

## Validation of Analytical Methods for Arsenic and Mercury Determination in Aquacultured Milkfish

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Toxic elements are considered one of the major sources of pollution in the aquatic environment. Aquacultural produce may be introduced to hazardous toxic elements, such as arsenic (As) and mercury (Hg) in surface water due to domestic and industrial wastes. To ensure food safety and consumer protection of milkfish (*Chanos chanos*) products in the Philippines, toxic elements in milkfish are being tested by chemical testing laboratories. The reliability and accuracy of the method of these laboratories can be demonstrated thru the z-scores obtained in joining Proficiency Testing (PT) schemes. The National Metrology Laboratory of the Philippines is a PT provider in the Philippines for toxic elements.

In this study, the method for As and Hg in milkfish were validated using Hydride Vapor Generator-Atomic Absorption Spectrophotometer (HVG-AAS) and Direct Mercury Analyzer (DMA) respectively. Validation parameters, such as linearity and working range, detection limit, repeatability, and trueness were evaluated based on AOAC criteria. The working range obtained for As via HVG-AAS is 0.53 – 2.68 µg/kg (R=0.9969) with a method detection limit (MDL) = 0.12 µg/kg and method quantitation limit (MQL) = 0.22 µg/kg. On the other hand, the working range for Hg via DMA is 0.52 – 100.9 µg/kg (R=1.000) with MDL = 0.31 µg/kg and MQL = 0.47 µg/kg. The repeatability expressed as RSD (n=7) for As (low level: 3.88%, high level: 1.38%) and Hg determination (low level: 7.30%, high level: 6.57%) passed the acceptance criteria based on the Horwitz equation. These values correspond to target concentration levels of As and Hg in the PT material. Trueness for As was done thru recovery by spiking (n=7) with recovery ranges of 80-110% and 97-102% for 50% and 200% spiking, respectively. For Hg, trueness by bias (n=7) was conducted using an APMP CRM and DORM-4 CRM with observed recovery ranges of 105-109% and 101-109%, respectively. The initial results for intermediate precision also showed minimal variations. In summary, the validated HVG-AAS and DMA methods proved to be reliable and efficient techniques for determining As and Hg in milkfish and are fit-for-purpose for assessment studies of As and Hg in milkfish PT material.

**Keywords:** method validation, arsenic, mercury, food safety, milkfish

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