Application Brief

Geochemistry, Mining and Metals

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Measurement of Multiple Heavy Metals in Plating Wastewater Using Flame AAS

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Complying with environmental standards

Metal plating is a process that uses electrochemical methods to treat material surfaces. As the process varies with the material to be plated, the pollutants in the liquid waste, and their concentrations, vary considerably. Plating wastewater is generally complex in composition, often containing elements such as Cr, Cd, Pb, Cu, Zn, and Ni.

Plating companies must comply with environmental standards for discharges from their facilities. These standards for wastewater include the Integrated Wastewater Discharge Standard (GB 8978-2002), Emission Standard of Pollutants for Electroplating (GB 21900-2008) and others. These standards require the concentration of these elements to be within the range of 0.1–2.0 mg/L. This concentration makes the analysis suitable for flame AAS or by ICP-OES. AAS instruments are popular due to their low capital cost and low running costs. However, analysts working in the plating industry or those in labs providing discharge sample testing, face a large number of samples containing varying concentrations of elements, complex test requirements and a heavy workload. Conventional AAS is slow as each sample must be measured multiple times, once for each element.

Figure 1. A SIPS accessory, fitted to the front of an Agilent AA 240FS flame atomic absorption spectrometer.

The Agilent AA 240FS flame atomic absorption spectrometer enables rapid sequential analysis of multiple elements in a single sample aspiration. The Agilent Sample Introduction Pump System (SIPS) accessory provides automation for flame AA analysis, increasing sample throughput, simplifying sample preparation and instrument operation. The accessory is available in a single pump version (SIPS 10) and a dual pump version (SIPS 20). The SIPS 20 enables automatic preparation of standard solutions from a single standard solution and auto dilution of over-range samples. The SIPS 20 also automates the injection of standard additions and spikes (for spike recovery measurements) and can add matrix modifiers and internal standards.

Analysis of four elements in a certified reference material

In this study, an Agilent 240FS instrument, fitted with a SIPS 20 accessory, was used to determine the concentrations of Cr, Cu, Ni and Zn in samples of a mixed water certified reference material 200931 (Institute of Reference Materials, China Ministry of Environmental Protection) and real plating bath waste water discharge samples. The samples were evaluated in accordance with the requirements of the measurement standards: Integrated Wastewater Discharge Standard (GB 8978-2002) and Emission Standard of Pollutants for Electroplating (GB 21900-2008). Table 1 summarizes the instrument settings used for the analysis.

Table 1. Instrument settings for the determination of the four elements.

Test item	Air flow (L/min)	Acetylene flow (L/min)	Characteristic wavelength (nm)	Slit width (nm)	Lamp current (mA)
Cr	13.5	3.3	357.9	0.2	7.0
Cu	13.5	2.0	324.8	0.5	4.0
Ni	13.5	2.1	232.0	0.2	4.0
Zn	13.5	2.0	213.9	1.0	5.0

The standards were prepared by the SIPS 20 accessory from one multielement stock solution and used to create a calibration curve for each element. All calibrations had excellent linearity, as shown in Figure 2 and the correlation coefficients in Table 2.

Table 2. Correlation coefficients for each calibration curve.

Element	Correlation Coefficient (r^2)
Cr	0.9999
Cu	0.9998
Ni	0.9996
Zn	1.0000

Sample analysis

The results from the analysis of the Mixed Water 200931 CRM as well as the plating bath waste water samples are shown in Table 3. The concentrations of the four elements were determined sequentially in a single aspiration, providing high analytical efficiency. The sample results showed excellent accuracy, with all four elements for the CRM recovered within ± 2%. The plating wastewater sample was measured, as received, for the four elements Cr, Cu, Ni and Zn. A spike recovery test (1.5 mg/L spike) on the sample was performed to ensure the accuracy of the method. Recovery rates for the plating wastewater samples were between 91-99% demonstrating the suitability of the 240FS and SIPS 20 when analyzing real wastewater samples. A precision of 0.45%–1.52%, met the QC requirements of the wastewater standard. The method showed many advantages such as fast analysis, excellent repeatability, and low cost.

Table 3. Sample measurement results.

	200931 CRM			Plating Wastewater Sample		
	Certified Value (mg/L)	Measured Value (mg/L)	Recovery	Measured Value (mg/L)	Measured Spike (mg/L)	Spike Recovery
Cr	0.590	0.582	99%	0.06	1.42	91%
Cu	0.591	0.596	101%	0.12	1.55	95%
Ni	0.681	0.679	100%	0.08	1.49	94%
Zn	0.297	0.302	98%	0.31	1.79	99%

Multiple analyses of the mixed water CRM were performed to investigate the precision over time. Figure 2 shows the short term precision of six measurements, with %RSDs for all elements between 0.5 and 1.5%, demonstrating excellent repeatability.

