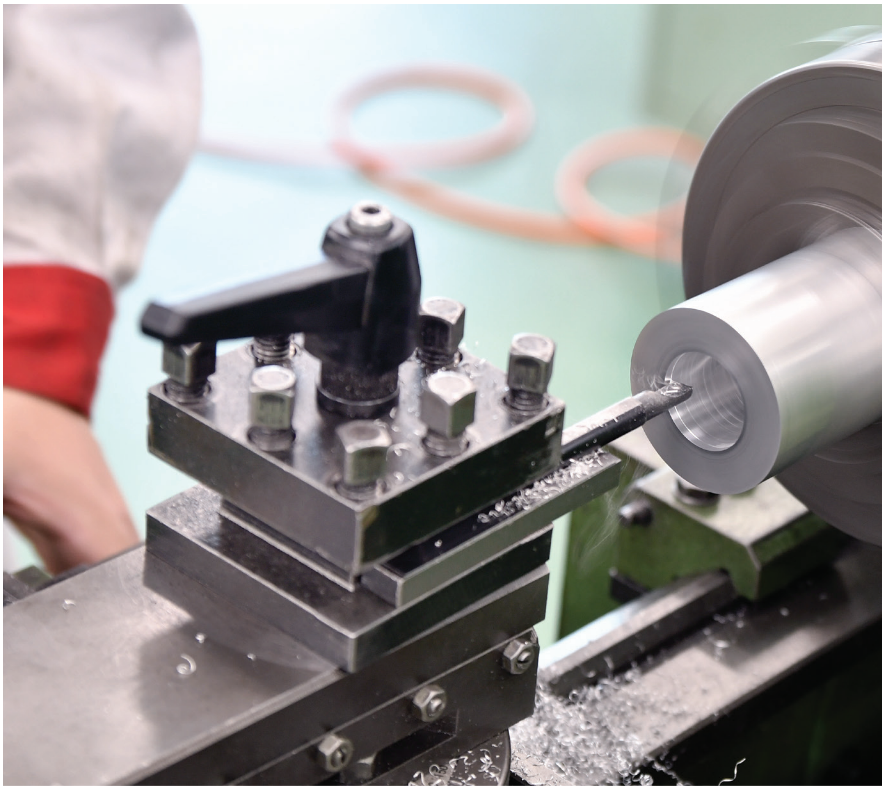

APPLICATION NOTE

ICP-Optical Emission Spectroscopy

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Analysis of Impurities in Aluminum with the Avio 220 Max ICP-OES Following London Metal Exchange Guidelines

Introduction

Aluminum (Al) is a common metal used in a wide variety of applications and products, including, but not limited to, transportation, construction, electrical, and consumer products. This varied and popular use is due to Al's unique characteristics: lightweight, strong, resistant to corrosion, non-toxic, high thermal and electrical conductivity, as well as easily machined, formed, and drawn. This versatility is further increased by combining Al with other metals to create aluminum-based alloys. In addition, Al can easily be recycled without losing any of its desirable properties.

Because of its popularity, there are many grades of Al available. The London Metal Exchange (LME) lists specifications for two commonly-used Al purities: 99.5% and 99.7%[1], with each having up to six inorganic impurities specified. ICP-OES is the best technique for measuring these impurities at these levels due to its ability to easily handle high-matrix samples and simplicity of operation.

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PerkinElmer's Avio® 220 Max hybrid simultaneous ICP-OES is the ideal choice for this application. Its optical design provides excellent light throughput, which is important for measuring lower concentrations with shorter analytical times.[2] The Avio 220 Max ICP-OES also uses a CCD detector providing simultaneous background and analyte measurement, which is important for samples such as metal matrices.[3]

This work describes the analysis of impurities in Al at the LME specifications using the Avio 220 Max ICP-OES.

Experimental

Sample Preparation

All analyses were performed in 1% solutions of Al to simulate digests diluted 100x with 5% nitric acid (v/v). To check for accuracy, elemental spikes were added to the 1% Al solution at the levels set in the "Special Contract Rules for High Grade Primary Aluminum", as shown in Table 1. Calibration standards were prepared in 1% Al solutions at 0.5, 2, and 8 ppm to overcome matrix effects on the plasma; 5% HNO_3 was used as the blank. Scandium (Sc) was added as an internal standard to all standards and samples.

