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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 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific safety precautions are described in Section 5 and 6.3.1.

2. Referenced Documents

- 2.1 ASTM Standards: ³
- E 7 Terminology Relating to Metallography
 E 407 Test Methods for Microetching Metals and Alloys

3. Terminology

- 3.1 *Definitions*—All terms used in this guide are either defined in Terminology E 7 or are discussed in 3.2.
 - 3.2 Definitions of Terms Specific to This Standard:
- 3.2.1 *electrolytic polish (electropolish)*—A method of polishing metals and alloys in which material is removed from the surface by making the metal the anode in an electrolytic bath.

4. Significance and Use

- 4.1 Advantages of Electrolytic Polishing:
- 4.1.1 For some metals, a high quality surface finish can be produced that is equivalent to, or better than, that which can be obtained by mechanical methods.
- 4.1.2 Once procedures have been established, satisfactory results can be obtained rapidly with reproducibility.
- 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.

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

TABLE 1 Electrolytes for Electropolishing

Class	Use	Formula		Cell Voltage	Time	Remarks
Group	I (Electrolytes Composed of Perchloric	Acid and Alcohol With or With	out Organic Additions	s)		
-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			35 to 65 12 to 35 20 to 60	15 to 60 s 15 to 60 s 	
-2	Mg and high Mg alloys stainless steel and aluminum	ethanol (95 %)	800 mL 200 mL	35 to 80	15 to 60 s	nickel cathode
-3	stainless steel	perchloric acid (60 %) ethanol (95 %) perchloric acid (65 %)	940 mL 60 mL	30 to 45	15 to 60 s	
-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
-5	steels—stainless, alloy, high-speed; Fe, Al, Zr, Pb	ethanol (95 %)	558-700 mL 100 mL	15 to 50	15 to 60 s	universal electrolyte comparable to I-4
tps:/ -6	Al, Al-Si alloys	perchloric acid (30 %) 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
-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	
-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	, , , , , , , , , , , , , , , , , , , ,		25 to 35 58 to 66 70 to 75	30 to 60 s 45 s 15 s	polish only polish and etch simultaneously
10	aluminum	methanol (absolute) nitric acid perchloric acid (60 %)	950 mL 15 mL 50 mL	30 to 60	15 to 60 s	,
-11	steels—carbon, alloy, stainless Ti, high-temperature alloys, Pb, Mo	methanol (absolute) butylcellosolve perchloric acid	600 mL 360 mL 60 mL	30–40	5–60 s	good all purpose electropolish
-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.
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.
-14	Ni, Ag, or Cu alloys Cd	ethanol (95 %) butylcellosolve perchloric acid	700 mL 100 mL 200 mL	70–80	15 s	



Class	Use	Formula		Cell Voltage	Time	Remarks
I-15	Mo and Mo alloys	methanol (absolute)	600 mL		20 s	Mix methanol and water, add
		water	13 mL			perchloric acid carefully. Add
		butylcellosolve	360 mL			butylcellosolve before use.
		perchloric acid	47 mL			
	II (Electrolytes Composed of Perchloric		, , ,			
l-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
l-2	Zr, Ti, U, steel—carbon and alloy	acetic acid (glacial)	900 mL	12 to 70	0.5 to 2 min	
-	zi, ii, o, otoor barborrana andy	perchloric acid (60 %)	100 mL	12 10 70	0.0 to 2 mm	
I-3	U, Zr, Ti, Al, steel—carbon and	acetic acid (glacial)	800 mL	40 to 100	1 to 15 min	
	alloy	perchloric acid (60 %)	200 mL			
I-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
I-6	Cr	perchloric acid (60 %) acetic acid (glacial)	350 mL 1000 mL	30–50	2-3 min	can lawar valtaga to 25 V by adding
1-0	CI	perchloric acid	5 mL	30-30	2-3 111111	can lower voltage to 25 V by adding 5–15 % water.
