High Performance Graphite Furnace Tube for Determination of Lead in Blood

Application Note

Clinical Research and Pharmaceutical

Authors

Kai Robinson,

John Sanders,

James Barker

Introduction

With the development of GF-AAS there has been continued development of graphite tubes and platforms. Early tubes used uncoated graphite, had poor lifetime, memory and low sensitivity. Development of pyrolytically coated tubes improved resistance to oxidation. Coated tubes produced superior lifetime, sensitivity and precision of analytes.

The next advance in tube design was the introduction of a graphite platform ("L'vov platform") onto which the sample is injected for atomization. The use of a platform delays atomization until the gas environment has reached a stable temperature relative to the tube wall. This prevents condensation and recombination of the analytes in the gas phase.

A variety of platform designs have been utilized over the years, some of these having limitations such as small sample volume and restricted atomization temperature limits.

This application note describes a graphite tube design that addresses these limitations, resulting in improved sensitivities and detection limits compared to previous tube designs. This design is called the Agilent Omega Platform Tube.

Exposure to lead has adverse effects on human health. The Centers for Disease Control and Prevention (CDC) state that a blood lead level of greater than 10 µg/dL (equivalent to 0.48 µmol/L) is of concern and acknowledges studies indicating evidence of adverse effects in children at lower levels [1].

In this application note the Agilent Omega Platform Tubes are evaluated for the determination of lead in blood. Deuterium and Zeeman background correction are two commonly used techniques that are suitable for this application. Zeeman background correction is used for background correction in this application. This complex matrix requires good control of the furnace temperature profile.

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Instrumentation

The instrumentation used for the determination of lead in blood was an Agilent 280Z AA with Zeeman background correction. The Agilent 280Z AA is configured for 8 lamps in fixed positions. Agilent UltrAA lamps, which boost the emission intensity for reduced noise and enhanced sensitivity, can be used with this instrument.

The spectrometer was equipped with an Agilent GTA 120 graphite tube atomizer and an Agilent PSD 120 Programmable Sample Dispenser. The Agilent PSD 120 provides the capacity for up to 135 solutions and allows automatic standard preparation and over-range dilution.

The furnace viewing camera, Tube-CAM, is a standard feature of the Agilent 280Z AA. Tube-CAM provides real time viewing inside the graphite tube via SpectrAA software. Still images and videos can be recorded throughout an analysis. Tube-CAM enables the dispensing height to be set accurately, confirmation of furnace conditions during method development, and monitoring of potential matrix buildup inside the tube after the atomization stage.

Table 1 lists the instrument settings and Table 2 lists the furnace settings used in the analysis. The inert gas used was high-purity argon.

Table 1. Instrument Settings

	Instrument Parameters	Settings
Measurement	Replicates	2
	Mode	Peak Area
Optical	Lamp	Pb UltrAA
	Wavelength	283.3 nm
	Slit Width	0.5R
	Lamp Current	10.0 mA
Standards	Standard 1	0.0483 µmol/L
	Standard 2	0.0966 µmol/L
	Standard 3	0.1449 µmol/L
	Standard 4	0.2419 µmol/L
	Standard 5	0.3382 µmol/L
Sampler	Total Volume	10 µL
	Sample Volume	10 µL
	Number of Injections	1

Table 2. Furnace Settings

Step	Temp (°C)	Time (s)	Flow (L/min)	Read
1	80	5	0.3	
2	95	40	0.3	
3	120	5	0.3	
4	250	5	0.3	
5	250	5	0.3	
6	400	10	0.3	
7	700	10	0.3	
8	700	10	0.3	
9	700	2	0	
10	2200	0.7	0	Yes
11	2200	1.5	0	Yes
12	2600	0.4	0.3	
13	2600	2	0.3	

Materials and Reagents

A non-ionic surfactant was used to hemolyze the blood samples. A phosphate chemical modifier was used to permit the use of a higher ashing temperature, reduce matrix interferences and to stabilize the lead signal [2].

