APPLICATION NOTE

Atomic Absorption

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The Analysis of Precious Metals in Mining with the PinAAcle 500 Atomic Absorption Spectrometer

Introduction

When mining for precious metals, ores are extracted from the ground and subjected to various sample preparation procedures in order to remove the metals of interest. A commonly used procedure to isolate metals from the ore is fire-assay, which leaves a matrix-free "button" of the metal. This button is then dissolved in the appropriate acids and analyzed. By knowing the amount of sample used in the sample preparation and the analytical results, the concentration of the metals in the ground can be determined. These analyses are typically done with flame atomic absorption (AA) spectrometry due to its low cost, analytical speed, simplicity, and robustness. This work will focus on the analysis of precious metals in simulated digested precious metal buttons, with an added emphasis on assessing the lowest limits which can be accurately measured.

Experimental

All analyses were performed on the PerkinElmer PinAAcle™ 500 AA spectrometer operating with 10 cm burner head, according to the conditions in Table 1. All samples and standards were introduced manually using self-aspiration through the high-sensitivity nebulizer with the spacer removed. The gas flow rates were optimized to give the highest sensitivity and a steady signal. For all analyses, a three-second integration time and three replicates were used.

Standards were prepared in either 2% HNO_3 (copper, silver) or 15% aqua regia (gold, palladium, platinum) to simulate the dissolution of a button sample after fire-assay sample preparation.

Results and Discussion

The ability to accurately measure low concentrations was assessed by establishing low-level calibration curves with regression values > 0.999. The lowest standards were those whose absorptions were greater than the blank and gave relative standard deviations (RSDs) < 5%. For every element, lower concentrations could be measured, but the RSDs were greater than 5%, a result of statistics when considering small numbers. Table 2 shows the calibration standards, with the resulting calibration curves appearing in Figure 1. To assess the accuracy of the measurements, a standard at the mid-point of each calibration curve was measured, with typical results shown in Table 3.

Table 1. PinAAcle 500 AA spectrometer instrumental conditions.

Parameter	Gold (Au)	Palladium (Pd)	Platinum (Pt)	Copper (Cu)	Silver (Ag)
Wavelength (nm)	242.80	244.79	265.94	324.75	328.07
Slit (nm)	0.7	0.2	0.7	0.7	0.7
Lamp	HCL	HCL	HCL	HCL	HCL
Air Flow (L/min)	4.40	4.40	4.40	4.40	7.80
Acetylene Flow (L/min)	1.58	2.02	2.02	1.86	2.02

Table 2. Low-level calibrations.

Element	Calibration Standards (µg/L)	Calibration Type
Au	50, 60, 70, 80 50, 60,	Linear Through
Pd	70, 80 500, 750,	Zero Linear
Pt	1000, 1250 10, 20,	Through Zero
Cu	30, 40 5, 10 20, 30	Linear Through
Ag		Zero Linear
		Through Zero
		Linear Through
		Zero

Table 3. Mid-level standard quantitative read back.

Element	Standard (µg/L)	Read-Back (µg/L)	% Recovery
Au	65	67.7	104
Pd	65	69.2	106
Pt	850	836	98
Cu	25	24.1	96
Ag	15	14.5	97

