Monitoring of electroless plating baths by capillary electrophoresis

Application Note

Chemical

Authors

Tomoyoshi Soga
Institute for Advanced Biosciences,
Keio University, Japan

Maria Serwe
Agilent Technologies
Waldronn, Germany

Abstract

Electroless plating is mainly used for the plating of non-metals, for example, ceramics and plastics, and allows the plating of complex shaped parts with a uniform film-thickness. In addition to metal cations, the bath solutions contain additives such as reducing agents (which drive the plating reaction) and organic acids (as buffering and/or metal complexing agents). Inorganic anions are also present as counter-ions of the plating metals. These ions can easily be monitored using capillary electrophoresis (CE) with indirect UV detection.

Agilent Technologies

Experimental

Anion analysis was performed using the Agilent Capillary Electrophoresis system equipped with diode array detection and Agilent ChemStation software. The analysis uses the Agilent Plating Bath Analysis Kit (Agilent part number 5064-8228).

Prior to first use, a new capillary was flushed with run buffer for 15 minutes (at 1 bar). Between analyses the capillary was flushed for 4 minutes from an extra buffer vial into waste. Buffer vials were replaced after 10 runs when using 2 mL-vials and after 5 runs when using 1 mL-vials. Sample preparation consisted simply of dilution with water.

Equipment

- Agilent Capillary Electrophoresis system
- Agilent ChemStation
- Agilent Plating Bath Analysis Kit

Results and discussion

Figure 1 shows the analysis of two different plating baths. Electroless nickel-plating baths contain nickel sulfate or nickel chloride, together with hypophosphite as the reducing agent. Formate, present in the electroless copper-plating bath, is an oxidation product of formaldehyde, which is used as a reducing agent. The assay was linear over the range 10–100 ppm with $r^2 > 0.999$. The method detection limit was 1–2 ppm. For the analysis of the electroless nickel-plating bath repeatability (n = 8) was < 0.1 % RSD for migration times and < 4.5 % RSD for peak area. The assay also allows the analysis of iron (II) and iron (III) in electro-plating with direct UV detection at 230 nm (data not shown).

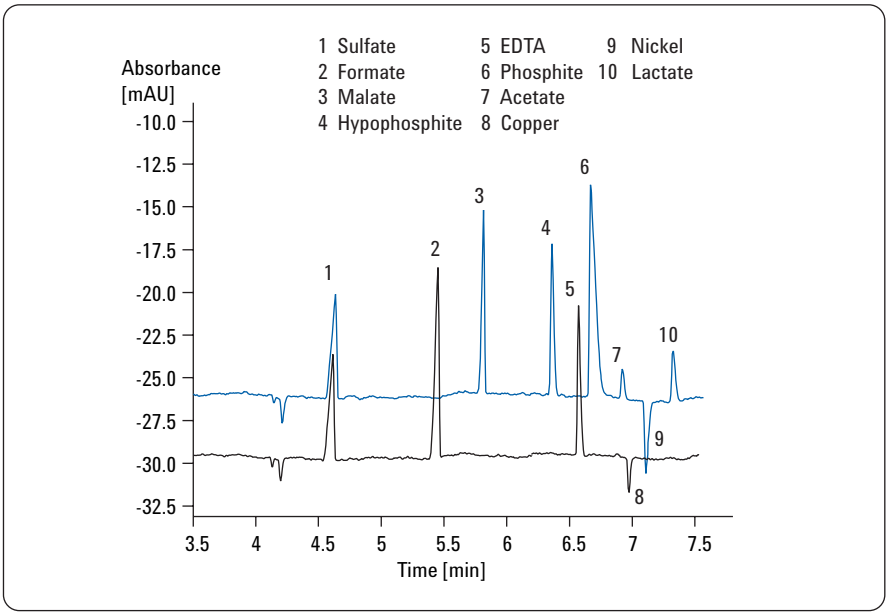

Figure 1
Analysis of electroless nickel- or copper-plating baths.

Chromatographic conditions

Sample:	Electroless nickel-and copper-plating bath, 1:500 diluted with water
Injection:	8 seconds at 50 mbar
Capillary:	Fused silica capillary, total length 80.5 cm, effective length 72 cm, internal diameter 50 µm (Agilent part number G1600-62211)
Buffer:	Agilent Plating Bath Analysis Buffer (Agilent part number 5064-8236)
Voltage:	-25 kV
Temperature:	20 °C
Detection:	Signal 350/20 nm, reference 275/10 nm

In the plating bath industry, the monitoring of additives in bath solutions or waste is essential for quality control, cost saving and environmental concerns. Electroless plating bath samples have presented a number of challenges to ion chromatography. CE, in contrast, allows a quick determination of all major components with only minor sample preparation.

www.agilent.com/chem/ce

Published November 1, 2008
Publication Number 5989-9812EN

Agilent Technologies