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Standard Guide on Metallographic Laboratory Safety[1]

This standard is issued under the fixed designation E2014; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide outlines the basic safety guidelines to be used in a metallographic laboratory. Safe working habits are discussed for various tasks performed in a metallographic laboratory.

1.2 The sections appear in the following order:

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E3 Guide for Preparation of Metallographic Specimens

E7 Terminology Relating to Metallography

E340 Practice for Macroetching Metals and Alloys

E407 Practice for Microetching Metals and Alloys

E883 Guide for Reflected–Light Photomicrography

E1180 Practice for Preparing Sulfur Prints for Macrostructural Evaluation

E1558 Guide for Electrolytic Polishing of Metallographic Specimens

2.2 *ANSI Standard:*[3]

ANSI/AIHA Z9.5 Laboratory Ventelation

2.3 *NFPA Standard:*[4]

NFPA 45 Standard on Fire Protection for Laboratories Using Chemicals

NFPA 70E Standard for Electrical Safety in the Workplace

3. Terminology

3.1 *Definitions*—All terms used in this guide are either defined in Terminology E7 or are discussed in 3.2.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *agreement state*—a government body that has been granted regulatory authority over by-product materials and radiation-producing devices by the United States Nuclear Regulatory Commission (USNRC).

4. Significance and Use

4.1 This guide is intended as a summary of safety practices in the metallography laboratory. It can be used as a training

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

[4] Available from National Fire Protection Association (NFPA), 1 Batterymarch Park, Quincy, MA 02169-7471, http://www.nfpa.org.

reference for those new to the field of metallography and as a refresher to those who are experienced.

4.2 This guide is not intended to be inclusive of all safety concerns encountered in a metallographic laboratory. Several books that provide safety information are available (**1-15**).[5]

4.3 Before operating any equipment, it is advisable to read and understand the accompanying manuals and to follow any specified safety guidelines.

4.4 Safety data sheets (SDS) for chemicals being used in a laboratory should be on file and readily accessible. When working with any chemicals, especially for the first time, one should review the SDS supplied by the manufacturer and follow any safety guidelines suggested. The most current and applicable SDS should be on file for a given product or chemical.

5. General

5.1 Before operating any equipment, read and understand the accompanying manuals and follow any specified safety guidelines.

5.2 It is strongly recommended to read and understand Safety Data Sheets (SDS) for chemicals used in the laboratory. When working with any chemical, especially for the first time, review the SDS supplied by the manufacturer and follow any safety and environmental guidelines provided. Each laboratory is responsible for ensuring that the SDS they access is up-to-date.

5.3 Easily accessible emergency eyewash and showers are required where chemicals are stored, handled, or utilized. Consult the applicable regulatory agencies.

5.4 It is recommended to complete a Standard Operating Procedure (SOP), including a Job Safety Analysis (JSA) for every task or practice performed, listing any potential hazards and describing the safe operating practice to mitigate those hazards.

5.5 Be aware of the nature of the specimen material submitted for examination to ensure that it in itself does not pose a safety risk during storage or sample preparation. For example, see 7.5.

6. Heat Treating

6.1 The use of *Personal Protective Equipment* is strongly recommended. The minimum protection includes safety shoes (with metatarsal if required), safety glasses, and heat resistant gloves. When utilizing furnaces at high temperatures, >1038°C (>1900°F), a heat resistant suit and full face shield is well advised in addition to the gloves and glasses.

6.2 *Furnace Load Weights*—The weight limit that can be safely manually loaded into a furnace and unloaded at temperature should be established by the laboratory manager. The weight limit for manual loading is determined for specific procedures. This may increase or decrease depending on availability of equipment to aid in loading or unloading material; configuration of samples, which could make loading and unloading more or less risky; or the ability to utilize more than one technician in loading and unloading, or combinations thereof.

6.3 *Furnace Loading and Unloading*—When working with a furnace that requires removal of a thermocouple prior to loading or unloading, or both, handle the thermocouple with heat resistant gloves or tongs.

6.3.1 Material should be loaded into the furnace with tongs. The tongs should have the proper configuration for the specimen to be loaded. Properly maintain the tongs.

6.3.2 When several small or oddly shaped specimens are to be heat treated, a tray or wire basket may facilitate loading and unloading.

6.3.3 **Warning—**The surface of an oil quench bath can ignite during quenching. Use caution avoid being burnt. Do not lean over the oil quench tank when pieces are being quenched. Cover the tank quickly to extinguish any flames. Care should be given not to remove samples too quickly from the oil quench, as they may ignite.

6.4 Salt bath furnaces are set up in accordance with the manufacturer's instructions, along with state and local environmental regulations. Exhaust systems are required for gases generated. Some government organizations may regulate exhaust system specifications.

