APPLICATION NOTE

ICP-Optical Emission Spectrosco

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Analysis of Copper-Gold Ore with the Avio 220 Max ICP-OES

Introduction

The extraction of metals from ore at a mining plant requires the routine analysis of various samples. These include the feed (crushed ore), the tails (waste ore after processing) and the concentrate (final product). For a copper-gold mine, the metals that require analysis can include, not just copper (Cu) and gold (Au), but also silver (Ag) if present at significant levels. Other elements that can be associated with the ore, including iron (Fe) and arsenic (As), may also need to be measured.

Flame atomic absorption spectrometers (AAS) are often used by mining laboratories to perform this analysis. Flame AAS is a relatively simple, robust, low-cost technique. However, it has several limitations including being slower for analysis of multiple elements, having a narrow linear range which necessitates frequent dilutions, as well as poor detection limits for some elements, such as arsenic.

In this work, reference materials were analyzed for copper, iron, silver and arsenic using PerkinElmer's Avio® 220 Max hybrid simultaneous ICP-OES. The Avio 220 Max is ideal for laboratories looking to improve productivity for analyses usually performed with flame AAS. The instrument's dual view plasma enables the analysis of both trace and major elements in the same method, while its unique double-monochromator optical system with CCD detector provides excellent detection limits. In addition, the Avio 220 Max ICP-OES can be switched off completely when not being used (saving power and argon), with a startup time of only 10 minutes.

Experimental

Sample Preparation

Six certified reference materials (CRMs) were analyzed: GBM305-4 and GBM313-3 (Geostats Pty. Ltd. Western Australia, Australia) and OREAS 501B, 502B, 503B and 504B (Ore Research & Exploration, Pty. Ltd., Victoria, Australia). These represent feed ore samples at a copper-gold mine. The CRMs were digested using aqua-regia (made from analytical reagent-grade acids) in an SPB sample preparation block (PerkinElmer Inc, Shelton, Connecticut USA). For each sample, 0.4 g was weighed into a 50 mL polypropylene test tube, followed by the addition of 5 mL of analytical-grade nitric acid and 15 mL hydrochloric acid. Each tube was gently swirled and allowed to sit for 10 minutes before being placed in the sample preparation block set to a temperature of 110 oC, covered with polypropylene watch glass, and allowed to digest for one hour. The tubes were then removed, and the samples allowed to cool for 30 minutes before diluting to the 50 mL mark with deionized (DI) water, capping and shaking. An internal standard was not used in this work, although it could be to further improve data quality.

Calibration Standards

Calibration standards were prepared in 30% v/v hydrochloric acid to match the acid content of the prepared samples. The concentrations are shown in Table 1. For the calibration blank, an undigested acid blank (i.e. nitric and hydrochloric acid) was used.

Table 1. Calibration Standards (mg/L)

Element	Standard 1	Standard 2	Standard 3	Standard 4
Silver	0.2	0.4	0.8	1.2
Arsenic	10	20	30	40
Copper	5	10	50	100
Iron	50	100	150	200

Instrument and Method Parameters

Analysis was performed using an Avio 220 Max hybrid simultaneous ICP-OES using the conditions in Table 2. A PEEK low-flow Gemcone nebulizer was used due to its high resistance to blockages, which may occur from both high dissolved solids and any residual particulates in the digested samples. The RF power was set to 1500 watts, the plasma gas to 10 L/min and the nebulizer gas to 0.60 L/min. The lower plasma gas flow is a benefit of the Avio Max's patented Flat Plate™ plasma technology. Plus, the incorporation of PlasmaShear™ prevents sample deposition on the interface optic, eliminating the need for cleaning and performing maintenance on the interface region. This is especially important when running high-matrix samples, such as ores.

Due to the high sensitivity of the Avio 220 Max ICP-OES, only short read times of 1-2 seconds were used for each element. The total analysis time per sample was two minutes, which includes uptake, read delay and a 30-second autosampler wash.

All instrument control and data acquisition was done with Syngistix™ for ICP software.

Table 2. Avio 220 Max ICP-OES Sample Introduction and Instrument Conditions

Parameter	Value
Nebulizer	PEEK Low-flow Gemcone
Spray Chamber	Baffled Glass Cyclonic 1-
Torch	slot Quartz 2.0 mm ID
Injector	Alumina 1-2 seconds (2
Read Time	replicates) 1500 watts 10
RF Power	L/min 0.2 L/min 0.6
Plasma Gas Flow	L/min
Auxiliary Gas Flow	
Nebulizer Gas Flow	

The wavelengths and plasma views used are shown in Table 3. Axial mode provides the ability to measure lower concentrations and was, therefore, chosen for silver and arsenic, while radial mode provides a higher linear calibration range and was used for copper and iron.

