

# Method Validation for the Determination of Total Mercury in Foodstuffs by Direct Mercury Analysis (DMA)

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Key words: Mercury, DMA, foodstuffs, method validation, rapid and precise.

Mercury is a toxic metal that can be transformed by bacteria into methylmercury, a more harmful and bioavailable form, which accumulates in the food chain, particularly in fish and shellfish. Monitoring mercury levels is essential for ensuring food safety, especially to protect vulnerable groups, such as pregnant women, due to its potential impact on foetal development and neurological function. As it is well known, most of the total mercury present in aquatic food is in the form of methylmercury, which reinforces the importance of accurate and reliable analysis, as it's the form most concerning to human health.

The purpose of this study was to validate a method for mercury measurements in food samples, to ensure its accuracy, reliability, and suitability, for applications in food safety, quality control, and regulatory compliance.

A method for total mercury determinations in food matrices was validated, using a Direct Mercury Analyser (DMA-80), based on thermal decomposition, amalgamation, and atomic absorption spectrometry (AAS).

The validated method covers two calibration ranges: from 2.5-15 ng and 30-1000 ng, using two separate calibration curves. Trueness, repeatability and intermediate precision (within-laboratory precision) were assessed using a certified reference material (CRM), NIST 1566b Oyster Tissue, and through interlaboratory comparison tests. Trueness, repeatability, and intermediate precision showed uncertainty values of 11%, 5%, and 8%, respectively. The detection and quantification limits were determined as 0.0014 mg.Kg<sup>-1</sup> and 0.0038 mg.Kg<sup>-1</sup>. These levels are significantly lower than the maximum levels established by Regulation (EU) 2023/915, which sets mercury limits of 0.1 mg.Kg<sup>-1</sup> for food supplements and salt, 0.3 or 0.5 mg.Kg<sup>-1</sup> for most fish, molluscs and bivalves, and 1.0 mg.Kg<sup>-1</sup> for large predatory fish. Quality control tools included blanks for contamination checks and two standards measured in each curve, during every experiment, to verify calibration accuracy. Additionally, CRMs must be within their established reference values, and interlaboratory test results must meet a z-score between -2 and 2 to be considered acceptable.

In conclusion, the analysed method is rapid and precise, requires minimal sample preparation, and is suitable for several matrices, with low detection limits and strong analytical performance.

#### References:

Commission Regulation (EU) 2023/915 of 25 April 2023 on maximum levels for certain contaminants in foodstuffs. Available at: <https://eur-lex.europa.eu/legal-content/EN/TXT/?uri=CELEX:32023R0915>.