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Standard Guide for Electrolytic Polishing of Metallographic Specimens¹

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1. Scope

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

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

- 1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.
- 1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific safety precautions are described in Section 5 and 6.3.1.

2. Referenced Documents

2.1 ASTM Standards:³

E7 Terminology Relating to Metallography

E407 Practice for Microetching Metals and Alloys

3. Terminology

- 3.1 Definitions—All terms used in this guide are either defined in Terminology E7 or are discussed in 3.2.
- 3.2 Definitions of Terms Specific to This Standard:
- 3.2.1 *electrolytic polish (electropolish)*—A method of polishing metals and alloys in which material is removed from the surface by making the metal the anode in an electrolytic bath.

4. Significance and Use

- 4.1 Advantages of Electrolytic Polishing:
- 4.1.1 For some metals, a high quality surface finish can be produced that is equivalent to, or better than, that which can be obtained by mechanical methods.
 - 4.1.2 Once procedures have been established, satisfactory results can be obtained rapidly with reproducibility.
 - 4.1.3 There can be a marked saving of time if many specimens of the same material are polished sequentially.
- 4.1.4 Electropolishing a selected area on the surface of a relatively large metal part can be accomplished nondestructively, that is, without the need for sectioning to remove a piece.
 - 4.1.5 Soft, single-phase metals, which may be difficult to polish by mechanical methods, may be successfully electropolished.
- 4.1.6 The true microstructure of a specimen can be obtained because artifacts (such as disturbed metal, scratches, and mechanical twins) produced on the surface, even by careful grinding and mechanical polishing operations, can be removed. These features are important in low-load hardness testing, X-ray diffraction studies, and in electron microscopy, where higher resolution puts a premium on undistorted metal surfaces.
- 4.1.7 After electropolishing is completed, etching can often be accomplished by reducing the voltage (generally to about one-tenth that required for polishing) for a short time before it is turned off.

Note 2—Not all electropolishing solutions produce good etching results.

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² The **boldface** numbers in parentheses refer to the references at the end of this standard.

³ For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.



- 4.2 Disadvantages of Electrolytic Polishing:
- 4.2.1 Many of the chemical mixtures used in electropolishing are poisonous or dangerous if not properly handled (see Section 5). These hazards are similar to those involved in the mixing and handling of etchants, see Test Methods E407.
 - 4.2.2 In multi-phase alloys, the polishing rate of each phase may be different. The result may be a non-planar surface.
- 4.2.3 Electropolished surfaces may be slightly undulated rather than perfectly planar and, therefore, may not be suitable for examination at all magnifications.
- 4.2.4 The rate of polishing in areas adjacent to various inhomogeneities, such as nonmetallic inclusions and voids, is usually greater than that in the surrounding matrix and tends to exaggerate the size of the inclusions and voids.
- 4.2.5 Dimples, pits, and waviness limit applications involving surface phenomena, coatings, interfaces, and cracks. Edges tend to be attacked preferentially, resulting in edge rounding.
 - 4.2.6 Artifacts may be produced by electropolishing.
 - 4.2.7 Specimen mounting materials may react with the electrolyte.
 - 4.2.8 The electropolished surfaces of certain materials may be passive and difficult to etch.
- 4.2.9 Metal removal rates by electropolishing are usually quite low, typically about 1 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 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.



