

VOLTAMMETRIC MICROCELL FOR TRACE ELEMENTS DETECTION

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The monitoring of trace metals in fresh and sea water is essential to a better understanding of the aquatic ecosystems. Previously, we have shown that the voltammetry stripping technique used with Ir/Hg microelectrodes arrays is ideally suited for in-situ trace elements monitoring [1].

In this paper, the development of a novel voltammetric system is reported. The key component of the system is a gel-integrated Hg-plated Ir voltammetric cell, coupled to a permeation liquid membrane (PLM) allowing in-situ separation[2]. PLM is based on a selective complexation of the analyte by a hydrophobic ligand (carrier) dissolved in a water immiscible organic solvent. This allows to improve the overall analytical performance of the system, in terms of detection limit and speciation capability.

The voltammetric microcell is realized on a Si/Si₃N₄ substrate by thin-film technology. It consists of eight Ir microdisks ($\varnothing 5\mu\text{m}$) and an Ir auxiliary electrode (about 1mm^2). A 20nm thin Ta adhesion layer is evaporated prior to the evaporation of a 130nm thin Ir layer. A 200nm thin Si₃N₄ top passivation layer is then deposited by low pressure chemical deposition. Each microdisk is located in a 32nL microwell formed by a 200μm thick SU8 ring. Then an antifouling agarose gel is deposited from a 1.5% agarose solution + 10 % C18 at 80°C into the microwells. Finally, a Hg microdrops are electrodeposited through the agarose gel. The cross-section of the voltammetric cell, showing the different technological steps, is depicted in figure 1. A picture of the device is given in figure 2.

Preliminary results are shown in figures 3. A concentration ([Cd]=750 nM) pulse is alternately flashed into the voltammetric cell. The measurements are performed by Square Wave Anodic Stripping Voltammetry (SWASV). The electrode stability vs. time was found to be excellent in the time range of about 9 hours (Fig.4).

Ref:

[1] J.Pei et al., Individually addressable gel-integrated voltammetric microelectrode array for high resolution measurement of concentration profiles at interfaces, Anal.Chem. 2001, 73, 2273-2281.

[2] J.Buffle et al., IUPAC series on "Analytical and Physical chemistry of environmental systems, vol.6, chapter 10 (2000), Wiley, Chichester.

Agarose gel
Hg μdisks
SU8 ring
LPCVD Si ₃ N ₄
Ta/Ir or Ti/Ir
LPCVD Si ₃ N ₄
Silicon

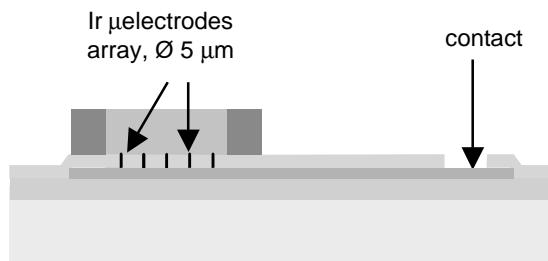


Fig.1: Schematic diagram of the cross-section of the voltammetric microcell

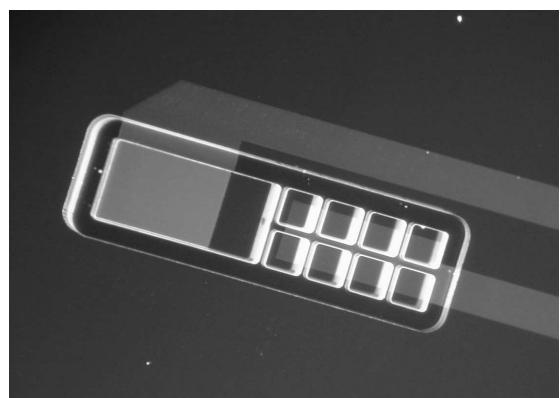


Fig.2: Photograph of the voltammetric microcell with eight Ir micro-electrodes and their corresponding 32 nL SU8 microwells, the larger Ir band (left) is an auxiliary electrode.

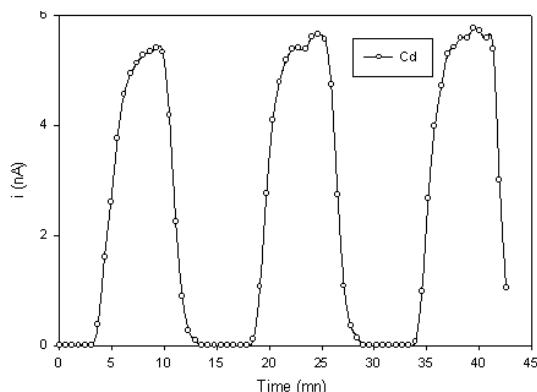


Fig.3: Alternate pulse concentration flowing into the voltammetric cell. The solutions were made of 10^{-2}M Na₄P₂O₇, pH=6.0, with and without

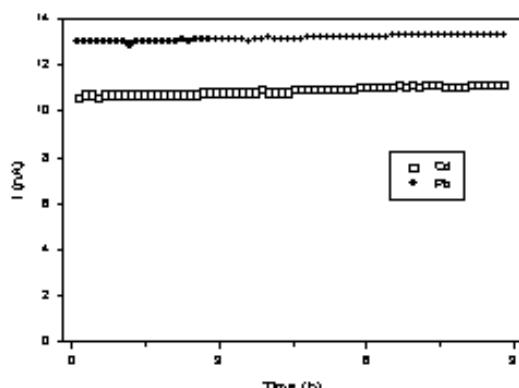


Fig.4: Electrode stability, measurements achieved in a batch solution with a 100nM concentration of Pb and of Cd.