CAPSULES
preliminary notes and applications from Bioanalytical Systems, Inc.

Anodic Stripping Voltammetry of Pb & Cd

Key Terms
Anodic Stripping Voltammetry, ASV, Trace Metal Ion Determination, Pb Determination, Cd Determination, CV-27 Voltammograph, BAS 100.

Anodic Stripping Voltammetry (ASV) can both qualitatively and quantitatively determine the metal ions in a variety of samples including hair, blood cells, urine, water, and electroplating baths (1). In this determination the metals of interest are first codeposited with mercury onto the electrode. The deposited metals are then stripped off into the solution by scanning the electrode potential. Determination of trace metals at sub-ppb levels is common with this technique. Stripping voltammetry has proved to be a sensitive, precise, and economic electroanalytical tool for detecting trace metals. Table 1 is a comparison between the sensitivities for ASV and atomic absorption for some elements readily determined by ASV (2). ASV can be done with either the CV-27 or the BAS 100.

The simplest potential wave form used for ASV is a linear sweep. Figure 1 shows a linear scan stripping voltammogram of 100 ppb Pb^{2+} and Cd^{2+} obtained with the CV-27 Voltammograph. This was a completely manual experiment.

The Anodic Stripping Voltammetry procedure can be fully automated with the BAS 100. Automation includes nitrogen purging, solution stirring, preconcentration, and triggering of the potential scan. In addition, sensitivity and detection limits can be improved by the availability of differential pulse and square wave sweeps. A differential pulse stripping voltammogram of 100 ppb Cd^{2+} and 100 ppb Pb^{2+} is shown in Figure 2. Differential pulse stripping voltammetry discriminates against background current better than linear scan voltammetry; however, lower scan rates employed increase the time of the experiment. Square wave techniques have similar sensitivities to differential pulse techniques but allow faster sweep rates.

Table 1. Comparison of ASV and Flameless AAS (2)

	Sensitivity, ng mL^{-1}	
Element	ASV	AAS
Ag	0.05	0.01
Bi	0.005	0.4
Cd	0.05	0.008
Cu	0.03	0.06
Hg	0.05	8
In	0.03	0.04
Pb	0.05	0.2
Sb	0.01	0.5
Sn	0.2	0.2
Ti	0.1	0.1
Zn	0.03	0.003

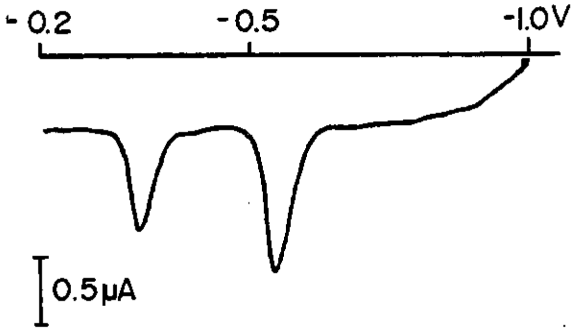

Figure 1. Linear sweep stripping voltammogram of 100 ppb Pb^{2+} and Cd^{2+} obtained with a CV-27. Deposition time 240 sec., scan rate 5 mV s^{-1}

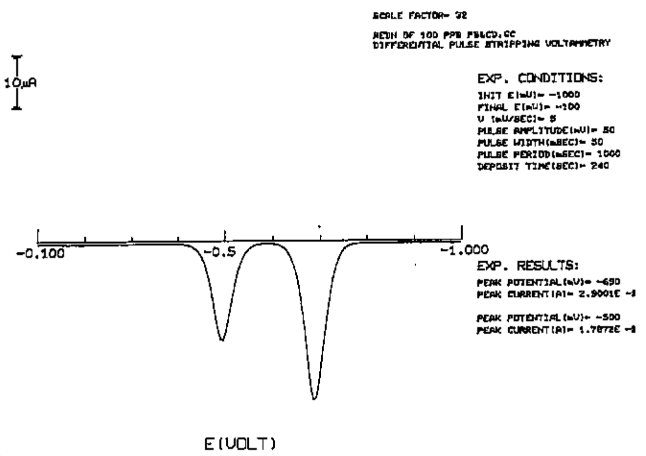

Figure 2. Differential pulse stripping voltammogram of 100 ppb Pb^{2+} and Cd^{2+} obtained with a BAS 100. Deposition time 240 sec., scan rate 5 mV s^{-1}.

Experimental Considerations

The stripping voltammograms were obtained with in situ deposition of mercury onto a stationary glassy carbon electrode.

All operating conditions, including the electrode position in the cell and the rotation speed of the stirring bar, must remain unchanged throughout the entire stripping experiment to obtain reproducible data.

Detection limits(which can be as low as 10^{-9} to 10^{-10} M) are limited by impurities in the background. To minimize background interference and sample contamination, clean the cell with concentrated HNO_3 and all other glassware with 6 M HNO_3 (3,4). Do not allow smoking in the laboratory. Buffer solutions used for sample dilution should be pre-electrolyzed over a mercury pool to remove trace metal ion impurities (use the BAS Pre-electrolysis Clean Up Cell).

For the determination of very low concentrations, longer deposition times can be used to increase the signal.

The stripping curve is usually quantitated by the standard-addition method. The sample is spiked with a solution of approximately the same concentration ratio of Pb^{2+} to Cd^{2+} as the sample.

References

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