Determination of major and minor elements in geological samples using the 4100 MP-AES

Application note

Geochemistry, metals and mining

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Introduction

Establishing the elemental composition and grade of base metal ores is carried out at different stages of mining and processing. It is used in advanced exploration to confirm the feasibility of mining the ore, and in the assay of the ore concentrate. The determination of the concentration of base metals requires a technique with good accuracy and precision that is capable of generating and managing data from the analysis of large numbers of samples. Consequently, there are also very high expectations in terms of sample throughput and reliability.

In addition, ore-grade samples contain a range of concentrations. It would not be unusual to see high percentage level analytes present alongside low parts per million level analytes. A sample with 40% copper would not be unexpected, and as such, any analysis technique used needs to be able to cope with this range of concentrations.

Typically, the determination of major and minor elements in geological samples is performed using flame atomic absorption spectroscopy (FAAS) and/or inductively coupled plasma optical emission spectroscopy (ICP-OES). Agilent has developed a new instrument, the 4100 Microwave Plasma Atomic Emission Spectrometer (MP-AES), that out-performs FAAS in terms of analytical performance, and offers lower operating costs and unattended overnight operation. The 4100 MP-AES is a highly automated multi-element system with high matrix tolerance and high sample throughput. This ensures that challenging samples can be analyzed quickly and reliably with little operator training and minimal method development.

Experimental

Instrumentation

The 4100 MP-AES is a fast sequential multi-element analytical technique that uses a microwave-induced plasma to provide analysis of liquid sample using a conventional sample introduction system. As MP-AES relies on the generation of a microwave plasma using nitrogen, no flammable gases such as acetylene are required. This reduces running costs and improves lab safety. Nitrogen can be supplied from bottled gas or the Agilent 4107 Nitrogen Generator. This alleviates the difficulty and costs in sourcing gases such as acetylene, specially in remote locations.

The potential of the Agilent 4100 MP-AES for the analysis of ore-grade base metal samples was investigated following preparation of the samples by a four-acid digestion procedure.

Samples and sample preparation

Ore-grade samples require different digestion techniques to geochemical exploration samples in order to dissolve the high metal concentrations and retain them in solution. As a result, dilution factors are greater than with geochemical exploration samples, resulting in higher detection limits, and also higher upper limits.

The preparation method used involves taking 0.4 g of sample and performing a four-acid (HNO_3-$HClO_4$-HF-HCl) digestion. The process is completed by further addition of hydrochloric acid and deionized water, followed by cooling to room temperature. The resulting solution was diluted to 100 mL with de-ionized water resulting in a final matrix of 30% HCl. This represents a 250-fold sample dilution.

The 4100 MP-AES operating parameters were optimized as shown in Table 1.

Table 1. Agilent 4100 MP-AES operating parameters

Instrument parameter	Setting
Nebulizer pressure	80–240 kPa
Read time	3 s (10 s for Ag)
Stabilization time	15 s
Background correction	Auto

Results and discussion

Method detection limits (MDLs)

Method detection limits (MDLs) for silver, copper, molybdenum, nickel, lead, and zinc were determined. Table 2 shows that excellent MDLs can be obtained using the 4100 MP-AES.

Table 2. MDLs for digestions of ore-grade base metal samples

Analyte	Wavelength (nm)	MDL (ppm)
Ag	328.068	0.04
Cu	327.395	0.1
Mo	379.398	0.7
Ni	352.453	0.4
Pb	405.781	0.4
Zn	213.857	0.7

Quantitative analysis

A batch of base metal ore sample digests were prepared using the sample preparation method. These sample were analysed by the 4100 MP-AES using a blank and three matrix-matched standard solutions.

Accuracy

The results obtained by the 4100 MP-AES for Ag, Cu, Mo, Ni, Pb and Zn were plotted against the expected results — see Figures 1 to 7. The graphs show good correlation (accuracy) over the calibration range, up to ~4% for copper, indicating that MP-AES is a suitable technique for the application.

