



Standard Practice for Preparation of Copper and Copper-Base Alloys for Electroplating and Conversion Coatings¹

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This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This practice is intended to serve as a guide for the proper preparation of copper and its alloys for electroplating and conversion coating. This practice is also suitable for use before autocatalytic plating. Only alloys containing at least 50 mass % copper are considered within the scope of this practice.

1.2 The wide variety of methods of mechanical finishing are not considered strictly as preparation for electroplating or conversion coating and consequently are described only briefly.

1.3 Details of electroplating and subsequent treatments for applying conversion coatings are not within the scope of this practice.

1.4 *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.* For a specific hazard statement, see 6.5.2.

2. Referenced Documents

2.1 *ASTM Standards:*²

D322 Test Method for Gasoline Diluent in Used Gasoline Engine Oils by Distillation

3. Significance and Use

3.1 The proper preparation of copper and copper alloy surfaces for electroplating, conversion coating, or autocatalytic plating is often critical to the performance of the coatings.

¹ This practice is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.02 on Pre Treatment.

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² 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.

3.2 This practice outlines procedures required to produce satisfactory coatings on surfaces of copper and copper alloy surfaces.

4. Process Chemicals

4.1 All process chemicals are of technical grade or better. Acid solutions are prepared from grade chemicals as listed in Appendix X1.

4.2 *Purity of Water*—High quality water is not normally required to make up and maintain the solutions utilized in this practice. If reused or recycled water from waste treatment processes or from other in-plant sources is to be used, it should be relatively free of chromium salts, oil, wetting agents, or insoluble materials. Excessively hard water can decrease the life and performance of many cleaning solutions and make parts more difficult to rinse completely.

5. General Considerations

5.1 *Removal of Oxides*—Oxides can be removed from as fabricated, annealed, or heat-treated alloys by abrasive methods such as tumbling, burnishing, and emery set-up wheel polishing and by chemical methods, such as deoxidizing solutions, bright dips, and cyanide dips. The choice of method is dependent on the resultant surface finish required, amount of oxide to be removed, and the end-use properties of the article finished.

5.2 *Castings and Forgings*—Castings and forgings requiring abrasive methods to produce a desired surface finish do not necessarily need pickling or bright dipping. If pickled, bright dipped, or deoxidized, however, castings and other porous parts should be thoroughly rinsed between operations to avoid or minimize staining or stain spots. Castings or forgings processed in solutions containing wetting agents, which are in many proprietary products or which may be added by the individual, usually require greater care in rinsing.

5.3 *Stampings and Drawn Products*—Stampings and drawn work follow the same rule as castings and forgings except, for economy considerations, it may be advisable to pickle or deoxidize before abrasive finishing if heavy oxides are present.

5.4 *Cold-Headed and Progressive Die Products*—Cold-headed products and progressive die products often require relief annealing to avoid subsequent season cracking.

5.5 *Screw Machine Products*—Screw machine products may be readily electroplated with only mild cleaning and acid dipping as they are produced from the machines. Abrasive methods may be applied as appropriate before cleaning and acid dipping.

6. Preparation for Electroplating and Conversion Coating

6.1 Outlines of Typical Preparatory Cycles:

- 6.1.1 Vapor degrease or alkaline clean or emulsion soak clean, or tumble clean,
- 6.1.2 Rinse,
- 6.1.3 Alkaline electroclean,
- 6.1.4 Rinse,
- 6.1.5 Acid dip,
- 6.1.6 Rinse, and
- 6.1.7 Electroplate or conversion coat in an acid solution.

NOTE 1—A bright dip, electropolish, or deoxidization may be added after step 6.1.4 or 6.1.2.4 followed by two agitated and running rinses prior to step 6.1.5.

NOTE 2—If chromium compounds are used in the bright dip or alternatives in Note 1, additional steps will be required to ensure complete chromium removal from surfaces before any plating process.

NOTE 3—Additional information on procedures for cleaning of copper or copper alloys prior to electroplating may be found in Practice D322.

6.2 *Precleaning*—Solvent or solvent-alkali emulsion-soak cleaners can be used if the parts being electroplated can be rinsed easily and completely; otherwise, mild alkaline cleaners and vapor degreasing should be used.

6.3 *Electrocleaning*—To produce the chemically clean surface required for electroplating or subsequent coatings, an electrolytic cleaner may be used with the part as the anode or cathode. Anodic cleaning, particularly of brass, may cause slight tarnishing or etching if applied for a prolonged time (more than a few seconds) or at too high or too low an operating temperature. A contaminated cathodic cleaner may form a smut film on the work. The voltage at the source is usually 6 to 8 V. Separate solutions should be used for anodic and cathodic cleaning. When a part is properly cleaned, it will show a continuous liquid film upon removal from rinses after acid dip solutions.

NOTE 4—Proprietary cleaners which are used in accordance with the supplier's recommendations are preferred.

