भारतीय मानक ब्यूरो

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भारतीय मानक प्रारूप

धातु विज्ञान के नमूने की तैयारी के लिए अभ्यास संहिता
भाग 2 इलेक्ट्रोलाइटिक पॉलिशिंग
(पहला पुनरीक्षण)

Draft *Indian Standard*

CODE OF PRACTICE FOR PREPARATION OF METALLOGRAPHIC SPECIMENS
PART 2 ELECTROLYTIC POLISHING
(First Revision)

ICS 77.080.20

FOREWORD

(*formal clauses will be added later*)

Electropolishing is the preparation of smooth, scratch-free surfaces by anodic solution of a metal in an electrolytic bath. By correct adjustment of the composition of the electrolyte, temperature, voltage, current density, and time, the removal of material from a metallic surface can be controlled so as to affect mainly the protruding portions, leaving finally an approximately plane surface suitable for metallographic observation.

Some useful information regarding conditions of electropolishing and advantages and limitations of this process are given in Appendices A and B respectively.

This standard was originally published in 1975. This revision has been brought out to bring the standard in the latest style and format of the Indian Standards. In addition to this, references clause has been added.

This standard was originally published in eleven parts. The committee later decided to consolidate standards on etchants of all nonferrous materials. Consequently, Parts III, IV, VI, VII, VIII, IX, X, and XI have been withdrawn and superseded by Part III Etchants for non-ferrous materials.

The remaining parts of this standard are as follows:

Part II Electrolytic polishing

Part III Non-ferrous alloys (Superseded by Parts III, IV, VI, VII, VIII, IX, X, and XI)

Part V Ferrous alloys

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Doc No.: MTD 22 (26067)
July 2024

Draft *Indian Standard*

CODE OF PRACTICE FOR PREPARATION OF METALLOGRAPHIC SPECIMENS

PART 2 ELECTROLYTIC POLISHING

(*First Revision*)

1 SCOPE

This draft standard (Part 2) covers electrolytic polishing of metal specimens for metallographic observation.

2 REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreement based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
MTD/22/21991	Code of practice for preparation of metallographic specimens: Part 1 General features (*first revision*)

3 MECHANISM OF ELECTROLYTIC POLISHING

3.1 Recommended methods of selection, size, cutting, cleaning and mounting of metallographic specimens in IS 7739 (Part 1).

3.2 The general mechanism of electrolytic polishing of a metallographic specimen is associated with dissolution of the specimen metal during anodic polarization of the specimen in a suitable electrolyte. The solution of the specimen metal during the process gives rise to the formation of a boundary layer of film of electrolyte immediately adjacent to the specimen surface that is of different composition and of higher electrical resistance than the remainder of the electrolyte. This layer of the boundary film is thinner at the protruding ridges of the ground surface, the concentration gradient is steeper, and the electrical resistance is lower, than that part of the boundary film associated with the valleys. As a consequence, under a given potential the current density at the peaks and ridges will be greater than at the valleys; and if the applied potential is sufficiently high, metal from the ridges will go into solution at a faster rate than metal from more depressed regions. This differential in the rates of dissolution causes a gradual smoothing out, or polishing, of the ground surface.

4 PREPARATION OF THE SPECIMEN SURFACE FOR ELECTROPOLISHING

4.1 The surface of the specimen to be electro polished shall be made plane by grinding on a series of emery papers or silicon carbide papers of decreasing grit size. For most metals and alloys, grinding through No. 000 emery paper is adequate preparation. After grinding, all debris shall be washed from the surface of the specimen and a final rinse shall be given with ether or other appropriate organic solvents to ensure complete removal of oily substances that might otherwise interfere with uniform polishing.

4.2 Small specimens shall be mounted by conventional mounting procedures for ease in handling for mechanical preparation and subsequent electro-polishing. Electrical contact can be made through a small hole drilled through the back of the mount into the metal.

The possibility of violent reaction between plastics and certain electrolytes shall be taken into consideration, while mounting specimens in plastic. Mounting of specimens in dissimilar metals is not desirable because the metal in contact with the electrolyte is likely to interfere with polishing and also because fusible mounting alloys containing bismuth may be dangerously reactive in electrolytes containing oxidizing agents.

5 APPARATUS

5.1 For the electropolishing of metal specimens in appropriate electrolyte, a suitable electrolysis cell, and a controllable power supply are needed. Simple laboratory apparatus can be assembled to perform the function.

5.2 Whenever an attempt is made to polish large surface areas, the problems of obtaining sufficient current density and cooling of the specimen and electrolyte become very troublesome. An adequate mass of electrolyte should be used so that local overheating does not occur. Supplementary cooling may be required. In general, electropolishing of areas larger than 1 cm^2 is not recommended for metallographic work because of the increased difficulties encountered, but it is usually possible to polish larger areas.

5.3 Many variations are possible in the actual design of a cell for electrolysis. A simple arrangement is shown in Fig. 1. Since the current density is critical, it is advantageous to predetermine the approximate area to be polished so that the conditions for optimum results can be achieved. This variable can be controlled by the use of simple fixed masks such as are used in the commercial apparatus, or by the use of protecting masks of insulating paints or tapes.