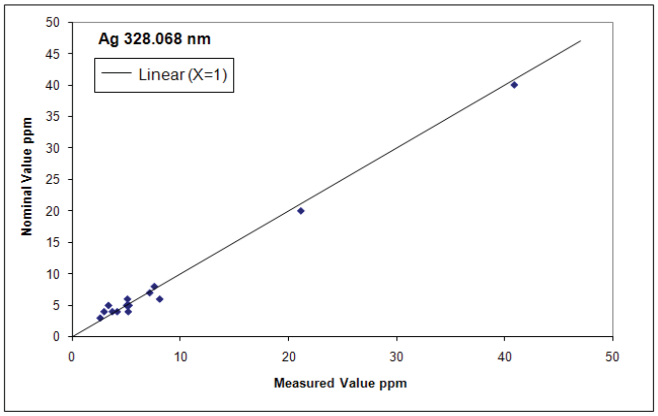

Figure 1. MP-AES measured results for Ag compared to nominal values

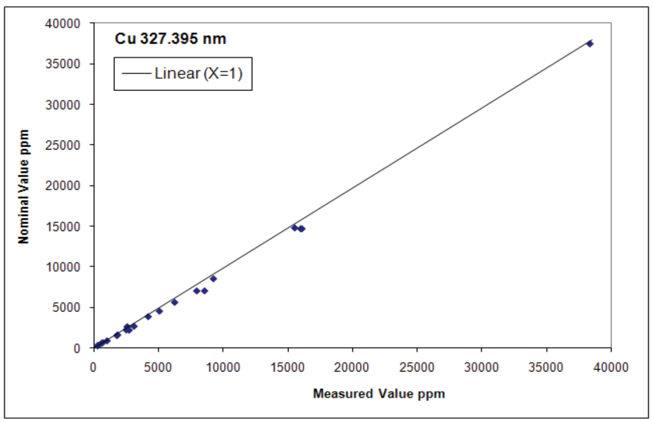

Figure 2. MP-AES measured results for Cu compared to nominal values

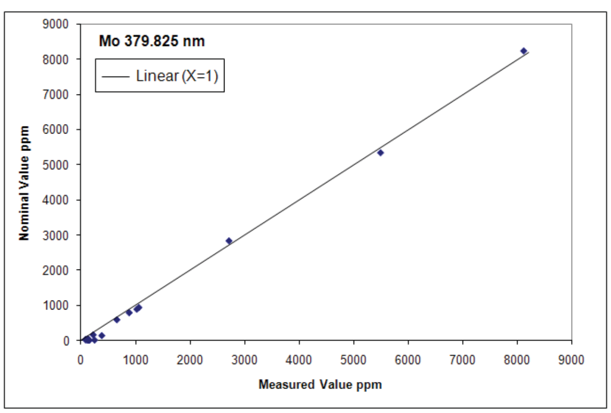

Figure 3. MP-AES measured results for Mo compared to nominal values

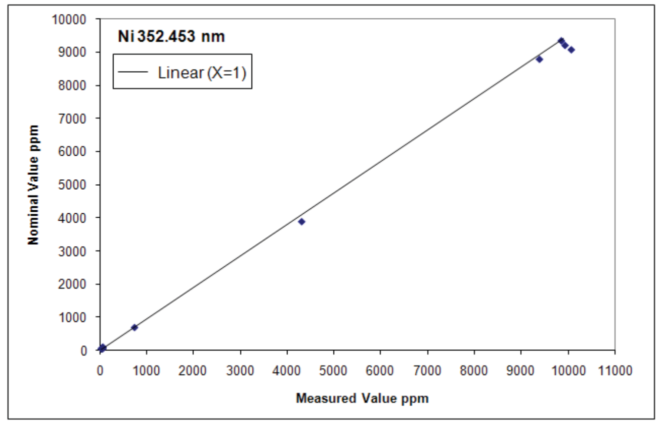

Figure 4. MP-AES measured results for Ni compared to nominal values

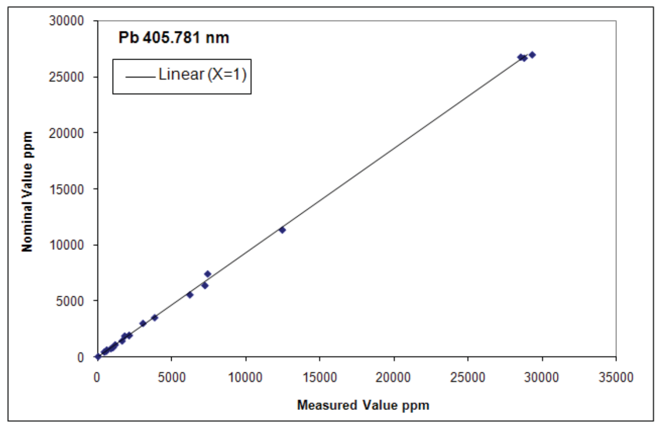

Figure 5. MP-AES measured results for Pb compared to nominal values

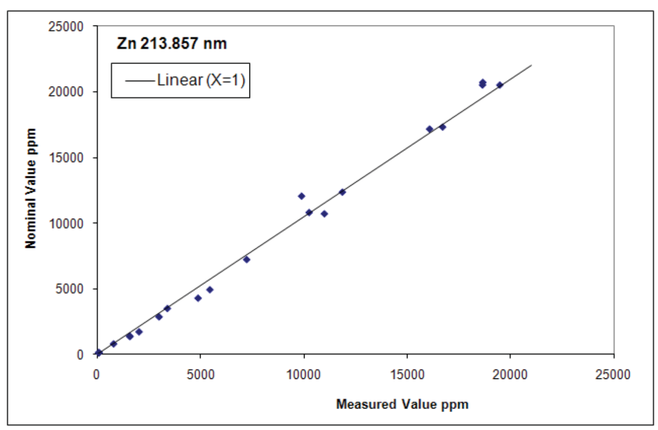

Figure 6. MP-AES measured results for Zn at the 213.857 nm wavelength compared to nominal values

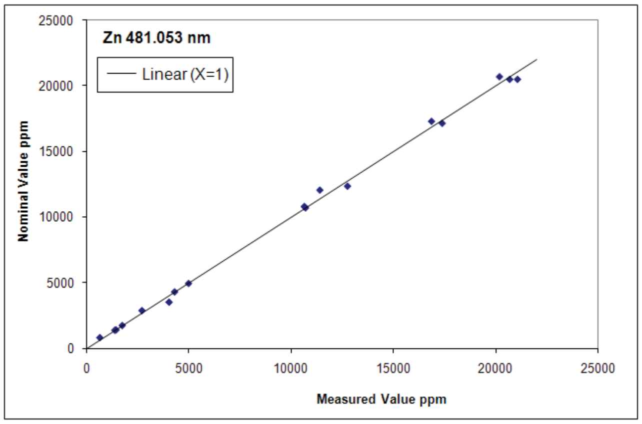

Figure 7. MP-AES measured results for Zn at the 481.053 nm wavelength compared to nominal values

Conclusions

A series of geological samples has been successfully analyzed using the Agilent 4100 MP-AES, following a sample preparation procedure that is commonly used to prepare base metal ores for analysis by geochemical laboratories. Quantitative results were obtained for 6 elements present in a batch of 23 base metal ore samples, with measured concentrations ranging from less than 1 ppm to 29%. The comparative results for Ag, Cu, Mo, Ni, Pb and Zn obtained using MP-AES show that the 4100 MP-AES is well suited to this application. It also offers additional benefits over comparative techniques such as flame AAS, through lower operating costs, improved safety and the ability to perform the analysis using unattended overnight operation.

Operating costs can be further reduced by directly generating nitrogen from compressed air using an Agilent 4107 Nitrogen Generator. This is particularly advantageous in areas where gas supplies are expensive, difficult to obtain or pose logistics challenges in transporting bulk cylinders to site. The 4100 MP-AES also offers the possibility of installations in remote locations. This enables laboratories to analyze samples locally at the source rather than transporting the samples to a central laboratory for analysis, as is the current practice. Furthermore, the Agilent 4100 MP-AES has the lowest operating costs of comparable techniques such as flame AA, and by using non-flammable gases, removes safety concerns associated with acetylene and nitrous oxide.

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