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Extraction of methylmercury from sediment for species-specific isotope ratio analysis

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Isotope ratio analysis (IRA) of mercury allows for the investigation of biogeochemical processes and source tracing of mercury in the environment. When the percentage of methylmercury is low in a sample, the isotope ratios can be significantly different than that of mercury [1]. Limited studies have investigated species-specific IRA of mercury due to analytical challenges with quantitatively isolating methylmercury from the matrix, especially soil and sediment. For precise isotope ratio measurements, a much larger quantity of methylmercury is required in comparison to quantification analysis [2]. Also, the extraction method must not cause fractionation [1]. We analysed previously published methods for extraction of methylmercury from soil and sediments and evaluated their suitability as an extraction method for IRA of methylmercury.

All the extractants were analysed using gas chromatography-multicollector inductively coupled plasma mass spectrometry (GC-MC-ICP-MS). The first method tested was a two-step acid extraction [3]. In this method, complex forming reagents were used to extract methylmercury back into a final aqueous solution used for measurements. However, used reagents caused matrix effects, and extracted methylmercury was not completely ethylated to be volatilized for gas chromatography. Acid extraction was therefore not considered a viable method for our method of analysis. Another method tested was an alkaline extraction using KOH-methanol [4]. This method was initially designed for ~0.5g of soil but was successfully scaled up to process larger quantities that may be needed to have sufficient mass of methylmercury for IRA. Eventually, distillation was also tested for isolation of methylmercury from sediment and was successfully scaled up for larger masses of soil. All tested methods were evaluated for fractionation of mercury isotopes during extraction. The development of an extraction method for methylmercury that accounts for the analytical challenges will allow for future investigations into the cycling of methylmercury in the environment.

[1] Donovan et al (2016), *Enviro. Sci. Tech.* 50, 1691-1702 [2] Dzurko, Foucher & Hintelmann (2009), *Anal. Bioanal. Chem*. 393, 345-355. [3] Masbou, Point & Sonke (2013), *Anal. Atom. Spec.* 28, 1620. [4] Tang, Tang & Lei (2022), *Enviro. Sci. 119*, 166–174.