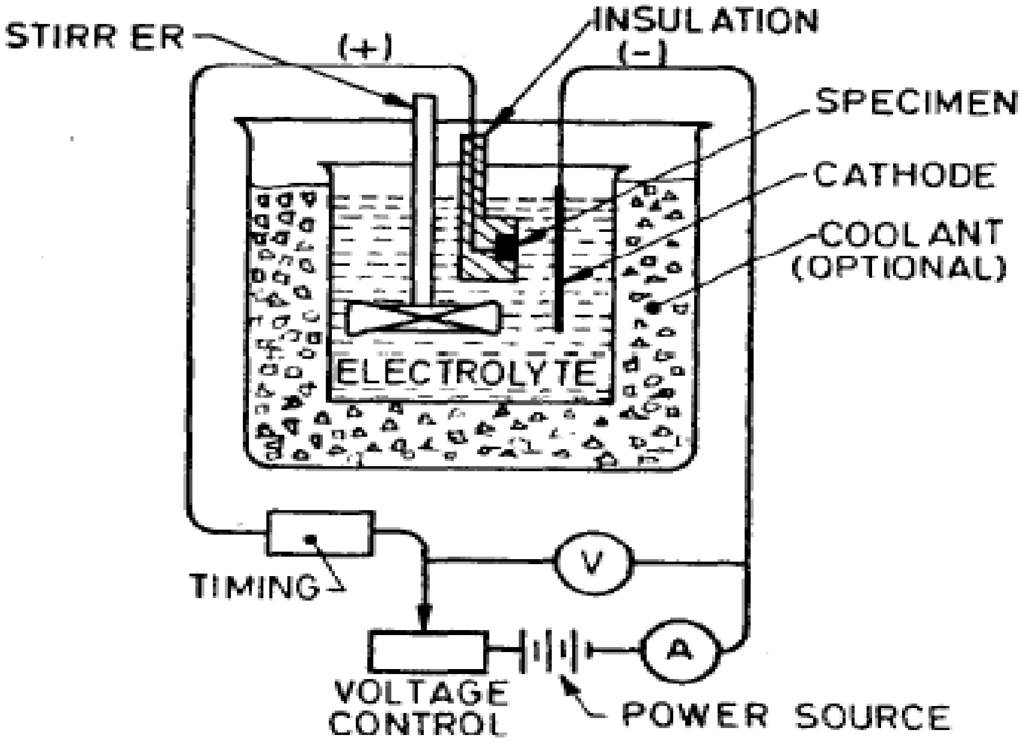
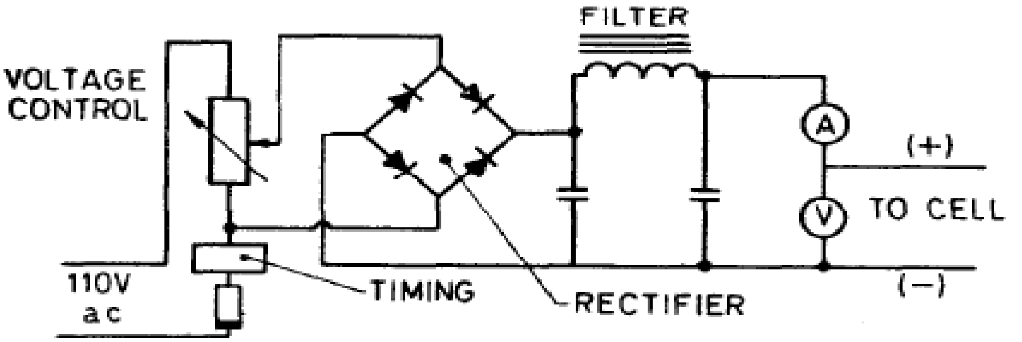
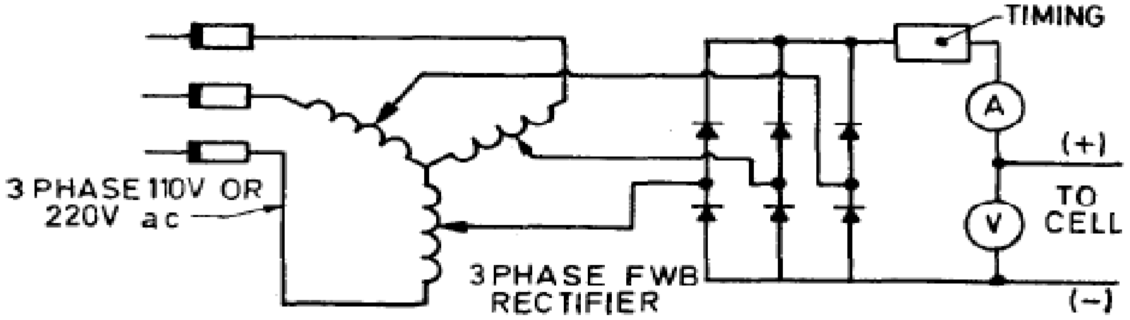


FIG. 1 SIMPLE ARRANGEMENT FOR ELECTROPOLISHING

5.4 Suitable power sources may be batteries, rectifier power supplies, or direct-current generators. Since as much as 150 V may be required for the polishing of some metals, precautions shall be taken to avoid electrical shock. The ideal power source would be a true direct-current potential with a closely controlled voltage. When ordinary line direct-current power is used, the voltage and current characteristics needed for electropolishing can be controlled only by the use of resistance. One of the best ways of obtaining the power for electropolishing is to rectify the output of a variable-voltage transformer (*see* Fig. 2). When a single-phase, full-wave, bridge- rectifier circuit is used, the output current is not satisfactory for all electro- polishing unless the ripple voltage is reduced to some small value by filtering. This is because a particular ionization level shall often be maintained within close limits in the cell. Sufficient filtering for small amounts of power is obtained with small chokes and capacitors. When heavy currents are needed, a better source of power is a three-phase, full-wave, bridge rectifier.



SINGLE PHASE FULL WAVE BRIDGE POWER SUPPLY



3 PHASE POWER SUPPLY

Three variable voltage transformers ganged (star if 220 V, delta if 110 V).

