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## **ACCEPTED MANUSCRIPT**

### Speciation of mercury in water and fish samples by HPLC-ICP-MS after

#### magnetic solid phase extraction

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### Abstract

In this paper, Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub>@ $\gamma$ -mercaptopropyltrimethoxysilane ( $\gamma$ -MPTS) magnetic nanoparticles was prepared and a new method of magnetic solid phase extraction (MSPE)-high performance liquid chromatography (HPLC)-inductively coupled plasma mass spectrometry (ICP-MS) was developed for the speciation of mercury including inorganic mercury (Hg<sup>2+</sup>), methylmercury (MeHg<sup>+</sup>) and phenylmercury (PhHg<sup>+</sup>) in environmental water, wastewater, tap water and fish samples. A rapid separation of three target mercury species was achieved in 8 min by employing relatively high ratio of methanol in HPLC mobile phase. Various parameters affecting Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub>@ $\gamma$ -MPTS-based MSPE of target mercury species have been investigated. Under the optimized conditions, the limits of detection for Hg<sup>2+</sup>, MeHg<sup>+</sup> and PhHg<sup>+</sup> were in the range of 0.49-0.74 ng L<sup>-1</sup>. The intra- and inter-day relative standard deviations (n=5) were less than 9.0% and 12%, respectively. The developed MSPE-HPLC-ICP-MS method was validated by the speciation of mercury in the Certified Reference Material of DORM-2 dogfish as well as real-world samples including environmental water, wastewater, tap water and fish samples, and it has the advantages of simple operation, rapid separation, high sensitivity, high enrichment factor and is suitable for the analysis of mercury species in samples with complex matrix.

Graphical abstract

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