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## Standard Guide on Metallographic Laboratory Safety<sup>1</sup>

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### 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:

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### 2. Referenced Documents

#### 2.1 *ASTM Standards:*

E 3 Methods of Preparation of Metallographic Specimens<sup>2</sup>

E 7 Terminology Relating to Metallography<sup>2</sup>

E 340 Test Method for Macroetching Metals and Alloys<sup>2</sup>

E 407 Practice for Microetching Metals and Alloys<sup>2</sup>

E 883 Guide for Reflected-Light Photomicrography<sup>2</sup>

E 1180 Practice for Preparing Sulfur Prints for Macrostructural Examination<sup>2</sup>

E 1558 Guide to Electrolytic Polishing of Metallographic Specimens<sup>2</sup>

<sup>1</sup> 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.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 03.01.

### 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).<sup>3</sup>

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

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

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 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.

## 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.

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

Recommended Practice	Discouraged Practice
Develop SOPs and JSAs	Lift specimens $\geq$ 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	

**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.

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.

**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)	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