APPLICATION NOTE

Atomic Absorption

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Determination of Arsenic and Nickel in Serum by Graphite Furnace Atomic Absorption Spectrometry

Introduction

Two common techniques for trace element determinations in complex matrices are graphite furnace atomic absorption spectrometry (GFAAS) and inductively coupled plasma mass spectrometry (ICP-MS). While both techniques can successfully perform these analyses, GFAAS has the advantage of being lower cost and more well-established, while ICP-MS is capable of higher sample throughput, especially as the number of analytes increases. The appropriate technique depends on the needs of the lab.

Both GFAAS and ICP-MS have been used to determine the trace elemental content of serum, a complex sample which contains high levels of both salts and biomolecules. These matrix components can cause spectral interferences, instrumental drift, and/or instrumental contamination, depending on the elements measured, the instrumental technique used, the number of samples analyzed, and the analytical conditions. This work describes methodology for the determination of arsenic (As) and nickel (Ni) in serum via graphite furnace atomic absorption spectrometry.

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Experimental

Standard and Sample Preparation

Calibration standards were prepared in a pooled serum sample (i.e. a combination of several clean serum samples), which were diluted with a mixture of 0.1% Triton™ X-100 + 0.2% (v/v) nitric acid. The blank, standards, and sample all used the same dilution ratio: 0.4 mL serum plus 0.8 mL diluent.

Calibration standards were prepared by serial dilutions from 1000 ppm stock solutions to yield the final concentrations shown in Table 1. These levels were chosen to cover the expected concentrations in the serum sample.

Table 1. As and Ni Calibration Standards for Serum Analysis.

Arsenic		Nickel	
Standard Number	Concentration (µg/L)	Standard Number	Concentration (µg/L)
1	5	1	1
2	10	2	2
3	20	3	4
		4	8

Instrumental Conditions

All analyses were performed on the PerkinElmer PinAAcle™ 900Z atomic absorption spectrometer (Part No. N3160060), using Zeeman background correction. For optimal performance, different instrumental conditions were used for As and Ni, as shown in Tables 2 and 3. The matrix modifier used for As determination was prepared by combining 1 mL of a 1% $Pd(NO_3)_2$ solution with 0.1 mL of a 2% $Mg(NO_3)_2$ solution and diluting to 10 mL with deionized water. For Ni measurements, a matrix modifier was not necessary. The argon (Ar) flow is used to remove residues from the graphite tube during pyrolysis and is turned off during the atomization and reading step (Step 7 in Table 3), while air is used to burn off residual carbon to prevent build up in the graphite tube. Step 5 is included in the furnace program to extend the tube life: by switching to argon before atomization, oxygen is removed from the tube, thus improving tube lifetime.

Table 2. PinAAcle 900Z Instrumental Conditions for As and Ni in Serum.

Parameter	Conditions for Arsenic	Conditions for Nickel
Lamp Type	Electrodeless Discharge	Hollow Cathode
Wavelength (nm)	193.70	232.00
Slit (nm)	0.7	0.2
Read Time (s)	3	5
Baseline Offset Correction Time (s)	2	2
Graphite Tube	End Cap	THGA
Matrix Modifier	0.1% $Pd(NO_3)_2$ + 0.02% $Mg(NO_3)_2$	None
Matrix Modifier Volume (µL)	4	---
Sample Volume (µL)	15	20
Replicates	2	2

Table 3. Furnace Program for Measurement of As and Ni in Serum.

Step	Temperature (°C)	Ramp (s)	Hold (s)	Gas Flow (mL/min)	Gas
1	110	1	30	250	Argon
2	130	20	30	250	Argon
3	300	20	5	250	Air
4	550	20	30	250	Air
5	550	1	15	250	Argon
6	1250	10	20	250	Argon
7	2000	0	3	0	Argon
8	2450	1	3	250	Argon

Results and Discussion

Figure 1 shows the calibration curves for both As and Ni, which demonstrates the linearity of the methodology, with correlation coefficients greater than 0.997 for each element when using Linear, Calculated Intercept.

