



Designation: ~~E1558-99 (Reapproved 2004)~~ Designation: E 1558 – 09

Standard Guide for Electrolytic Polishing of Metallographic Specimens¹

This standard is issued under the fixed designation E 1558; 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 deals with electrolytic polishing as a means of preparation of specimens for metallographic purposes. Procedures are described for polishing a variety of metals.

NOTE 1—References (1-133)² on electrolytic polishing will provide the reader with specific information beyond the scope of this guide.

1.2

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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. Specific safety precautions are described in Section 5 and 6.3.1.

2. Referenced Documents

2.1 ASTM Standards:³

E 7 Terminology Relating to Metallography

E 407 Test Methods Practice for Microetching Metals and Alloys

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 electrolytic polish (electropolish)—A method of polishing metals and alloys in which material is removed from the surface by making the metal the anode in an electrolytic bath.

4. Significance and Use

4.1 Advantages of Electrolytic Polishing:

4.1.1 For some metals, a high quality surface finish can be produced that is equivalent to, or better than, that which can be obtained by mechanical methods.

4.1.2 Once procedures have been established, satisfactory results can be obtained rapidly with reproducibility.

4.1.3 There can be a marked saving of time if many specimens of the same material are polished sequentially.

4.1.4 Electropolishing a selected area on the surface of a relatively large metal part can be accomplished nondestructively, that is, without the need for sectioning to remove a piece.

4.1.5 Soft, single-phase metals, which may be difficult to polish by mechanical methods, may be successfully electropolished.

4.1.6 The true microstructure of a specimen can be obtained because artifacts (such as disturbed metal, scratches, and mechanical twins) produced on the surface, even by careful grinding and mechanical polishing operations, can be removed. These features are important in low-load hardness testing, X-ray diffraction studies, and in electron microscopy, where higher resolution puts a premium on undistorted metal surfaces.

4.1.7 After electropolishing is completed, etching can often be accomplished by reducing the voltage (generally to about one-tenth that required for polishing) for a short time before it is turned off.

NOTE 2—Not all electropolishing solutions produce good etching results.

4.2 Disadvantages of Electrolytic Polishing:

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, 2004. Published July 2004. Originally approved in 1993. Last previous edition approved in 1999 as E 1558 – 99(2004).

² The boldface numbers in parentheses refer to the references at the end of this standard.

³ 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.

4.2.1 Many of the chemical mixtures used in electropolishing are poisonous or dangerous if not properly handled (see Section 5). These hazards are similar to those involved in the mixing and handling of etchants, see Test Methods E 407.

4.2.2 In multi-phase alloys, the polishing rate of each phase may be different. The result may be a non-planar surface.

4.2.3 Electropolished surfaces may be slightly undulated rather than perfectly planar and, therefore, may not be suitable for examination at all magnifications.

4.2.4 The rate of polishing in areas adjacent to various inhomogeneities, such as nonmetallic inclusions and voids, is usually greater than that in the surrounding matrix and tends to exaggerate the size of the inclusions and voids.

4.2.5 Dimples, pits, and waviness limit applications involving surface phenomena, coatings, interfaces, and cracks. Edges tend to be attacked preferentially, resulting in edge rounding.

4.2.6 Artifacts may be produced by electropolishing.

4.2.7 Specimen mounting materials may react with the electrolyte.

4.2.8 The electropolished surfaces of certain materials may be passive and difficult to etch.

4.2.9 Metal removal rates by electropolishing are usually quite low, typically about 1 $\mu\text{m}/\text{min}$, and all of the prior induced damage from cutting and grinding may not be removed if preparation is stopped after a 600-grit SiC grind and electropolishing times are short.

4.2.10 A large number of electrolytes may be needed to polish the variety of metals encountered by a given laboratory. Considerable time may be required to develop a procedure for a new alloy.

5. General Safety Precautions

5.1 Before using or mixing any chemicals, all product labels and pertinent Material Safety Data Sheets (MSDS) should be read and understood concerning all of the hazards and safety precautions to be observed. Users should be aware of the type of hazards involved in the use of all chemicals used, including those hazards that are immediate, long-term, visible, invisible, and with or without odors.

5.1.1 Consult the product labels and MSDS for recommendations concerning proper protective clothing.

5.1.2 All chemicals are potentially dangerous. All persons using any electrolyte should be thoroughly familiar with all of the chemicals involved and the proper procedure for handling, mixing, and disposing of each chemical, as well as any combinations of those chemicals.