Instrumentation

All analyses were performed on an Avio 220 Max ICP-OES using the parameters in Table 2 and wavelengths in Table 3. The standard sample introduction components were used, along with a torch position of -3. The wavelengths selected are interference-free. Due to the high sensitivity of the Avio 220 Max, all analytes were measured with a read time of 0.2 seconds, resulting in an analytical time of one minute per sample. In addition, the sensitivity allowed all reading to be accomplished in radial mode, thereby minimizing matrix effects caused by the Al and simplifying the analysis.

Results and Discussion

For accuracy determination, 1% Al solutions were spiked at the specification levels shown in Table 1 and measured. Figure 1 shows that all recoveries are within 5% of their true values, demonstrating the accuracy of the methodology. With the proper selection of wavelengths and analytical conditions, a 1% Al matrix does not inhibit impurity analysis.

Table 2. Avio 220 Max ICP-OES Instrumental Parameters.

Parameter	Value
Nebulizer	MEINHARD® K-1
Spray Chamber	Baffled glass cyclonic
RF Power	1500 W
Injector	2.0 mm ceramic
Plasma Flow	8 L/min
Auxiliary Flow	0.2 L/min
Nebulizer Flow	0.65 L/min
Torch Position	-3
Sample Uptake Rate	1 mL/min
Replicates	2
Plasma View	Radial
Read Time	0.2 sec

Table 3. Elements and Wavelengths.

Element	Wavelength (nm)
Fe	238.206
Si	251.614
Ga	417.207
Zn	206.200
V	292.466
Mg	285.214
Cu	327.393
Sc (Int Std)	361.383

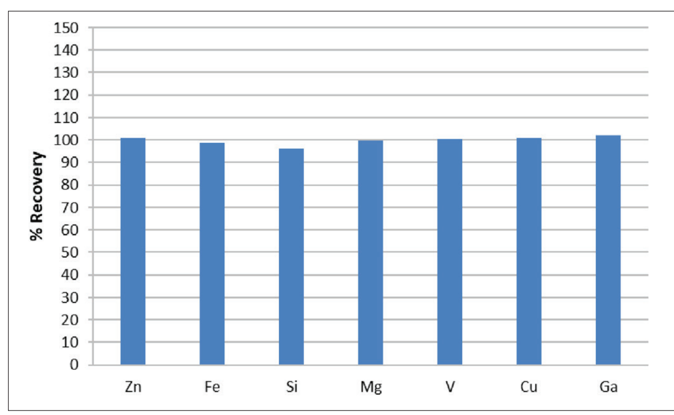

Figure 1. Analyte recoveries in 1% aluminum at the concentrations specified in LME's Special Contract Rules for High Grade Purity Aluminum.

Table 1. LME Special Contract Rules for High Grade Primary Aluminum and Associated Concentrations in Solution with 100x Dilution.

	P1020A: International Designations and Chemical Composition Limits for Unalloyed Aluminum		**GB/T 1196-2008:** Unalloyed Aluminum Ingots for Remelting	
Element	**Specification (wt %)**	**Concentration in 1% Solution (mg/L)**	**Specification (wt %)**	**Concentration in 1% Solution (mg/L)**
Fe	0.20	20	0.20	20
Si	0.10	10	0.10	10
Ga	0.04	4	0.03	3
Zn	0.03	3	0.03	3
V	0.03	3	---	---
Mg	---	---	0.02	2
Cu	---	---	0.01	1

With the accuracy established, the stability was assessed by measuring 1% aluminum samples over four hours with a one-minute rinse between samples and monitoring the internal standard signal. The resulting stability appears in Figure 2 and shows signal variations of less than 4%, demonstrating the excellent stability of the system in the presence of a high-matrix sample.

