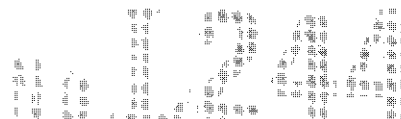


## HIGH PERFORMANCE LIQUID CHROMATOGRAPHY



## Analysis of Plating Solutions

Analysis of plating solution needs to be carried out periodically during the preparation of the plating solution or plating work for improving the stability and productivity of the product. In the plating solution, various substances are contained including not only the main metal element but also a complexing agent,

a buffer, or a reduction agent. Analysis of these compounds can be made with high accuracy and in a short time by using the HPLC. Shown here are analyses of cyanide, organic compounds and formaldehydes.

### ■ Analysis of Cyanide

Cyanide, which is contained in a plating solution of precious metal such as gold plating solution or silver plating solution, is an important target of analysis for not only quality control but the control of the plating solution. Cyanide can be analyzed by the cyanide analysis system using the post-column derivatization method. In this method, cyanide in a sample solution is separated by the column, and is derivatized into cyan chloride with chloramine-T, then it is reacted with a solution of pyridine-4-carboxylic acid and pyrazolone; absorbance of the blue solution thus obtained is subjected to measurement at 638nm.

Shown in Fig.1 and Fig.2 are examples of analyses for cyanide contained in a gold plating solution and silver plating solution.

Table 1 Analytical Conditions

(for separation)	
Column	: Shim-pack ANINO-Na (6mm I.D. × 100mmL.)
Mobile Phase	: 10mM Sodium Tartarate buffer (pH4.2)
Flow Rate	: 0.6mL/min.
Temperature	: 50°C
(for detection)	
<chlorination>	
Reagent	: 200mM Sodium Phosphate buffer containing 2mM Chloramine T (pH6.8)
Flow Rate	: 0.3mL/min.
Temperature	: 100°C
<coloring>	
Reagent	: A: 100mM 1-Phenyl-3-methyl-5-pyrazolone Ethanol Solution B: 20mM Pyridine-4-carboxylic Acid Aq. Solution A/B=1/3 (v/v)
Flow Rate	: 0.3mL/min.
Temperature	: 100°C
Detector	: SPD-10AV Wavelength 638nm

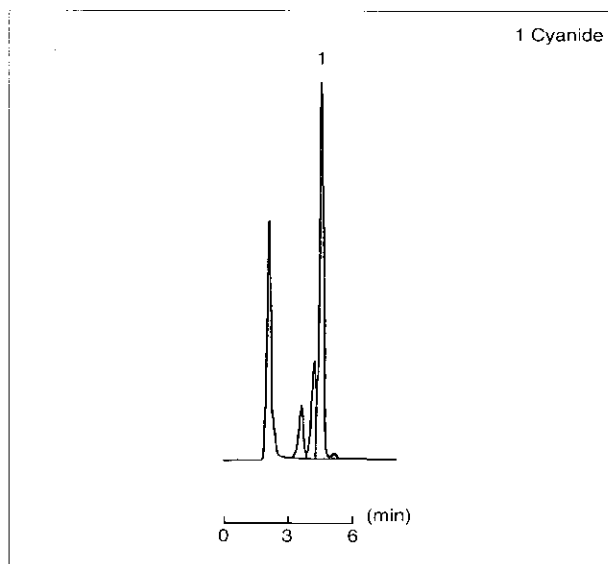


Fig.1 Chromatogram of Cyanide in Gold Plating Solution

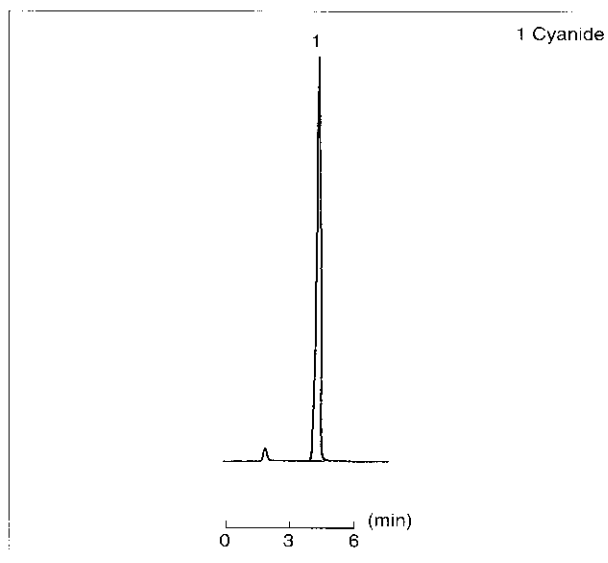


Fig.2 Chromatogram in Cyanide in Silver Plating Solution

## ■ Analysis of Organic Acids

Organic acids are added to the plating solution as a complexing agent or a buffer, and for the selective and high-sensitive analysis of these, an organic acid analysis system is applicable. Shown in Fig.3 is analysis of various organic acids in a nickel plating solution, and in Figure 4, analysis of citric acid.