6.4.1 Personal protective equipment requirements include a heat resistant apron, sleeve protection, safety glasses, safety shoes (with metatarsal if required), and a heat resistant full face shield.

6.4.2 The start up of a salt bath has to be done by a properly trained technician.

6.4.3 When long cycles with little attention are performed, it is recommended to use temperature control devices with alarms with limits and shutdown capability.

6.4.4 Keep all water or moisture away from the salt bath. Clean pieces for heat treat to remove cutting fluids or other residual fluids. After cleaning, dry all pieces thoroughly.

6.4.5 Clean and dry baskets before each use. Salt dried on the basket can absorb moisture.

6.4.6 To avoid electric shock, do not place metal, baskets, or sludging tools between electrodes while the power is on.

6.4.7 Wear a full face mask when filling furnace with dry salts. An appropriate respiratory mask may also be required as per the applicable SDS.

6.4.8 Keep the work area clean. Do not keep boxes, pallets, or other highly flammable items in the work area. Keep floors clean to avoid slips, trips, or falls.

6.4.9 Keep salt containers tightly closed to avoid moisture adsorption by the dry salt.

6.4.10 Establish periodic checks and safe procedures for electrodes, sludging, rectifying, and the use of graphite rods.

6.5 *Induction Furnace*—When operating induction furnaces, the additional following precautions should be followed:

6.5.1 Persons with Pace Makers should stay well away from induction furnaces and other equipment designed to create strong magnetic fields.

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[5] The boldface numbers in parentheses refer to the list of references at the end of this standard.

6.5.2 No jewelry (rings, watches, bracelets) or other metal should be worn when operating the induction furnace or when remaining in close proximity. Voltage can be induced in these items to cause severe burns as the metal items heat up.

6.5.3 Standard precautions for arc flash protection as outlined in NFPA 70E should be followed as necessary for the high voltage and high power induction equipment.

6.6 A summary of recommended safe practices can be found in Table 1.

7. Specimen Sectioning and Rough Grinding (16)

7.1 Safety glasses should be worn when operating sectioning equipment. Do not wear ties, loose fitting clothing, and jewelry as they can become tangled in the moving parts of equipment. Tie back long hair. Auditory protection (ear plugs or other protection) may be needed if the noise level exceeds recommended guidelines or regulatory requirements. Ensure that there is sufficient lighting to clearly see the work area.

7.2 The cutting blade of a band saw is exposed and can be dangerous. Use the appropriate blade type and cutting settings for an effortless cut. Let the blade do the cutting and do not force the part into the blade. A guiding device, such as a block of wood, can be used to maneuver flat and stable specimens thereby reducing the possibility of hand injury. Round stock, pipe and curved specimens that can be destabilized by the downward drag of the blade need to be securely clamped in an appropriate jig or vice to prevent severe injury from an ejected specimen or broken blade.

7.3 The major danger from abrasive saw operation is from flying debris from a broken wheel. This danger is normally eliminated in modern equipment by shielding the cutting area from the operator. A cutoff saw must never be operated without a shield in place. Use cut-off wheels rated for the RPM of the cut-off machine. Also, select the cutoff wheel appropriate for the material being cut to prevent overheating and catastrophic wheel breakage. It is recommended to leave the cover open for the blade to dry out.

7.4 Sectioning often results in the formation of burrs on the edges of the specimen. These burrs can be quite sharp and potentially dangerous. Burrs can be removed by filing or grinding. Wear the appropriate approved cut resistant gloves when handling cut specimens.

7.5 Wet rough grinding is always preferred over dry grinding because of the reduced amount of dust and heat damage to the specimen. The dusts generated from grinding any metal are a health hazard. Metals such as beryllium, magnesium, lead, titanium, manganese, and silver, are extremely toxic and must be ground wet under a ventilation hood. An appropriate respiratory mask and a full face shield is also advisable.

TABLE 1 A Summary of Recommended and Discourage Practices When Heat treating

Recommended Practice	Discouraged Practice
Develop SOPs and JSAs	Lift specimens ≥ 10 kg
Wear	
safety shoes	Lean body over bath or quench tank
safety glasses or face shield	
heat resistant gloves	Leave tank contents uncovered
Use tongs, tray, or wire basket when handling samples	Contact electrodes with conductive material
Keep specimens and surrounding area free of moisture and debris	Loose clothing near automated furnace belts

7.6 Recommended procedures for the metallographic preparation of various materials can be found in Guide E3.

8. Specimen Mounting

8.1 A safety concern while using older mounting presses without integrated cooling is the potential of burns from contact with hot equipment or specimens. Newer mounting presses with integrated cooling systems do not typically have these issues. Wear insulated gloves to protect hands as needed.

8.2 Prolonged contact with many mounting materials can cause irritation to the eyes and skin. Consult the product label and SDS of the specific mounting material, and use the recommended practices and protective equipment.

