APPLICATION NOTE

ICP-Optical Emission Spectroscopy

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Analysis of Impurities in Nickel with the Avio 550 Max ICP-OES Following London Metal Exchange Guidelines

Introduction

Nickel (Ni) is one of the most widely used metals due to its corrosion resistance and strength at high and low temperatures. It is most frequently used in the production of steel but is also an important component in a variety of other alloys, as well as being commonly used in electronics, plating, and rechargeable batteries. These varied uses require nickel of different purities: in some applications, high-purity nickel is required, while for others, lower-grade nickel will suffice.

The London Metal Exchange (LME) issues specifications for a variety of purities for different metals. This work focuses on the analysis of contaminants in nickel with PerkinElmer's Avio® 550 Max ICP optical emission spectrometer (ICP-OES), using "Special Contract Rules for Primary Nickel"[1] as a guideline for the analytes and required concentrations.

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Experimental

Samples

All analyses were performed in 1% solutions of Ni to simulate digests diluted 100 times with 5% nitric acid (v/v). To check for accuracy, elemental spikes were added to the 1% Ni solution at the levels set in the "Special Contract Rules for Primary Nickel"[1] for ASTM Standard Specification for Nickel (99.80%). Analyses were performed against external calibration curves composed of standards prepared in 5% HNO_3 at 0.25, 0.5, and 1.0 ppm.

Instrumentation

All analyses were performed on the Avio 550 Max fully simultaneous ICP-OES using the parameters in Table 1 and the analytes and wavelengths in Table 2. The standard sample introduction configuration and parameters were used for all analyses, with the torch position set to -4. Combining the simultaneous analysis of the Avio 550 Max with the low argon consumption (9 L/min total) provides significant cost savings, when considering the cost of argon.

Results and Discussion

Table 3 shows the ASTM Standard Specification for Nickel at 99.80% weight percent[1], along with the concentrations in a 1% Ni solution (equivalent to a 100x dilution). A 1% Ni solution was spiked with the elements at the listed concentrations, with the recoveries appearing in Figure 1. All recoveries are within 10%, demonstrating the ability to accurately measure these elements at low concentration levels.

For Bi and Sn, there were significant spectral interferences from the Ni matrix. However, by applying a multicomponent spectral fitting (MSF) model[2] to these elements, the effects of the interferences were removed, allowing for accurate measurements at the required levels.

Table 1. Avio 550 Max ICP-OES Instrumental Parameters and Conditions.

Parameter	Value
Nebulizer	MEINHARD® K-1
Spray Chamber	Baffled glass cyclonic
RF Power	1500 W
Injector	2.0 mm ceramic
Plasma Gas Flow	8 L/min
Aux Gas Flow	0.2 L/min
Nebulizer Gas Flow	0.60 L/min
Torch Position	-4
Sample Uptake Rate	1.0 mL/min
Sample Uptake Tubing	Black/Black (0.76 mm id)
Internal Standard Tubing	Green/Orange (0.38 mm id)
Drain Tubing	Red/Red (1.14 mm id)
Replicates	3
Plasma View	Axial
Integration Time	5 sec (min and max)
Integration Range	0.5 – 2 sec

Table 2. Elements and Wavelengths.

Element	Wavelength (nm)
As	188.979
Bi	306.766
Co	238.892
Cu	324.752
Fe	238.204
Mn	259.372
P	178.221
Pb	283.306
S	181.975
Sb	206.836
Si	288.158
Sn	283.998
Zn	206.200
Sc (int std)	361.383

Table 3. London Metal Exchange Impurity Levels in 99.80% Ni.

Element	Specification (wt %)	Concentration in 1% Ni Solution (mg/L)
As	0.005	0.5
Bi	0.005	0.5
Co	0.15	15
Cu	0.02	2
Fe	0.02	2
Mn	0.005	0.5
P	0.005	0.5
Pb	0.005	0.5
S	0.01	1
Sb	0.005	0.5
Si	0.005	0.5
S	0.01	1
Sn	0.005	0.5
Zn	0.005	0.5

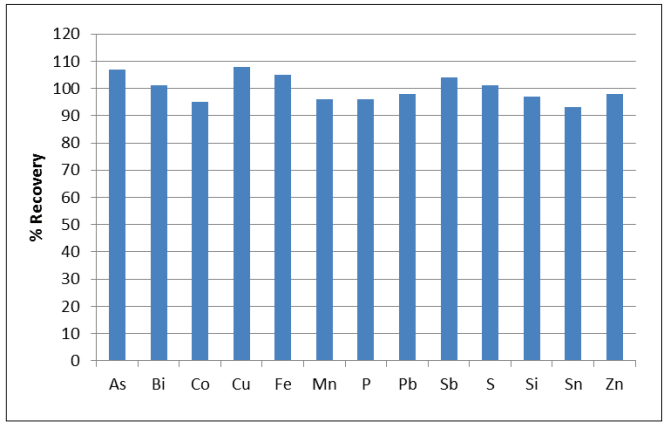

Figure 1. Recovery of analyte spikes in 1% Ni. Spike concentrations are shown in Table 3.

