



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 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 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.

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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.

4.1.2 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 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 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 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:*



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 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	nickel cathode
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	one of the best formulas for universal use
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	universal electrolyte comparable to I-4
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	particularly good with Al-Si alloys
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	three-second cycles repeated at least seven times to prevent heating
	germanium titanium zirconium aluminum			25 to 35 58 to 66 70 to 75 30 to 60	30 to 60 s 45 s 15 s 15 to 60 s	polish only polish and etch simultaneously
I-10		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 (60 %)	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.
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	Mix ethanol and water, add perchloric acid carefully. Then, add butylcellosolve before use.
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	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) 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	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

TABLE 1 *Continued*

Class	Use	Formula		Cell Voltage	Time	Remarks
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			
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 pyrophosphoric acid	1 L	...	10 min	good for all austenitic heat resistant alloys, 38°C plus
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—0 to 27°C
		sulfuric acid	250 mL		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—0 to 27°C
		sulfuric acid	100 mL			
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			

TABLE 1 *Continued*

Class	Use	Formula	Cell Voltage	Time	Remarks
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 , 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 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 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 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 to 60 min 0.5 to A/cm ² , 38 to 54°C
VI-12	tantalum	hydrofluoric acid sulfuric acid	100 mL 900 mL	...	9 min graphite cathode, 0.1 A/cm ² , 32 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 to 49°C
VI-14	zinc	water chromic acid sulfuric acid sodium dichromate acetic acid (glacial) hydrogen peroxide (30 %)	800 mL 100 g 46 mL 310 g 96 mL 260 mL	...	0.002 A/cm ² , 21 to 49°C
VI-15	stainless steel	(Caution) hydrofluoric acid sulfuric acid	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 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 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)	900 mL 100 mL 60 g	30 to 60	1 to 6 min (Caution) Anhydrous aluminum chloride is extremely dangerous to handle.
VI-22	uranium	zinc chloride (anhydrous) acetic acid (glacial) distilled water chromic acid	250 g 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. 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 to 3 min (Caution) Anhydrous aluminum chloride is extremely dangerous to handle. Use below 16°C.

TABLE 1 *Continued*

Class	Use	Formula		Cell Voltage	Time	Remarks
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	980 mL	7	30 s	polish 30 s but allow to remain in electrolyte until brown film is dissolved
VI-26	Sb	hydrochloric acid methanol (absolute)	20 mL 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.
VI-27	Sb	sulfuric acid hydrochloric acid ethanol (95 %)	50 mL 30 mL 30 mL			good for polarized light work
		glycerol	30 mL			
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 methanol (absolute)	640 mL 150 mL 1000 mL			
VI-31	Nb	hydrochloric acid water	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 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 , 38 to 49°C
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

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 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