8.3 When using castable resins, it is recommended to work (mixing, pouring and setting) in a fume hood. The corrosive, explosive, and carcinogenic properties vary widely with different castable resin components, and users need to be aware of the most current and applicable SDS information and of occupational health and safety issues relating to each component separately and mixed.

9. Mechanical Grinding/Polishing

9.1 Hand injury is common during grinding or polishing. For manual grinding or polishing of small or irregularly-shaped specimens consider mounting specimen to accommodate handling.

9.2 For semi-automatic and automatic grinder/polisher, ensure that all moving parts have come to rest before mounting, removing specimen holders or before cleaning the equipment. Lock-out procedures should be applied prior to the cleaning of equipment.

9.3 When using grinding or polishing equipment, do not wear ties, loose fitting jewelry, or loose fitting clothing, which could become entangled with the equipment.

9.4 Some lubricating liquids and polishing suspensions used during grinding and polishing are flammable. Use caution. Read the manufacturer's product label and most current and applicable SDS before using such products.

9.5 Dried colloidal silica may contain crystalline silica, which is considered as carcinogenic. If an accumulation of dried colloidal silica is to be removed, wear the required safety equipment and in particular a face mask with the appropriate filter. A dust mask is not sufficient. It is preferable to clean up colloidal silica while it is still wet.

10. Chemical Safety

10.1 Before using or mixing any chemicals, read and understand all product labels and pertinent SDS. The appropriate type of protective clothing will depend on the task being performed and the chemicals used. (see applicable SDS).

10.1.1 The hazards involved with handling chemicals are numerous and often specific to the chemicals being used. It is advisable that users are aware if there are dangers (with or without an odor) that are visible or invisible, with immediate or long term consequences.

10.2 The safe use, storage, and disposal of chemicals become more complex whenever they are combined or mixed. Experimentation with new combinations of chemical reagents is not recommended unless conducted by a person knowledgeable in chemistry. Table 2 shows some chemicals often encountered in a metallography laboratory known to be incompatible with one another.

10.2.1 It is recommended to mix small quantities and store in glass-stopper bottles. Exceptions include fluorides and strong alkaline solutions, which must be stored in polyethylene or another appropriate container recommended by the manufacturer for that specific chemical. Replace evaporated chemicals, if needed, to maintain filled capacity.

10.2.2 Storing incompatible chemicals in the same cabinet is not recommended.

10.2.3 Ventilated (connected to a fume hood), fire or explosion proof cabinets may be required. Consult the most current SDS for storage recommendations.

10.2.4 It is a good practice to dispose of spent or exhausted chemicals promptly, following all applicable regulations. Keep track of the shelf life of all chemicals.

10.3 *Specific Chemical Precautions.* Paragraphs 10.3.1 – 10.3.12 give specific safety situations that are often encountered in a metallographic laboratory and known to be dangerous.

10.3.1 The addition of sulfuric acid to water produces an extremely exothermic (heat generated) reaction. The solution must be cooled during mixing. While cooling, the acid must be slowly poured into the water or solvent with constant stirring. Spattering of the solution must be avoided. Concentrated and dilute solutions of sulfuric acid strongly attack the skin, are very hygroscopic, and vigorously attack most plastics.

10.3.2 The addition of crystalline chromium trioxide to water forms chromic acid; a strong oxidizing agent. The reaction liberates free oxygen, which can be an incendiary in the presence of oxidizable materials.

TABLE 2 Listing of Some Chemical Combinations Known to be Dangerous Encountered in Metallographic Laboratories

Chemical	Do Not Mix with the Following:	Use in Metallography
Acetic acid	Glycol, hydroxyl compounds, nitric acid, peroxides, permanganates	Chemical polishing
Acetone	Concentrated solutions of nitric and sulfuric acids	Degreasing, cleaning, etchants
Chromic acid	Acetic anhydride, flammable liquids, glycerol	Electropolishing
Hydrogen peroxide	Flammable liquids, organic materials	Chemical polishing, etchants
Nitric acid (concentrated)	Acetic acid, chromic acid, flammable liquids, higher alcohols	Chemical polishing, etchants
Perchloric acid	Acetic anhydride, alcohol, some organics, oil and grease	Electropolishing
Sulfuric acid	Methanol, chlorate, perchlorate and permanganate compounds	Etchants

10.3.2.1 Chromic acid cannot be safely mixed with acetic acid or most organic liquids, such as alcohols or glycerol.

10.3.2.2 Chromic acid solutions cannot be used in contact with plastic parts as it will cause their eventual destruction.

10.3.2.3 Prevent chromic acid contact with the skin since repeated exposure to even dilute solutions will cause persistent and painful ulcers that are slow to heal.