5.1.3

5.1.3 When pouring, mixing, or etching, always use the proper protective equipment (glasses, gloves, apron, etc.) and it is strongly recommended to always work under a certified and tested fume hood. This is imperative with etchants that give off noxious odors or toxic vapors. In particular, note that solutions containing perchloric acid must be mixed and used in an exclusive hood equipped with a wash down feature to avoid accumulation of explosive perchlorates.

5.1.4 Table 1 includes specific safety precautions for the mixing or use of some electrolytes. The user should take care to observe each of these specific precautions.

TABLE 1 Electrolytes for Electropolishing

Class	Use	Formula		Cell Voltage	Time	Remarks
Group I (Electrolytes Composed of Perchloric Acid and Alcohol With or Without Organic Additions)						
I-1	Al and Al alloys with less than 2-percent Si	ethanol (95%)	800 mL	30 to 80	15 to 60 s	
		distilled water	140 mL			
I-1	steels—carbon, alloy, stainless Pb, Pb-Sn, Pb-Sn-Cd, Pb-Sn-Sb Zn, Zn-Sn-Fe, Zn-Al-Cu Mg and high Mg alloys	perchloric acid (60%)	60 mL	35 to 65	15 to 60 s	nickel-cathode
				12 to 35	15 to 60 s	
I-2	stainless steel and aluminum	ethanol (95%)	800 mL	35 to 80	15 to 60 s	
I-3	stainless steel	perchloric acid (60%)	200 mL	30 to 45	15 to 60 s	
		ethanol (95%)	940 mL			
I-4	steel, cast iron, Al, Al alloys, Ni, Sn, Ag, Be, Ti, Zr, U, heat-resisting alloys	perchloric acid (65%)	60 mL	30 to 65	15 to 60 s	one of the best formulas for universal use
		ethanol (95%) 2-butoxy ethanol	700 mL-100 mL			
I-5	steels—stainless, alloy, high-speed; Fe, Al, Zr, Pb	perchloric acid (30%)	200 mL	15 to 50	15 to 60 s	universal electrolyte comparable to I-4
		ethanol (95%) glycerin perchloric acid (30%)	700 mL-100 mL			
I-6	Al, Al-Si alloys	acid (30%)	200 mL	35 to 60	15 to 60 s	particularly good with Al-Si alloys
		ethanol (95%) diethyl ether	760 mL-190 mL			
I-7	Mo, Ti, Zr, U-Zr alloy	perchloric acid (30%)	50 mL	60 to 150	5 to 30 s	
		methanol (absolute) 2-butoxy ethanol perchloric acid (60%)	600 mL-370 mL			
I-8	Al-Si alloys	perchloric acid (60%)	30 mL	50 to 100	5 to 60 s	
		methanol (absolute) glycerin	840 mL-125 mL			
I-9	vanadium germanium titanium zirconium	perchloric acid (65%)	35 mL	30	3 s	three-second cycles repeated at least seven times to prevent heating
		methanol (absolute) 2-butoxy ethanol perchloric acid (65%)	600 mL-350 mL			
			60 mL	25 to 35-58 to 30 to 60 s-45	66-70 to 75 s-15 s	polish only polish and etch simultaneously

