Analysis of Lead and Cadmium in Electroless Nickel Plating Solution (Flame Method)

Heavy metals including lead (Pb) and cadmium (Cd) were traditionally used as the stabilizer in plating solutions. However, with the enforcement of regulations such as RoHS, most plating solutions are currently lead-free or heavy metal-free and Pb, Cd, etc. are controlled as impurities.

In the analysis of impurities such as Pb and Cd in nickel plating solution, the absorption line of the main component contained in high concentration (nickel) causes spectral interference when the deuterium lamp correction method is used. Therefore, the correction for the measured value is known to be difficult.

By using ZA3000 with the polarized Zeeman correction method, accurate quantitative analysis is possible as the background correction can be performed without being affected by the absorption line of nickel, the main component.

ZA3000 Atomic Absorption Spectrophotometer

Analysis of Lead in Electroless Nickel Plating Solution (Flame Method)

- ✓ Electroless nickel plating solutions (lead-free and with lead addition) diluted to four times with purified water were used as the analysis samples.
- ✓ In the addition recovery test, a good result was obtained with the recovery rate higher than 94%.

■ Analytical Conditions

Table 1 Analytical Conditions for Pb

Element	Pb	Atomizer	STD Burner
Instrument	ZA3000	Flame	Air-C2H2
Atomization	Flame	Fuel (C_2H_2)	2.0 L/min
Wavelength	283.3 nm	Oxidant(Air)	160 kPa 15.0 L/min
Lamp Current	7.5 mA		
Slit Width	1.3 nm	Burner Height	7.5 mm

Table 2 Measurement Parameters for Pb

Meas. Mode	Working Curve
Signal Mode	BKG Correction
Curve Order	Linear
Calculation	Integration
Time Constant	1.0 sec
Calculation Time	5.0 sec
Delay Time	5.0 sec

■ Analysis Result

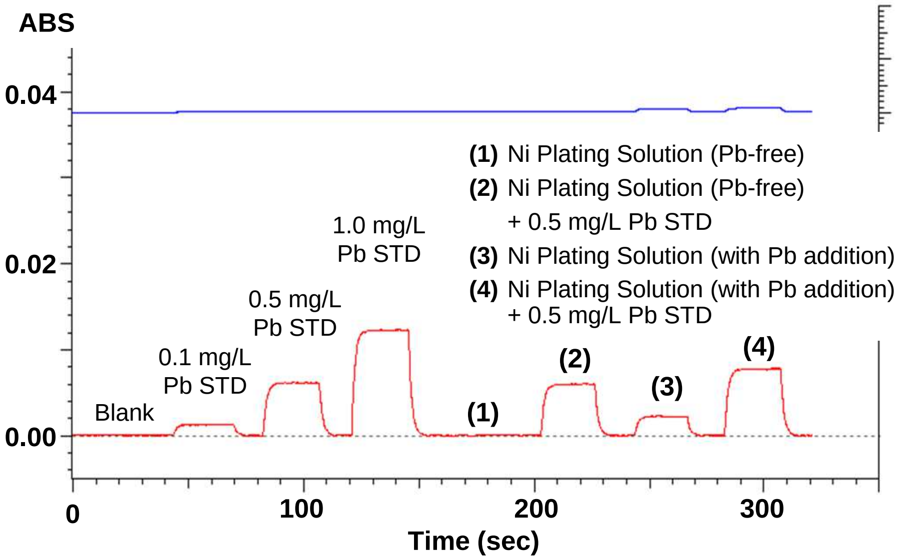

Figure 1 Atomic Absorption Signal Profile for Pb

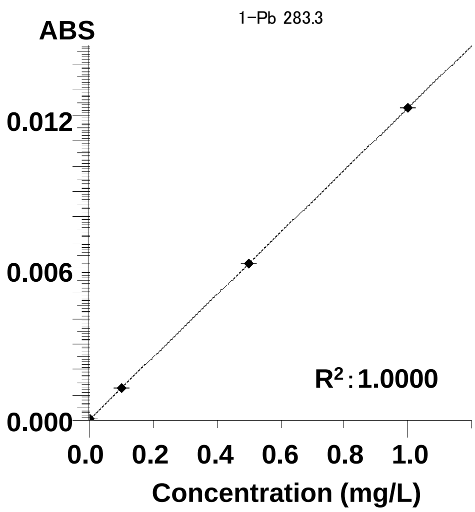

Figure 2 Calibration Curve of Pb

Table 3 Concentration and Absorbance of Each Sample

ID	Sample	Concentration (mg/L)	Absorbance
STD 1	Blank	0.00	0.00006
STD 2	0.1 mg/L Pb	0.10	0.00127
STD 3	0.5 mg/L Pb	0.50	0.00616
STD 4	1.0 mg/L Pb	1.00	0.01228
(1)	Pb-free Ni plating solution	N.D.(<0.02)*	0.00008
(2)	Pb-free Ni plating solution + Pb STD 0.5 mg/L	0.49	0.00600
(3)	Ni plating solution with Pb addition	0.17	0.00218
(4)	Ni plating solution with Pb addition + Pb STD 0.5 mg/L	0.64	0.00783

Table 4 Analysis Result of Pb in Electroless Ni Plating Solution

Sample	Analysis result (mg/L)	Addition concentration (mg/L)	Recovery rate (%)	Stock solution concentration (mg/L)
Pb-free electroless Ni plating solution	N.D. (<0.02)*	-	98	N.D. (<0.08)*
	0.49±0.01	0.5		
Electroless Ni plating solution with Pb addition	0.17±0.01	-	94	0.68±0.01
	0.64±0.01	0.5		

*: The lower limit of detection (3σ) calculated from the data obtained this time was 0.02 mg/L, indicating that the analysis result is less than 0.08 mg/L (0.02 × 4).

