DOI: http://doi.org/10.21698/simi.2025.ab44

MERCURY SPECIATION IN SEDIMENT SAMPLES USING HPLC-ICP-MS

Alexandru Paul Rus, Anda-Gabriela Tenea, Cristina Dinu, Ana Ioana Paris, Gabriela Geanina Vasile

National Research and Development Institute for Industrial Ecology – ECOIND Bucharest, 57 73 Drumul Podul Dambovitei, district 6, 060652, Bucharest, alexandru.rus@ecoind.ro, Romania

Keywords*: Hg^{2+}, methyl-Hg, sediment, speciation.*

Introduction

Mercury is a highly toxic element that poses significant risks to both the environment and human health, largely due to its ability to bioaccumulate. While environmental assessments and health risk evaluations often focus primary on total mercury (Hg) levels, it is important to identify the different chemical forms. In particular, alkylated mercury compounds—especially methylmercury (methyl-Hg)—are far more toxic than inorganic mercury species and should be prioritized in such analyses [1].

This study aimed to develop, optimize, and validate an analytical method for the simultaneous quantification of mercury species—namely inorganic mercury (Hg^{2+}) and methyl-Hg—in sediments.

Materials and methods

The analysis was conducted using a high-performance liquid chromatography system (Agilent 1260 Infinity II HPLC) coupled with an inductively coupled plasma mass spectrometer (Agilent 7850 ICP-MS). Chromatographic separation was achieved with a Thermo Scientific Hypersil Gold column (150 mm × 4.6 mm) operated at ambient temperature. $Hg(NO_3)_2$ in HNO_3 2 mol/L (Supelco, Germany) and methyl-Hg chloride 95% purity (LGC, Germany) were used as certified reference materials. 1 g/L L-cysteine represent the mobile phase.

For the pretreatment of sediment samples, the following reagents were used: 69% nitric acid, 0.5% hydrochloric acid, toluene, sodium sulphate, and sodium acetate. All reagents were HPLC-grade.

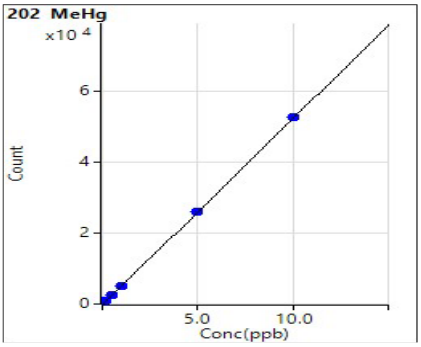
Results and conclusions

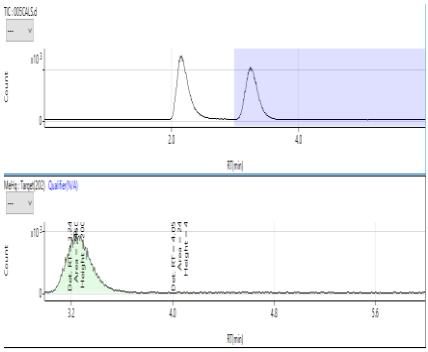
Calibration curves for both Hg^{2+} and methyl-Hg were established using a five-point linear range from 0.2 to 10.0 µg/L The mobile phase was delivered at a flow rate of 1.0 mL/min. The total run time per sample was 7 minutes (figures 1 (a) and 2 (a)). The chromatograms for Hg^{2+} and methyl-Hg are presented in figures 1 (b) and 2 (b).

The performance parameters were evaluated for 4 isotopes (199, 200, 201, 202).

The experimental validation data yielded the following results: a limit of quantification of 0.4 µg/kg, a precision of 8%, and an uncertainty of 22%. The best results were obtained for isotope 202 for both species.

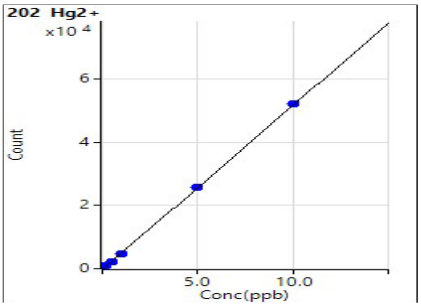
The method was applied on three different sediments collected from Olt River. The recovery percentage was situated in the range 74% ÷ 99%.

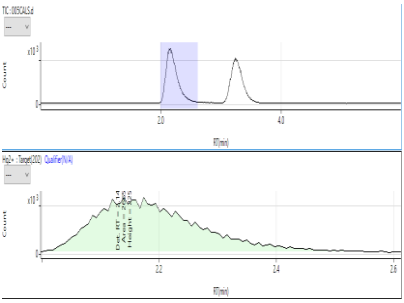




(a) y = 5258.3398 * x + 14.9667, R = 1.0000 (b) 5 µg/L (202) methyl-Hg Chromatogram

Figure 1. (a) Linear regression curve for methyl-Hg; (b) chromatogram for isotope 202





(a) y = 5198.1757*x, R = 1.0000 (b) 5 µg/L (202) Hg^{2+} Chromatogram

Figure 2. (a) Linear regression curve for Hg^{2+}; (b) chromatogram for isotope 202

The performance parameter values indicate that the method is suitable for sediment quality control.

References

[1] *Anil, A.S.; Alam, S.; Thakur, L.K.*, J. Food Compos. Anal., 129, 2024, https://doi.org/10.1016/j.jfca.2024.106092.

Acknowledgement

This work was carried out through the "Nucleu" Program within the National Research Development and Innovation Plan 2022-2027 with the support of Romanian Ministry of Research, Innovation and Digitalization, contract no. 3N/2022, Project code PN 23 22 01 01.