Analysis of Platinum Group Elements in Water Samples by Energy Dispersive X-Ray Fluorescence

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Abstract

For the last years, Platinum Group Metals (PGM) have been included in the list of the ten most expensive metals.Their important applications in high-tech products as well as their excellent catalytic properties have attracted a particular interest in these metals, including in the recycling sector, while their potential environmental impact is increasingly being investigated [1]. Energy Dispersive X-Ray Fluorescence (EDXRF) could be a very interesting method for PGM analysis, especially from their L x-ray lines (for Pt, Ir, Os). Considering liquid samples, the expected concentrations are very low, so an improvement of the minimum detection limit is needed. In recent years, the application of novel complexation membranes has achieved an excellent improvement of these detection limits in the case of aqueous solutions (including seawater) [2]. In this work we present the first results from the application of this preconcentration method to the analysis of PGE; among them, emphasis was given on the analysis of platinum [3] which was used as a "paradigm" for all the others. At the same time, the experimental selections given, and the difficulties encountered in such analyses are highlighted.

Experimental

Liquid membrane was prepared and left to dry in different ways. The solidified membrane was immersed in aquatic solution spiked with Pt standard solution.

A very thin layer of free membrane (1cm* 1cm*1mm) swirling throughout the solution stirred by a magnetic stirrer and drops of the liquid membrane on the surface of the magnet stir (Fig.1) on the solution was examined among other ways, and gave adequate and similar results. The membrane was dissolved and left to be solidified again as a small spot on a cup with a thin Mylar bottom. Pt recovery was determined with EDXRF.

Figure 1: solidified drops on a magnetic stirrer

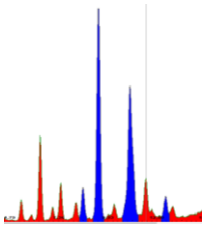

Figure 3. Analysis of 250 mL tap water spiked with 20 ppb Pt for 5 min. KCl at 40 g/L concentration was added in the sample (pH=4).

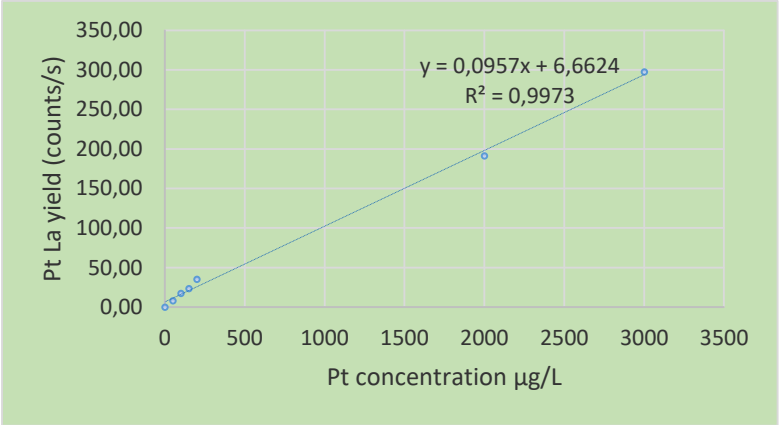

Figure 2: Linearity of the Pt La X-ray yield with the presence of KCl in acidified samples of tap water

Results and Conclusions

Figure 3 shows the spectrum from the analysis of tap water sample, doped with platinum of 20 ppb concentration under the conditions indicated in the figure caption. The La platinum yield is given in Fig 2. A very good linearity for a wide range of concentrations (0-3000 µg/L) was achieved.

The recovery of Pt was almost similar both in magnet deposition as well as with free membranes and was depending on the volume of the water.

In high aquatic volume (2 L) the minimum detection limit was 79 ppt while the recovery is less. In small aquatic volume (100 mL) there is a very good recovery (90 %), with a minimum detection limit of 320 ppt (Table 1).

The results were achieved for 24 hours equilibration time. The effort to improve is ongoing for all PGM.

Table 1: Recivery and minimum detection limit of Pt in 100 mL, 250 mL and 2 L water volume.

Drinking water spiked with Pt (20 ppb)	Recovery Pt	Minimum detection limit Pt in part per trillion (ppt)
100 mL	90%	320 ppt
250 mL	80%	160 ppt
2L		79 ppt

References

[1] I. Yakoumis,M. Panou, A.Moschovi, D. Panias, Clean. Eng. Technol. A Rev. (2021) 100112.

[2] N. Kallithrakas-Kontos, P. Boultadaki and S. Foteinis, Data in brief 29 (2020) 105236.

[3] G. Vlamaki, N. Kallithrakas-Kontos, I. Queralt, M. Hidalgo, X-Ray Spectrom. 2023 1-11