

NATIONAL RADIO ASTRONOMY OBSERVATORY  
Charlottesville, Virginia

## NRAO CHEMICAL LAB REPORT NO. 12

TITLE: Chemical Lab Procedures, 1997

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DATE: January 6, 1997

### I. METAL CLEANING AND PREPARATION

Oils, greases, abrasives, oxide films, metal chips and the like need to be removed before a piece can be electroplated. For most work we ultrasound clean using an alkaline soap mixture, ~ pH 11.35, (125 g.  $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$  plus 50 g. sodium lauryl sulfate per gallon). After a rinse, a mixture of 2-1/2% Olin's Polytergent B-300, 2-1/2% sulfamic acid, 22% butyl cellosolve, and 10% acetic acid, in water, pH 1.85 max., (hereafter called general metal cleaner) is used. Another rinse is applied, usually followed by immediate plating! In rare cases involving oils and abrasives, where little in-shop cleaning has been performed and days to weeks have passed before parts are received for plating, what appears to be a film has formed, which, fortunately, comes off well in a brief methyl ethyl ketone preclean dip, followed by a water rinse.

COPPER, BRASS, TELLURIUM COPPER, BERYLLIUM COPPER, PHOSPHOR BRONZE, MILD STEEL, NICKEL and SILVER, with or without SOLDER<sup>1</sup> or BRAZING, are cleaned and prepared using this procedure.

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<sup>1</sup> Solder darkens, presenting a bad appearance, but is ideally suited for ensuing plating.

Copper electroforms wherein some of the copper but not all has been grown should, if removed from the electroforming bath for processing, be anodically electro-cleaned, producing vigorous bubbling, in phosphoric acid without rinsing afterwards. Reentering with adherent phosphoric acid prevents lamination of the copper otherwise produced.

ALUMINUM MANDRELS used in electroforming are cleaned in a different way to be considered in the section on electroforming.

## II. BATH FORMULATIONS, MAINTENANCE, QUANTITATIVE ANALYSIS, AND OPERATIONAL PARAMETERS

### NRAO Acid Copper Sulfate Electroplating/Electroforming Bath

Discussion: This is a self-leveling copper sulfate-sulfuric acid electroforming/electroplating bath incorporating proprietary brighteners/levelers. The anodes used are Univertical's oval cross-section 10"-long phosphorized copper, the pH is in the vicinity of 1.0, agitation is vigorous, filtration is by a pump with a polyester wound cartridge, operational temperature is room temperature or slightly warmed in winter. Currently at NRAO, two of these baths are used containing about 55 gallons each, using four, triple-bagged anodes, each of one square foot surface area. (Of course, the number of anodes should be increased/decreased according to need.) These baths are housed in 24" x 24" x 24" PVC-lined steel cubical tanks, as in Figure 1. Note: Nylon should not be used in connection with these baths, as it is attacked by the bath, producing an oily scum.

NOTE: Filter-Pump  
Not Illustrated

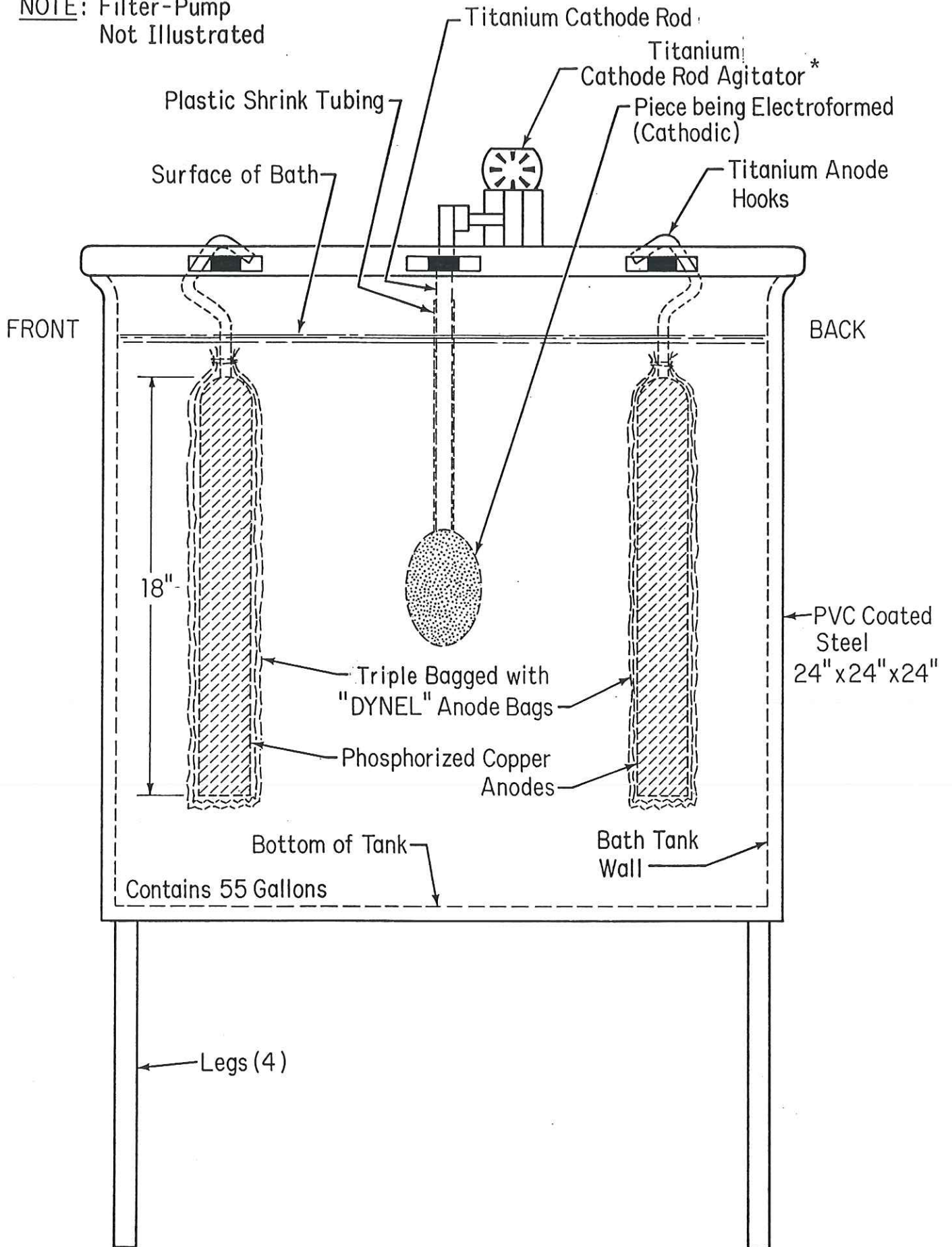


Fig. 1. Large acid-copper-sulfate electroforming bath.