FIG.2 SCHEMATIC ARRANGEMENT FOR RECTIFYING ALTERNATING CURRENT POWER SOURCE

5.5 Some of the electrolytes are strong etchants of the metal unless the polishing current is flowing. Therefore, the provision for rapid insertion and removal of the specimen is imperative.

5.6 Another critical factor in successful polishing is the amount of agitation or flow between specimen and cathode. Flow should not be so vigorous that it interferes with the formation of a polishing film but should be sufficient to promote the formation of a uniform film. Generally there is an optimum condition which shall be experimentally determined for each case.

5.7 Additional design features that are desirable in apparatus for the electro polishing of metallographic specimens are as follows:

a) Separation of power and control portions from the polishing cell to protect them from corrosive fumes and solutions,
b) Meters for the continuous indication of voltage and current,
c) Polishing cycle controlled by an automatic timer,
d) Provision for escape of gases formed while polishing,
e) Provision for the polishing of one or more preselected small areas on a larger specimen,
f) Convenient changing of electrolytes and washing and drying of the apparatus, and
g) Parts of apparatus in contact with electrolyte adequately resistant to corrosion.

6 PROCEDURE

6.1 The specimen to be polished is made the anode in an electrolytic cell, containing a suitable electrolyte, and the polishing is carried out under the specified conditions. The formulae of electrolytes and the conditions of their use for the polishing of various metals are given in Table 1.

6.1.1 The electrolytes are intended to be used at ordinary room temperature, unless otherwise stated. A stainless steel cathode shall be used, unless otherwise stipulated.

6.1.2 A list of the most common troubles likely to be encountered in electropolishing and suggestions for solving the problems are given in Table 2.

Table 1 Electrolytes for Electropolishing

(*Clause* 6.1 *and* 6.2)

SL NO. (1)	CLASS (2)	USE (3)	FORMULA (4)		CELL VOLTAGE (5)	TIME (6)	REMARK (7)
Group 1 (Electrolytes Composed of Perchloric Acid and Alcohol with or without Organic Additions)							
i)	I-1	Al and Al alloys with less than 2 percent Si	Ethanol (95 percent)	800 ml	30 to 80		—
			Distilled water	140 ml			
			Perchloric acid (60 percent)	60 ml			
		Steel- carbon, alloys, stainless Pb, Pb-Sn, Pb-Sn-Cd, Pb-Sn-SdSb Zn, Zn-Sn-Fe, Zn-Al-Cu Mg and high Mg alloys	—		30 to 65	15 to 60 s	—
					12 to 35	15 to 60 s	
					20 to 60	—	
					—	—	Nickel cathode
ii)	I-2	Stainless steel and aluminium	Ethanol (95 percent)	800 ml	35 to 80	15 to 60 s	—
			Perchloric acid (60 percent)	20 ml			
iii)	I-3	Stainless steel	Ethanol (95 percent)	940 ml	15 to 45 s	15 to 60 s	—
			Perchloric acid (65 percent)	60 ml			

iv)	I-4	Steel, cast iron, Al and Al alloys Ni, Sn, Ag, Be, Ti, Zr, U, heat-resisting alloys	Ethanol (95 percent)	700 ml	30 to 65	15 to 60 s	One of the best formulac for universal use
			2-Butoxy ethanol	100 ml	—	—	—
			Perchloric acid (30 percent)	200 ml			
v)	I-5	Steel –stainless, alloy, high-speed ; Fe, Al, Zr, Pb	Ethanol (95 percent	700 ml	15 to 50	15 to 60 s	Universal electrolyte comparable to I-4
			Glycerine	100 ml			
			Perchloric acid (30 percent)	200 ml			
vi)	I-6	Al, Al-Si alloys	Ethanol (95 percent	760 ml	35 to 60	15 to 60 s	Particularly good with Al-Si alloys
			Diethyl ether	190 ml			
			Perchloric acid (30 percent)	50 ml			
vii)	I-7	Mo, Ti, Zr, U-Zr	Methanol (absolute)	600 ml	60 to 150	5 to 30 s	—
			2-Butoxy ethanol	370 ml			
			Perchloric acid (60 percent)	30 ml			
viii)	I-8	Al-Si alloys	Methanol (absolute)	840 ml	50 to 100	5 to 60 s	
			Glycerine	152 ml			
			Perchloric acid (65 percent)	35 ml			

ix)	I-9	Vanadium	Methanol (absolute)	590 ml	30	3 s	Three-second cycles repeated at least seven times to prevent heating
			2-Butoxy ethanol	350 ml			
			Perchloric acid (65 percent)	60 ml			
		Germanium	–	–	25 to 35	30 to 60 s	
		Titanium			38 to 66	45 s	Polishing only
		Zirconium			70 to 75	15 s	Polishing and etch simultaneously
x)	I-10	Aluminium	Methanol (absolute)	950 ml	30 to 60	15 to 60 s	–
			Nitric acid	15 ml			
			Perchloric acid (60 percent)	50 ml			
Group II (Electrolytes Composed of Perchloric acid Glacial Acetic Acid in varying proportions)							
xi)	II-1	Cr, Ti, Zr, U	Acetic acid (glacial)	940 ml	20 to 60	1 to 5 min	Good general purpose electrolyte
		Fe, steel-carbon, alloys, stainless	Perchloric acid (60 percent	60 ml	–	–	
xii)	II-2	Zr, Ti, U, steel-carbon and alloy	Acetic acid (glacial)	900 ml	12 to 70	0.5 to 2 min	–
			Perchloric acid (60 percent)	100 ml			
xiii)	II-3		Acetic acid (glacial)	800 ml	40 to 100	1 to 15 min	–