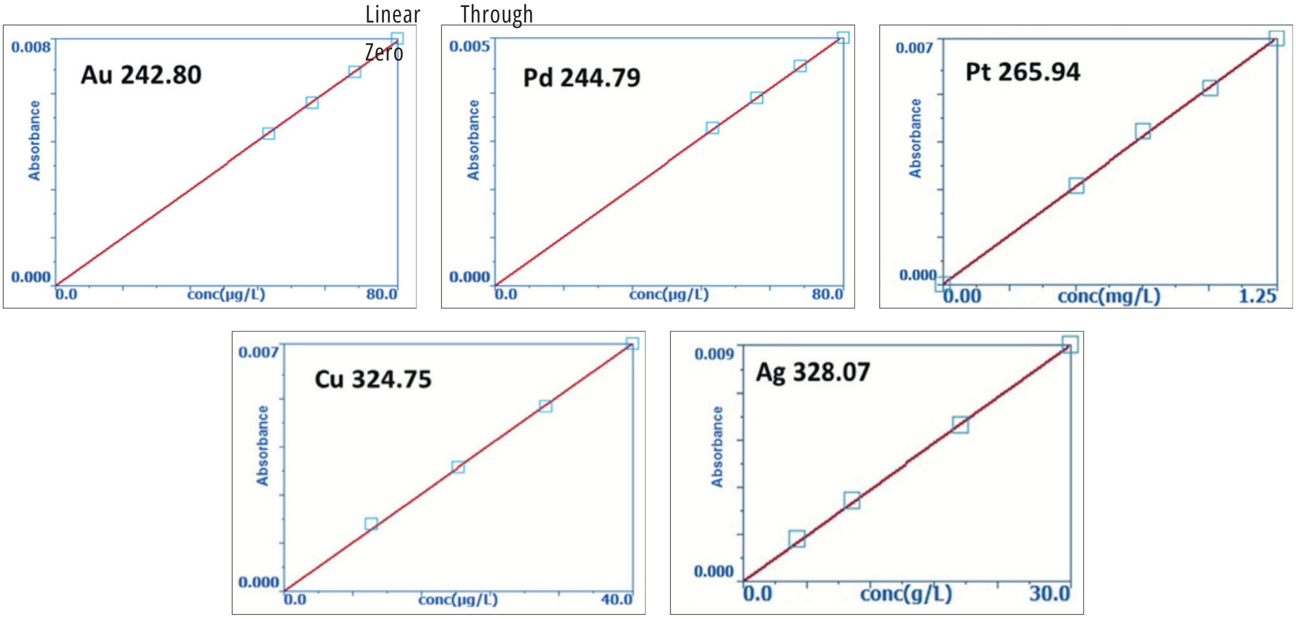

Figure 1. Low-level calibration curves for gold, palladium, platinum, copper, and silver.

Because of the importance of measuring precious metals at low levels, detection limits were determined under the same instrumental conditions and timings as the calibration and quantitation studies (Table 4) using the following formula:

$$\text{Detection Limit} = \frac{(3*SD*CC)}{0.0044}$$

SD = Standard deviation
CC = Characteristic concentration

The characteristic concentration is determined by running a standard, recording the absorbance, and using the following formula:

$$\text{Characteristic Concentration} = (\text{Conc}*0.0044)/\text{Abs}$$

Conc = Concentration of the standard
Abs = Absorbance

An explanation of the “0.0044” constant can be found elsewhere1.

Conclusion

This work has demonstrated the ability of the PinAAcle 500 AA spectrometer to accurately measure low-level gold, palladium, platinum, copper, and silver in matrices which result from the fire-assay preparation of ore samples. The Syngistix Touch™ software operated from the PinAAcle’s large touchscreen display allows for simple operation when analyzing samples. If more flexibility is desired, Syngistix™ for AA software can run from PinAAcle’s on-board computer – this full-featured software provides flexibility for method development, allows off-line post-analysis data reprocessing, and enhanced reporting capabilities, among other features.

In addition to enhanced software capabilities, the PinAAcle 500 has also been optimized for use in corrosive environments with samples prepared in highly acidic matrices. Examples include acid-resistant coatings on the flame shield and instrument boards.
With its high corrosion resistance, flexible software options, and enhanced analytical capabilities, the PinAAcle 500 is an excellent instrument for measuring precious metals in a mining environment.

References

1. “Sensitivity Versus Detection Limit”, Technical note, PerkinElmer, Inc.

Table 4. Detection limits.

Element	Matrix	Detection Limit (µg/L)
Au	15% aqua regia	10.5
	1% HNO_3	8.2
Pd	15% aqua regia	13.7
	1% HNO_3	14.2
Pt	15% aqua regia	56.2
	1% HNO_3	56.5
Cu	1% HNO_3	2.1
Ag	1% HNO_3	1.4

Consumables Used

Component	Part Number
Au Hollow Cathode Lamp	N3050107
Pd Hollow Cathode Lamp	N3050158
Pt Hollow Cathode Lamp	N3050162
Cu Hollow Cathode Lamp	N3050121
Ag Hollow Cathode Lamp	N3050102
Nebulizer Capillary Tubing	09908265
High-Sensitivity Nebulizer with Tantalum Capillary	N3160152
Au 1000 mg/L Standard	N9303759 (125 mL) N9300121 (500 mL)
Pd 1000 mg/L Standard	N9303789 (125 mL) N9300138 (500 mL)
Pt 1000 mg/L Standard	N9303791 (125 mL) N9300140 (500 mL)
Cu 1000 mg/L Standard	N9300183 (125 mL) N9300114 (500 mL)
Ag 1000 mg/L Standard	N9300171 (125 mL) N9300151 (500 mL)
Autosampler Tubes	B0193233 (15 mL) B0193234 (50 mL)

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