The following reagents were used for preparation of calibration solutions and for sample preparation:

- Lead standard solution, 1000 mg/L (SpectrosoL, BDH Laboratory Supplies, Poole, BH15 1TD, England)
- Non-ionic Surfactant (Triton X-100, LABCHEM, AJAX Chemicals, Sydney, NSW, Australia)
- High-purity nitric acid (Ultrapur, Merck, Kilsyth, Victoria, Australia)
- Ammonium dihydrogen phosphate (Suprapur, Merck, Kilsyth, Victoria, Australia)
- Class 1 Water (MΩ/cm)
- Whole Blood Metals Control (Lyphochek Levels 1, 2 and 3, BIO-RAD Laboratories, Irvine, CA, USA)
- Porcine Blood (EDTA Anticoagulated, Gamma Irradiated, QCT Quality Control Technologies Pty Ltd, Newcastle, NSW, Australia)

Sample Preparation

A non-ionic surfactant/phosphate solution was prepared from 0.5 g ammonium dihydrogen phosphate and 5 mL of 1% (v/v) Triton X-100 solution made to 100 mL with Class 1 water.

A 1 mg/L lead solution (4.83 µmol/L Pb) was prepared in 0.1% nitric acid. This solution was then used to prepare the calibration solutions and matrix spike sample.

Five calibration solutions and a blank were prepared by diluting 1 mL of porcine blood and the required amount of 1 mg/L lead solution to 10 mL in non-ionic surfactant/phosphate solution.

Lyphochek Whole Blood Metal Controls were reconstituted per included instructions. The Certified Reference Material (CRM) solutions were then prepared by diluting 0.5 mL of reconstituted Lyphochek to 5 mL in non-ionic surfactant/phosphate solution.

A Lyphochek Level 1 spike was prepared by diluting 0.5 mL of Lyphochek Level 1 and 20 µL of 1 mg/L Pb solution to 5 mL in non-ionic surfactant/phosphate solution.

All calibration solutions and CRM solutions were prepared fresh daily.

The rinse solution for the PSD-120 programmable sample dispenser was 0.1% nitric acid and 0.1% non-ionic surfactant in Class 1 water. An alternate rinse solution of 0.1% nitric acid and 10% ethanol in Class 1 water could be used.

Results and Discussion

Accurate results were obtained for the spiked sample and CRMs (refer to Table 3). The method exhibited excellent correlation coefficient $R^2 = 0.9992$ (refer to Figure 1). The Omega Platform Tube exhibited good performance for this difficult matrix with minimal carbon buildup (refer to Figure 2).

The temperature profile of the standard and sample show the high signal to noise ratios obtained with the Omega Platform Tube (refer to Figures 3 and 4).

No carry-over was observed between samples. After each analytical sequence the dispenser capillary was soaked in aqua-regia for 1 minute to remove any protein and to ensure that the capillary remained hydrophobic. This is a recommend practice for this matrix.

Table 3. Spiked Sample and CRM Results

Sample	Pb Conc. (µmol/L)	Recovery (%)
Porcine blood	0	N/A
Porcine blood spike (0.14 µmol/L Pb)	0.14	100
Lypocheck 1 (0.41 µmol/l Pb)	0.40	98
Lypocheck 2 (1.34 µmol/L Pb)	1.31	98
Lypocheck 3 (2.50 µmol/L Pb)	2.63	105

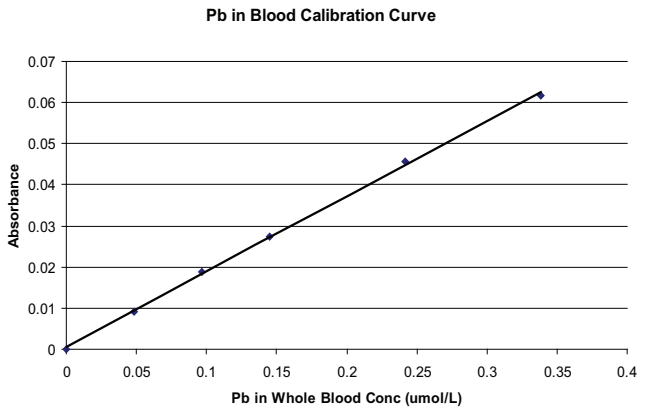

Figure 1. Calibration curve for Pb in blood showing excellent correlation coefficient, $R^2 = 0.9992$.

