



# Florida CirTech, Inc.

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## Technical Bulletin CirEtch 130 Micro-Etch

### I. Description

CirEtch 130 is an easy to use, stabilized, hydrogen peroxide/sulfuric acid, copper micro etch. It has been designed to be used in the printed circuit industry. It can be used to increase the surface bonding of copper to copper, preventing poor adhesion of copper and blisters and peelers. It's varied etching characteristic makes it an excellent etchant for the pre-plate line as well as for the pattern plating line. CirEtch 130 is ideal for use with many final finishes, such as immersion silver, ENIG, and immersion tin. Cir130 produces the proper surface topography to enhance solderability.

CirEtch 130 is very easy to waste treat, thereby having an advantage over persulfate compounds. The copper metal can be removed from solution by a simple pH adjustment, giving you a pure cupric sulfate crystal that can be easily disposed. The working solution has an extended life over dry powders.

### II. Operating Parameters

|             |                                          |
|-------------|------------------------------------------|
| Make-up     | See Section IV. Control Parameters       |
| Temperature | 70 to 110°F (22 to 43°C)                 |
| Time        | 1 to 4 minutes                           |
| Process     | Batch Tank                               |
| Agitation   | Recommended                              |
| Ventilation | Recommended                              |
| Tanks       | Polypropylene, Polyethylene              |
| Heaters     | Quartz, Teflon, Stainless Steel          |
| Racks       | Stainless Steel, Plastisol Covered Steel |

### III. Physical Properties

|                  | <u>CIR130A</u>      | <u>CIR130M</u> | <u>CIR130W</u>   |
|------------------|---------------------|----------------|------------------|
| Specific gravity | 1.18 – 1.20         | 0.99 – 1.01    | 0.99 – 1.01      |
| Appearance       | Clear, liquid       | Clear liquid   | Clear liquid     |
| PH               | 1.0 – 2.0           | 6.0 – 8.0      | 6.0 – 8.0        |
| Odor             | Acrid, peroxide     | None           | None             |
| Flash Point      | NA                  | NA             | NA               |
| Contents         | Stabilized peroxide | Organics       | Fume suppressant |

### IV. Control Procedures

#### Make-up

The make ups listed in the table below are recommendations for typical operation. These concentrations can be adjusted to get the desired etch rate.

| Component                        | Pattern Plate Line Make-up | Final Finish Make-up  |
|----------------------------------|----------------------------|-----------------------|
| Water                            | 80% by volume              | 70% by volume         |
| Sulfuric acid, 94 – 98%, reagent | 10% by volume              | 10% by volume         |
| CIR130A                          | 5% by volume               | 10% by volume         |
| CIR130M                          | 5% by volume               | 10% by volume         |
| CIR130W (optional)               | 25 mL per 100 gallons      | 25 mL per 100 gallons |

CIR130A is a stabilized hydrogen peroxide. CIR130M contains stabilizers, surfactants and other organics. It is normally only used for bath makeup. CIR130W is a fume suppressant, and is an optional component. CIR130W can be added if the user desires.

CirEtch 130 activity is controlled by analysis and replenishment. The etch rate should be measured regularly, and replenishments made to maintain the etch rate. Hydrogen peroxide will slowly breakdown and leave the bath as a gas. After long periods of inactivity, analysis and replenishment of CIR130A should be done before use.

Etch rate is influenced by many factors. Increasing the CIR130A concentration will increase the etch rate. Increasing the temperature will also increase the etch rate, but increased temperatures will increase the breakdown rate of hydrogen peroxide. Sulfuric acid concentration should be maintained to ensure copper solubility.

The copper concentration of the working solution is dependent on sulfuric acid content and temperature. The maximum copper sulfate pentahydrate concentration is near 300 grams per liter. Above this level, copper sulfate pentahydrate crystals will precipitate out of solution. Additional crystals will form if the solution is cooled.

#### Copper Reclamation

The copper etched can be reclaimed as copper sulfate pentahydrate. When the copper sulfate pentahydrate in solution reaches 300 g/L (40 ounces per gallon) as determined by analysis, transfer the solution to a holding tank. Using the procedure below, determine the sulfuric acid content. Add the necessary amount of sulfuric acid to bring the bath to a concentration of 10% by volume. Allow the bath to cool to room temperature. The copper sulfate will crystallize and precipitate from solution. The liquid can now be transferred back into the processing tank. Analyze for CIR130A, add the necessary amount of concentrate and the bath is then ready for use. The copper sulfate pentahydrate crystals should be dried and disposed of in accordance with appropriate regulations.