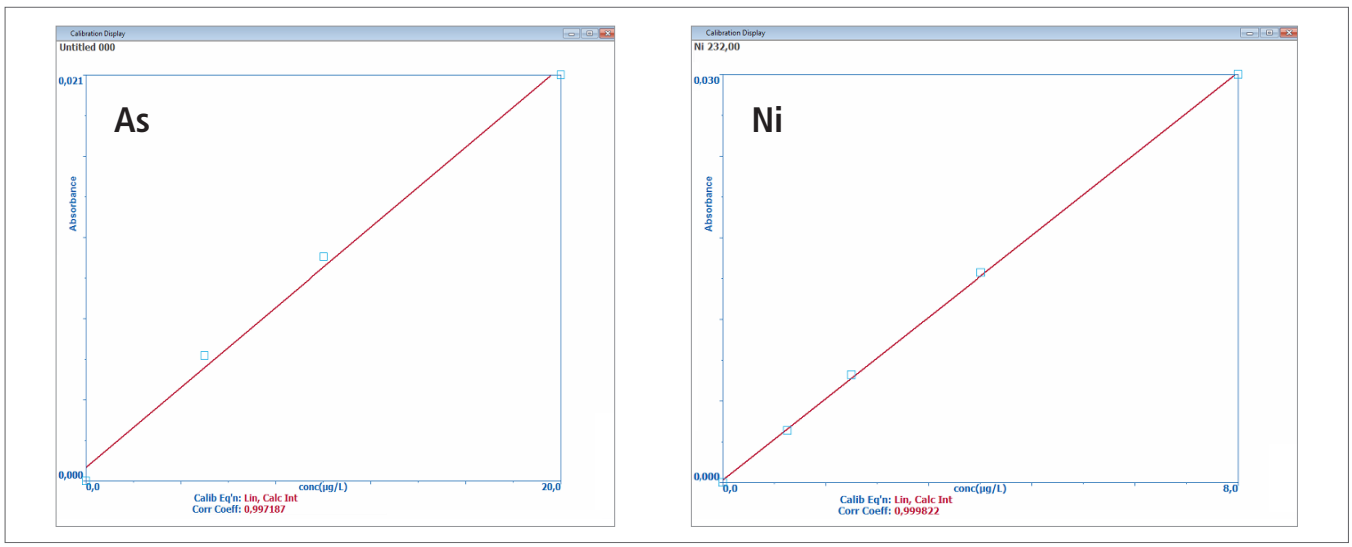

Figure 1. Calibration curves for As and Ni in serum.

Performance of the methodology was assessed through reference materials and spike recoveries. Seronorm™ Trace Elements in Serum (Lot Number 1309416) has a certified range of 7.9 – 11.9 µg/L for Ni. When determined by this methodology, a value of 10.6 µg/L was obtained, well within the certified range, demonstrating the accuracy of the methodology for Ni.

Since Seronorm™ serum does not have a certified As concentration, the accuracy of the methodology was assessed by spike recoveries. A typical sample was spiked with 10 µg/L of both As and Ni and analyzed. The results are shown in Table 4; with recoveries within 5% of the spiked values for both elements, the accuracy of the methodology is demonstrated.

Table 4. As and Ni Spike Recoveries in Serum.

Element	Spike Level (µg/L)	Sample (µg/L)	Spiked Sample (µg/L)	% Recovery
As	10.0	0.37	10.7	103%
Ni	10.0	0.93	11.4	105%

With the accuracy established, the methodology was applied to the analysis of serum samples from industrial workers. Of the fifty samples analyzed, all showed Ni levels below 1 µg/L, while As appeared in only five samples at levels greater than 0.15 µg/L. These results indicate that the workers had minimal exposure to As and Ni during the jobs.

Conclusions

This work has demonstrated the ability of GFAAS to determine arsenic and nickel in serum, providing an alternative to ICP-MS instrumentation. The advantages of GFAAS include lower cost, smaller sample volume, and no sample pretreatment apart from dilution. By applying the appropriate furnace conditions, accurate results at low concentrations in complex samples can be attained.

Instrument Options

Model	Part Number
PinAAcle 900Z Zeeman Furnace AA Spectrometer (IVD)	N3160060
PinAAcle 900T Combined Flame & Zeeman Furnace AA Spectrometer (IVD)	N3160082

Consumables Used

Component	Part Number
1% $Pd(NO_3)_2$ Matrix Modifier Solution	B0190635
1% $Mg(NO_3)_2$ Matrix Modifier Solution	B0190634
THGA Graphite Tubes	B3000641 (pack of 5) B0504033 (pack of 20) N3110147 (pack of 100)
THGA Graphite Tubes with Endcaps	B3000653 (pack of 5) B3000655 (pack of 20)
Autosampler Cups, 1.2 mL, Polypropylene	B0510397 (bag of 2000)
Autosampler Tray, 132 Positions for 1.2 mL Autosampler Cups	B3001506
1000 ppm Arsenic Standard	N9300180 (125 mL) N9300102 (500 mL)
1000 ppm Nickel Standard	N9300177 (125 mL) N9300136 (500 mL)
As Electrodeless Discharge Lamp	N3050605
Ni Hollow Cathode Lamp	N3050152

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