TABLE 1—Continued

Class	Use	Formula	Cell Voltage	Time	Remarks	
I-10	aluminum	methanol (absolute) nitric acid perchloric acid (60%)	950 mL 15 mL 50 mL	30 to 60	15 to 60 s	
I-11	steels—carbon, alloy, stainless Ti, high-temperature alloys, Pb, Mo	methanol (absolute) butylcellosolve perchloric acid	600 mL 360 mL 60 mL	30–40	5–60 s	good all-purpose electropolish
I-12	Al and Al alloys	ethanol (95%) perchloric acid	1000 mL 200 mL 10		2 min	not good for Al-Cu and Al-Si alloys. Black film forms. Peel off after 1–1.5 min and polish 1 min more. Mix ethanol and water, add perchloric acid carefully. Then, add butylcellosolve before use.
I-13	steel, Al, Ni, Sn, Ti, Be stainless steel Al ₃ Ni	ethanol (95%) butylcellosolve water perchloric acid	700 mL 100 mL 137 mL 62 mL	20	20 s	
I-14	Ni, Ag, or Cu alloys Gd	ethanol (95%) butylcellosolve perchloric acid	700 mL 100 mL 200 mL	70–80	15 s	
I-15	Mo and Mo alloys	methanol (absolute) water butylcellosolve perchloric acid	600 mL 13 mL 360 mL 47 mL		20 s	Mix methanol and water, add perchloric acid carefully. Add butylcellosolve before use.
Group II (Electrolytes Composed of Perchloric Acid and Glacial Acetic Acid in Varying Proportions)						
II-1	Cr, Ti, Zr, U, Fe, steel—carbon, alloy, stainless	acetic acid (glacial) perchloric acid (60%)	940 mL 60 mL	20 to 60	1 to 5 min	good general-purpose electrolyte
II-2	Zr, Ti, U, steel—carbon and alloy	acetic acid (glacial) perchloric acid (60%)	900 mL 100 mL	12 to 70	0.5 to 2 min	
II-3	U, Zr, Ti, Al, steel—carbon and alloy	acetic acid (glacial) perchloric acid (60%)	800 mL 200 mL	40 to 100	1 to 15 min	
II-4	Ni, Pb, Pb-Sb alloys	acetic acid (glacial) perchloric acid (60%)	700 mL 300 mL	40 to 100	1 to 5 min	
II-5	3-percent Si-Fe	acetic acid (glacial) perchloric acid (60%)	650 mL 350 mL	∞	5 min	0.06 A/cm ²
II-6	Cr	acetic acid (glacial) perchloric acid	1000 mL 5 mL	30–50	2–3 min	can lower voltage to 25 V by adding 5–15% water.
II-7	Hf, steel—carbon and alloy	acetic acid (glacial) perchloric acid	1000 mL 50 mL	∞	∞	Used to polish Hf wires.
Group III (Electrolytes Composed of Phosphoric Acid in Water or Organic Solvent)						
III-1	cobalt	phosphoric acid (85%)	1000 mL	1–2	3 to 5 min	
III-2	pure copper	distilled water phosphoric acid (85%)	175 mL 825 mL	1.0 to 1.6	10 to 40 min	copper cathode
III-3	stainless, brass, Cu and Cu alloys except Sn-bronze	water phosphoric acid (85%)	300 mL 700 mL	1.5 to 1.8	5 to 15 min	copper cathode
III-4	alpha or alpha plus beta brass, Cu-Fe, Cu-Co, Co, Cd	water phosphoric acid (85%)	600 mL 400 mL	1 to 2	1 to 15 min	copper or stainless steel cathode
III-5	Cu, Cu-Zn	water pyrophosphoric acid	1000 mL 580 g	1 to 2	10 min	copper cathode
III-6	steel	diethylene glycol monoethyl ether phosphoric acid (85%)	500 mL 500 mL	5 to 20	5 to 15 min	120°F
III-7	Al, Ag, Mg	water ethanol (95%) phosphoric acid (85%)	200 mL 380 mL 400 mL	25 to 30	4 to 6 min	aluminum cathode, 100 to 110°F
III-8	uranium	ethanol (absolute) glycerin (cp) phosphoric acid (85%)	300 mL 300 mL 300 mL	∞	∞	
III-9	Mn, Mn-Cu alloys	ethanol (95%) glycerin phosphoric acid (85%)	500 mL 250 mL 250 mL	18	∞	
III-10	Cu and Cu-base alloys	distilled water ethanol (95%) phosphoric acid (85%)	500 mL 250 mL 250 mL	∞	1 to 5 min	
III-11	stainless steel	ethanol (absolute), to pyrophosphoric acid	1 L 400 g	∞	10 min	good for all austenitic heat resistant alloys, 100°F plus
III-12	Mg-Zn	ethanol (95%) phosphoric acid (85%)	625 mL 375 mL	1.5 to 2.5	3 to 30 min	
III-13	uranium	ethanol (95%) ethylene glycol phosphoric acid (85%)	445 mL 275 mL 275 mL	18 to 20	5 to 15 min	0.03 A/cm ²
III-14	Al-Mg alloys	water ethanol (95%) phosphoric acid (85%)	250 mL 380 mL 400 mL	50–60	2 min	
III-15	Cu-Pb alloys	ethanol (95%) phosphoric acid (85%)	620 mL 380 mL			good up to 30% Pb
III-16	Neptunium	ethanol (95%) glycerol phosphoric acid (85%)	400 mL 400 mL 800 mL			after 600-grit SiC, use 6-μm diamond on nylon before electropolishing.
Group IV (Electrolytes Composed of Sulfuric Acid in Water or Organic Solvent)						
IV-1	stainless steel	water sulfuric acid	250 mL 750 mL	1.5 to 6	1 to 2 min	
IV-2	stainless steel, Fe, Ni	water sulfuric acid	400 mL 600 mL	1.5 to 6	2 to 6 min	
IV-3	stainless steel, Fe, Ni, Mo	water sulfuric acid	750 mL 250 mL	1.5 to 6	2 to 10 min 0.3 to 1 min	particularly good for sintered Mo—32 to 80°F
IV-4	molybdenum	water sulfuric acid	900 mL 100 mL	1.5 to 6	0.3 to 2 min	particularly good for sintered Mo—32 to 80°F

