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StandardGuide for Electrolytic Polishing of Metallographic Specimens[1]

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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 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]
E7 Terminology Relating to Metallography
E407 Practice for Microetching Metals and Alloys

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 *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:*

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 E407.

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.

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[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.

4.2.5 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 μm/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 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.

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

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.

5.3 Many of the electrolytes in the following listing can be exceedingly dangerous if carelessly handled. The pertinent safety precautions for each class of electrolyte should be read before any electrolyte is mixed or used.

5.4 Electrolytes containing perchloric acid and acetic anhydride are very dangerous to mix and may be unpredictable in use. Many industrial firms and research laboratories forbid the use of such mixtures. Certain cities also have ordinances prohibiting the use of such potentially explosive mixtures. These facts are considered sufficient reason for recommending against their use.

5.5 Mixtures of oxidizable organic compounds and powerful oxidizing agents are always potentially dangerous. After some use, any electrolyte will become heavily laden with ions of the metals polished. These ions may interfere with further polishing or catalyze the decomposition of the electrolyte. The electrolyte then must be discarded in accordance with appropriate regulations.

5.6 Most electrolytes (with few exceptions) should be mixed and stored in clean glass containers and never be in contact with foreign materials or organic compounds. The exceptions are those electrolytes containing fluorides and strong alkaline solutions that should be mixed and stored in polyethylene or other appropriate material containers. Electrolytes must never be allowed to become concentrated by evaporation. All electrolytes should be discarded appropriately as soon as they have exceeded their immediate usefulness.

5.7 Specimens mounted in bismuth or bismuth-containing metals must not be electropolished in perchloric acid solutions because this mounting medium may react explosively with the electrolyte. Likewise, bismuth or bismuth-containing alloys must not be electropolished in solutions containing perchloric

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) Use in a washdown/perchloric rated fume hood.						
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			
		perchloric acid (60 %)	60 mL			
	steels—carbon, alloy, stainless			35 to 65	15 to 60 s	
	Pb, Pb-Sn, Pb-Sn-Cd, Pb-Sn-Sb			12 to 35	15 to 60 s	
	Zn, Zn-Sn-Fe, Zn-Al-Cu			20 to 60	...	
	Mg and high Mg alloys			...	...	nickel cathode
I-2	stainless steel and aluminum	ethanol (95 %)	800 mL	35 to 80	15 to 60 s	
		perchloric acid (60 %)	200 mL			
I-3	stainless steel	ethanol (95 %)	940 mL	30 to 45	15 to 60 s	
