New methodology for determination of gold and precious metals using the Agilent 4100 MP-AES

Application note

Geochemistry, metals and mining

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Introduction

The accurate and precise determination of gold and other precious metals (PMs) is a vital task for companies involved in PM production and the contract or service laboratories that support them. This might involve the processing of hundreds of samples typical of exploration and mining activities, or supplying the quality control assay of the final product from PM producers or recyclers. PMs are also produced as a by-product of copper or nickel processing.

Today's analysts have access to a range of elemental analytical techniques including flame and graphite furnace atomic absorption spectroscopy (AA), inductively coupled plasma optical emission spectroscopy (ICP-OES) and ICP mass spectroscopy (ICP-MS). The choice of method will depend on a number of factors, for example, the technology that is available, the skill of the operator, sample type, cost of analysis, sample throughput requirements and the objective of the analytical result.

Agilent has identified an increased need for multi-element determinations over a wide dynamic range at an attractive overall cost of analysis, taking into account instrument supplies, consumables, power and labor, and has developed a new instrument to meet this need.

The Agilent 4100 Microwave Plasma-Atomic Emission Spectrometer (MP-AES) (Figure 1) is a low-cost, highly automated technique that is suitable for the trace analysis of PMs in samples typically found in mining and exploration activities. 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, especially in remote locations.

Figure 1. Agilent 4100 MP-AES

This application note describes the analysis of PM samples prepared by fire assay using the Agilent 4100 MP-AES.

Experimental

Instrumentation

The Agilent 4100 MP-AES is a fast sequential multi-element analytical technique that has a microwave-induced nitrogen plasma at its heart. As a result running costs are significantly reduced as only nitrogen is required for plasma operation.

The 4100 MP-AES uses Agilent's unique Microwave Excitation Assembly to create a concentrated axial magnetic field around a conventional torch. This focuses and contains the microwave energy where it is needed to produce a toroidal plasma with a cooler central channel that is suitable for stable introduction of liquid samples using a conventional sample introduction system.

Samples and sample preparation

A series of samples that are normally analyzed by flame AA were prepared using fire assay. A 30 g rock sample was heated with flux to over 1,000 °C. The process yields a small silver sphere, which was then dissolved in 4 mL of 25% aqua regia. The 4100 MP-AES operating parameters were then optimized, as shown in Table 1.

Table 1. Agilent 4100 MP-AES operating parameters

Instrument parameter	Setting
Analytes (wavelength)	Au (267.595), Pt (265.945), Pd (363.470)
Nebulizer pressure	140–240 kPa
Read time	3 s
No. of replicates	3
Sample uptake delay	10 s
Stabilization time	5 s
Background correction	Auto

Method detection limits

Method Detection Limits (MDLs) for gold, platinum and palladium were determined by measuring two sets of ten method blanks twice, on non-consecutive days, using the conditions as defined in the analytical method. The MDL was calculated as the 3 sigma standard deviation of the twenty concentration results.

The MDLs listed in Table 2 are sufficiently low for this type of analysis.

Table 2. MDLs for Au, Pt and Pd in fire assay samples

Analyte	Wavelength (nm)	MDL (µg/L)
Au	267.595	4
Pt	265.945	13
Pd	363.470	0.7

Linear range

The concentration or working range of an analytical technique is the range of concentrations that can be measured accurately without the need to recalibrate or dilute the sample. The linear range for Au, Pt and Pd was investigated using the 4100 MP-AES. A series of standards was prepared at concentrations of 2, 7, 70, 90, 100, 110 and 120 mg/L, and analyzed using the 4100 MP-AES. The calibration graphs obtained are shown in Figures 2, 3 and 4. These show that the linearity for all three analytes was acceptable up to a concentration of 120 mg/L, which exceeds the requirements of the application.

Sample volume

The analysis of PMs is volume-sensitive. The typical total sample volume available for analysis is around 4 mL. By using an Agilent SPS 3 Sample Preparation System connected to the 4100 MP-AES, the method cycle time (sample to sample) was 55 s and the sample volume consumed during analysis was 1.8 mL.

Accuracy

In order to test the ability of the 4100 MP-AES to analyze PMs at variable concentrations, a batch of Certified Reference Materials (CRMs) was analyzed.

Figure 2 Calibration graph for Au at the 267.595 nm wavelength

Figure 3. Calibration graph for Pt at the 265.945 nm wavelength

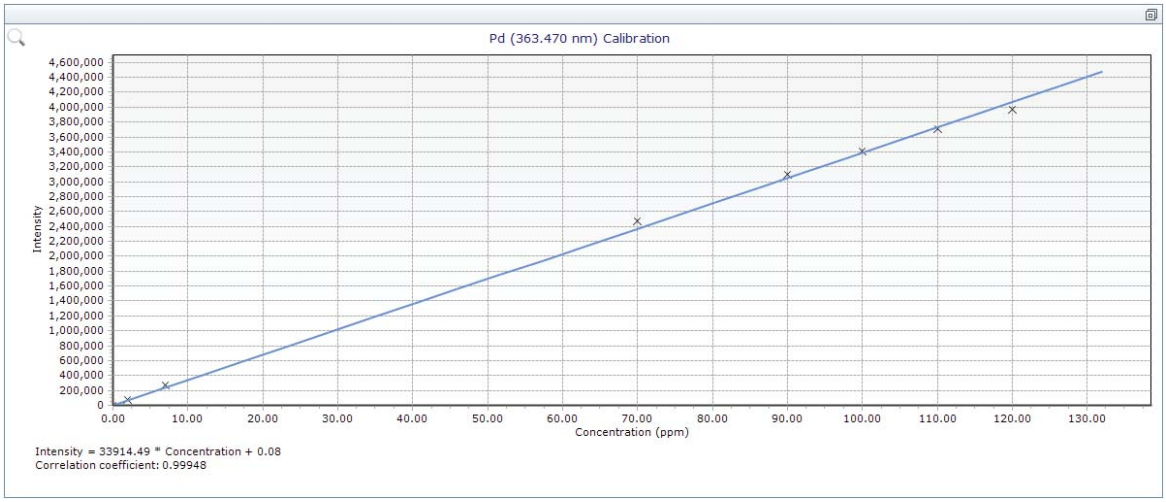

Figure 4. Calibration graph for Pd at the 363.469 nm wavelength

These were custom CRMs that had been professionally prepared from solid ore samples and certified through a round robin test process. They are not commercially available. The results listed in Table 4 show excellent agreement (accuracy) between the 4100 MP-AES measured results and the certified values.