TABLE 1—Continued

Class	Use	Formula	Cell Voltage	Time	Remarks	
IV-5	stainless steel	water-glycerin-sulfuric acid 70 mL 200 mL 720 mL	1.5 to 6	0.5 to 5 min		
IV-6	stainless steel, aluminum	water-glycerin-sulfuric acid 220 mL 200 mL 580 mL	1.5 to 12	1 to 20 min		
IV-7	molybdenum	methanol (absolute)-sulfuric acid	6 to 18	0.5 to 1.5 min	32 to 80°F	
IV-8	Ni-base superalloys	methanol (absolute)-sulfuric acid	800 mL 200 mL 30	20 s	for alloy-625	
Group V (Electrolytes Composed of Chromic Acid in Water)						
V-1	stainless steel	water-chromic acid	830 mL 620 g	1.5 to 9	2 to 10 min	
V-2	Zn, brass	water-chromic acid	830 mL 170 g	1.5 to 12	10 to 60 s	
Group VI (Mixed Acids or Salts in Water or Organic Solvent)						
VI-1	stainless steel	phosphoric acid (85%)-sulfuric acid	600 mL 400 mL		
VI-2	stainless steel	water-phosphoric acid (85%)-sulfuric acid	150 mL 300 mL ... 550 mL	2 min	0.3 A/cm ²	
VI-3	stainless and alloy steel	water-phosphoric acid (85%)-sulfuric acid	240 mL 420 mL ... 340 mL	2 to 10 min	0.1 to 0.2 A/cm ²	
VI-4	stainless steel	water-phosphoric acid (85%)-sulfuric acid	330 mL 550 mL ... 120 mL	1 min	0.05 A/cm ²	
VI-5	bronze (to 9% Sn)	water-phosphoric acid (85%)-sulfuric acid	450 mL 390 mL ... 160 mL	1 to 5 min	0.1 A/cm ²	
VI-6	bronze (to 6% Sn)	water-phosphoric acid (85%)-sulfuric acid	330 mL 580 mL ... 90 mL	1 to 5 min	0.1 A/cm ²	
VI-7	steel	water-glycerin-phosphoric acid (85%)-sulfuric acid	140 mL 100 mL ... 430 mL 330 mL	1 to 5 min	1 to 5 A/cm ² , 100°F plus	
VI-8	stainless steel	water-glycerin-phosphoric acid (85%)-sulfuric acid	200 mL 590 mL ... 100 mL 110 mL	5 min	1 A/cm ² , 80 to 120°F	
VI-9	stainless steel	water-chromic acid-phosphoric acid (85%)-sulfuric acid	260 mL 175 g ... 175 mL 580 mL	30 min	0.6 A/cm ² , 80 to 120°F	
VI-10	stainless steel	water-chromic acid-phosphoric acid (85%)-sulfuric acid	175 mL 195 g ... 460 mL 390 mL	60 min	0.5 A/cm ² , 80 to 120°F	
VI-11	stainless and alloy steel	water-chromic acid-phosphoric acid (85%)-sulfuric acid	240 mL 80 g 650 ... mL 130 mL	5 to 60 min	0.5 to A/cm ² , 100 to 130°F	
VI-12	tantalum	hydrofluoric acid-sulfuric acid	100 mL 900 mL ...	9 min	graphite cathode, 0.1 A/cm ² , 90 to 100°F	
VI-13	stainless steel	water-hydrofluoric acid-sulfuric acid	210 mL 180 mL ... 610 mL	5 min	0.5 A/cm ² , 70 to 120°F	
VI-14	zinc	water-chromic acid-sulfuric acid-sodium dichromate-acetic acid (glacial)	800 mL 100 g 46 ... mL 310 g 96 mL	...	0.002 A/cm ² , 70 to 100°F	
VI-15	stainless steel	hydrogen peroxide (30%)-sulfuric acid	260 mL 240 mL ... 500 mL	5 min	0.5 A/cm ² (Caution) Dangerous	
VI-16	stainless steel	water-hydrofluoric acid-sulfuric acid	520 mL 80 mL ... 400 mL	½ to 4 min	0.08 to 0.3 A/cm ²	
VI-17	stainless steel	water-chromic acid-nitric acid-hydrochloric acid-sulfuric acid	600 mL 180 g 60 ... mL 3 mL 240 mL	...		
VI-18	bismuth	glycerin-acetic acid (glacial)-nitric acid	750 mL 125 mL 125 mL	12	1 to 5 min	0.5 ± A/cm ² (Caution) This mixture will decompose vigorously after a short time. Do not try to keep.
VI-19	magnesium	ethylene glycol-monoethyl-ether-hydrochloric acid	900 mL 100 mL	50 to 60	10 to 30 s	Bath should be stirred. Cool-cracked ice below 35°F
VI-20	molybdenum, sintered and cast	methanol (absolute)-hydrochloric acid-sulfuric acid	685 mL 225 mL 90 mL	19 to 35	20 to 35 s	Mix slowly. Heat is developed. Avoid contamination with water. Below 35°F.
Group VI (Mixed Acids or Salts in Water or Organic Solvent)—Continued						
VI-21	titanium	ethanol (95%)-n-butyl alcohol-aluminum chloride (anhydrous)-zinc chloride (anhydrous)	900 mL 100 mL 60 g 250 g	30 to 60	1 to 6 min	(Caution) Anhydrous aluminum chloride is extremely dangerous to handle.
VI-22	uranium	acetic acid (glacial)-distilled water-chromic acid	750 mL 210 mL 180 g	80	5 to 30 min	The chromic acid is dissolved in the water before adding to the acetic acid. Below 35°F.
VI-23	pure zinc	ethanol (95%)-aluminum chloride (anhydrous)-zinc chloride (anhydrous)-distilled water-n-butyl alcohol	720 mL 50 g 225 g 160 mL 80 mL	25 to 40	0.5 to 3 min	(Caution) Anhydrous aluminum chloride is extremely dangerous to handle. Below 60°F.
VI-24	zirconium. Polish and etch simultaneously	glycerin-(Caution) hydrofluoric acid-nitric acid	870 mL 43 mL 87 mL	9 to 12	1 to 10 min	(Caution) will decompose on standing, dangerous if kept too long
VI-25	bismuth	saturated solution KI in distilled water-hydrochloric acid	980 mL 20 mL	7	30 s	polish 30 s but allow to remain in electrolyte until brown film is dissolved

