

CHAPTER 2

EXPERIMENT



2.1 Chemicals

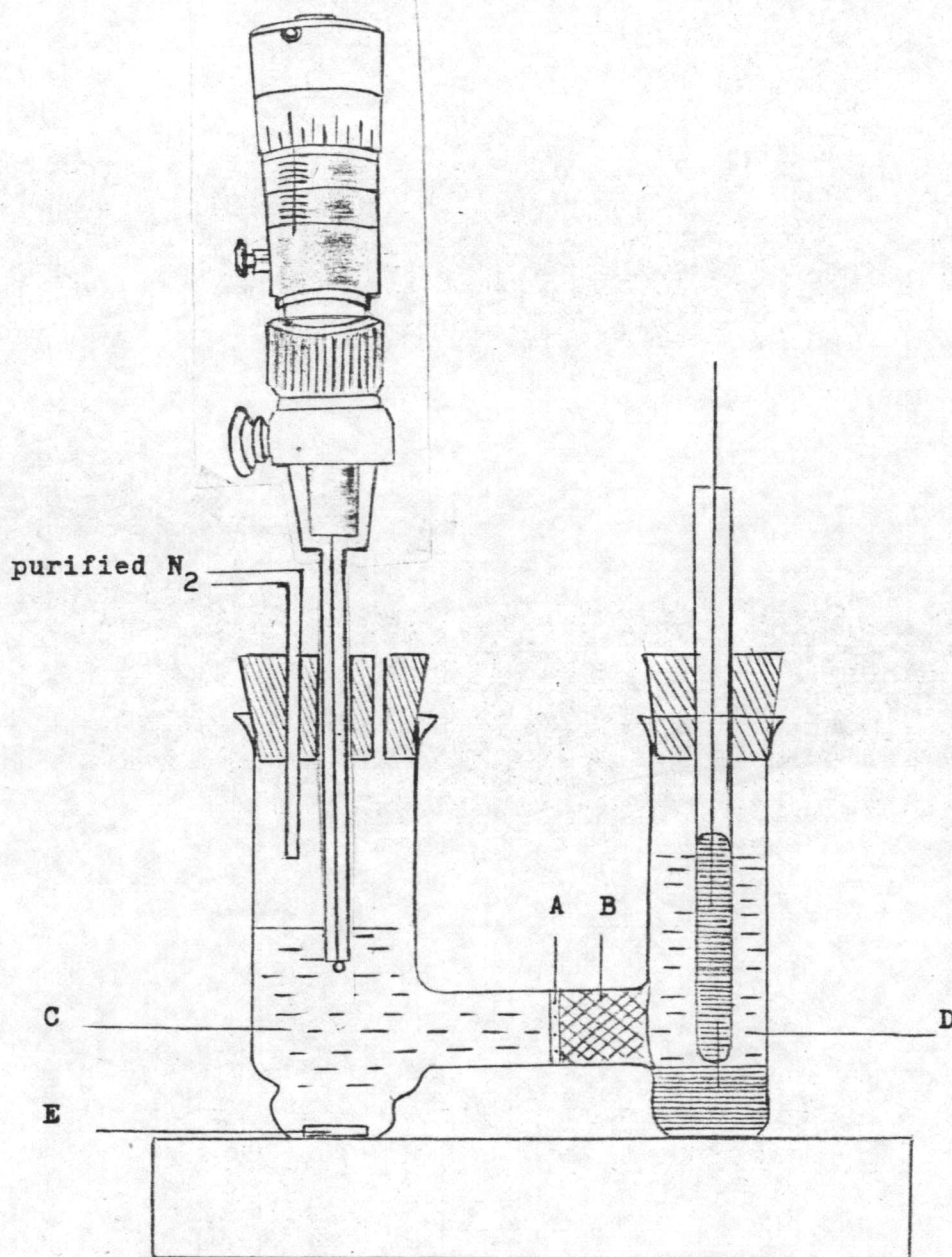
All chemicals used were of reagent grade, no further purification of these compounds was attempted unless otherwise stated. Hygroscopic compounds were kept in a desiccator over the anhydrous silica gel. $\text{Pb}(\text{NO}_3)_2$ was dried at 110°C and stored in the desiccator until used.

2.2 Apparatus

The cathodic voltammogram and anodic stripping voltammogram were obtained with a Radiometer Copenhagen Polariter type P04g. The cell employed in all anodic stripping analyses is a two compartment cell (H-shaped cell): one compartment served for reference electrode, saturated calomel electrode (SCE), and another compartment served for the test solution.

The hanging mercury drop electrode (Princeton Applied Research Model 9923) is used as working electrode.