TABLE 1 Electrolytes for Electropolishing

Group I (I-1		Formula		Cell Voltage	Time	Remarks
I-1	(Electrolytes Composed of Perchloric Aci					ted fume hood.
	Al and Al alloys with less than	ethanol (95 %)	800 mL	30 to 80	15 to 60 s	
	2 percent Si	distilled water perchloric acid (60 %)	140 mL 60 mL			
	steels—carbon, alloy, stainless	perchionic acid (60 %)	00 IIIL	35 to 65	15 to 60 s	
	Pb, Pb-Sn, Pb-Sn-Cd, Pb-Sn-Sb			12 to 35	15 to 60 s	
	Zn, Zn-Sn-Fe, Zn-Al-Cu			20 to 60		
	Mg and high Mg alloys					nickel cathode
I-2	stainless steel and aluminum	ethanol (95 %)	800 mL	35 to 80	15 to 60 s	
		perchloric acid (60 %)	200 mL			
I-3	stainless steel	ethanol (95 %)	940 mL	30 to 45	15 to 60 s	
		perchloric acid (65 %)	60 mL			
I-4	steel, cast iron, Al, Al alloys, Ni,	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
	heat-resisting alloys	perchloric acid (30 %)	200 mL		4= 4 00	
I-5	steels—stainless, alloy,	ethanol (95 %)	700 mL	15 to 50	15 to 60 s	universal electrolyte comparable to
	high-speed; Fe, Al, Zr, Pb	glycerin	100 mL			1-4
I-6	AL ALSi allove	perchloric acid (30 %) ethanol (95 %)	200 mL 760 mL	35 to 60	15 to 60 c	particularly good with Al Si alloys
1-0	Al, Al-Si alloys	diethyl ether	190 mL	35 to 60	15 to 60 s	particularly good with Al-Si alloys
		perchloric acid (30 %)	50 mL			
I-7	Mo, Ti, Zr, U-Zr alloy	methanol (absolute)	600 mL	60 to 150	5 to 30 s	
	Wo, 11, 21, 0 21 alloy	2-butoxy ethanol	370 mL	00 10 100	0 10 00 0	
		perchloric acid (60 %)	30 mL			
l - 8	Al-Si alloys	methanol (absolute)	840 mL	50 to 100	5 to 60 s	
	•	glycerin	125 mL			
		perchloric acid (65 %)	35 mL			
I-9	vanadium	methanol (absolute)	590 mL	30	3 s	three-second cycles repeated at
		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
I-10	aluminum	methanol (absolute)	950 mL	30 to 60	15 to 60 s	
		nitric acid	15 mL			
1 44	ataala aarban allau atainlaaa	perchloric acid (60 %)	50 mL	20 40	F 60 a	good all numana alastropolish
I-11	steels—carbon, alloy, stainless Ti, high-temperature alloys, Pb,	methanol (absolute) butylcellosolve	600 mL 360 mL	30–40	5–60 s	good all purpose electropolish
	Mo	perchloric acid	60 mL			
l-12	Al and Al alloys	ethanol (95 %)	1000 mL	10	2 min	not good for Al-Cu and Al-Si alloys.
1 12	Al and Al alloys	perchloric acid	200 mL	10	2 111111	Black film forms. Peel off after 1–1.5
		A STM E1	559 00(201/			min and polish 1 min more.
I-13	steel, Al, Ni, Sn, Ti, Be	ethanol (95 %)	700 mL	[±] / 20	20 s	Mix ethanol and water, add
	stainless steel eh ai/catalog/s	butylcellosolve be 2561	d-ee100 mLf76			perchloric acid carefully. Then, add
	Al ₃ Ni	water	137 mL			butylcellosolve before use.
		perchloric acid	62 mL			
		poromono dola	02 IIIL			
l-14	Ni, Ag, or Cu alloys	ethanol (95 %)	700 mL	70–80	15 s	
I-14	Ni, Ag, or Cu alloys Cd	ethanol (95 %) butylcellosolve	700 mL 100 mL	70–80	15 s	
	Cd	ethanol (95 %) butylcellosolve perchloric acid	700 mL 100 mL 200 mL	70–80		
		ethanol (95 %) butylcellosolve perchloric acid methanol (absolute)	700 mL 100 mL 200 mL 600 mL	70–80	15 s 20 s	Mix methanol and water, add
	Cd	ethanol (95 %) butylcellosolve perchloric acid methanol (absolute) water	700 mL 100 mL 200 mL 600 mL 13 mL	70–80		perchloric acid carefully. Add
	Cd	ethanol (95 %) butylcellosolve perchloric acid methanol (absolute) water butylcellosolve	700 mL 100 mL 200 mL 600 mL 13 mL 360 mL	70–80		,
l-15	Cd Mo and Mo alloys	ethanol (95 %) butylcellosolve perchloric acid methanol (absolute) water butylcellosolve perchloric acid	700 mL 100 mL 200 mL 600 mL 13 mL 360 mL 47 mL		20 s	perchloric acid carefully. Add butylcellosolve before use.
I-15 Group II	Cd Mo and Mo alloys (Electrolytes Composed of Perchloric Ac	ethanol (95 %) butylcellosolve perchloric acid methanol (absolute) water butylcellosolve perchloric acid and Glacial Acetic Acid in Val	700 mL 100 mL 200 mL 600 mL 13 mL 360 mL 47 mL rying Proportions) U	Use in a washdo	20 s wn/perchloric ra	perchloric acid carefully. Add butylcellosolve before use.
I-15 Group II	Cd Mo and Mo alloys (Electrolytes Composed of Perchloric AcCr, Ti, Zr, U,	ethanol (95 %) butylcellosolve perchloric acid methanol (absolute) water butylcellosolve perchloric acid sid and Glacial Acetic Acid in Val	700 mL 100 mL 200 mL 600 mL 13 mL 360 mL 47 mL rying Proportions) U		20 s	perchloric acid carefully. Add butylcellosolve before use.
