notes and applications from Bioanalytical Systems, Inc.

Determination of Copper, Lead, Cadmium, and Zinc in River Water

Purpose

Environmental analysis of heavy metals in natural waters by stripping voltammetry has been reported thoroughly over the last few decades [1-3]. The determination of heavy metals by electrochemical methods in general is integral in many studies in pollution and toxicology, including such topics as the uptake, accumulation, transport, and toxic effects on organisms. Of particular interest is the amount of heavy metals to which mankind is exposed through bioaccumulation in the food chain [4].

One of the most useful electrochemical methods is differential pulse stripping voltammetry. DPSV differs from conventional linear sweep stripping voltammetry by its potential excitation waveform, which translates to lower detection limits and increased sensitivity. As with all anodic stripping voltammetry techniques, a potential is applied at which the metal species in solution will be reduced and concentrated onto the working electrode. The potential is then scanned positively and the metal is oxidized, and "stripped" off the electrode back into solution. The potential at which the metal strips can be used to qualitatively identify the metal. The resulting stripping current is proportional to the concentration of the metal being stripped and can be used for quantitative determinations.

Sample Preparation

A sample of river water and silt was obtained and diluted with 0.1 M HCl (Ultrex IITM, J.T. Baker) in a 4:1 ratio. A 5 mL aliquot of this solution was filtered and placed in a low-volume glass CGME cell (soaked overnight in 0.3 M nitric acid; rinsed with D.I. water). The sample in the cell was then deaerated for 10 minutes.

Conditions

The working electrode was a Controlled Growth Mercury Electrode (CGME), placed in the static drop mode (SMDE). An Ag/AgCl reference and platinum

auxiliary electrode were used along with a BAS 100B/W Electrochemical Workstation. The deposition potential was set at -1100 mV for 600 seconds and the potential was then scanned to +100 mV. A background check was made by deoxygenating a 5 mL aliquot of 0.1 M HCl and repeating the above procedure.

Results

A standard addition procedure was performed to quantitate the metals found in the initial scan. A stock solution was made such that a 10 uL aliquot would raise the concentration of the sample by 20 ppb Cu, Pb, Zn, and 5 ppb Cd when added to the cell. Three standard additions were made; the following voltammograms were recorded [F1] and calibration curves constructed. An example of the calibration curve is shown in F2.

The specified metals were present in the river water at the following concentrations:

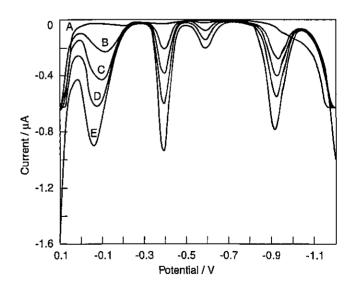


Figure 1. Voltammograms of (a) background, (b) sample, (c) addition 1, (d) addition 2, and (e) addition 3.

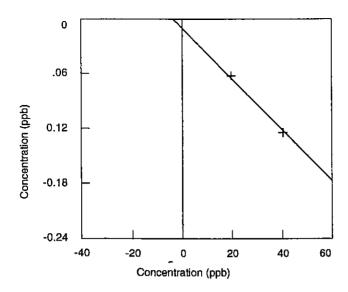


Figure 2. Example of standard addition curve (Cd shown).

References

- Wang, J. Stripping Analysis: Principles, Instrumentation, and Applications. 1985, VCH Publishers, Deerfield Beach, FL. pp 109-127.
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