

AMERICAN SOCIETY FOR TESTING AND MATERIALS 100 Barr Harbor Dr., West Conshohocken, PA 19428 Reprinted from the Annual Book of ASTM Standards. Copyright ASTM

Standard Guide on Metallographic Laboratory Safety¹

This standard is issued under the fixed designation E 2014; 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 covers the outline of 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 This guide 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.

1.3 The sections appear in the following order:

	Section
Scope	1
Referenced Documents	2
Terminology	3
Significance and Use	4
Heat Treating	5
Specimen Preparation/Sectioning	6
Specimen Mounting	7
Mechanical Grinding/Polishing	8
Chemical Safety	9
Electrolytic Polishing/Etching	10
Sulfur Printing	11
Laboratory Ventilation/Fume Hoods	12
Chemical Spills	13
Photography	14
X ray/Electron Microscopy	15
Keywords	16

2. Referenced Documents

- 2.1 ASTM Standards:
- E 3 Methods of Preparation of Metallographic Specimens²
- E 7 Terminology Relating to Metallography²
- E 340 Test Method for Macroetching Metals and Alloys²
- E 407 Practice for Microetching Metals and Alloys²
- E 883 Guide for Reflected-Light Photomicrography²
- E 1180 Practice for Preparing Sulfur Prints for Macrostructural Examination²
- E 1558 Guide to Electrolytic Polishing of Metallographic Specimens²

3. Terminology

3.1 *Definitions*—All terms used in this guide are either defined in Terminology E 7 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 manual 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 (1-16).³

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 Material safety data sheets (MSDS) 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 MSDS supplied by the manufacturer and follow any safety guidelines suggested. The most current and applicable MSDS should be on file for a given product or chemical.

5. Heat Treating

5.1 *Personal Protective Equipment*, should be utilized. The minimum protection should include safety shoes, safety glasses, and heat resistant gloves. When utilizing furnaces at high temperatures (>1900°C), a heat resistant suit and face shield should be used in addition to gloves and glasses.

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

5.3 Furnace Loading and Unloading—When working with a furnace that requires a removal of a thermocouple prior to

¹ This test method is under the jurisdiction of ASTM Committee E-4 on Metallography and is the direct responsibility of Subcommittee E04.17 on Laboratory Evaluation and Safety.

Current edition approved April 10, 1999. Published July 1999.

² Annual Book of ASTM Standards, Vol 03.01.

³ The boldface numbers in parentheses refer to the list of references at the end of this standard.

loading, care should be taken to handle the thermocouple with heat resistant gloves or tongs.

5.3.1 Material should be loaded into the furnace with tongs. The tongs used should have the proper configuration for the specimen to be loaded. Tongs should be properly maintained.

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

5.3.3 The surface of an oil quench bath can ignite during quenching. Caution should be taken to avoid being burnt. Do not lean over the oil quench tank when pieces are being quenched, and cover the tank as quickly as possible to extinguish the flames.

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

5.4.1 Personal protective equipment should include a heat resistant apron, sleeve protection, safety glasses, safety shoes, and face shield.

5.4.2 The start up of a salt bath should be performed by a properly trained technician.

5.4.3 When long cycles with little attention are performed, the temperature control devices should have alarms with limits and shutdown capability.

5.4.4 Keep all water or moisture away from and out of the salt bath. Clean the pieces to remove cutting fluids or any other residuals. After cleaning, dry all the pieces thoroughly.

5.4.5 Clean and dry baskets before each use. Moisture may build up on the salt dried on the basket.

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

5.4.7 Wear a face mask when filling furnace with dry salts.

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

5.4.9 Keep salt containers tightly closed to avoid moisture adsorption in dry salt.

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

5.5 A standard operating procedure (SOP), including a job safety analysis (JSA), should be completed for every heat treating practice performed, listing any potential hazards and describing the safe operating practice. A summary of recommended safe practices can be found in Table 1.

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

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

6. Specimen Preparation/Sectioning (17)

6.1 Safety glasses should always be worn when operating sectioning equipment. Ties, loose fitting clothing, and jewelry can become tangled in the moving parts of equipment and should not be worn.

6.2 The cutting blade of a bandsaw is exposed and can be dangerous. A guiding device, such as a block of wood, should be used to maneuver the specimen, thereby, reducing the possibility of hand injury.

6.3 The major danger from abrasive saw operations 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 should never be operated without a shield in place.

6.4 Sectioning often results in the formation of burrs on the edges of the specimen. These burrs can be quite sharp and potentially dangerous. The burrs should be carefully removed by filing or grinding.

