Application Brief

Environmental

Analysis of Pb and Cr in Soil using Graphite Furnace Atomic Absorption Spectrometry

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Heavy metal pollution

Heavy metal pollution is a serious environmental problem. Heavy metals enter soil through wastes, irrigation water, air pollution, and settling of airborne particles. By migrating and accumulating in crop products, they can work their way through the food chain.

The major heavy metal pollutants, lead (Pb) and chromium (Cr) are required to be quantified in soil during annual monitoring in China. The limits of these two elements in soil is strictly specified in the environmental standards for soil.

Quantifying Cr in soil

The large number of soil samples to be measured and the importance of accurate results drives the need for a simple, precise, and reliable analysis method. The determination of Cr in soil is often conducted by diphenylcarbazide spectrophotometry, flame atomic absorption spectrophotometry, inductively coupled plasma atomic emission spectrometry, or inductively coupled plasma mass spectrometry. The first two methods are the standard methods in China and the United States for determining Cr in soil.

Flame atomic absorption spectrophotometry is characterized by simple, easy, and fast operation, and is a standard method widely applied in the determination of Cr in soil. However, this method requires high flame temperature, leading to serious interference. Ammonium chloride can to be added to eliminate such interference, resulting in relatively low measured values for Cr. Graphite furnace atomic absorption spectroscopy (GFAAS) features high atomization temperature and high sensitivity. This method has become a standard method for the determination of Cr in food, aquatic products, and drinking water. The determination of Cr in soil and other media using the graphite furnace techqniue is specified in US EPA Method 7010. So far, China has not yet established a standard method for the determination of Cr in soil by graphite furnace atomic absorption method.

Quantifying Pb in soil

Graphite furnace atomic absorption spectrophotometry is commonly used to measure trace levels of Pb in soil. However, the analysis by graphite furnace atomic absorption often suffers from heavy interference due to background absorption caused by the complex composition of the soil matrix. This high background introduces error into measurements.

Agilent 280Z AA graphite furnace atomic absorption spectrometer was used to determine the Pb content in the soil. During the detection, the Zeeman background correction was applied to remove the background interference from the soil matrix and measure accurately the response for Pb in the soil samples.

This study aimed to establish a method for the determination of Cr in soil using GFAAS, which may fill the gap in existing standards. The study also aimed to improve the analysis of Pb in soil by addressing the matrix interference problems.

Analyses of Pb and Cr

In this study, a nitric acid–hydrogen peroxide–hydrofluoric acid mixture was used for the microwave digestion of a Chinese certified reference material of soil (GBW07409), followed by acid removal, and analysis on Agilent 280Z graphite furnace atomic absorption spectrometer, using the conditions listed in Table 1.

Table 1. Instrument conditions and temperature program.

Element	Wavelength (nm)	Slit width (nm)	Lamp current (mA)	Drying temperature (°C)/Time (s)	Ashing temperature (°C)/Time (s)	Atomization temperature (°C)/Time (s)	Temp during interference elimination (°C)/Time (s)	Argon flow rate (mL·min^{-1})	Injection volume (µL)
Pb	283.3	0.5	8.0	85–120/55	600/9.2	2100/2.9	2100/2	300	10
Cr	429.0	0.5	7.0	85–120/55	1000/8	2600/2.8	2600/2	300	10

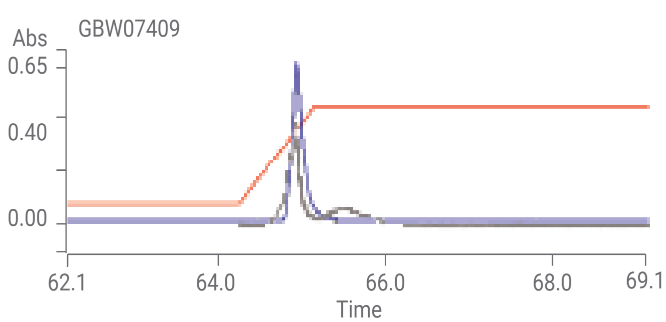

Figure 1. The signal generated by total Pb in 10 µL of the digest of reference soil material GBW07409 on Agilent 280Z graphite furnace atomic absorption spectrometer.

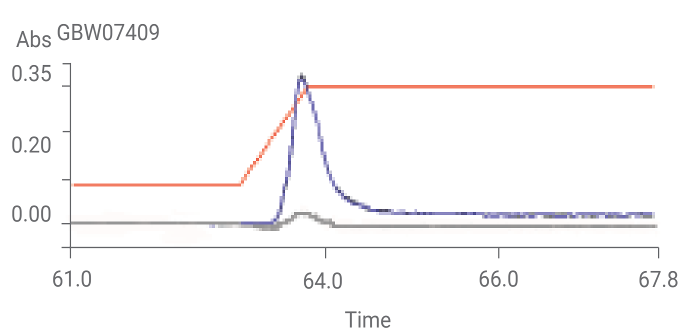

Figure 2. The signal generated by total Cr in 10 µL of the digest of reference soil material GBW07409 on Agilent 280Z graphite furnace atomic absorption spectrometer.

The samples were all injected into the graphite tube using the PSD 120 autosampler. The solution injection was monitored by the HD Tube-CAM that provides real time viewing of the samples throughout the analysis, important to control the drying and ashing steps. 20 g/L ammonium dihydrogenphosphate was used as the matrix modifier, which removed a large amount of organic matrix during the ashing process, and optimized the ashing temperature of Pb and Cr. The Agilent Zeeman background correction was applied to separate the Pb analytical signal from the background adsorption that couldn't be removed using modifiers. Optimum analytical conditions were found using the Surface Response Methodology tool in the SpectrAA software suppled with the Agilent 280Z. The tool finds the optimal relationship between the ashing temperature, atomization temperature and analyte absorbance. Figure 1 shows the a signal profile of the Pb peak, displaying the high sensitivity response for Pb (blue line) with freedom from the significant background interferences (gray line) that occur in soil samples.

The spectra of Cr in the sample are shown in Figure 2, which too shows high sensitivity and freedom from interferences.

The measured concentrations, compared to the certified Pb and Cr concentrations for the sample are shown in Table 2. The measured results were within ±10% of the certified values. These results show the effectiveness of the Agilent Zeeman background correction and the importance of an optimized furnace method.

Table 2. Analytical results of national certified reference material of soil (GBW07409).

Element	Certified value (mg/kg)	Mean measured value (mg/kg)	Recovery (%)
Pb	16.3±2.4	17.5	107
Cr	26.4±2.1	25.0	95

The results show the high sensitivity and accuracy of the method. The results also demonstrate that the Agilent 280Z graphite furnace atomic absorption spectrometer is capable of accurately quantifying samples with volumes as low as 10 µL. This method is able to complete the detection of one element in 3 minutes, which meets the needs of typical soil laboratories.

The analytical conditions developed with Surface Response Methodology tool and Tube-Cam feature provide the correct furnace method with the optimal drying, ashing, and atomization temperatures. This optimization ensures high sensitivity, high accuracy, and high reproducibility of analysis. The strong elimination of matrix interferences by Zeeman background correction makes Agilent 280Z graphite furnace system ideal for determining Pb and Cr in complex soil matrices. The instrument serves as a reliable and secure monitoring technology that supports the prevention and controlling efforts against soil pollution and helps make solid progresses in soil treatment and remediation.

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