10.3.2.4 Chromium trioxide is poisonous to humans and is a carcinogen.

10.3.3 When preparing solutions containing mixed acids, salts in water, or solutions with organics, the acid must be added slowly to the solvent with constant stirring.

10.3.4 Prevent skin contact with acid fluorides since exposure to them, which may pass unnoticed at the time, will result in serious burns later. Extreme caution must be used when handling hydrofluoric acid. The use of a full face shield, HF resistant (neoprene) apron and arm-length gloves are strongly recommended when handling concentrated HF acid.

10.3.4.1 Hydrofluoric acid will ulcerate the skin even if it is immediately washed off with water. In order to neutralize the HF acid, it is necessary to flush the area with water followed by the use of a neutralizing cream, such as calcium gluconate gel (following the recommended instructions – note that calcium gluconate has a fairly short shelf life and must be regularly replenished), after which a physician must be immediately consulted. If left untreated, the acid will continue to penetrate the soft tissue until sequestered by combining with the calcium in the bone.. In severe cases sufficient calcium can be depleted from the body to provoke cardiac arrest.

10.3.4.2 Hydrofluoric acid attacks glass and must therefore be used and stored only in containers made of polyethylene or TFE-fluorocarbon.

10.3.5 In mixtures containing anhydrous aluminum chloride, extreme care must be exercised. The reaction between this compound and water during mixing can be violent (exothermic – produces heat).

10.3.6 Chromates and dichromates cannot be safely mixed with most organic liquids but can be mixed with saturated organic acids. Prevent contact with the skin.

10.3.7 The use of cyanide compounds by anyone not properly trained and familiar with them is extremely dangerous. Cyanides are among the quickest acting and most potent poisons that are likely to be encountered in the laboratory. Cyanide is so quick-acting and deadly that the administration of an antidote is usually ineffectual. Extreme care must be taken so that no droplet of solution or salt crystal is ever left around where it can be accidently ingested.

10.3.8 Solutions containing alkali hydroxides aggressively attack the skin, so avoid contact. Their dissolution in water produces heat.

10.3.9 Hydroxides must be added to water slowly until the desired concentration is reached. If the temperature becomes excessive, allow the solution to cool to ambient before adding more hydroxide.

10.3.10 Mixtures of nitric acid and methanol are relatively safe. Mixtures of up to 33 % nitric acid can be stored safely. When mixing, always add the acid slowly to the alcohol with constant stirring.

10.3.10.1 Nitric acid cannot be safely stored in a tightly stoppered bottle with any higher alcohol than methanol except in very dilute solutions. For example, do not store solutions of more than 3 % nitric acid in ethanol. Under certain conditions, extremely unstable or explosive nitro compounds such as azides or fulminates can be formed in alcohol base nitric acid solutions. The spontaneous decomposition of the mixture can also be catalyzed by impurities or heat.

10.3.10.2 Solutions containing more than 3 % nitric acid in an alcohol other than methanol have to be discarded or diluted to less than 3% nitric acid as soon as they have served their immediate purpose. Due to their dangerous nature, if their use cannot be avoided, use only small quantities electrolytically.

10.3.10.3 Nitric acid dried in cotton can yield gun cotton which is potentially explosive. Rinse the cotton thoroughly in water to remove residual acid. Do not dispose in municipal waste.

10.3.11 When dry, picric acid is a dangerous explosive. Purchase only small quantities. Drying of the material must be avoided during handling, storage, and disposal. If a bottle of dry picric acid is found, contact the local bomb disposal resources. Do not attempt to move the bottle.

10.3.11.1 Picric acid in ethanol can be stored safely as long as the ethanol is not allowed to evaporate completely, including, for example, crusting in an etching beaker, on a counter top, or at the top of a storage container.

10.3.11.2 Cautionary statements concerning the use of perchloric acid can be found in 11.5.

10.3.12 Refer to Section 14 for Chemical Spill controls.

10.4 Recommended chemistries, procedures, and practices for macroetching and microetching can be found in Test Method E340 and Practice E407, respectively.

11. Electrolytic Polishing/Etching

11.1 Many electrolytes used for polishing and etching can be extremely dangerous if handled carelessly. Read and understand the pertinent and most current SDS for all chemicals before any electrolyte is mixed or used.

11.2 Wear safety glasses and protective gloves when electrolytic polishing, etching, and preparing the solutions. A laboratory coat is also advisable.

11.3 With a few exceptions, most electrolytes have to be mixed and stored in clean glass containers. Exceptions include fluorides and strong alkaline solutions, which must be stored in polyethylene or another appropriate container recommended by the manufacturer for that specific chemical.

11.4 Electrolytes must never be allowed to concentrate due to evaporation. As soon as they exceed their usefulness, immediately discard electrolytes following all applicable regulations.