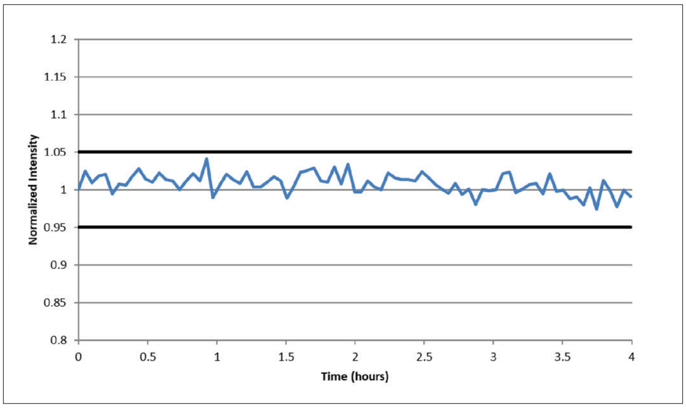

Figure 2. Internal standard (Sc) stability over a four-hour analysis of 1% Al.

Finally, detection limits were determined by performing seven consecutive measurements of a 1% Al solution and multiplying the standard deviation by 3.14. As shown in Figure 3, the detection limits are below the specified levels, providing confidence that the specified concentrations can be easily measured.

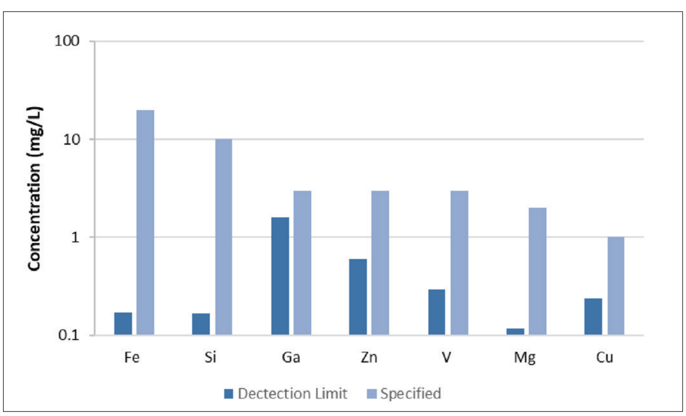

Figure 3. Detection limits in 1% Al (corrected for 100x dilution) compared to specification limits.

Conclusion

This work demonstrates the ability of the Avio 220 Max hybrid simultaneous ICP-OES, with simultaneous background measurement, to accurately determine impurities in aluminum, as specified in the London Metal Exchange. The required detection limits were easily met using short read times and a total analysis time of only one minute per sample. The methodology proved to be stable over four hours (the maximum time evaluated) due to instrument design considerations which prevent sample build-up on the interface.

Consumables Used

Component	Part Number
Sample Uptake Tubing: Black/Black (0.76 mm id) PVC, Flared	N0777043
Drain Tubing: Gray/Gray (1.30 mm id), Santoprene	N0777444
Aluminum Standard, 10,000 µg/mL	N9304111 (125 mL) N9304110 (500 mL)
Gallium Standard, 10,000 µg/mL	N9303772 (125 mL) N9300119 (500 mL)
Iron Standard, 1000 µg/mL	N9303771 (125 mL) N9300126 (500 mL)
Magnesium Standard, 1000 µg/mL	N9300179 (125 mL) N9300131 (500 mL)
Scandium Standard, 1000 µg/mL	N9303798 (125 mL) N9300148 (500 mL)
Silicon Standard, 1000 µg/mL	N9303799 (125 mL) N9300150 (500 mL)
Zinc Standard, 1000 µg/mL	N9300178 (125 mL) N9300168 (500 mL)
Autosampler Tubes, case of 500	B0193233 (15 mL) B0193234 (50 mL)

References

1. "Special Contract Rules for High Grade Primary Aluminum", The London Metal Exchange.
2. "The Avio 220 Max ICP-OES: A Unique Double-Monochromator Optical System", Technical Note, PerkinElmer, 2020.
3. "Avio 220 Max ICP-OES Custom-Designed Solid-State Detector with Hybrid Simultaneous Analysis", Technical Note, PerkinElmer, 2021.

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