\*Motor is brushless: Dayton shaded-pole gear motor, Model 3M137B.



Maintenance of Acid Copper Bath: Hull cell tests are performed as needed to determine necessary changes in brightener levels. The 267 ml. Hull cell is used, at 2 amps per panel for 5 minute intervals, on highly polished, commercially-available<sup>2</sup> brass panels using a corrugated copper anode. Usually frequent small additions of brightener are made according to the "trained-eye" principle obviating the necessity of hull-cell tests.

Occasionally major adjustments or checks are necessary in order to keep the acid copper bath operating at its best (for example, being more than 100% efficient, copper concentration tends to increase with time). Then quantitative analysis by wet-test methods is used. The following procedures apply to determine copper-metal concentration, sulfuric acid concentration, and - very rarely - chloride-ion concentration:

1. COPPER METAL CONCENTRATION: Carefully pipette a 5.0 ml. sample of the bath into a 500 ml. Erlenmeyer flask and add 100 mls. distilled water. Add ammonium hydroxide (approximately 2-5 mls.) until solution turns a deep blue. Gently boil until the deep color disappears, using a few boileezers, since foaming tends to be vigorous. Add glacial acetic acid until the solution turns acid (5-10 mls., as determined by pH paper test of vapor boiling off). Add 2 mls. more glacial acetic acid. Boil an additional 2 minutes, then cool and add 4 grams potassium iodide (amount not critical as it is used as an indicator). Titrate with 0.1 N sodium thiosulfate solution until the solution turns a pale yellow color. Add 2 mls. starch solution (not critical) and titrate to a white endpoint. The ounces per gallon of copper equals 0.17 times the number of mls. of thiosulfate.

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<sup>2</sup> McGean-Rohco, 2910 Harvard Ave., Cleveland, OH 44109.

2. SULFURIC ACID CONCENTRATION: Carefully pipette a 10.0 ml. sample of the bath into a 500 ml. Erlenmeyer and add 150 mls. distilled water. Titrate with a 1.0 N sodium hydroxide solution with rapid mechanical stirring until the first faint sign of a permanent turbidity appears. The ounces-per-gallon of sulfuric acid equals 0.426 times the number of mls. of sodium hydroxide solution.

3. CHLORIDE ION CONCENTRATION<sup>3</sup>: (gravimetric determination) Pipette accurately a 100 ml. sample of the bath into a 250 ml. beaker. Add 10 mls. nitric acid and 5 mls. 10% silver nitrate. Boil and stir until the silver chloride is coagulated. Carefully filter through a fine sintered-glass weighing crucible, and thoroughly dry at about 120°C for two hours. Cool and determine the weight by subtracting the tare weight. Mg/L Cl equals wt. AgCl times 0.2474 times 4000.

#### Modified ASTM Rochelle Copper Cyanide Flash Bath

Composition and Formulation: 8 liters distilled water  
add 899 grams sodium cyanide  
slowly with stirring add 756 grams CuCN  
add 1080 grams Rochelle Salts (Na-K Tartrate)  
add water to make 18 liters  
adjust free cyanide to 0.50 ounces/gallon  
(NaCN raises, CuCN lowers)  
adjust pH to 9.5-10.0 with NaHCO<sub>3</sub>

Operational Parameters: This depends upon the particular use to which the bath is going to be put. For flashing zincated aluminum, individual current densities and

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<sup>3</sup> This test is only necessary if obvious anode polarization is taking place, resulting in no current flow. Generally, chloride is provided by replacing evaporated water with tap, not distilled water. Excessive chloride ion deactivates the anodes, sending current flow to zero and voltage reading to maximum on power supply.

times depend upon the aluminum's geometrical configurations, but the norm is 24 ASF for 45 seconds or more where appropriate and "burning" of sharp projections is not a factor, requiring a lower current or a shorter time period. If current, on the other hand, is too low, adhesion suffers, producing blistering that may not be noticed until the piece is then put in a warmer bath. Unequal thermal expansions produce this effect. For thin coatings over silver, brass, etc., a lower current density may be used.

Other operational parameters remain constant, regardless of the use to which the bath is put. These are as follows:

Temperature:	room temperature (40°C if critical)
Agitation:	magnetic stirring
Filtration:	batch
Anodes:	6 (9" x 0.5" diameter) OFHC anodes evenly spaced around inside perimeter of glass beaker

#### Quantitative Analysis:

1. Free Cyanide: Pipette a 10.0 ml. sample into a 250 ml. Erlenmeyer flask. Add 90 mls. distilled water, also 5 mls. 10% KI solution. Titrate with 0.10 N AgNO<sub>3</sub> solution until a faint yellow turbidity persists. NaCN in ounces/gallon equals 0.131 times number of mls. silver nitrate solution. Normal bath color is a pale, straw yellow. If the bath persists in a blue color, then the free cyanide is about zero. Adjust it!

2. Rochelle Salts: This test is not normally required or performed. However, if desired, there is a satisfactory procedure in *Metal Finishing Guidebook and Directory*.



3. Carbonates: The same as is the case with Rochelle Salts. It should be noted that carbonate comes from electrolytic and/or thermal oxidation of cyanide. Carbonate concentration has a lot to do with plate quality. If plate quality becomes poor and pH adjustment or free cyanide adjustment doesn't improve matters much, it is likely the case that carbonate contamination has ruined the bath.