Analysis of Cadmium in Electroless Nickel Plating Solution (Flame Method)

✓Electroless nickel plating solutions (lead-free and with lead addition) diluted to four times with purified water were used as the analysis samples.

✓In the addition recovery test, a good result was obtained with the recovery rate higher than 93%.

■ Analytical Conditions

Table 5 Analytical Conditions for Cd

Element	Cd	Atomizer	STD Burner
Instrument	ZA3000	Flame	Air-C2H2
Atomization	Flame	Fuel(C_2H_2)	1.8 L/min
Wavelength	228.8 nm	Oxidant(Air)	160 kPa
Lamp Current	7.5 mA		15.0 L/min
Slit Width	1.3 nm	Burner Height	5.0 mm

Table 6 Measurement Parameters for Cd

Meas. Mode	Working Curve
Signal Mode	BKG Correction
Curve Order	Linear
Calculation	Integration
Time Constant	1.0 sec
Calculation Time	5.0 sec
Delay Time	5.0 sec

■ Analysis Result

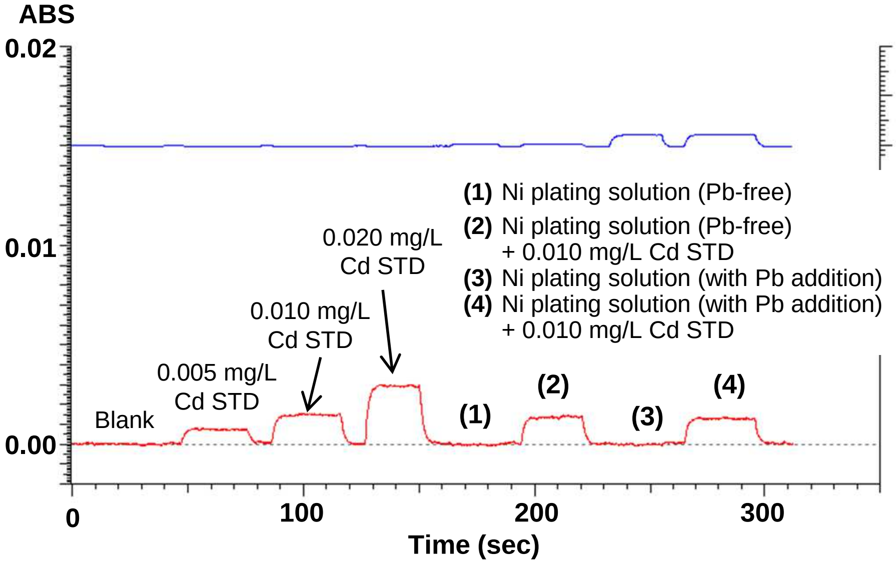

Figure 3 Atomic Absorption Signal Profile for Cd

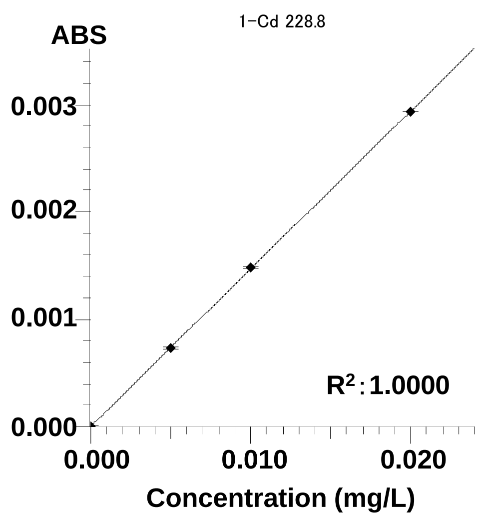

Figure 4 Calibration Curve of Cd

Table 7 Concentration and Absorbance of Each Sample

ID	Sample	Concentration (mg/L)	Absorbance
STD 1	Blank	0.000	0.00000
STD 2	0.005 mg/L Cd	0.005	0.00073
STD 3	0.010 mg/L Cd	0.010	0.00148
STD 4	0.020 mg/L Cd	0.020	0.00293
(1)	Pb-free Ni plating solution	N.D.(<0.001)**	-0.00001
(2)	Pb-free Ni plating solution + STD 0.01 mg/L	0.0096	0.00141
(3)	Ni plating solution with Pb addition	N.D.(<0.001)**	0.00001
(4)	Ni plating solution with Pb addition + STD 0.01 mg/L	0.0093	0.00137

Table 8 Analysis Result of Cd in Electroless Ni Plating Solution

Sample	Analysis Result (mg/L)	Addition Concentration (mg/L)	Recovery rate (%)	Stock Solution Concentration (mg/L)
Pb-free electroless Ni plating solution	N.D. (<0.001)**	-	96	N.D. (<0.004)**
	0.0096±0.0001	0.01		
Electroless Ni plating solution with Pb addition	N.D. (<0.001)**	-	93	N.D. (<0.004)**
	0.0093±0.0001	0.01		

**: The lower limit of detection (3σ) calculated from the data obtained this time was 0.001 mg/L, indicating that the analysis result is less than 0.004 mg/L (0.001 × 4).

[KEY WORDS]

Material/Processing Material Related, Industrial Chemicals, Nickel Plating Solution, Lead, Pb, Cadmium, Cd, Flame, AA, ZA3000, Material

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