CAPSULE 283

CAPSULES
preliminary notes and applications from Bioanalytical Systems, Inc.

Determination of Lead Using Differential Pulse Stripping Voltammetry: Lead in Paint

Purpose

The amount of lead in a sample of yellow paint pigment was determined using the Controlled Growth Mercury Electrode (CGME) and Differential Pulse Stripping Voltammetry (DPSV).

Electrochemical methods provide numerous advantages for the determination of trace metals in a wide variety of matrices. Unlike other methods, such as atomic absorption spectroscopy (AAS) and inductively coupled plasma mass spectrometry (ICP-MS), electrochemistry is relatively space- and cost-efficient [1]. As demand for on-site and point-source environmental analyses increases, so too does demand for portable, reliable instrumentation that can be operated by non-specialists. Electrochemical techniques are more than capable of meeting many of the needs of the environmental market. Products are currently being developed and tested in many environmental, industrial, and clinical settings [2]. Electrochemical methods, such as anodic stripping voltammetry (ASV), compare favorably with spectroscopic methods for both plant and animal tissue, blood, ground water and drinking water analyses [3,4].

One of the most discussed and insidious metals is lead. Introduced into the environment by many different means, it persists in nature and continues to contaminate living organisms in both rural and urban settings. A cumulative toxin, lead has been indicted in many cases of child poisoning, with effects ranging from mild retardation to death. In 1917, the medical community discovered that most cases of childhood lead poisoning are caused by the ingestion of paint chips [5].

Method

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

Procedure

SAFETY NOTE: All solutions containing HCl were prepared in a fume hood. Any dilutions involving concentrated HCl must be done slowly and very carefully to prevent fuming and spattering of solution.

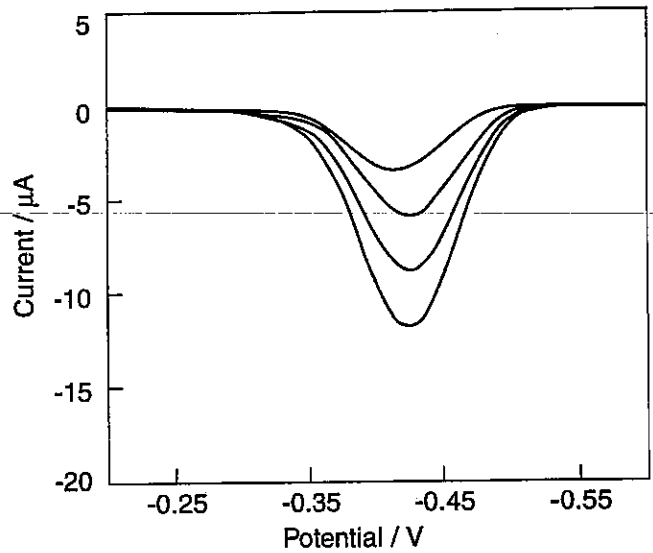
A sample of hardened nitrocellulose lacquer pigment (0.0046 g) was placed in a clean flask (soaked in 0.3 M nitric acid, rinsed with D.I. water) with 2 mL of 12 M hydrochloric acid (Ultrex™ II, J.T. Baker). The contents of the flask were stirred vigorously with a magnetic stir bar for approximately 4 hours. The contents were transferred to a 100-mL polypropylene volumetric flask and diluted to the mark with D.I. water. A blank of 0.24 M hydrochloric acid solution was used to generate a background voltammogram.

The working electrode was a BAS CGME, placed in the static mercury drop electrode (SMDE) mode. The CGME and Ag/AgCl reference and platinum wire auxiliary electrode were connected to the 100B/W Electrochemical Workstation. All solutions used in this experiment were deaerated by sparging with argon for 10 minutes. All glassware and plastic were soaked in 0.3 M nitric acid and rinsed with D.I. water prior to analysis. Experimental parameters are shown in T1.

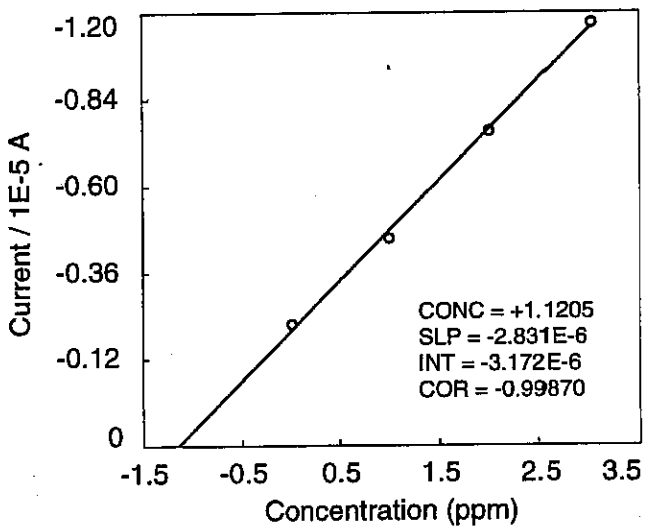
DEPOSITION E (mV)	-650
FINAL E (mV)	+100
SCAN RATE (mV/s)	4
DEPOSITION TIME (s)	120
PULSE AMPLITUDE (mV)	50
PULSE WIDTH (ms)	50
PULSE PERIOD (ms)	250

T1. Experimental Parameters for DPSV

No lead was found in the background solution. The amount of lead in the pigment solution was determined by use of a standard addition curve. After the initial voltammogram was recorded, an addition was made by micro-pipetting 10 µL of lead atomic absorption standard (1 mg Pb(II)/mL, Aldrich) into the sample solution. After stirring the solution thoroughly, the voltammogram was recorded for this and all subsequent standard additions (F1). By processing the data with the standard addition software on the 100B/W, the sample concentration was obtained with the curve shown in F2.



F1. Voltammogram overlay of sample and three standard additions.



F2. Standard addition curve using BAS 100 B/W analysis software.

Results and Discussion

The duration of the acid digestion of the pigment sample was determined by multiplying the length of time needed for all of the yellow color to fade from the pigment (2 hours) by two. Although this was an arbitrary time, other samples that were digested for longer periods showed no significant increase in lead concentration. The concentration of lead in the sample was determined to be 1.1 ppm. Considering that 0.0046 g of pigment was used, the total amount of lead released by acid digestion was 0.00012 g. The paint pigment used was 2.4% lead, in the form of the yellow chromate salt.

1) *B. Feldman, et al., Anal. Chem. 66 (1994): 1983.*
2) *J. Wang, Analyst 119 (1994): 763-766.*
3) *D. Jagner, L. Renman, and Y. Wang, Electroanalysis 5 (1993): 283-288.*
4) *H.W. Numberg, Electrochim. Acta 22 (1977): 935-949.*
5) *M. Jandreski, Clin. Chem. News 8 (1994): 8-13.*