4.5.01

AOAC Official Method 920.39
Fat (Crude) or Ether Extract in Animal Feed

First Action 1920
Final Action

Use method **A** or **C** for feed ingredients and mixed feeds other than (*1*) baked and/or expanded, (*2*) dried milk products, (*3*) containing urea, or (*4*) mixed feeds that have at least 20% of crude fat derived from baked and/or expanded, or dried milk products.

A. Indirect Method

Determine moisture as in 934.01 (*see* 4.1.03) or 920.36 (*see* 4.1.05); then extract dried substance as in **C**, and dry again. Report loss in weight as ether extract.

Direct Method

B. Reagent

Anhydrous ether.—Wash commercial ether with 2 or 3 portions H_2O, add solid NaOH or KOH, and let stand until most of H_2O is abstracted from the ether. Decant into dry bottle, add small pieces of carefully cleaned metallic Na, and let stand until H_2 evolution ceases. Keep ether, thus dehydrated, over metallic Na in loosely stoppered bottles. (*Caution: See* Appendix B, safety notes on sodium metal and diethyl ether.)

C. Determination

(Large amounts H_2O-soluble components such as carbohydrates, urea, lactic acid, glycerol, and others may interfere with extraction of fat; if present, extract 2 g test portion on small paper in funnel with five 20 mL portions H_2O prior to drying for ether extraction.)

Extract ca 2 g test portion, dried as in 934.01 (*see* 4.1.03) or 920.36 (*see* 4.1.05), with anhydrous ether. Use thimble with porosity permitting rapid passage of ether. Extraction period may vary from 4 h at condensation rate of 5–6 drops/s to 16 h at 2–3 drops/s. Dry extract 30 min at 100°C, cool, and weigh.

References: *JAOAC* **64,** 351(1981); **65,** 289(1982).
ISO 6492:1999(E).

4.1.03

AOAC Official Method 934.01
Loss on Drying (Moisture) at 95 –100°C for Feeds
Dry Matter on Oven Drying at 95 –100°C for Feeds

First Action 1934
Final Action

*Codex-Adopted–AOAC Method**

(This method is not applicable to feeds containing >5% urea.)

Dry test portion containing ca 2 g dry material to constant weight at 95 –100°C under pressure 100 mm Hg (ca 5 h). For feeds with high molasses content, use temperature 70°C and pressure 50 mm Hg. Use covered Al dish 50 mm diameter and 40 mm deep. Report loss on drying (LOD) as estimate of moisture content.

Calculations

$$\%\ (\text{w/w})\ \text{LOD} = \%\ (\text{w/w})\ \text{moisture} = 100 \quad \frac{\text{wt loss on drying, g}}{\text{wt test portion, g}}$$

$$\%\ \text{Dry matter} = 100 - \%\ \text{LOD}$$

References: *JAOAC* **17,** 68(1934); **51,** 467(1968);
60, 322(1977).

Revised: March 1998

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* Adopted as Codex Defined Method (Type I) for gravimetry in the loss of drying in special foods.

31.4.02

AOAC Official Method 963.15
Fat in Cacao Products

Soxhlet Extraction Method
First Action 1963
Final Action 1973

Office International du Cacao et du Chocolat–AOAC Method

(Applicable to cacao products with or without milk ingredients or to products prepared by cooking with sugar and H_2O, and drying.)

A. Apparatus and Reagents

(**a**) *Soxhlet apparatus*.—With standard taper joints, siphon capacity ca 100 mL (33 80 mm thimble), 250 mL Erlenmeyer, and regulated heating mantle.

(**b**) *Petroleum ether*.—Distilled in glass, bp 30 –60°C.

B. Determination

Prepare test sample as in 970.20(**b**)(*1*) (*see* 31.1.01). Accurately weigh 3–4 g chocolate liquor, 4–5 g cocoa, 4–5 g sweet chocolate, or 9–10 g milk chocolate into 300–500 mL beaker. Add slowly, while stirring, 45 mL boiling water to give homogeneous suspension. Add 55 mL ca 8M HCl (2 + 1) and few defatted SiC chips or other antibumping agent, and stir. Cover with watch glass, bring slowly to boil, and boil gently 15 min. Rinse watch glass with 100 mL H_2O. Filter digest through 15 cm S&S 589 medium fluted paper, or equivalent, rinsing beaker 3 times with H_2O. Continue washing until last portion of filtrate is Cl-free as determined by addition of 0.1M $AgNO_3$. Transfer wet paper and residue to defatted extraction thimble and dry 6–18 h in small beaker at 100°C. Place glass wool plug over paper.

Add few defatted antibumping chips to 250 mL Erlenmeyer and dry 1 h at 100°C. Cool to room temperature in desiccator and weigh. Place thimble containing dried residue in Soxhlet, supporting it with spiral or glass beads. Rinse digestion beaker, drying beaker, and watch glass with three 50 mL portions petroleum ether, and add washings to thimble. Reflux digested residue 4 h adjusting heat so that extractor siphons 30 times/h or condensation rate of 5–6 drops/s.