### **V. Analysis**

#### Copper Etch Rate

1. Weigh a 3"x3" copper clad, double-sided coupon to 4-decimal places, and record the weight A.
2. Run the coupon through the Cir130 bath, and measure the time in minutes.
3. Rinse the coupon thoroughly and dry it.
4. Weigh it again to 4-decimal places, and record the weight B.
5. Calculation:

$$\text{Etch rate (microinches per min)} = (A - B) \times 379 / (\text{minutes in solution})$$

The etch rate can vary based on temperature and CIR130A concentration. Increase the temperature and/or CIR130A content to increase the etch rate.

#### Copper Etch Depth

Etch depth is the total amount of copper removed during the entire immersion time. Etch depth can be calculated as shown below.

$$\text{Total etch depth (microinches)} = (\text{etch rate}) \times (\text{minutes in solution})$$

Typical etch depths are shown below.

Etch depth for pattern plating: 20 – 40 microinches total  
Etch depth for final finish lines: 40 – 80 microinches total

Total etch depth can be increased simply by increasing the immersion time, and more copper will be removed.

#### CIR130A Concentration

1. Pipet 1.0 mL of the working solution into a 250 mL Erlenmeyer flask.
2. Add 50-75 mL of DI water and 4 to 5 drops of Ferroin indicator.
3. Titrate with 0.1 N Ceric ammonium sulfate solution to a pale blue endpoint.
4. Calculation:

$$\text{CIR130A content (\% by vol)} = (\text{mLs of Ceric ammonium sulfate}) \times 0.33$$

Additions of CIR130A should be made to maintain the desired etch rate.

#### Sulfuric Acid Concentration

1. Pipet 1.0 mL of the working solution into a 250 mL Erlenmeyer flask.
2. Add 50-75 mL of DI water and 3 to 5 drops of Methyl orange indicator.
3. Titrate with 0.1 N Sodium hydroxide solution to a yellow endpoint.
4. Calculation:

$$\text{Sulfuric acid (\% by vol)} = (\text{mLs of 0.1 N sodium hydroxide}) \times 0.28$$

Additions of sulfuric acid should be made to keep the concentration in the range of 8 – 10% by volume. This maintains the maximum solubility of copper in the bath. Use 94 – 98% reagent grade sulfuric acid for additions.

#### Copper Sulfate Pentahydrate Concentration

1. Pipet 1.0 mL of the working solution into a 250 mL Erlenmeyer flask.
2. Add 5 mL of ammonium hydroxide solution.
3. Add 50-75 mL of DI water and 4 - 6 drops of Pan indicator.
4. Titrate with 0.05 M EDTA to an apple green endpoint.
5. Calculation:

$$\text{Copper sulfate pentahydrate (g/L)} = (\text{mLs of 0.05 M EDTA}) \times 12.1$$

Copper sulfate pentahydrate will precipitate out above the saturation limit, which is near 300 g/L. These copper sulfate crystals can be removed from the solution, and the bath re-used as described in Section IV. Control Procedures.

## **VI. Safety and Storage**

CIR130A is a strong oxidizing solution containing hydrogen peroxide. IT CAUSES EYE AND SKIN INJURY. EFFECTS MAY BE DELAYED. When handling concentrate or working solution, wear protective clothing, gloves and chemical safety goggles. Use in a well-ventilated area. Avoid mists. AVOID CONTACT WITH COMBUSTIBLE MATERIALS. AVOID CONTAMINATION FROM ANY SOURCE. (DUST AND ORGANIC MATERIAL) SUCH CONTAMINATION MAY CAUSE RAPID DECOMPOSITION, GENERATION OF LARGE QUANTITIES OF OXYGEN GAS AND HIGH PRESSURES.

Working solutions containing CE-130 are acidic and should be handled in a manner similar to that of sulfuric acid. Exposed areas should be flushed immediately with copious amounts of cold, clean water for approximately 15 minutes. Seek medical attention promptly in case of over exposure or injury. Store CIR130 components in their original, vented containers. Keep away from sunlight and temperature extremes.

## **VII. Waste Treatment**

CIR130 spent or working solution is an acidic copper etchant. Copper can be removed from solution by precipitation. This can be accomplished by raising the pH of the solution to above 10 with dilute caustic soda. A mild exothermic reaction will occur and a precipitate will form. This precipitate can be removed by filtration. It will contain copper hydroxide sludge. The clear solution remaining can be decanted to the sewer. Observe local waste treatment and disposal regulations. Please ask a Florida CirTech technical sales rep. for more information regarding waste treatment of this chemistry and our complete line of waste treatment line if additional help or information is desired.

### **VIII. Miscellaneous**

Components are available in 5 and 55 gallon drums. Consult MSDS sheets for additional information.

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