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Determination of Mercury in Real Samples Using a Gold-modified Glassy Carbon Electrode

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Mercury and its compounds are highly toxic, even at low concentrations, they accumulate in vital organs and tissues and cause kidney injury, central nervous system disorders, intellectual deterioration and even death. For these reasons, there is an increasing necessity for quantification of mercury in different samples, such as in environmental compartments, food, humans and pharmaceuticals. Therefore it is important to develop sensitive analytical methods for its determination.

The aim of this work is to evaluate the applicability on different real samples of a procedure for the determination of aqueous Hg(II) by anodic stripping voltammetry at a gold-modified glassy carbon electrode. Modification with gold was performed by dipping the electrode into a 50 mg/l HAuCl₄ solution and applying a potential of 0.80 V for 6 min. From a previous study¹ optimal values of the square wave parameters for the stripping scan were: step potential 0.004 V, frequency 150 Hz, amplitude 0.003 V. The highest signal with the best baseline shape was obtained with a deposition potential of 0 V. As expected, the height of Hg peak increased with increasing deposition time; a value of 120 s was found to be suitable for concentrations up to 50 µg/l. The renewable gold surface permits to eliminate memory effects, to maintain a stable baseline and response, and to avoid frequent mechanical cleaning steps.

An ocular lubricant gel containing 2x10⁻³% (w/w) of Thimerosal (sodium ethylmercurithiosalicylate, C₉H₉HgNaO₂S), equivalent to 0.98 mg of Hg for 100 g of gel, was purchased from a local chemist. The concentration of mercury found (1.00 ± 0.06 mg/100 g) was in good agreement with the expected values.

Some certified samples were analysed after dissolution in microwave oven: a) City Waste Incineration Ash (BCR 176, [Hg] = 31.4 mg/kg), b) Tuna Fish (ISPRA T 22, [Hg] = 4.43 mg/kg), c) Sea lettuce (BCR 279, [Hg] = 0.05 mg/kg). Two different solutions were tested for the digestion, 6 ml HNO₃/1 ml H₂O₂ and 3 ml HNO₃/3 ml H₂O₂: the latter permitted to obtain the best results. The concentrations found for Incineration Ash, [Hg] = 31.7 ± 3.4 mg/kg, and for Tuna Fish, [Hg] = 4.33 ± 0.38 mg/kg were in good agreement with the certified values. The sample of sea lettuce contains a great amount of I⁻, 154 mg/kg, that strongly interfered with voltammetric determination of Hg. The presence of I⁻ caused a decrease of the background current, because of its interaction with the gold surface, a sharp decrease of the mercury signal and a loss of linearity, which hindered the quantification of the analyte, due to the low solubility of Hg₂I₂ (K_{sp} = 1.1 × 10⁻²⁸)¹.

The determinations were performed also with cold vapor atomic absorption spectrometry and graphite furnace atomic absorption spectrometry to assess advantages and disadvantages of using voltammetry determination in comparison with other techniques.

¹ Abollino, O.; Giacomino, A.; Malandrino, M.; Piscioneri, G.; Mentasti, E.; *Electroanalysis*, 2008, 1, 75-83.