		perchloric acid (65 %)	60 mL			
I-4	steel, cast iron, Al, Al alloys, Ni, Sn, Ag, Be, Ti, Zr, U, heat-resisting alloys	ethanol (95 %)	700 mL	30 to 65	15 to 60 s	one of the best formulas for universal use
		2-butoxy ethanol	100 mL			
		perchloric acid (30 %)	200 mL			
I-5	steels—stainless, alloy, high-speed; Fe, Al, Zr, Pb	ethanol (95 %)	700 mL	15 to 50	15 to 60 s	universal electrolyte comparable to I-4
		glycerin	100 mL			
		perchloric acid (30 %)	200 mL			
I-6	Al, Al-Si alloys	ethanol (95 %)	760 mL	35 to 60	15 to 60 s	particularly good with Al-Si alloys
		diethyl ether	190 mL			
		perchloric acid (30 %)	50 mL			
I-7	Mo, Ti, Zr, U-Zr alloy	methanol (absolute)	600 mL	60 to 150	5 to 30 s	
		2-butoxy ethanol	370 mL			
		perchloric acid (60 %)	30 mL			
I-8	Al-Si alloys	methanol (absolute)	840 mL	50 to 100	5 to 60 s	
		glycerin	125 mL			
		perchloric acid (65 %)	35 mL			
I-9	vanadium	methanol (absolute)	590 mL	30	3 s	three-second cycles repeated at least seven times to prevent heating
		2-butoxy ethanol	350 mL			
		perchloric acid (65 %)	60 mL			
	germanium			25 to 35	30 to 60 s	
	titanium			58 to 66	45 s	polish only
	zirconium			70 to 75	15 s	polish and etch simultaneously
I-10	aluminum	methanol (absolute)	950 mL	30 to 60	15 to 60 s	
		nitric acid	15 mL			
		perchloric acid (60 %)	50 mL			
I-11	steels—carbon, alloy, stainless Ti, high-temperature alloys, Pb, Mo	methanol (absolute)	600 mL	30–40	5–60 s	good all purpose electropolish
		butylcellosolve	360 mL			
		perchloric acid	60 mL			
I-12	Al and Al alloys	ethanol (95 %)	1000 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.
		perchloric acid	200 mL			
I-13	steel, Al, Ni, Sn, Ti, Be stainless steel Al_3 Ni	ethanol (95 %)	700 mL	20	20 s	Mix ethanol and water, add perchloric acid carefully. Then, add butylcellosolve before use.
		butylcellosolve	100 mL			
		water	137 mL			
		perchloric acid	62 mL			
I-14	Ni, Ag, or Cu alloys Cd	ethanol (95 %)	700 mL	70–80	15 s	
		butylcellosolve	100 mL			
		perchloric acid	200 mL			
I-15	Mo and Mo alloys	methanol (absolute)	600 mL		20 s	Mix methanol and water, add perchloric acid carefully. Add butylcellosolve before use.
		water	13 mL			
		butylcellosolve	360 mL			
		perchloric acid	47 mL			
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)	940 mL	20 to 60	1 to 5 min	good general-purpose electrolyte
		perchloric acid (60 %)	60 mL			
II-2	Zr, Ti, U, steel—carbon and alloy	acetic acid (glacial)	900 mL	12 to 70	0.5 to 2 min	
		perchloric acid (60 %)	100 mL			
II-3	U, Zr, Ti, Al, steel—carbon and alloy	acetic acid (glacial)	800 mL	40 to 100	1 to 15 min	
		perchloric acid (60 %)	200 mL			
II-4	Ni, Pb, Pb-Sb alloys	acetic acid (glacial)	700 mL	40 to 100	1 to 5 min	
		perchloric acid (60 %)	300 mL			
II-5	3 percent Si-Fe	acetic acid (glacial)	650 mL	...	5 min	0.06 A/cm^2
		perchloric acid (60 %)	350 mL			
II-6	Cr	acetic acid (glacial)	1000 mL	30–50	2–3 min	can lower voltage to 25 V by adding 5–15 % water.
		perchloric acid	5 mL			
II-7	Hf, steel—carbon and alloy	acetic acid (glacial)	1000 mL	...	...	Used to polish Hf wires.
		perchloric acid	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	175 mL	1.0 to 1.6	10 to 40 min	copper cathode
		phosphoric acid (85 %)	825 mL			
III-3	stainless, brass, Cu and Cu alloys except Sn bronze	water	300 mL	1.5 to 1.8	5 to 15 min	copper cathode
		phosphoric acid (85 %)	700 mL			

