

Designation: E 1558 – 99

# Standard Guide for Electrolytic Polishing of Metallographic Specimens<sup>1</sup>

This standard is issued under the fixed designation E 1558; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

# 1. Scope

1.1 This guide deals with electrolytic polishing as a means of preparation of specimens for metallographic purposes. Procedures are described for polishing a variety of metals.

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

1.2 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:

E 3 Methods of Preparation of Metallographic Specimens<sup>3</sup>

E 7 Terminology Relating to Metallography<sup>3</sup>

E 407 Test Methods for Microetching Metals and Alloys<sup>3</sup>

### 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: bd2be

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.

5 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.

<sup>&</sup>lt;sup>1</sup> This guide is under the jurisdiction of ASTM Committee E-4 on Metallography and is the direct responsibility of Subcommittee E04.01 on Sampling, Specimen Preparation, and Photography.

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 $<sup>^{2}\,\</sup>mathrm{The}$  boldface numbers in parentheses refer to the references at the end of this standard.

<sup>&</sup>lt;sup>3</sup> Annual Book of ASTM Standards, Vol 03.01.

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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  $\mu$ 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 2 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 2	Electrolytes	for Electro	polishing
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alloys with less than nt Si ırbon, alloy, stainless ı, Pb-Sn-Cd, Pb-Sn-Sb	c Acid and Alcohol With or Wi ethanol (95 %) distilled water perchloric acid (60 %)	thout Organic Addition 800 mL 140 mL 60 mL	30 to 80 35 to 65	15 to 60 s	
nt Ši Irbon, alloy, stainless I, Pb-Sn-Cd, Pb-Sn-Sb	distilled water	140 mL			
, Pb-Sn-Cd, Pb-Sn-Sb			35 to 65	45 4 00	
· · ·				15 to 60 s	
			12 to 35	15 to 60 s	
-Fe, Zn-Al-Cu			20 to 60		
gh Mg alloys					nickel cathode
steel and aluminum	ethanol (95 %)	800 mL	35 to 80	15 to 60 s	
	perchloric acid (60 %)	200 mL			
steel	ethanol (95 %) perchloric acid (65 %)	940 mL 60 mL	30 to 45	15 to 60 s	
iron, Al, Al alloys, Ni,	ethanol (95 %)	700 mL	30 to 65	15 to 60 s	one of the best formulas for
Sn, Ag, Be, Ti, Zr, U,	2-butoxy ethanol	100 mL			universal use
isting alloys	perchloric acid (30 %)	200 mL			
ainless, alloy,	ethanol (95 %)	700 mL	15 to 50	15 to 60 s	universal electrolyte comparable to
eed Fe Al 7r Ph	glycerin	100 mL			I-4
$\Box \Box U, I \Box, \Box, \Box, \Box I, \Box U$	perchloric acid (30 %)	200 mL			
sis ai	ting alloys	titing alloys perchloric acid (30 %) nless, alloy, ethanol (95 %) ed; Fe, Al, Zr, Pb glycerin	titing alloys perchloric acid (30 %) 200 mL nless, alloy, ethanol (95 %) 700 mL ed; Fe, Al, Zr, Pb glycerin 100 mL	titing alloysperchloric acid (30 %)200 mLnless, alloy,ethanol (95 %)700 mL15 to 50ed; Fe, Al, Zr, Pbglycerin100 mL	titing alloysperchloric acid (30 %)200 mL200 mLnless, alloy,ethanol (95 %)700 mL15 to 5015 to 60 sed; Fe, Al, Zr, Pbglycerin100 mL

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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
itting or etching at edges of specimen	too viscous or thick film	(1) decrease voltage
		(2) increase agitation
		(3) use less viscous electrolyte
udge settling on surface	insoluble anode product	(1) try new electrolyte
		(2) increase temperature
		(3) increase voltage
oughness or matte surface	insufficient or no polishing film	(1) increase voltage
		(2) use more viscous electrolyte
aviness or streaks on polished surface	<ol> <li>insufficient time</li> </ol>	<ol> <li>increase or decrease agitation</li> </ol>
	(2) incorrect agitation	(2) better preparation
	(3) inadequate preparation	(3) increase voltage and decrease time
aine on natiohad aurface	(4) too much time	(d) remains an eximen while surrent is still an
ains on polished surface	attack after polishing current is off	<ol> <li>remove specimen while current is still on</li> <li>the last spreak to be structure.</li> </ol>
analishad anota (bullaavaa)		(2) try less corrosive electrolyte
npolished spots (bullseyes)	gas bubbles	(1) increase agitation
access in relief	incufficient poliching film	(2) decrease voltage
hases in relief	insufficient polishing film	(1) increase voltage
		(2) better preparation
tting	(1) too long poliphing	(3) decrease time
tting	(1) too long polishing	(1) better preparation
	(2) too high voltage	(2) decrease voltage
		(3) decrease time
		(4) try different electrolyte