I-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 Phospho	ric Acid in Water or Organic Solve	ent)			
II-1	cobalt	phosphoric acid (85 %)	1000 mL	1.2	3 to 5 min	
II-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	water	300 mL	1.5 to 1.8	5 to 15 min	copper cathode
0	alloys except Sn bronze	phosphoric acid (85 %)	700 mL	1.0 to 1.0	0 10 10 11111	coppor daniedo
II-4	alpha or alpha plus beta brass,	water	600 mL	1 to 2	1 to 15 min	copper or stainless steel cathode
	Cu-Fe, Cu-Co, Co, Cd	phosphoric acid (85 %)	400 mL			
II-5	Cu, Cu-Zn	water	1000 mL	1 to 2	10 min	copper cathode
		pyrophosphoric acid	580 g		E . 4E .	10005
II-6	steel	diethylene glycol monoethyl ether	500 mL	5 to 20	5 to 15 min	120°F
		phosphoric acid (85 %)	500 mL			
II-7	Al, Ag, Mg	water	200 mL	25 to 30	4 to 6 min	aluminum cathode, 100 to 110°F
,	,	ethanol (95 %)	380 mL			
		phosphoric acid (85 %)	400 mL			
II-8	uranium	ethanol (absolute)	300 mL			
		glycerin (cp)	300 mL			
	Mar Mar Overallassa	phosphoric acid (85 %)	300 mL	10		
II-9	Mn, Mn-Cu alloys	ethanol (95 %) glycerin ASTM E155	500 mL 250 mL	18		
		phosphoric acid (85 %)	250 mL			
II-10	Cu and Cu-base alloys Catalog/S	distilled water	-ae 500 mL	92ab-e504	1 to 5 min	
	,	ethanol (95 %)	250 mL			
		phosphoric acid (85 %)	250 mL			
II-11	stainless steel	ethanol (absolute), to	1 L		10 min	good for all austenitic heat resistant
		pyrophosphoric acid	400 g			alloys, 100°F plus
II-12	Mg-Zn	ethanol (95 %)	625 mL	1.5 to 2.5	3 to 30 min	
II-13	uranium	phosphoric acid (85 %) ethanol (95 %)	375 mL 445 mL	18 to 20	5 to 15 min	0.03 A/cm ²
11-10	uramum	ethylene glycol	275 mL	10 10 20	3 10 13 111111	0.03 A/GIII
		phosphoric acid (85 %)	275 mL			
II-14	Al-Mg alloys	water	250 mL	50-60	2 min	
	9	ethanol (95 %)	380 mL			
		phosphoric acid (85 %)	400 mL			
II-15	Cu-Pb alloys	ethanol (95 %)	620 mL			good up to 30 % Pb
11.40	Nestunium	phosphoric acid (85 %)	380 mL			often COO mult COO
III-16	Neptunium	ethanol (95 %) glycerol	400 mL 400 mL			after 600-grit SiC, use 6-µm diamond on nylon before
		phosphoric acid (85 %)	800 mL			electropolishing.
		iroup IV (Electrolytes Composed of		later or Organic	Solvent)	
V-1	stainless steel	water	250 mL	1.5 to 6	1 to 2 min	
		sulfuric acid	750 mL		=	
V-2	stainless steel, Fe, Ni	water	400 mL	1.5 to 6	2 to 6 min	
		sulfuric acid	600 mL			
V-3	stainless steel, Fe, Ni, Mo	water	750 mL	1.5 to 6	2 to 10 min	particularly good for sintered
		sulfuric acid	250 mL		Mo—	Mo—32 to 80°F
V/ 4	no ob do do no uno	water	0001	1 E to 0	0.3 to 1 min	portional or and for electrical
IV-4	molybdenum	water	900 mL	1.5 to 6	0.3 to 2 min	particularly good for sintered
		sulfuric acid	100 mL			Mo—32 to 80°F



Class	Use	Formula		Cell Voltage	Time	Remarks
IV-5	stainless steel	water	70 mL	1.5 to 6	0.5 to 5 min	
		glycerin	200 mL			
		sulfuric acid	720 mL			
V-6	stainless steel, aluminum	water	220 mL	1.5 to 12	1 to 20 min	
		glycerin sulfuric acid	200 mL 580 mL			
V-7	molybdenum	methanol (absolute)	875 mL	6 to 18	0.5 to 1.5 min	32 to 80°F
-	,	sulfuric acid	125 mL	- 10 10		
V-8	Ni-base superalloys	methanol (absolute)	800 mL	30	20 s	for alloy 625
		sulfuric acid	200 mL			
