Application Brief

Food Testing and Agriculture

Agilent

Trusted Answers

Determination of Multiple Elements in Aquatic Products Using a Graphite Furnace Atomic Absorption Spectroscopy

Monitoring pollutants in ecosystems

The measurement of heavy metals in fish and other seafood is required to monitor the presence of contaminants in the ecosystem as well as prior to consumption. Seafood products have a complex, salty composition. This complex matrix causes significant matrix effects when analyzing samples with a graphite furnace AA system (GFAAS), requiring accurate background correction.

In this study, Pb, Cd, Cu, Co, and Ni were measured in two standard reference materials: Tort 2 Lobster pancreas and CRM No 278R Mussel tissue. To optimize the analytical signal and remove chemical interferences, two matrix modifiers were used.

Removing matrix effects and automating standard and sample preparation

The samples were frozen and then dried and homogenized. 10 mg of the dried sample was added to 100 µL of nitric acid. The mixture was placed in a closed vessel and digested at 80 °C for 3 hours, prior to cooling and diluting with ultra-pure water to bring the volume to 2 mL. The concentration of nitric acid in the sample solution was about 3% after the dilution.

An Agilent 240Z graphite furnace AA system with Transverse Zeeman Background Correction technology was used for the analysis, with the conditions shown in
Table 1. The longitudinally heated graphite furnace with the Constant Temperature Zone (CTZ) design creates a constant temperature in the graphite tube, providing uniform heating of the sample. The transverse AC modulated Zeeman background correction applies a uniform magnetic field across the atomizer, correcting the background absorption caused by complex samples.

The Agilent 240Z includes the PSD 120 autosampler, a standard feature on all Agilent GFAAS instruments. The PSD 120 can accurately prepare calibration standards and perform sample preparations including modifier addition and sample dilution. The operator simply prepares a single stock standard of the highest standard concentration, and the PSD 120 will automatically prepare the other standards to create a calibration curve. The PSD 120 can also automatically dilute overrange samples and present them for repeat analysis.

Result accuracy

Table 2 compares the measured concentrations with the certified concentrations supplied with the reference materials. The measured concentrations showed a high level of accuracy, being within ±10% of the certified values.

Figure 1 shows the signal from three of the analyte elements; Pb, Cd, and Ni in the mussel tissue sample. The complex matrix of seafood samples results in a large background signal (grey line). This background varies for each analyte at the different wavelengths and the different ash and atomization temperatures used. The Agilent 240Z using the transverse Zeeman background correction is able to correct this large background signal across the full wavelength range. The analyte peak (black line) shows the high sensitivity performance of the 240Z achieved using the CTZ with furnace optimized geometry.

Figure 2 shows the calibration curves for Pb, Cd and Ni, which were all prepared from a single bulk standard by the PSD 120 autosampler. Each calibration shows a high degree of linearity.

The results from about multiple replicate runs (as indicated in Table 2) were stable and reliable. The method is applicable to the determination of trace heavy metals in complex matrices such as aquatic products. As a minimal amount of sample was required for the preparation procedure, the method is very suitable for applications such as the monitoring of heavy metals in biological samples with a small portions of samples.

Table 1. Instrument conditions.

	Cd	Cu	Pb	Co	Ni	Cr
Wavelength (nm)	228.8	327.4	283.3	242.5	232.0	357.9
Lamp current (mA)	4.0	4.0	10.0	7.0	4.0	7.0
Slit width (nm)	0.5	0.5	0.5	0.2	0.2	0.2
Sample volume (µL)	5	10	20	40	20	10
Modifier 1* (µL)	5	-	5	-	8	5
Modifier 2** (µL)	5	-	5	-	4	5

Notes: Modifier 1: 0.4 mg/mL $Pd(NO_3)_2$
Modifier 2: 2 g/L $Mg(NO_3)_2$

Table 2. The certified concentrations and the measured concentrations of the two seafood reference materials.

Element	Tort-2 Certified value (mg/kg)	Tort-2 Measured value (mg/kg)	n*	Recovery (%)	CRM 278R Certified value(mg/kg)	278R Measured value (mg/kg)	n	Recovery (%)
Cd	26.7±0.6	25.7±0.92	45	96	0.348±0.007	0.31±0.01	54	90
Cu	106±10	109±4	50	103	9.45±0.13	9.1±0.4	53	96
Pb	0.35±0.13	0.36±0.04	47	103	2.00±0.04	1.8±0.1	51	91
Co	0.51±0.09	0.55±0.02	49	107	n/a	0.34±0.01	56	n/a
Ni	2.5±0.19	2.30±0.05	49	92	n/a	0.94±0.04	52	n/a