		U, Zr, Ti, Al, steel-carbon and alloy	Perchloric acid (60 percent)	200 ml			
xiv)	II-4	Ni, Pb, Pb-Sb alloys	Acetic acid (glacial)	700 ml	40 to 100	1 to 5 min	
			Perchloric acid (60 percent)	300 ml			
xv)	II-5	3 percent Si- Fe	Acetic acid (glacial)	650 ml	–	5 min	0.06 A/cm^2
			Perchloric acid (60 percent)	350 ml			
Group III (*Electrolytes Composed of Phosphoric Acid in Water or Organic Solvent*)							
xvi)	III-1	Cobalt	Phosphoric acid (85 percent)	1000 ml	1.2	3 to 5 min	–
xvii)	III-2	Pure copper	Distilled water	175 ml	1.0 to 0.6	10 to 40 min	Copper cathode
			Phosphoric acid (85 percent)	825 ml			
xviii)	III-3	Stainless, brass, Cu and Cu alloys except Sn bronze	Water	300 ml	1.5 to 1.8	5 to 15 min	Copper cathode
xix)			Phosphoric acid (85 percent)	700 ml			
xx)	III-4	Alpha pr alpha plus beta brass	Water	600 ml	1 to 2	5 to 15 min	Copper or stainless steel cathode
		Cu-Fe, Cu-Co, Cd	Phosphoric acid (85 percent)	400 ml			

xxi)	III-5	Cu, Cu-Zn	Water	1000 ml	1 to 2	10 min	Copper cathode
			Phosphoric acid (85 percent)	580 ml			
xxii)	III-6	Steel	Diethylene glycol-monoethylether	500 ml	5 to 20	5 to 15 min	49°C
			Phosphoric acid (85 percent)	500 ml			
xxiii)	III-7	Al, Ag, Mg	Water	200 ml	25 to 30	4 to 6 min	Aluminium cathode 38 to 43°C
			Ethanol (95 percent)	380 ml			
			Phosphoric acid (85 percent)	400 ml			
xxiv)	III-8	Uranium	Ethanol (absolute)	300 ml	–	–	–
			Glycerine (cp)	300 ml			
			Phosphoric acid (85 percent)	300 ml			
xxv)	III-9	Mn, Mn-Cu alloys	Ethanol (95 percent) ’	500 ml	18	–	–
			Glycerine	250 ml			
			Phosphoric acid (85 percent)	250 ml			
xxvi)	III-10	Cu and Cu-base alloys	Distilled water	250 ml	–	1 to 5 min	–
			Ethanol (95 percent)	250 ml			

			Phosphoric acid (85 percent)	250 ml			
xxvii	III-11	Stainless steel	Ethanol (absolute) Pyrophosphoric acid	 400 g	—	10 min	Good for all austenitic heat resistant alloys, 38°C plus
xxviii	III-12	Mg-Zn	Ethanol (95 percent) Phosphoric acid (85 percent)	625 ml 375 ml	1.5 to 2.5	3 to 30 min	—
xxix)	III-13	Uranium	Ethanol (95 percent) Ethylene glycol Phosphoric acid (85 percent)	445 ml 275 ml 275 ml	18 to 20	50 to 15 min	0.03 A/cm^3

Group IV (Electrolytes Composed of Sulphuric Acid in Water or Organic Solvent)

xxx)	IV-1	Stainless steel	Water Sulphuric acid	250 ml 750 ml	1.5 to 6	1 to 2 min	—
xxxi)	IV-2	Stainless steel, Fe, Ni	Water Sulphuric acid	400 ml 600 ml	1.5 to 6	2 to 6 min	—
xxxii	IV-3	Stainless steel, Fe, Ni, MO	Water Sulphuric acid	750 ml 250 ml	1.5 to 6	2 to 10 min Mo-0.3 to 1 min	Particularly good for sintered Mo, Mo-0 to 27°C
xxxiii	IV-4	Molybdenum	Water	900 ml	1.5 to 6	0.3 to 2 min	

			Sulphuric acid	100 ml			Particularly good for sintered Mo, Mo-0 to 27°C
xxxiv	IV-5	Stainless steel	Water	70 ml	1.5 to 6	0.5 to 5 min	–
			Glycerine	200 ml			
			Sulphuric acid	720 ml			
xxxv	IV-6	Stainless steel, aluminium	Water	220 ml	1.5 to 12	1 to 20 min	–
			Glycerine	200 ml			
			Sulphuric acid	580 ml			
xxxvi	IV-7	Molybdenum	Methanol (absolute)	875 ml	6 to 18	0.5 to 1.5 min	0 to 27°C
			Sulphuric acid	125 ml			

Group V (Electrolytes Composed of Chromic Acid in Water)

xxxvi	V-1	Stainless steel	Water	830 ml	1.5 to 9	2 to 10 min	–
			Chromic acid	620 g			
xxxvi	V-2	Zn, brass	Water	830 ml	1. to 12	10 to 60 s	–
			Chromic acid	170 g			

Group VI (Mixed Acids or Salts in Water or Organic Solutions)

xxxix	VI-1	Stainless steel	Phosphoric acid (85 percent)	600 ml	–	–	–
			Sulphuric acid	400 ml			
xl)	VI-2	Stainless steel	Water	150 ml	–	2 min	0.3 A/cm^3

