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Standard Guide on Metallographic Laboratory Safety[1]

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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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E3 Guide for Preparation of Metallographic Specimens
E7 Terminology Relating to Metallography
E340 Test Method 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

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 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 **(1-14)**.[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. General

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

[1] This guide is under the jurisdiction of ASTM Committee E04 on Metallography and is the direct responsibility of Subcommittee E04.17 on Criteria for Metallographic Laboratory Evaluation and Safety.

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

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

5.3 Easily accessible emergency eyewash and showers are required where chemicals are manipulated and used. 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, handle the thermocouple with heat resistant gloves or tongs.

6.3.1 Material should be loaded into the furnace with tongs. The tongs used need to 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 The surface of an oil quench bath can ignite during quenching. Be cautious 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 any flames.

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 to remove cutting fluids or any other residuals. After cleaning, dry all the 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 MSDS.

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

7. Specimen Sectioning and Rough Grinding (15)

7.1 Safety glasses need to 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. 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

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
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	

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.

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, 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.

7.6 Recommended procedures for the metallographic preparation of various materials can be found in Methods 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 if needed.

8.2 Prolonged contact with many mounting materials can cause irritation to the eyes and skin. Consult the product label and MSDS 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 MSDS information and of occupational health and safety issues relating to each component separately and mixed.

9. Mechanical Grinding/Polishing

9.1 Injury to operator's hands is very common during grinding or polishing. For manual grinding or polishing of small or irregularly-shaped specimens consider mounting them to accommodate handling.

9.2 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. Lock-out procedures may need to 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 of the lubricating liquids and polishing suspensions used during grinding and polishing are flammable so be cautious. Read the specific manufacturer's product label and most current and applicable MSDS 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 and most current MSDS. The appropriate type of protective clothing will depend on the task being performed and the chemicals used. (as per the applicable MSDS).

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 only mix small quantities and to store them 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 It is a recommended practice to not store incompatible chemicals in the same cabinet.

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

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

10.2.4 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 (anything less than 100 % concentrated) 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.

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 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 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 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 MSDS for all chemicals before any electrolyte is mixed or used.