#### Sel-Rex BDT 200 Sulfite Gold Plating Bath

Discussion: This is an excellent gold plating bath, much superior for most purposes in the electronics field (excepting wire-bonding use or whisker posts and related applications which tend to suffer from galling) to the cyanide gold baths. It can also be a very annoying bath if not properly understood and carefully maintained! It is self-levelling and produces a very bright, pure, amorphous, yellow gold (99.9%) plate of moderate hardness (knoop hardness 130 to 190). It should be noted that due to the bath's alkalinity, glass is gradually dissolved. Because of this and gradual contamination, the bath should be totally replaced every couple of years. Please note that the fresh bath is water white. Bath brightener contains compound arsenic.

Some of the problems that can arise: peeling and blistering<sup>4</sup> of gold off base metal, patches of black, pink appearance on high current areas, organic cellophane-like pieces floating in bath, etc. Below is a trouble-shooting chart to demonstrate what to do to treat a symptom and what causes the symptom:

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<sup>4</sup> When attempting to use BDT-200 over a layer of copper from the acid copper baths, blistering may result, perhaps caused by surface brighteners, pH effects and/or physical surface effects. Use an intermediate layer of Pur-A-Gold 125.

<u>PROBLEM</u>	<u>PROBABLE CAUSE</u>	<u>PROBABLE SOLUTION</u>
peeling or blistering of gold	lack of compatibility with underlying layer; or immersion layer formed before current flow commenced	plate a layer of metal in between from a bath that is compatible with both; enter bath electrically hot
patches of black	incomplete cleaning or too low a pH	adjust pH to 9.5 to 10.0 and/or clean part a little better
pink appearance on high current areas	gold level in bath is too low or current is too high or insufficient brightener	recalculate current. If current is okay, analyze gold content and make necessary additions.
brassy-reddish appearance	specific gravity too high, due to buildup and dissolved glass	use up as much gold as possible, increasing temperature and/or dilution, then replace
lint and particles	gradual buildup with time	filter bath using pleated papers or, if vacuum filtering, using glass fiber filter disc.



cellophane-like pieces floating in bath	probably electrolytic oxidation-reduction reaction of organics in bath and/or salt buildup; dissolved glass reprecipitating	filter bath, probably need to use up gold and then replace bath.
yellow color of bath	pH too low or copper contamination	adjust bath w. 20% NaOH. If contaminated, reclaim gold and replace bath.
gassing or brown powdery deposits	current is way too high, probably by factor of 10X or more	check scale! reduce current!

Operational Parameters: See the chart below. The temperature recommended by the Sel-Rex people is 35-55°C. We use 40°.

Gold content-----0.7 to 2.0 recommended oz./gal. If less than 0.5, problems begin to manifest themselves, e.g., pink in high current area.

pH-----9.5 (adjust up w. 20% NaOH, down ONLY IF ABSOLUTELY NECESSARY w. 5% H<sub>2</sub>SO<sub>4</sub> very carefully, as gold tends to precipitate out due to some sulfite-forming sulfurous acid, which reduces some gold ions to the metallic state.

Filtration-----batch vacuum filtered through glass-fiber filter.

Specific gravity----8°-35° Baume (preferably lower end).

Anode-----Platinized Niobium.

Current density-----3 ASF, straight DC, entering bath electrically "hot" using voltage regulation according to Figure 2.

