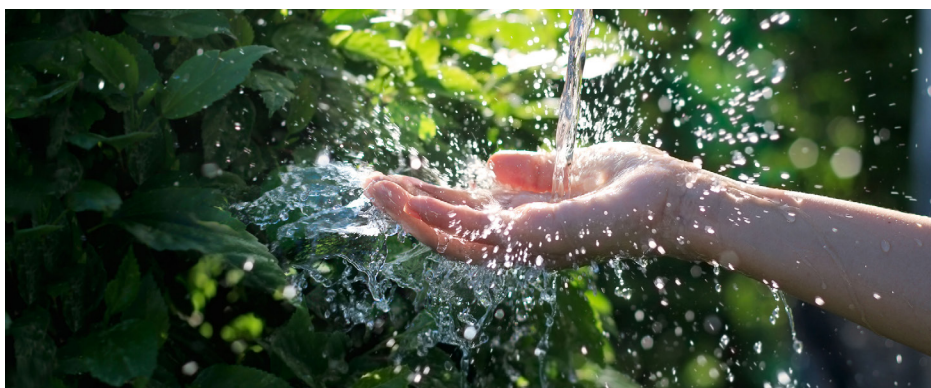


Measuring Cadmium in Water

Using intelligent optimization of analysis parameters



Element: Cd

Matrix: Water

Modifier: $\text{NH}_4\text{H}_2\text{PO}_4$ +
 $\text{Mg}(\text{NO}_3)_2$

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

Contamination of drinking water may occur as a result of the presence of cadmium as an impurity in the zinc of galvanized pipes or cadmium-containing solders in fittings, water heaters, water coolers and taps. In 2011, WHO confirmed the guideline value for Cadmium in drinking water as $3 \mu\text{g}/\text{L}$ (1). With its great sensitivity, graphite furnace is a cost-effective technique for such analysis.

The Agilent 200 AA Series simplifies graphite furnace method optimization. The 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 and their SRM Wizard can automatically determine the best ash and atomize temperatures. Please refer to *Optimizing GFAAS ashing and atomizing temperatures using Surface Response Methodology* (2).

Example analysis

Furnace measurements were performed using an Agilent 240Z AAS with transverse Zeeman background correction. The instrument features the highly sensitive and accurate Agilent GTA 120 Graphite Tube Atomizer and an Agilent PSD 120 Programmable Sample Dispenser autosampler. An extraction/LED accessory (refer to Figure 3) removes fumes at source during the furnace operation. It also provides optimum viewing to easily align the capillary with the injection hole.

The 240Z AA comprises a Zeeman effect with longitudinal graphite tube heating and a Constant Temperature Zone (CTZ) design for best sensitivity.

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

Analytical conditions

Parameter	Setting
Hollow Cathode Lamp	Cadmium (part no. 5610100800)
Graphite tube platform	Omega (part no. 6310003700)
Wavelength	228.8 nm
Slit Width	0.5 nm
Lamp current	4 mA
Mode	Peak Area

Chemical modifier: 1 mL $\text{NH}_4\text{H}_2\text{PO}_4$ 5 g/L + 0.2 mL $\text{Mg}(\text{NO}_3)_2$ 10 g/L (Part No. 5190-8337 and 5190-8338)

Standard solution: 2.50 $\mu\text{g}/\text{L}$ Cd

Standard Reference Material: , SPS SW1: $0.50 \pm 0.01 \mu\text{g}/\text{L}$ Cd (from LGC Standards)

Method optimization

Dry steps were optimized by using the integrated camera (refer to Figure 1).



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

Ash and atomize temperatures were optimized by a chemometric method included in the instrument software, the Surface Response Methodology tool (SRM).

Tests were performed on a standard reference material and on a spiked water sample. All measurements were made using the Peak Area calculation.

With the SRM Wizard, only 3 clicks were needed to develop the model and, based on the results of the chemometric analysis, the Surface Response Methodology tool determined the optimum conditions, shown in Tables 1 & 2 and in Figure 2.

Table 1. Experimental design factors

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

Table 2. The optimum conditions determined by SRM for the standard ad sample were very similar, indicating that the modifier mix was ideally suited for the application.

Temperature	Standard	Sample
Ash (°C)	570	568
Atomize (°C)	1533	1577

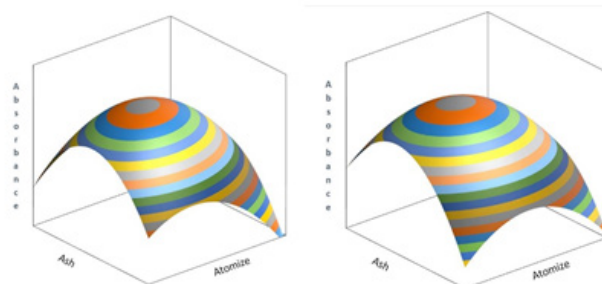


Figure 2. The Surface Response Methodology tool in the software uses chemometrics to automatically optimize the ash and atomize temperatures. Shown here is the plot for the standard reference material (left) and the spiked sample (right).

Results

- Characteristic concentration in peak area: 0.035 µg/L.
- Characteristic Mass in peak area: 0.57 pg
- Instrument Detection Limit for 20 µL: 0.011 µg/L
- % recovery SPS-SW1: 98.1 %
- % recovery bottled water spiked with 1.0 µg/L: 101.7 %

Conclusion

The Agilent 240Z system delivered an excellent recovery and detection limit for cadmium in drinking water, with the method being applicable for a wide range of worldwide standards.

With the most sensitive Zeeman workhead, local fume extraction, Tube-Cam and SRM wizard, the 240Z provides a very easy way to determine the appropriate furnace conditions. It is a cost effective solution, lowering argon consumption and increasing graphite tube lifetime. The system is a cost effective and accurate solution for laboratories routinely performing such testing.

References

1. World Health Organization 2011, WHO/SDE/WSH/03.04/80/Rev/1 - Cadmium in Drinking-water
2. Optimizing GFAAS ashing and atomizing temperatures using Surface Response Methodology, Agilent publication number 5991-9156EN.

More information

The optional fume extraction accessory (Figure 3) includes a mirror and lights to provide a clear view of the injection hole, making alignment of the autosampler capillary easy. The accessory connects to the exhaust system to remove vapors at the source.

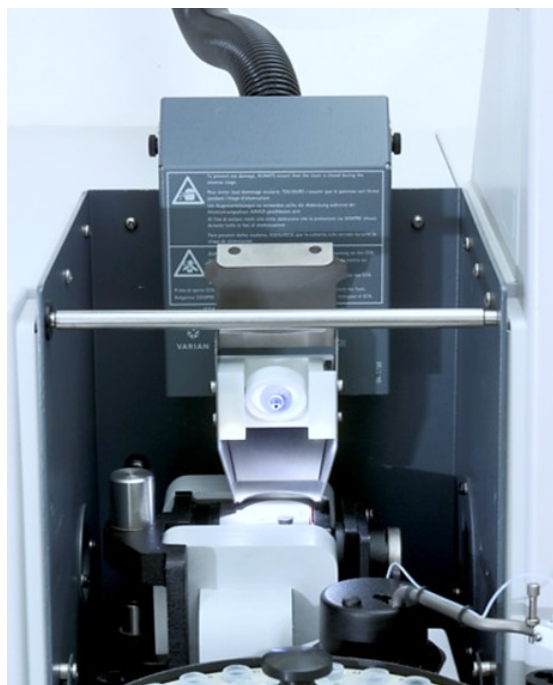


Figure 3. The optional extraction/LED accessory for the Agilent furnace.

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