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Standard Guide for Electrolytic Polishing of Metallographic Specimens¹

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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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific safety precautions are described in Section 5 and 6.3.1.

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:*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:*

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.

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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.

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.

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 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.

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 %) distilled water perchloric acid (60 %)	800 mL 140 mL 60 mL	30 to 80	15 s 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 s to 60 s 15 s to 60 s
I-2	stainless steel and aluminum	ethanol (95 %) perchloric acid (60 %)	800 mL 200 mL	35 to 80	15 s to 60 s
I-3	stainless steel	ethanol (95 %) perchloric acid (65 %)	940 mL 60 mL	30 to 45	15 s 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 s 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 s 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 s 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 s to 30 s
I-8	Al-Si alloys	methanol (absolute) glycerin perchloric acid (65 %)	840 mL 125 mL 35 mL	50 to 100	5 s 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 s 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 s to 60 s
I-11	steels—carbon, alloy, stainless Ti, high-temperature alloys, Pb, Mo	methanol (absolute) butylcellosolve perchloric acid (60 %)	600 mL 360 mL 60 mL	30–40	5 s–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 min 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 min 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 min to 15 min
II-4	Ni, Pb, Pb-Sb alloys	acetic acid (glacial) perchloric acid (60 %)	700 mL 300 mL	40 to 100	1 min 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 min–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 min to 5 min
III-2	pure copper	distilled water phosphoric acid (85 %)	175 mL 825 mL	1.0 to 1.6	10 min to 40 min
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 min to 15 min