Table 4. Results for Au, Pt and Pd obtained using the 4100 MP-AES compared to certified reference values

Gold	CRM certified value (mg/L)	MP-AES result (mg/L)
CRM 1	19.8	19.3
CRM 2	7.9	7.4
CRM 3	23.1	22.7
CRM 4	5.6	5.7
CRM 5	57.8	55.3
CRM 6	3.1	3.3
CRM 7	35.9	35.4
CRM 8	8.4	8.9

Platinum	CRM certified value (mg/L)	MP-AES result (mg/L)
CRM 6	0.74	0.75
CRM 7	35.6	35.9
CRM 8	9.0	9.5

Palladium	CRM certified value (mg/L)	MP-AES result (mg/L)
CRM 6	3.21	3.4
CRM 7	44.4	44.0
CRM 8	35.0	36.5

Recovery of unknown samples

A batch of unknown samples was analyzed for gold content using the 4100 MP-AES, and the results compared with the data obtained from analysis using conventional flame AA. The comparison can be seen in Table 5.

A typical spectrum of a sample containing approximately 40 ppm of gold can be seen in Figure 5. This demonstrates excellent signal to noise ratio with the flat baseline and the narrow emission peak confirming there are no spectral interferences.

Table 5. Results for gold in unknown samples, comparing the 4100 MP-AES with flame AA

Sample	MP-AES result (mg/L)	Flame AA result (mg/L)	Agreement with AA result (%)
1	0.09	0.09	100
2	0.85	0.84	101
3	5.3	5.1	104
4	13.7	14.4	95
5	20.8	21.8	95
6	4.3	4.1	105
7	1.0	1.0	100

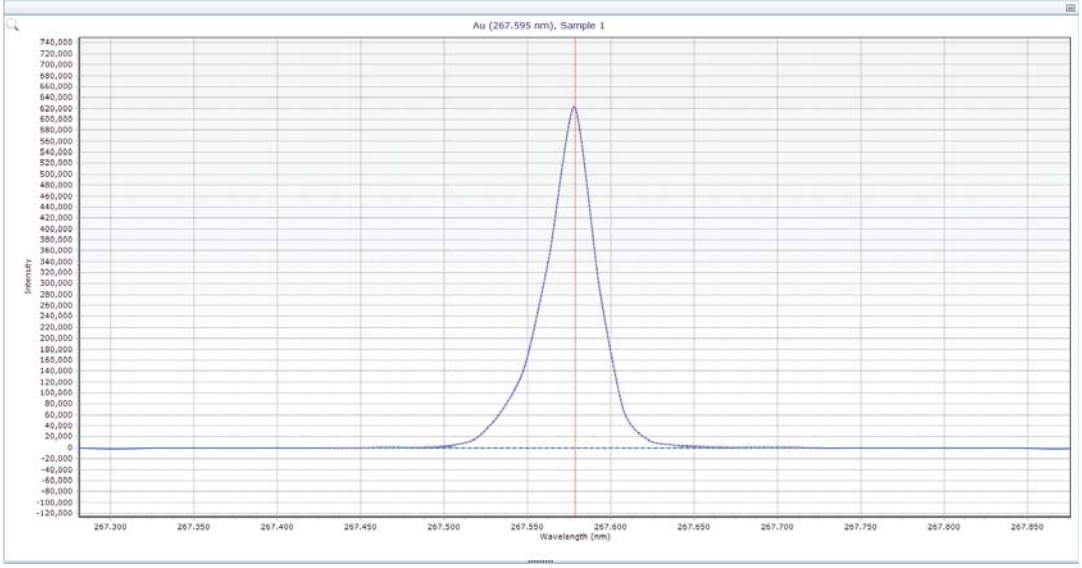

Figure 5. A typical PM sample spectrum for Au at the 267.595 nm wavelength

Conclusions

Following a thorough evaluation of the performance of the Agilent 4100 MP-AES, it is apparent that the 4100 MP-AES offers many advantages for the analysis of gold and other precious metals compared to conventional analysis techniques such as flame AA. The 4100 MP-AES offers superior sensitivity, an increased linear dynamic range and improved speed of analysis. In addition, the 4100 MP-AES more than doubles the measurement speed of conventional AA systems. It also offers exceptional accuracy as demonstrated by the excellent agreement with the certified values for several CRMs and real samples. Furthermore, the 4100 MP-AES has the lowest running costs of all of today's atomic spectroscopy techniques, due to reduced gas costs. Its use of non-combustible nitrogen also ensures that, unlike flame AA, the 4100 MP-AES can provide safer, multi-element unattended overnight operation.

The 4100 MP-AES also offers the option of installation at remote locations. This enables laboratories to analyze samples at the source rather than shipping the samples to a central laboratory for analysis, as is the current practice. In other instances where remote analysis is already performed using flame AA, the MP-AES provides the user with capability to analyze the samples in a safer environment, without the need for flammable gases such as acetylene.

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