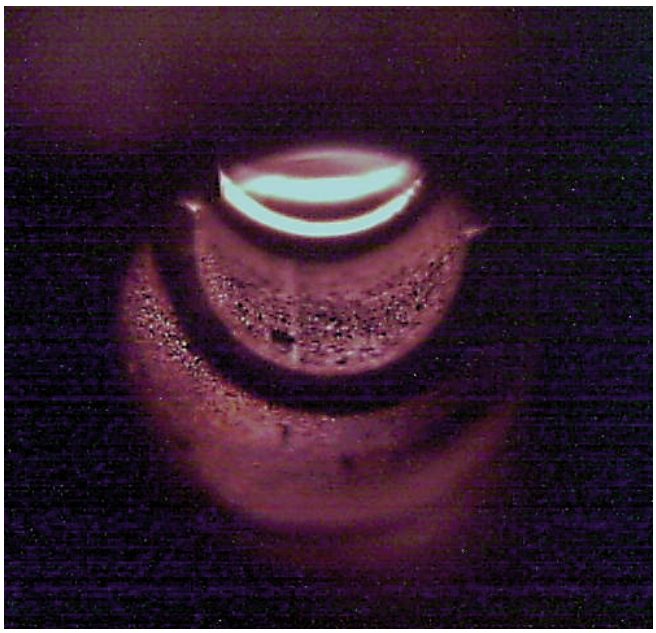

Figure 2. Image of Omega Platform Tube from the Agilent Tube-CAM viewing camera at the completion of the work.

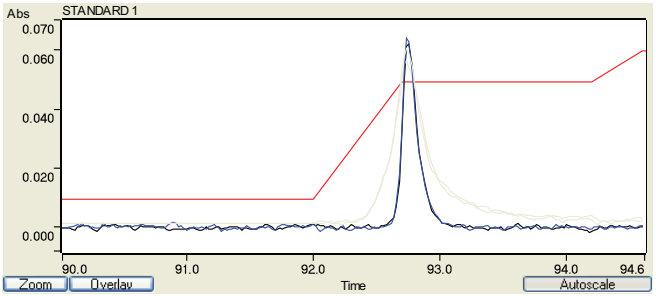

Figure 3. Temperature Profile for 0.0483 µmol/L Pb in blood standard.

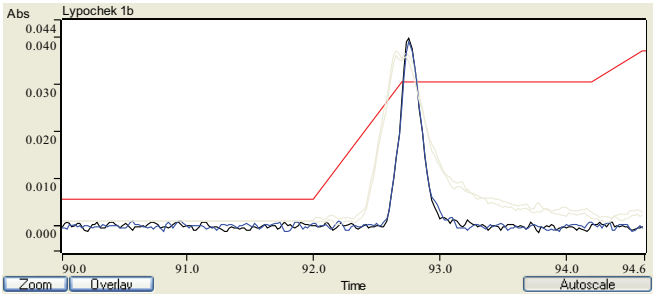

Figure 4. Temperature Profile for Lypocheck 1 SRM.

Conclusion

The high performance Agilent Omega Platform Tube has been successfully used for the determination of lead in blood by graphite furnace AAS using an Agilent 280Z AA. The complex sample matrix produced broad-band molecular absorption which was accurately corrected using Zeeman background correction. Excellent recoveries for spiked samples (100%) and standard reference materials (98% at 0.41 µmol/L) were obtained.

The benefits of the Agilent Omega Platform Tube design are applicable to a variety of other analyses.

References

1. J. L. Gerberding, et al., Preventing Lead Poisoning in Young Children, A Statement by the Centers for Disease Control, Public Health Service, Atlanta, GA (5th Revision August 2005). U.S. Department of Health and Human Services, Centers for Disease Control and Prevention Web site. Available at: http://www.cdc.gov/nceh/lead/ACCLPP/recommend.htm Accessed 20th June, 2008.
2. Hinderberger, E. J., Kaiser, M. L., and Koirtyohann, S. R. At. Spectrosc. 1981, 2 (1), 1-7.

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