		Group V (Electrolytes C	composed of Chromic	c Acid in Water)		
/-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 of	or Salts in Water or 0	Organic Solvent)		
/I-1	stainless steel	phosphoric acid (85 %)	600 mL			
/1-2	stainless steel	sulfuric acid water	400 mL 150 mL		2 min	0.3 A/cm ²
1-2	Stall 11633 StCC1	phosphoric acid (85 %)	300 mL	•••	£ 111111	0.0 A/GIII
		sulfuric acid	550 mL			
′I-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			
/I-4	stainless steel	water	330 mL		1 min	0.05 A/cm ²
		phosphoric acid (85 %) sulfuric acid	550 mL 120 mL			
′I-5	bronze (to 9 % Sn)	water	450 mL		1 to 5 min	0.1 A/cm ²
	5101120 (10 0 70 011)	phosphoric acid (85 %)	390 mL	مآم	1 10 0 111111	0.1700111
		sulfuric acid	160 mL			
/I-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		411.	4 5 4 2 40005 1
VI-7	steel	water glycerin	140 mL 100 mL		1 to 5 min 1 to	to 5 A/cm ² , 100°F plus
		phosphoric acid (85 %)	430 mL			
		sulfuric acid	330 mL			
/I-8	stainless steel	water	200 mL		5 min	1 A/cm ² , 80 to 120°F
		glycerin	590 mL			
		phosphoric acid (85 %)	58-100 mL 04)			
// 0	//	sulfuric acid	110 mL		70 4506.0	0.6 A/cm ² , 80 to 120°F
/I-9 S	stainless steel teh ai/catalo	g/star water s/sist/d233ac l chromic acid	175 g	92ab-e504	30 min	0.6 A/CIII , 80 to 120 F
		phosphoric acid (85 %)	175 mL			
		sulfuric acid	580 mL			
/I-10	stainless steel	water	175 mL		60 min	0.5 A/cm ² , 80 to 120°F
		chromic acid	105 g			
		phosphoric acid (85 %)	460 mL 390 mL			
/1_11	stainless and allow steel	Sulfuric acid			5 to 60 min	0.5 to 4/cm² 100 to 130°E
/I-11	stainless and alloy steel	water	240 mL		5 to 60 min	0.5 to A/cm ² , 100 to 130°F
/I-11	stainless and alloy steel	water chromic acid	240 mL 80 g		5 to 60 min	0.5 to A/cm², 100 to 130°F
/I-11	stainless and alloy steel	water	240 mL		5 to 60 min	0.5 to A/cm ² , 100 to 130°F
	stainless and alloy steel tantalum	water chromic acid phosphoric acid (85 %) sulfuric acid hydrofluoric acid	240 mL 80 g 650 mL 130 mL 100 mL		5 to 60 min 9 min	graphite cathode, 0.1 A/cm², 90 to
/I-12	tantalum	water chromic acid phosphoric acid (85 %) sulfuric acid hydrofluoric acid sulfuric acid	240 mL 80 g 650 mL 130 mL 100 mL 900 mL		9 min	graphite cathode, 0.1 A/cm², 90 to 100°F
/I-12		water chromic acid phosphoric acid (85 %) sulfuric acid hydrofluoric acid sulfuric acid water	240 mL 80 g 650 mL 130 mL 100 mL 900 mL 210 mL			graphite cathode, 0.1 A/cm², 90 to
/l-12	tantalum	water chromic acid phosphoric acid (85 %) sulfuric acid hydrofluoric acid sulfuric acid water hydrofluoric acid	240 mL 80 g 650 mL 130 mL 100 mL 900 mL 210 mL 180 mL		9 min	graphite cathode, 0.1 A/cm², 90 to 100°F
/I-12 /I-13	tantalum stainless steel	water chromic acid phosphoric acid (85 %) sulfuric acid hydrofluoric acid sulfuric acid water hydrofluoric acid sulfuric acid	240 mL 80 g 650 mL 130 mL 100 mL 900 mL 210 mL 180 mL 610 mL		9 min 5 min	graphite cathode, 0.1 A/cm², 90 to 100°F 0.5 A/cm², 70 to 120°F
/I-12 /I-13	tantalum	water chromic acid phosphoric acid (85 %) sulfuric acid hydrofluoric acid sulfuric acid water hydrofluoric acid	240 mL 80 g 650 mL 130 mL 100 mL 900 mL 210 mL 180 mL		9 min	graphite cathode, 0.1 A/cm², 90 to 100°F