CHARACTERISTICS BDT -200 GOLD BATH CURRENT VS VOLTAGE  
 DC 3 ASF, 40°C, BAUME 30, pH 9.5, ~2 OZ AU/CAL

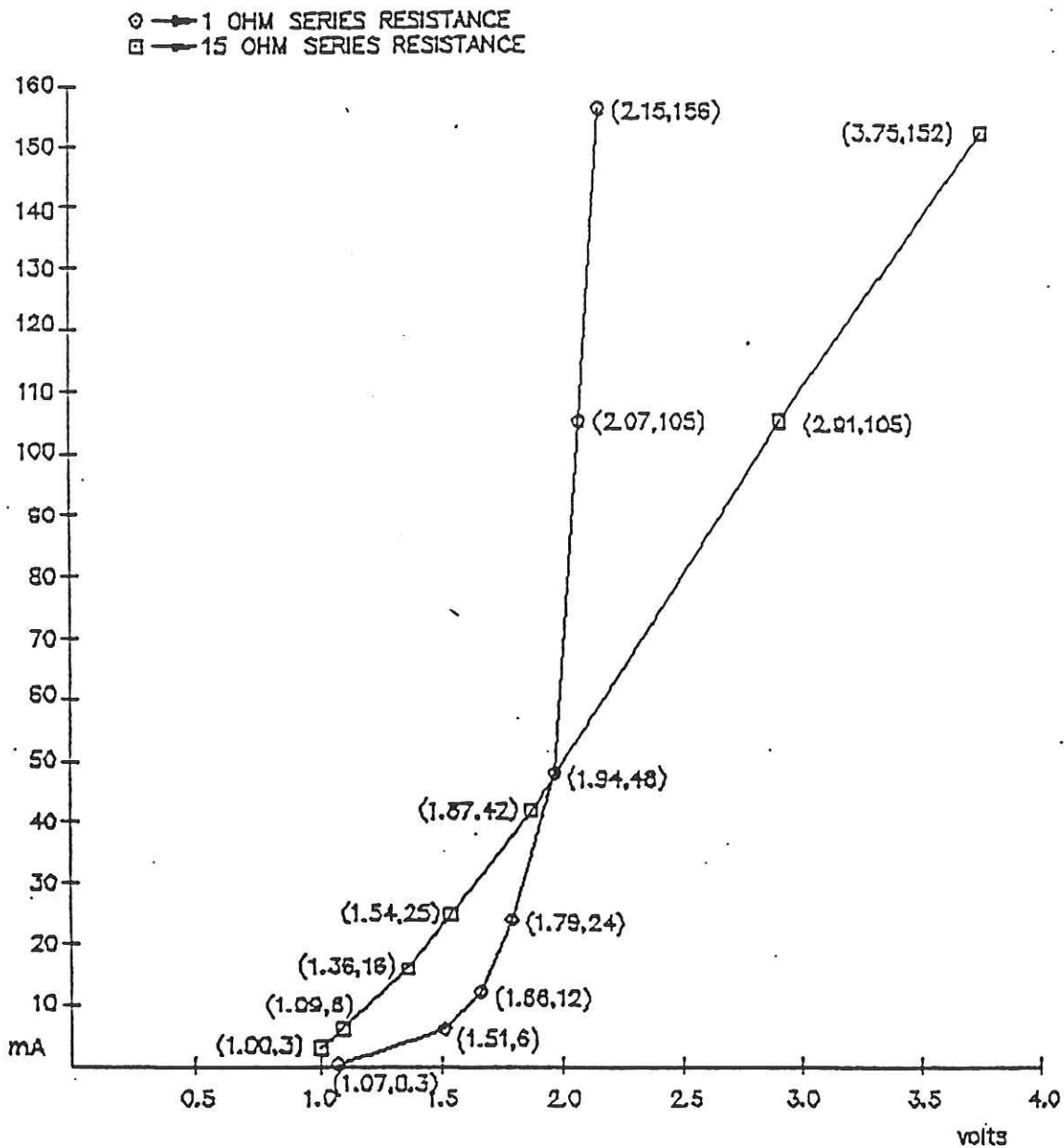


Fig. 2

Plating rate-----0.0001" in 14 minutes @ 3 ASF or 1 troy ounce Au in  
250 amp minutes.

It ought to be noted that the literature reports a sulfite gold, produced by the Engelhard company, that can be used in wirebonding.

Quantitative Analysis of BDT 200 Bath: This consists of a gold metal determination, modifying the procedure used for gold metal determination of a cyanide-gold bath. The modified procedure is as follows:

1. Accurately pipette 10.0 mls. gold bath into a 500 ml. Erlenmeyer. In a hood, add 15 mls. conc. HCl, and evaporate with stirring to a syrup (DON'T ALLOW TO BOIL DRY). Since some sulfurous acid is generated, some of the gold will come out as finely divided metal. Add 5 mls. HNO<sub>3</sub> to the syrup and 15 mls. HCl. Boil to a syrup again. Add another 15 mls. HCl, boil to a syrup again. Repeat one more time. This drives off nitrogenous compounds. Add 150 mls. distilled to final syrup. Add 25 mls. of 20% KI and 2 mls. 1% starch indicator solution. Titrate w. 0.01 N sodium thiosulfate to colorless endpoint, using good lab procedure. Record number of milliliters thiosulfate used.

2. Now, to standardize the thiosulfate solution used, weigh out 0.0500 grams fine gold wire. Dissolve in minimum aqua regia necessary, then go through a boiling down and three HCl additions with boiling down, as above. Titrate and record this number. Then, the gold in the bath can be calculated thusly:

troy oz. Au/gallon of bath =

$(0.608/\text{mls. thiosulfate for } 0.0500 \text{ g. Au}) \times \text{mls. for sample}$

For example, if titration of bath took 48 mls. thiosulfate and standardizing took 37 mls. for the 0.0500 grams wire, then

$\text{tr. oz./gal. Au} = (0.608/37) \times 48 = 0.79$

### Pur-A-Gold 125 Cyanide Gold Plating Bath

Discussion: This is a very soft (Knoop 50-80) high purity (99.99+%) gold bath producing crystalline deposits suitable for wirebonding and in the manufacture of coil stock (which undergoes considerable twisting and flexing). It is NOT self-levelling and is not as suitable as the BDT-200 bath for most purposes. It is, however, a somewhat "forgiving" bath as far as part cleanliness goes.

It has a phosphate-citrate base and, hence, pH can be adjusted as for the Aurobond TC bath.

#### Operational Parameters:

Gold content-----0.7 - 2.0 troy oz./gal.  
pH-----5.75 ( $\text{Na}_3\text{PO}_4$  ↑, citric acid ↓)  
Temperature-----60°C (important for best results)  
Anode-----platinized niobium  
Current density-----3 ASF<sup>5</sup> straight DC  
Plating rate-----50 microinches/6 minutes

Two of these baths are maintained. One is strictly for wirebonding applications, and is replaced without replenishing by a fresh bath. The other is replenished for general soft gold applications.

### NRAO Bright Nickel Bath

#### Composition:

999 g.  $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$   
120 g.  $\text{H}_3\text{BO}_3$   
28 g. sodium paraphenolsulfonate  
132 mls. 40% formaldehyde  
to 4 liters -- adjust pH to 4.0  
with  $\text{H}_2\text{SO}_4$  or  $\text{NaHCO}_3$

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<sup>5</sup> When plating thick layers where edge buildup is a problem, reduce current density slightly.



Use: Bright nickel is for corrosion resistance and physical appearance. It usually requires some gentle polishing with a cream and polishing rag. It can be plated right on copper or zincated aluminum. Caution must be used to insure that plating times aren't too long and current densities not too high. Parameters: 50°C, pH 4.0, 32-1/2 ASF, nickel anode, 7 minutes maximum, vigorous agitation and uniform exposure of surfaces approximating "conforming anode" (due to poor throwing power).

#### NRAO Electroless Copper Composition<sup>6</sup>

##### Composition:

###### Part A (one) -

In one liter volumetric in 300 mls. water dissolve 13.3 g. copper lactate, then dissolve 18 g. EDTA·2Na, bring to one liter

###### Part B (two) -

Per 100 mls. Part A, add, only upon using, 2 grams paraformaldehyde flakes, then concentrated KOH solution and adjust pH to 12.5

Use: Quartz windows (washer-shaped) get their inner and outer surfaces plated with copper for soldering them into place as vacuum-tight windows for microwave radiation. For adhesion of the initial electroless copper (which is prerequisite to electrolytic copper deposition), the surface of the quartz must be roughened. This substrate (already roughened by manufacturer) is then pretreated as follows:

- 1) 5 minute soaking in activator consisting of 20 g. SnCl<sub>2</sub> in 500 mls. water adjusted by HCl or KOH to pH 1.25.
- 2) GENTLE water rinse.