TABLE 1 *Continued*

Class	Use	Formula		Cell Voltage	Time	Remarks
III-4	alpha or alpha plus beta brass, Cu-Fe, Cu-Co, Co, Cd	water	600 mL	1 to 2	1 to 15 min	copper or stainless steel cathode
		phosphoric acid (85 %)	400 mL			
III-5	Cu, Cu-Zn	water	1000 mL	1 to 2	10 min	copper cathode
		pyrophosphoric acid	580 g			
III-6	steel	diethylene glycol monoethyl ether	500 mL	5 to 20	5 to 15 min	49°C
		phosphoric acid (85 %)	500 mL			
III-7	Al, Ag, Mg	water	200 mL	25 to 30	4 to 6 min	aluminum cathode, 38 to 43°C
		ethanol (95 %)	380 mL			
		phosphoric acid (85 %)	400 mL			
III-8	uranium	ethanol (absolute)	300 mL	...	...	
		glycerin (cp)	300 mL			
		phosphoric acid (85 %)	300 mL			
III-9	Mn, Mn-Cu alloys	ethanol (95 %)	500 mL	18	...	
		glycerin	250 mL			
		phosphoric acid (85 %)	250 mL			
III-10	Cu and Cu-base alloys	distilled water	500 mL	...	1 to 5 min	
		ethanol (95 %)	250 mL			
		phosphoric acid (85 %)	250 mL			
III-11	stainless steel	ethanol (absolute), to	1 L	...	10 min	good for all austenitic heat resistant alloys, 38°C plus
		pyrophosphoric acid	400 g			
III-12	Mg-Zn	ethanol (95 %)	625 mL	1.5 to 2.5	3 to 30 min	
		phosphoric acid (85 %)	375 mL			
III-13	uranium	ethanol (95 %)	445 mL	18 to 20	5 to 15 min	0.03 A/cm^2
		ethylene glycol	275 mL			
		phosphoric acid (85 %)	275 mL			
III-14	Al-Mg alloys	water	250 mL	50–60	2 min	
		ethanol (95 %)	380 mL			
		phosphoric acid (85 %)	400 mL			
III-15	Cu-Pb alloys	ethanol (95 %)	620 mL			good up to 30 % Pb
		phosphoric acid (85 %)	380 mL			
III-16	Neptunium	ethanol (95 %)	400 mL			after P1200–grit SiC, use 6-µm diamond on nylon before electropolishing.
		glycerol	400 mL			
		phosphoric acid (85 %)	800 mL			
	Group IV (Electrolytes Composed of Sulfuric Acid in Water or Organic Solvent)					
IV-1	stainless steel	water	250 mL	1.5 to 6	1 to 2 min	
		sulfuric acid	750 mL			
IV-2	stainless steel, Fe, Ni	water	400 mL	1.5 to 6	2 to 6 min	
		sulfuric acid	600 mL			
IV-3	stainless steel, Fe, Ni, Mo	water	750 mL	1.5 to 6	2 to 10 min	particularly good for sintered Mo—
		sulfuric acid	250 mL		Mo—	0 to 27°C
					0.3 to 1 min	
IV-4	molybdenum	water	900 mL	1.5 to 6	0.3 to 2 min	particularly good for sintered Mo—
		sulfuric acid	100 mL			0 to 27°C
IV-5	stainless steel	water	70 mL	1.5 to 6	0.5 to 5 min	
		glycerin	200 mL			
		sulfuric acid	720 mL			
IV-6	stainless steel, aluminum	water	220 mL	1.5 to 12	1 to 20 min	
		glycerin	200 mL			
		sulfuric acid	580 mL			
IV-7	molybdenum	methanol (absolute)	875 mL	6 to 18	0.5 to 1.5 min	0 to 27°C
		sulfuric acid	125 mL			
IV-8	Ni-base superalloys	methanol (absolute)	800 mL	30	20 s	for alloy 625
		sulfuric acid	200 mL			
	Group V (Electrolytes Composed of Chromic Acid in Water)					
V-1	stainless steel	water	830 mL	1.5 to 9	2 to 10 min	
		chromic acid	620 g			
V-2	Zn, brass	water	830 mL	1.5 to 12	10 to 60 s	
		chromic acid	170 g			
	Group VI (Mixed Acids or Salts in Water or Organic Solvent)					
VI-1	stainless steel	phosphoric acid (85 %)	600 mL	...	...	
		sulfuric acid	400 mL			
VI-2	stainless steel	water	150 mL	...	2 min	0.3 A/cm^2
		phosphoric acid (85 %)	300 mL			
		sulfuric acid	550 mL			
VI-3	stainless and alloy steel	water	240 mL	...	2 to 10 min	0.1 to 0.2 A/cm^2
		phosphoric acid (85 %)	420 mL			
		sulfuric acid	340 mL			
VI-4	stainless steel	water	330 mL	...	1 min	0.05 A/cm^2
		phosphoric acid (85 %)	550 mL			
		sulfuric acid	120 mL			
VI-5	bronze (to 9 % Sn)	water	450 mL	...	1 to 5 min	0.1 A/cm^2
		phosphoric acid (85 %)	390 mL			
		sulfuric acid	160 mL			

