Designation: E2014 – 17

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:

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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, health, and environmental 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

[1] This guide is under the jurisdiction of ASTM Committee E04 on Metallography and is the direct responsibility of Subcommittee E04.01 on Specimen Preparation.

Current edition approved June 1, 2017. Published July 2017. Originally approved in 1999. Last previous edition approved in 2011 as E2014 – 11. DOI: 10.1520/E2014-17.

[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.

[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.

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

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

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.