

Determination of mercury in sediment samples

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Introduction

Due to its high eco-toxicity and potential harmfulness to humans, much attention has been paid to the determination of mercury (Hg) and its monitoring is now a common consideration in most environment quality assessment. Cold vapour atomic absorption spectrometry (CVAAS) is the most widely used technique for this purpose because of its high sensitivity, low interferences and simplicity. The technique is commonly used in conjunction with of flow injection analysis (FIA) to improve sample throughput. Sequential injection (SI) analysis is sometimes used as an alternative to FIA. But it usually requires complex hardware configuration and control system. It also often achieve much lower sample through put and lower sensitivity.

Aim of study. To develop a simple and robust SIA system for a more efficient vapour generation of Hg; to optimise operation and performance, in terms of sample throughput, accuracy and precision, and reagent consumption. The apply the system to the quantification of Hg in a river sediment reference material and lake sediment samples.

Novel SIA design

A SIA system was assembled in our laboratory by using a peristaltic pump and two 3-way 2-position solenoid valves as illustrated below.

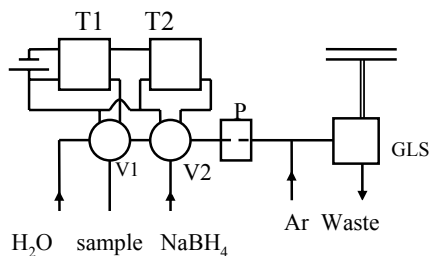


Figure 1. Diagram of the SIA/CV-AAS system. T1, T2, timer; V1, V2, solenoid valves; P, peristaltic pump; Ar, argon flow; GLS, gas-liquid separator.

Each of the valves was controlled by a digital timer. Tygon pump tubing of 0.065" in diameter was used for the pumping of liquid.

Analysis of mercury

Sediment sample was dried, homogenized and passed through 63 μm Nylon sieve. One gram of the sample was digested with Aqua regia. Hg was determined with the system with NaBH_4 as reductant and quantified by standard calibration.

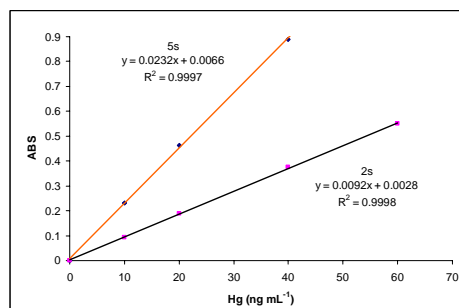


Figure 2 Calibration curves obtained for Hg with sampling times of 2 and 5 seconds

Results and discussion

A detection limit of 0.2 $\mu\text{g/L}$ was achieved with a precision of 1.8% and a throughput of 180 s h^{-1} ; dynamic (linear) range was 0 to 60 $\mu\text{g/L}$, better than previously. Lower sensitivity can be achieved by reducing sample aspiration time (Figure 2).

Application to a reference sediment material gave a good agreement with certified value.

Table 1 Variation of analytical results with reference material

Measured value in average (n=6, $\mu\text{g g}^{-1}$)	11.45 ± 0.32
RSD (% , n=6)	1.78
Certified value ($\mu\text{g g}^{-1}$)	11.31 ± 1.17
Recovery (%)	101.2 ± 2.8

Application to lake sediment samples

Hg in sediment samples collected from the Gippsland Lakes were determined

using the system and the results are shown in Figure 3.

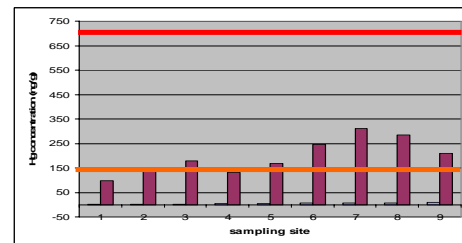


Figure 3 Spatial variation of Hg in Gippsland Lakes

The results in Figure 3 show that in most cases Hg level in sediment in Gippsland Lakes are above the effect range low value of 150 ng/g (indicated by the orange line in Fig.3) but well below the effect range median value of 710 ng/g (the red line) of Australia and New Zealand Sediment Quality Guideline . This means the aquatic organisms, including fish in the Lakes are to some extent in the risk of Hg contamination. Therefore, check of Hg in commercial aquatic products from the Lakes is necessary.

Conclusions

Simple and robust SIA system enabled substantial improvement in sensitivity and sample throughput for Hg determination by CVAAS. A dynamic (linear) range of 0-60 ng mL^{-1} was achieved with a sampling time of 2 seconds; A detection limit of 0.2 ng mL^{-1} was accomplished; Common heavy metals such as Cu, Pb, Zn, Co, Ni, Fe and Mn did not interfere with Hg measurement. The method was Successfully applied to the determination of mercury in sediment samples. Elevated Hg level in the sediment in Gippsland Lakes has been detected.

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