Application Brief

Environmental

Measuring Lead in Water

Utilizing intelligent optimization of analysis parameters

Element: Pb
Matrix: Water
Modifier: $NH_4H_2PO_4$ + $Mg(NO_3)_2$

Instrumentation: Agilent 240Z Graphite Furnace AAS

Standards:

ISO 15586:2003

U.S. EPA Method 200.9

IS 10500; IS 14543, IS 13428

GB 5749-2006

3113 B:2012

Introduction

The major sources of human exposure to lead (Pb) are food and drinking water. In 2008, the World Health Organization released a guideline value of 10 µg/L as the maximum level of lead in drinking water. Monitoring the amount of lead in drinking water is a critical function of governments and other organizations around the world.

In order to measure Pb within the limits required by the different regulations, atomic absorption, combined with graphite furnace methods is most frequently used. However, optimization of methods for graphite furnace systems can be difficult, due to the complexity of the atomization process.

The Agilent 200 AA Series significantly simplifies graphite furnace method implementation through intelligent ashing and atomization optimizations. Furthermore, Agilent AA instruments feature the Stabilized Temperature Platform Furnace (STPF) concept. The instruments' Tube-CAM video can be used to control the injection and dry steps of the measurement. The SRM Wizard, an integrated feature of the instrument software, can automatically determine the best ash and atomize temperatures. Please see *Optimizing GFAAS ashing and atomizing temperatures using Surface Response Methodology* (2).

Example analysis

Furnace measurements were performed using an Agilent 240Z Atomic Absorption Spectrometer (AAS) with transverse Zeeman background correction. The instrument features the highly sensitive and accurate Agilent GTA 120 Graphite Tube Atomizer and the Agilent PSD 120 Programmable Sample Dispenser autosampler.

An extraction/LED accessory (see Figure 3) allows for improved fume removal compared to standard ventilation ducts. It also provides optimum viewing to easily align the capillary with the injection hole.

The instrument uses the patented Zeeman effect with longitudinal graphite tube heating and a Constant Temperature Zone (CTZ) design for best sensitivity and characteristic masses.

Atomization for Lead was from a pyrolytic platform Omega tube. The inert gas used was 99.99% pure argon.

Analytical Conditions

Table 1 summarizes the analytical conditions, including lamp type, tube type, and instrument parameters.

Table 1. Analytical Conditions

Parameter	Setting
Lamp	UltrAA Lamp Pb (Agilent part number 5610108200)
Graphite tube platform	Omega (Agilent part number 6310003700)
Wavelength	283.3 nm
Slit Width	0.5 nm
Lamp current	10 mA
Mode	Peak Area

Standard solution: 50 µg/L Pb

Standard Reference Material: SPS SW2: 25.0 ± 0.1 µg/L Pb (from LGC Standards)

Method optimization

Dry steps were optimized by using the integrated camera. Optimization of the ash and atomize temperatures (see Figure 1) was done using the using an intelligent chemometric method, included in the instrument software (the Surface Response Methodology tool). Tests were performed on a standard reference material and on a spiked water sample. All measurements were made using the Peak Area calculation.

Figure 1. The view inside the tube, using the integrated camera. The probe is dispensing the sample into the tube.

With the SRM Wizard, only 3 clicks were needed to develop the model. Based on the results of the chemometric analysis, the Surface Response Methodology tool determined the optimum conditions, shown in Tables 2 & 3 and in Figure 2.

Table 2. Experimental design factors

Ash: 700 °C	**Atomize 1600 °C**
Change: 200 °C	**Change: 250 °C**

Table 3. The optimum conditions determined for the standard and sample were similar, indicating that the modifier mix was suited to the application.

Temperature	Standard	Sample
Ash (°C)	598	614
Atomize (°C)	1435	1476

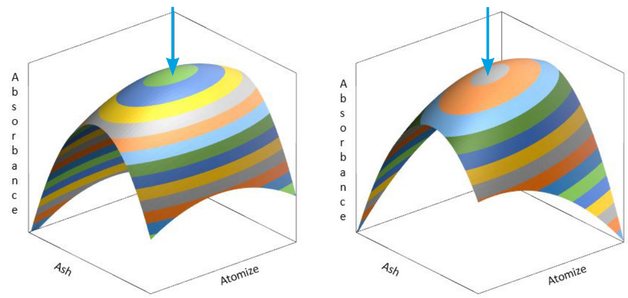

Figure 2. The Surface Response Methodology software tool uses chemometrics to automatically optimize the ash and atomize temperatures. Shown here is the plot for the standard reference material (left) and the spiked sample (right). The arrows indicate the optimum temperatures.

Results

- Characteristic concentration in peak area: 0.85 µg/L
- Characteristic Mass in peak area: 13.9 pg
- Instrumental Detection limit for 20 µL: 0.15 µg/L
- % recovery of SRM: 100.1 %
- % recovery of water spiked with 25 µg/L: 103.6%

Conclusion

The lead in drinking water method, applicable for a wide range of worldwide standards, was optimized automatically on the Agilent 240Z GFAA system. The method exceeded the required performance for detection limits and accuracy.

With the most sensitive Zeeman workhead, local fume extraction, LED lighted injection hole, Tube-Cam, and SRM wizard, the 240Z has the perfect tools for ease of use and method optimization. The 240Z is a cost effective solution, lowering argon consumption and increasing graphite tube lifetime without compromising accuracy and precision. It is ideal for any laboratory performing routine testing that wishes to minimize initial setup and optimization.

References

1. World Health Organization 2011, WHO/SDE/WSH/03.04/09/Rev/1 - Lead in Drinking-water

2. Optimizing GFAAS ashing and atomizing temperatures using Surface Response Methodology, Agilent publication number 5991-9156EN.

Optimize fume extraction for improved laboratory safety

The optional fume extraction accessory (Figure 3) includes an LED lighted mirror to provide a clear view of the graphite tube injection hole. Alignment of the autosampler capillary is worry-free while the exhaust system removes ashing vapors at their source. The extraction accessory provides flexibility of the furnace placement eliminating the need for direct position under the exhaust fan.

Figure 3. The optional extraction/LED accessory for the Agilent furnace.

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