Cadmium Determination in Crab Meat using Graphite Furnace Atomic Absorption Spectroscopy

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Key Words
Cadmium, crab meat, Food

Goal

This application note describes the analysis of cadmium in crab meat by graphite furnace atomic absorption spectroscopy (GFAAS) following microwave assisted acid digestion.

Introduction

Cadmium is a heavy metal that occurs naturally in the environment. However, waste incineration and use of fertilizers have increased the background levels of cadmium in water, soil, and living organisms[1]. Cadmium has a very toxic effect on the human kidney and can lead to bone demineralisation[2]. The European Food Safety Authority (EFSA) Panel on contaminants in the food chain established a tolerable weekly intake (TWI) for cadmium to be 2.5 µg per kilogram of bodyweight – this is an amount that can be consumed weekly over a lifetime without appreciable risk to health[3]. This information, put it in real terms for a person of 60 kg, means that they can consume up to 0.15 mg of cadmium per week with no ill effect.

A survey was carried out by the United Kingdom Food Standard Agency (FSA) to investigate cadmium levels present in crab meat across 399 samples in September 2013. The FSA found significant levels present in some of the samples analyzed. As a result of this survey the FSA has convened a working group to look at ways to reduce cadmium levels in meat from crabs during processing[3].

Method

Instrumentation

Graphite furnace atomic absorption spectrometry has a high sensitivity and is a recognised technique for cadmium analysis in a variety of sample matrices. A Thermo Scientific™ iCE™ 3500 AA was used for the GFAAS measurements of cadmium in different crab meat samples. The system allows cadmium determination in samples with complex matrix without affecting the data obtained. The Ash/Atomise wizard in the Thermo Scientific™ SOLAAR™ software was used to determine the spectrometer parameters for cadmium measurements (Figure 1).

The transient height signal measurement was selected and each measurement was performed in triplicate. The final set of spectrometer parameters used is shown in Table 1.

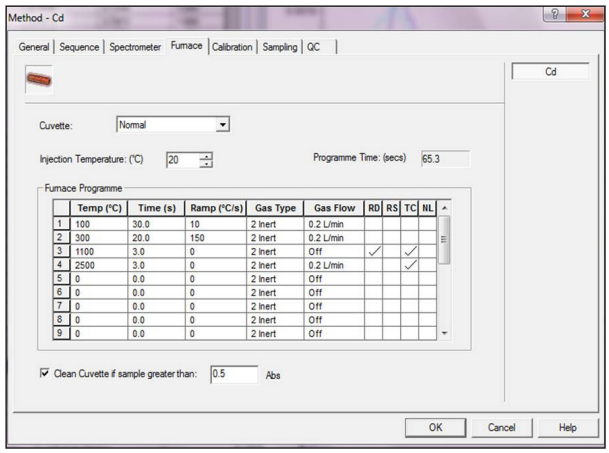

Figure 1: Furnace temperature parameters.

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Table 1. Instrument settings for the iCE 3500 AAS.

Parameter	Value
Wavelength	228.8 nm
Band pass	0.5 nm
Background Correction (BGC)	Combined BGC
Lamp Current	50%
Signal Measurement	Peak Height
Measurement Time	3 s
Replicates	3

It was expected that there would be significant background effects, which if not corrected could result in false readings. Combined Deuterium and Zeeman background correction was therefore used throughout to monitor possible interferences. Due to the challenging characteristics of the sample matrix, an extra drying step was added to facilitate complete drying of the samples.

Electrographite (Normal) cuvettes were used throughout the analysis. The final total injected/working volume used in this work was 20 µL. It was found to be beneficial to select the slow solution uptake and slow solution injection functions for all samples as some of the samples were more viscous than others. Intelligent dilution was used when analyzing samples to ensure all measurements were within the calibration range (Figure 2).

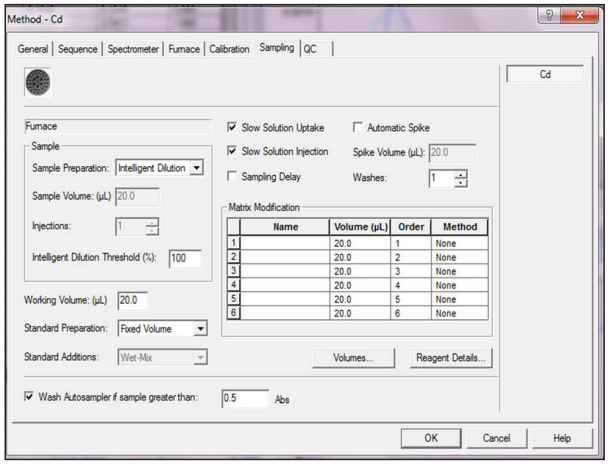

Figure 2: Final Sampling Parameters.

