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Validation of a method for the analysis of total mercury in fish muscle by cold vapor atomic absorption spectroscopy (CVAAS) with a direct mercury analyzer (DMA)

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Mercury (Hg) pollution is a global environmental issue due to its adverse effects on human health and the environment. (1, 2, 3) Mercury can be released into various environmental compartments as a result of anthropogenic activities, such as gold mining (4), fossil fuel combustion (3), and burning of solid waste (5). This metal accumulates in living organisms and can reach toxic levels in the food chain, particularly in fish and other aquatic animals.

The Minamata Convention was adopted in 2013, and Costa Rica has been a signatory since 2016. This Convention aims to raise awareness about the health problems resulting from Hg exposure. Therefore, evaluation of this metal in environmental samples is essential to monitor and prevent human exposure.

In this context, direct analysis of Hg in fish samples has become a valuable tool for the evaluation of environmental contamination. Direct mercury analyzers (DMA) allow accurate and rapid measurement of mercury concentration in both solid and liquid samples, without the need for a prior digestion process. This significantly reduces analysis time and costs while increasing the accuracy and reproducibility of the results compared to Atomic Absorption Spectrometry (AAS), a reliable and widely-used technique for measuring metals, including mercury.

This study presents the validation of a method using DMA to determine total mercury in fish muscle. The equipment features detection cells of 1 cm and 10 cm to quantify high and low concentrations, respectively. The considered parameters were: linearity, limit of detection (LOD), limit of quantification (LOQ), intermediate repeatability, and recovery. The validation was performed on fish muscle using the European reference material (ERM-BB422) at both high and low concentrations.

The linearity of the method was verified through fitting the ordinary least squares (OLS) model and graphical analysis of the residuals. The calibration curves exhibited homoscedastic behavior across all calibration points as proven through a Bartlett test. A linear fit (r=0.99) was obtained for low and high concentrations evaluated, while sensitivities of the calibration curves remained constant over months Intermediate repeatability (sr) using spike samples was 90-105% for the low concentration range and 81-109% for the high concentration range, with both exhibiting a coefficient of variation (% CV: 7-14).

The limits of detection for the low and high level were 3.1 $\mu g \cdot kg^{-1}$ and 0.057 $\mu g \cdot kg^{-1}$ respectively. The limits of quantification were 9.4 $\mu g \cdot kg^{-1}$ and 0.174 $\mu g \cdot kg^{-1}$, respectively. These detection/quantification limits are low enough to measure concentrations relevant for fish in Costa Rica, considering that the maximum recommended concentration for mercury in fish is 500 $\mu g \cdot kg^{-1}$.

Finally, the recovery with low and high concentration using ERM-BB422 was (628.3 ± 0.4 $\mu g \cdot kg^{-1}$) and (629.781 ± 0.006 $\mu g \cdot kg^{-1}$) with 5% error for both concentrations.

The method demonstrated the necessary features for monitoring mercury and preventing human exposure in the target matrix.

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