I-15 Group II II-1	Cd Mo and Mo alloys (Electrolytes Composed of Perchloric Ac Cr, Ti, Zr, U, Fe, steel—carbon, alloy, stainless	ethanol (95 %) butylcellosolve perchloric acid methanol (absolute) water butylcellosolve perchloric acid dand Glacial Acetic Acid in Var acetic acid (glacial) perchloric acid (60 %)	700 mL 100 mL 200 mL 600 mL 13 mL 360 mL 47 mL rying Proportions) U 940 mL 60 mL	Use in a washdo 20 to 60	20 s wn/perchloric ra 1 to 5 min	perchloric acid carefully. Add butylcellosolve before use.
Group II	Cd Mo and Mo alloys (Electrolytes Composed of Perchloric AcCr, Ti, Zr, U,	ethanol (95 %) butylcellosolve perchloric acid methanol (absolute) water butylcellosolve perchloric acid did and Glacial Acetic Acid in Val acetic acid (glacial) perchloric acid (60 %) acetic acid (glacial)	700 mL 100 mL 200 mL 600 mL 13 mL 360 mL 47 mL rying Proportions) U 940 mL 60 mL 900 mL	Use in a washdo	20 s wn/perchloric ra	perchloric acid carefully. Add butylcellosolve before use.
Group II II-1 II-2	Cd Mo and Mo alloys (Electrolytes Composed of Perchloric Ac Cr, Ti, Zr, U, Fe, steel—carbon, alloy, stainless Zr, Ti, U, steel—carbon and alloy	ethanol (95 %) butylcellosolve perchloric acid methanol (absolute) water butylcellosolve perchloric acid did and Glacial Acetic Acid in Var acetic acid (glacial) perchloric acid (60 %) acetic acid (glacial) perchloric acid (60 %)	700 mL 100 mL 200 mL 600 mL 13 mL 360 mL 47 mL rying Proportions) U 940 mL 60 mL 900 mL 100 mL	Use in a washdo 20 to 60 12 to 70	20 s wn/perchloric ra 1 to 5 min 0.5 to 2 min	perchloric acid carefully. Add butylcellosolve before use.
Group II II-1 II-2	Cd Mo and Mo alloys (Electrolytes Composed of Perchloric Active Cr., Ti, Zr, U, Fe, steel—carbon, alloy, stainless Zr, Ti, U, steel—carbon and alloy U, Zr, Ti, Al, steel—carbon and	ethanol (95 %) butylcellosolve perchloric acid methanol (absolute) water butylcellosolve perchloric acid cid and Glacial Acetic Acid in Val acetic acid (glacial) perchloric acid (60 %) acetic acid (60 %) acetic acid (60 %) acetic acid (glacial)	700 mL 100 mL 200 mL 600 mL 13 mL 360 mL 47 mL rying Proportions) U 940 mL 900 mL 100 mL 800 mL	Use in a washdo 20 to 60	20 s wn/perchloric ra 1 to 5 min	perchloric acid carefully. Add butylcellosolve before use.
Group II II-1 II-2 II-3	Cd Mo and Mo alloys (Electrolytes Composed of Perchloric Action Cr, Ti, Zr, U, Fe, steel—carbon, alloy, stainless Zr, Ti, U, steel—carbon and alloy U, Zr, Ti, Al, steel—carbon and alloy	ethanol (95 %) butylcellosolve perchloric acid methanol (absolute) water butylcellosolve perchloric acid dand Glacial Acetic Acid in Val acetic acid (glacial) perchloric acid (60 %) acetic acid (glacial) perchloric acid (60 %) acetic acid (glacial) perchloric acid (60 %)	700 mL 100 mL 200 mL 600 mL 13 mL 360 mL 47 mL rying Proportions) U 940 mL 60 mL 900 mL 100 mL 800 mL 200 mL	Use in a washdo 20 to 60 12 to 70 40 to 100	20 s wn/perchloric ra 1 to 5 min 0.5 to 2 min 1 to 15 min	perchloric acid carefully. Add butylcellosolve before use.
Group II II-1 II-2 II-3	Cd Mo and Mo alloys (Electrolytes Composed of Perchloric Active Cr., Ti, Zr, U, Fe, steel—carbon, alloy, stainless Zr, Ti, U, steel—carbon and alloy U, Zr, Ti, Al, steel—carbon and	ethanol (95 %) butylcellosolve perchloric acid methanol (absolute) water butylcellosolve perchloric acid cid and Glacial Acetic Acid in Val acetic acid (glacial) perchloric acid (60 %) acetic acid (60 %) acetic acid (60 %) acetic acid (glacial)	700 mL 100 mL 200 mL 600 mL 13 mL 360 mL 47 mL rying Proportions) t 940 mL 60 mL 900 mL 100 mL 800 mL 200 mL 700 mL	Use in a washdo 20 to 60 12 to 70	20 s wn/perchloric ra 1 to 5 min 0.5 to 2 min	perchloric acid carefully. Add butylcellosolve before use.
Group II II-1 II-2 II-3	Cd Mo and Mo alloys (Electrolytes Composed of Perchloric Action Cr, Ti, Zr, U, Fe, steel—carbon, alloy, stainless Zr, Ti, U, steel—carbon and alloy U, Zr, Ti, Al, steel—carbon and alloy Ni, Pb, Pb-Sb alloys	ethanol (95 %) butylcellosolve perchloric acid methanol (absolute) water butylcellosolve perchloric acid dand Glacial Acetic Acid in Var acetic acid (glacial) perchloric acid (60 %)	700 mL 100 mL 200 mL 600 mL 13 mL 360 mL 47 mL rying Proportions) U 940 mL 60 mL 900 mL 100 mL 800 mL 200 mL	Use in a washdo 20 to 60 12 to 70 40 to 100	20 s wn/perchloric ra 1 to 5 min 0.5 to 2 min 1 to 15 min	perchloric acid carefully. Add butylcellosolve before use.