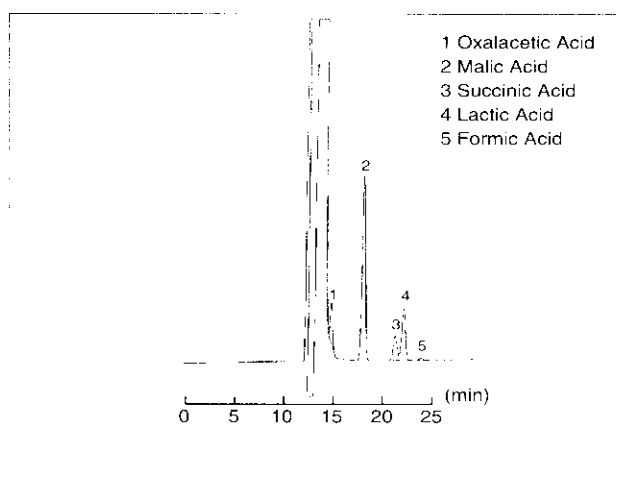


Fig.3 Chromatogram of Organic Acids in Nickel Plating solution

## ■ Analysis of Formaldehyde

Fig.5 shows an analysis of formaldehyde which is used as a reducing agent. The sample was derivatized with 2,4-DNPH (dinitrophenylhydrazine), separated with the column, and was subjected to absorbance measurement at 360nm. Using a copper pyrophosphate plating solution, derivatization was automatically performed through automatic sample injection by SIL-10A auto injector.

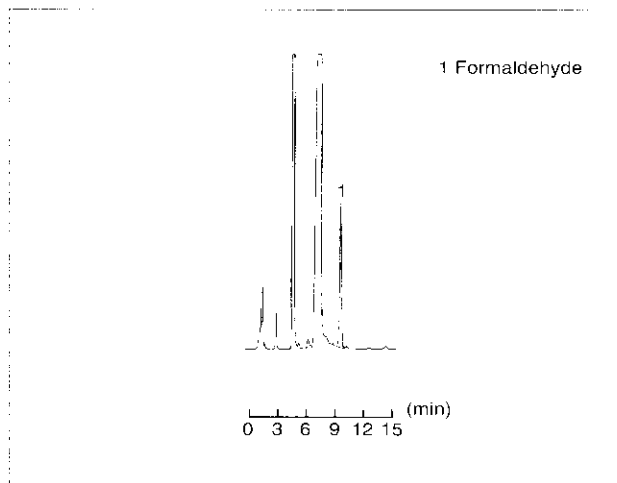


Fig. 5 Chromatogram of Formaldehyde in Copper Pyrophosphate Plating Solution

Table 2 Analytical Conditions

(for separation)	
Column	: Shim-pack SCR-102H (8mm I.D. × 300mmL.) × 2 (Fig.3) × 1 (Fig.4)
Guard Column	: Guard Column SCR-102H (6mm I.D. × 50mmL.)
Mobile Phase	: 5mM p-Toluensulfonic Acid
Flow Rate	: 0.8mL/min.
Temperature	: 40°C
(for detection)	
Reagent	: 5mM p-Toluensulfonic Acid, 20mM Bis-Tris and 100 μM EDTA · 2Na
Flow Rate	: 0.8mL/min.
Temperature	: 43°C
Detector	: CDD-6A

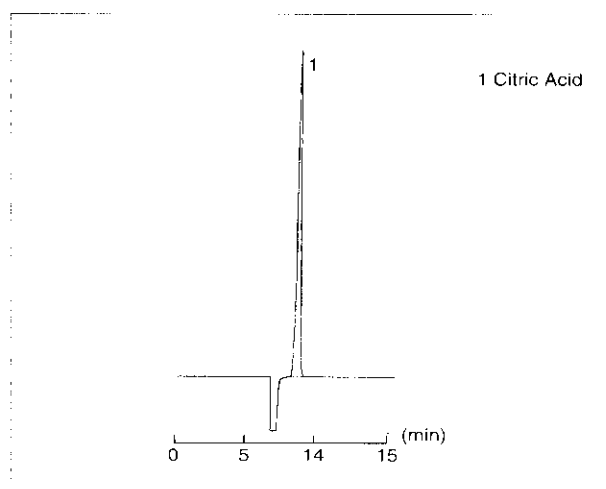


Fig.4 Chromatogram of Citric Acid in Gold Plating Solution

Table 3 Analytical Conditions

Column	: STR ODS-II (4.6mm I.D. × 150mmL.)
Mobile Phase	: Acetonitrile/Water=45/55 (v/v)
Flow Rate	: 0.8mL/min.
Temperature	: 40°C
Detector	: SPD-10A Wavelength 360nm

Table 4 Automatic Derivatization Procedure

Sample 300μL (*1)
Reagent 300μL (*2)
Mix
Wait 10min. under 30°C
Inject 20μL

(\*1) Sample was diluted to 1/10 with Water

(\*2) 2,4-DNPH/Acetonitrile (0.5mg/ml) solution containing 3% (v/v) Phosphoric Acid

\*Precious metal plating solutions;

By courtesy of Tokyo metropolitan Industry Technology Center.

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