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 min to 15 min	copper or stainless steel cathode
III-5	Cu, Cu-Zn	phosphoric acid (85 %)	400 mL	1 to 2	10 min	copper cathode
III-6	steel	water	1000 mL			
		pyrophosphoric acid	580 g			
		diethylene glycol monoethyl ether	500 mL	5 to 20	5 min to 15 min	49 °C
III-7	Al, Ag, Mg	phosphoric acid (85 %)	500 mL			
		water	200 mL	25 to 30	4 min to 6 min	aluminum cathode, 38 °C to 43 °C
		ethanol (95 %)	380 mL			
III-8	uranium	phosphoric acid (85 %)	400 mL	
		ethanol (absolute)	300 mL			
		glycerin (cp)	300 mL			
III-9	Mn, Mn-Cu alloys	phosphoric acid (85 %)	300 mL	18	...	
		ethanol (95 %)	500 mL			
		glycerin	250 mL			
III-10	Cu and Cu-base alloys	phosphoric acid (85 %)	250 mL	...	1 min to 5 min	
		distilled water	500 mL			
		ethanol (95 %)	250 mL			
III-11	stainless steel	phosphoric acid (85 %)	250 mL	...	10 min	good for all austenitic heat resistant alloys, 38 °C plus
		ethanol (absolute), to pyrophosphoric acid	1 L			
III-12	Mg-Zn	ethanol (95 %)	400 g	1.5 to 2.5	3 min to 30 min	
		phosphoric acid (85 %)	625 mL			
III-13	uranium	ethanol (95 %)	375 mL	18 to 20	5 min to 15 min	0.03 A/cm ²
		ethylene glycol	445 mL			
		phosphoric acid (85 %)	275 mL			
III-14	Al-Mg alloys	water	275 mL	50–60	2 min	
		ethanol (95 %)	250 mL			
		phosphoric acid (85 %)	380 mL			
III-15	Cu-Pb alloys	ethanol (95 %)	400 mL			good up to 30 % Pb
		phosphoric acid (85 %)	620 mL			
III-16	Neptunium	ethanol (95 %)	380 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 min to 2 min	
		sulfuric acid	750 mL			
IV-2	stainless steel, Fe, Ni	water	400 mL	1.5 to 6	2 min to 6 min	
		sulfuric acid	600 mL			
IV-3	stainless steel, Fe, Ni, Mo	water	750 mL	1.5 to 6	2 min to 10 min	particularly good for sintered Mo— 0 °C to 27 °C
		sulfuric acid	250 mL			
IV-4	molybdenum	water	900 mL	1.5 to 6	0.3 to 1 min	particularly good for sintered Mo— 0 °C to 27 °C
		sulfuric acid	100 mL			
IV-5	stainless steel	water	70 mL	1.5 to 6	0.5 min to 5 min	
		glycerin	200 mL			
		sulfuric acid	720 mL			
IV-6	stainless steel, aluminum	water	220 mL	1.5 to 12	1 min to 20 min	
		glycerin	200 mL			
		sulfuric acid	580 mL			
IV-7	molybdenum	methanol (absolute)	875 mL	6 to 18	0.5 min to 1.5 min	0 °C 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 min to 10 min	
		chromic acid	620 g			
V-2	Zn, brass	water	830 mL	1.5 to 12	10 s 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 min to 10 min	0.1 A/cm ² 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 min 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 phosphoric acid (85 %) sulfuric acid	330 mL 580 mL 90 mL	...	1 min 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 min to 5 min	1 A/cm ² to 5 A/cm ² , 38 °C 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 , 27 °C to 49 °C
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 , 27 °C to 49 °C
VI-10	stainless steel	water chromic acid phosphoric acid (85 %) sulfuric acid	175 mL 105 g 460 mL 390 mL	...	60 min	0.5 A/cm ² , 27 °C to 49 °C
VI-11	stainless and alloy steel	water chromic acid phosphoric acid (85 %) sulfuric acid	240 mL 80 g 650 mL 130 mL	...	5 min to 60 min	0.5 to A/cm ² , 38 °C to 54 °C
VI-12	tantalum	hydrofluoric acid sulfuric acid	100 mL 900 mL	...	9 min	graphite cathode, 0.1 A/cm ² , 32 °C to 38 °C
VI-13	stainless steel	water hydrofluoric acid sulfuric acid	210 mL 180 mL 610 mL	...	5 min	0.5 A/cm ² , 21 °C to 49 °C
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 , 21 °C to 49 °C
VI-15	stainless steel	hydrogen peroxide (30 %) (Caution) hydrofluoric acid 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 A/cm ² 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 min 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 s to 30 s	Bath should be stirred. Cool with cracked ice below 2 °C
VI-20	molybdenum, sintered and cast	methanol (absolute) hydrochloric acid sulfuric acid	685 mL 225 mL 90 mL	19 to 35	20 s to 35 s	Mix slowly. Heat is developed. Avoid contamination with water. Use below 2 °C.
Group VI (Mixed Acids or Salts in Water or Organic Solvent)— <i>Continued</i>						
VI-21	titanium	ethanol (95 %) <i>n</i> -butyl alcohol aluminum chloride (anhydrous) (add very slowly) (Caution) zinc chloride (anhydrous)	900 mL 100 mL 60 g 250 g	30 to 60	1 min 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 min to 30 min	The chromic acid is dissolved in the water before adding to the acetic acid. Use below 2 °C.
VI-23	pure zinc	ethanol (95 %) aluminum chloride (anhydrous) (Caution) zinc chloride (anhydrous) distilled water <i>n</i> -butyl alcohol	720 mL 50 g 225 g 160 mL 80 mL	25 to 40	0.5 min to 3 min	(Caution) Anhydrous aluminum chloride is extremely dangerous to handle. Use below 16 °C.
VI-24	zirconium. Polish and etch simultaneously	glycerin (Caution) hydrofluoric acid nitric acid	870 mL 43 mL 87 mL	9 to 12	1 min 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	300 mL 50 mL	6–10 2 min– 4 min	pure Sb. Use Pt cathode and anode lead wires. Agitate bath. Do not touch polished surface with cotton.
VI-27	Sb	hydrochloric acid ethanol (95 %) glycerol	30 mL 30 mL 30 mL		good for polarized light work
VI-28	Bi	phosphoric acid sulfuric acid water	100 mL 30 mL 200 mL		good for polarized light work
VI-29	Cr	phosphoric acid sulfuric acid water	100 mL 200 mL 210 mL	18	stir bath or specimen
VI-30	Ge	phosphoric acid sulfuric acid	640 mL 150 mL		
VI-31	Nb	methanol (absolute) hydrochloric acid water	1000 mL 10 mL 300 mL	40	polish to α -alumina before electropolishing
VI-32	Nb	sulfuric acid hydrofluoric acid methanol (absolute)	100 mL 100 mL 940 mL	50–60	10 s
VI-33	Ni-base superalloy	sulfuric acid hydrofluoric acid methanol (absolute) hydrochloric acid	50 mL 15 mL 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 40 g 50 g	7.5	2 min 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 A/cm ² 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 , 38 °C to 49 °C
VII-5	tungsten, lead	water to sodium hydroxide	1000 mL 100 g	...	8 min to 10 min graphite cathode, 0.03 A/cm ² 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 A/cm ² 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 s to 60 s very useful but dangerous

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 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 °C 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 perchloric acid should be added to the acetic with stirring. These mixtures are normally perfectly safe to mix and use but, nonetheless, great care should be exercised with them. Temperatures must never be allowed to exceed 29 °C. They are flammable and must be guarded against fire or the evaporation of the acetic acid. Plastic parts are likely to be quickly damaged by exposure to such mixtures.