11.5 *Safety Precautions for Electrolytes Containing Perchloric Acid:*

11.5.1 Electrolytes containing perchloric and acetic anhydride are very dangerous to mix and can be unpredictable in use. Many research laboratories, industrial firms, and local governments forbid the use of such mixtures. Their use is not recommended.

11.5.2 When mixing perchloric acid and glacial acetic acid, the perchloric must be added to the acetic acid with stirring. Solution temperatures must never be allowed to exceed 29°C (85°F). Plastic material is susceptible to damage from these chemicals and is not recommended.

11.5.3 Electrolytes composed of perchloric acid and alcohol, with or without other organic additions, are safe to mix, provided the following safety precautions are followed.

11.5.3.1 Electro-polishing specimens or mounting materials containing bismuth in perchloric acid solutions is not recommended as reaction with Bi could result in an explosion.

11.5.3.2 Similarly, specimens mounted in organic mounting compounds, such as Bakelite (phenolic resins), must not be electro-polished in electrolytes containing perchloric acid as they may also react explosively

11.5.3.3 Special fume hoods with sidewall water curtains for perchloric acid manipulation and use are available.

11.5.4 Electrolytes composed of phosphoric acid in water or an organic solvent are generally quite easy to prepare. During mixing, the acid must be slowly poured into the water or solvent with constant stirring to prevent the formation of a heavy layer of acid at the bottom of the vessel.

11.6 Recommended electro-polishing practices and a listing of electrolytes commonly encountered in metallography can be found in Guide E1558.

12. Sulfur Printing

12.1 The sulfur printing procedure exposes the operator to hazards associated with grinding, chemicals, and photographic solutions. Therefore, refer to those sections for additional safety precautions.

12.2 *Personal Protective Equipment*—Many different types of personal protective equipment are recommended for the sulfur printing process. For the initial handling and grinding of the specimen: safety glasses or a face shield, safety shoes, and protective gloves. When dealing with the sulfuric acid solution: safety glasses, sulfuric acid resistant gloves, and a laboratory coat.

12.3 Any contact with concentrated sulfuric acid must be avoided. When mixing the dilute sulfuric acid solution the concentrated sulfuric acid must be added to the water very slowly with constant stirring. When mixed with water, substantial heat will be generated. The use of tongs is strongly recommended when handling the print in the acid solution.

12.4 Sulfuric acid is extremely reactive with strong oxidizing agents.

12.5 Prolonged exposure to the solution can cause skin and eye irritation. An easily accessible emergency eyewash station and shower is recommended.

12.6 Recommendations for preparing sulfur prints for macrostructural examination can be found in Practice E1180.

13. Laboratory Ventilation/Fume Hoods

13.1 *Fume Hood*—Fully intergrated fume hood shall have air circulated into a laboratory from a non-laboratory source and then exhausted to the outside of the building. Exhaust ventilation is located such that other ventilation devices do not inhibit its operation. Exhaust filters need to be cleaned or changed at regular intervals, as recommended by the manufacturer and as required by local and federal guidelines.

13.1.1 Any exhaust ventilation system is installed in accordance with any applicable statutory regulations.

13.1.2 Duct work material and fan components need to be selected as a function of the type of dust or fume that it will be exposed to. The duct work and fan require periodic inspection for checking the presence of corrosion and accumulations.

13.2 *Ductless Fume Hood*—Ductless fume hoods are ventilated enclosures that have their own exhaust fan that draws air out of the hood, passes it through an air cleaning filter and then discharges the cleaned exhaust air back into the workplace. Ductless fume hoods are "recirculating" exhaust systems used for contaminant control and use a variety of filters for air cleaning purposes, depending on the air contaminant(s).

13.2.1 Filter types include carbon filters and HEPA particular filters. The carbon filters can be designed for removal of specific molecule types (or combinations of types) including acids, inorganics, organics, solvents, organic vapors, and radioactive iodine. When multiple molecule types and particles are expected to be used in the same ductless fume hood, filters of different types can be stacked in series for comprehensive removal of the target molecules and particles. When using multiple filters in series it is important to position the HEPA filter (if used) first in the airflow to prevent premature saturat ion or clogging of the molecule filters.

13.2.2 The collection efficiency of these filters decreases over time, therefore a preventive maintenance program in accordance with the manufacturer's recommendations must be implemented to ensure optimum performance of the system and effective employee protection. It is advisable to institute an air sampling procedure that will allow the user to test the quality of the air that has passed through the filter. Such testing can be performed based on hours of use of the fume hood. Ductless fume hoods are typically equipped with an air sampling port, and the ductless fume hood manufacturer typically offers ancillary products to enable the air quality testing. The decrease in efficiency of the filter over time is not necessarily linear, and is not necessarily equal for all molecule types. It is possible for a filter to have sufficient efficiency for one molecule type, and degraded efficiency for the removal of other molecule types. It is important to test the removal efficiency of the molecule types that will be used.

