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Title:

Accurate area determination of a solid gold electrode coupled with anodic stripping voltammetry for reliable measurements of mercury content in foodstuffs

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## Abstract:

Mercury is one of the most toxic elements even at ultratrace levels. Fish accumulates substantial concentrations of this metal in its tissues becoming the primary source of mercury in food chain for humans. For this reason, it is important to develop reliable methods for its determination. Electrochemistry offers attractive routine analysis capabilities, with low-cost instrumentation and applicability to *in situ* measurements. In particular, anodic stripping voltammetry (ASV), in specific conditions, has the characteristics to become a primary method of measurement, where all the variables are completely understood and traceable to International System of Units (SI). An effective determination of the available electrode area is the first factor to investigate in these measurements. The area of a solid gold electrode (SGE) was determined with two voltammetric techniques: one consists of cyclic voltage ramps at different scan rates, referred to the Randles-Sevcik equation; the other consists of voltage sweeps at different electrode rotation rates, based on the Levich equation. In both cases, a diffusion controlled process is developed. The redox system ferrocene/ferrocenium was chosen for its well-known reversible process of diffusioncontrolled single electron transfer. Acetonitrile and tetrabutylammonium esafluorophosphate were used as solvent and supporting electrolyte respectively. SGE, Ag/AgCl/KCl and graphite wire configuration was adopted.

The estimated areas were compared with that obtained by the elaboration of electrode images taken with a scanning electron microscopy.

ASV coupled with SGE was used firstly for the determination of mercury in tuna certified reference materials (BCR463 and BCR464) and, then, in fresh matrices.

Repeatability, linearity, accuracy and detection limit of the procedure were evaluated.

The medium exchange technique was also tested to reduce matrix effects. Consistent results were obtained.

Aim of this work is the establishment of the traceability to SI units of the ASV in order to apply it to food analysis.