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Application News

No.A506

Spectrophotometric Analysis

Analysis of the Lead Content of a Coffee Beverage by Graphite Furnace Atomic Absorption Spectrometry and Introduction of the GFA-TV Graphite Furnace Atomizer Camera

Introduction

Graphite furnace atomic absorption spectrometry (GFAAS, electrically heated atomic absorption, flameless atomic absorption) is capable of highly sensitive analysis of many different elements. GFAAS can also remove organic materials in particular from samples during analysis as it performs atomization via drying and ashing steps using a temperature program. GFAAS can consequently be used to analyze beverages, water-soluble foods, biological samples, organic solvents, and water-soluble organic compounds without needing to perform degradation pretreatments. However, the properties of these untreated samples differ from those of aqueous solutions subjected to degradation pretreatment, and corresponding care is needed during sample injection and drying.
This Application News describes the direct analysis of lead in a commercially available coffee beverage without pretreatment using GFAAS, and introduce the GFA-TV graphite furnace atomizer camera–a convenient optional product that allows observation inside the furnace during sample injection and drying.

Analytical Samples and Analysis Method

The amount of lead (Pb) in a commercially available coffee beverage was measured using a standard spiking method. Spiked concentrations were 10, 20, and 30 ppb.

Analysis Method and Conditions

Analysis was performed using an AA-7000 atomic absorption spectrophotometer connected to a GFA-7000 graphite furnace atomizer and an ASC-7000 autosampler.
The main analytical conditions are shown in Table 1. The sample injection volume was 10 µL, and 5 µL of palladium nitrate solution was added automatically (Pd concentration 100 mg/L) as a matrix modifier.

Table 1 Main Analytical Conditions

Element Measured	Pb
Analysis Wavelength	283.3 nm
Slit Width	0.7 nm
Lighting Mode	BGC-SR
Temperature Program	<table><tr><th></th><th>Temp.</th><th>Time (sec)</th><th>Heat Mode</th><th>Flow Rate</th></tr><tr><td>1</td><td>60</td><td>3</td><td>RAMP</td><td>0.10</td></tr><tr><td>2</td><td>120</td><td>10</td><td>RAMP</td><td>0.10</td></tr><tr><td>3</td><td>250</td><td>10</td><td>RAMP</td><td>0.10</td></tr><tr><td>4</td><td>400</td><td>10</td><td>RAMP</td><td>0.20</td></tr><tr><td>5</td><td>400</td><td>15</td><td>STEP</td><td>0.20</td></tr><tr><td>6</td><td>1200</td><td>10</td><td>RAMP</td><td>0.20</td></tr><tr><td>7</td><td>1200</td><td>15</td><td>STEP</td><td>0.20</td></tr><tr><td>8</td><td>1200</td><td>3</td><td>STEP</td><td>0.00</td></tr><tr><td>9</td><td>2200</td><td>5</td><td>STEP</td><td>0.00</td></tr><tr><td>10</td><td>2500</td><td>2</td><td>STEP</td><td>0.20</td></tr></table> Atomization stage No. 9
Graphite Tube	Platform

GFA-TV Graphite Furnace Atomizer Camera

Using a graphite furnace, a small amount of sample (from several µL to several tens of µL) is injected into a graphite tube and atomized by heating. Consequently, sample injection and drying conditions have a substantial effect on resulting measurements.
The furnace camera can be used to observe clearly the conditions inside the graphite tube in real time, and allows easy monitoring of adjustments to the nozzle injection position, and of sample injection and drying.

Fig. 1 shows an installed GFA-TV.
Fig. 2 shows the screen shown when adjusting nozzle position. Nozzle depth adjustment is made easy because of the ability to view images from the camera inside the furnace.
Fig. 3, 4, and 5 show images captured by the GFA-TV during observation of sample injection, drying, and ashing. Being able to observe injection and drying in real time simplifies the investigation of analytical conditions such as the temperature program and injection volume.

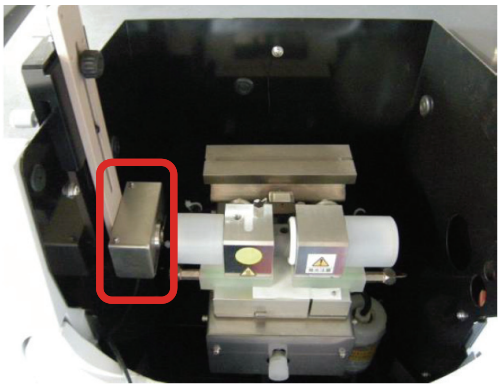

Fig. 1 An Installed GFA-TV

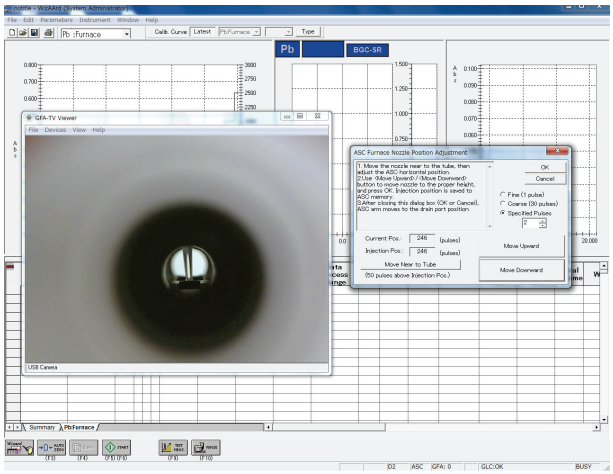

Fig. 2 Using the GFA-TV During Nozzle Position Adjustment

Fig. 3 to 5 show images captured by the GFA-TV during observation of sample injection, drying, and ashing, respectively.
Fig. 6 shows the peak profiles obtained for Pb in a coffee beverage, and Fig. 7 shows the calibration curve obtained for reference spiked samples.