In order to purify nitrogen gas before being used to deaerate the test solution, three bubbling towers were placed in the gas line. The first tower contained amalgamated zinc metals in a vanadous chloride solution, for reducing any contaminated



The H-type voltammetric cell used throughout this study. The letter labels are A-the sintered glass, B-the agar plug, C-the test solution, D-the calomel half cell, and E-the magnetic stirring bar.

oxygen gas. The second tower contained a 4 % alkaline solution for neutralization of the nitrogen gas and the last one was a trapped tower for prevention of any solution from the second tower to flow into the test solution.

All potentials in this work were measured against SCE. The pH measurements were obtained with a pH meter (Radiometer Copenhagen type PHM 28).

2.3 Procedure

2.3.1 double deionized water

Double deionized water was obtained by passing the distilled water through a series of three columns (3.5 cm, I.D. and 55 cm, long). Amberite IR-45 (OH), anionic resin, was packed in the first column, Amberite IR-120, (H), **cationic** resin, was in the second column and the last column was one half filled with the anionic resin and another half with the cationic resin.

2.3.2 standard lead solution

A $1.00 \times 10^{-2} \text{ M Pb(NO}_3)_2$ solution was prepared by dissolving 0.03312 g $\text{Pb(NO}_3)_2$ in a 100.0 cm^3 volumetric flask. The percentage of the lead (II) ion in the reagent grade of $\text{Pb(NO}_3)_2$, determined by **amperometric titration using** hanging mercury drop electrode, was found to be 99.99.

A series of standard solutions containing 0.99×10^{-8} to 1.19×10^{-6} M $\text{Pb}(\text{NO}_3)_2$ in 0.02 M KNO_3 and 0.10 M HNO_3 was also prepared by successive dilution of 1.00×10^{-2} M $\text{Pb}(\text{NO}_3)_2$ solution.

2.4 Preparation of the vegetable sample for determining lead

Before the vegetable was ashed, it was carefully shaken and rinsed with tap water to remove surface soil, then dried in air for about half an hour, and weighed (using 50-100 g sample) in a porcelain basin. These were performed on coded samples so that lead contents were not influenced by possible experimental bias. The basin with sample was dried overnight in an oven of 120°C and placed in a 250°C furnace. The temperature of this furnace was raised up slowly (50 increments) to 350°C and held until smoking ceased. The temperature was then increased to 450°C in ca. 75 increments. The sample was ashed at this temperature for 6 hours and was removed from the furnace. If ash still contained excess C particles (i.e., ash is grey rather than white), wet the sample with minimum amount of water followed by dropwise addition of HNO_3 ($0.5\text{-}3 \text{ cm}^3$). Then the sample was dried on water bath, transferred to furnace at 250°C , slowly increased temperature to 450°C , and continued heating for 1-2 hours. If the ash was grey, the sample was repeated with the HNO_3 treatment and ashing until C-free residue was obtained. After finishing, the basin with ash was cool down in a desiccator and was weighed accurately again. The method mentioned above is from Official Methods of Analysis of the Association of Official

Analytical Chemists (65).

2.5 Test solution

The ashed sample was dissolved with 5 cm³ of the double deionized water and warmed to aid solution. The deionized water, KNO₃ and HNO₃ were added to the sample solution to have the final concentrations of 0.02 M KNO₃ and 0.10 M HNO₃ in a 50.0 cm³ volumetric flask.

2.6 Voltammetric and stripping analysis

Before the test solution was placed in the cell for either voltammetry or stripping analysis, the test compartment was washed with 0.1 M HNO₃ and twice with the double deionized water. Then a 10.0 cm³ test solution was transferred to the cell, and N₂ gas was bubbled through the solution for 10 minutes. Then the N₂ gas inlet tube was adjusted to let N₂ flow gently above and across the solution surface, and the HMDE was inserted in the test compartment.

To obtain the deposition potential for Pb(II) solution, a cathodic voltammogram of Pb(II) solution must be recorded and its peak potential was measured.

For deposition of Pb(Hg), the stirrer moter was turned on, a potential of -0.60 V was set and the electrolysis was performed for 20 minutes.

In cathodic and anodic voltammetry, the desired potential

range, current sensitivity, scan rate and polarity were set on the instrument and the voltammogram was scanned.

After recording the voltammogram, the exact volume of known concentration of standard Pb(II) solution was added to the sample solution (0.01-1.00 cm³). The volume of standard Pb(II) solution added is depended upon the estimation of lead content in sample, it should be sufficient to approximately double level of each analyte presented. The ASV of this solution was performed again. From the measurement of the anodic peak currents of the sample solution and the sample solution which added the standard solution, the concentration of Pb(II) ion in the sample was determined by calculating from equations 5 and 6 (see page 19).