TABLE 1 *Continued*

Class	Use	Formula		Cell Voltage	Time	Remarks
VI-6	bronze (to 6 % Sn)	water	330 mL	...	1 to 5 min	0.1 A/cm^2
		phosphoric acid (85 %)	580 mL			
		sulfuric acid	90 mL			
VI-7	steel	water	140 mL	...	1 to 5 min	1 to 5 A/cm^2, 38°C plus
		glycerin	100 mL			
		phosphoric acid (85 %)	430 mL			
		sulfuric acid	330 mL			
VI-8	stainless steel	water	200 mL	...	5 min	1 A/cm^2, 27 to 49°C
		glycerin	590 mL			
		phosphoric acid (85 %)	100 mL			
		sulfuric acid	110 mL			
VI-9	stainless steel	water	260 mL	...	30 min	0.6 A/cm^2, 27 to 49°C
		chromic acid	175 g			
		phosphoric acid (85 %)	175 mL			
		sulfuric acid	580 mL			
VI-10	stainless steel	water	175 mL	...	60 min	0.5 A/cm^2, 27 to 49°C
		chromic acid	105 g			
		phosphoric acid (85 %)	460 mL			
		sulfuric acid	390 mL			
VI-11	stainless and alloy steel	water	240 mL	...	5 to 60 min	0.5 to A/cm^2, 38 to 54°C
		chromic acid	80 g			
		phosphoric acid (85 %)	650 mL			
		sulfuric acid	130 mL			
VI-12	tantalum	hydrofluoric acid	100 mL	...	9 min	graphite cathode, 0.1 A/cm^2, 32 to 38°C
		sulfuric acid	900 mL			
VI-13	stainless steel	water	210 mL	...	5 min	0.5 A/cm^2, 21 to 49°C
		hydrofluoric acid	180 mL			
		sulfuric acid	610 mL			
VI-14	zinc	water	800 mL	...	...	0.002 A/cm^2, 21 to 49°C
		chromic acid	100 g			
		sulfuric acid	46 mL			
		sodium dichromate	310 g			
		acetic acid (glacial)	96 mL			
VI-15	stainless steel	hydrogen peroxide (30 %) **(Caution)**	260 mL	...	5 min	0.5 A/cm^2 **(Caution) Dangerous**
		hydrofluoric acid	240 mL			
		sulfuric acid	500 mL			
VI-16	stainless steel	water	520 mL	...	½ to 4 min	0.08 to 0.3 A/cm^2
		hydrofluoric acid	80 mL			
		sulfuric acid	400 mL			
VI-17	stainless steel	water	600 mL	...	...	
		chromic acid	180 g			
		nitric acid	60 mL			
		hydrochloric acid	3 mL			
		sulfuric acid	240 mL			
VI-18	bismuth	glycerin	750 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.
		acetic acid (glacial)	125 mL			
		nitric acid	125 mL			
VI-19	magnesium	ethylene-glycol-monoethyl ether	900 mL	50 to 60	10 to 30 s	Bath should be stirred. Cool with cracked ice below 2°C
		hydrochloric acid	100 mL			
VI-20	molybdenum, sintered and cast	methanol (absolute)	685 mL	19 to 35	20 to 35 s	Mix slowly. Heat is developed. Avoid contamination with water. Use below 2°C.
		hydrochloric acid	225 mL			
		sulfuric acid	90 mL			
	Group VI (Mixed Acids or Salts in Water or Organic Solvent)— *Continued*					
VI-21	titanium	ethanol (95 %)	900 mL	30 to 60	1 to 6 min	**(Caution)** Anhydrous aluminum chloride is extremely dangerous to handle.
		n-butyl alcohol	100 mL			
		aluminum chloride (anhydrous) (add very slowly) **(Caution)**	60 g			
		zinc chloride (anhydrous)	250 g			
VI-22	uranium	acetic acid (glacial)	750 mL	80	5 to 30 min	The chromic acid is dissolved in the water before adding to the acetic acid. Use below 2°C.
		distilled water	210 mL			
		chromic acid	180 g			
VI-23	pure zinc	ethanol (95 %)	720 mL	25 to 40	0.5 to 3 min	**(Caution)** Anhydrous aluminum chloride is extremely dangerous to handle. Use below 16°C.
		aluminum chloride (anhydrous) **(Caution)**	50 g			
		zinc chloride (anhydrous)	225 g			
		distilled water	160 mL			
		n-butyl alcohol	80 mL			
VI-24	zirconium. Polish and etch simultaneously	glycerin **(Caution)**	870 mL	9 to 12	1 to 10 min	**(Caution)** will decompose on standing, dangerous if kept too long
		hydrofluoric acid	43 mL			
		nitric acid	87 mL			
VI-25	bismuth	saturated solution KI in distilled water	980 mL	7	30 s	polish 30 s but allow to remain in electrolyte until brown film is dissolved
		hydrochloric acid	20 mL			