Table 3. Wavelengths and Plasma View

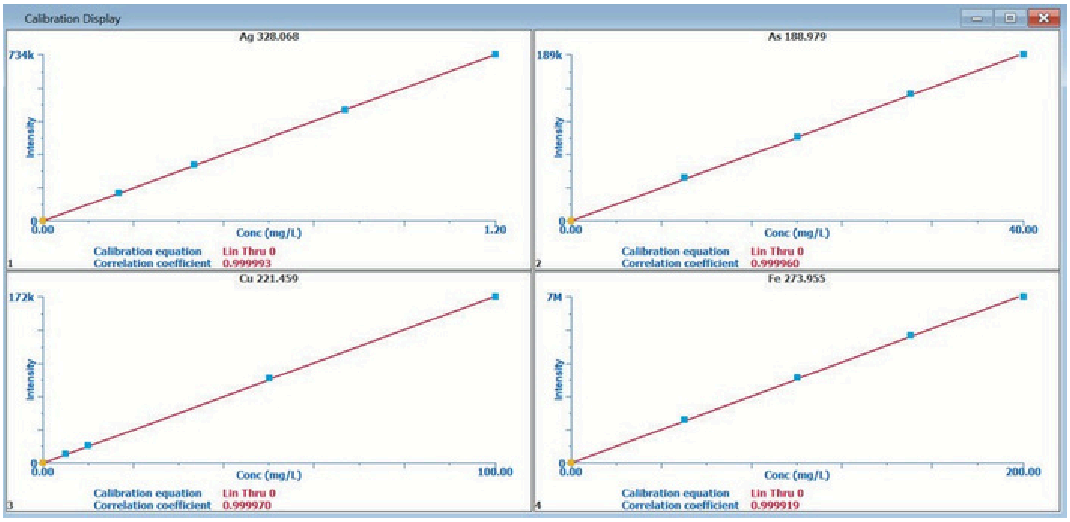
Element	Wavelength	Plasma View
Silver	Ag 328.068	Axial
Arsenic	As 188.979	Axial
Copper	Cu 221.459	Radial
Iron	Fe 273.955	Radial

Results and Discussion

The calibration graphs are shown in Figure 1. All wavelengths had excellent linearity with correlation coefficients > 0.9999 using a linear-through-zero equation.

An emission spectrum for GBM313-3 is shown in Figure 2, where the grey bars represent the pixels on the CCD detector used for the analyte peak measurement, and the green crosses are the background correction (BGC) points. Due to the superior sensitivity of the Avio 220 Max ICP-OES, a clear peak is obtained for 0.031 mg/L Ag at Ag 328.068 nm. Silver also has potential spectral interferences from adjacent wavelengths given the high concentrations of iron, copper and titanium, but, as seen in the silver spectrum, these elements do not interfere with silver due to the excellent resolution of the Avio 220 Max.

Figure 3 displays the results of silver, arsenic, copper, and iron from the six GBM and OREAS reference materials analyzed. All results are well within the uncertainty limits. Copper ranged from 0.25% to 1.05% (wt%). The results for silver were between 0.7 and 4.1 ppm and showed that the interferences from high Fe, up to 7% in sample, were not significant. The arsenic results were also within the expected limits at both the low concentrations in OREAS (10-20 ppm) and at the higher levels present in the GBM CRM samples.



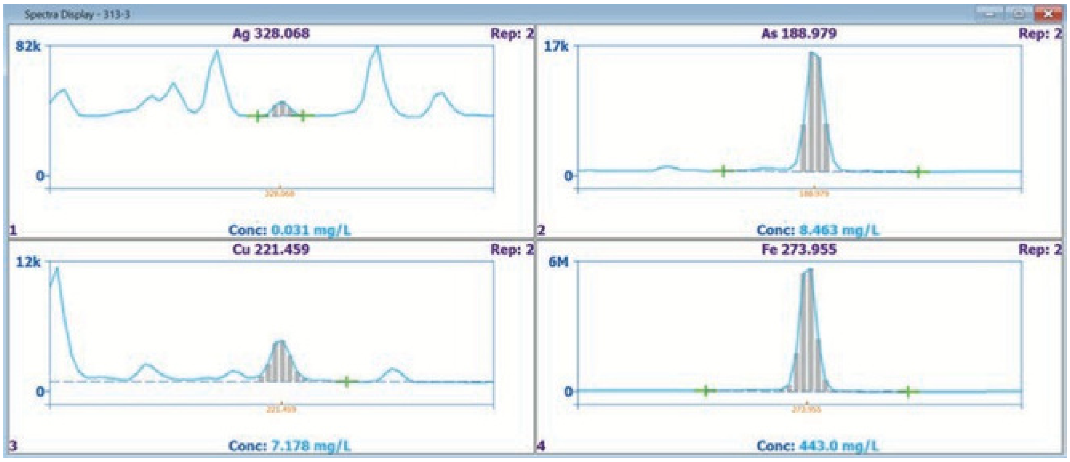
Ag 328.068 — Calibration equation: Lin Thru 0; Correlation coefficient: 0.999993