			Phosphoric acid (85 percent) Sulphuric acid	300 ml 550 ml			
xli)	VI-3	Stainless and alloys steel	Water Phosphoric acid (85 percent) Sulphuric acid	240 ml 420 ml 340 ml	–	2 to 10 min	0.1 to 0.2 A/cm^2
xlii)	VI-4	Stainless steel	Water Phosphoric acid (85 percent) Sulphuric acid	330 ml 550 ml 120 ml	–	1 min	0.05 A/cm^2
xliii)	VI-5	Bronze (to 9 percent Sn)	Water Phosphoric acid (85 percent) Sulphuric acid	450 ml 390 ml 160 ml	–	1 to 5 min	0.1 A/cm^2
xliv)	VI-6	Bronze (to 6 percent Sn)	Water Phosphoric acid (85 percent) Sulphuric acid	330 ml 580 ml 90 ml-	–	1 to 5 min	0.1 A/cm^2
xlv)	VI-7	Steel	Water Glycerine Phosphoric acid (85 percent)	140 ml 100 ml 430 ml	–	1 to 5 min	1 to 5 A/cm^2, 38°C plus

			Sulphuric acid	330 ml			
xlvi)	VI-8	Stainless steel	Water Glycerine Phosphoric acid (85 percent) Sulphuric acid	200 ml 590 ml 100 ml 110 ml	—	5 min	1 A/cm^2, 27 to 49°C plus
xlvii)	VI-9	Stainless steel	Water Chromic acid Phosphoric acid (85 percent) Sulphuric acid	260 ml 175 ml 175 ml 580 ml	—	30 min	0.6 A/cm^2, 27 to 49°C plus
xlviii	VI-10	Stainless steel	Water Chromic acid Phosphoric acid (85 percent) Sulphuric acid	175 ml 105 g 460 ml 390 ml	—	60 min	0.5 A/cm^2, 27 to 49°C plus
xlix)	VI-11	Stainless and alloys steel	Water Chromic acid Phosphoric acid (85 percent) Sulphuric acid	240 ml 80 g 650 ml 130 ml	—	5 to 60 min	0.5 to A/cm^2, 38 to 55°C plus
l)	VI-12	Tantalum	Hydrofluoric acid Sulphuric acid	100 ml 90 ml	—	9 min	Graphite, cathode 0.1 A/cm^2, 32 to 38°C
li)	VI-13	Stainless steel	Water	210 ml	—		0.5 A/cm^2, 21 to 49°C

			Hydrofluoric acid Sulphuric acid	180 ml 610 ml			
lii)	VI-14	Zinc	Water Chromic acid Sulphuric acid Sodium dichromate Acetic acid (glacial)	800 ml 100 ml 46 ml 310 g 96 ml	—	—	0.002 A/cm^2, 21 to 38°C
liii)	VI-15	Stainless steel	Hydrogen peroxide (30 percent) (Caution) Hydrofluoric acid Sulphuric acid	260 ml 240 ml 500 ml	—	5 min	0.5 A/cm^2 *Caution*: Dangerous
liv)	VI-16	Stainless steel	Water Hydrofluoric acid Sulphuric acid	520 ml 80 ml 400 ml	—	½ to 4 min	0.08 to 0.3 A/cm^2
lv)	VI-17	Stainless steel	Water Chromic acid Nitric acid Hydrochloric acid Sulphuric acid	600 ml 180 g 60 g 3 ml 240 ml	—	—	—
lvi)	VI-18	Bismuth	Glycerine Acetic acid (glacial)	750 ml 125 ml	12	1 to 5 min	0.5 ± A/cm^2 *Caution*: This mixture will decompose

			Nitric acid	125 ml			vigorously after a short time. Do not try to keep
lvii)	VI-19	Magnesium	Ethylene glycol-monoethylether Hydrochloric acid	900 ml 100 ml	50 to 60	10 to 30 s	Bath should be stirred. Cool cracked ice below 1.5°C
lviii)	VI-20	Molybdenum, sintered and cast	Methanol (absolute) Hydrochloric acid Sulphuric acid	685 ml 225 ml 90 ml	19 to 35	20 to 35 s	Mix slowly. Heat is developed. Avoid contamination with water below 1.5°C
lix)	VI-21	Titanium	Ethanol (95 percent) N-butyl alcohol Aluminium chloride (anhydrous) (add very slowly) (Caution)	900 ml 100 ml 60 g	30 to 60	1 to 6 min	Caution : Anhydrous aluminium chloride is extremely dangerous to handle
lx)	VI-22	Uranium	Zinc chloride (anhydrous)	250 g	80	5 to 30 min	The chromic acid is dissolved in the water

			Acetic acid (glacial)	750 ml			before adding to the acetic acid below 1.5°C
			Distilled water	210 ml			
			Chromic acid	180 g			
lxi)	VI-23	Pure zinc	Ethanol (95 percent)	720 ml	25 to 40	0.5 to 3 min	Caution : Anhydrous aluminium chloride is extremely dangerous to handle below 15°C
			Aluminium chloride (anhydrous) (Caution)	50 g			
			Zinc chloride (anhydrous)	225 g			
			Distilled water	160 ml			
			N-butyl alcohol	80 ml			
lxii)	VI-24	Zirconium Polish and etch simultaneously	Glycerine (Caution)	870ml	9 to 12	1 to 10 min	Caution : Will decompose on standing, dangerous if kept too long
			Hydrofluoric acid	43 ml			
			Nitric acid	87 ml			
lxiii)	VI-25	Bismuth	Saturated solution of KI in distilled water	980 ml	7	30 s	Polish 30 s but allow to remain in electrolyte until brown film is dissolved
			Hydrochloric acid	20 ml			