TABLE 1—Continued

Class	Use	Formula	Cell Voltage	Time	Remarks	
VI-26	Sb	methanol (absolute) sulfuric acid hydrochloric acid	300 mL 50 mL 30 mL	6–10	2–4 min	pure Sb. Use Pt cathode and anode lead wires. Agitate bath. Do not touch polished surface with cotton. good for polarized light work
VI-27	Sb	ethanol (95%) glycerol phosphoric acid-sulfuric acid	30 mL 30 mL 100 mL 30 mL			good for polarized light work
VI-28	Bi	water-phosphoric acid-sulfuric acid	200 mL 100 mL 200 mL			good for polarized light work
VI-29	Cr	water-phosphoric acid-sulfuric acid	210 mL 640 mL 150 mL	18		stir bath or specimen
VI-30	Ge	methanol (absolute) hydrochloric acid	1000 mL 10 mL			
VI-31	Nb	water-sulfuric acid-hydrofluoric acid	300 mL 100 mL 100 mL	40		polish to α -alumina before electropolishing
VI-32	Nb	methanol (absolute) sulfuric acid hydrofluoric acid	940 mL 50 mL 15 mL	50–60	10 s	
VI-33	Ni-base superalloy	methanol (absolute) hydrochloric acid	170 mL 30 mL	30	20 s	for Waspaloy and IN-100 mod. Etch at 5 V for 4 s.
Group VII (Alkaline Electrolytes)						
VII-1	gold	water-to-potassium cyanide potassium carbonate-gold chloride	1000 mL 80 g 407.5 g 50 g	407.5	2 to 4 min	graphite cathode
VII-2	silver	water-to-sodium cyanide potassium ferrocyanide	1000 mL 100 g 100 g	2.5	To 1 min	graphite cathode
VII-3	silver	water-to-potassium cyanide-silver cyanide-potassium dichromate	1000 mL 400 g 280 g 280 g	∞	To 9 min	graphite cathode, 0.003 to 0.009 A/cm ²
VII-4	tungsten	water-to-trisodium phosphate	1000 mL 160 g	∞	10 min	graphite cathode, 0.09 A/cm ² , 100 to 120 F
VII-5	tungsten, lead	water-to-sodium hydroxide	1000 mL 100 g	∞	8 to 10 min	graphite cathode, 0.03 to 0.06 A/cm ²
VII-6	zinc, tin	water-to-potassium hydroxide	1000 mL 200 g	2 to 6	15 min	copper cathode, 0.1 to 0.2 A/cm ²
VII-7	W	water-sodium hydroxide	1000 mL 20 g		5 min	
Group VIII (Mixture of Methyl Alcohol and Nitric Acid)						
VIII-1	Ni, Cu, Zn, Monel, brass, Ni-chrome, stainless steel	methanol (absolute) nitric acid	660 mL 330 mL	40 to 70	10 to 60 s	very useful but dangerous