/I-12 /I-13	tantalum stainless steel	water chromic acid phosphoric acid (85 %) sulfuric acid hydrofluoric acid sulfuric acid water hydrofluoric acid sulfuric acid sulfuric acid	240 mL 80 g 650 mL 130 mL 100 mL 900 mL 210 mL 180 mL 610 mL 800 mL		9 min 5 min	graphite cathode, 0.1 A/cm², 90 to 100°F 0.5 A/cm², 70 to 120°F
/I-12 /I-13	tantalum stainless steel	water chromic acid phosphoric acid (85 %) sulfuric acid hydrofluoric acid sulfuric acid water hydrofluoric acid sulfuric acid water chromic acid sulfuric acid sulfuric acid sulfuric acid sulfuric acid sulfuric acid sodium dichromate	240 mL 80 g 650 mL 130 mL 100 mL 900 mL 210 mL 180 mL 610 mL 800 mL 100 g 46 mL 310 g		9 min 5 min	graphite cathode, 0.1 A/cm², 90 to 100°F 0.5 A/cm², 70 to 120°F
/I-12 /I-13 /I-14	tantalum stainless steel zinc	water chromic acid phosphoric acid (85 %) sulfuric acid hydrofluoric acid sulfuric acid water hydrofluoric acid sulfuric acid water chromic acid sulfuric acid sulfuric acid sulfuric acid sulfuric acid sulfuric acid sodium dichromate acetic acid (glacial)	240 mL 80 g 650 mL 130 mL 100 mL 900 mL 210 mL 180 mL 610 mL 800 mL 100 g 46 mL 310 g 96 mL		9 min 5 min 	graphite cathode, 0.1 A/cm², 90 to 100°F 0.5 A/cm², 70 to 120°F 0.002 A/cm², 70 to 100°F
VI-12 VI-13 VI-14	tantalum stainless steel	water chromic acid phosphoric acid (85 %) sulfuric acid hydrofluoric acid sulfuric acid water hydrofluoric acid sulfuric acid water chromic acid sulfuric acid sulfuric acid sulfuric acid sulfuric acid sulfuric acid sulfuric acid sodium dichromate acetic acid (glacial) hydrogen peroxide (30 %)	240 mL 80 g 650 mL 130 mL 100 mL 900 mL 210 mL 180 mL 610 mL 800 mL 100 g 46 mL 310 g		9 min 5 min	graphite cathode, 0.1 A/cm², 90 to 100°F 0.5 A/cm², 70 to 120°F
/I-12 /I-13 /I-14	tantalum stainless steel zinc	water chromic acid phosphoric acid (85 %) sulfuric acid hydrofluoric acid sulfuric acid water hydrofluoric acid sulfuric acid water chromic acid sulfuric acid sulfuric acid sulfuric acid sulfuric acid sulfuric acid sodium dichromate acetic acid (glacial) hydrogen peroxide (30 %) (Caution)	240 mL 80 g 650 mL 130 mL 100 mL 900 mL 210 mL 180 mL 610 mL 800 mL 100 g 46 mL 310 g 96 mL 260 mL		9 min 5 min 	graphite cathode, 0.1 A/cm², 90 to 100°F 0.5 A/cm², 70 to 120°F 0.002 A/cm², 70 to 100°F
/I-12 /I-13 /I-14	tantalum stainless steel zinc	water chromic acid phosphoric acid (85 %) sulfuric acid hydrofluoric acid sulfuric acid water hydrofluoric acid sulfuric acid sulfuric acid sulfuric acid sulfuric acid sulfuric acid sulfuric acid sodium dichromate acetic acid (glacial) hydrogen peroxide (30 %) (Caution) hydrofluoric acid	240 mL 80 g 650 mL 130 mL 100 mL 900 mL 210 mL 180 mL 610 mL 800 mL 100 g 46 mL 310 g 96 mL 260 mL		9 min 5 min 	graphite cathode, 0.1 A/cm², 90 to 100°F 0.5 A/cm², 70 to 120°F 0.002 A/cm², 70 to 100°F
VI-12 VI-13 VI-14 VI-15	tantalum stainless steel zinc stainless steel	water chromic acid phosphoric acid (85 %) sulfuric acid hydrofluoric acid sulfuric acid water hydrofluoric acid sulfuric acid water chromic acid sulfuric acid sulfuric acid sulfuric acid sulfuric acid sodium dichromate acetic acid (glacial) hydrogen peroxide (30 %) (Caution) hydrofluoric acid sulfuric acid	240 mL 80 g 650 mL 130 mL 100 mL 900 mL 210 mL 180 mL 610 mL 800 mL 100 g 46 mL 310 g 96 mL 260 mL		9 min 5 min 5 min	graphite cathode, 0.1 A/cm², 90 to 100°F 0.5 A/cm², 70 to 120°F 0.002 A/cm², 70 to 100°F 0.5 A/cm² (Caution) Dangerous
