Mercury determination at trace levels using membrane preconcentration and benchtop total reflection X-ray fluorescence analysis

E. Marguí, I. Queralt, M. Guerra, N. Kallithrakas-Kontos

PII: S0584-8547(18)30029-6
DOI: doi:10.1016/j.sab.2018.07.020
Reference: SAB 5495

To appear in: *Spectrochimica Acta Part B: Atomic Spectroscopy*

Received date: 16 January 2018
Revised date: 13 June 2018
Accepted date: 16 July 2018

Please cite this article as: E. Marguí, I. Queralt, M. Guerra, N. Kallithrakas-Kontos , Mercury determination at trace levels using membrane preconcentration and benchtop total reflection X-ray fluorescence analysis. Sab (2018), doi:10.1016/j.sab.2018.07.020

Mercury determination at trace levels using membrane preconcentration and benchtop total reflection X-ray fluorescence analysis

E. Marguí[1]*, I. Queralt[2], M. Guerra[3], N. Kallithrakas-Kontos[4]

[1] *Department of Chemistry, University of Girona, Faculty of Sciences, C/M.Aurèlia Campmany, 69,17003 Girona, Spain*

[2] *Institute of Environmental Assessment and Water Research. IDAEA-CSIC. Jordi Girona 18-26, 08034 Barcelona, Spain.*

[3] *LIBPhys-UNL, Departamento de Física. Universidade Nova de Lisboa, 2829-516 Caparica, Portugal*

[4] *Analytical and Environmental Chemistry Laboratoy, Technical University of Crete, University Campus, GR-73100 Chania, Greece*

**Corresponding author. E-mail: eva.margui@udg.edu*

Abstract

At present, there is a considerable interest in Hg monitoring in water samples due to its widespread occurrence and the high toxicity of most of its compounds. Hg determination in water samples by means of total reflection X-ray fluorescence spectrometry (TXRF) entails some difficulties due to the high vapour pressure and low boiling point of this element that produce evaporation and loss of Hg from the surface of the reflector during the drying process, commonly employed in sample preparation in TXRF analysis.

In a former work we demonstrated the usefulness of PVC-based membranes functionalized with dithizone for Hg preconcentration. Limits of detection as low as 0.8 $\mu g \cdot L^{-1}$ Hg were obtained using a 1kW TXRF system. The main goal of the present research was to improve the extraction system and to apply it in combination with low power TXRF instrumentation (50W). In this sense, some extraction parameters as well as measurement conditions such as the volume of membrane deposited on the quartz reflector, the agitation speed during extraction, the aqueous sample volume and the measurement time were carefully evaluated to obtain the maximum sensitivity for Hg determination. Using the best analytical conditions, a limit of detection for Hg of 0.3 $\mu g \cdot L^{-1}$ was obtained which is adequate according to the limits of current legislation on the quality of water for human consumption. Additionally, a morphological study of the preconcentrated sample on the quartz reflector was performed by optical microscopy and information regarding Hg distribution along the membrane was obtained by µ-XRF analysis. It was found that Hg is not homogenously accumulated on the membrane and thus, the size of the membrane on the reflector is a critical parameter that has to be controlled to ensure the complete exposure of the loaded membrane to the X-ray beam. This fact is of special relevance when using this preconcentration approach in combination with micro-TXRF. Finally, to further prove applicability of the TXRF method for real water sample analysis, different types of spiked water samples were analysed.

Keywords: mercury, preconcentration, membranes, TXRF, water samples