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<sup>6</sup>A new bath stabilized with mercaptobenzothiazole is under consideration.

- 3) 5 minute soaking in catalyst consisting of 1 g.  $\text{PdCl}_2$ /500 mls. water adjusted to pH 4.0 with KOH (not  $\text{NH}_4\text{OH}$ ) or HCl.
- 4) GENTLE water rinse.

Then immerse the part(s) immediately in the freshly prepared electroless copper until it is pretty much "spent." Rinse thoroughly with water and proceed to next step.

#### Hard Anodizing Bath (for Aluminum)

Composition: This bath consists of 20% concentrated sulfuric acid by volume in distilled water.

Use: Keeping bath at 0°C sharp, reverse polarity (making the piece the anode) is used at 15 ASF. The piece is sufficiently anodized when the voltage rises to 75V. The piece, when properly done, will be a light charcoal color. Anodizing is used at NRAO to produce pieces that are thermally conductive electric insulators.

#### Stainless Steel Activation Nickel

Composition:

8 oz./qt.  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$

4 liq. oz./qt. HCl (conc.)

Use: Cleaned stainless is plated for three minutes @ 100 ASF at room temperature.

Please note that this bath has been largely discontinued in favor of other procedures.<sup>7</sup>

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<sup>7</sup> See Section VI, Miscellaneous Information, #7, at end of this paper for exception.

## Miscellaneous Baths for Possible Occasional Future Use

### 1. Electroless nickel:

Composition: 30 g.  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$   
(per liter) 11.9 g. potassium citrate  
10.0 g. sodium hypophosphite

Use: Using same procedure as for electroless copper activating-catalyzing

pH 5.85

temperature 85°C sharp

growth rate estimated 0.2 mils/hr.

## III. GENERAL PLATING PROCEDURES

Most often, pieces which are received in the plating lab consist of the following metals: copper, leaded brass, phosphor-bronze, tellurium-copper, silver, beryllium-copper, oxygen-free-high-conductivity (OFHC) copper and extra-tough pitch (ETP) copper. These metals are generally plated in the same manner, using the same bath combinations. General plating procedure is as follows:

1. Ultra-sound clean in alkaline soap cleaner/water rinse
2. Ultra-sound clean in general metal cleaner/rinse
3. Copper cyanide strike 10 seconds @ 12 ASF (if not pure copper)/water rinse (this step eliminates corrosion of non-copper metals, e.g., zinc through pores in the gold electroplate). USE THIS STEP ONLY WHEN ESSENTIAL.
4. Gold plate to desired microinches (usually 75 minimum; however, less if dimensions are critical, or 100 or more if high durability or good heat properties required - 250 or more of cyanide gold for wirebonding)
5. Water-rinse, air-dry

#### IV. SPECIAL PLATING PROCEDURES (INCLUDES ELECTROFORMING)

A number of different, special plating procedures are called for. A number of these are listed below:

1. Copper plating stainless steel and Kovar
2. Preparation for electroforming on aluminum types 7075, 2024, and 6061
3. Nickel electroforming

These will be considered one at a time below:

1. Copper plating stainless steel: A significant improvement has been made in this particular area. Nascent hydrogen is plated onto the stainless steel piece using 30% H<sub>2</sub>SO<sub>4</sub>, a platinized-niobium anode and a current density of 125 ASF for four minutes. Then the steel is IMMEDIATELY immersed in acid copper bath and plated. Five to ten minutes @ 30 ASF in the acid copper bath is the prescribed treatment for preparing stainless steel for soldering (approximately 100 to 200 μinches). Excess copper can be "painted off" with concentrated nitric acid.

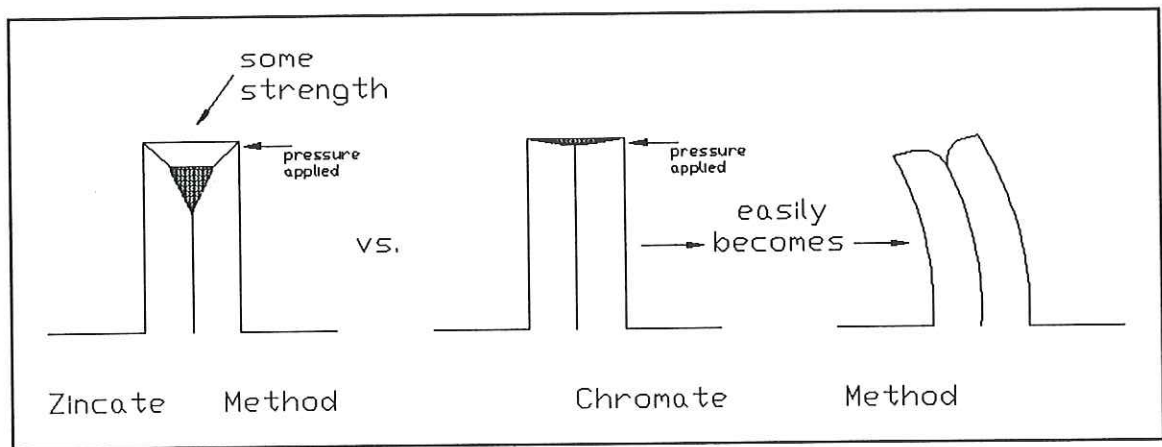
2. Preparation for electroforming on aluminum types 7075, 2024, and 6061: Two methods for copper electroforming are used, depending on what the particular job calls for: (I) a zincate immersion plate method, standard in the industry, and (II) a chromate dip preparation method, as developed by V. Lakshminarayanan<sup>8</sup> of the Raman Research Institute in India and modified for NRAO use. The former method provides an adherent copper which tends to grow with a current distribution that is very geometry dependent, but that is nevertheless very useful in, for example, the production of corrugated feedhorns with high groove aspect ratio (depth to width) of the mandrel. The latter method of chromating leaves a microscopically uniform peppered appearance of what seems to be either chromium sesquioxide or a hydrated

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<sup>8</sup>narayan@rri.ernet.in



version of the sesquioxide. The copper conforms but is very weakly adherent; current distribution is not so geometrically dependent, so that, for instance, flat surfaces do not tend to grow so cup-shaped when non-conforming anodes are used, minimizing machining. Even grooves tend to close, filled with metal rather than trapped bath. For very thin grooves, however, the finished copper lacks strength and is easily pushed over as two adjacent copper sheets.