VI-11 VI-12 VI-13 VI-14 VI-15	tantalum stainless steel zinc	water chromic acid phosphoric acid (85 %) sulfuric acid hydrofluoric acid sulfuric acid water hydrofluoric acid sulfuric acid sulfuric acid sulfuric acid sulfuric acid sulfuric acid sulfuric acid sodium dichromate acetic acid (glacial) hydrogen peroxide (30 %) (Caution) hydrofluoric acid	240 mL 80 g 650 mL 130 mL 100 mL 900 mL 210 mL 180 mL 610 mL 800 mL 100 g 46 mL 310 g 96 mL 260 mL		9 min 5 min 	graphite cathode, 0.1 A/cm², 90 to 100°F 0.5 A/cm², 70 to 120°F 0.002 A/cm², 70 to 100°F



			00			
Class	Use	Formula		Cell Voltage	Time	Remarks
VI-17	stainless steel	water	600 mL			
		chromic acid	180 g			
		nitric acid	60 mL			
		hydrochloric acid	3 mL			
		sulfuric acid	240 mL			2.5
VI-18	bismuth	glycerin	750 mL	12	1 to 5 min	0.5 ± A/cm² (Caution) This mixture
		acetic acid (glacial)	125 mL			will decompose vigorously after a
VI-19	magnesium	nitric acid ethylene-glycol-monoethyl ether	125 mL 900 mL	50 to 60	10 to 30 s	short time. Do not try to keep. Bath should be stirred. Cool cracked
VI 15	magnesiam	hydrochloric acid	100 mL	30 10 00	10 10 00 3	ice below 35°F
VI-20	molybdenum, sintered and cast	methanol (absolute)	685 mL	19 to 35	20 to 35 s	Mix slowly. Heat is developed. Avoid
	•	hydrochloric acid	225 mL			contamination with water. Below
		sulfuric acid	90 mL			35°F.
		Group VI (Mixed Acids or Salts in V	Nater or Organic	c Solvent)—Con	ntinued	
VI-21	titanium	ethanol (95 %)	900 mL	30 to 60	1 to 6 min	(Caution) Anhydrous aluminum
		n-butyl alcohol	100 mL			chloride is extremely dangerous to
		aluminum chloride (anhydrous)	60 g			handle.
		(add very slowly) (Caution)				
V/I 00		zinc chloride (anhydrous)	250 g	00	E to 00	The character and in discretized in the
VI-22	uranium	acetic acid (glacial)	750 mL	80	5 to 30 min	The chromic acid is dissolved in the
		distilled water chromic acid	210 mL 180 g			water before adding to the acetic acid. Below 35°F.
VI-23	pure zinc	ethanol (95 %)	720 mL	25 to 40	0.5 to 3 min	(Caution) Anhydrous aluminum
0	F	aluminum chloride (anhydrous)	50 g	20 10 10	2.0 .0 0 11111	chloride is extremely dangerous to
		(Caution)	9			handle. Below 60°F.
		zinc chloride (anhydrous)	225 g			
		distilled water	160 mL			
		n-butyl alcohol	80 mL			
VI-24	zirconium. Polish and etch	glycerin (Caution)	870 mL	9 to 12	1 to 10 min	(Caution) will decompose on
	simultaneously	hydrofluoric acid	43 mL			standing, dangerous if kept too long
		nitric acid	87 mL	us		
VI-25	bismuth	saturated solution KI in distilled	980 mL	7	30 s	polish 30 s but allow to remain in
		water	00-0			electrolyte until brown film is
VI-26	Sb	hydrochloric acid	20 mL 300 mL	6–10	2-4 min	dissolved
VI-20	30	methanol (absolute) sulfuric acid	50 mL	0-10	2-4/11111	pure Sb. Use Pt cathode and anode lead wires. Agitate bath. Do not
		hydrochloric acid	30 mL			touch polished surface with cotton.