Group II II-1 II-2 II-3	Cd Mo and Mo alloys (Electrolytes Composed of Perchloric Action Cr, Ti, Zr, U, Fe, steel—carbon, alloy, stainless Zr, Ti, U, steel—carbon and alloy U, Zr, Ti, Al, steel—carbon and alloy	ethanol (95 %) butylcellosolve perchloric acid methanol (absolute) water butylcellosolve perchloric acid and Glacial Acetic Acid in Val acetic acid (glacial) perchloric acid (60 %) acetic acid (glacial) perchloric acid (60 %) acetic acid (glacial) perchloric acid (60 %) acetic acid (60 %) acetic acid (glacial)	700 mL 100 mL 200 mL 600 mL 13 mL 360 mL 47 mL rying Proportions) U 940 mL 60 mL 900 mL 100 mL 800 mL 200 mL 700 mL 300 mL	Use in a washdo 20 to 60 12 to 70 40 to 100 40 to 100	wn/perchloric ra 1 to 5 min 0.5 to 2 min 1 to 15 min 1 to 5 min 1 to 5 min	perchloric acid carefully. Add butylcellosolve before use. Ited fume hood. good general-purpose electrolyte
Group III II-1 II-2 II-3 II-4 II-5	Cd Mo and Mo alloys (Electrolytes Composed of Perchloric Action Cr, Ti, Zr, U, Fe, steel—carbon, alloy, stainless Zr, Ti, U, steel—carbon and alloy U, Zr, Ti, Al, steel—carbon and alloy Ni, Pb, Pb-Sb alloys	ethanol (95 %) butylcellosolve perchloric acid methanol (absolute) water butylcellosolve perchloric acid did and Glacial Acetic Acid in Var acetic acid (glacial) perchloric acid (60 %) acetic acid (glacial)	700 mL 100 mL 200 mL 600 mL 13 mL 360 mL 47 mL rying Proportions) U 940 mL 60 mL 900 mL 100 mL 800 mL 200 mL 700 mL 300 mL 300 mL	Use in a washdo 20 to 60 12 to 70 40 to 100 40 to 100	wn/perchloric ra 1 to 5 min 0.5 to 2 min 1 to 15 min 1 to 5 min 1 to 5 min	perchloric acid carefully. Add butylcellosolve before use. Ited fume hood. good general-purpose electrolyte
Group III II-1 II-2 II-3 II-4 II-5	Cd Mo and Mo alloys (Electrolytes Composed of Perchloric AcCr, Ti, Zr, U, Fe, steel—carbon, alloy, stainless Zr, Ti, U, steel—carbon and alloy U, Zr, Ti, Al, steel—carbon and alloy Ni, Pb, Pb-Sb alloys 3 percent Si-Fe	ethanol (95 %) butylcellosolve perchloric acid methanol (absolute) water butylcellosolve perchloric acid did and Glacial Acetic Acid in Var acetic acid (glacial) perchloric acid (60 %)	700 mL 100 mL 200 mL 600 mL 13 mL 360 mL 47 mL rying Proportions) U 940 mL 900 mL 100 mL 800 mL 200 mL 700 mL 300 mL 300 mL 350 mL	Use in a washdo 20 to 60 12 to 70 40 to 100 40 to 100 	wn/perchloric ra 1 to 5 min 0.5 to 2 min 1 to 15 min 1 to 5 min 5 min	perchloric acid carefully. Add butylcellosolve before use. Ited fume hood. good general-purpose electrolyte 0.06 A/cm²
Group II II-1 II-2 II-3 II-4 II-5	Cd Mo and Mo alloys (Electrolytes Composed of Perchloric AcCr, Ti, Zr, U, Fe, steel—carbon, alloy, stainless Zr, Ti, U, steel—carbon and alloy U, Zr, Ti, Al, steel—carbon and alloy Ni, Pb, Pb-Sb alloys 3 percent Si-Fe	ethanol (95 %) butylcellosolve perchloric acid methanol (absolute) water butylcellosolve perchloric acid acetic acid (glacial) perchloric acid (60 %) acetic acid (glacial) perchloric acid (acid)	700 mL 100 mL 200 mL 600 mL 13 mL 360 mL 47 mL rying Proportions) U 940 mL 900 mL 100 mL 800 mL 200 mL 700 mL 300 mL 350 mL 350 mL	Use in a washdo 20 to 60 12 to 70 40 to 100 40 to 100 	wwn/perchloric ra 1 to 5 min 0.5 to 2 min 1 to 15 min 1 to 15 min 1 to 5 min 5 min	perchloric acid carefully. Add butylcellosolve before use. Ited fume hood. good general-purpose electrolyte 0.06 A/cm² can lower voltage to 25 V by adding
Group II II-1 II-2 II-3 II-4 II-5 II-6	Cd Mo and Mo alloys (Electrolytes Composed of Perchloric Ad Cr, Ti, Zr, U, Fe, steel—carbon, alloy, stainless Zr, Ti, U, steel—carbon and alloy U, Zr, Ti, Al, steel—carbon and alloy Ni, Pb, Pb-Sb alloys 3 percent Si-Fe Cr Hf, steel—carbon and alloy	ethanol (95 %) butylcellosolve perchloric acid methanol (absolute) water butylcellosolve perchloric acid acetic acid (glacial) perchloric acid (60 %) acetic acid (glacial) perchloric acid (glacial) perchloric acid (glacial) perchloric acid (glacial) perchloric acid acetic acid (glacial)	700 mL 100 mL 200 mL 600 mL 13 mL 360 mL 47 mL rying Proportions) L 940 mL 60 mL 900 mL 100 mL 800 mL 200 mL 700 mL 300 mL 300 mL 650 mL 1000 mL 1000 mL 1000 mL 5 mL	Use in a washdo 20 to 60 12 to 70 40 to 100 40 to 100 30–50	wn/perchloric ra 1 to 5 min 0.5 to 2 min 1 to 15 min 1 to 5 min 5 min 2–3 min	perchloric acid carefully. Add butylcellosolve before use. ted fume hood. good general-purpose electrolyte 0.06 A/cm² can lower voltage to 25 V by adding 5–15 % water.
