Introduction

The analysis of plating bath solutions is necessary for routine bath monitoring and for quality assurance of electroplates. Since the specifications and the cost associated with plated products has increased, the need to analyze for formulations, exhaustible components, and the contaminants has also increased. Typical components of plating baths are metal salts, reducing agents, complexing agents, stabilizers, and pH adjustment buffers. Ion chromatography (IC) provides a simple method for monitoring these compounds.

Either Ion Chromatography method, single-column ion chromatography or suppressor-based ion chromatography, can be used for the analysis of plating bath solutions. The Alltech Electrochemically Regenerated Ion Suppressor (ERIS™ 1000HP Autosuppressor) is designed to improve the detection sensitivity and baseline stability when either anions or cations are analyzed. The ERIS 1000HP converts high conductivity mobile phase into low conducting or non-conducting species, while converting analyte cations into high conductivity bases or analyte anions into high conductivity acids.

Single-column methods for cation analyses have sensitivities that are comparable to that of suppressor-based IC methods. Single-column methods for cation analyses require a high conductivity mobile phase to provide a large difference in detector response between the sample and the mobile phase ions. Anion analyses by single-column IC have detection limits which are less sensitive than suppressor-based IC methods. High or low conductivity mobile phases are used to provide a difference in signal response between the sample and the mobile phase for single-column methods for anion analyses. The Alltech Conductivity Detector can electronically suppress highly conductive mobile phases; thus, it is ideal for single-column IC methods.

Results and Discussion

Chromium electroplating baths contain high concentrations of inorganic, organic salts, and organic additives. The balance of these species is critical in maintaining the quality of plating baths. Chromate and sulfate are the two most common analyzed species in chromium electroplating baths because their ratio in the bath is critical. Figure 1 shows a chromatogram of chromate plating bath.

Copper plating solutions are used in the manufacturing of printed circuit boards and for plating prior to nickel plating. Ion chromatography methods have been used for the analysis of electroless copper, copper pyrophosphate baths, and acid copper sulfate baths. Figure 2 shows a chromatogram of a copper electroless plating bath. Both EDTA and copper EDTA complex are components of this bath and can be separated using the Wescan Anion/R column in the single-column IC mode.

Nickel plating solutions are used for resistance to corrosion and electroforming. Ion chromatography has been used to optimize plating parameters during nickel sulfamate, Watt’s nickel bath, electroless nickel baths, and electroless nickel and cobalt baths. Figure 3 shows a chromatogram of the anions in a nickel plating bath solution. Figure 4 shows a chromatogram of the trace cations in a nickel plating bath solution. This sample was treated with the Sample Concentrator and Neutralizer (SCAN 1000) to remove the high concentration of boric acid in the original sample. Since nickel hydroxide, which is partially insoluble, is formed, most of the nickel has been removed.

Conclusion

Ion chromatography provides efficient methods for analyzing the major constituents and containate ions in plating bath solutions. The use of ion chromatography will ensure efficient coating and corrosion resistance.
### Electroless Copper Bath

1. EDTA
2. CuEDTA

**Figure 2**

- **Column:** Wescan Anion/R, 100 x 4.1mm
- **Mobile Phase:** 5mN Sulfuric Acid
- **Flowrate:** 1.0mL/min
- **Detector:** Conductivity, Range 10

### Nickel Plating Bath

1. Hypophosphite
2. Phosphate and Phosphite
3. Sulfate

**Figure 3**

- **Column:** Allsep Anion, 100 x 4.6mm
- **Mobile Phase:** 0.85mM NaHCO₃:0.9mM Na₂CO₃
- **Flowrate:** 1.2mL/min
- **Detector:** Suppressed Conductivity

### Treated Ni Plating Bath

1. Sodium
2. Potassium
3. Nickel

**Figure 4**

- **Column:** Universal Cation, 100 x 4.6mm
- **Mobile Phase:** 3mM Methanesulfonic Acid
- **Flowrate:** 1.0mL/min
- **Detector:** Conductivity

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### Description

<table>
<thead>
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<tbody>
<tr>
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<td>Wescan Anion / R, 100 x 4.1mm</td>
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