

CHAPTER 4
EXPERIMENTAL

(4) EXPERIMENTAL

4.1 APPARATUS, CHEMICAL AND INSTRUMENTS.

The apparatus that are needed for this project is liked the following:

- (i) Beakers;
- (ii) Micro-pipettes: 50 μ L;
- (iii) Pipettes;
- (iv) Spatula;
- (v) Volumetric flasks: 100 mL, 50 mL;
- (vi) Stirrer.

The following Table 4.0 shows the chemical reagents and instruments are used in this project (Reference 52):

Table 4.0: The chemical reagents and instruments are used in this project.

Chemical reagents	Manufacturer
Ammonium hydroxide	R & M Chemicals (U.K.)
Ammonium chloride	Hamburg Chemicals GmbH
Nitric acid (16 M)	R & M Chemicals (U.K.)
Potassium nitrate (powder)	RIEDEL-DE HAEN AG SEELZE-HANNOVER (Germany)
Potassium chloride (powder)	RIEDEL-DE HAEN AG SEELZE-HANNOVER (Germany)
Lead standard solution (1000 ppm)	J.T. Baker (U.S.A)
Cadmium standard solution (1000 ppm)	J.T. Baker (U.S.A)
Chemical reagents	Manufacturer
Zinc standard solution (1000 ppm)	J.T. Baker (U.S.A)

Nitrogen gas	NISSAN-INDUSTRIAL OXYGEN INCORPORATED BHD (Shah Alam, Malaysia)
Deionised water	
Instruments	Manufacturer
Electro-balance	Denver Instrument Company (U.S.A)
BAS 100 B/W Electrochemical Workstation	Bioanalytical Systems, Inc (Indiana, U.S.A)

Source: Analysis of selected trace metals in water using Differential Pulse Polarography and Anodic Stripping Voltammetry (2000).

4.2 SOLUTION PREPARATION

4.2.1 PREPARATION OF SUPPORTING ELECTROLYTES

(i) 0.1 M Nitric acid, HNO₃

About 10 mL of 16-M of nitric acid is pipetted into the volumetric flask (100 mL).

The deionised water is added until the level of 100 mL and the concentration is 1.6 M.

Then, about 6.25 mL of the previous solution (1.6 M) is pipetted into the 100 mL of another volumetric flask. The deionised water is added until the level of 100 mL. The concentration is 0.1 M.

(ii) 0.1 M Potassium nitrate, KNO₃

About 1.01 g of potassium nitrate is weighted and transferred into the 100 mL volumetric flask. The deionised water is added until the level of 100 mL and shake well.

(iii) **0.1 M Potassium chloride, KCl**

About 0.755 g of potassium chloride is weighted and transferred into the 100 mL volumetric flask. The deionised water is added until the level of 100 mL and shake well.

(iv) **0.1 M Ammonium hydroxide-ammonium chloride solution, NH₄OH-NH₄Cl**

About 53.49 g ammonium chloride and 72.5 mL of ammonium hydroxide solution are added into a 1-liter volumetric flask. The deionised water is added until the level of 1-liter and shakes well. The concentration is 1.0 M.

Then, 10 mL of the 1.0 M ammonium hydroxide-ammonium chloride solution is pipetted into a 100 mL volumetric flask. The deionised water is added until the level of 100 mL and shake well. The final concentration is 0.1 M.

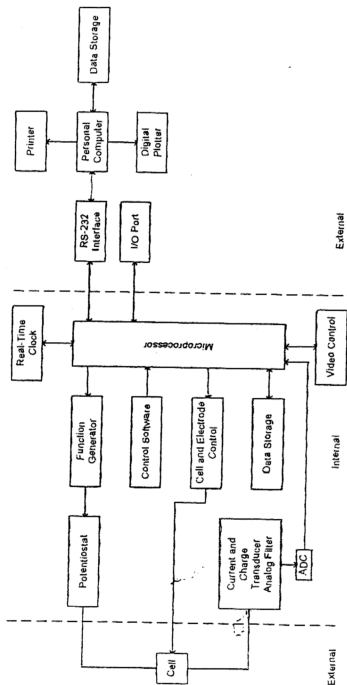
4.2.2 PREPARATION OF STANDARD SOLUTIONS (LEAD, CADMIUM, ZINC) IN VARIOUS CONCENTRATION.

By using the stock solutions of the metals (lead, cadmium, zinc) with concentration 1000 ppm to prepare the standard solutions into suitable concentrations. The concentrations have used in this project are such as 50 ppm, 5.0 ppm, 2.0 ppm, 0.5 ppm, 0.05 ppm and 0.02 ppm.

4.3 INSTRUMENTAL

The instrument has used for this trace metal analysis in the BAS B/W Electrochemical Workstation. It is made up of a personal computer (PC), an electrochemical analyser and a cell stand. The following diagram shows the operation of the BAS 100 B/W Electrochemical Workstation (Reference 52).

Figure 2.1: The Block diagram of the BAS 100 B/W Electrochemical Workstation.
 Source: Analysis of selected trace metals in water using Differential Pulse Polarography and Anodic Stripping Voltammetry (2000).



4.4 PROCEDURE:

4.4.1 Determination of the half-wave potential for metals (lead, cadmium, and zinc) in the four different types of the supporting electrolytes by using DPP mode.