Sample Preparation

Standards

A cadmium stock standard solution containing 1000 mg/L of cadmium was diluted with a pre-mixed solution of deionised water and analytical grade concentrated nitric acid to provide a working standard of 10 µg/L in 2% (w/v) HNO_3. The calibration blank solution used throughout was a 2% w/v HNO_3 solution.

Samples

A total of 3 different brands of crab product were investigated in this study, representing all the main types of crab meat samples that are commercially available. The crab meat was weighed (1g) and transferred into a digestion vessel. The microwave digestion vessels containing the samples were placed in a fume extraction hood before adding concentrated HNO_3 (5ml) and DI ultra-pure water (4ml). The vessels were left for at least 30 minutes without their lids on to allow gases to escape. After this time the vessels were placed into a microwave digestion system and digested using the parameters in Table 2.

Table 2. Microwave digestion program used for the samples.

Step	Time (min)	Temp. 1 (°C)	Temp. 2 (°C)	Pressure (bar)	Power (W)
1	15	200	110	45	1500
2	15	200	110	45	1500

This procedure was used to prepare the sample blanks and spikes, and all the samples were prepared in duplicate unless stated otherwise.

Results

The calibration curve was obtained using the autosampler with the top standard of 10 µg/L normal method of quadratic least squares fit. Typical calibration curve obtained during the analysis using this method had an R^2 factor of 0.9995 or greater. An example of a typical calibration curve is given in Figure 3.

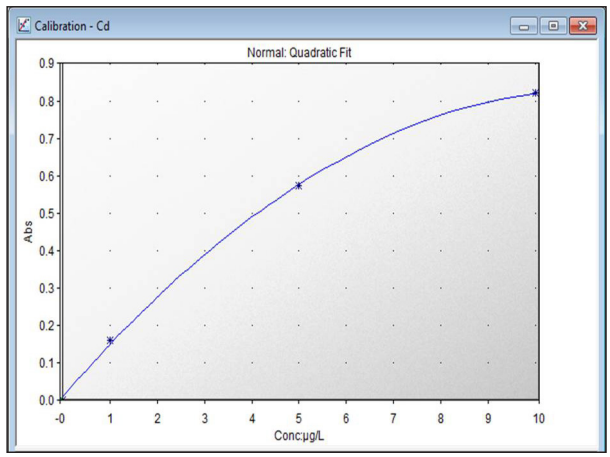

Figure 3: The calibration curve for cadmium.

The results indicate that these crab samples do exceed the recommended provisional tolerable weekly intake (PTWI) of 2.5 µg per kilogram of bodyweight for cadmium. The cadmium levels found in these types of food are high, however the average consumption of crab is relatively low and too sporadic to cause any real concern (Table 3).

Table 3. Results show the measured concentration of cadmium as well as expected and measured concentrations with percentage spike recovery for three separate crab samples (*all data was calculated from 3 replicate readings for each solution using transient peak height measurements).

Sample	Measured concentration sample (mg/kg)	Expected concentration spike (mg/kg)	Measured concentration spike (mg/kg)	Spike recovery (%)
Tinned crab*	0.0005	0.5005	0.4663	93.2
Dressed crab*	0.9641	1.46	1.4594	100.0
Crab pâté*	0.6143	1.06	1.0919	103.0

Conclusion

The Thermo Scientific iCE 3500 AA demonstrates an ideal solution for cadmium determination in crab samples. As a complete automatic system, the iCE 3500 AA is able to analyze cadmium in a wide range of crab meats with varying concentrations of salt, protein and sugar with the aid of acid digestion. The optimization wizards within the SOLAAR software make method development simple and ensure optimum analytical conditions. Significant background effects were accurately corrected for by the combined background correction included as standard in the iCE 3500 AA instrument.

References

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2. "Safety and Health Topics | Cadmium". Osha.gov. Retrieved 2013-07-08.
3. Food survey information sheet number 01/13 September 2013 cadmium in brown meat from crabs and products made with the brown meat from crabs sponsored by FSA (Food Standard Agency).

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