NOTE 1—Careful consideration of the type of fume hood for the application must be performed.

13.3 It is recommended for any procedures that produce unpleasant odors or potentially hazardous fumes, vapors, or gases or both to be carried out in a fume hood. If corrosive emissions are produced, the hood should be lined with a corrosion-resistant material.

13.3.1 Fume hood emissions need to be exhausted outside the building in such a manner as to avoid any possible entry into building air intakes, and be far removed from outside pedestrian traffic areas.

13.3.2 Typically, fume hoods are not designed to provide protection from explosions. When flammable liquids or compressed gasses are used, a negative air pressure needs to be maintained in the laboratory environment.

13.3.3 Exhaust velocities have to be sufficient to minimize any deposition of emissions in the system and to prevent any escape of emissions from the hood face into the lab, but low enough to prevent vortices from forming in front of the technician, and to satisfy all applicable regulations. It is good practice to periodically y (at least every 3 months - U.S. OSHA, 29 CFR Part 1910 Recommended (**15**)) check the capture velocity of emissions at the hood face. Velocities need be in the range from 0,3 to 0,5 m/s (60 to 100 feet per minute) (U.S. OSHA, 29 CFR Part 1910 Recommended (**15**). Smoke testing can be used to ensure adequate capture and to locate any dead or turbulent areas.

13.3.3.1 Fume hood efficiency is affected by how far the front face window is lowered. Read and understand the manufacturer's operating instructions before using a fume hood.

13.3.4 Perform periodic maintenance at least annually in accordance with the manufacturer's instructions. If deficiencies are noted during inspection, terminate any activity using the hood until repairs are made. A visual lock-out procedure is recommended to ensure that the ventilation / fume hood is not inadvertently used.

13.3.5 Periodic maintenance and inspection should be performed in accordance with the manufacturer's instructions. If deficiencies are noted during inspection, any activity using the hood should be terminated until repairs are made.

13.3.6 Many older fume hoods do not have air flow monitoring devices. However, OSHA, ANSI/AIHA Z9.5 and NFPA 45 require new and remodeled hood to be equipped with air flow monitoring devices. In situations in which toxic gasses are being used, a low airflow or loss-of-airflow alarm system should to be installed to warn personnel of a change in exhaust capacity.

13.3.7 Industrial hygiene testing should be performed periodically. A test box is worn by the technician for up to 8 working hours. Filter should be sent to an independent test facility.

14. Chemical Spills

14.1 It is important to have a chemical spill cleanup procedure in place to handle small, medium, and large spills.

14.1.1 The procedures is not based strictly on volume of substance spilled, but also on how hazardous is the spilled substance.

14.1.2 For small quantities, and less dangerous materials, a trained person could contain and clean up a spill.

14.1.3 For larger quantities, and more dangerous materials, containment and clean up requires the assistance of hazardous material experts.

14.1.4 All laboratory personnel need to be aware of the established procedures developed for a specific laboratory's requirements, and any telephone numbers of people who are to be notified in the event of a spill need to be posted for easy access.

14.1.5 A chemical spill kit equipped to handle the common types of spills (acids, caustics, oxidizers, reducers, and solvents) need to be easily accessible in every metallographic laboratory.

14.2 Personal protection equipment must be worn when handling any type of chemical spill.

14.2.1 The minimum equipment consists of safety glasses or a face shield, safety gloves appropriate to the chemical nature of the spill, safety shoes, and a laboratory coat. Respiratory equipment may be necessary for certain types of spills.

14.2.2 In addition to personal protection equipment, it is important for the laboratory personnel to be aware of the proper first aid procedures in case of accidental splashing, inhalation, or swallowing.

14.3 There are six basic steps for handling a chemical spill. They are: *(1)* ensure that the contaminated area is safe before entering, *(2)* identify the nature of the spill, *(3)* choose the spill cleanup method, *(4)* treat the spill, and *(5)* restore the area and *(6)* dispose of the spill residue.

14.3.1 Isolate the spill area until the substance can be properly cleaned up. Depending on the size of the spill, the appropriate personnel are notified and any additional personal safety equipment required is obtained.

14.3.2 Before the spill can be cleaned up, it is important to properly identify the chemical nature of the spill, especially if neutralization is needed. The most current and applicable SDS have to be consulted to provide the necessary information concerning the chemicals involved.

14.3.3 There are two methods of chemical clean up: sorption and chemical neutralization.

14.3.3.1 Sorbents come in two forms, loose and pads, and can be used on acids, bases (caustics), and organics (flammables), as long as the sorbent is not reactive with the spilled substance. A disadvantage of sorbents is that they do not neutralize or eliminate the potential chemical hazard associated with the spilled substance.

