Aluminium Determinations in Parenteral Solutions

Application Note

Atomic Absorption

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Introduction

An Agilent atomic absorption spectrometer was used to evaluate the performance for the determination of aluminium in parenteral solutions. A wide range of test samples were run on the instrument over a six month period. Results, based upon recoveries of aluminium supplements, were satisfactory for quantitative measurements. The preferred furnace arrangement for the assays was a pyrolytically coated tube without a platform.

Agilent Technologies

Experimental

Instrumentation

The instrument was an Agilent SpectrAA-400 Zeeman Atomic Absorption Spectrometer with autosampler and IBM PS/2 Model 3286 computer. The graphite tube was partitioned pyrolytic coated and normal inert gas was argon. A platform was used for part of this study.

Table 1 contains spectrometer parameters for the aluminium determinations along with typical calibration and sample volumes programmed into the autosampler. The first experiments were performed using a furnace program supplied by Varian in Wood Dale, Illinois, but the program was later modified. The furnace parameters are listed in Table 2.

Table 1. Spectrometer and Autosampler Conditions

Instrument parameters			
Lamp current (mA)	10		
Slit width (nm)	0.5		
Slit height	Normal		
Wavelength (nm)	309.3		
Sample introduction	Sampler automixing		
Measurement time (seconds)	3.0		
Replicates	2		
Background correction	On		
Maximum absorbance	2.00		
Sampler volumes	**Solution**	**Blank**	**Modifier**
Blank	0	15	5
Standard 1	4	11	5
Standard 2	6	9	5
Standard 3	10	5	5
Sample	10	5	5
Multiple inject	No		
Hot inject	No		
Pre inject mod.	No		

Table 2a. Graphite Furnace Parameters– Initial Temperature Program for Aluminium Assays

Step	Temp °C	Time	Gas flow	Gas type	Read
1	95	5.0	3.0	Normal	No
2	95	40.0	3.0	Normal	No
3	120	10.0	3.0	Normal	No
4	500	15.0	3.0	Normal	No
5	1600	10.0	3.0	Normal	No
6	1600	20.0	3.0	Normal	No
7	1600	2.0	0.0	Normal	No
8	2600	0.5	0.0	Normal	Yes
9	2600	4.0	0.0	Normal	Yes
10	2700	2.0	3.0	Normal	No

Table 2b. Graphite Furnace Parameters – Modified Temperature Program

Step	Temp °C	Time	Gas flow	Gas type	Read
1	95	5.0	0.0	Normal	No
2	95	40.0	3.0	Normal	No
3	120	20.0	3.0	Normal	No
4	500	10.0	3.0	Normal	No
5	500	20.0	3.0	Normal	No
6	1400	20.0	3.0	Normal	No
7	1400	20.0	3.0	Normal	No
8	1400	2.0	0.0	Normal	No
9	2500	0.6	0.0	Normal	Yes
10	2500	3.0	0.0	Normal	Yes
11	2500	2.0	3.0	Normal	No

Test solutions

Dianeal (peritoneal dialysis) solution, low Ca, 1.5% dextrose[1]

Dianeal solution, low Ca, 2.5% dextrose[1]

Dianeal solution, low Ca, 3.5% dextrose[1]

Heparin, sodium, 5 units/mL in 0.9% NaCl[1]

10% Travasol (amino acid injection) solution with electrolytes[1]

2.75% Travasol solution with 10% dextrose[1]

15% Novamine (amino acid injection) solution[2]

Amino acid mixture B powder - a blend of 12 amino acids

Amino acid mixture C powder - a blend of 13 amino acids

[1] Product of Baxter Healthcare Corporation, Deerfield, Illinois
[2] Product of Kabi Pharmacia, Clayton, NC

Reagents

Deionized distilled water, NANOpure II, Sybron/ Barnstead

Nitric acid, J. T. Baker Chemical Co., Ultrex

Magnesium nitrate, recrystallized from reagent grade

1000 mg/L aluminium, commercial standard, Ricca Chemical Co.

A working calibration standard of 10 ng/mL was prepared each day by successive dilutions of the aluminium stock solution in 0.14 M nitric acid.

A matrix modifier solution was added to standard and sample injections. The modifier solution contained 22 mL of water, 2 mL of nitric acid and 1 mL of a 20% w/v solution of the recrystallized magnesium nitrate. Each injection included five microlitres of the modifier, adding 40 micrograms of magnesium nitrate. The purity of the magnesium nitrate was essential to the success of the assay, otherwise the reagent blanks were too high to obtain a standard calibration.