Both methods have their place. An example of a useful application of the chromating method may be seen in Figure 3.

Where zincating solutions are currently used to prepare aluminum for copper plating, here is the general procedure:

#### I. Zincating Method

1. Immerse with swirling into an alkaline cleaning dip consisting of 44 grams  $\text{Na}_2\text{CO}_3$  + 44 grams  $\text{Na}_3\text{PO}_4$  per 1/2 gallon solution pH 11.9 for 6-15 minutes at room temperature. Should appear blue like steel.
2. Water rinse.

3. Amchem nitric deoxidizer #4<sup>9</sup> dip with swirling 10 seconds.
4. Carefully and thoroughly water rinse.
5. Zincate, as will be described below.
6. Quickly but thoroughly water rinse.
7. Copper cyanide flash<sup>10</sup>.
8. Acid-copper-sulfate plate 1-5 minutes @ 20 ASF
9. Water rinse.
10. Pur-A-Gold 125 one to two minutes.
11. BDT 200 plate (generally 300 to 500 microinches).
12. Water rinse.
13. Pur-A-Gold 125 again, as above.
14. Water rinse.
15. Electroform<sup>11</sup> in acid-sulfate-copper @ 10 to 20 ASF for first 24-48 hours, then up to 20-30 ASF thereafter, OR electroform in acid nickel sulfamate (Sel-Rex "Sulfamex" bath) @ 30 ASF -- other current densities only if compressive or tensile stress desirable.

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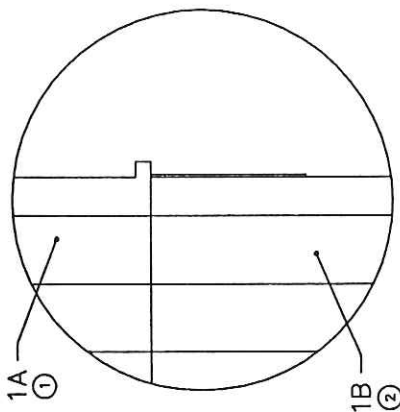
<sup>9</sup> Amchem Nitric Deoxidizer #4 is manufactured by Amchem Products, Inc., Ambler, PA 19002.

<sup>10</sup> We have the bath short-circuited with proper current on just prior to flashing for quick response time. Otherwise, the zincate layer partially deteriorates, resulting in blistering between aluminum and copper when the mandrel is placed in hot gold baths (differing thermal expansions).

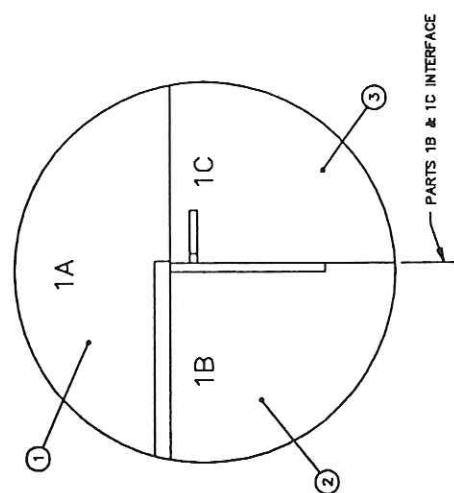
<sup>11</sup> For some electroform geometries (normally using the zincate process), cleavage planes and grooves are a serious problem. This usually can be handled by (1) machining a layer off after partial growth (smearing the cleavage line with copper in the process), (2) solder filleting using a solder pot (not a torch), or (3) filleting with silver conductive epoxy. In the second case, a copper cyanide flash is necessary before reentry into the electroforming copper bath. In the third case, a light sanding is required to expose metal particles, followed by a copper cyanide flash, and then reentry into the electroforming bath.

**MATERIAL LIST**

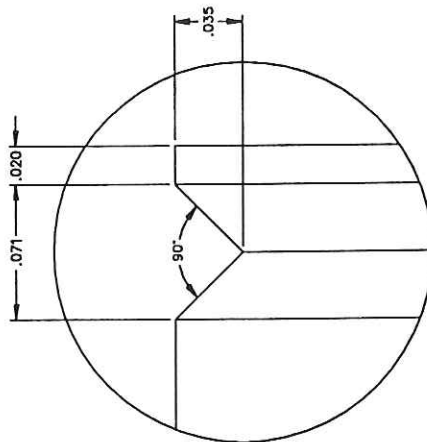
ITEM	QTY	DESCRIPTION	REMARKS
1	1	PART 1A	B20002F002
2	1	PART 1B	B20002F003
3	1	PART 1C	B20002F004
4	4	STAINLESS STEEL DOWEL - 3/32 DIA. X 1/2 LONG	LIGHT DRIVE FIT
5	4	ST. ST. SOCKET HEAD CAP SCREW - 4-40UNC-2A X 1/2	



