

EXPANDING OPPORTUNITIES

Determination of Methylmercury in Fish and Seafood with the Milestone DMA-80



What is mercury speciation?

Mercury speciation is an analytical process that separates and measures the different mercury species in a test sample.

Mercury exists in three forms: elemental mercury, inorganic mercury compounds (primarily mercury chloride), and organic mercury compounds (primarily methyl mercury).

All forms are quite toxic, and each form exhibit different health effects.

Methylmercury

Methylmercury is the most highly researched form of mercury present in nature.

It is an organic mercury specie commonly found in fish and other animal tissues.

Methylmercury is mobile and easily absorbed, but it is difficult for the organisms to eliminate.

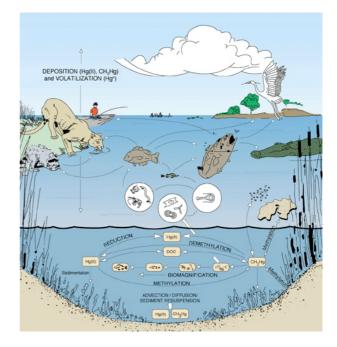
Instead, Methylmercury accumulates in biological tissues.

Animals higher on the food chain tend to have more Methylmercury in their tissues than those lower on the food chain.

This process is known as bioaccumulation.

Bioaccumulation can result in fish having over a million times higher Methylmercury concentration than the water in which they swim.

The significant bioaccumulation of Methylmercury in fish and seafood has resulted in a serious food safety problem.



The Mercury Cycle

Determination of Methylmercury

Through mercury speciation analysis, chemists gain access to critical data.

Understanding the ratio of Methylmercury to inorganic mercury facilitates a more complete understanding of toxic effects and risk to the environment and the human body. Several papers summarizing and discussing the different analytical approaches used to determine Methylmercury have been published. From an analytical point of view, Methylmercury determination is frequently performed by coupling gas chromatography (GC) or high performance liquid chromatography (HPLC) to different detectors such as electron impact-mass spectrometry (EI-MS), inductively coupled plasmamass spectrometry (ICP-MS), microwave induced plasma-atomic emission spectrometry (MIP-AES), cold vapor-atomic absorption spectrometry (CV-AAS) and cold vapor-atomic fluorescence spectrometry (CV-AFS).

> "Solid samples cannot be directly injected into a chromatographic system necessitating the extraction of mercury species from the samples. The mercury species are either leached out from the solid sample or the whole sample is digested or dissolved in acidic or alkaline media. As samples from non-polluted areas contain low mercury levels the risk of contaminating samples during sampling, storage and pre-treatment is large.

> Because of this, extreme precaution is necessary to avoid contamination from sample containers and reagents. As each step in an analysis may contribute to errors it is generally advantageous to include as few steps as possible."

On the reliability of methods for the determination of mercury based on chromatographic separation coupled to atomic spectrometric detection. Johanna Qvarnstrom, Umea University, Sweden

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A collaborative trial, IMEP-115, was conducted by the European Union Reference Laboratory for Heavy Metals in Feed and Food (EURL-HM), to determine the performance characteristics of an analytical method for the determination of methyl mercury in seafood.

The method is based on a double liquid-liquid extraction, first with an organic solvent and then with a cysteine solution.

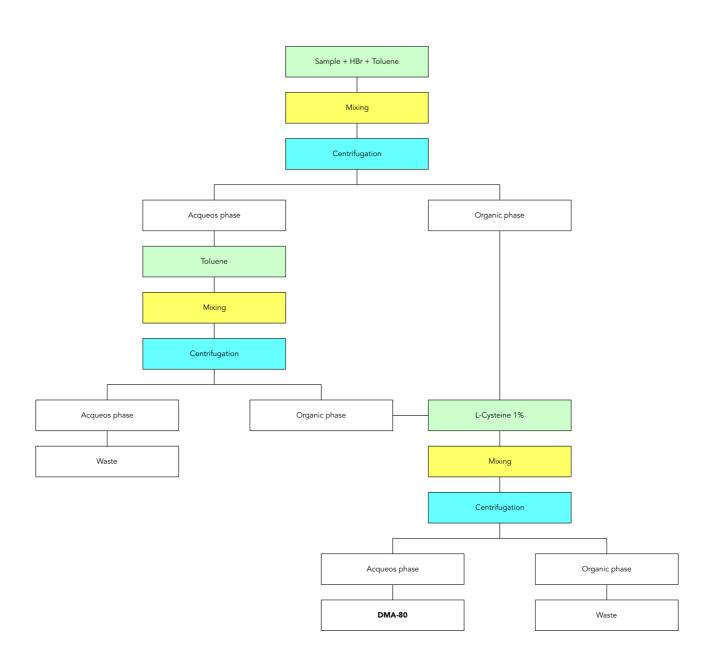
The final quantification is done with a direct mercury analyzer, and the Milestone DMA-80 is explicitly mentioned in the method.

The well-known advantages of the DMA-80 do apply also for mercury speciation, namely:

- No preliminary digestion step
- Ease of use
- High productivity
- Low cost of analysis

The analytical method would extract other organic mercury species in case may be present, but it can be all considered as Methylmercury because it represents almost the totally of the organic mercury in food samples.

A simplified diagram of the method is shown in this report.



IMEP-115 diagram

Results

Fifteen laboratories, from ten European countries, registered for participation.

All were experienced in the direct determination of mercury.

Five test items, covering a reasonable concentration range, were selected.

The five test items were certified reference materials: dogfish liver (NRCC, DOLT-4), lobster hepatopancreas (NRCC, TORT-2), organics in freeze-dried mussel tissue (NIST, SRM 2974a), oyster tissue (NIST, SRM 1566b) and tuna fish (EC-JRC-IRMM, ERM CE464).

The repeatability relative standard deviation ranged from 3.9 to 12.3 % while the reproducibility relative standard deviation ranged from 8.4 to 24.8 %.

The method demonstrates to have acceptable precision for all test materials, thus it fit for its intended analytical purpose.

Sample	DOLT-4 Dogfish Liver	TORT-2 Lobster Hepatopancre as	SRM 2974a Mussel Tissue	SRM 1566b Oyster Tissue	ERM CE464 Tuna Fish
Certified MeHg (mg/kg)	1,33 ± 0,13	0,152 ± 0,013	0,069 ± 0,0008	0,0132 ± 0,0007	5,12 ± 0,34
Found MeHg (mg/kg)	1,13 ± 0,40	0,147 ± 0,030	0,071 ± 0,016	0,019 ± 0,010	4,47 ± 0,76

Conclusions

As a result of the statistical evaluation of the present collaborative trial study, the proposed method fits its intended analytical purpose for the determination of methyl mercury by direct analysis in different seafood test samples.

The method proves to have adequate trueness and precision for the methyl mercury determination (expressed as Hg) ranging from 0.012 to higher than 5 mg/kg.

All method performance characteristics related to trueness and precision estimated in the present collaborative trial show values which are within acceptance levels as laid down in European legislation.

References

Report EUR 25830 EN. IMEP-115: Determination of Methylmercury in Seafood.

Joint Research Centre Technical Report. Determination of Methylmercury in seafood by direct mercury analysis: Standard operating procedure.



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