Reagents and samples were prepared exclusively in plastic labware. The autosampler vials were soaked in a 1.4 M nitric acid solution and rinsed with water before use.

Sample Preparation

Initially, samples of Dianeal solution and heparin were tested directly against external standards with no pretreatment. The samples were supplemented with 10 ng/mL of aluminium added by the autosampler to determine analytical recoveries. The experiments were run with and without a platform installed in the graphite tube, using the first temperature program detailed in Table 2.

Powdered samples of amino acids were prepared by adding five grams to a 100 mL polymethylpentene volumetric flask containing one mL of nitric acid in about 50 mL of water. The sample was allowed to dissolve and the flask filled to volume with water. Some of the amino acid samples were run using the first temperature program listed in Table 2, then the program was modified and all further work done using the second temperature program.

The technique for the more viscous samples in-volved diluting in the autosampler cups with a 0.14 M nitric acid solution. The following dilutions were made using micropipeters with disposable plastic tips: 10% Travasol with Electrolytes 1+2, 15% Novamine 1+3, Travasol/Dextrose 1+1.

Results

Data obtained for the test solutions under various conditions are presented in Table 3. The data is listed in chronological order of the experiments. The somewhat poorer recoveries at the top of the Table may be due in part to the learning curve of the instrument operator.

Table 3. Aluminium Assay Results

Test solutions	**%Recovery for tube wall**	
Dianeal, low Ca, 1.5% dextrose	82, 79	
Dianeal, low Ca, 2.5% dextrose	76	
Dianeal, low Ca, 3.5% dextrose	61	
	%Recovery for platform	
	Run #1	**Run #2**
Dianeal, low Ca, 1.5% dextrose	82, 85	86, 87
Dianeal, low Ca, 2.5% dextrose	76	103
Dianeal, low Ca, 3.5% dextrose	55	82
Heparin, sodium in saline	105,117	
Amino acid mixture C,	#1	92, 86
	#2	82, 81
Modified temperature program		
1400 °C ash, 2500 °C atomize	**%Recovery for platform**	
10% Travasol w/electrolyte	113, 106	
Amino acid mix C, 5% w/v	93, 104	
Amino acid mix B, 5% w/v	90	
Amino acid mix B, 5% w/v	112, 104, 102	
Amino acid mix B, 5% w/v	109, 112	
Amino acid mix B, 5% w/v	110, 106	
	%Recovery for tube wall	
15% Novamine	103, 104, 103	
15% Novamine	106, 112, 115	
15% Novamine	109, 103, 105	
Dianeal, low Ca	98	
Dianeal, low Ca	97	
Travasol/dextrose	95	
Amino acid mix B, 5% w/v	112, 104	
Amino acid mix B, 5% w/v	109, 112	
Amino acid mix B, 5% w/v	110, 106	

A more moderate ashing temperature of 1400 °C appeared to improve the instrument performance, even for samples such as Dianeal that contain electrolytes.

The sensitivity of the assay, as calculated by the characteristic mass, was 14 pg (for 0.0044 absor-bance). Determinations at the ultra-trace level may be performed with such an instrument sensitivity.

There is no obvious benefit revealed in the data by using a platform instead of using tube wall injections. It may be that the massive centre section of the Agilent graphite tube design allows for performance resembling that of a stabilized temperature furnace. Injections onto the tube wall generally allowed for more reproducible absorbance readings than those on a platform. Since tube wall injections were free of problems such as test solution running off the platform, they offered a more trouble-free option for routine instrument operation.

Figure 1 contains absorbance profiles of injections onto the tube wall for an 8 ng/mL calibration stan-dard (top) and a sample of amino acid mixture B supplemented with 6 ng/mL of aluminium. The peaks have similar gausian shapes with no obvious shoulders suggesting interferences. Injections onto a platform gave much shallower and broader absorbance profiles.

Conclusion

The experiments demonstrate that the performance of the Agilent SpectrAA-400 Zeeman Atomic Absorption Spectrometer is sufficient to assay a variety of parental solutions containing organic and inorganic components. Aluminium determinations at the ultra-trace level can be made reliably with the system.