NOTE 5—The formula of a typical electrolytic cleaner composition is given in Appendix X1.

6.4 *Tumble Cleaning*—Tumble cleaning is an alternative process that can be used as a substitute for precleaning or electrocleaning depending on the parts being processed. A typical tumble cleaner is listed in Appendix X1. Proprietary cleaners are available and are generally considered preferable.

6.5 *Tarnish and Stain Removal, Deoxidizing, and Neutralizing:*

6.5.1 *Acid Dipping*—After the work has been thoroughly cleaned and rinsed, it must be acidified to neutralize any residual alkali before it enters an electroplating bath. Thus,

before nickel, copper, tin, chromium, and similar plating solutions, an acid dip is used. The most common dips used are sulfuric acid, 50 mL/L to 100 mL/L by volume, or hydrochloric acid, 100 mL/L to 200 mL/L by volume. Fluoboric acid, 50 mL/L to 100 mL/L by volume, can be used before fluoborate electroplating solutions. These solutions are maintained at room temperature. Leaded copper alloys form insoluble salts in either sulfuric or hydrochloric acid, and therefore should be pickled in fluoboric acid, 20 mL/L to 50 mL/L by volume, or nitric acid, 100 mL/L to 200 mL/L by volume.

6.5.2 *Cyanide Dipping*—After the work has been cleaned, acid pickled, or acid dipped, and thoroughly rinsed, it is sometimes immersed in a 15 to 45 g/L sodium cyanide solution to remove slight tarnish. This step is more common if the first electroplating solution also contains cyanide. (**Warning**—One should provide adequate rinsing before and after any cyanide-containing solution before going into an acid solution.)

NOTE 6—When processing parts containing lead as an alloy constituent, such as free-machining brass, care should be taken not to allow lead to accumulate in a cyanide solution beyond 50 mg/L as Pb.

6.5.3 *Deoxidizing and Bright Dipping :*

6.5.3.1 Deoxidizing is usually performed to activate the surface of the part by the removal of the oxide coating. Strong oxidizing solutions such as mixtures of sulfuric acid and hydrogen peroxide are used as pickling agents. Most of these compositions are proprietary.

6.5.3.2 Bright dipping is primarily used to improve the surface luster of the work. It also serves as a deoxidizing solution. While proprietary processes are available one non-proprietary composition which can produce good surface luster (not mirror brightness) is included in Appendix X1. The bright dipped parts should be rinsed thoroughly by immersion in several water rinses with constant agitation. A mild alkaline solution may be employed to neutralize residual acids before immersion in any cyanide-containing solution. An excess of hydrochloric acid in the bright dip composition must be avoided; otherwise, a dull finish will result.

7. Striking

7.1 *Copper Strike*—In order to prevent peeling, a copper strike is used before silver or nickel electroplating of leaded copper alloys and work that has been soft soldered. A nickel strike (see Appendix X2) may be used in addition to the copper strike before silver electroplating. A standard cyanide copper strike may be employed (see Note 6). All soldered surfaces and the basis metal must be completely covered with copper. This requires up to 1 min at 3 to 6 V.

7.2 *Silver Strike*—It is always necessary to apply a silver strike to the work before it enters a silver electroplating solution. The power source should be on and the electrical circuit connection made (for “live” entry) before immersing the work in either the silver strike or silver electroplating solution.

7.3 *Nickel Strike*—In order to obtain adhesion on alloys containing nickel, or chromium and iron, or both, one of the nickel strike solutions described in Appendix X1 is used. Additional thicknesses of nickel for the purpose of diffusion control, etc., may be applied.

7.4 *Gold Strike*—It is always necessary to apply a gold strike to the work before it enters a good electroplating solution. A standard soft gold strike may be employed.

8. Keywords

8.1 activation; cleaning; copper; deoxidation; preparation; striking

APPENDIXES

(Nonmandatory Information)

XI. CLEANING AND PICKLING SOLUTIONS

X1.1 *Process Chemicals*—All process chemicals are of technical grade or better. Acid solutions are prepared from the following technical grade chemicals:

Glycolic acid (CH ₂ OH COOH)	67 mass %, density 1.3 g/mL
Sulfuric acid (H ₂ SO ₄)	93 mass %, density 1.83 g/mL
Hydrochloric acid (HCl)	31 mass %, density 1.16 g/mL
Nitric acid (HNO ₃)	67 mass %, density 1.40 g/mL
Phosphoric acid (H ³ PO ₄)	75 mass %, density 1.5 g/mL
Fluoboric acid (HBF ₄)	48 mass %, density 1.38 g/mL

X1.2 *Tumble Cleaning*—A typical tumble cleaner is as follows:

	Mass %
Mild soap chips or powder	12.5
Trisodium phosphate (Na ₃ PO ₄ ·12H ₂ O)	12.5
Sodium carbonate (Na ₂ CO ₃)	75

This mixture is used in a solution concentration of from 30 to 45 g/L at 80 to 90°C.