# TABLE 1 Electropolishing Procedural Problems and Corrections

# TABLE 2 Continued

Class	Use	Formula		Cell Voltage	Time	Remarks
I-6	AI, AI-Si alloys	ethanol (95 %)	760 mL	35 to 60	15 to 60 s	particularly good with Al-Si alloys
		diethyl ether	190 mL			
		perchloric acid (30 %)	50 mL			
I-7	Mo, Ti, Zr, U-Zr alloy	methanol (absolute)	600 mL	60 to 150	5 to 30 s	
	-	2-butoxy ethanol	370 mL			
		perchloric acid (60 %)	30 mL			
I-8	Al-Si alloys	methanol (absolute)	840 mL	50 to 100	5 to 60 s	
	2	glycerin	125 mL			
		perchloric acid (65 %)	35 mL			
-9	vanadium	methanol (absolute)	590 mL	30	3 s	three-second cycles repeated at
		2-butoxy ethanol	350 mL			least seven times to prevent heating
		perchloric acid (65 %)	60 mL			
	germanium			25 to 35	30 to 60 s	
	titanium			58 to 66	45 s	polish only
	zirconium			70 to 75	15 s	polish and etch simultaneously
-10	aluminum	methanol (absolute)	950 mL	30 to 60	15 to 60 s	·····
		nitric acid	15 mL			
		perchloric acid (60 %)	50 mL			
-11	steels-carbon, alloy, stainless	methanol (absolute)	600 mL	30–40	5–60 s	good all purpose electropolish
	Ti, high-temperature alloys, Pb,	butylcellosolve	360 mL	00 10	0 00 0	geod an purpose electropenen
	Mo	perchloric acid	60 mL			
-12	Al and Al alloys	ethanol (95 %)	1000 mL	10	2 min	not good for Al-Cu and Al-Si alloys.
12		perchloric acid	200 mL	10	2 11111	Black film forms. Peel off after 1–1.
		percilione acid	200 IIIL			min and polish 1 min more.
-13	steel, Al, Ni, Sn, Ti, Be	ethanol (95 %)	700 mL	20	20 s	Mix ethanol and water, add
1-13	stainless steel	butylcellosolve	100 mL	20	20 5	,
	Al <sub>3</sub> Ni	water	137 mL			perchloric acid carefully. Then, add butylcellosolve before use.
		perchloric acid	62 mL			
-14	Ni. Ag or Cu ollovo		700 mL	70–80	15 s	
-14	Ni, Ag or Cu alloys Cd	ethanol (95 %) butylcellosolve	100 mL	70-00	15.5	
	Cu		200 mL			
45		perchloric acid			00 -	Mission attraction of supervision and a
-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			
Group	II (Electrolytes Composed of Perchloric A	Acid and Glacial Acetic Acid in	Varying Proportions	)	7	
I-1	Cr, Ti, Zr, U	acetic acid (glacial)	940 mL	20 to 60	1 to 5 min	good general-purpose electrolyte
	Fe, steel-carbon, alloy, stainless	perchloric acid (60 %)	60 mL			
I-2	Zr, Ti, U, steel-carbon and alloy	acetic acid (glacial)	900 mL	12 to 70	0.5 to 2 min	
	· · · ·	perchloric acid (60 %)	100 mL			
1-3	U, Zr, Ti, Al, steel—carbon and	acetic acid (glacial) ASI	800 mL	40 to 100	1 to 15 min	
	alloy	perchloric acid (60 %)	200 mL			
1-4	Ni, Pb, Pb-Sb alloys	acetic acid (glacial)	700 mL a9-	40 to 100	1 to 5 min	
• •		perchloric acid (60 %)	300 mL	10 10 100		
I-5	3 percent Si-Fe	acetic acid (glacial)	650 mL		5 min	0.06 A/cm <sup>2</sup>
. 0		perchloric acid (60 %)	350 mL		0 11111	0.0070011
-6	Cr	acetic acid (glacial)	1000 mL	30–50	2–3 min	can lower voltage to 25 V by adding
	<u>.</u>	perchloric acid	5 mL	50 50	2 0 11111	5–15 % water.
I-7	Hf, steel-carbon and alloy	acetic acid (glacial)	1000 mL			Used to polish Hf wires.
1.7						used to polish i il wiles.
		perchloric acid	50 mL			