includes specific safety precautions for the mixing or use of some electrolytes. The user should take care to observe each of these specific precautions.

5.2 Some basic suggestions for the handling and disposal of electrolytes and their ingredients are as follows:

5.2.1 When pouring, mixing, or using electrolytes, always use the proper protective equipment (eyewear, gloves, apron, and so forth):

5.2.2 Use proper devices (glass or plastic) for weighing, measuring, mixing, containing, and storage of solutions.

5.2.3 When mixing electrolytes, always add reagents to the solvent unless specific instructions indicate otherwise.

5.2.4 When using an electrolyte, always avoid direct physical contact with the electrolyte and the specimen. Use tongs or some other indirect method of handling specimens.

5.2.5 In general, it is good practice to work under a properly designed chemical fume hood, and it is imperative with those electrolytes that give off noxious odors or toxic vapors.

5.2.1 As previously stated, it is good practice to always work under a certified fume hood when mixing and utilizing any electrolyte and it is imperative with those electrolytes that give off noxious odors or toxic vapor. Additionally, the electrolytes in Groups I and II must be treated with extra caution because dried perchlorates can accumulate in hood ductwork and on work surfaces creating the potential for a powerful accidental explosion. Therefore, these electrolytes must only be used in an exclusive hood equipped with a wash down feature. To avoid the accumulation of explosive, dry perchlorates, the hood should undergo a wash down cycle following each use.

5.2.2 When pouring, mixing, or using electrolytes, always use the proper protective equipment (eyewear, gloves, apron, and so forth):

5.2.3 Use proper devices (glass or plastic) for weighing, measuring, mixing, containing, and storage of solutions.

5.2.4 When mixing electrolytes, always add reagents to the solvent unless specific instructions indicate otherwise.

5.2.5 When using an electrolyte, always avoid direct physical contact with the electrolyte and the specimen. Use tongs or some other indirect method of handling specimens.

5.2.6 Methanol is a cumulative poison hazard. Where ethanol or methanol are listed as alternates, ethanol is the preferred solvent. Methanol should be used in a properly designed chemical fume hood.

5.2.7 All spills should be cleaned up and disposed of properly, no matter how small the spill.

5.2.8 Properly dispose of all solutions that are not identified by composition and concentration.

5.2.9 Store, handle, and dispose of chemicals according to the manufacturer's recommendations. Observe printed cautions on reagent containers.

5.2.10 Information pertaining to the toxicity hazards and working precautions of chemicals, solvents, acids, bases, and so forth, being used (such as MSDS) should be available for rapid consultation.