6.5 Wet grinding is always preferred over dry grinding because of the reduced amount of dust and reduced heat damage to the specimen. The dusts generated from grinding any metal are a health hazard. Those generated by grinding materials, such as beryllium, magnesium, lead, manganese, and silver, are extremely toxic. These materials should be ground wet under a ventilation hood, and a face mask should be worn.

6.6 A SOP, including a JSA, should be completed for each piece of equipment being used while sectioning or preparing specimens, listing any potential hazards and describing the safe operating procedure. Recommended procedures for the metallographic preparation of various materials can be found in Methods E 3.

7. Specimen Mounting

7.1 The major safety concern while using either automatic or manual mounting presses involves the potential of burns from contact with hot equipment or specimens. Operators should wear insulated gloves to protect hands.

7.2 Prolonged contact with many mounting materials can cause irritation to the eyes and skin; gloves are recommended. Also prolonged exposure to the vapors and dust generated during the mounting procedure is not recommended. Consult the product label and MSDS of the specific mounting material, and use the protective equipment recommended.

7.3 When using castable resins, work should be completed under a fume hood. The corrosive, explosive, and carcinogenic properties vary widely with different castable resin components, and users should be aware of the most current and applicable MSDS information and occupational health and safety issues relating to each component separately and mixed.

7.4 A SOP, including a JSA, should be completed for each piece of equipment used for every mounting routine performed. The SOP should list any potential hazards and describe the safe operating practice.

8. Mechanical Grinding/Polishing

8.1 Injury to operator's hands is a very common injury received during grinding or polishing. For manual polishing, small or irregularly-shaped specimens should be mounted to accommodate handling. For semi-automatic and automatic

procedures, ensure that all moving parts have come to rest before mounting or removing specimen holders or before cleaning the equipment, or both.

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

8.3 Some of the lubricating liquids and polishing suspensions used during grinding and polishing are flammable and caution should be taken. The specific manufacturer's product label and most current and applicable MSDS should be read before it is used.

8.4 A SOP, including a JSA, should be completed for all mechanical grinding and polishing equipment, listing any potential hazards and describing the safe operating practice.

9. Chemical Safety

9.1 Before using or mixing any chemicals, all product labels and pertinent and most current MSDS should be read and understood. The appropriate type of protective clothing will depend on the task being performed. Again, consult the product label and the most current and applicable MSDS for recommendations concerning protective clothing.

9.2 The safe use, storage, and disposal of chemicals becomes 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.

9.3 The hazards involved with handling chemicals are numerous and often specific to the chemicals being used. Users should be aware if there are dangers that are long term, immediate, visible, invisible, and with or without odor.

9.4 Only small quantities should be mixed and stored in glass-stoppered bottles filled to capacity. Exceptions include fluorides and strong alkaline solutions, which should be stored to polyethylene or another appropriate container recommended by the manufacturer for that specific chemical. Any evaporated solvents should be replaced to maintain a filled capacity.

9.5 Chemicals should always be protected from heat and fire.

9.6 Spent or exhausted chemicals should be disposed of promptly, following all applicable regulations.

TABLE 2 Listing of Some Chemical Combinations Encountered in a Metallographic Laboratory Known to Be Dangerous

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) Perchloric acid	Acetic acid, chromic acid, flammable liquids, higher alcohols Acetic anhydride, alcohol, some	Chemical polishing, etchants Electropolishing
Sulfuric acid	organics, oil and grease Methanol, chlorate, perchlorate and permanganate compounds	Etchants

9.7 *Specific Chemical Precautions*—Paragraphs 9.7.1-9.7.13 list some specific safety situations that are often encountered in a metallographic laboratory and known to be dangerous.

9.7.1 The addition of sulfuric acid to water produces an extremely exothermic reaction. The solution must be cooled during mixing. While cooling, the acid must be slowly poured in the water or solvent with constant stirring. Spattering of the solution must be avoided. Dilute solutions (anything less than 100 % concentrated) of sulfuric acid strongly attack the skin, are very hygroscopic, and vigorously attack most plastics.

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

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

9.7.2.2 Chromic acid solutions cannot be used in contact with plastic parts without their eventual destruction.

9.7.2.3 Care should be taken to 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.

9.7.2.4 Chromium trioxide is poisonous to humans and a carcinogen.

9.7.3 When preparing solutions containing mixed acids, salts in water, or other organic solutions, the acid should be added slowly to the solvent with constant stirring.

