure, especially in concentrated form. These chemicals include organic Cl, carbamate, and organic P insecticides, mercurials, arsenicals, and nicotine. As an example, organic P family of pesticides is consistently highly toxic, not only by oral ingestion, but dermally and by inhalation as well. Observe following minimum precautions at all times. Consult safety data sheets or labels for additional information.

#### **Petroleum Ether**

Extremely flammable. Use effective fume removal device. Avoid static electricity.

CAS-8030-30-6 (petroleum ether)

#### **Phosphotungstic Acid**

Emits highly toxic fumes when heated to decomposition or in strong alkali.

CAS-12067-99-1 (phosphotungstic acid)

#### **Pyridine**

Toxic. Flammable. Use effective fume removal device. Releases toxic cyanides when heated to decomposition.

CAS-110-86-1 (pyridine)

#### **Radioactive Chemicals**

Consult NIST Handbook No. 92, "Safe Handling of Radioactive Materials" (available as NCRP Report No. 30 from National Council on Radiation Protection, Publications Department, 4201 Connecticut Ave, NW, Washington, DC 20008, USA) and NCRP Report No. 39, "Basic Radiation Protection Criteria," before handling these materials.

#### **Silver Iodate**

Powerful oxidizing agent. Can initiate combustion in contact with organic material (e.g., paper or cloth). Can react vigorously with reducing agents. Handle as noted for oxidizers.

CAS-7783-97-3 (silver iodate)

#### **Silver Nitrate**

Powerful oxidizing agent; strongly corrosive. Dust or solid form is hazardous to eyes. Handle as noted for oxidizers.

CAS-7761-88-8 (silver nitrate)

#### **Sulfur Dioxide**

Toxic gas. Forms  $\text{H}_2\text{SO}_3$  in contact with moisture. Use effective fume removal device to remove  $\text{SO}_2$  vapors released by reaction or from gas cylinder. Avoid contact with skin, eyes, and respiratory tract.

CAS-7446-09-5 (sulfur dioxide)

#### **Toxic Dusts**

Use gloves and goggles to avoid contact with skin and eyes. Use effective fume removal device or other respiratory protection.

#### **Uranyl Acetate**

Highly toxic. Avoid skin contact and breathing dusts.

CAS-541-09-3 (uranyl acetate)

### **Special References**

#### **Chemical Safety**

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- (3) "Prudent Practices in the Laboratory: Handling and Disposal of Chemicals" (1995) National Research Council, National Academy Press, Washington, DC 20005, USA.
- (4) "Flammable and Combustible Liquids Code Handbook" (1984) 2nd Ed., National Fire Protection Association, Boston, MA 02110, USA.
- (5) "Chemical Protective Clothing, Volumes 1 and 2" (1990) J.S. Johnson & K.J. Anderson (Eds.), American Industrial Hygienists Association, Fairfax, VA 22031, USA.
- (6) "Health and Safety for Toxicity Testing" (1984) D.B. Walters & C.W. Jameson, Butterworth Publishers, Stoneham, MA, USA.
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- (9) "Safe Practices in Chemical Laboratories" (1989) Royal Society of Chemistry, Springer Verlag New York, Inc., Secaucus, NJ 07096-2485, USA.
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- (13) "Onderzoek Kwaliteit Zuurkasten" (1985) G. Bolkesteijn, Technical University Twente, Enschede, The Netherlands (printed in Dutch).
- (14) "Handboek Bedrijfsveiligheid" (1986) 4 Volumes, Kluwer, Deventer, The Netherlands.
- (15) "La Securite dans les Laboratoires Utilisant des Substances Chimiques: Guide Practique" (1983) Centre National de Prevention et de Protection (CPP), Paris, France, p. 305 (printed in French).
- (16) "Zuurkasten In Laboratoria: Hoe zijn ze, Hoe blijven ze en hoe moeten we er mee werken" (1982) A.G. Kroes, Cursus Hogere Veiligheidskunde, Royal Shell Laboratory Safety, Environment and Security, Amsterdam, The Netherlands, 68 pp (printed in Dutch).
- (17) Gosselin, Smith, & Hodge (1984) "Clinical Toxicology of Commercial Products (Home and Farm)," 5th Ed., The Williams and Wilkens Co., Baltimore, MD 21202, USA.
- (18) "Farm Chemicals Handbook 2000" (1999) Meister Publishing Co., 37733 Euclid Ave, Willoughby, OH 44094, USA.
- (19) "Recognition and Management of Pesticide Poisonings," (1998) 5th Ed., U.S. Environmental Protection Agency, National Service Center for Environmental Publications, Cincinnati, OH 45242-2419, USA.
- (20) "Citizens Guide to Pest Control and Pesticide Safety" (1995) U.S. Environmental Protection Agency, National Service Center for Environmental Publications, Cincinnati, OH 45242-2419, USA.

#### **Laboratory Safety**

- (1) "CRC Handbook of Laboratory Safety" (1995) Furr, A.K. (Ed.), CRC Press LLC Headquarters, 2000 NW Corporate Blvd, Boca Raton, FL 33431, USA.
- (2) "Handbook of Laboratory Waste Disposal" (1985) M.J. Pitt & E. Pitt (Eds), Ellis Horwood Ltd., Halstead Press—John Wiley & Sons, New York, NY 10158-0012, USA.
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- (4) "Radionucleotiden-Laboratoria; Richtlijnen voor Inrichting van en Werken in Radio-Nucleotiden-Laboratoria" (1983) Ministerie van Volkshuisvesting, Ruimtelijke Ordening, en Milieubeheer, Publication 83-02, The Hague, The Netherlands, 44 pp (printed in Dutch).

#### **Toxic Solvents**

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