As 188.979 — Calibration equation: Lin Thru 0; Correlation coefficient: 0.999960

Cu 221.459 — Calibration equation: Lin Thru 0; Correlation coefficient: 0.999970

Fe 273.955 — Calibration equation: Lin Thru 0; Correlation coefficient: 0.999919

Figure 1. Calibration plots for Ag, As, Cu, Fe.



Ag 328.068 — Conc: 0.031 mg/L

As 188.979 — Conc: 8.463 mg/L

Cu 221.459 — Conc: 7.178 mg/L

Fe 273.955 — Conc: 443.0 mg/L

Figure 2. GBM313-3 Spectra for silver, arsenic, copper, and iron.

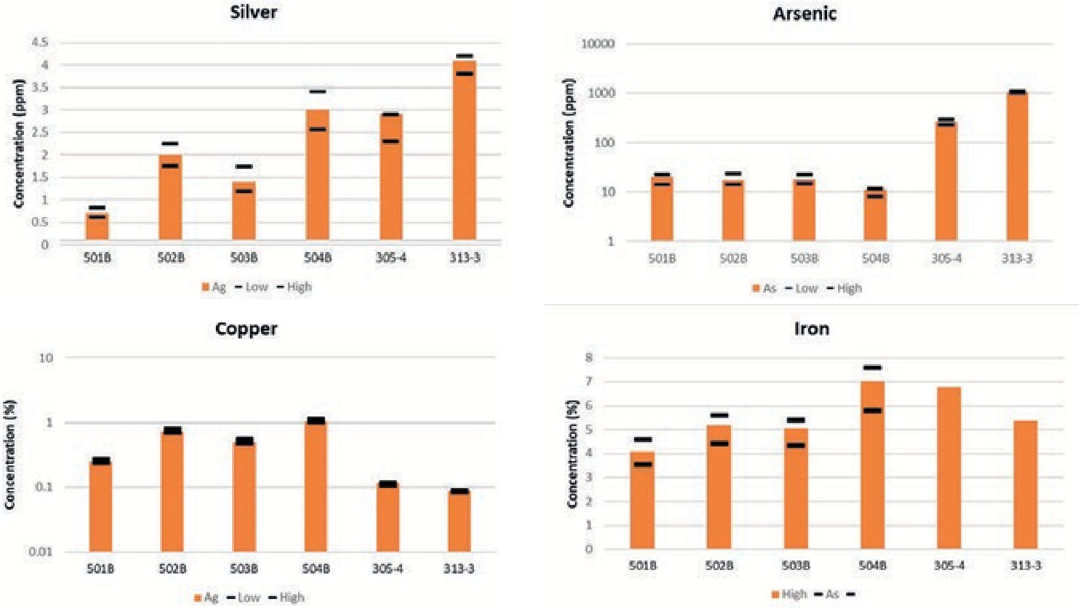
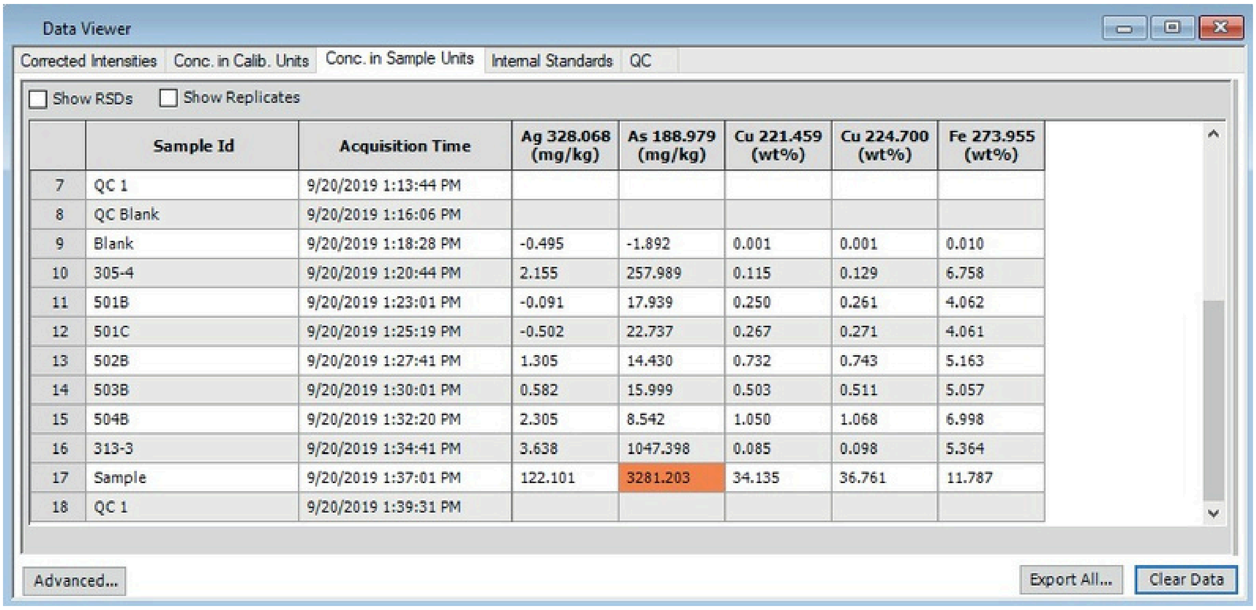