VI-27	Sb	ethanol (95 %)	30 mL			good for polarized light work
		glycerol	30 mL			
		phosphoric acid	100 mL			
		sulfuric acid STM F1558	30 mL			
VI-28	Bi	water	200 mL			good for polarized light work
		phosphoric acid 233ac11-a sulfuric acid	200 mL 3 C-			
VI-29	Cr	water	210 mL	18		stir bath or specimen
VI-23	OI .	phosphoric acid	640 mL	10		sui batti of specimen
		sulfuric acid	150 mL			
VI-30	Ge					
	Ge	methanol (absolute)	1000 mL			
VI-31	de	methanol (absolute) hydrochloric acid				
	Nb	hydrochloric acid water	1000 mL 10 mL 300 mL	40		polish to α -alumina before
		hydrochloric acid water sulfuric acid	1000 mL 10 mL 300 mL 100 mL	40		polish to α -alumina before electropolishing
\/I 60	Nb	hydrochloric acid water sulfuric acid hydrofluoric acid	1000 mL 10 mL 300 mL 100 mL 100 mL		40 -	polish to α-alumina before electropolishing
VI-32		hydrochloric acid water sulfuric acid hydrofluoric acid methanol (absolute)	1000 mL 10 mL 300 mL 100 mL 100 mL 940 mL	40 50–60	10 s	polish to α -alumina before electropolishing
VI-32	Nb	hydrochloric acid water sulfuric acid hydrofluoric acid methanol (absolute) sulfuric acid	1000 mL 10 mL 300 mL 100 mL 100 mL 940 mL 50 mL		10 s	polish to $\alpha\text{-alumina}$ before electropolishing
	Nb Nb	hydrochloric acid water sulfuric acid hydrofluoric acid methanol (absolute) sulfuric acid hydrofluoric acid	1000 mL 10 mL 300 mL 100 mL 100 mL 940 mL 50 mL 15 mL	50–60		electropolishing
VI-32 VI-33	Nb	hydrochloric acid water sulfuric acid hydrofluoric acid methanol (absolute) sulfuric acid	1000 mL 10 mL 300 mL 100 mL 100 mL 940 mL 50 mL		10 s 20 s	polish to $\alpha\text{-alumina}$ before electropolishing for Waspaloy and IN-100 mod. Etch at 5 V for 4 s.
	Nb Nb	hydrochloric acid water sulfuric acid hydrofluoric acid methanol (absolute) sulfuric acid hydrofluoric acid methanol (absolute) hydrochloric acid	1000 mL 10 mL 300 mL 100 mL 100 mL 940 mL 50 mL 15 mL 170 mL 30 mL	50–60 30		electropolishing for Waspaloy and IN-100 mod. Etch
VI-33	Nb Ni-base superalloy	hydrochloric acid water sulfuric acid hydrofluoric acid methanol (absolute) sulfuric acid hydrofluoric acid methanol (absolute) hydrochloric acid Group VII (Alk	1000 mL 10 mL 300 mL 100 mL 100 mL 940 mL 50 mL 15 mL 170 mL 30 mL	50-60 30	20 s	electropolishing for Waspaloy and IN-100 mod. Etch at 5 V for 4 s.
	Nb Nb	hydrochloric acid water sulfuric acid hydrofluoric acid methanol (absolute) sulfuric acid hydrofluoric acid methanol (absolute) hydrochloric acid Group VII (Alk	1000 mL 10 mL 300 mL 100 mL 100 mL 940 mL 50 mL 15 mL 170 mL 30 mL aline Electrolyte	50–60 30		electropolishing for Waspaloy and IN-100 mod. Etch
VI-33	Nb Ni-base superalloy	hydrochloric acid water sulfuric acid hydrofluoric acid methanol (absolute) sulfuric acid hydrofluoric acid methanol (absolute) hydrochloric acid Group VII (Alk water to potassium cyanide	1000 mL 10 mL 300 mL 100 mL 100 mL 940 mL 50 mL 15 mL 170 mL 30 mL aline Electrolyte:	50-60 30	20 s	electropolishing for Waspaloy and IN-100 mod. Etch at 5 V for 4 s.