5.8.4 *Group III—(Electrolytes Composed of Phosphoric Acid in Water or Organic Solvents):*

5.8.4.1 These mixtures are generally quite easy to prepare. In mixing, they are handled exactly as a mineral acid; namely, the acid must be slowly poured into the water or solvent with constant stirring to prevent the formation of a heavy layer of acid at the bottom of the vessel. Some solid phosphoric acids are quite energetic in their combination with water, requiring extra care in their mixing.

5.8.5 *Group IV—(Electrolytes Composed of Sulfuric Acid in Water or Organic Solvents):*

5.8.5.1 The addition of sulfuric acid to water produces an extremely exothermic reaction. The acid must always be poured into the water slowly and with constant stirring. Cooling is necessary. Great care should be taken to prevent spattering. Even dilute solutions of sulfuric acid strongly attack the skin or clothing. Such solutions are also very hygroscopic. They vigorously attack most plastics. The mixtures of sulfuric acid with other inorganic acids are generally more useful as electrolytes.

5.8.6 *Group V—(Electrolytes Composed of Chromium Trioxide in Water):*

5.8.6.1 The addition of crystalline chromium trioxide (CrO_3) to water is simple, since very little heat is developed. The resulting chromic acid is a powerful oxidant. Under certain conditions it will liberate considerable quantities of free oxygen. It is generally dangerous, and possibly incendiary, in the presence of oxidizable materials. It cannot be safely mixed with most organic liquids, such as alcohols or glycerol. It can be safely mixed with certain saturated organic acids, but should not be mixed with acetic acid. Chromic acid solutions cannot be used in contact with plastic parts without their eventual destruction. Care should be taken to prevent its contact with the skin since repeated exposure to even dilute solutions of chromic acid or the chromates will cause persistent and painful ulcers that are slow to heal. Chromium trioxide is a human poison and is a carcinogen.

5.8.7 *Group VI—(Mixed Acids or Salts in Water or Organic Solutions):*

5.8.7.1 These mixtures are safe to mix and use providing the mixing is done properly. It must be remembered that in all cases, the acid is added to the solvent slowly and with constant stirring. If sulfuric acid is in the formula, it is added last and with particular care. If hydrofluoric acid or fluorides are part of a formula, polyethylene or other similar hydrofluoric acid-resistant vessels should be used. 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. In those electrolytes containing anhydrous aluminum chloride, extreme care must be exercised. The reaction between this compound and water is almost explosive. 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.

5.8.8 *Group VII—(Alkaline Electrolytes):*

5.8.8.1 These mixtures can be grouped into two general categories, those containing cyanide, and those not containing cyanide.

(1) The use of cyanide by anyone not properly trained and familiar with it is extremely dangerous. Cyanides are among the quickest acting and most potent poisons 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 that no droplet of the solution or crystal of the salt is ever left around where it can be accidentally picked up and carried to the mouth.

(2) Solutions of the alkali hydroxides are very useful for the polishing of certain amphoteric metals. Their attack on the skin is drastic, so great care should be exercised in their use. The dissolution of alkali hydroxides, such as NaOH, in water produces substantial heat. Add the pellets to the water a little bit at a time with constant stirring until the required concentration is obtained. If the temperature becomes excessive, allow the solution to cool back to ambient before adding more hydroxide.

5.8.9 *Group VIII—(Mixtures of Methyl Alcohol and Nitric Acid):*

5.8.9.1 Nitric acid can be mixed with methanol with apparent safety (mixtures with up to about 33 % nitric acid can be safely stored). This is done by adding the acid to the alcohol with careful stirring. HNO_3 cannot be safely mixed and stored with any higher alcohol except in very dilute solutions (for example, do not store solutions of more than 3 % HNO_3 in ethanol). Under certain conditions, extremely unstable or explosive nitro compounds, azides or fulminates can be formed in alcoholic HNO_3 solutions. The spontaneous decomposition of the mixture can also be catalyzed by impurities or heat. It should always be discarded as soon as it has served its immediate purpose. Due to its dangerous nature, it should not be employed if its use can be avoided.

6. Apparatus

6.1 For the electropolishing of metal specimens in an appropriate electrolyte, a suitable electrolysis cell and a controllable power supply are needed. Simple laboratory apparatus, such as shown in Fig. 1, can be assembled to perform this function. Many such arrangements are described in the literature. There are also several commercially available models of electropolishing apparatus for either laboratory or field use.

6.2 Whenever an attempt is made to polish large surface areas, the problems of obtaining sufficient current density and cooling of the specimen and electrolyte become troublesome. An adequate volume of electrolyte should be used so that