X1.3 *Electrocleaning*—A typical formula is as follows:

	Mass %
Sodium carbonate (Na ₂ CO ₃)	40 to 50
Trisodium phosphate (Na ₃ PO ₄ ·12H ₂ O)	25 to 50
Sodium hydroxide (NaOH)	10 to 25
Surface active agent (Low foam or No Foam Type)	1
Total of individual ingredients adjusted to give 100 %	

This mixture is used in a solution concentration of 30 to 45 g/L.

Temperature	60 to 71°C
Current density	1 to 3 A/dm ²
Time	1 to 3 min cathodic and 5 to 10 s anodic

X1.4 Acid Pickles:

X1.4.1 *“Fire Off” or Scale Removal Dip*

This dip is used to remove heavy oxide layers on small parts that can be handled in bulk. A typical example is the following mixture:

Sulfuric acid	(50 mL/L) to (300 mL/L)
Nitric acid	(150 mL/L) to (600 mL/L)
Water	remainder
Temperature	18 to 20°C

NOTE X1.1—Lead sulfate may form on the small globules of lead present in leaded brass and can cause porosity and blistering of the subsequent electroplate. Diluted nitric acid (without sulfuric acid) has been used successfully, as has fluoboric acid, in scale removal.

X1.4.2 For copper and copper-base alloys (except beryllium copper) to remove oxides, sulfuric acid (100 mL/L to 400 mL/L) by volume is used. These solutions may be used from

room temperature to 80°C. Time required may have to be 10 min or longer. In some instances the smuts developed in beryllium and tellurium copper can be easily and completely removed by a short immersion in 30 to 35 % by vol (22°Be) HCl at ambient temperature.

X1.4.3 To remove oxides from beryllium copper alloys, proprietary sulfuric acid/hydrogen peroxide mixtures have been found to be preferable to the conventional bright dip solutions described in X1.4.1 and X1.4.5. Reduced porosity of the electroplated coating has been found when these proprietary deoxidizing solutions are employed instead of bright dips.

X1.4.4 When heat treated, beryllium copper may form a beryllium oxide film. -min dip in a solution of the following composition can be used to ensure complete removal of this film:

Sodium hydroxide	500 g/L
Water	To balance
Temperature	130°C

NOTE X1.2—This procedure is normally used by the producers of beryllium copper alloys.

X1.4.5 *Nitric-Phosphoric Acid Pickle*

This pickle is also used to remove oxides and scale. The parts should be clean and dry before being placed in the solution.

Nitric acid	250 mL/L
Phosphoric acid	750 mL/L

X1.5 *Electropolishing*—Electropolishing is used to impart a bright smooth finish to the part. It is accomplished by making the work anodic at 3 to 6 A/dm² in a solution as follows:

Glycolic acid	350 mL/L
Phosphoric acid	550 mL/L
Sulfuric acid	190 mL/L
Water	Balance

X1.6 *Bright Dip Solution*—A formula for a bright dip is given as follows:

Sulfuric acid	600 mL/L to 750 mL/L
Nitric acid	200 mL/L to 350 mL/L
Hydrochloric acid	1 mL/L
Water	To 1 L

Agitate the work constantly while immersed for from 5 to 105 s. A mild alkaline solution can be used after rinsing the bright dip solution from the part to ensure neutralization if a cyanide plating solution follows.

X2. NICKEL STRIKE SOLUTIONS

X2.1 Woods Nickel Strike—A low pH nickel strike of the following composition may be substituted for a copper strike, especially prior to silver electroplating:

Nickel chloride (NiCl ₂ ·6H ₂ O)	240 g/L
Hydrochloric acid	120 g/L
Water	balance
Cathodic current density	5.4 to 10.8 A/dm ²
Temperature	18 to 25°C
Time	2 min

X2.2 Low pH Sulfamate Nickel Strike—This solution has been useful when plating on assemblies of mixed alloy components where copper or copper alloys have been joined to high-nickel steels or other passive metals.

Nickel Sulfamate (Ni (SO ₃ NH ₂) ₂)	320 g/L
Nickel as metal	76 g/L
Boric acid (H ₃ BO ₃)	30 g/L
Water	balance
pH (lowered with sulfamic acid)	1.5
Cathode current density	2–10 A/dm ²
Time	2 min
Temperature	18 to 25°C

X2.3 Glycolate Nickel Strike—Glycolate nickel strike is used on complex parts to increase uniformity and coverage in low current density areas.

Nickel Acetate (Ni(CH ₃ COO) ₂)·4H ₂ O	65 gm/L
Boric Acid	45 gm/L
Hydroxyacetic Acid (Glycolic Acid)	60 mL/L
Sacharin (stress reducer)	1.5 gm/L
pH	6.2
Temperature	Ambient
Current Density	2.7 A/dm ²
Anodes	Nickel (inert)
Time	5 min

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