

Determination of methylmercury in fish muscle by GC-AFS

Rostislav Červenka^{1,2}, Josef Komárek^{1,2} and Veronika Czibulková²

¹Masaryk University, Faculty of Science, RECETOX, Kamenice 126/3, 625 00 Brno, Czech Republic

²Masaryk University, Faculty of Science, Department of Chemistry Kotlářská 2, 611 37 Brno, Czech Republic

Methylmercury is the most toxic form of mercury present in aquatic ecosystems. Ingestion of fish muscle forms an exposure pathway of mercury to humans and the determination of methylmercury in fish is therefore important. The widely used analytical procedures for methylmercury determination are based on the coupling of gas chromatography (GC) with element specific detection methods. These procedures involve a number of discrete analytical steps, comprising the extraction of the mercury species from a solid sample, preconcentration techniques, modification of analytes through derivatization, separation by chromatography and detection. Atomic fluorescence spectrometry (AFS) affords a high degree of element specificity.

In this study the techniques of headspace solid phase microextraction (SPME) and liquid – liquid extraction (LLE) for preconcentration and sampling of methylmercury in the injection port of gas chromatograph were compared. Aqueous derivatization with sodium tetrapropylborate was performed. Propylated analytes were by SPME headspace sampled with a polydimethylsiloxane-coated fused-silica fiber, by LLE extracted into isoctane phase. Both techniques are very reproducible. To detection AFS with pyrolysis of mercury species was used.

For the isolation of methylmercury ultrasonication and microwave-assisted extractions in the presence of extraction agents, such as mixture HCl and NaCl, KOH in methanol and tetramethylammonium hydroxide were tested. Extraction efficiency of the total mercury was determined using the AMA 254 mercury analyzer, extraction yields of methylmercury by GC-AFS. The precipitated matter appeared by treatment of sample for adjustment of pH to 5 and lower extraction yields caused probably by retention of methylmercury in precipitate were obtained. The pH of the sample is an important factor influencing derivatization. Two-fold microwave-assisted extraction, Folch method extraction (chloroform : methanol, 2:1), the use of three-fold extraction by acetone and enzymatic hydrolysis with protease were tested to increasing of extraction yields of methyl mercury. Certified Reference Material DORM-2 and real sample of fish muscle were analyzed.

Acknowledgement

This work was supported by project MSM 0021622412 of Ministry of Education, Youth and Sports of the Czech Republic.