Group III II-1 III-2 III-3 III-4 III-5 III-6 III-7 Group III	Cd Mo and Mo alloys (Electrolytes Composed of Perchloric Actor, Ti, Zr, U, Fe, steel—carbon, alloy, stainless Zr, Ti, U, steel—carbon and alloy U, Zr, Ti, Al, steel—carbon and alloy Ni, Pb, Pb-Sb alloys 3 percent Si-Fe Cr Hf, steel—carbon and alloy	ethanol (95 %) butylcellosolve perchloric acid methanol (absolute) water butylcellosolve perchloric acid did and Glacial Acetic Acid in Var acetic acid (glacial) perchloric acid (60 %) acetic acid (glacial) perchloric acid (glacial) perchloric acid acetic acid (glacial) perchloric acid	700 mL 100 mL 200 mL 600 mL 13 mL 360 mL 47 mL rying Proportions) U 940 mL 60 mL 900 mL 100 mL 800 mL 200 mL 700 mL 350 mL 1000 mL 1000 mL 5 mL 1000 mL	Use in a washdo 20 to 60 12 to 70 40 to 100 40 to 100 30–50	wn/perchloric ra 1 to 5 min 0.5 to 2 min 1 to 15 min 1 to 5 min 5 min 2–3 min	perchloric acid carefully. Add butylcellosolve before use. ted fume hood. good general-purpose electrolyte 0.06 A/cm² can lower voltage to 25 V by adding 5–15 % water.
Group II II-1 II-2 II-3 II-4 II-5 II-6 II-7 Group III	Cd Mo and Mo alloys (Electrolytes Composed of Perchloric Active Cr, Ti, Zr, U, Fe, steel—carbon, alloy, stainless Zr, Ti, U, steel—carbon and alloy U, Zr, Ti, Al, steel—carbon and alloy Ni, Pb, Pb-Sb alloys 3 percent Si-Fe Cr Hf, steel—carbon and alloy	ethanol (95 %) butylcellosolve perchloric acid methanol (absolute) water butylcellosolve perchloric acid did and Glacial Acetic Acid in Val acetic acid (glacial) perchloric acid (60 %) acetic acid (glacial) perchloric acid acetic acid (glacial) perchloric acid Acid in Water or Organic Solven phosphoric acid (85 %)	700 mL 100 mL 200 mL 600 mL 13 mL 360 mL 47 mL rying Proportions) U 940 mL 900 mL 100 mL 800 mL 200 mL 300 mL 300 mL 350 mL 1000 mL 5 mL 1000 mL 50 mL	Use in a washdo 20 to 60 12 to 70 40 to 100 40 to 100 30–50	wn/perchloric ra 1 to 5 min 0.5 to 2 min 1 to 15 min 1 to 5 min 5 min 2–3 min	perchloric acid carefully. Add butylcellosolve before use. Ited fume hood. good general-purpose electrolyte 0.06 A/cm² can lower voltage to 25 V by adding 5–15 % water. Used to polish Hf wires.
Group II II-1 II-2 II-3 II-4 II-5 II-6 II-7 Group III	Cd Mo and Mo alloys (Electrolytes Composed of Perchloric Actor, Ti, Zr, U, Fe, steel—carbon, alloy, stainless Zr, Ti, U, steel—carbon and alloy U, Zr, Ti, Al, steel—carbon and alloy Ni, Pb, Pb-Sb alloys 3 percent Si-Fe Cr Hf, steel—carbon and alloy	ethanol (95 %) butylcellosolve perchloric acid methanol (absolute) water butylcellosolve perchloric acid did and Glacial Acetic Acid in Val acetic acid (glacial) perchloric acid (60 %) acetic acid (glacial) perchloric acid (glacial) perchloric acid (glacial) perchloric acid acetic acid (glacial) perchloric acid acetic acid (glacial) perchloric acid Acid in Water or Organic Solven phosphoric acid (85 %) distilled water	700 mL 100 mL 200 mL 600 mL 13 mL 360 mL 47 mL rying Proportions) U 940 mL 900 mL 900 mL 100 mL 300 mL 650 mL 350 mL 1000 mL 5 mL 1000 mL 1000 mL 1000 mL	Use in a washdo 20 to 60 12 to 70 40 to 100 40 to 100 30–50	wn/perchloric ra 1 to 5 min 0.5 to 2 min 1 to 15 min 1 to 5 min 5 min 2–3 min	perchloric acid carefully. Add butylcellosolve before use. ted fume hood. good general-purpose electrolyte 0.06 A/cm² can lower voltage to 25 V by adding 5–15 % water.
II-1 II-2 II-3 II-4 II-5 II-6	Cd Mo and Mo alloys (Electrolytes Composed of Perchloric Active Cr, Ti, Zr, U, Fe, steel—carbon, alloy, stainless Zr, Ti, U, steel—carbon and alloy U, Zr, Ti, Al, steel—carbon and alloy Ni, Pb, Pb-Sb alloys 3 percent Si-Fe Cr Hf, steel—carbon and alloy	ethanol (95 %) butylcellosolve perchloric acid methanol (absolute) water butylcellosolve perchloric acid did and Glacial Acetic Acid in Val acetic acid (glacial) perchloric acid (60 %) acetic acid (glacial) perchloric acid acetic acid (glacial) perchloric acid Acid in Water or Organic Solven phosphoric acid (85 %)	700 mL 100 mL 200 mL 600 mL 13 mL 360 mL 47 mL rying Proportions) U 940 mL 900 mL 100 mL 800 mL 200 mL 300 mL 300 mL 350 mL 1000 mL 5 mL 1000 mL 50 mL	Use in a washdo 20 to 60 12 to 70 40 to 100 40 to 100 30–50	wn/perchloric ra 1 to 5 min 0.5 to 2 min 1 to 15 min 1 to 5 min 5 min 2–3 min	perchloric acid carefully. Add butylcellosolve before use. Ited fume hood. good general-purpose electrolyte 0.06 A/cm² can lower voltage to 25 V by adding 5–15 % water. Used to polish Hf wires.