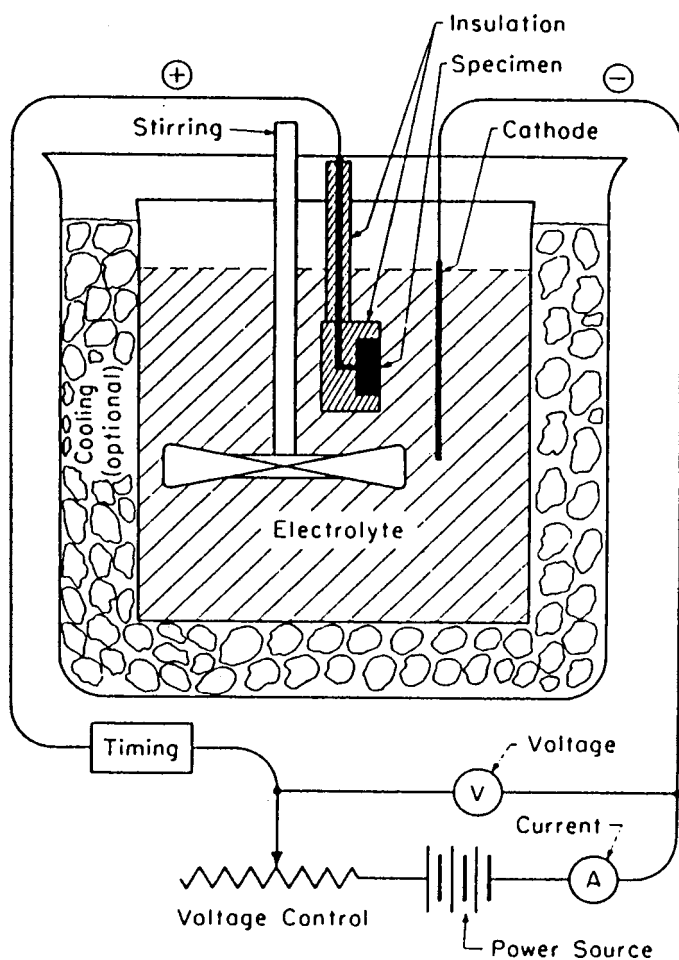


FIG. 1 Simple Arrangement for Electropolishing

cooling of the specimen and electrolyte become troublesome. An adequate volume of electrolyte should be used so that localized overheating does not occur. Supplementary cooling may be required. In general, electropolishing of areas larger than 1 cm² is not recommended for metallographic work because of the increased difficulties encountered, but it is possible to polish larger areas.

6.3 Many variations are possible in the design of a cell for electrolysis. Because the current density is critical, predetermine the approximate area to be polished so that the conditions for optimum results can be achieved. This variable can be controlled by the use of simple fixed masks, such as are used in commercial units, or by the use of protecting masks of insulating paints or tapes.

6.3.1 The material used for the mask may be important as reactions with the electrolyte may occur which may simply degrade the mask material, or may lead to explosions (see discussions of electrolyte types I, II, V, and VIII in 5).

6.4 Suitable power sources may be batteries, rectifier power supplies, or direct-current generators. Because as much as 150 V dc may be required to polish some metals, precautions must be taken to avoid electrical shock. The ideal power source would be a true direct current supply with a closely controlled voltage. When alternating-current converted to direct-current power is used, the voltage and current characteristics needed

for electropolishing can be controlled only by the use of resistance. One of the best ways of obtaining the power for electropolishing is to rectify the output of a variable-voltage transformer. When a single-phase, full wave, bridge-rectifier circuit is used, the output current is not satisfactory for all electropolishing unless the ripple voltage is reduced to some small value by filtering. This is because a particular ionization level must often be maintained within close limits in the electrolytic cell. Sufficient filtering for small amounts of power is obtained with small chokes and capacitors. When heavy currents are needed, a better source of power is a three-phase, full-wave, bridge rectifier.

6.5 Certain electrolytes are strong etchants of the metal when the polishing current is off. Therefore, the provision for rapid insertion and removal of the specimen is necessary.

6.6 The electrolyte flow rate may be an important variable. Certain electrolytes polish best when a vigorous flow rate is employed, using special electropolishing cells, while other electrolytes may require gentle agitation or no agitation at all. When using pumped solutions with specially designed polishing cells, the manufacturer's practice for adjustment of the flow rate should be followed.

6.7 The cathode material should be relatively inert in the electrolyte and, for best results, should be more noble than the metal or alloy being electropolished. A stainless steel cathode is recommended for all of the electrolytes in Table 1 where no specific recommendation is given. Other useful cathode materials include copper, nickel, graphite and platinum. The surface area of the cathode should be much greater than that of the anode (surface to be polished).

6.8 Additional design features that are desirable in apparatus for the electropolishing of metallographic specimens are as follows:

6.8.1 Separation of the power and control portions from the polishing cell to protect them from corrosive fumes and solutions;

6.8.2 Meters for the continuous indication of voltage and current;

6.8.3 Polishing cycle controlled by an automatic timer;

6.8.4 Appropriate fume hood to vent gases formed while polishing;

6.8.5 Convenient changing of electrolytes and washing and drying of the apparatus; and

6.8.6 Corrosion resistant parts which are in contact with electrolyte.

7. Procedure

7.1 The metallographer may be required to electropolish some metal or alloy which has not previously been encountered. There are several general principles which can be applied advantageously in these cases. The problem should be viewed in comparison with known procedures and information gained through previous experience. It is generally helpful to compare the relationship of the major component with elements of the same general group in the periodic arrangement of the elements, and to study the phase diagram, if available, to predict the number of phases and their characteristics. Single-phase alloys are generally easier to electropolish than multi-phase

alloys. Minor alloying elements may affect the material's response to polishing in a given electrolyte.

7.2 The conditions required for optimum polishing of a metal in a given electrolyte can be ascertained by plotting current density versus voltage curves. The resulting curves will approximate one of the two forms shown in Fig. 2. Curve I is typical of electrolytes that either polish over a very wide range or will not polish at all. Curve II is characteristic of electrolytes that form an ionic film. The dotted portion of the curve is added in recognition of certain published data and the observation that the formation of a polishing film requires finite time. Polishing will occur between B and C and is usually best slightly before C.