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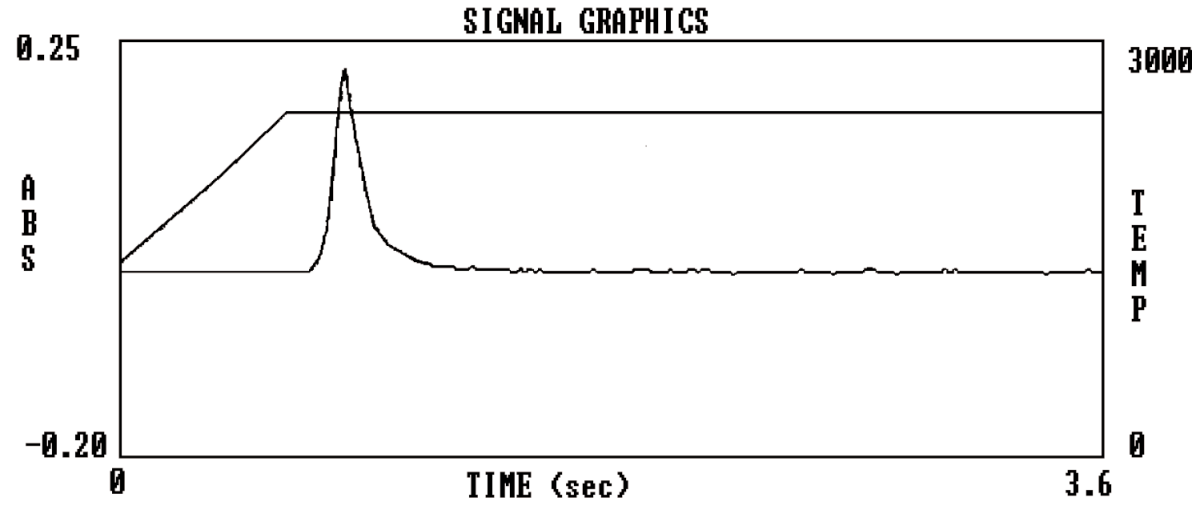

Figure 1a. Absorbance profiles for aluminium –8 ng/mL aluminium calibration standard.

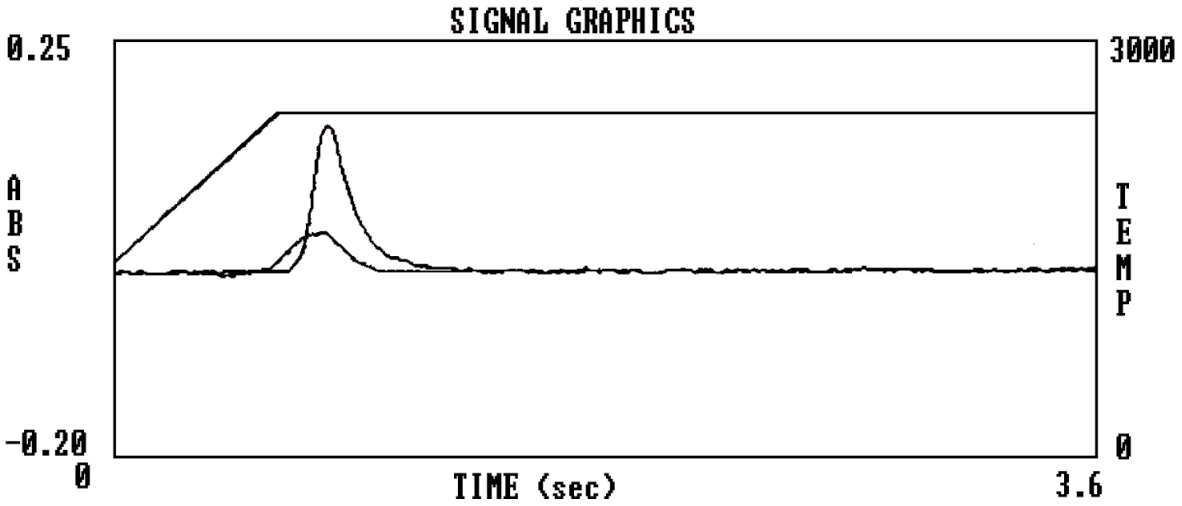

Figure 1b. Absorbance profiles for aluminium – 5% amino acid solution with 6 ng/mL aluminium added.

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Printed in the USA
November 1, 2010
AA109

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