TABLE 1 Continued

		IADEL I	Continued			
Class	Use	Formula		Cell Voltage	Time	Remarks
III-4	alpha or alpha plus beta brass, Cu-Fe, Cu-Co, Co, Cd	water phosphoric acid (85 %)	600 mL 400 mL	1 to 2	1 to 15 min	copper or stainless steel cathode
III-5	Cu, Cu-Zn	water	1000 mL	1 to 2	10 min	copper cathode
III-6	steel	pyrophosphoric acid diethylene glycol monoethyl ether	580 g 500 mL	5 to 20	5 to 15 min	49°C
		phosphoric acid (85 %)	500 mL			
III-7	Al, Ag, Mg	water ethanol (95 %)	200 mL 380 mL	25 to 30	4 to 6 min	aluminum cathode, 38 to 43°C
		phosphoric acid (85 %)	400 mL			
III-8	uranium	ethanol (absolute)	300 mL			
		glycerin (cp) phosphoric acid (85 %)	300 mL 300 mL			
I-9	Mn, Mn-Cu alloys	ethanol (95 %)	500 mL	18		
		glycerin	250 mL			
III-10	Cu and Cu-base alloys	phosphoric acid (85 %) distilled water	250 mL 500 mL		1 to 5 min	
	ou and ou bace anoye	ethanol (95 %)	250 mL	•••	1 10 0 111111	
		phosphoric acid (85 %)	250 mL			
I-11	stainless steel	ethanol (absolute), to pyrophosphoric acid	1 L 400 g	•••	10 min	good for all austenitic heat resista alloys, 38°C plus
I-12	Mg-Zn	ethanol (95 %)	400 g 625 mL	1.5 to 2.5	3 to 30 min	alloys, 36 C plus
=		phosphoric acid (85 %)	375 mL			
I-13	uranium	ethanol (95 %)	445 mL	18 to 20	5 to 15 min	0.03 A/cm ²
		ethylene glycol phosphoric acid (85 %)	275 mL 275 mL			
I-14	Al-Mg alloys	water	250 mL	50-60	2 min	
	,	ethanol (95 %)	380 mL			
1.15	Cu Dh alleus	phosphoric acid (85 %)	400 mL			good up to 20.9/ Db
I-15	Cu-Pb alloys	ethanol (95 %) phosphoric acid (85 %)	620 mL 380 mL			good up to 30 % Pb
II-16	Neptunium	ethanol (95 %)	400 mL			after P1200-grit SiC, use 6-µm
		glycerol	400 mL			diamond on nylon before
		phosphoric acid (85 %) Group IV (Electrolytes Composed of S	800 mL	later or Organic	Solvent)	electropolishing.
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 sulfuric acid	400 mL 600 mL	1.5 to 6	2 to 6 min	
V-3	stainless steel, Fe, Ni, Mo	water	750 mL	1.5 to 6	2 to 10 min	particularly good for sintered Mo-
		sulfuric acid	250 mL		Mo—	0 to 27°C
V-4	molybdenum	water ASTM E155	900 mL	1.5 to 6	0.3 to 1 min 0.3 to 2 min	particularly good for sintered Mo-
https	::/standards.iteh.ai/catalog/	16 14 /11 // 4 0 5 C 4 1	100 mL 776	-206d-2821	0.0 10 2 11111	0 to 27°C
V-5	stainless steel	water	70 mL	1.5 to 6	0.5 to 5 min	
		glycerin	200 mL			
V-6	stainless steel, aluminum	sulfuric acid water	720 mL 220 mL	1.5 to 12	1 to 20 min	
	ctallinoso ctool, alaminam	glycerin	200 mL	1.0 10 12	1 10 20 111111	
		sulfuric acid	580 mL			
V-7	molybdenum	methanol (absolute)	875 mL	6 to 18	0.5 to 1.5 mir	0 to 27°C
V-8	Ni-base superalloys	sulfuric acid methanol (absolute)	125 mL 800 mL	30	20 s	for alloy 625
		sulfuric acid	200 mL			
		Group V (Electrolytes Comp				
/ -1	stainless steel	water chromic acid	830 mL 620 g	1.5 to 9	2 to 10 min	
/-2	Zn, brass	water	830 mL	1.5 to 12	10 to 60 s	
		chromic acid	170 g			
// 4	stainless steel	Group VI (Mixed Acids or Sa phosphoric acid (85 %)				
/I-1	Stainless steel	sulfuric acid	600 mL 400 mL			
					2 min	0.3 A/cm ²
/1-2	stainless steel	water	150 mL	•••		
/1-2	stainless steel	phosphoric acid (85 %)	300 mL			
		phosphoric acid (85 %) sulfuric acid	300 mL 550 mL		2 to 10 min	0.1 to 0.2 A/om ²
	stainless steel stainless and alloy steel	phosphoric acid (85 %) sulfuric acid water	300 mL 550 mL 240 mL		2 to 10 min	0.1 to 0.2 A/cm ²
		phosphoric acid (85 %) sulfuric acid	300 mL 550 mL		2 to 10 min	0.1 to 0.2 A/cm ²
′I-3		phosphoric acid (85 %) sulfuric acid water phosphoric acid (85 %) sulfuric acid water	300 mL 550 mL 240 mL 420 mL 340 mL 330 mL		2 to 10 min 1 min	0.1 to 0.2 A/cm ² 0.05 A/cm ²
/I-3	stainless and alloy steel	phosphoric acid (85 %) sulfuric acid water phosphoric acid (85 %) sulfuric acid water phosphoric acid (85 %)	300 mL 550 mL 240 mL 420 mL 340 mL 330 mL 550 mL			
/I-3 /I-4	stainless and alloy steel	phosphoric acid (85 %) sulfuric acid water phosphoric acid (85 %) sulfuric acid water phosphoric acid (85 %) sulfuric acid	300 mL 550 mL 240 mL 420 mL 340 mL 330 mL 550 mL 120 mL		1 min	0.05 A/cm ²
VI-2 VI-3 VI-4 VI-5	stainless and alloy steel	phosphoric acid (85 %) sulfuric acid water phosphoric acid (85 %) sulfuric acid water phosphoric acid (85 %)	300 mL 550 mL 240 mL 420 mL 340 mL 330 mL 550 mL			