9.7.4 Particular care should be taken to avoid skin contact with acid fluorides since exposure to them, which may pass unnoticed at the time, may result in serious burns later. Extreme caution should be used when handling hydrofluoric acid.

9.7.4.1 Hydrofluoric acid will ulcerate the skin if not immediately washed off with water. Flushing the area with water must be followed by the use of a neutralizing cream, such as calcium glutamate gel, after which a physician should be consulted as soon as possible. If left untreated, the acid will attack the skin to the bone.

9.7.4.2 Hydrofluoric acid, which attacks glass, should be used and stored only in containers made of polyethylene or TFE-fluorocarbon.

9.7.5 In those mixtures containing anhydrous aluminum chloride, extreme care must be exercised. The reaction between this compound and water during mixing can be violent. While mixing, the acid must be slowly poured into the water with constant stirring.

9.7.6 Chromates and dichromates cannot be safely mixed with most organic liquids but can be mixed with saturated organic acids. Care should be taken to prevent contact with the skin.

9.7.7 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 crystal of salt is ever left around where it can be accidently ingested.

9.7.8 Solutions containing alkali hydroxides aggressively attack the skin, so contact should be avoided. Their dissolution in water produces heat.

9.7.9 Hydroxides should 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.

9.7.10 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.

9.7.10.1 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, azides, or fulminates can be formed in alcoholic nitric acid solutions. The spontaneous decomposition of the mixture can also be catalyzed by impurities or heat.

9.7.10.2 Solutions containing more than 3 % nitric acid in an alcohol other than methanol should always be discarded as soon as they have served their immediate purpose. Due to their dangerous nature, only small quantities should be employed electrolytically if their use can not be avoided.

9.7.11 When dry, picric acid is a dangerous explosive. Only small quantities should be purchased. Drying of the material must be avoided during handling, storage, and disposal.

9.7.11.1 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.

9.7.12 Cautionary statements concerning the use of perchloric acid can be found in 10.5.

9.7.13 Recommended chemistries, procedures, and practices for macroetching and microetching can be found in Test Method E 340 and Practice E 407, respectively.

10. Electrolytic Polishing/Etching

10.1 Many electrolytes used for polishing and etching can be extremely dangerous if handled carelessly. The pertinent and most current MSDS for all chemicals should be read before any electrolyte is mixed or used.

10.2 Safety glasses and protective gloves should be worn whenever electrolytic polishing or etching.

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

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

10.5 Safety Precautions for Electrolytes Containing Perchloric Acid:

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

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

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

10.5.3.1 Electro-polishing specimens or mounting materials containing bismuth in perchloric acid solutions might result in an explosion.

10.5.3.2 Similarly, specimens mounted in organic mounting compounds, such as Bakelite, must not be electro-polished in electrolytes containing perchloric acid as they may also react explosively.

10.5.4 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 in the water or solvent with constant stirring to prevent the formation of a heavy layer of acid at the bottom of the vessel.

10.6 A SOP, including a JSA, should be completed for each piece of electrolytic etching or polishing machine used, listing any potential hazards and describing the safe operating practice. Recommended electro-polishing practices and a listing of electrolytes commonly encountered in metallography can be found in Guide E 1558.

11. Sulfur Printing

11.1 The sulfur printing procedure exposes the operator to hazards associated with grinding, chemicals, and photographic solutions. Therefore, refer to those sections as well for more detailed safety precautions.

11.2 *Personal Protective Equipment*—Many different types of personal protective equipment can and should be utilized during the sulfur printing process. For the initial handling and grinding of the specimen, safety glasses or a face shield, safety shoes, and protective gloves may be worn. When dealing with the sulfuric acid solution, safety glasses, rubber gloves, and a lab coat should be utilized.

11.3 The primary safety concern in the area of sulfur printing deals with the use of sulfuric acid. Any contact with concentrated sulfuric acid must be avoided. When mixing the dilute sulfuric acid solution used to make sulfur prints, the concentrated sulfuric acid should be added to the water very slowly with constant stirring. Sulfuric acid is extremely reactive with strong oxidizing agents. When mixed with water, substantial heat will be generated. The use of safety glasses and rubber gloves is recommended. Tongs should be used when handling the print in the acid solution.

11.4 Prolonged exposure to the solution could cause skin and eye irritation. The use of safety glasses and rubber gloves is still recommended when working with this chemical for an extended period of time.

11.5 A SOP, including a JSA, should be completed, listing any potential hazards and describing the safe operating practice used while sulfur printing. Recommendations for preparing sulfur prints for macrostructural examination can be found in Practice E 1180.

