

**CHELATE ASSISTED, PRESSURIZED, LIQUID EXTRACTION
FOR THE REMOVAL OF ADSORBED METAL CONTAMINANTS
FROM SOILS**

**By
Karen L. Marshall**

**Dissertation submitted to the Faculty of the
Virginia Polytechnic Institute and State University
In partial fulfillment of the requirements for the degree of**

DOCTOR OF PHILOSOPHY

IN

CHEMISTRY

**G. Long, Chairman
M. Anderson
L. Taylor
K. Brewer
J. Viers**

**March 3, 2000
Blacksburg, Virginia**

CHELATE ASSISTED, PRESSURIZED, LIQUID EXTRACTION FOR THE REMOVAL OF ADSORBED METAL CONTAMINANTS FROM SOILS

By

Karen L. Marshall
Chairman: Gary L. Long
Department of Chemistry

ABSTRACT

Chelate Assisted, Pressurized, Liquid Extraction (CAPLE) has been developed in our laboratory as an efficient, separation-based, extraction methodology for heavy metals in soils. Unlike current extraction methods used in environmental determination of contaminated soils, CAPLE is able to selectively remove adsorbed metals from the soil matrix without requiring the total destruction of the sample. By not fracturing the soil matrix particles, as with hot acid digestion methods, geologically bound metals are not liberated in the CAPLE process. This unique feature of CAPLE allows us to quantify levels of contaminant metals and correlate them to anthropological activity in the area.

CAPLE requires the use of a modified supercritical fluid extractor for operation with water at sub-critical levels. The extraction of the sorbed metals is facilitated by the use of a chelating agent. Metal determinations are performed by atomic absorption (FAAS or GFAAS) or ICP emission spectrometry.

CAPLE has been subjected to a variety of experimental conditions in order to elucidate the strengths and possible weaknesses of the extraction technique. The uses of the chelating agents (type and concentration) have been optimized. Possible release of metals from the resulting ionic strength of the chelating

solutions have been shown not to be a factor. Both pressure and temperature effects have been studied and adjusted for optimal conditions.

The majority of the research lies in the application of CAPLE to a variety of soil conditions. The effect on particle size of the soil and soil coating (humic acid and iron oxides) has been studied. In all soil systems and coatings studied, CAPLE could be optimized to completely remove chemisorbed metals. Tests of CAPLE on Cu-sludge amended soils provided excellent agreement with traditional methods of soil analysis. Not only was good agreement obtained between the recoveries of the methods, but CAPLE was also found to be much faster, more environmentally friendly, and much less prone to sample loss or sample contamination compared to traditional soil extraction methods.

A final portion of this work involves a rigorous statistical analysis of CAPLE to a sequential extraction method. Since a Standard Reference Material (SRM) has not been provided for chemisorbed metals onto soils, a comparative analysis was chosen to validate the technique. Using the Cu-amended soils, CAPLE was found to effectively liberate all chemisorbed metals as compared to the sequential extraction technique. There was no statistical difference in recovery between the two extraction methods.

CAPLE is shown in this work to be a viable extraction method for analyzing contaminant metals in soils. It is a rapid and efficient technique. Unlike traditional digestion methods, it is able to differentiate anthropological metals from geologically occurring metals. Its ease of use, coupled with simplicity of instrumental design and analytical reagents make it an attractive extraction technique for environmental analysis.