VI-33	Nb Ni-base superalloy	hydrochloric acid water sulfuric acid hydrofluoric acid methanol (absolute) sulfuric acid hydrofluoric acid methanol (absolute) hydrochloric acid Group VII (Alk water to potassium cyanide potassium carbonate	1000 mL 10 mL 300 mL 100 mL 100 mL 940 mL 50 mL 15 mL 170 mL 30 mL aline Electrolyte 1000 mL 80 g 40 g	50-60 30	20 s	electropolishing for Waspaloy and IN-100 mod. Etch at 5 V for 4 s.
VI-33 	Nb Ni-base superalloy gold	hydrochloric acid water sulfuric acid hydrofluoric acid methanol (absolute) sulfuric acid hydrofluoric acid methanol (absolute) hydrochloric acid Group VII (Alk water to potassium cyanide polassium carbonate gold chloride	1000 mL 10 mL 300 mL 100 mL 100 mL 940 mL 50 mL 15 mL 170 mL 30 mL aline Electrolyte 1000 mL 80 g 40 g 50 g	50–60 30 es) 7.5	20 s	electropolishing for Waspaloy and IN-100 mod. Etch at 5 V for 4 s. graphite cathode
VI-33	Nb Ni-base superalloy	hydrochloric acid water sulfuric acid hydrofluoric acid methanol (absolute) sulfuric acid hydrofluoric acid methanol (absolute) hydrochloric acid Group VII (Alk water to potassium cyanide potassium carbonate	1000 mL 10 mL 300 mL 100 mL 100 mL 940 mL 50 mL 15 mL 170 mL 30 mL aline Electrolyte 1000 mL 80 g 40 g 50 g 1000 mL	50-60 30	20 s	electropolishing for Waspaloy and IN-100 mod. Etch at 5 V for 4 s.
VI-33 	Nb Ni-base superalloy gold	hydrochloric acid water sulfuric acid hydrofluoric acid methanol (absolute) sulfuric acid hydrofluoric acid methanol (absolute) hydrochloric acid Group VII (Alk water to potassium cyanide potassium carbonate gold chloride water to	1000 mL 10 mL 300 mL 100 mL 100 mL 940 mL 50 mL 15 mL 170 mL 30 mL aline Electrolyte 1000 mL 80 g 40 g 50 g	50–60 30 es) 7.5	20 s	electropolishing for Waspaloy and IN-100 mod. Etch at 5 V for 4 s. graphite cathode
VI-33 	Nb Ni-base superalloy gold	hydrochloric acid water sulfuric acid hydrofluoric acid methanol (absolute) sulfuric acid hydrofluoric acid methanol (absolute) hydrochloric acid Group VII (Alk water to potassium cyanide potassium carbonate gold chloride water to sodium cyanide	1000 mL 10 mL 300 mL 100 mL 100 mL 940 mL 50 mL 15 mL 170 mL 30 mL aline Electrolyte 1000 mL 80 g 40 g 50 g 1000 mL 1000 mL	50–60 30 es) 7.5	20 s	for Waspaloy and IN-100 mod. Etch at 5 V for 4 s.
VI-33 VII-1 VII-2	Nb Ni-base superalloy gold silver	hydrochloric acid water sulfuric acid hydrofluoric acid methanol (absolute) sulfuric acid hydrofluoric acid methanol (absolute) hydrochloric acid Group VII (Alk water to potassium cyanide potassium carbonate gold chloride water to sodium cyanide potassium ferrocyanide water to potassium ferrocyanide	1000 mL 10 mL 300 mL 100 mL 100 mL 100 mL 940 mL 50 mL 15 mL 170 mL 30 mL 20 g	50–60 30 9s) 7.5	20 s 2 to 4 min To 1 min	for Waspaloy and IN-100 mod. Etch at 5 V for 4 s. graphite cathode graphite cathode
VI-33 VII-1 VII-2	Nb Ni-base superalloy gold silver	hydrochloric acid water sulfuric acid hydrofluoric acid methanol (absolute) sulfuric acid hydrofluoric acid methanol (absolute) hydrochloric acid Group VII (Alk water to potassium cyanide potassium carbonate gold chloride water to sodium cyanide potassium ferrocyanide water to potassium ferrocyanide silver cyanide	1000 mL 10 mL 300 mL 100 mL 100 mL 100 mL 940 mL 50 mL 15 mL 170 mL 30 mL 20 g	50–60 30 9s) 7.5	20 s 2 to 4 min To 1 min	for Waspaloy and IN-100 mod. Etch at 5 V for 4 s. graphite cathode graphite cathode graphite cathode, 0.003 to 0.009