**DETAIL "Y"**  
SCALE: 20X SIZE

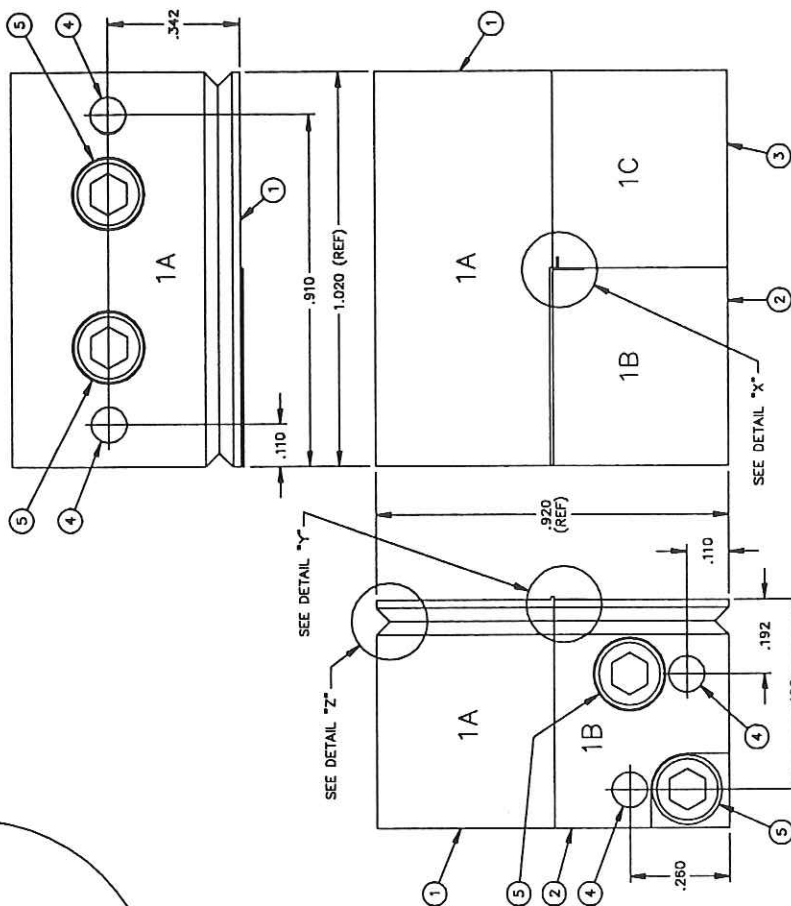


**DETAIL "X"**  
SCALE: 20X SIZE



**DETAIL "Z"**  
SCALE: 20X SIZE

NOTE: THIS V GROOVE SHALL EXTEND THE ENTIRE PERIMETER OF THE PART 1 ASSEMBLY. SEE NOTE 2 AT RIGHT.



UNLESS OTHERWISE SPECIFIED TOLERANCES

FRACTION	DECIMAL	FINISH
1/4	±.0005	AS MACHINED
1/8	±.0005	AS MACHINED
1/16	±.0005	AS MACHINED
1/32	±.0005	AS MACHINED
1/64	±.0005	AS MACHINED
1/128	±.0005	AS MACHINED
1/256	±.0005	AS MACHINED
1/512	±.0005	AS MACHINED
1/1024	±.0005	AS MACHINED

ALL DIMENSIONS ARE IN INCHES

- NOTES**
- △ THE OVERALL PATTERN BLOCK DIMENSIONS ARE .010 LARGER ON EACH SIDE THAN THE FINAL COPPER BODY.
  - △ THE V GROOVE SHOWN IS USED TO PROVIDE AN ANCHOR FOR THE ELECTROFORMED COPPER. THIS PREVENTS THE COPPER AND ALUMINUM PATTERN BLOCK FROM SEPARATING WHEN MACHINING AWAY MOST OF THE ALUMINUM.

NATIONAL RADIO ASTRONOMY OBSERVATORY  
CHARLOTTEVILLE, VA. 22903

TITLE: **PART 1**  
**ELECTROFORMING PATTERN BLOCK ASSEMBLY**

PROJECT: 585 GHZ SIS MIXER (VERSION I)

DESIGN	BRADLEY/PAM	4-6-93	COMPUTER DRAWING	CDL X20002\F001
DRAWN	DGS	5-26-93	MATERIAL	
SHEET	1 OF 1	FINISH		

SCALE: 4X SIZE & NOTED  
DWG. NO.: C20002F001  
REVISION

The zincating solution and procedure are listed below:

Zincate Solution Composition

Add 525 g. NaOH + 100 g. ZnO to  
a solution of 10 g. Rochelle salts  
+  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ , 1 g. in  $\text{H}_2\text{O}$   
make up to one liter

Technique

Zincate only once for 5-10 seconds.  
Rinse quickly. Plate in cyanide  
copper @ 24 ASF seconds or more,  
depending on whether there are  
projections, deep recesses, etc.

II. Chromating Method (all operations at room temperature)

1. Routinely clean masked mandrel using general purpose procedure. Then for one minute immerse with swirling into room temperature 10% aqueous sodium hydroxide.
  2. Water rinse briefly.
  3. Desmut 5 seconds in Amchem nitric deoxidizer #4.
  4. Thoroughly but quickly rinse.
  5. Lightly swirl in a solution consisting of 5%  $\text{Na}_2\text{CO}_3 \cdot 4\text{H}_2\text{O}$  and 2% NaOH, both by weight, for six minutes.
  6. Rinse in two waters with no swirling (which would disturb fragile chromium oxide coating).
  7. Quickly place moist substrate in small acid copper electroforming bath surrounded with large excess of anode surface area with only agitation enough to keep substrate from forming "burned" deposit. Use 30-40 ASF for the first few minutes, then lower to 20-25 ASF.
3. Nickel Electroforming: Currently (but only rarely) one nickel electroforming bath is used - Sel-Rex "Sufamex" acid sulfamate bath (with a small fraction of 1% sodium lauryl sulfate added). To quote the specs supplied, it "produces dull, pure, ductile, fine-grain, low-stress nickel deposits."



To the bath, as provided by the manufacturer, after dummmying for 24 hours, is added 0.1 g/liter of sodium lauryl sulfate, to eliminate hydrogen pitting and increase bath efficiency.

Parameters for use are as follows:

Temperature-----50°C; varied if stresses and other property  
changes desired  
Current Density-----30 ASF; more or less if various stresses desirable  
Agitation-----very vigorous  
Filtration-----batch  
pH-----3.25 adjusted with sulfamic acid ↓ or nickel  
carbonate ↑. If nickel carbonate is needed, add to  
a portion of bath, making a slurry and then  
gradually add back to bath.  
Current-----Straight DC (at present)  
Addition Agents-----Sulfamex Anode Activator

The electroforming is done after preparing the aluminum according to standard procedure involving alkaline cleaner, Amchem deoxidizer, copper striking, copper sulfate plating and gold plating. 10% sodium hydroxide is used to dissolve out the aluminum after electroforming and machining since nickel dissolves rapidly in hydrochloric acid. The standard flash dissolver is then used, followed by gold plating.

