BASi Tech Notes

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Lead in Lake Water: Using the Controlled Growth Mercury Electrode (CGME) and Gold Electrode with Differential Pulse Stripping Voltammetry (DPSV)

1018

Purpose

The concentration of Pb(II) in a water sample collected from the southern shoreline of Lake Michigan is determined using the BASi Controlled Growth Mercury Electrode (CGME) and a BASi 1.6mm diameter gold voltammetry electrode (MF-2014). Sensitivity, reproducibility, and ease of use of the electrodes are compared.

Anodic stripping voltammetry was used extensively for trace determination of heavy metals in natural and marine waters [1-3]. Atomic absorption spectroscopy (AAS) is more Widely used for these types of measurements, but requires more sample preparation than stripping analysis. Advantages of stripping voltammetry include superior detection limits, a high degree of reproducibility [4], and the possibility for on-site testing and speciation studies (AAS can only be used to determine the total amount of metal).

Instrumentation

All results were obtained using either the BAS CV-50 Voltammetric Analyzer or the BAS 100B/W Electrochemical Workstation and Differential Pulse Stripping Voltammetry (DPSV). DPSV differs from conventional linear sweep voltammetry by its potential excitation waveform. This difference translates to increased sensitivity and lower detection limits.

Procedure

Water samples were collected in a 125-mL PPCO (polypropylene copolymer) screw-top bottle and stored in a refrigerator for approximately two days prior to analysis.

Sixty microliters each of ammonium hydroxide (Optima™, Fischer) and concentrated HCI (Ultrex™ II, J.T. Baker) were added to 10 mL of lake water (final pH=4). These electrolytes were chosen due to their high degree of purity and for the ease of sample pretreatment. A background solution was made by the same method with D.L water used in place of lake water.

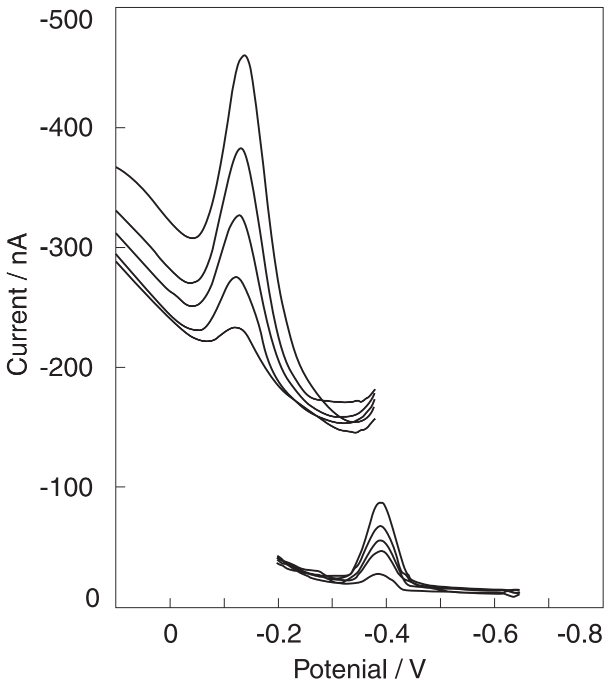
A platinum (auxiliary) and Ag/AgCI (reference) electrode were placed in a low-volume glass CGME cell. The lake water sample was transferred to the cell and deaerated with argon for approximately 10 minutes. The CGME in the static mercury drop electrode (SMDE) mode was used in conjunction with the BAS 100B/W. The DPSV technique was chosen and the experimental parameters are summarized in T1.

T1. Experimental parameters for DPSV.

Initial E (mV)	-650
Final E (mV)	+100
Scan Rate (mV/s)	4
Deposition Time (s)	120
Pulse Amplitude (mV)	50
Pulse Width (ms)	50
Pulse Period (ms)	2

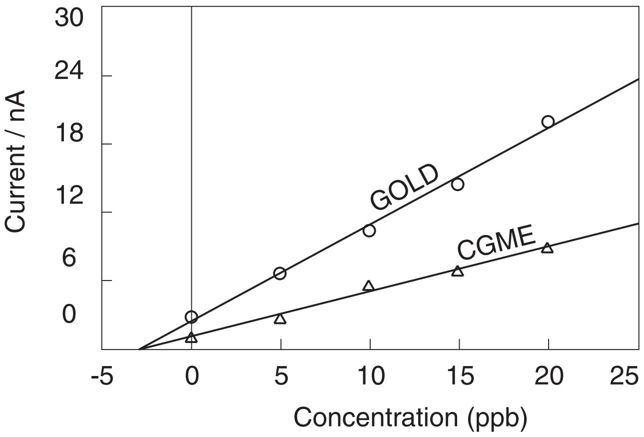
(Note: Initial potential is the same as the deposition potential.)

For all runs, the BASi 1.6mm diameter gold voltammetry electrode was placed in the sample cell and used under the same experimental parameters as the CGME. Each sample, including the background solution, was analyzed in a tandem sequence by the CGME and the gold disk. Lead concentration was determined by standard addition. Four additions, each of which increased the sample cell lead concentration by 5 parts per billion (ppb), were made. F1 shows the sample plus standard addition curves for voltammograms run on both the CGME and gold electrode. A standard addition curve was generated for each electrode, as shown in F2.



Fl. Voltammogram overlay of sample and standard additions for lake water sample. Peaks on the left obtained with the gold disk electrode; CGME results on the right.

F2. Standard addition curve for lake water sample using 1.6mm diameter gold electrode and the CGME.



Results

No lead was found in the background solution. A lead peak (-395 mV vs. Ag/AgCl with the CGME; 130 mV with the Au electrode) was found in the lake water sample; lead concentration was 2.4 ppb with the CGME and 2.7ppb with the gold disk. The slope (sensitivity) of the standard addition curve using the gold electrode was over twice that of the CGME. The difference in background currents is due to the different electrode materials and their relative surface areas. The main convenience associated with the CGME was the fresh electrode surface provided by each new drop, so no electrochemical cleaning was necessary. Although it was necessary to electrochemically clean the gold electrode after each run, its inherent sensitivity makes its use for lead determinations very enticing.

References

1) J.E Podolski and G.E. Glass, Anal. Chim. Acta 101 (1978):79.

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4) J. Wang, "Stripping Analysis: Principles, Instrumentation, and Applications," VCH Publishers, Deerfield Beach (1985).