Group VII (Alkaline Electrolytes)

lxiv)	VII-1	Gold	Water	1000 ml	7.5	2 to 4 min	Graphic cathode

			Potassium cyanide Potassium carbonate Gold chloride	80 g 40 g 50 g			
lxv)	VII-2	Silver	Water Sodium cyanide Potassium Ferro cyanide	1000 ml 100 g 100 g	2.5	To 1 min	Graphic cathode
lxvi)	VII-3	Silver	Water Potassium cyanide Silver cyanide Potassium dichromate	1000 ml 400 g 280 g 280 g	–	To 9 min	Graphic cathode, 0.003 to 0.009 A/cm^2.
lxvii)	VII-4	Tungsten	Water Trisodium phosphate	1000 ml 160 g	–	10 min	Graphic cathode, 0.09 A/cm^2, 3 to 49°C
lxviii	VII-5	Tungsten, Lead	Water Sodium hydroxide	1000 ml 100 g	–	8 to 10 min	Graphic cathode, 0.03 to 0.06 A/cm^2
lxix)	VII-6	Zinc, tin	Water Potassium hydroxide	1000 ml 200 g	2 to 6	15 min	Copper cathode, 0.003 to 0.009 A/cm^2.

Group VIII (Mixture of Methyl Alcohol and Nitric Acid

lxx)	VIII-l	Ni, Cu, Zn, Monel, Brass, Nichrome, Stainless steel	Methanol (absolute) Nitric acid	660 ml 330 ml	40 to 70	10 to 60 s	Very widely useful but dangerous

Table 2 Electopolishing Procedural Problems and Corrections
(*Clause* 6.1.2)

Sl No.	Trouble	Possible Cause	Suggested Correction
(1)	(2)	(3)	(4)
i)	Centre of specimen deeply etched	No polishing film at centre of specimen	1) Increase voltage 2) Decrease agitation 3) Use more viscous electrolyte
ii)	Pitting or etching at edges of specimen	Too viscous thick film	1) Decrease voltage 2) Increase agitation 3) Use less viscous electrolyte
iii)	Sludge setting on surface	Insoluble anode product	1) Try new electrolyte 2) Increase temperature 3) Increase voltage
iv)	Roughness or matt surface	Insufficient or no polishing film	1) Increase voltage 2) Use more viscous electrolyte
v)	Waviness or streak on polishing surface	1) Insufficient time 2) Incorrect agitation 3) Inadequate preparation 4) Too much time	1) Increase or decrease agitation 2) Better preparation 3) Increase voltage and decrease time
vi)	Satins on polishing surface	Attack after polishing current is off	1) Remove specimen while current is still on 2) Try less corrosive electrolyte
vii)	Unpolished spots (bulls eyes)	Gas bubbles	1) Increase agitation 2) Decrease voltage
viii)	Phases in relief	Insufficient polishing film	1) Increase voltage 2) Better preparation 3) Decrease time
ix)	Pitting	1) Too long polishing 2) Too high voltage	1) Better preparation 2) Decrease voltage 3) Decrease time 4) Try different electrolyte

6.2 Safety Precautions — Most of the electrolytes, specified in Table 1, can be exceedingly dangerous if carelessly handled. The instructions given in **6.2.1** to **6.2.4.8** should be read before any electrolyte is mixed or used.

6.2.1 Mixtures of perchloric acid and acetic anhydride are very dangerous to mix and even more unpredictable to use. Many industrial firms and research laboratories forbid the use of such mixtures. Some cities also have ordinances prohibiting the use of such potentially explosive mixtures. These facts are considered sufficient reason for recommending against their use.

6.2.2 It should be borne in mind that mixture of oxidizable organic com pounds 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 efficient polishing or catalyse the decomposition of the electrolyte. The electrolyte must be discarded and flushed down a chemical waste with adequate water before this occurs.

6.2.3 Electrolytes, with few exceptions, should be mixed and stored in clean glass containers and never in contact with foreign or organic materials. They shall never be put aside and allowed to become concentrated by evaporation. Unless there is definite reason to do otherwise, all electrolytes should be discarded as soon as they have passed their immediate usefulness.

6.2.4 The electrolytes recommended for use are classified in.to eight groups. Their chemical components are listed in the order of mixing. Although this is contrary to common practice, it is done to prevent possibly dangerous mistakes. Unless other instructions are given, the electrolytes are intended to be used in the ordinary room temperature range from 18 to about 32°C. The use of a stainless steel cathode is also presumed unless otherwise stipulated.

6.2.4.1 *Group I (electrolytes composed of perchloric acid and alcohol with or without organic additions)* — These electrolytes are believed to be safe to mix and use provided the following safety precautions are strictly observed:

a) The baths are only compounded in small quantities and stored glass stoppered bottle of such size that the bottles are completely filled by the electrolytes.
b) Any evaporated solvents are promptly replaced by keeping the bottle filled.
c) Spent or exhausted baths are promptly discarded.
d) No departure is ever allowed from the prescribed formula, the method of mixing it, or the strength of the acid used.
e) The electrolytes are always protected from heat or fire.

6.2.4.2 *Group II (electrolytes composed of perchloric acid glacial acetic acid in varying proportions*) — Very little heat is developed when perchloric acid is mixed with glacial acetic acid. In mixing, the perchloric acid should be added to the acetic acid with stirring. These mixtures are supposed to be perfectly safe to mix and use but it is, nonetheless, felt that great care should be exercised with them. Temperatures shall never be allowed to exceed 30°C. They are flammable and shall be guarded against fire or the evaporation of the acetic acid to such mixtures.