TABLE 1 Continued

Class	Use	Formula		Cell Voltage	Time	Remarks
VI-6	bronze (to 6 % Sn)	water	330 mL		1 to 5 min	0.1 A/cm ²
		phosphoric acid (85 %)	580 mL			
		sulfuric acid	90 mL			
VI-7	steel	water	140 mL		1 to 5 min	1 to 5 A/cm ² , 38°C plus
		glycerin	100 mL			
		phosphoric acid (85 %)	430 mL			
		sulfuric acid	330 mL			
VI-8	stainless steel	water	200 mL		5 min	1 A/cm ² , 27 to 49°C
		glycerin	590 mL			
		phosphoric acid (85 %)	100 mL			
		sulfuric acid	110 mL			
VI-9	stainless steel	water	260 mL		30 min	0.6 A/cm ² , 27 to 49°C
		chromic acid	175 g			
		phosphoric acid (85 %)	175 mL			
		sulfuric acid	580 mL			
VI-10	stainless steel	water	175 mL		60 min	0.5 A/cm ² , 27 to 49°C
		chromic acid	105 g			•
		phosphoric acid (85 %)	460 mL			
		sulfuric acid	390 mL			
VI-11	stainless and alloy steel	water	240 mL		5 to 60 min	0.5 to A/cm ² , 38 to 54°C
*	stannoso and anoy stool	chromic acid	80 g		0 10 00 111111	0.0 10 7 0111 , 00 10 0 1 0
		phosphoric acid (85 %)	650 mL			
		sulfuric acid	130 mL			
VI-12	tantalum	hydrofluoric acid	100 mL		9 min	graphite cathode, 0.1 A/cm ² , 32 to
V I- I Z	tantatum	•			3 111111	38°C
VI 40		sulfuric acid	900 mL		F!	
VI-13	stainless steel	water	210 mL		5 min	0.5 A/cm ² , 21 to 49°C
		hydrofluoric acid	180 mL			
		sulfuric acid	610 mL			2 2 2 4 4 2 2
VI-14	zinc	water	800 mL			0.002 A/cm ² , 21 to 49°C
		chromic acid	100 g			
		sulfuric acid	46 mL			
		sodium dichromate	310 g			
		acetic acid (glacial)	96 mL			
VI-15	stainless steel	hydrogen peroxide (30 %)	260 mL	0	5 min	0.5 A/cm ² (Caution) Dangerous
		(Caution)				
		hydrofluoric acid	240 mL			
		sulfuric acid	500 mL			
VI-16	stainless steel	water	520 mL	ΔΥΙΈΔΙΧ	1/2 to 4 min	0.08 to 0.3 A/cm ²
		hydrofluoric acid	80 mL			
		sulfuric acid	400 mL			
VI-17	stainless steel	water	600 mL			
		chromic acid	180 g			
		nitric acid AS IM E1558	60 mL			
			3 mL			
		sulfuric acid	240 mL			
VI-18	bismuth	glycerin	750 mL	12	1 to 5 min	$0.5 \pm A/cm^2$ (Caution) This mixture
	5.5	acetic acid (glacial)	125 mL			will decompose vigorously after a
		nitric acid	125 mL			short time. Do not try to keep.
// 10	magnasium		900 mL	E0 to 60	10 to 20 o	Bath should be stirred. Cool with
VI-19	magnesium	ethylene-glycol-monoethyl ether		50 to 60	10 to 30 s	
// 00	and the demonstration of the state of the st	hydrochloric acid	100 mL	10 1 05	00 +- 05	cracked ice below 2°C
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. Use below
		sulfuric acid	90 mL		. ,	2°C.
		Group VI (Mixed Acids or Salts in Wa				(2 11) 1 1 1 1 1 1 1
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)				
		zinc chloride (anhydrous)	250 g			
VI-22	uranium	acetic acid (glacial)	750 mL	80	5 to 30 min	The chromic acid is dissolved in the
		distilled water	210 mL			water before adding to the acetic
		chromic acid	180 g			acid. Use below 2°C.
VI-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)	9			handle. Use below 16°C.
		zinc chloride (anhydrous)	225 g			
		distilled water	160 mL			
		n-butyl alcohol	80 mL			
/1 24	ziroonium Polich and atah	•		9 to 12	1 to 10 min	(Caution) will decompose on
VI-24	zirconium. Polish and etch	glycerin (Caution)	870 mL	9 10 12	1 to 10 min	(Caution) will decompose on
	simultaneously	hydrofluoric acid	43 mL			standing, dangerous if kept too long
// 6=		nitric acid	87 mL			" 00 . "
VI-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