7.3 After the polishing range is determined, other constants such as preparation, electrolyte flow and time can be determined experimentally. In the majority of cases, a mechanically prepared surface, as produced by grinding up to a 600-grit silicon carbide finish, is sufficient; however, with some alloys, a mechanically polished surface is desirable. Metal removal rates by electropolishing are low, typically around 1 μm per minute. To remove all prior damage from grinding, either some rough polishing should be performed after the P1200-grit grinding step, or the electropolishing time may be extended,

which may not be desirable. The surface to be electropolished should be thoroughly cleaned to obtain uniform attack by the electrolyte.

7.4 Small specimens may be mounted in compression mounting media or in castable mounting media for ease in handling for mechanical preparation. Both types of mounting media are generally impervious to attack by the electrolyte (see 5.7). When mounted samples are to be electropolished, electrical contact can be made through a small hole drilled through the back of the mount into the metal.

7.5 To aid in selecting or developing an electrolyte for a new metal or alloy, the following characteristics should be considered:

7.5.1 The electrolyte should be somewhat viscous.

7.5.2 The electrolyte must be a good solvent of the anode under electrolysis conditions. Insoluble products that form adherent deposits on the face of the specimen may prevent polishing.

7.5.3 It is desirable that the electrolyte not attack the metal when the current is not flowing. However, this condition is not always achieved.

7.5.4 One or more ions of large radii [example: $(\text{PO}_4)^{-3}$, $(\text{ClO}_4)^{-1}$, or $(\text{SO}_4)^{-2}$ (and occasionally certain large organic molecules)] are usually required in electrolytes.

7.5.5 The electrolyte should be simple to mix, stable, and safe to handle.

7.5.6 It is desirable that the electrolyte function at room temperature and not be sensitive to temperature changes. If electropolishing is being done on a continuing basis, the electrolyte can be cooled by placing the cell in a water-cooled container having a water inlet and outlet.

7.6 The cathode material and size, and the anode-to-cathode spacing and orientation, are important variables. The cathode material should be more noble than the anode material (that is, the surface to be polished) and the cathode surface area should be substantially greater than the anode surface area to be polished. Generally, the cathode and anode surfaces should be aligned parallel to each other with a spacing of about 10–20 mm.

7.7 The depth of the specimen below the electrolyte surface during polishing may also influence polishing results. There should be adequate electrolyte above the area to be polished for best results.

7.8 Table 2 shows a listing of some of the most common troubles likely to be encountered in electropolishing and suggestions for solving the problems.

7.9 Table 1 contains a listing of the formulas and conditions for use of electrolytes suggested for the polishing of various metals. Table 3 is a cross listing of the various electrolytes for specific metals and generally for their alloys.

8. Keywords

8.1 electrolytic polishing; electropolishing; polishing; specimen preparation (metallographic)