TABLE 2 Continued

Class	Use	Formula		Cell Voltage	Time	Remarks
Group	III (Electrolytes Composed of Phospho	oric Acid in Water or Organic Solven	it)			
II-1	cobalt	phosphoric acid (85 %)	1000 mL	1.2	3 to 5 min	
II-1 II-2		distilled water		1.2 1.0 to 1.6		connor acthoda
111-2	pure copper		175 mL	1.0 10 1.0	10 10 40 11111	copper cathode
		phosphoric acid (85 %)	825 mL			
II-3	stainless, brass, Cu and Cu	water	300 mL	1.5 to 1.8	5 to 15 min	copper cathode
	alloys except Sn bronze	phosphoric acid (85 %)	700 mL			
111-4	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			
III-5	Cu, Cu-Zn	water	1000 mL	1 to 2	10 min	copper cathode
	60, 60 Zh	pyrophosphoric acid	580 g	1 10 2	10 min	
ui c	ataal			E to 20	E to 1E min	120°E
III-6 st	steel	diethylene glycol monoethyl	500 mL	5 to 20	5 to 15 min	120°F
		ether	500 ml			
		phosphoric acid (85 %)	500 mL			
-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			
III-8	uranium	ethanol (absolute)	300 mL			
		glycerin (cp)	300 mL			
		phosphoric acid (85 %)	300 mL			
111-9	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	
111-10	eu and eu-base anoys		250 mL		1 to 5 min	
		ethanol (95 %)				
		phosphoric acid (85 %)	250 mL			
III-11	stainless steel	ethanol (absolute), to	1 L		10 min	good for all austenitic heat resistar
		pyrophosphoric acid	400 g			alloys, 100°F 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 <sup>2</sup>
		ethylene glycol	275 mL			
		phosphoric acid (85 %)	275 mL			
III-14	Al-Mg alloys	water	250 mL	50-60	2 min	
	A my anoys	ethanol (95 %)	380 mL		2 11011	
			400 mL			
	On Phaellene	phosphoric acid (85 %)				need on the 20 % Dh
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 600-grit SiC, use 6-µm
		glycerol	400 mL			diamond on nylon before
		phosphoric acid (85 %)	800 mL			electropolishing.
		Group IV (Electrolytes Composed of	Sulfuric Acid in '	Water or Organic	: Solvent)	
	G					4/95/90m_0155X_00
V-1	htt <del>ne//etandarde iteh ai/cat</del> a	water	250 ml	1.5 to 6	1 to 2 min	
V-1	G stainless steel	water sulfuric acid	250 mL	1.5 to 6	1 to 2 min	
	stainless steel	sulfuric acid	750 mL			
	htt <del>ne//etandarde iteh ai/cat</del> a	sulfuric acid water	750 mL 400 mL	1.5 to 6	1 to 2 min 2 to 6 min	
IV-2	stainless steel ands. itch. av cata stainless steel, Fe, Ni	sulfuric acid water sulfuric acid	750 mL 400 mL 600 mL	1.5 to 6	2 to 6 min	
V-2	stainless steel	sulfuric acid water sulfuric acid water	750 mL 400 mL 600 mL 750 mL		2 to 6 min 2 to 10 min	particularly good for sintered
IV-2	stainless steel ands. itch. av cata stainless steel, Fe, Ni	sulfuric acid water sulfuric acid	750 mL 400 mL 600 mL	1.5 to 6	2 to 6 min 2 to 10 min Mo—	particularly good for sintered Mo—32 to 80°F
IV-2	stainless steel ands. itch. av cata stainless steel, Fe, Ni	sulfuric acid water sulfuric acid water	750 mL 400 mL 600 mL 750 mL	1.5 to 6	2 to 6 min 2 to 10 min	
IV-2 IV-3	stainless steel ands. itch. av cata stainless steel, Fe, Ni	sulfuric acid water sulfuric acid water	750 mL 400 mL 600 mL 750 mL	1.5 to 6	2 to 6 min 2 to 10 min Mo—	
IV-2 IV-3	stainless steel ands. iteh. aveau stainless steel, Fe, Ni stainless steel, Fe, Ni, Mo	sulfuric acid water sulfuric acid water sulfuric acid	750 mL 400 mL 600 mL 750 mL 250 mL	1.5 to 6 1.5 to 6	2 to 6 min 2 to 10 min Mo— 0.3 to 1 min	Mo-32 to 80°F