6.2.4.3 *Group III* (*electrolytes composed of phosphoric acid in water or organic solvent*) —These mixtures are generally quite easy to prepare. In mixing they are handled exactly as any mineral acid, namely, the acid shall be slowly poured into the water or solvent with constant stirring to prevent the formation of a heavy layer of acid at the bottom of the vessel. Some solid phosphoric acids are quite energetic in their combination with water.

6.2.4.4 *Group IV (electrolytes composed of sulphuric acid in water or organic solvent*) — The dilution of sulphuric acid with water is somewhat difficult due to the extremely exothermic reaction. The acid must always be poured into the water slowly and with constant stirring. Great care should be taken to prevent spattering. Even dilute solutions of sulphuric acid strongly attack the skin or clothing. Such solutions are also very hygroscopic. They vigorously attack most plastic. The mixtures of sulphuric acid with other in organic acids arc generally more useful as electrolytes.

6.2.4.5 *Group V (electrolytes composed of chormic acid in water)* — The addition of crystalline chromic acid to water is simple since very little heat is developed. Chromic acid is powerful oxidant and under certain condition it will liberate considerable quantities of free oxygen. It is generally dangerous and possibly incendiary in the presence of oxidizable materials. It cannot be safely mixed with most organic liquids. It can generally be mixed with saturated organic acids. It can generally be Chromic acid solutions cannot be used in contact with plastic parts without their eventual destruction. Care should be taken to prevent its contact with the skin since repeated exposure to even dilute solutions of chromic acid or the chromates will cause persistent and painful ulcers that are difficult to heal.

6.2.4.*6 Group VI (mixed acids or salts in water or organic solutions*) — These mixtures are safe to mix and use provided the mixing is properly done. It must be remembered that in all cases the acid is added to the solvent slowly and with constant stirring. If sulphuric acid is in the formula, it is added last and with particular care.

If hydrofluoric acid or fluorides are part of a formula, polyethylene or other similar hydrofluoric acid-resistant vessels should be used. Particular care should be taken to avoid skin contact with acid fluorides since exposure 10 them, which may pass unnoticed at the time, may result in serious burns later. In those electrolytes containing anhydrous aluminium chloride extreme care must be exercised. The reaction between this compound and water is almost explosive. Chromic acid cannot b: safely mixed with most organic liquids but can be mixed with saturated organic acids. Care should be taken to prevent contact with the skin.

6.8.4.7 *Group VII (alkaline electrolytes)* — These mixtures can b-grouped in two general categories, those containing cyanide, and those not containing cyanide.

Caution — The use of cyanide by anyone not properly trained and familiar with it is extremely dangerous. Cyanides are among the quickest acting and most potent poisons likely to be encountered in the laboratory. Cyanide is so quick acting and deadly that the administration of an antidote is usually ineffectual. Extreme care must be taken that 110 droplet of the solution or crystal of the salt is ever left around where it can be accidentally picked up and carried to the mouth. Should any spillage occur, as much as possible should be mopped up with a sponge and water. The remainder can then be instantly destroyed by washing the area with very dilute nitric acid.

Solutions of the alkali hydroxides are very useful for the polishing of certain amphoteric metals. Their attack on the skin is drastic, so great, care should be exercised in their use.

6.2.4.8 *Group VIII (mixture of methyl alcohol and nitric acid)* — Nitric acid can be mixed with methanol with apparent safety. This is done by adding the acid to the alcohol with careful stirring. It cannot safely be mixed with any higher alcohol except in very dilute solutions. If pure chemicals are used, the mixture of nitric acid and methanol is quite stable provided it is never heated or confined in any way. Do not store in a closed container. Under certain conditions, extremely unstable or explosive nitro compounds, azides or fulminates can be formed. The spontaneous decomposition of the mixture can also be catalyzed by impurities or heat. It should always be discarded as soon as it has served its immediate purpose. For certain cases this is an extremely useful electrolyte. Due to its dangerous nature, it should not be employed if its use can be avoided.

APPENDIX A

(*foreword*)

RECOMMENDATIONS FOR DETERMINING THE CONDITIONS OF ELECTROPOLISHING

A-1 Very frequently the metallographer is asked to electropolish some metal or alloy which he has not previously handled. There are several general principles which can be applied advantageously in these cases. The problem should be viewed in comparison with known procedures and information gained through previous experience. It is generally helpful to compare the position of the major component with elements of the same general group in the periodic arrangement of the elements, and to study the phase diagram, if available, to predict the number of phases and their characteristics. Single-phase alloys are generally easy to electropolish whereas multiphase alloys are likely to be difficult or impossible to polish. Minor alloying additions to the metal may profoundly affect its response to polishing in a given electrolyte.

A-2 The possibility of polishing a metal and the conditions for polishing a metal in a given electrolyte can sometimes be ascertained by plotting current versus voltage curves. The resulting curves will approximate one of the two forms shown in Fig. 3. Curve I is typical of electrolytes that polish over a very wide range or will not polish at all. Curve II is characteristic of electrolytes that form an ionic film. The dotted portion of the curve is added in recognition of certain published data and the observation that the formation of a polishing film requires finite time. Polishing will occur between B and C is usually best at or slightly beyond C.

A-3 After the polishing range is determined, the other constants, such as preparation, agitation and time can be determined experimentally. The amount of preparation depends on the nature of the specimen and on the results desired. Often this can be accomplished through the use of No. 0 emery paper on a powder grinder. In other cases it is necessary or desirable to have a good mechanical polish before starting to electropolish. The surface to be polished, should be clean in all respects to obtain uniform attack.