It took less than one minute to measure all four elements in a sample. The method showed many advantages; fast, high efficiency analysis, excellent repeatability and low cost, making the Agilent 240FS instrument, fitted with a SIPS accessory, ideal for monitoring of multiple heavy metal elements in plating wastewater.

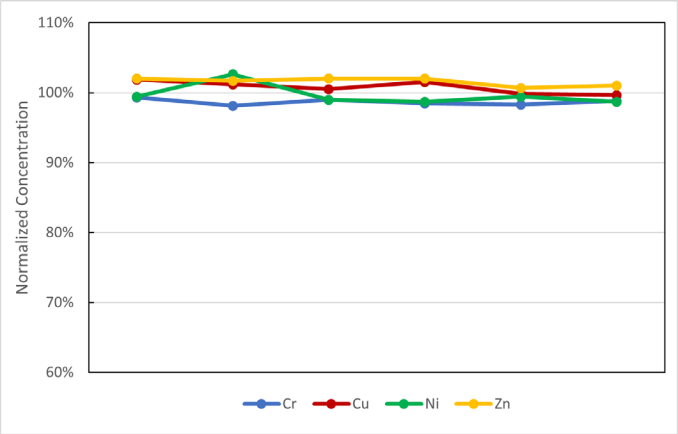

Figure 2. Short term precision of six measurements, with %RSDs for all elements between 0.5 and 1.5%.

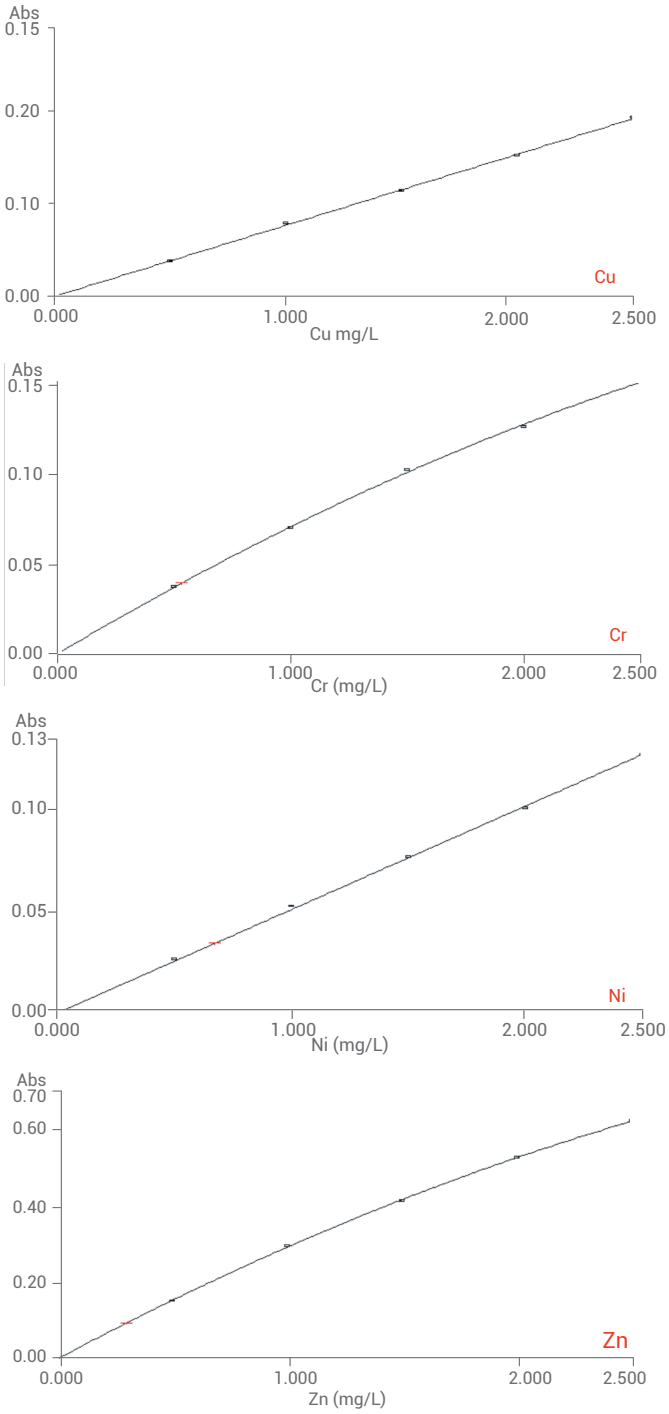

Figure 3. Standard curves for the four elements show excellent linearity (the highest concentration point of each element is 2.5 mg/L).

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