1. Rinse the glass cell and the electrodes with deionised water and next with the supporting electrolyte.
2. Fill in 10 mL of the supporting electrolyte into the glass cell.
3. Place the glass cell in the cell stand.
4. Turn on the nitrogen gas.
5. Switch on the PC, electrochemical analyzer and cell stand.
6. Turn on the **Purge** switch at the cell stand for 5 minutes.
7. Go to the **File** menu and select **Setup** icon.
8. Set the parameters like the following:

i) Comm. Port:	1
ii) Band Rate:	19200
iii) Line Freq.:	60 Hz
iv) Cell stand:	CGME
v) Knock and Audible	
vi) PreDrops:	1
vii) Oxidation Current Polarity:	Minus
9. Go to **Method** and click the **Select Mode**. Select the **Pulse Technique:** and **Differential Pulse Polarography (DPP)**.
10. Set the **General** parameters for Initial E, Final E, and Sensitivity.
11. Set the **Specific** parameters like the following:

i) Scan rate:	2 mV/s
ii) Pulse amplitude:	50 mV
iii) Sample width:	17 ms
iv) Pulse width:	50 ms

- v) Drop time: 1000 ms
vi) Quiet time: 5 s

12. After purging, turn the **Purge** to **Blanket** throughout the analysis.
13. Run the polarogram for the supporting electrolyte.
14. Record the peak current.
15. Add 50 μL of the standard solution (50 ppm) into the 10 mL supporting electrolyte.
16. Run the polarogram for the standard solution.
17. Identify the metals by checking the peak potential. Record the peak currents for each metal at different concentrations.
18. Repeat the step 15 a few times.
19. Draw the graph of peak current versus the concentration for each metal.
20. Repeat from the step 1 until the step 19 for another types of supporting electrolyte.

4.4.2 Determination of the detection limits for the metals (lead, cadmium, and zinc) by using the DPP mode.

1. Repeat all the steps from the part (1) *Determination of the half-wave potential for metals in different types of supporting electrolytes* except no need to draw the graph.
2. Use the standard solution (5.0 ppm) for lead and cadmium; (2.0 ppm) for zinc.
3. Repeat a few times for step 2.

4.4.3 Determination of optimum conditions for the DPSV mode.

1. Rinse the glass cell and the electrodes with the deionised water and then supporting electrolyte.
2. Fill in the glass cell with 10 mL of supporting electrolyte.

3. Place the glass cell in the cell stand.
4. Put in a stirrer and turn on the **Stir** button. Set the stir rate at 100 rpm.
5. Turn on the nitrogen gas.
6. Switch on the PC, electrochemical analyzer and cell stand.
7. Turn on the **Purge**.
8. Go to the **File** menu and select the **Setup** icon.
9. Set the parameters like the following:

i) Comm. Port:	1
ii) Band Rate:	19200
iii) Line Freq.:	60 Hz
iv) Cell Stand:	SMDE
v) Audible	
vi) PreDrops:	0
vii) Oxidation Current Polarity:	Minus
10. Go to **Method** menu and click the **Select Mode**. Select the **Stripping Techniques** and **Differential Pulse Stripping Voltammetry (DPSV)**.
11. Set the **General** parameters for Initial E, Final E, Rotation Rate, Sensitivity and Deposition Time.
12. Set the **Specific** parameters for Scan rate, Pulse amplitude, Sample width, Pulse width, Pulse period, and Quiet time.
13. After purging, turn on the **Purge** to **Blanket** throughout analysis.
14. Change the parameters, such as Drop size, Scan rate, Stir rate, Deposition time, and Quiet time.
15. Add in the certain concentration metals into the 10 mL supporting electrolyte.
16. Run the analysis.

4.4.4 Determination of the half-wave potential for the metal (lead, cadmium, zinc) in the four different types of supporting electrolytes by using DPSV mode.

1. Repeat the part (3)-*Determination of optimum conditions for DPSV mode* from step 1 to step 13.
2. Run the voltammogram for the supporting electrolyte.
3. Record the peak current.
4. Add 50 μL of the standard solutions (0.5 and 0.05 ppm) into the 10 mL supporting electrolyte.
5. Run the analysis.
6. Identify the metals by checking the peak potential. Record the peak current for each metals at different concentrations.
7. Repeat the step 4 a few times.
8. Draw the graph of peak current versus the concentration for each metal.
9. Repeat from step 1 until the end for another types of supporting electrolytes.

4.4.5 Determination of the detection limits for the metals (lead, cadmium, and zinc) by using the DPSV mode.

1. Repeat the part (4)-*Determination of the half-wave potential for the metals (lead, cadmium, and zinc) in the four different types of supporting electrolyte by using the DPSV mode* from step 1 to step 3.
2. Add 50 μL of the standard solution (0.02 ppm) into the 10 mL supporting electrolyte.

3. Run the voltammogram.
4. Record the peak current for each metal.
5. Repeat a few times from step 2 until the end.

4.4.6 Determination the concentration of the metals (lead, cadmium, zinc) in river water by using DPSV mode.

SAMPLING:

1. Wash 3 PE bottles with water and then fill in the dilute hydrochloric acid. Leave them overnight.
2. Take the three samples from 3 sampling points like following:

STATION 1	STATION 2	STATION 3
PLACE: mosque	Behind the Nanking Vegetation	Damansara Intan SS2
DATE: 16-1-2001	16-1-2001	16-1-2001
TIME: 3.00 pm	3.25 pm	4.05 pm

ANALYTICAL PROCEDURES:

1. Rinse the glass cell and the electrodes with deionised water and then supporting electrolyte.
2. Fill in the glass cell with 6 mL of sample and 4 mL of supporting electrolyte.
3. Set the **General** and **Specific** parameters according the optimum conditions have determined in part (3)-*Determination of optimum conditions for DPSV mode.*
4. After purging, turn on the **Purge** to **Blanket** throughout the analysis.
5. Run the voltammogram for each station.