IV-2 IV-3 IV-4	stainless steel ands. ttoh. av cata stainless steel, Fe, Ni stainless steel, Fe, Ni, Mo molybdenum	sulfuric acid water sulfuric acid water sulfuric acid water	750 mL 400 mL 600 mL 750 mL 250 mL 900 mL 100 mL	1.5 to 6 1.5 to 6 1.5 to 6	2 to 6 min 2 to 10 min Mo— 0.3 to 1 min 0.3 to 2 min	Mo-32 to 80°F particularly good for sintered
IV-2 IV-3 IV-4	stainless steel ands. iteh. aveau stainless steel, Fe, Ni stainless steel, Fe, Ni, Mo	sulfuric acid water sulfuric acid water sulfuric acid water sulfuric acid water	750 mL 400 mL 600 mL 750 mL 250 mL 900 mL 100 mL 70 mL	1.5 to 6 1.5 to 6	2 to 6 min 2 to 10 min Mo— 0.3 to 1 min	Mo-32 to 80°F particularly good for sintered
IV-2 IV-3 IV-4	stainless steel ands. ttoh. av cata stainless steel, Fe, Ni stainless steel, Fe, Ni, Mo molybdenum	sulfuric acid water sulfuric acid water sulfuric acid water sulfuric acid water glycerin	750 mL 400 mL 600 mL 750 mL 250 mL 900 mL 100 mL 70 mL 200 mL	1.5 to 6 1.5 to 6 1.5 to 6	2 to 6 min 2 to 10 min Mo— 0.3 to 1 min 0.3 to 2 min	Mo-32 to 80°F particularly good for sintered
IV-2 IV-3 IV-4 IV-5	stainless steel ands. Itch. av cata stainless steel, Fe, Ni stainless steel, Fe, Ni, Mo molybdenum stainless steel	sulfuric acid water sulfuric acid water sulfuric acid water sulfuric acid water glycerin sulfuric acid	750 mL 400 mL 600 mL 750 mL 250 mL 900 mL 100 mL 70 mL 200 mL 720 mL	1.5 to 6 1.5 to 6 1.5 to 6 1.5 to 6	2 to 6 min 2 to 10 min Mo— 0.3 to 1 min 0.3 to 2 min 0.5 to 5 min	Mo-32 to 80°F particularly good for sintered
V-2 V-3 V-4 V-5	stainless steel ands. ttoh. av cata stainless steel, Fe, Ni stainless steel, Fe, Ni, Mo molybdenum	sulfuric acid water sulfuric acid water sulfuric acid water sulfuric acid water glycerin sulfuric acid water	750 mL 400 mL 600 mL 750 mL 250 mL 900 mL 100 mL 70 mL 200 mL 720 mL 220 mL	1.5 to 6 1.5 to 6 1.5 to 6	2 to 6 min 2 to 10 min Mo— 0.3 to 1 min 0.3 to 2 min	Mo-32 to 80°F particularly good for sintered
V-2 V-3 V-4 V-5	stainless steel ands. Itch. av cata stainless steel, Fe, Ni stainless steel, Fe, Ni, Mo molybdenum stainless steel	sulfuric acid water sulfuric acid water sulfuric acid water sulfuric acid water glycerin sulfuric acid water glycerin	750 mL 400 mL 600 mL 750 mL 250 mL 900 mL 100 mL 70 mL 720 mL 220 mL 220 mL	1.5 to 6 1.5 to 6 1.5 to 6 1.5 to 6	2 to 6 min 2 to 10 min Mo— 0.3 to 1 min 0.3 to 2 min 0.5 to 5 min	Mo-32 to 80°F particularly good for sintered
IV-2 IV-3 IV-4 IV-5 IV-6	stainless steel ands. Itch. av cata stainless steel, Fe, Ni stainless steel, Fe, Ni, Mo molybdenum stainless steel stainless steel, aluminum	sulfuric acid water sulfuric acid water sulfuric acid water sulfuric acid water glycerin sulfuric acid water glycerin sulfuric acid	750 mL 400 mL 600 mL 750 mL 250 mL 900 mL 100 mL 70 mL 200 mL 220 mL 220 mL 220 mL 280 mL	1.5 to 6 1.5 to 6 1.5 to 6 1.5 to 6 1.5 to 12	2 to 6 min 2 to 10 min Mo— 0.3 to 1 min 0.3 to 2 min 0.5 to 5 min 1 to 20 min	Mo—32 to 80°F particularly good for sintered Mo—32 to 80°F