12. Laboratory Ventilation/Fume Hoods

12.1 Air should circulate into a laboratory from a nonlaboratory source and then exhausted to the outside of the building. Exhaust ventilation should be located such that other ventilation devices do not inhibit the operation of the device. Exhaust filters should be cleaned or changed at regular intervals.

12.1.1 Any exhaust ventilation system should be installed in accordance with any applicable statutory regulations.

12.1.2 Duct work should be inspected periodically for corrosion and cleanliness.

12.2 Any procedures that produce unpleasant odors or potentially hazardous fumes, vapors, or gases or both should be conducted in a fume hood. If corrosive emissions are produced, the hood should be lined with a corrosion-resistant material. The corrosion-resistant material should not react with any exhaust fumes produced in the hood.

12.2.1 Fume hood emissions should be exhausted outside the building in such a manner as to avoid air intakes to the building, not recirculated to other inside areas.

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

12.2.3 Exhaust velocities should be sufficient to minimize any deposition of emissions in the system to prevent any escape of emissions from the hood face and to satisfy all state and federal regulations.

12.2.4 The capture velocity of emissions at the hood face should be monitored periodically. Velocities should be in the range from 80 to 120 feet per minute. Smoke testing can be used to ensure adequate capture and to locate any dead or turbulent areas.

12.2.5 Periodic maintenance 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.

12.2.6 In situations in which toxic gasses are being used, a low airflow or loss-of-airflow alarm system should be installed to warn personnel of a change in exhaust capacity.

13. Chemical Spills

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

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

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

13.1.3 For larger quantities, and more dangerous materials, containment and clean up might require the assistance of hazardous material experts.

13.1.4 All laboratory personnel should 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 should be posted for easy access.

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

13.2 Personal protection equipment should be used when handling any type of chemical spill.

13.2.1 The minimum equipment should consist of safety glasses or a face shield, rubber gloves, safety shoes, and a laboratory coat. Respiratory equipment may be necessary for certain types of spills.

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

13.3 There are five basic steps for handling a chemical spill. They are (1) ensure contaminated area is safe before entering, (2) identify the spill, (3) choose the spill cleanup method, (4) treat the spill, and (5) restore the area.

13.3.1 The safety steps involve isolating the area of the spill until the substance can be properly cleaned up. Depending on the size of the spill, the proper personnel should be notified; and finally, any additional personal safety equipment required should be obtained.

13.3.2 Before the spill can be cleaned up, it is important to properly identify the spill, especially if neutralization is the chosen method. The most current and applicable MSDS provide the necessary information concerning the chemicals.

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

13.3.3.1 Sorbents come in two forms—loose and pads—and can be used on acids, caustics, and 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 hazard associated with the spilled substance.

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

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

13.3.4.1 For a sorbent, the agent should be mixed into the spill until all is absorbed.

13.3.4.2 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 should be checked once the neutralization appears to be complete.

13.3.5 To restore the area, all traces of the spill should be cleaned up and the area neutralized. This is important, especially if a sorbent was used to clean up the main spill. If the pH is in the range from 2 to 12, then the material can be disposed of. If the pH does not fall into this range, additional neutralization is necessary.

13.3.5.1 The residue should be properly disposed of in accordance with company policy and applicable statutory

regulations. Any barriers that were erected to isolate the area need to be put away. If the spill was large enough to deplete the stock of chemicals in the chemical spill cleanup kit, then replacement materials should be ordered at this time.

14. Photography

14.1 The primary safety concern in the area of photography deals with the chemicals used in the developing of negatives and the processing of prints. The manufacturers of photographic chemicals have evaluated the hazards associated with their products and have listed this information on the product label or on printed material provided with the product. It is strongly recommended that this literature be read and the guidelines followed. The user should also be familiar with the MSDS provided with each product.

14.2 Many of the chemicals used in photographic work can cause eye and skin irritation after prolonged contact. The use of safety glasses and rubber gloves when working repeatedly with these chemicals for an extended period of time is recommended.

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

14.4 Information related to reflected light photomicrography can be found in Guide E 883.

15. X-ray Diffraction/Electron Microscopy

15.1 An electron microscope presents several potential safety hazards that must be recognized before it is operated. These microscopes all operate at dangerously high voltages and all produce hazardous X rays when energized.

15.2 Many electron microscopes are also equipped with energy dispersive X-ray detectors, which require the handling of liquid nitrogen on a regular basis. Liquid nitrogen should always be transported in an approved cryogenic container.

