Mercury speciation analysis in marine samples by HPLC-ICPMS

Mercury (Hg) is a naturally occurring element, which is found in the earth’s crust and can be released into the environment through both natural and anthropogenic processes. Mercury exists as elemental mercury (metallic), inorganic mercury and organic mercury (primarily methylmercury). Methylmercury is highly toxic, particularly to the nervous system, and the developing brain is thought to be the most sensitive target organ for methylmercury toxicity. Methylmercury bioaccumulates and biomagnifies along the food chain and it is the most common mercury species in fish and seafood. Human exposure to methylmercury is mainly from fish and other seafood consumption. A simple method for the determination of methylmercury in marine based foods and feeds has been developed and in-house validated. The applied HPLC-ICPMS method was inspired by Vallant et al (2007). Samples were extracted with 5 M hydrochloric acid by sonication. Hereby the protein-bound mercury species are released. The extracts were then centrifuged (10 min at 3170 x g) and the supernatant decanted (extraction step was repeated twice). The combined extracts were added 10 M sodium hydroxide to increase pH, following further dilution in the mobile phase and filtering prior to analysis. Analysis of mercury species were performed using HPLC-ICPMS equipped with a MicroMist nebuliser. Typical plasma conditions were 1500 W RF power, 15 l/min, 0.97 l/min and 0.17 l/min for plasma, carrier and makeup gas, respectively. Analysis was performed in the time resolved analysis mode monitoring the 202Hg, 198Hg, 35Cl (m/z) with 1 s (Hg) and 0.01 s (Cl) integration time per data point. Separation of inorganic mercury and methylmercury was obtained on a polymer-based cation-exchange column (150×2.1 mm id, 10 μm) using isocratic elution (0.2 ml/min at 40 °C). The mobile phase (pH~3) consisted of L-cysteine (0.5% w/w), pyridine (50 mmol/L), methanol (5% v/w) and formic acid (0.8% v/w). Total run time 10 min. External calibration standards (0–10 μg/L) were run before and after the samples in order to quantify the methylmercury species by peak height (m/z 202). The methylmercury method was validated by triplicate analysis of certified reference materials (DORM-2, TORT-2 and DORM-3) and 4 other fish and feed samples of marine origin, repeated on 3 different days. The limit of detection and quantification were 0.027 and 0.054 mg/kg, respectively. The limits were calculated as three and ten times the standard deviation at intra-laboratory reproducibility conditions of a natural fortified sample with low content (0.06 mg/kg) divided by average recoveries for certified reference materials. Mean recoveries of the reference materials were 94–102%. The in-house reproducibility standard deviations were less than ≤12% for samples containing 0.15 to 4.47 mg/kg and less than ≤20% for samples with 0.06 mg/kg. Vallant B, Kadnar R and Goessler W (2007) J Anal Atom Spectrom 22, 322–325. Acknowledgement: Funding from the European Community's Seventh Framework Programme (FP7/2007-2013) under grant agreement n° 211326.

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