IV-2 IV-3 IV-4 IV-5 IV-6	stainless steel ands. Itch. av cata stainless steel, Fe, Ni stainless steel, Fe, Ni, Mo molybdenum stainless steel	sulfuric acid water sulfuric acid water sulfuric acid water sulfuric acid water glycerin sulfuric acid water glycerin sulfuric acid methanol (absolute)	750 mL 400 mL 600 mL 750 mL 250 mL 900 mL 100 mL 70 mL 200 mL 200 mL 200 mL 200 mL 280 mL 875 mL	1.5 to 6 1.5 to 6 1.5 to 6 1.5 to 6	2 to 6 min 2 to 10 min Mo— 0.3 to 1 min 0.3 to 2 min 0.5 to 5 min	Mo—32 to 80°F particularly good for sintered Mo—32 to 80°F
IV-2 IV-3 IV-4 IV-5 IV-6 IV-7	stainless steel ands. Itch. av cata stainless steel, Fe, Ni stainless steel, Fe, Ni, Mo molybdenum stainless steel stainless steel, aluminum	sulfuric acid water sulfuric acid water sulfuric acid water sulfuric acid water glycerin sulfuric acid water glycerin sulfuric acid methanol (absolute) sulfuric acid	750 mL 400 mL 600 mL 750 mL 250 mL 900 mL 100 mL 70 mL 200 mL 220 mL 220 mL 220 mL 280 mL	1.5 to 6 1.5 to 6 1.5 to 6 1.5 to 6 1.5 to 12	2 to 6 min 2 to 10 min Mo— 0.3 to 1 min 0.3 to 2 min 0.5 to 5 min 1 to 20 min	Mo—32 to 80°F particularly good for sintered Mo—32 to 80°F
IV-2 IV-3 IV-4 IV-5 IV-6 IV-7	stainless steel ands. Itch. av cata stainless steel, Fe, Ni stainless steel, Fe, Ni, Mo molybdenum stainless steel stainless steel, aluminum	sulfuric acid water sulfuric acid water sulfuric acid water sulfuric acid water glycerin sulfuric acid water glycerin sulfuric acid methanol (absolute)	750 mL 400 mL 600 mL 750 mL 250 mL 900 mL 100 mL 70 mL 200 mL 200 mL 200 mL 200 mL 280 mL 875 mL	1.5 to 6 1.5 to 6 1.5 to 6 1.5 to 6 1.5 to 12	2 to 6 min 2 to 10 min Mo— 0.3 to 1 min 0.3 to 2 min 0.5 to 5 min 1 to 20 min	Mo—32 to 80°F particularly good for sintered Mo—32 to 80°F
IV-2 IV-3 IV-4 IV-5 IV-6 IV-7	stainless steel ands. Itch. av cata stainless steel, Fe, Ni stainless steel, Fe, Ni, Mo molybdenum stainless steel stainless steel, aluminum molybdenum	sulfuric acid water sulfuric acid water sulfuric acid water sulfuric acid water glycerin sulfuric acid water glycerin sulfuric acid methanol (absolute) sulfuric acid	750 mL 400 mL 600 mL 750 mL 250 mL 900 mL 100 mL 70 mL 200 mL 220 mL 220 mL 200 mL 580 mL 875 mL 125 mL	1.5 to 6 1.5 to 6 1.5 to 6 1.5 to 6 1.5 to 12 6 to 18	2 to 6 min 2 to 10 min Mo— 0.3 to 1 min 0.3 to 2 min 0.5 to 5 min 1 to 20 min 0.5 to 1.5 min	Mo—32 to 80°F particularly good for sintered Mo—32 to 80°F 32 to 80°F
IV-2 IV-3 IV-4 IV-5 IV-6 IV-7	stainless steel ands. Itch. av cata stainless steel, Fe, Ni stainless steel, Fe, Ni, Mo molybdenum stainless steel stainless steel, aluminum molybdenum	sulfuric acid water sulfuric acid water sulfuric acid water sulfuric acid water glycerin sulfuric acid water glycerin sulfuric acid methanol (absolute) sulfuric acid methanol (absolute)	750 mL 400 mL 600 mL 750 mL 250 mL 900 mL 100 mL 70 mL 200 mL 200 mL 200 mL 200 mL 875 mL 125 mL 800 mL 200 mL	1.5 to 6 1.5 to 6 1.5 to 6 1.5 to 6 1.5 to 12 6 to 18 30	2 to 6 min 2 to 10 min Mo— 0.3 to 1 min 0.3 to 2 min 0.5 to 5 min 1 to 20 min 0.5 to 1.5 min	Mo—32 to 80°F particularly good for sintered Mo—32 to 80°F 32 to 80°F