15.2.1 Anyone handling liquid nitrogen should wear safety glasses with side shields to prevent liquid nitrogen from splashing into their eyes. Gloves should also be worn to prevent burns to the skin.

15.3 All electrical cabling should be neatly routed in a safe manner and periodically inspected for evidence of deterioration.

15.3.1 All safety interlocks should be kept in good working order and never by-passed.

15.3.2 Troubleshooting of these systems should be performed by a person knowledgeable in high voltage systems.

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	

15.4 Laboratory equipment that generates X rays from a radio-active 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 must be operated in a safe manner, and the facility, equipment, and procedures should meet the requirements of the controlling agency. Usually, compliance means the following:

15.4.1 The equipment must be registered with the controlling agency.

15.4.2 A radiation safety program should be established for the facility. The program should define responsibilities and assign them to specific individuals. The program should be documented and in the form of a radiation control manual. The radiation control manual will detail the entire radiation safety program.

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

15.4.4 Other documents and notices, such as licenses, employee monitoring records, and safety procedures and policies, should be posted.

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

15.4.6 Radiation levels should 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 should not be considered as a control badge. If dosimeters are used, all readings must be logged, documented, and reported.

15.4.7 A training program should be established covering the nature of radiation, operation of equipment, hazards from exposure, and proper safety practices. The program should be documented and given 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.

15.5 Equipment that generates X rays from a nonradioactive source generally is regulated by individual states. It is the responsibility of laboratory personnel to ensure that all federal and state regulatory laws are followed.

15.6 A SOP, including a JSA, should be completed for all the equipment and any procedures relating to X-ray diffraction or electron microscopy, listing any potential hazards and describing the safe operating practice.

16. Keywords

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

働 E 2014

REFERENCES

- (1) Bretherick, L., *Handbook of Reactive Chemical Hazards*, 2nd Edition, Butterworths, London, 1979.
- (2) Diberardinis, L.J. et al, *Guidelines for Laboratory Design: Health & Safety Considerations*, J. Wiley & Sons, New York, N. Y., 1989.
- (3) Fuscaldo, A. A. et al, eds., Laboratory Safety: Theory & Practice, Academic Press, San Diego, Calif., 1980.
- (4) Gersey, E., Wilkerson, A., and Party, E., *Laboratory Safety in Practice*, Van Nostrand Reinhold, New York, N.Y., 1991.
- (5) Keith Furr, A., ed., *Handbook of Laboratory Safety*, 3rd Edition, CRC Press, Boca Raton, Fla., 1989.
- (6) Kelsey, C. A., and Gardner, A. F., eds., *Radiation Safety for Laboratory Technicians*, Warren H. Green, Inc., St. Iouis, Mo., 1983.
- (7) Pal, S. B., ed., Handbook of Laboratory Health & Safety Measures, Kluwer Acedemic, Norwell, Mass., 1985.
- (8) Patty, F.A., ed., *Industrial Hygiene and Toxicology*, Vol.II- Toxicology, 3rd Edition, Wiley-Interscience, New York, N.Y., 1980.
- (9) Proctor, N. and Hughes, J., *Chemical Hazards in the Workplace*, J. B. Lippincott Co., Philadelphia, PA., 1978.

- (10) Prudent Practices for Disposal of Chemicals from Laboratories, National Academy Press, Washington, D. C., 1983.
- (11) Prudent Practices for Handling Hazardous Chemicals in Laboratories, National Academy Press, Washington, D. C., 1981.
- (12) Rayburn, S. R., *The Foundations of Laboratory Safety*, Brock-Springer Series in Contemporary Bioscience, Springer-Verlag, New York, N. Y., 1989.
- (13) Sax, N. I., *Dangerous Properties of Industrial Materials*, 5th Edition, Van Nostrand Reinhold Co., New York, N. Y., 1979.
- (14) Steere, N. V., ed., *Handbook of Laboratory Safety*, 2nd Edition, CRC Press, Boca Raton, Fla., 1971.
- (15) Van Houten, N., *Laboratory Safety Standards for Industry, Research, Academe*, Sci-Tech Publications, Lake Isabella, Calif., 1990.
- (16) Metals Handbook, Heat Treating, Vol. 4, ASM, Metals Park, OH, 1991.
- (17) Vander Voort, G. F., *Metallography, Principles and Practice*, New York, NY, 1984, pp. 279-280.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 100 Barr Harbor Drive, West Conshohocken, PA 19428.