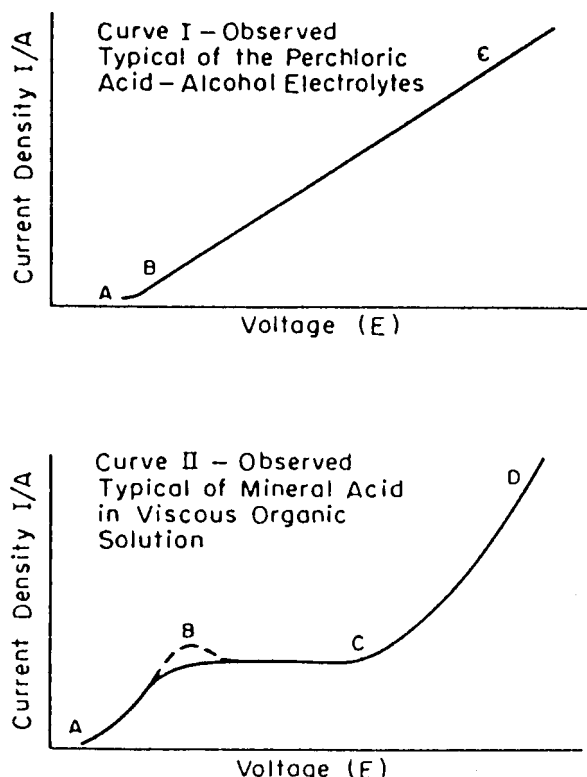


FIG. 2 Typical Curves for Determining Conditions for Electropolishing

TABLE 2 Electropolishing Procedural Problems and Corrections

Trouble	Possible Cause	Suggested Correction
Center of specimen deeply etched	no polishing film at center of specimen	(1) increase voltage (2) decrease agitation (3) use more viscous electrolyte
Pitting or etching at edges of specimen	too viscous or thick film	(1) decrease voltage (2) increase agitation (3) use less viscous electrolyte
Sludge settling on surface	insoluble anode product	(1) try new electrolyte (2) increase temperature (3) increase voltage
Roughness or matte surface	insufficient or no polishing film	(1) increase voltage (2) use more viscous electrolyte
Waviness or streaks on polished surface	(1) insufficient time (2) incorrect agitation (3) inadequate preparation (4) too much time	(1) increase or decrease agitation (2) better preparation (3) increase voltage and decrease time
Stains on polished surface	attack after polishing current is off	(1) remove specimen while current is still on (2) try less corrosive electrolyte
Unpolished spots (bullseyes)	gas bubbles	(1) increase agitation (2) decrease voltage
Phases in relief	insufficient polishing film	(1) increase voltage (2) better preparation (3) decrease time
Pitting	(1) too long polishing (2) too high voltage	(1) better preparation (2) decrease voltage (3) decrease time (4) try different electrolyte

TABLE 3 Suggested Electrolytes for Metals and Generally Their Alloys

Metal or Alloy	Electrolyte No.
Aluminum	I-1, I-2, I-4, I-5, I-6, I-8, I-10, I-12, I-13, II-3, III-7, III-14, IV-6
Aluminum-silicon alloys	I-6, I-8
Antimony	II-4, VI-26, VI-27
Beryllium	I-4, I-13
Bismuth	VI-18, VI-25, VI-28
Cadmium	I-14, III-4
Cast iron	I-4, II-1
Chromium	II-1, II-6, VI-29, VIII-1
Cobalt	I-5, II-3, III-1, III-4, VIII-1
Copper	III-2, III-3, III-4, III-5, III-10, III-15, VIII-1
Copper-zinc alloys	III-3, III-4, III-5, III-10, V-2, VIII-1
Copper-tin alloys	III-10, VI-5, VI-6, VIII-1
Copper-nickel alloys	III-3, III-10, VIII-1
Germanium	I-9, VI-30
Gold	VII-1
Hafnium	II-7, VII-11
Iron, pure	I-5, II-1, IV-2, IV-3
Iron-silicon alloys	I-5, I-6, I-8, II-5
Iron-copper alloys	III-3, III-4
Iron-nickel alloys	I-5, II-1, II-2, II-4, IV-3, VIII-1
Lead	I-1, I-5, I-11, II-4, VII-5
Magnesium	I-1, III-7, III-12, VI-19
Manganese	III-9
Molybdenum	I-7, I-11, I-15, IV-4, IV-7, VI-20
Neptunium	III-16
Nickel-chromium	II-4, VIII-1
Nickel	I-4, I-13, I-14, II-4, IV-2, VIII-1
Nickel aluminide	I-13
Niobium	VI-31, VI-32
Silicon	VII-5
Silver	I-14, III-7, VII-1, VII-2, VII-3
Steel, austenitic, stainless, and super alloys	I-1, I-2, I-3, I-4, I-5, I-11, I-13, II-1, II-2, II-3, III-3, III-6, III-11, IV-1, IV-2, IV-3, IV-5, IV-6, IV-8, V-1, VI-1, VI-2, VI-3, VI-4, VI-7, VI-8, VI-9, VI-10, VI-11, VI-13, VI-15, VI-16, VI-17, VI-33, VIII-1
Steel, carbon and alloy	I-1, I-2, I-4, I-5, I-11, I-13, II-1, II-2, II-3, II-5, II-7, III-6, VI-3, VI-11
Tantalum	VI-12
Tin	I-4, I-13, VI-5, VI-6, VII-6
Titanium	I-4, I-9, I-13, II-1, II-2, II-3
Tungsten	VII-4, VII-5, VII-7
Uranium	I-4, I-7, II-1, II-2, II-3, III-8, III-13
Vanadium	I-9
Zinc	I-1, I-5, III-12, V-2, VI-14, VI-23, VII-6, VIII-1
Zirconium	I-4, I-7, I-9, II-2, VI-24

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