IV-2 IV-3 IV-4 IV-5 IV-6 IV-7 IV-8	stainless steel ands. Itch. ar cata stainless steel, Fe, Ni stainless steel, Fe, Ni, Mo molybdenum stainless steel stainless steel, aluminum molybdenum Ni-base superalloys	sulfuric acid water sulfuric acid water sulfuric acid water sulfuric acid water glycerin sulfuric acid water glycerin sulfuric acid methanol (absolute) sulfuric acid methanol (absolute) sulfuric acid Group V (Electrolytes Com	750 mL 400 mL 600 mL 750 mL 250 mL 900 mL 100 mL 70 mL 200 mL 220 mL 220 mL 200 mL 580 mL 875 mL 125 mL 800 mL 200 mL	1.5 to 6 1.5 to 6 1.5 to 6 1.5 to 6 1.5 to 12 6 to 18 30	2 to 6 min 2 to 10 min Mo— 0.3 to 1 min 0.3 to 2 min 0.5 to 5 min 1 to 20 min 0.5 to 1.5 min 20 s	Mo—32 to 80°F particularly good for sintered Mo—32 to 80°F 32 to 80°F
IV-2 IV-3 IV-4 IV-5 IV-6 IV-7 IV-8	stainless steel ands. Itch. av cata stainless steel, Fe, Ni stainless steel, Fe, Ni, Mo molybdenum stainless steel stainless steel, aluminum molybdenum	sulfuric acid water sulfuric acid water sulfuric acid water sulfuric acid water glycerin sulfuric acid water glycerin sulfuric acid methanol (absolute) sulfuric acid methanol (absolute) sulfuric acid Group V (Electrolytes Com water	750 mL 400 mL 600 mL 750 mL 250 mL 900 mL 100 mL 70 mL 200 mL 200 mL 200 mL 200 mL 580 mL 875 mL 125 mL 800 mL 200 mL 900 mL 830 mL	1.5 to 6 1.5 to 6 1.5 to 6 1.5 to 6 1.5 to 12 6 to 18 30	2 to 6 min 2 to 10 min Mo— 0.3 to 1 min 0.3 to 2 min 0.5 to 5 min 1 to 20 min 0.5 to 1.5 min	Mo—32 to 80°F particularly good for sintered Mo—32 to 80°F 32 to 80°F
IV-2 IV-3 IV-4 IV-5 IV-6 IV-7 IV-8 	stainless steel ands. tich. ar cata stainless steel, Fe, Ni stainless steel, Fe, Ni, Mo molybdenum stainless steel stainless steel, aluminum molybdenum Ni-base superalloys stainless steel	sulfuric acid water sulfuric acid water sulfuric acid water sulfuric acid water glycerin sulfuric acid water glycerin sulfuric acid methanol (absolute) sulfuric acid methanol (absolute) sulfuric acid Group V (Electrolytes Com water chromic acid	750 mL 400 mL 600 mL 750 mL 250 mL 900 mL 100 mL 70 mL 200 mL 200 mL 200 mL 200 mL 580 mL 875 mL 125 mL 800 mL 200 mL 900 mL 830 mL 620 g	1.5 to 6 1.5 to 6 1.5 to 6 1.5 to 6 1.5 to 12 6 to 18 30 ic Acid in Water) 1.5 to 9	2 to 6 min 2 to 10 min Mo— 0.3 to 1 min 0.3 to 2 min 0.5 to 5 min 1 to 20 min 0.5 to 1.5 min 20 s 2 to 10 min	Mo—32 to 80°F particularly good for sintered Mo—32 to 80°F 32 to 80°F
IV-1 IV-2 IV-3 IV-4 IV-5 IV-6 IV-7 IV-8 V-1 V-1 V-2	stainless steel ands. Itch. ar cata stainless steel, Fe, Ni stainless steel, Fe, Ni, Mo molybdenum stainless steel stainless steel, aluminum molybdenum Ni-base superalloys	sulfuric acid water sulfuric acid water sulfuric acid water sulfuric acid water glycerin sulfuric acid water glycerin sulfuric acid methanol (absolute) sulfuric acid methanol (absolute) sulfuric acid Group V (Electrolytes Com water	750 mL 400 mL 600 mL 750 mL 250 mL 900 mL 100 mL 70 mL 200 mL 200 mL 200 mL 200 mL 580 mL 875 mL 125 mL 800 mL 200 mL 900 mL 830 mL	1.5 to 6 1.5 to 6 1.5 to 6 1.5 to 6 1.5 to 12 6 to 18 30	2 to 6 min 2 to 10 min Mo— 0.3 to 1 min 0.3 to 2 min 0.5 to 5 min 1 to 20 min 0.5 to 1.5 min 20 s	Mo—32 to 80°F particularly good for sintered Mo—32 to 80°F 32 to 80°F