TABLE 1 *Continued*

Class	Use	Formula		Cell Voltage	Time	Remarks
VI-26	Sb	methanol (absolute)	300 mL	6–10	2–4 min	pure Sb. Use Pt cathode and anode lead wires. Agitate bath. Do not touch polished surface with cotton.
		sulfuric acid	50 mL			
		hydrochloric acid	30 mL			
VI-27	Sb	ethanol (95 %)	30 mL			good for polarized light work
		glycerol	30 mL			
		phosphoric acid	100 mL			
		sulfuric acid	30 mL			
VI-28	Bi	water	200 mL			good for polarized light work
		phosphoric acid	100 mL			
		sulfuric acid	200 mL			
VI-29	Cr	water	210 mL	18		stir bath or specimen
		phosphoric acid	640 mL			
		sulfuric acid	150 mL			
VI-30	Ge	methanol (absolute)	1000 mL			
		hydrochloric acid	10 mL			
VI-31	Nb	water	300 mL	40		polish to α-alumina before electropolishing
		sulfuric acid	100 mL			
		hydrofluoric acid	100 mL			
VI-32	Nb	methanol (absolute)	940 mL	50–60	10 s	
		sulfuric acid	50 mL			
		hydrofluoric acid	15 mL			
VI-33	Ni-base superalloy	methanol (absolute)	170 mL	30	20 s	for Waspaloy and IN-100 mod. Etch at 5 V for 4 s.
		hydrochloric acid	30 mL			
		Group VII (Alkaline Electrolytes)				
VII-1	gold	water to	1000 mL	7.5	2 to 4 min	graphite cathode
		potassium cyanide	80 g			
		potassium carbonate	40 g			
		gold chloride	50 g			
VII-2	silver	water to	1000 mL	2.5	To 1 min	graphite cathode
		sodium cyanide	100 g			
		potassium ferrocyanide	100 g			
VII-3	silver	water to	1000 mL	...	To 9 min	graphite cathode, 0.003 to 0.009 A/cm^2
		potassium cyanide	400 g			
		silver cyanide	280 g			
		potassium dichromate	280 g			
VII-4	tungsten	water to	1000 mL	...	10 min	graphite cathode, 0.09 A/cm^2, 38 to 49°C
		trisodium phosphate	160 g			
VII-5	tungsten, lead	water to	1000 mL	...	8 to 10 min	graphite cathode, 0.03 to 0.06 A/cm^2
		sodium hydroxide	100 g			
VII-6	zinc, tin	water to	1000 mL	2 to 6	15 min	copper cathode, 0.1 to 0.2 A/cm^2
		potassium hydroxide	200 g			
VII-7	W	water	1000 mL		5 min	
		sodium hydroxide	20 g			
		Group VIII (Mixture of Methyl Alcohol and Nitric Acid)				
VIII-1	Ni, Cu, Zn, Monel, brass, Ni-chrome, stainless steel	methanol (absolute)	660 mL	40 to 70	10 to 60 s	very useful but dangerous
		nitric acid	330 mL			

acid. Specimens mounted in organic mounting compounds, such as Bakelite, must not be electropolished in electrolytes containing perchloric acid as they may also react explosively.

5.8 *Specific Safety Precautions for Each Group of Electrolytes:*

5.8.1 The electrolytes recommended for use are classified into eight groups. Their chemical components are listed in the order of mixing. This ordering has been done to prevent possibly dangerous reactions. Unless other instructions are specifically given, the electrolytes are intended to be used in the temperature range from about 18 to 27°C. Cooling may be necessary to maintain this range during use.

5.8.2 *Group I—(Electrolytes Composed of Perchloric Acid and Alcohol (Methanol or Ethanol) With or Without Organic Additions):*

5.8.2.1 These electrolytes are believed to be safe to mix and use provided the following safety precautions are followed. Use these electrolytes in an exclusive hood equipped with a wash down feature. The hood should undergo a wash down cycle following each use to avoid accumulation of explosive, dry perchlorates. Only small quantities should be mixed and stored in glass-stoppered bottles filled to capacity. Any evaporated solvents should be replaced to keep the bottle filled. Spent or exhausted polishing baths are to be promptly discarded in a manner consistent with prevailing regulations. The electrolytes are always to be protected from heat or fire.

NOTE 3—In this, and all the following formulations, the term 95 % ethanol refers to a specifically denatured alcohol which is composed of 95 parts by volume absolute ethanol and 5 parts by volume absolute methanol. In case this formulation is not available, the use of 100 % absolute ethanol is advised. Alcohol formulations containing benzene, gasoline, or other denaturing substances are likely to cause difficulties and their use is not recommended.

5.8.3 *Group II—(Electrolytes Composed of Perchloric Acid and Glacial Acetic Acid):*

5.8.3.1 Use these electrolytes in an exclusive hood equipped with a wash down feature. The hood should undergo a wash down cycle following each use to avoid accumulation of explosive, dry perchlorates. Very little heat is developed when perchloric acid is mixed with glacial acetic acid. In mixing, the