VI-33 VII-1 VII-2 VII-3	Nb Ni-base superalloy gold silver	hydrochloric acid water sulfuric acid hydrofluoric acid methanol (absolute) sulfuric acid hydrofluoric acid methanol (absolute) hydrochloric acid Group VII (Alk water to potassium cyanide potassium carbonate gold chloride water to sodium cyanide potassium ferrocyanide water to potassium ferrocyanide silver cyanide potassium cyanide silver cyanide potassium dichromate	1000 mL 10 mL 300 mL 100 mL 100 mL 940 mL 50 mL 15 mL 170 mL 30 mL 1000 mL 80 g 40 g 50 g 1000 mL 100 g 1000 g 1000 mL 400 g 280 g 280 g	50–60 30 9s) 7.5	20 s 2 to 4 min To 1 min To 9 min	for Waspaloy and IN-100 mod. Etch at 5 V for 4 s. graphite cathode graphite cathode graphite cathode, 0.003 to 0.009 A/cm²
VI-33 VII-1 VII-2	Nb Ni-base superalloy gold silver	hydrochloric acid water sulfuric acid hydrofluoric acid methanol (absolute) sulfuric acid hydrofluoric acid methanol (absolute) hydrochloric acid Group VII (Alk water to potassium cyanide potassium carbonate gold chloride water to sodium cyanide potassium ferrocyanide water to potassium cyanide potassium cyanide silver cyanide potassium cyanide silver cyanide potassium dichromate water to	1000 mL 10 mL 300 mL 100 mL 100 mL 940 mL 50 mL 15 mL 170 mL 30 mL 1000 mL 80 g 40 g 1000 mL 100 g 1000 g 1000 mL 400 g 280 g 280 g 280 g 1000 mL	50–60 30 9s) 7.5	20 s 2 to 4 min To 1 min	for Waspaloy and IN-100 mod. Etch at 5 V for 4 s. graphite cathode graphite cathode graphite cathode, 0.003 to 0.009 A/cm² graphite cathode, 0.09 A/cm², 100 to
VI-33 VII-1 VII-2 VII-3	Nb Ni-base superalloy gold silver	hydrochloric acid water sulfuric acid hydrofluoric acid methanol (absolute) sulfuric acid hydrofluoric acid methanol (absolute) hydrochloric acid Group VII (Alk water to potassium cyanide potassium carbonate gold chloride water to sodium cyanide potassium ferrocyanide water to potassium ferrocyanide silver cyanide potassium cyanide silver cyanide potassium dichromate	1000 mL 10 mL 300 mL 100 mL 100 mL 940 mL 50 mL 15 mL 170 mL 30 mL 1000 mL 80 g 40 g 50 g 1000 mL 100 g 1000 g 1000 mL 400 g 280 g 280 g	50–60 30 7.5 2.5	20 s 2 to 4 min To 1 min To 9 min	for Waspaloy and IN-100 mod. Etch at 5 V for 4 s. graphite cathode graphite cathode graphite cathode, 0.003 to 0.009 A/cm²

Class	Use	Formula		Cell Voltage	Time	Remarks	
VII-6 VII-7	zinc, tin	water to potassium hydroxide water sodium hydroxide	1000 mL 200 g 1000 mL 20 g	2 to 6	15 min 5 min	copper cathode, 0.1 to 0.2 A/cm ²	
	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	

- 5.2 Some basic suggestions for the handling and disposal of electrolytes and their ingredients are as follows:
- 5.2.1 When pouring, mixing, or using electrolytes, always use the proper protective equipment (eyewear, gloves, apron, and so forth).
- 5.2.2 Use proper devices (glass or plastic) for weighing, measuring, mixing, containing, and storage of solutions.
- 5.2.3 When mixing electrolytes, always add reagents to the solvent unless specific instructions indicate otherwise.
- 5.2.4 When using an electrolyte, always avoid direct physical contact with the electrolyte and the specimen. Use tongs or some other indirect method of handling specimens.
- 5.2.5 In general, it is good practice to work under a properly designed chemical fume hood, and it is imperative with those electrolytes that give off noxious odors or toxic vapors.
- 5.2.6 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:
- 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 65 to 80°F. 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. 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 Very little heat is developed when perchloric acid is mixed with glacial acetic acid. In mixing, the perchloric acid