

Automated multiple Determination of Hg-Species in Marine Biota by GC-CVAFS¹ after TMAH Digestion² and Solvent Stripping

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Cold Vapour AFS can be run with high sensitivity, is easily to be handled and – last but not least – is one of the definitely low budget techniques. These characteristics make it be the detection method of choice in the special field of gas chromatographic speciation analysis of mercury in marine biota.

In order to complete this method's advantages, we did some efforts to find out a convenient serial digestion procedure and to make it's GC sampling mode fit for liquid injection. Complete speciation can be achieved in one unique procedure in which methylmercury, inorganic and elementary Hg as well as other volatile species are quantified. Varying some experimental details enables total mercury to be determined via one GC-Signal.

Our analytical procedures include following steps:

Speciation of methylmercury and Hg²⁺:

Digestion by TMAH, alkylation by STEB³ (alt.: STPB⁴), solvent-thermo stripping by N₂ into n-decane, automatic liquid injection, GC on a 5 µm wide-bore capillary column, AFS-detection as Hg⁰ via pyrolysis in a 920 °C quartz cell

Total mercury:

Generally cold vapour AAS is the method of choice for the determination of total mercury due to its quick and simple performance. Nevertheless GC-VCAFS can also be applied for this purpose via ethylation of Hg²⁺.

For both ethylated species limits of detection and quantification can be established in the sub-ng/g range. Thus the mercury burden of nearly every type of marine biota can be speciated quantitatively. Thus the up to now used measuring of merely total mercury in fish can be replaced or completed, providing more detailed analytical and toxicological information.

However: Due to the time consuming (because necessarily moderate) digestion conditions the total procedure of our speciation analysis takes all in all 18 hours time. This is much too long, when fresh fish has immediately to be checked prior to or while being marketed. In cases like these CV-AAS (available without any sample pre-treatment if necessary) should further on be preferred.

¹ GC-CVAFS: cold vapour atomic fluorescence spectrometry

² TMAH: tetra methyl ammonium hydroxide

³ STEB: sodium tetra ethyl borate

⁴ STPB: sodium tetra n-propyl borate