14.3.3.2 In chemical neutralization, caustic substances are added to acid spills; an acid is added to a caustic spill; a reducing substance is added to an oxidizer, and an oxidizer to a reducing substance. Chemical spill kits contain containers of such neutralizing substances.

14.3.4 For both methods of cleanup, the treating agent is applied to the spill in small amounts, working from the outside in toward the middle to prevent the spread of the spill.

14.3.4.1 For a sorbent, the agent is mixed into the spill until all is absorbed.

14.3.4.2 For the neutralization method, after the proper neutralizer is chosen, it is added to the spill and thoroughly mixed in. Heat and fumes may be given off. The pH of the spill is checked once the neutralization appears to be complete. Some products have a color indicator incorporated to indicate that the mixture's pH is neutral (pH of 7.0).

14.3.5 To restore the area, all traces of the spill are cleaned up and the area neutralized. This is important, especially if a sorbent was used to clean up the main spill. The pH of any residues must be neutral for safe disposal.

14.3.5.1 The residue is properly disposed of in accordance with company policy and applicable statutory regulations. Any barriers that were erected to isolate the area cleaned and neutralized if contaminated and put away. If the spill was large enough to deplete the stock of chemicals in the chemical spill cleanup kit, replenish them before any work is allowed to continue.

15. Photography

15.1 The primary safety concern in photography is with respect to the chemicals used for developing negatives and for processing prints. It is strongly recommended to read and understand product literature as well as the most recent SDS and to follow their guidelines.

15.2 Many of the chemicals used in photography can cause eye and skin irritation after prolonged contact. The use of safety glasses and safety gloves resistant to those chemicals when working repeatedly with these chemicals for an extended period of time is recommended. A laboratory coat is also advisable.

15.3 Chemical manufacturers also recommend several precautions to be observed when mixing or storing their chemicals. These precautions can be found in Table 3.

15.4 Safety Information related to reflected light photomicrography can be found in Guide E883.

16. X-ray Diffraction and Electron Microscopy

16.1 *Electron Microscope.* An electron microscope presents several potential safety hazards that must be recognized before it is operated. These microscopes all operate at dangerously high voltages. Some hazardous X-rays are produced when the electron beam strikes a target, but their strength may not warrant protective or monitoring measures. Consult the instrument's manufacturer.

16.2 Many electron microscopes are also equipped with SiLi energy dispersive X-ray detectors, which require the handling of liquid nitrogen on a regular basis. Liquid nitrogen must always be transported in an approved cryogenic container. Liquid nitrogen free Silicon Drift Detectors (SDDs) are now a standard option.

16.2.1 Anyone handling liquid nitrogen shall wear safety glasses with side shields to prevent liquid nitrogen from splashing into their eyes. A full face shield is a preferred option. Insulated gloves (cryogenic resistant) must also be worn to prevent burns to the skin.

16.3 Neatly and safely route all electrical cabling and periodically inspect them for evidence of deterioration.

TABLE 3 Recommended Precautions When Mixing and Storing Photographic Chemicals

Chemical	Precaution
Developers	Keep away from acids
Fixers	Keep away from alcohol
Activators	Keep away from acids
Photo-flo	Keep away from oxidizers
Stop bath	Keep away from alkalis

16.3.1 Keep all safety interlocks in good working order and never by-pass them.

16.3.2 Troubleshooting of these systems is to be performed by a person knowledgeable in high voltage systems, preferably the manufacturer's service specialists.

16.3.3 It is recommended to establish a visual lock-out procedure to ensure that the instrument cannot be inadvertently energized during maintenance.

16.4 *Radioactive Sources.* In the United States of America laboratory equipment that generates X-rays from a radioactive source comes under the auspices of the USNRC or the State Department of Health, if the state where the equipment is located is an agreement state. All such equipment shall be operated in a safe manner, and the facility, equipment, and procedures shall meet the requirements of the controlling agency. Usually, compliance means the following:

16.4.1 The equipment must be registered with the controlling agency.

16.4.2 A radiation safety program is established for the facility. This program defines responsibilities and assigns them to specific individuals. The program is documented and in the form of a radiation control manual. The radiation control manual will detail the entire radiation safety program.

16.4.3 Signs are to be posted indicating that radiation producing equipment is present. Sign size, configuration, colors, and content will be detailed in the controlling agency's regulations.

16.4.4 Other documents and notices, such as licenses, employee monitoring records, and safety procedures and policies, are to be posted.

16.4.5 Safety surveys are performed on a regular basis, usually quarterly. The surveys include the checking of interlocks, warning lights, and of posting requirements as well as the measurement of radiation levels, if any, using a radiation survey meter or Geiger counter. All surveys must be documented.