TABLE 1 *Continued*

Class	Use	Formula	Cell Voltage	Time	Remarks
Group I (Electrolytes Composed of Perchloric Acid and Alcohol With or Without Organic Additions) Use in a washdown/perchloric rated fume hood.					
I-1	Al and Al alloys with less than 2 percent Si	ethanol (95 %) distilled water perchloric acid (60 %)	800 mL 140 mL 60 mL	30 to 80	15 to 60 s
	steels—carbon, alloy, stainless Pb, Pb-Sn, Pb-Sn-Cd, Pb-Sn-Sb Zn, Zn-Sn-Fe, Zn-Al-Cu Mg and high Mg alloys			35 to 65 12 to 35 20 to 60	15 to 60 s 15 to 60 s ...
I-2	stainless steel and aluminum	ethanol (95 %) perchloric acid (60 %)	800 mL 200 mL	35 to 80	15 to 60 s
I-3	stainless steel	ethanol (95 %) perchloric acid (65 %)	940 mL 60 mL	30 to 45	15 to 60 s
I-4	steel, cast iron, Al, Al alloys, Ni, Sn, Ag, Be, Ti, Zr, U, heat-resisting alloys	ethanol (95 %) 2-butoxy ethanol perchloric acid (30 %)	700 mL 100 mL 200 mL	30 to 65	15 to 60 s
I-5	steels—stainless, alloy, high-speed; Fe, Al, Zr, Pb	ethanol (95 %) glycerin perchloric acid (30 %)	700 mL 100 mL 200 mL	15 to 50	15 to 60 s
I-6	Al, Al-Si alloys	ethanol (95 %) diethyl ether perchloric acid (30 %)	760 mL 190 mL 50 mL	35 to 60	15 to 60 s
I-7	Mo, Ti, Zr, U-Zr alloy	methanol (absolute) 2-butoxy ethanol perchloric acid (60 %)	600 mL 370 mL 30 mL	60 to 150	5 to 30 s
I-8	Al-Si alloys	methanol (absolute) glycerin perchloric acid (65 %)	840 mL 125 mL 35 mL	50 to 100	5 to 60 s
I-9	vanadium	methanol (absolute) 2-butoxy ethanol perchloric acid (65 %)	590 mL 350 mL 60 mL	30	3 s
	germanium titanium zirconium			25 to 35 58 to 66 70 to 75	30 to 60 s 45 s 15 s
I-10	aluminum	methanol (absolute) nitric acid perchloric acid (60 %)	950 mL 15 mL 50 mL	30 to 60	15 to 60 s
I-11	steels—carbon, alloy, stainless Ti, high-temperature alloys, Pb, Mo	methanol (absolute) butylcellosolve perchloric acid	600 mL 360 mL 60 mL	30–40	5–60 s
I-12	Al and Al alloys	ethanol (95 %) perchloric acid	1000 mL 200 mL	10	2 min
I-13	steel, Al, Ni, Sn, Ti, Be stainless steel Al ₃ Ni	ethanol (95 %) butylcellosolve water perchloric acid	700 mL 100 mL 137 mL 62 mL	20	20 s
I-14	Ni, Ag, or Cu alloys Cd	ethanol (95 %) butylcellosolve perchloric acid	700 mL 100 mL 200 mL	70–80	15 s
I-15	Mo and Mo alloys	methanol (absolute) water butylcellosolve perchloric acid	600 mL 13 mL 360 mL 47 mL		20 s
Group II (Electrolytes Composed of Perchloric Acid and Glacial Acetic Acid in Varying Proportions) Use in a washdown/perchloric rated fume hood.					
II-1	Cr, Ti, Zr, U, Fe, steel—carbon, alloy, stainless	acetic acid (glacial) perchloric acid (60 %)	940 mL 60 mL	20 to 60	1 to 5 min
II-2	Zr, Ti, U, steel—carbon and alloy	acetic acid (glacial) perchloric acid (60 %)	900 mL 100 mL	12 to 70	0.5 to 2 min
II-3	U, Zr, Ti, Al, steel—carbon and alloy	acetic acid (glacial) perchloric acid (60 %)	800 mL 200 mL	40 to 100	1 to 15 min
II-4	Ni, Pb, Pb-Sb alloys	acetic acid (glacial) perchloric acid (60 %)	700 mL 300 mL	40 to 100	1 to 5 min
II-5	3 percent Si-Fe	acetic acid (glacial) perchloric acid (60 %)	650 mL 350 mL	...	5 min
II-6	Cr	acetic acid (glacial) perchloric acid	1000 mL 5 mL	30–50	2–3 min
II-7	Hf, steel—carbon and alloy	acetic acid (glacial) perchloric acid	1000 mL 50 mL
Group III (Electrolytes Composed of Phosphoric Acid in Water or Organic Solvent)					
III-1	cobalt	phosphoric acid (85 %)	1000 mL	1.2	3 to 5 min
III-2	pure copper	distilled water phosphoric acid (85 %)	175 mL 825 mL	1.0 to 1.6	10 to 40 min