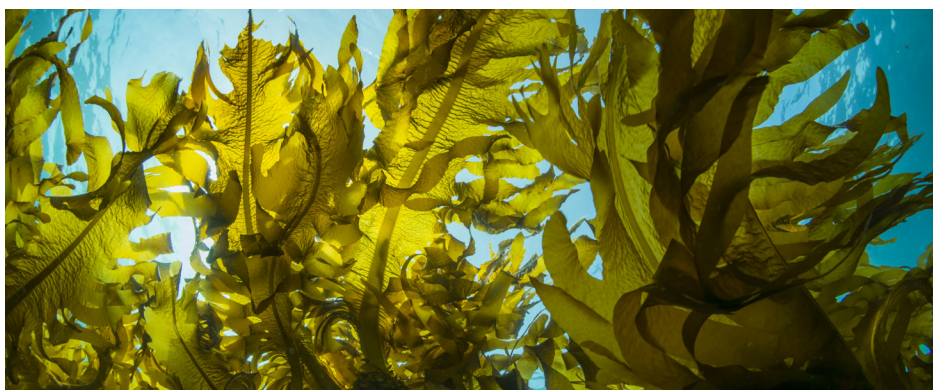**Agilent**

Trusted Answers

Determination of Pb and Cd in *Porphyra tenera* using Microwave Digestion and Graphite Furnace Atomic Absorption Spectrometry

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Measuring the impacts of offshore pollution

Urban industries are developing fast in China, especially the accelerated offshore deployment of heavy chemical industries, as well as petroleum and mining industries. These industries can discharge waste water and waste residues containing a large amount of heavy metals into the ocean, exacerbating marine pollution, especially in offshore waters.

Porphyra tenera is a type of algae that grows on offshore rocks. It contains abundant iodine, multiple vitamins, and microelements. Apart from being edible, it is also used in Chinese traditional medicine.

However, offshore pollution has caused serious threats to the *porphyra tenera* farming industry in recent years. Measuring the concentration of some elements in *porphyra tenera* is useful to evaluate the level of heavy metal pollution and the nutrient value of *porphyra tenera*.

Commonly used methods for detecting Pb and Cd in food include atomic absorption spectrometry (AAS), spectrophotometry–colorimetry, and inductively coupled plasma mass spectrometry (ICP-MS).

Graphite furnace atomic absorption spectrometry (GFAAS) has advantages of high precision and sensitivity in instrument detection, low detection limits, and using low sample volumes. GFAAS instruments are also relatively low cost.

When analyzing dehydrated marine products such as *porphyra tenera*, high levels of microelements and heavy metal elements can interfere with signal of the target elements. This interference results in large deviations in results.

The Agilent AA 280Z graphite furnace atomic absorption spectrometer features Zeeman background correction, which can correct for structured backgrounds and spectral interferences over the full wavelength range. The instrument can also add matrix modifiers online, which change the matrix composition of a sample. Modifiers can be used to adjust the volatility of the target elements to lower interferences, or to convert those elements to different chemical forms, thereby separating the background signal from the atomic absorption signals of the target elements. For complex matrices, matrix modifiers can be used to enhance the atomic absorption signal and reduce the background signal during the atomization stage.

In this study, samples of a certified reference material (GBW08521) were prepared using microwave digestion before graphite furnace atomic absorption method was used to determine the concentration of lead (Pb) and cadmium (Cd). The study aimed to determine if GFAAS can perform stable and accurate assay of elements in complex matrix food samples such as *porphyra tenera*.

Sample preparation

0.500 g of the sample was weighed into a microwave digestion vessel into which 5 mL of nitric acid was then added. The sample was digested according to the microwave digestion procedure under following conditions:

1. Heating to 120 °C in 5 min hold for 5 min;

2. Heating to 160 °C in 5 min hold for 10 min;

3. Heating to 180 °C in 5 min hold for 10 min.

The digestion vessel was removed after cooling, placed on an electric hotplate and heated to 140–160 °C until the sample was nearly dry, removing the acid. The digest was transferred to a 50 mL volumetric flask. The digestion vessel was rinsed 2 to 3 times with a small amount of water. The rinse solutions were then combined in the volumetric flask. The flask was then filled up to the mark with ultra pure water, and the solutions were well mixed for later use. A reagent blank was also prepared.

Standard curves

The Agilent AA 280Z Graphite Furnace Atomic Absorption spectrometer can automatically prepare multiple standard solutions from a single bulk concentration stock solution. Stock solutions of Pb and Cd (50 µg/L and 5.0 µg/L, respectively) were prepared using China national standard solutions of Pb and Cd. Ultra pure water was then used to prepare all the standards at the concentrations shown in Table 2 .