TABLE 2 Continued

Class	Use	Formula		Cell Voltage	Time	Remarks
		Group VI (Mixed Acids or Sa	Its in Water or	Organic Solvent)		
/I-1	stainless steel	phosphoric acid (85 %)	600 mL			
		sulfuric acid	400 mL			2
1-2	stainless steel	water	150 mL		2 min	0.3 A/cm <sup>2</sup>
		phosphoric acid (85 %) sulfuric acid	300 mL			
-3	stainless and alloy steel	water	550 mL 240 mL		2 to 10 min	0.1 to 0.2 A/cm <sup>2</sup>
0		phosphoric acid (85 %)	420 mL		2 10 10 11111	0.1 10 0.2 //0.11
		sulfuric acid	340 mL			
-4	stainless steel	water	330 mL		1 min	0.05 A/cm <sup>2</sup>
		phosphoric acid (85 %)	550 mL			
-		sulfuric acid	120 mL			24.44 2
-5	bronze (to 9 % Sn)	water	450 mL 390 mL		1 to 5 min	0.1 A/cm <sup>2</sup>
		phosphoric acid (85 %) sulfuric acid	160 mL			
6	bronze (to 6 % Sn)	water	330 mL		1 to 5 min	0.1 A/cm <sup>2</sup>
0		phosphoric acid (85 %)	580 mL			
		sulfuric acid	90 mL			
-7	steel	water	140 mL		1 to 5 min	1 to 5 A/cm <sup>2</sup> , 100°F plus
		glycerin	100 mL			
		phosphoric acid (85 %)	430 mL			
0	atainlaga ataol	sulfuric acid	330 mL		E min	$1  \text{A/om}^2$ 90 to $100^{\circ}\text{F}$
В	stainless steel	water glycerin	200 mL 590 mL		5 min	1 A/cm <sup>2</sup> , 80 to 120°F
		phosphoric acid (85 %)	100 mL			
		sulfuric acid	110 mL			
9	stainless steel	water	260 mL		30 min	0.6 A/cm <sup>2</sup> , 80 to 120°F
		chromic acid	175 g			
		phosphoric acid (85 %)	175 mL			
		sulfuric acid	580 mL			
10	stainless steel	water	175 mL		60 min	0.5 A/cm <sup>2</sup> , 80 to 120°F
		chromic acid	105 g			
		phosphoric acid (85 %) sulfuric acid	460 mL 390 mL			
11	stainless and alloy steel	water	240 mL		5 to 60 min	0.5 to A/cm <sup>2</sup> , 100 to 130°F
••		chromic acid	80 g			
		phosphoric acid (85 %)	650 mL			
		sulfuric acid	130 mL			
12	tantalum	hydrofluoric acid	100 mL		9 min	graphite cathode, 0.1 A/cm <sup>2</sup> , 90 to
4.0		sulfuric acid	900 mL		<u> </u>	100°F
13	stainless steel	water <u>ASTM</u>	210 mL	)	5 min	0.5 A/cm <sup>2</sup> , 70 to 120°F
		hydrofluoric acid	180 mL 610 mL			
14	zinc	water	800 mL	-49a4-au20	-acc102u	0.002 A/cm <sup>2</sup> , 70 to 100°F
		chromic acid	100 g			
		sulfuric acid	46 mL			
		sodium dichromate	310 g			
		acetic acid (glacial)	96 mL			
-15	stainless steel	hydrogen peroxide (30 %)	260 mL		5 min	0.5 A/cm <sup>2</sup> (Caution) Dangerous
		(Caution) hydrofluoric acid	240 mL			
		sulfuric acid	500 mL			
16	stainless steel	water	520 mL		1/2 to 4 min	0.08 to 0.3 A/cm <sup>2</sup>
		hydrofluoric acid	80 mL		/	
		sulfuric acid	400 mL			
17	stainless steel	water	600 mL			
		chromic acid	180 g			
		nitric acid	60 mL			
		hydrochloric acid	3 mL			
18	bismuth	sulfuric acid glycerin	240 mL 750 mL	12	1 to 5 min	$0.5 \pm A/cm^2$ (Caution) This mixture
10	biomutif	acetic acid (glacial)	125 mL	12		will decompose vigorously after a
		nitric acid	125 mL			short time. Do not try to keep.
19	magnesium	ethylene-glycol-monoethyl ether	900 mL	50 to 60	10 to 30 s	Bath should be stirred. Cool cracke
	-	hydrochloric acid	100 mL			ice below 35°F
-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.