Figure 3. Silver, arsenic, copper, and iron results in certified reference materials. The black bars represent the certificate tolerance limits for each reference material. Iron was not certified in GBM 305-4 and GBM 313-3.

Copper mines also need to monitor impurities that can be associated with the copper concentrate that is produced. Elements such as arsenic are considered "penalty elements" by the smelters which refine the concentrate into pure copper. At levels above 1000 mg/kg arsenic, the smelter may charge extra to process the concentrate. Syngistix software makes it easy to see, at a glance, if any elements are outside of a user-defined range. As shown in Figure 4, Syngistix automatically highlighted the sample which contained more than 1000 mg/kg of arsenic, giving a quick visual indication that this sample may not meet the required specification.



	Sample Id	Acquisition Time	Ag 328.068 (mg/kg)	As 188.979 (mg/kg)	Cu 221.459 (wt%)	Cu 224.700 (wt%)	Fe 273.955 (wt%)
7	QC 1	9/20/2019 1:13:44 PM					
8	QC Blank	9/20/2019 1:16:06 PM					
9	Blank	9/20/2019 1:18:28 PM	-0.495	-1.892	0.001	0.001	0.010
10	305-4	9/20/2019 1:20:44 PM	2.155	257.989	0.115	0.129	6.758
11	501B	9/20/2019 1:23:01 PM	-0.091	17.939	0.250	0.261	4.062
12	501C	9/20/2019 1:25:19 PM	-0.502	22.737	0.267	0.271	4.061
13	502B	9/20/2019 1:27:41 PM	1.305	14.430	0.732	0.743	5.163
14	503B	9/20/2019 1:30:01 PM	0.582	15.999	0.503	0.511	5.057
15	504B	9/20/2019 1:32:20 PM	2.305	8.542	1.050	1.068	6.998
16	313-3	9/20/2019 1:34:41 PM	3.638	1047.398	0.085	0.098	5.364
17	Sample	9/20/2019 1:37:01 PM	122.101	3281.203	34.135	36.761	11.787
18	QC 1	9/20/2019 1:39:31 PM					

Figure 4. Data Viewer in Syngistix software, automatically highlighting a sample which contains arsenic above a user-defined limit.

Conclusion

This work has shown the analytical-speed advantage of the Avio 220 Max hybrid simultaneous ICP-OES for multi-element analysis compared to the traditional flame AA technique, which can only measure one element at a time. Major and trace elements can be analyzed in the same method using dual view with an analysis time of only two minutes per sample for copper, iron, arsenic and silver. The excellent sensitivity of the Avio 220 Max is ideally suited for the accurate measurement of difficult elements at low levels. Plus, the Avio 220 Max ICP-OES has low operating costs due to the Flat Plate plasma technology and the ability to switch the instrument off when not analyzing samples, given its best-in-class startup time (10 minutes from cold start).

Consumables Used

Component	Part Number
Sample Uptake Tubing, Black/Black (0.76 mm id), PVC	09908587 09908585
Drain Tubing, Red/Red (1.14 mm id), PVC	N0770358 B0193222
Low-Flow GemCone Nebulizer	(15 mL)
Autosampler Tubes	B0193234 (50 mL) N9300171 (125 mL) N9300151 (500 mL)
Pure-Grade Silver Standard, 1000 mg/L	N9300180 (125 mL) N9300102 (500 mL)
Pure-Grade Arsenic Standard, 1000 mg/L	N9300183 (125 mL) N9300114 (500 mL)
Pure-Grade Copper Standard, 1000 mg/L	N9303171 (125 mL) N9300126 (500 mL)
Pure-Grade Iron Standard, 1000 mg/L	

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