## V. A NOTE ON AN ANOMALOUS PLATING BEHAVIOR

When a three-dimensional piece is plated, having length, width and thickness on the same order of magnitude dimensionally, it is a simple matter to calculate plating current necessary for any particular bath following specifications supplied by the manufacturer. For instance, a piece with a surface area of four square inches and simple geometry, in a bath normally used at 3 amps/square foot, would require 83 mA of current for proper plating. However, as thickness of a piece approaches zero (e.g., 3"x3"x1/32"), this calculated current proves excessive, generally producing "burning" of the deposit at corners, perhaps even along the edges. Thus, I propose the following equation to calculate current for parts possessing one dimension considerably smaller than the other two:

$$\text{actual current} = 2/3 \text{ calculated current} \left[ 1 + T \left( \frac{W + L}{W \times L} \right) \right]$$

where T = thickness,

W = width and

L = length.

This equation is practical. It is not theoretically derived, is not an attempted explanation of phenomena and, in fact, would only be directly applicable to thin plates of uncomplicated geometry.

Current assessment is critical when copper flashing zincated aluminum mandrels for electroforms since a narrow "window" of current range exists between (1) "burning" the deposit and (2) providing too little current, thus allowing the zincate to be somewhat attacked by the alkalinity of the copper cyanide bath before copper plate integrity can be established, resulting in poor adhesion and potential "blistering" of the deposit. The above equation should be applied to assist in preventing these situations.

## VI. WASTE DISPOSAL

Proper hazardous waste disposal is best effected by turning wastes over to an approved waste-disposal facility. NRAO uses the Special Materials Handling Facility at the University of Virginia. However, small quantities of some wastes can be disposed of in the laboratory following the procedures in *Prudent Practices for Disposal of Chemicals from Laboratories*, 1983 National Academy Press.

## VI. MISCELLANEOUS INFORMATION

1. Gold recovery: Gold should be recovered from BDT-200 bath by careful addition of  $H_2SO_4$  (in a HOOD! due to evolution of sulfur oxides) to pH 1.0 filtration, rinsing thoroughly and heating filtrate in appropriate crucible to 2100°F to melt.
2. Aluminum mandrel removal: For copper electroforms, 50:50 conc. HCl:H<sub>2</sub>O seems to work the best overall. The aluminum should be dissolved out at room temperature. Where small openings are involved using 2024 aluminum, ultrasound should be employed. Excess copper as noticed by dark yellow color causes immersion plating of copper on dissolving aluminum surfaces, slowing or stopping dissolution of remaining aluminum and thinly covering outside gold-plated surfaces. This comes from copper off piece and from copper released from alloyed aluminum and may even involve a "battery" effect. Also it should be noted that three molecules of HCl are consumed for each Al atom dissolved! In addition, conc. hydrochloric acid is only 35% hydrogen chloride to start with! So don't try to get by using too little acid; it won't work. Dissolving of additional aluminum slows when saturation of solution with  $Al^{+3}$  approaches. Also, don't think 100% acid works better - it doesn't!

3. Copper flash removal: A solution consisting of 15%  $\text{HNO}_3$  and 55% glacial acetic acid appears to work quite well for dissolving copper flash from and off electroform after aluminum has been removed. Chromic acid solution has been suggested as superior.

4. Gold-stripper: A suitable gold stripping composition consists of 30 g. sodium metanitrobenzenesulfonate plus 120 g. sodium cyanide in 1 liter of solution. It dissolves gold and copper off aluminum or Kovar at room temperature. For best results, don't heat unless absolutely necessary.

5. Scale buildup: The water distillation apparatus should occasionally be cleaned of scale by dismantling, soaking in concentrated HCl and scraping with wires and brushes until it is all removed.

6. Mild Steel: This is routinely cleaned, then CuCN flashed at 24 ASF for 30 seconds to one minute and plated in the acid copper bath.

7. Music wire and the steel of drill bits need to be plated with gold on occasion. Anodic cleaning at 1,500 ASF for one minute in conc.  $\text{H}_3\text{PO}_4$ , followed by a one-minute nickel flash (bath used described in Section II, page 16), and one-minute acid copper bath plating precedes gold plating.

8. Aluminum can be bright-dipped by immersion for exactly one minute in a 215° sharp mixture of 84 mls. phosphoric acid to 3.3 mls. nitric acid to 12.7 mls. glacial acetic acid. Results are excellent. Surfaces are brighter and cleaner than possible by machining.

9. Small gold pads of the thickness order of less than a mil for soldering to SIS junctions, can be formed by the following procedure:

- (1) Form a cured resist-pattern (American Hoechst Corp. AZ5413) soluble in organic solvent.



- (2) Don't clean the bare aluminum, thus preserving the aluminum oxide outer layer.
- (3) Plate one minute at 30 ASF in Aurobond TC.
- (4) Rinse.
- (5) Plate in BDT-200 at 1 or 2 ASF to required thickness.
- (6) Rinse.
- (7) Carefully flake off loosely adhering pads. If aluminum had been cleaned, pads would have partially stuck and been ruined upon removal.

10. Copper electroforms that have been removed midstream and that are found to have perhaps one or two little pits in the copper can be "patched" for further copper growth by filling with indium and sanding flush. Then a cyanide copper strike before reentry is sufficient for fine results.

11. Gold stripping off copper-flashed, zincated aluminum mandrels can be nicely accomplished using the following:

Potassium Ferrocyanide	50.3 g/L	49°C
Potassium Cyanide	15.0 g/L	6 volts
Potassium Carbonate	9.8 g/L	piece anodic pH 9.0

Adjust pH with monobasic potassium phosphate.

Frequent additions of cyanide and pH adjuster are commonly necessary.

12. Stainless steel electropolishing is performed with the following:

330 mls/L	85% Lactic Acid	85° C
400 mls/L	85% Phosphoric Acid (mixed)	300 ASF
135 mls/L	Conc. Sulfuric Acid	piece anodic

This dissolves approximately 0.1 mils/side/min. of stainless steel.