TABLE 1 Continued

Class	Use	Formula		Cell Voltage	Time	Remarks
VI-26	Sb	methanol (absolute)	300 mL	6–10	2-4 min	pure Sb. Use Pt cathode and anode
		sulfuric acid	50 mL			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	30 mL			
VI-28	Bi	water	200 mL			good for polarized light work
		phosphoric acid	100 mL			
		sulfuric acid	200 mL			
VI-29	Cr	water	210 mL	18		stir bath or specimen
		phosphoric acid	640 mL			·
		sulfuric acid	150 mL			
VI-30	Ge	methanol (absolute)	1000 mL			
		hydrochloric acid	10 mL			
VI-31	Nb	water	300 mL	40		polish to α-alumina before
		sulfuric acid	100 mL			electropolishing
		hydrofluoric acid	100 mL			g
VI-32	Nb	methanol (absolute)	940 mL	50-60	10 s	
		sulfuric acid	50 mL			
		hydrofluoric acid	15 mL			
VI-33	Ni-base superalloy	methanol (absolute)	170 mL	30	20 s	for Waspaloy and IN-100 mod. Etch
		hydrochloric acid	30 mL			at 5 V for 4 s.
			Alkaline Electrolytes)		at 5 7 10. 1 5.
VII-1	gold	water to	1000 mL	7.5	2 to 4 min	graphite cathode
	•	potassium cyanide	80 g			
		potassium carbonate	40 a			
		gold chloride	50 g			
VII-2	silver	water to	1000 mL	2.5	To 1 min	graphite cathode
		sodium cyanide	100 g			
		potassium ferrocyanide	100 g			
VII-3	silver	water to	1000 mL	`C.I.S	To 9 min	graphite cathode, 0.003 to 0.009
		potassium cyanide	400 g			A/cm ²
		silver cyanide	280 g			
		potassium dichromate	280 g			
VII-4	tungsten	water to	1000 mL		10 min	graphite cathode, 0.09 A/cm ² , 38 to
	3	trisodium phosphate	160 g			49°C
VII-5	tungsten, lead	water to	1000 mL	TIOXXI	8 to 10 min	graphite cathode, 0.03 to 0.06 A/cm ²
0	3 ,	sodium hydroxide	100 g			3 .p
VII-6	zinc, tin	water to	1000 mL	2 to 6	15 min	copper cathode, 0.1 to 0.2 A/cm ²
• 0	20,	potassium hydroxide	200 g	2 10 0		55pps: 54m545, 511 to 512775
VII-7	W	water A OTEN (E1	= = 01000 mL 1 /		5 min	
		sodium hydroxide	20 g			
https	s://standards_iteh_ai/catalc	Group VIII (Mixture of		Nitric Acid)	h0985c3d	h/astm-e1558-092014
VIII-1	Ni, Cu, Zn, Monel, brass,	methanol (absolute)	660 mL	40 to 70	10 to 60 s	very useful but dangerous
		nitric acid `	330 mL			

- 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: