



CAPSULES

preliminary notes and applications from Bioanalytical Systems, Inc.

Determination of Lead in Soil by Anodic Stripping Voltammetry: Sample Preparation

Purpose

The acute and chronic toxicity of lead in children is well established, and there is considerable interest in the determination of trace lead concentrations in both physiological and environmental matrices. However, measurements of lead concentrations using voltammetric methods are sensitive to organic interferences, and hence sample preparation is a crucial part of such measurements.

Reference

Digestion of Soil Samples for the Determination of Trace Amounts of Lead by Differential-Pulse Anodic Stripping Voltammetry, A.R. Fernando and J.A. Plambeck, *Analyst* 117 (1992) 39-42.

Method

The standard method for the preparation of soil samples involves grinding and sieving through successively finer meshes, followed by acid digestion (to remove organic compounds that can interfere with the analysis). In this study, various digestion methods were compared:

- 1) Successive digestion in nitric acid, perchloric acid and hydrofluoric acid in an open beaker, followed by heating to near dryness and re-dissolution in 1 M nitric acid.
- 2) Digestion in a nitric acid/hydrofluoric acid mixture in a bomb at 150 °C.
- 3) Microwave digestion in a nitric acid/hydrofluoric acid mixture.

Results

The bomb and microwave procedures provided solutions that were reactive toward mercury, which caused problems with the mercury electrode. This reactivity was attributed to the presence of hydroflu-

oric acid, and removal of the hydrofluoric acid required evaporation of the acid mixture in an open beaker; that is, the time advantage gained using the bomb and microwave methods was negated. Therefore, the open beaker digestion method was used.

The re-dissolution that followed the evaporation required 1 M nitric acid, which reduced the potential window. Therefore, three methods for lowering the acidity were tried: neutralization (e.g., with base), dilution with water, and dilution with a weak acid. The addition of base caused the appearance of an interfering peak, whereas dilution with water produced wide variations in the pH of the analyte solutions, which decreased the reproducibility of the experimental results. However, good results were obtained following dilution using 0.1 M acetic acid and 0.1 M potassium chloride at pH 3.

This method was tested using standard soil samples containing 21 ± 4 ppm of lead. The results obtained ranged from 19-24 ppm, with standard deviations of 1-2%; that is, the results obtained using this method were acceptable.