Determination of Low Levels of Arsenic using Flame AAS and Agilent UltrAA Lamps

Application Note

Atomic Absorption

Author

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Introduction

Nickel ore can be profitable to mine and process depending on the prevailing market value of the nickel. It can also be profitable depending on the levels of more valuable base metals such as cobalt that may be present and that can be extracted by the processing method. The value of the ore may be decreased however depending on the levels of "penalty" elements. Arsenic presents a unique case in that it is an unwanted impurity and at high levels (generally greater than 200 ppm in ore) may inhibit the efficiency of modern processing technologies.

Rapid screening of the ore is therefore required. Base metals such as iron, nickel, cobalt, copper, zinc can be quickly and easily determined by flame AAS. Arsenic presents a unique challenge because it is usually present at levels that make it difficult to determine by flame AAS.

The matrix to be analyzed and the relative slowness of both hydride and graphite atomizer techniques make them unattractive for this application. The UltrAA boosted discharge hollow cathode lamps allow arsenic to be determined by flame to the low levels required for the screening application in the presence of high levels of the other base metals, without the need to resort to separate extraction procedures for arsenic or the use of a different technique. That is, all elements – high level base metals and low level arsenic – can be determined from a single extracted solution by a single analytical technique.

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Experimental

Instrumentation

The following were used for this study:

- Agilent SpectrAA 55B Atomic Absorption Spectrometer, connected to a PC

- Agilent UltrAA lamp control module

- Arsenic UltrAA lamp

- Sample Introduction Pump System accessory (SIPS) for actual samples

The instrument parameters for sample measurement are listed in Table 1.

Table 1.   Instrument Parameters Used for Sample Measurements

Parameter	Value
Instrument mode	Absorbance
Sampling mode	Manual
Calibration mode	Concentration
Measurement mode	Integrate
Replicates standard	10
Replicates sample	10
Wavelength	189.0 nm
Slit width	1.0 nm
Lamp current	10.0 mA
Background correction	BC On
SIPS	On
Number SIPS standards	4
Measurement time	1.0 s
Pre-read delay	5 s
Flame type	N_2O/Acetylene
N_2O flow	9.00 L/min
Acetylene flow	6.80 L/min

Reagents and Solutions

Standards used were diluted from 1000 ppm AA standards supplied by BDH (Poole, England).

All acids used were AR grade supplied by BDH (Poole, England).

Sample Preparation

The general procedure is that the milled ore sample (1 g) is treated with a mixed acid reagent (nitric acid, hydrochloric acid, hydrofluoric acid, perchloric acid) with hydrofluoric

acid being used to remove silicates and the perchloric used to dissolve sulfates.

Warning: Observe all precautions when using hydrofluoric acid or perchloric acids.

The digest solution is taken to dryness, re-dissolved in 10 mL of 10% hydrochloric acid and made up to 100 mL with distilled water.

Calibration

A study was carried out to determine the best resonance line to use. Arsenic has three resonance lines, 189.0 nm, 193.7 nm and 197.2 nm. The UltrAA lamp boosts all three lines; the intensities as measured (without flame) by the relative instrument gain are similar (Table 2). The respective calibration graphs for each of the resonance lines using air-acetylene flame are compared in Figure 1.
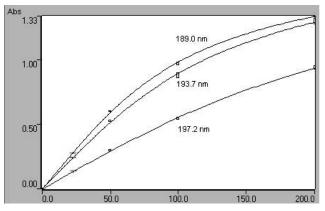

Figure 1:   Calibration graphs at the three resonance lines for arsenic using air-acetylene flame.

The respective calibration graphs for each of the resonance lines using nitrous oxide-acetylene flame are compared in Figure 2.
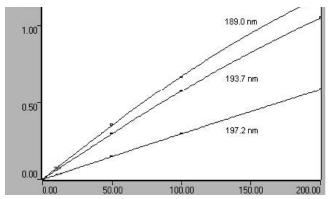

Figure 2.   Calibration graphs at the three resonance lines for arsenic using nitrous oxide-acetylene flame.

The characteristic concentrations (C.C.) for each resonance line using air-acetylene and nitrous oxide-acetylene flames were determined and are reported in Table 2.

Table 2. Characteristic Concentrations of Arsenic at Different Wavelengths and Different Flame Conditions

Wavelength (nm)	189.0	193.7	197.2
Gains (%)	54	52	47
C.C. mg/L (air-C_2H_2)	0.37	0.42	0.74
C.C. mg/L (N_2O-C_2H_2)	0.62	0.74	1.47

Although the most sensitive conditions are in the air-acetylene flame at 189.0 nm, the nitrous oxide-acetylene flame is generally preferred for the nickel ore samples. The nitrous oxide-acetylene flame provides the normal advantages of reduced interferences from the sample matrix because of its hotter temperature. Additionally, the nitrous oxide-acetylene flame tends to be more transparent than air-acetylene flame in the extreme ultra-violet region of the spectrum.

A variety of high nickel ores which contained low levels of arsenic (10–2500 ppm in ore) were analyzed (using SIPS). The instrument parameters are shown in Table 1. The results are shown in Table 3.

Table 3: Extract of Report for 26 Samples

Sample id	Concentration mg/L	Mean Abs	Recovery
Cal zero	0.0	-0.0033	
Standard	25.0	0.1717	
Standard	50.0	0.3342	
Standard	75.0	0.4805	
Standard	100.0	0.6186	
50 ppm Standard 2	49.1	0.3281	
Blank	0.2	0.0017	
Sample 001	13.3	0.0916	
Sample 001 dup	13.8	0.0950	104%
50ppm Standard 2	45.6	0.3064	91%
Sample 013	11.9	0.0816	
Sample 014	5.7	0.0395	
Sample 013 dup	11.5	0.0791	97%
Sample 014 dup	5.4	0.0372	95%
Sample 017	0.1	0.0007	
100 ppm Standard 4	92.6	0.5790	93%
Sample 023	23.4	0.1604	
Sample 024	12.2	0.0841	
Sample 025	0.6	0.0043	
Sample 026	0.9	0.0065	
100 ppm Standard 4	97.3	0.6029	97%
Sample 023 dup	23.6	0.1613	101%
Sample 024 dup	12.0	0.0828	98%
Sample 024 dup	11.6	0.0801	95%
Sample 023 dup	23.9	0.1634	102%
Blank	0.3	0.0019	

The limits of detection ranged from 0.05 to 0.14 mg/L in final solution. This equates to 5 to 14 ppm in ore.

Notable are the quality of the repeats, the recoveries of QCs and the ability to discriminate between samples with very low levels of analyte (< 1ppm in solution) without the need to modify the extraction procedure specifically for arsenic or to have a second extraction procedure for this element.

Conclusion

The combination of nitrous oxide-acetylene flame and UltrAA lamps with any SpectrAA instrument allows rapid and simple determination of low levels of arsenic in nickel ores on extractions that are common for the determination of the base metals. The high intensity UltrAA lamp allows full use of the more sensitive 189.0 nm and 193.7 nm lines not normally available on other systems.

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