Notes: n: number of replicate runs

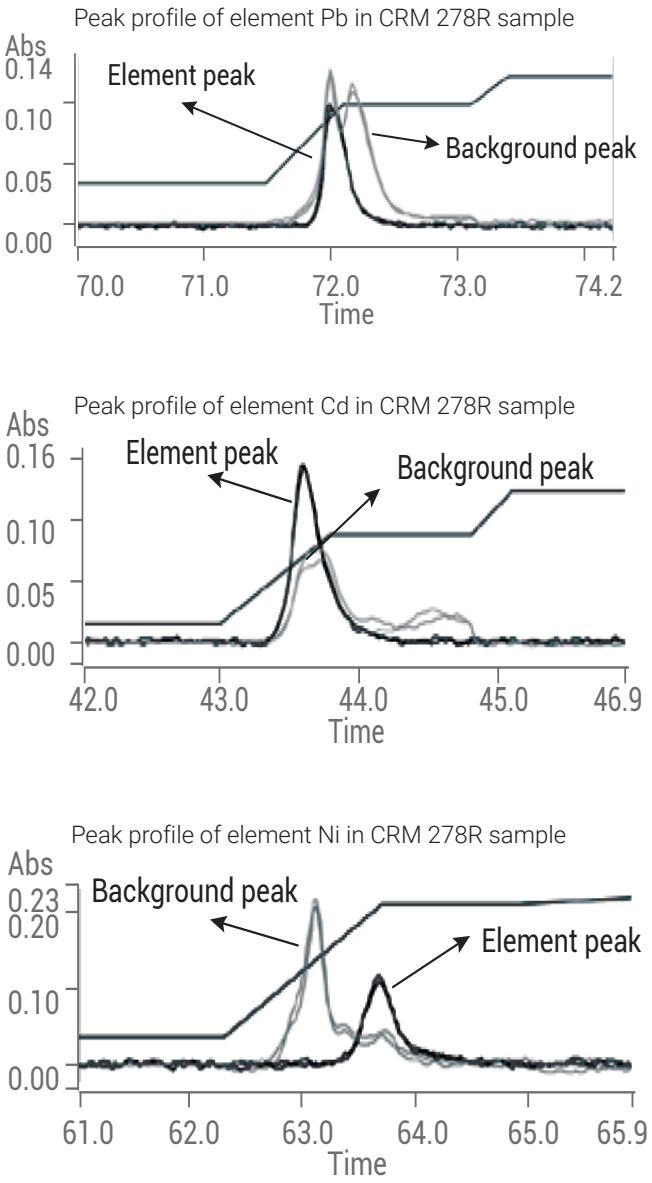

Figure 1. Peak signals for Pb, Cd and Ni in CRM 278R Mussel Tissue.

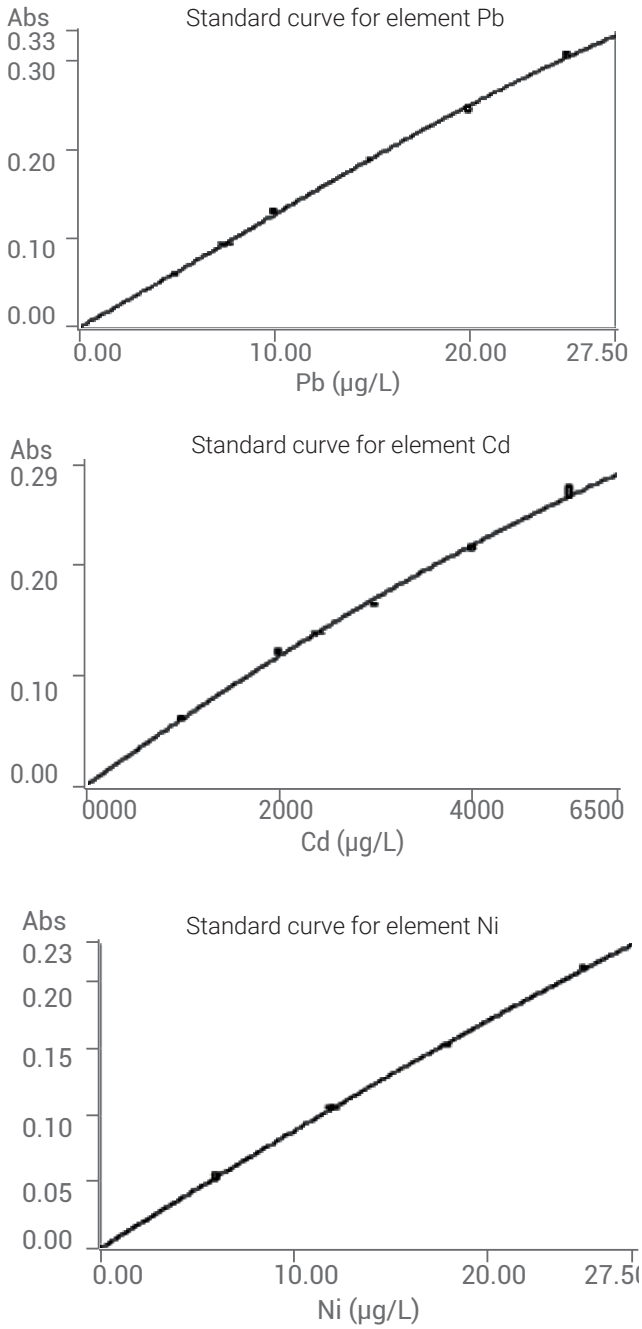

Figure 2. Calibration curves for Pb, Cd and Ni prepared using the PSD 120 autosampler.

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