The detection limits in solution were determined as three times the standard deviation of seven consecutive measurements of 1% Ni. Figure 2 displays the resulting detection limits along with the specification limits, accounting for the 100x dilution.

With the accuracy established, the stability was assessed by monitoring the internal standard signal over a six-hour analysis of 1% Ni. The results are plotted in Figure 3 and show a variation of less than 5% when normalized to the first reading, demonstrating the stability of the methodology.

Conclusions

This work has demonstrated the ability of PerkinElmer's Avio 550 Max fully simultaneous ICP-OES to analyze solutions of 1% nickel for elements at the levels specified by the London Metal Exchange. Spectral interferences from high matrix samples were overcome through the use of MSF, allowing all of the defined elements to be measured at their specified concentrations.

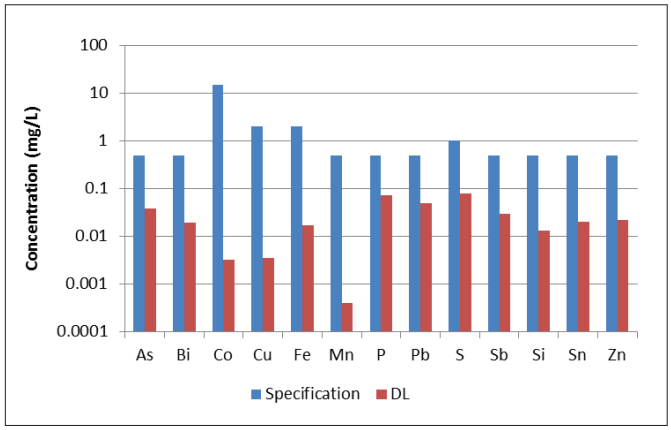

Figure 2. Detection limits in 1% Ni, along with the specification limits for 99.80% Ni, accounting for 100x dilution.

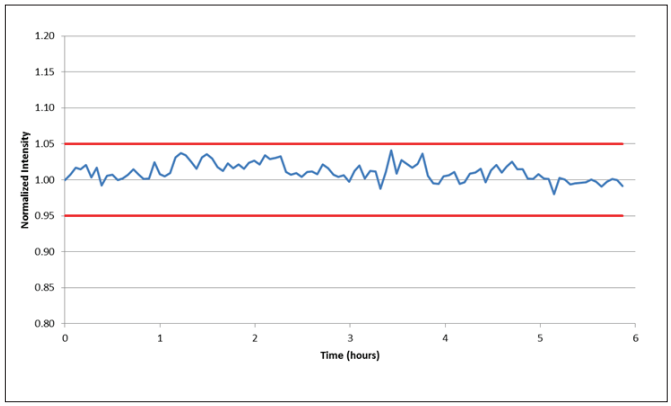

Figure 3. Internal standard (Sc) stability over a 6-hour analysis of 1% Ni solution. All data is normalized to the first reading.

References

1. "Special Contract Rules for Primary Nickel", The London Metal Exchange.
2. "Multicomponent Spectral Fitting"", Technical Note, PerkinElmer, 2016.

Consumables Used

Component	Part Number
Sample Uptake Tubing, Black/Black (0.76 mm id), PVC	N0777043 (flared) 09908587 (non-flared)
Drain Tubing, Red/Red (1.14 mm id), PVC	09908585
Internal Standard Tubing, Orange/Green (0.38 mm id), PVC	N0773111 (flared)
Antimony Standard, 1000 mg/L	N9300207 (125 mL) N9300101 (500 mL)
Arsenic Standard, 1000 mg/L	N9300180 (125 mL) N9300102 (500 mL)
Bismuth Standard, 1000 mg/L	N9303761 (125 mL) N9300105 (500 mL)
Cobalt Standard, 1000 mg/L	N9303766 (125 mL) N9300113 (500 mL)
Copper Standard, 1000 mg/L	N9300183 (125 mL) N9300114 (500 mL)
Iron Standard, 1000 mg/L	N9303771 (125 mL) N9300126 (500 mL)
Lead Standard, 1000 mg/L	N9300175 (125 mL) N9300128 (500 mL)
Manganese Standard, 1000 mg/L	N9303783 (125 mL) N9300132 (500 mL)
Phosphorus Standard, 1000 mg/L	N9303788 (125 mL) N9300139 (500 mL)
Scandium Standard, 1000 mg/L	N9303798 (125 mL) N9300148 (500 mL)
Silicon Standard, 1000 mg/L	N9303799 (125 mL) N9300150 (500 mL)
Sulfur Standard, 1000 mg/L	N9303796 (125 mL) N9300154 (500 mL)
Tin Standard, 1000 mg/L	N9303801 (125 mL) N9300161 (500 mL)
Zinc Standard, 1000 mg/L	N9300178 (125 mL) N9300168 (500 mL)
Autosampler Tubes	B0193233 (15 mL) B0193234 (50 mL)

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