

THE UNIVERSITY OF ALBERTA

TRACE HEAVY METAL DETERMINATION IN SOIL SAMPLES USING
DIFFERENTIAL PULSE ANODIC STRIPPING VOLTAMMETRY

BY

© ANGELO RANSIRIMAL FERNANDO

A THESIS

SUBMITTED TO THE FACULTY OF GRADUATE STUDIES AND RESEARCH IN
PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF
DOCTOR OF PHILOSOPHY

DEPARTMENT OF CHEMISTRY

411114

EDMONTON, ALBERTA

FALL 1988

CR



ABSTRACT

Studies reported in this thesis were carried out employing an anodic stripping system constructed via interfacing a micro computer (Analog Devices Macsym 150) to a commercially available polarographic analyzer (Princeton Applied Research Model 174A). All work was done using an automated hanging mercury drop electrode system (Princeton Applied Research Model 303).

Effects of the background electrolyte on differential pulse anodic stripping voltammetry were investigated. The presence of chloride ions in the solution was found to give higher sensitivity in the determination of cadmium than did the presence of nitrate ions. For lead, this situation was found to be reversed although the difference in sensitivities is not as significant as in the case of cadmium. Apart from the effect on sensitivity, the changing of chloride levels that can arise as a result of leaking from a chloride containing reference electrode was found to affect the precision of the analysis adversely.

Using a matrix exchange procedure and a two way analysis of variance process, oxygen interference in anodic stripping analysis was shown to be confined to the stripping or analysis step.

The applicability of differential pulse anodic stripping voltammetry for the measurement of trace heavy metals in soil samples was investigated by measuring the amount of lead in a sample of soil collected locally as well as in two certified reference soils (SO-1 and SO-2; Canadian Certified Reference Materials Project). Digestion of the soil sample in open Teflon beakers using a nitric : perchloric : hydrofluoric acid mixture was found to be superior to a microwave digestion using nitric and hydrofluoric acids. In the case of the local soil sample the presence of iron led to a noisy response during the analysis of the digested solution using differential pulse anodic stripping analysis.



Dilution of the final analytical solution with deaerated buffer was found to be an effective solution to this problem.