16.4.6 Radiation levels are to be monitored through the use of film badges or dosimeters. Each employee must have an individual film badge; badges must not be shared. It is recommended that a control badge be used to avoid the logging of background radiation in the records of the system. Badges may be used as area monitors; however, the area monitor cannot be considered as a control badge. If dosimeters are used, all readings must be logged, documented, and reported.

16.4.7 A training program should be established covering the nature of radiation, operation of equipment, hazards from exposure, and proper safety practices. The program is to be documented and available to all personnel who operate or service the equipment. An outline of the information included in the training program will be a part of the radiation control manual. Personnel may be required to pass regulatory agency proficiency requirements.

16.5 *Non-radiation Source.* In the United States of America equipment that generates X-rays from a non-radioactive source generally is regulated by individual states. It is the responsibility of laboratory personnel to ensure that all regulatory laws and regulations are followed.

17. Laboratory Ergonomics

17.1 Laboratory workstation and task ergonomics should be considered from the outset. Discomfort, pain and occupational disability may only become evident over prolonged and repeated exposure. A small discomfort can become a significant disability with repeated exposure over time.

17.1.1 Computer workstations should be designed from the chair and up to ensure that workers have the appropriate posture and body support to not stress muscles and joints. Particularly sensitive areas are the lower back (lumbar support in the chair) neck (proper positioning of the computer screen with respect to eye movement) shoulders and upper arms (chair armrest position with respect to the keyboard) and wrists (armrest and wrist supports sufficient to avoid localized pressure points).

17.2 Laboratory bench workstations should be designed such that the worker does not need to routinely (frequency and duration) bend over to accomplish the work while standing up, or that laboratory stools (with foot rests and lumbar support if needed) can be used with the worker's legs fitting comfortably underneath the work surface/counter.

17.3 Tools and tasks must be assessed for mechanical vibration to meet applicable regulatory guidelines and requirements.

17.4 Microscope workstations are to be similarly designed as for computer workstations such that the worker's eye level can be trained onto the microscopes eyepieces without straining or stressing muscles or joints.

18. Disposal of Residues

18.1 Many of the chemicals, products and specimen materials contained in the residues from sample preparation and etching are not environmentally friendly and their disposal in municipal waste or effluent lines may be prohibited by government legislations.

18.1.1 Disposal information may be available in the substance's applicable SDS, and consult the local environmental protection government agency for disposal guidelines. It may be necessary to dispose of certain substances in secured landfills and to pretreat or neutralize liquids before the runoff from sedimentation tanks is allowed into the municipal effluent waste lines. The resulting sludge may need to be disposed of in a secured landfill.

19. Keywords

19.1 chemical spills; ergonomics; etching; fume hoods; grinding; heat treating; metallography; SDS; photography; polishing; radiation; safety; specimen; sulfur printing; X-ray

REFERENCES

(1) Urben, p., *Handbook of Reactive Chemical Hazards*, 7th Edition, Elsevier, 2007.

(2) Diberardinis, L.J. et al, *Guidelines for Laboratory Design: Health & Safety Considerations*, 3rd Edition, J. Wiley & Sons, 2001.

(3) Fuscaldo, A. A. et al, eds., *Laboratory Safety: Theory & Practice*, Academic Press, 1980.

(4) Gersey, E., Wilkerson, A., and Party, E., *Laboratory Safety in Practice*, Van Nostrand Reinhold, 1991.

(5) Keith Furr, A., ed., *Handbook of Laboratory Safety*, 5th Edition, CRC Press, 2000.

(6) Kelsey, C. A., and Gardner, A. F., eds., *Radiation Safety for Laboratory Technicians*, Warren H. Green, Inc., 1983 .

(7) Pal, S. B., ed., *Handbook of Laboratory Health & Safety Measures*, Kluwer Acedemic, 1985.

(8) Patty, F.A., ed., *Industrial Hygiene and Toxicology*, Vol.II - Toxicology, 3rd Edition, John Wiley & Sons 1995.

(9) Proctor, N. and Hughes, J., *Chemical Hazards in the Workplace*, J. B. Lippincott Co., 1978.

(10) *Prudent Practices for Disposal of Chemicals from Laboratories*, National Academy Press, 1983.

(11) *Prudent Practices for Handling Hazardous Chemicals in Laboratories*, National Academy Press, 1981.

(12) Rayburn, S. R., *The Foundations of Laboratory Safety*, Brock-Springer Series in Contemporary Bioscience, Springer-Verlag, 1989.

(13) Sax, N. I., *Dangerous Properties of Industrial Materials*, 5th Edition, John Wiley & Sons, 2004.

(14) *Metals Handbook*, Heat Treating, Vol. 4, ASM International, 2008.

(15) OSHA Occupational Safety and Health Administration https://www.osha.gov

(16) Vander Voort, G. F., *Metallography, Principles and Practice*, ASM International, 1999.

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