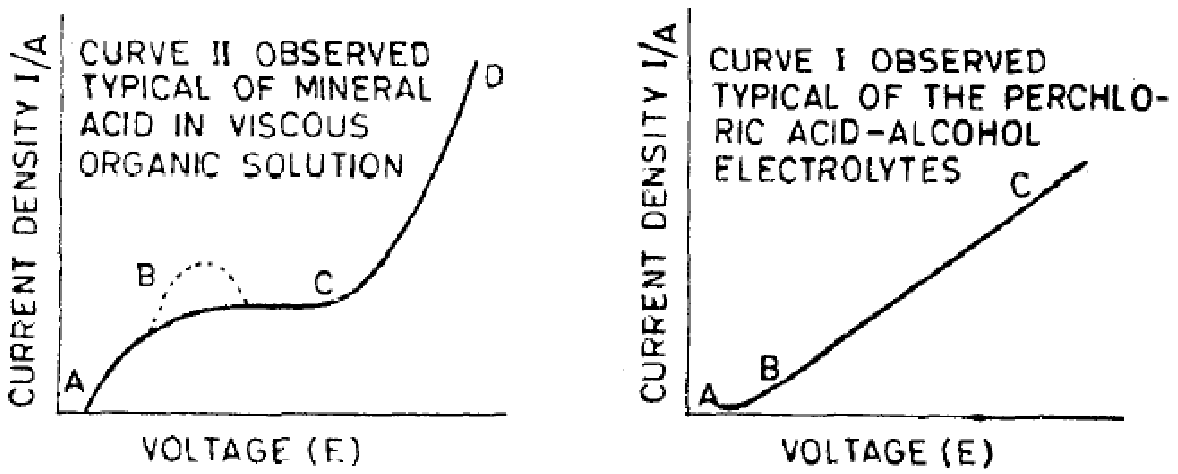


FIG. 3 TYPICAL CURVES FOR DETERMINING CONDITIONS FOR ELECTROPOLISHING

A-4 To aid in selecting or developing an electrolyte for a new metal or alloy or to achieve-a specific result, a few characteristics that may be desirable in an electrolyte are listed below:

a) The electrolyte should generally be somewhat viscous.
b) The electrolyte shall be a good solvent of the anode under electrolysis conditions. Insoluble products that form adherent deposits on the fact of the specimen may prevent polishing.
c) It is desirable that the electrolyte does not attack the metal when-no current is flowing. This condition is not always achieved.
d) The electrolyte usually should contain one or more ions of large radii, such as $(IO_4)^{-3}$, $(ClO_4)^{-1}$ or $(SO_4)^{-2}$ and sometimes large organic molecules.
e) The electrolyte should be simple to mix, stable, and safe to handle.
f) It is desirable that the electrolyte function at room temperature and not be sensitive to temperature changes, since it is difficult to control and maintain precise temperatures in an electrolyte during polishing.

APPENDIX B

(*foreword*)

ADVANTAGES AND DISADVANTAGES OF ELECTROPOLISMNG

B-1 ADVANTAGES

B-1.1 When properly applied, electropolishing can be an extremely useful tool for the metallographer. The principal advantages of the method are as follows:

a) For some metals, a high quality, finished surface can be produced that is equivalent to the best that can be obtained by mechanical methods.
b) Once a procedure has been established, good results can be obtained by even a new operator without the long training period so necessary to develop the physical skills for mechanical polishing.
c) There can be a marked saving of time if many specimens of the same material are to be polished sequentially.
d) The method is especially suited to the softer metals which may be difficult to polish by mechanical methods.
e) No scratches are produced in polishing.
f) Artifacts, such as disturbed metal or mechanical twins, produced on the surface even by careful grinding and polishing operations, should generally be removed. Thus, electropolishing may also be used to determine the true microstructure of a specimen as guide for mechanical preparation.
g) Surfaces resulting from electropolishing are completely unworked by the polishing procedures. This feature is important in low-load hardness testing or X-ray studies.
h) In some cases etching can be accomplished by simply reducing the voltage to approximately one-tenth the potential required for polishing and continuing electrolysis for a few seconds.
j) Electropolishing is frequently useful in electron metallography since the higher resolution puts a premium on clean, undistorted metal surfaces. It 1s sometimes convenient to electropolish so as to leave a residual etch of suitable depth. The very clean, slightly undercut surface may cause difficulties in replica stripping.

B-2 DISADVANTAGES

B-2.1 Metallographic preparation by electropolishing is subject to a number of limitations which should be recognized to prevent misapplication of the 21 method and disappointment in the results. The principal disadvantages include the following:

a) Many of the chemical mixtures used in electropolishing are poisonous or dangerous if not properly handled. These hazards are similar to those involved in the mixing and handling of etchants.
b) The conditions and electrolytes required to obtain a satisfactorily polished surface are different for different alloys, hence considerable time may be required to develop a procedure for a new alloy, if it can be developed at all. This limitation does not apply if appropriate procedures exist.

c) In multiphase alloys the rate of polishing of different phases may not be the same; at times this phenomenon may be an advantage.
d) A large number of electrolytes may be needed to polish the variety of metals encountered by a given laboratory.
e) Plastic or metal mounting materials may react with the electrolyte
f) Electropolished surfaces exhibit an undulating rather than a plane surface and may not in some cases be suited for examination at all I magnifications.
g) Edge effects limit applications involving small samples, surface phenomena, coatings, interfaces, and cracks.
h) Attack around non-metallic particles and adjacent metal, voids and various inhomogeneities may not be the same as that of the matrix, exaggerating the size of the voids and inclusions.
j) The electropolished surfaces of certain materials may be passive and difficult to etch.
k) Artifacts may occasionally be produced by electropolishing.