Remove flask, and evaporate solvent on steam bath. Dry flask at 100 –101°C to constant weight (1.5–2 h). Cool in desiccator to room temperature and weigh. Constant weight is attained when successive 1 h drying periods show additional loss of <0.05% fat. Fat, % = g fat 100/g test sample. Duplicate determinations should agree within 0.1% fat.

References: *JAOAC* **28,** 482(1945); **33,** 342(1950);
34, 442(1951); **53,** 490(1970).
Analytical Methods of the OICC (1963) Ref. 2,
p. 8a-E.

31.1.01

AOAC Official Method 970.20
Cacao Products

Preparation of Laboratory Sample
Procedure
First Action 1970

(**a**) *Powdered products.*—Mix thoroughly and preserve in tightly stoppered bottles.

(**b**) *Chocolate products.*—(*1*) Chill ca 200 g sweet or bitter chocolate until hard, and grate or shave to fine granular condition. Mix thoroughly and preserve in tightly stoppered bottle in cool place. Alternatively,

(*2*) Melt ca 200 g bitter, sweet, or milk chocolate by placing in suitable container and partly immersing container in bath at ca 50°C. Stir frequently until test portion melts and reaches temperature of 45°–50°C. Remove from bath, stir thoroughly, and while still liquid, remove test portion for analysis, using glass or metal tube, 4–10 mm diameter, provided with close-fitting plunger to expel test portion from tube, or disposable plastic syringe.

Reference: *JAOAC* **53,** 388(1970).

Appendix B: Laboratory Safety

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Office of the Texas State Chemist

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_2H_2 which is dissolved in solvent recommended by manufacturer. Open C_2H_2 tank stem valve only a quarter turn. Change tank when C_2H_2 pressure shows 75–100 lb. If instrument has a drain trap, ensure that it is filled with H_2O before igniting burner. Following repair to C_2H_2 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_2O 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_3 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_2H_2 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_2O 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_2O, 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 Kevlarä 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_2O 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_2O.

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 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, H_2S, flammable solvents, hydrazine, and metal powders (especially Zn, Al, and Mg). Gaseous nitrogen oxides from HNO_3 can cause severe lung damage. Copious fumes are evolved when concentrated 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 $HClO_4$ by immediate and thorough washing with large amounts of H_2O.

(**b**) Hoods, ducts, and other devices for removing $HClO_4$ vapor should be made of chemically inert materials and so designed that they can be thoroughly washed with H_2O. 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 $HClO_4$ 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 $HClO_4$, treat test portion first with HNO_3 to destroy easily oxidizable organic matter unless otherwise specified. *Do not evaporate to dryness.*

(**f**) Contact of $HClO_4$ solution with strong dehydrating agents, such as P_2O_5 or concentrated H_2SO_4, may result in formation of anhydrous $HClO_4$ which reacts explosively with organic matter and with reducing agents. Exercise special care in performing analyses requiring use of $HClO_4$ 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 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 H_2SO_4 to H_2O. 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. 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 H_2O. 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 H_2O. Protect skin and eyes when handling.

CAS-132-27-4 (sodium biphenyl)
CAS-124-41-4 (sodium methylate)
CAS-141-52-6 (sodium ethylate)

Sodium Peroxide

Less caustic than sodium and potassium hydroxides but reacts violently with H_2O, 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 alkalies as solids or concentrated solutions. Add pellets to H_2O, 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_2O or moisture, CO_2, 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 HNO_3. May react violently with 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 H_2O and store under H_2O 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 Cl_2. Considered to be carcinogenic. Use alternative if available.

CAS-71-43-0 (benzene)

Bromine and Chlorine

Hazardous with 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, N_2O_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 Cl_2, O_3, $LiAlH_4$, 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_3, and Na_2O_2. 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_2O. Burns in air when exposed to flame. Can be explosive in contact with $CHCl_3$ or CH_3Cl.

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_3, 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_2O, cool to room temperature, and add 50 mL 30% H_2O_2. (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_2O-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 $CHCl_3$, and KOH plus $CHCl_3$ or KOH plus $HClO_4$.

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_2SO_4, concentrated HNO_3, CrO_3.)

Can react violently with most metal powders, 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 H_2O. Strong oxidizing agent. May form explosive mixture with H_2SO_4 or $HClO_4$. 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 H_2O_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 H_2O_2. Hazardous with flammable liquids, aniline, and nitrobenzene. Since it slowly decomposes with evolution of O_2, provide stored H_2O_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 H_2SO_3 in contact with moisture. Use effective fume removal device to remove 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)

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(18) "Farm Chemicals Handbook 2000" (1999) Meister Publishing Co., 37733 Euclid Ave, Willoughby, OH 44094, USA.

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(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.

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