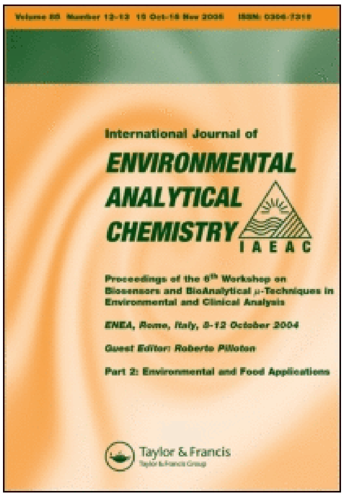

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The Analysis of Cadmium in Marine Samples

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The Analysis of Cadmium in Marine Samples

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This paper describes a procedure for extracting cadmium from seawater and from whole-organism digests of the gastropod mollusc *Mytilus planulatus* (common mussel) prior to analysis by atomic absorption spectrophotometry (AAS). The individual whole mussels were dehydrated to constant weight at 50°C, digested under simple reflux in nitric acid, and the solution buffered to pH 5 with sodium hydroxide and sodium citrate. The cadmium was extracted into dithizone in methyl isobutyl ketone and the organic layer stored in polyethylene containers for analysis by AAS. Seawater samples were treated in the same way, without dehydration. At each step in the sample preparation, the method was tested to ensure that no significant loss or contamination had occurred.

KEY WORDS: Cadmium, marine samples, solvent extraction, atomic absorption.

INTRODUCTION

Heavy metals in the marine environment have received increasing attention in recent years. This attention is a response to the increased knowledge of many undesirable effects of these metals on marine life, especially on organisms used as food for man. Cadmium has become a focus of attention because of its ubiquity and acute toxicity, but before research into its role in marine biological systems can be undertaken, a simple and reliable analytical technique is required. Methods for cadmium analysis in the parts per million range abound where the sample is in a soluble aqueous form, but the analysis of cadmium in marine samples requires sample preparation, concentration and separation from possible sources of interference. Where graphite furnace AAS is used, it has been shown that the presence of sodium chloride seriously interferes with the analysis of cadmium.[1] In addition, procedures must be adopted to minimize possible contamination and losses of cadmium during analysis. Loss and contamination can be caused by the use of high drying temperatures and inappropriate container materials.[2,3] The most suitable container material is polyethylene, which does not absorb cadmium in significant quantities.[4,5,6]

The method described (see Figure 1) uses wet ashing and solvent extraction, followed by atomic absorption spectrophotometric analysis to

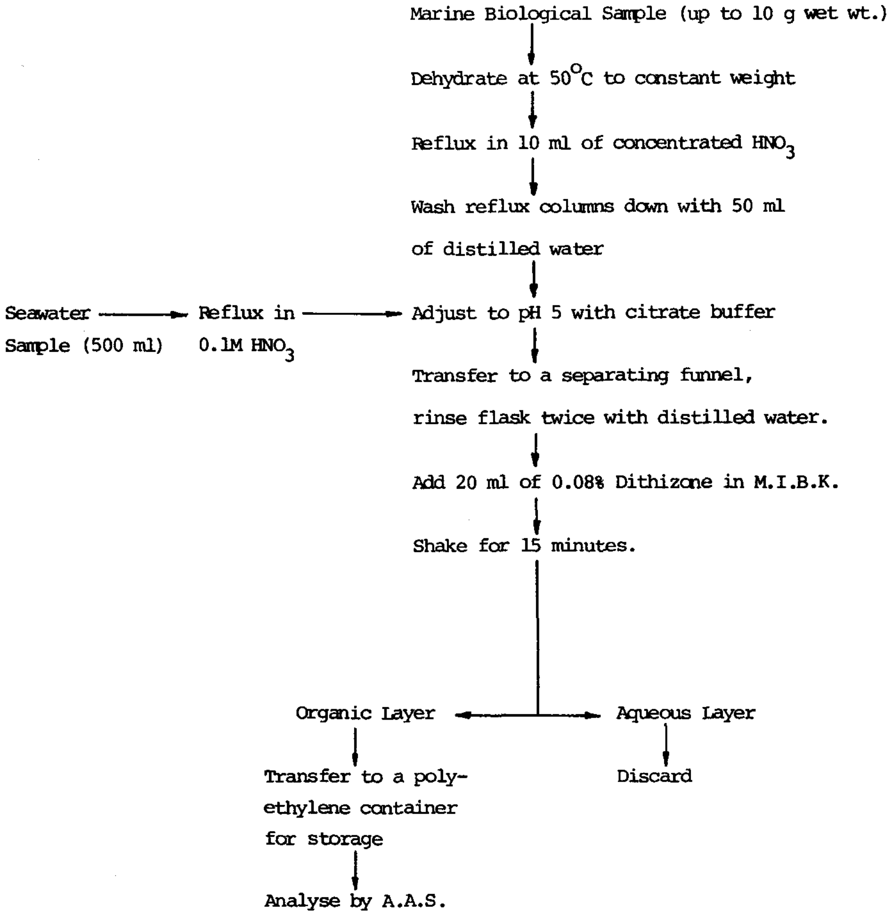

Figure 1 - Flow Chart showing procedure for sample preparation

provide a simple and reliable analytical technique for cadmium. This technique has been used to analyse trace cadmium concentrations in the mussel *Mytilus planulatus*, but is suitable for a wide range of biological materials.

EXPERIMENTAL

Sample preparation

Individual mussels (2–10 g wet weight), were dehydrated to constant weight (approx. 6 days) at 50°C, cooled, weighed and digested in 10 ml of nitric acid under simple reflux, (approx. 15 h). The digestion apparatus was washed with two 25 ml portions of distilled water to ensure maximum cadmium recovery and dissolve the white precipitate left after digestion. The pH was adjusted with sodium hydroxide and buffered to pH5† with sodium citrate. The cadmium in solution was then extracted into dithizone by shaking with 20 ml of 0.08% dithizone in methyl isobutyl ketone (MIBK) for 15 minutes. The cadmium in the MIBK layer was then analysed by either flame or carbon furnace AAS depending on the concentration.

At each step in the preparation, the optimum conditions were experimentally determined and rigorous procedures were adopted to ensure that no significant losses or contamination of sample had occurred. The extraction efficiency was found to be 98% under these conditions, which is consistent with an extraction constant[7] of $\log K = 2.14$. Complete experimental details of method validation can be obtained from the authors.

Standards

Analytical standards were prepared from a 1000 ppm stock solution of AR cadmium chloride in distilled water. These standards were then extracted by dithizone-MIBK in the same way as the sample digests.

Analysis

Samples in dithizone-MIBK were analysed on a Varian Techtron AA4 by AAS. In flame analysis, the sample was aspirated directly into an acetylene-air flame. For carbon-furnace analysis, a two-microlitre sample

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†A higher pH(6) would have been preferred, since extraction efficiency decreases below pH4, but a white precipitate forms which interferes with the extraction.

was placed into the carbon cup during the drying cycle, using an Oxford pipette.

ANALYTICAL RESULTS

Mussels collected from the same region of Port Phillip Bay (38° 19′ 50″ S, 144° 51′ 0″ E) were found to have a cadmium concentration of approximately 0.5 μg/g dry weight. A surprisingly high variability of ±0.4 μg/g was found in a group of 100 individuals ranging in size from 2–5 g dry weight with no correlation in cadmium concentration with size. Seawater samples from the same area were found to have a cadmium concentration of 0.05 to 0.1 ng/ml.

Acknowledgement

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