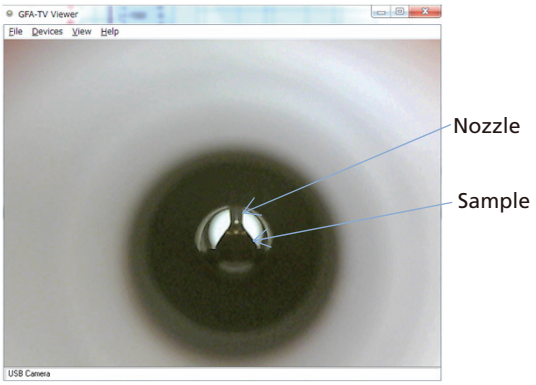

Fig. 3 Observing Injection with GFA-TV

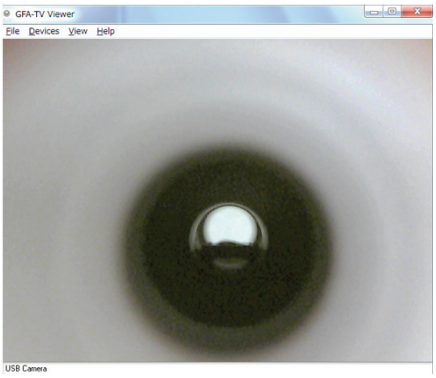

Fig. 4 Observing Drying with GFA-TV

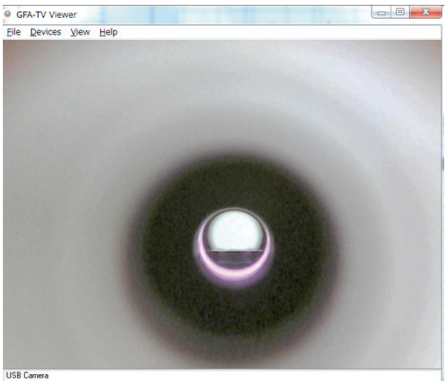

Fig. 5 Observing Ashing with GFA-TV

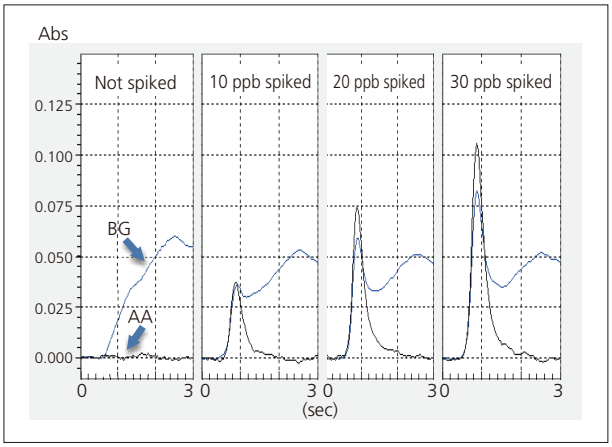

Fig. 6 Peak Profiles

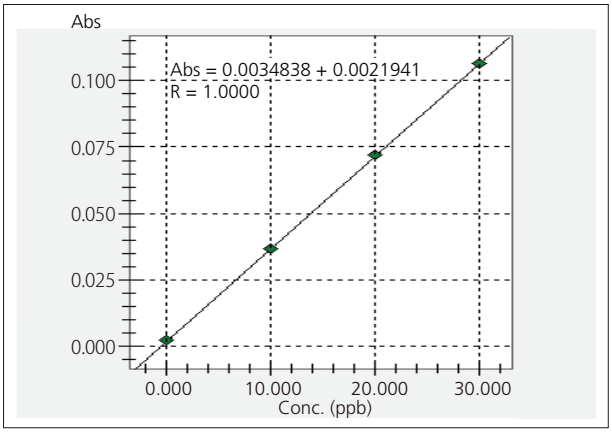

Fig. 7 Calibration Curve of Reference Spiked Concentrations

Analysis Results and Conclusion

We detected no Pb in the coffee beverage analyzed (below 3 ppb).
As shown in this experiment, a graphite furnace can be used to perform highly sensitive analysis without decomposition pretreatment of samples. This analytical method is suited to the measurement of small amounts of Na, Fe, and other elements for which contamination during pretreatment is a problem.
The GFA-TV graphite furnace atomizer camera also provides clear images of inside the graphite tube in real time that can be used to monitor nozzle injection position, sample injection, and sample drying, allowing for easy optimization of the injection position and temperature program.
The GFA-TV can be used not only with the AA-7000, but also with AA-6800, AA-6650, and AA-6300 instruments.

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