TABLE 2 Continued

		IABLE 2	Continueu			
Class	Use	Formula		Cell Voltage	Time	Remarks
		Group VI (Mixed Acids or Salts in	Water or Organ	nic Solvent)—Co	ontinued	
/I-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) zinc chloride (anhydrous)	250 a			
-22	uranium	acetic acid (glacial)	250 g 750 mL	80	5 to 30 min	The chromic acid is dissolved in the
. 22	didition	distilled water	210 mL	00	0 10 00 11111	water before adding to the acetic
		chromic acid	180 g			acid. Below 35°F.
I-23	pure zinc	ethanol (95 %)	720 mL	25 to 40	0.5 to 3 min	(Caution) Anhydrous aluminum
		aluminum chloride (anhydrous)	50 g			chloride is extremely dangerous to
		(Caution)				handle. Below 60°F.
		zinc chloride (anhydrous)	225 g 160 mL			
		distilled water n-butyl alcohol	80 mL			
I-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	0 10 12		standing, dangerous if kept too long
		nitric acid	87 mL			
′I-25	bismuth	saturated solution KI in distilled	980 mL	7	30 s	polish 30 s but allow to remain in
		water				electrolyte until brown film is
		hydrochloric acid	20 mL			dissolved
1-26	Sb	methanol (absolute)	300 mL	6–10	2–4 min	pure Sb. Use Pt cathode and anod
		sulfuric acid	50 mL			lead wires. Agitate bath. Do not
1.07	0h	hydrochloric acid	30 mL			touch polished surface with cotton.
1-27	Sb	ethanol (95 %) glycerol	30 mL 30 mL			good for polarized light work
		phosphoric acid	100 mL			
		sulfuric acid	30 mL			
′I-28	Ві	water	200 mL			good for polarized light work
		phosphoric acid 🦳 👘 🦷	100 mL			
	_	sulfuric acid	200 mL			
I-29	Cr	water	210 mL	18		stir bath or specimen
		phosphoric acid	640 mL			
I-30	Ge	sulfuric acid methanol (absolute)	150 mL 1000 mL			
1-30	Ge	hydrochloric acid	10 mL			
I-31	Nb	water	300 mL	e <sub>40</sub> e v		polish to $\alpha$ -alumina before
		sulfuric acid	100 mL			electropolishing
		hydrofluoric acid	100 mL			
1-32	Nb	methanol (absolute)	940 mL	50-60	10 s	
		sulfuric acid <u>ASTIM</u>	50 mL-99			
1-33	Ni-base superalloy	hydrofluoric acid methanol (absolute)	a 15 mL 170 mL a9-	-49304-ad2e	-20 s 1 b 2 d 3	for Waspaloy and IN-100 mod. Etcl
1-55	Ni-base superality	hydrochloric acid	30 mL	30	20 5	at 5 V for 4 s.
		,	kaline Electrolyte	(20		
'II-1	gold	water to	1000 mL	7.5	2 to 4 min	graphite cathode
		potassium cyanide potassium carbonate	80 g 40 g			
		gold chloride	40 g 50 g			
II-2	silver	water to	1000 mL	2.5	To 1 min	graphite cathode
		sodium cyanide	100 g	2.0	10 1 1111	graphile callede
		potassium ferrocyanide	100 g			
'II-3	silver	water to	1000 mL		To 9 min	graphite cathode, 0.003 to 0.009
		potassium cyanide	400 g			A/cm <sup>2</sup>
		silver cyanide	280 g			
		potassium dichromate	280 g		40 .	
11-4	tungsten	water to trisodium phosphate	1000 mL		10 min	graphite cathode, 0.09 A/cm <sup>2</sup> , 100 120 F
/11-5	tungsten, lead	water to	160 g 1000 mL		8 to 10 min	graphite cathode, 0.03 to 0.06 A/cm
		sodium hydroxide	100 g		5.6 10 1111	g.apinto bathbao, 0.00 to 0.00 A/01
11-6	zinc, tin	water to	1000 mL	2 to 6	15 min	copper cathode, 0.1 to 0.2 A/cm <sup>2</sup>
-		potassium hydroxide	200 g			
/11-7	W	water	1000 mL		5 min	
		sodium hydroxide	20 g			
		Group VIII (Mixture of M	ethyl Alcohol ar	nd Nitric Acid)		
/111-1	Ni, Cu, Zn, Monel, brass,	Group VIII (Mixture of M methanol (absolute)	660 mL	40 to 70	10 to 60 s	very useful but dangerous