Zeeman background correction mode was used to remove spectral interferences. 5 µL of palladium nitrate was introduced as the matrix modifier with the sample solution. The instrument operating conditions shown in Table 1 were used.

Table 1. Instrument operating conditions.

Element	Wave-length (nm)	Slit width (nm)	Lamp current (mA)	Integration method	Ashing temperature (°C)	Atomization temperature (°C)
Pb	283.3	0.5	10	Peak area	600	2300
Cd	228.8	0.5	5	Peak area	500	2000

Analysis results of the standards can be seen in Table 3.

Table 2. Concentrations of standards (µg/L) and linearity of the calibration curves.

Element	Std 1	Std 2	Std 3	Std 4	Std 5	Correlation coefficient
Pb	5.0	10	20	40	50	0.9998
Cd	0.5	1.0	2.0	4.0	5.0	0.9993

The peak signal of the *porphyra tenera* samples were consistent with those of the standard solutions, and the absorbance of replicates had a relative standard deviation of less than 1%, indicating that the method is effective in controlling and reducing interferences. The results are shown in Figures 1 to 4.

Precision and accuracy

Ten samples of the *porphyra tenera* reference material (0.500 g each) were accurately weighed, digested, and analyzed. The relative standard deviation (%RSD) of the measurement of these replicate samples was then calculated, see Table 4.

Table 4. Precision and recovery (n = 6).

Element	Measured Value (mg/kg)	RSD (%)	Certified Value (mg/kg)	Mean Recovery (%)
Pb	0.78	1.35	0.81	96.3
Cd	5.1	0.48	5.2	98.0

Method suitability

The study results demonstrate that the Agilent 280Z method is able to effectively measure elements in complex dehydrated matrices using Zeeman background correction. The results of the national certified reference materials demonstrate high precision and accuracy, making the method suitable for monitoring coastal marine ecosystems as well as the quality and safety of *porphyra tenera* products.
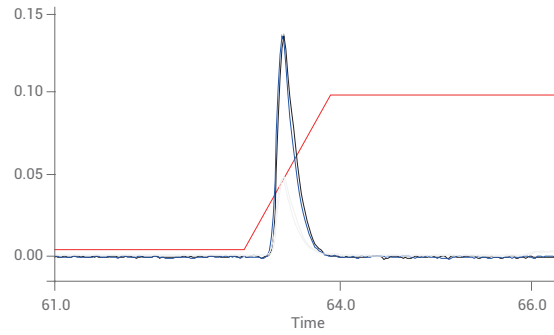

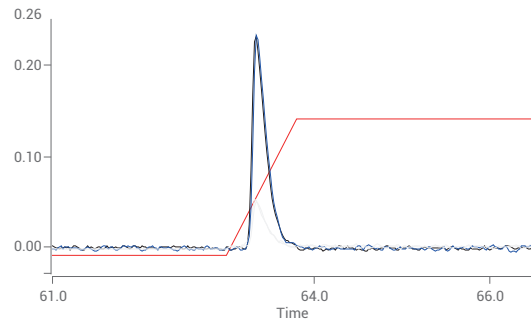**Figure 1.** Measured signal of Pb standard solution.
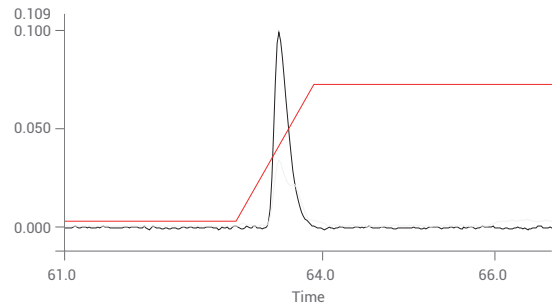

Figure 2. Measured signal of Cd standard solutions.

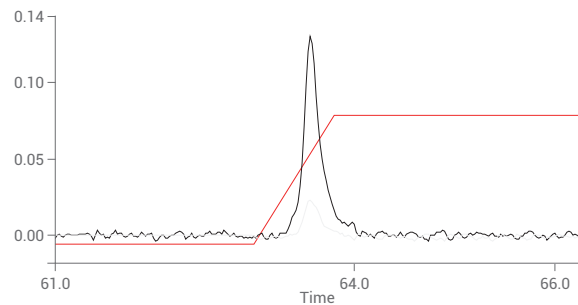**Figure 3.** Measured signal of Pb in Porphyra tenera samples.

Figure 4. Measured signal of Cd in Porphyra tenera samples.

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