Experiment no 4

Object : To determine the concentration of nitrate ions in environmental samples using cyclic voltammetry (CV), a technique that measures the current response of an electrochemical cell to a varying potential.

**Materials and Equipment**

1. **Electrochemical Workstation** (e.g., Potentiostat/Galvanostat)
2. **Three-Electrode System**:

**Working Electrode**: Glassy Carbon Electrode (GCE)

**Reference Electrode**: Silver/Silver Chloride (Ag/AgCl)

**Counter Electrode**: Platinum Wire or Mesh

1. **Electrolyte Solution**: 0.1 M PBS Phosphate Buffer Solution (pH 7.0),
2. **Nitrate Standard Solutions**: Sodium nitrate,Various concentrations (e.g., 1 mM, 5 mM, 10 mM)
3. **Environmental Sample**: Water sample with potential nitrate contamination
4. **Deionized Water**
5. **Glassware**: Beakers, pipettes, and volumetric flasks
6. **pH Meter**
7. **Protective Equipment**: Lab coat, gloves, safety glass

**Theory**- Cyclic voltammetry (CV) is an electrochemical technique used to study the redox behavior of chemical species by sweeping the potential of a working electrode between two set values in a cyclic manner and measuring the resulting current. In CV, the potential (E) of the working electrode is varied linearly over time, and the resulting current (I) is recorded to generate a voltammogram. The typical cyclic voltammetry experiment involves applying a potential E(t)that changes according to the following equation:

E(t)=E0+νt

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Cyclic voltammetry involves applying a voltage to an electrode immersed in an electrolyte solution, and seeing how the system responds. In CV, a linear sweeping voltage is applied to an aqueous solution containing the compound of interest. A linear sweeping voltage is defined by the voltage (or potential) being varied linearly at the speed of the scan rate Figure 1 illustrates the voltage variation. The voltage flips sign and the potential reverses when it reaches a maximum value, becoming the maximum voltage. The potential of the working electrode is swept linearly between two values (the start and end potentials) at a constant rate (scan rate). This sweep can be forward (positive potential) and reverse (negative potential), creating a cyclic waveform.  
As seen in Figure 1, the changeover occurs at the peak. The procedure can then be carried over again in a cyclical or periodic fashion.

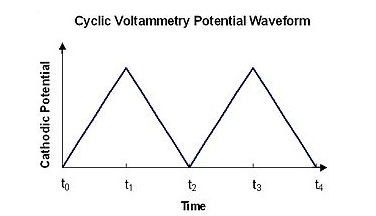


Figure 1.1: Potential versus time program for cyclic voltammetry showing the forward and reversed linear potential ramp.

**Procedure**

* Potential versus time program illustrating the forward and reversed linear potential ramp for cyclic voltammetry.
* One milliliter (mM) of nitrite is added to a 25 or 50 milliliter (mL) PBS solution.
* A cyclic voltammetry measurement with a scan rate of 50 mV/s will be made between 0 and 1.2 V.
* At about 1.0 V, the nitrite oxidation peak is seen. CV was first recorded without nitrite being added to PBS. and after that, CV recorded PBS solution containing nitrite.
* In conclusion, the distinct peak of nitrite oxidation was visible by comparison. in order to gauge the various nitrite concentrations.
* The technique of linear sweep voltammetry was effective.
* Here, LSV will first be recorded in the PBS solution at a scan rate of 10 mV/s at the potential zone mentioned above.

**References**

* Bard, A. J., & Faulkner, L. R. (2001). Electrochemical Methods: Fundamentals and Applications. John Wiley & Sons.
* Eggins, B. R. (2002). Chemical Sensors and Biosensors. Wiley.
* S radha

Here are some objective-type (multiple-choice) questions for assessing understanding of cyclic voltammetry (CV) and its applications.

**Pre-Test Questions**

1. **What is the primary purpose of cyclic voltammetry?**

A) To measure the pH of a solution

B) To analyze the thermal properties of a substance

C) To study the redox behavior of chemical species

D) To determine the molecular weight of a compound

**Answer: C) To study the redox behavior of chemical species**

1. **Which electrode in a three-electrode system provides a stable reference potential?**

A) Working Electrode

B) Counter Electrode

C) Reference Electrode

D) Auxiliary Electrode

**Answer: C) Reference Electrode**

1. **In cyclic voltammetry, what does the scan rate refer to?**

A) The rate at which the potential is changed

B) The speed of the reaction at the electrode

C) The rate at which the sample is introduced

D) The rate of current decay over time

**Answer: A) The rate at which the potential is changed**

1. **What is typically observed in a voltammogram as a result of an oxidation reaction?**

A) A peak at a negative potential

B) A peak at a positive potential

C) A flat baseline

D) No current response

**Answer: B) A peak at a positive potential**

**5. Which of the following can affect the peak current in a cyclic voltammogram?**

A) Temperature

B) Scan rate

C) Concentration of the analyte

D) All of the above

**Answer: D) All of the above**

**Post-Test Questions**

1. **When preparing a calibration curve for nitrate analysis using cyclic voltammetry, what is plotted on the x-axis?**

A) Peak Current

B) Peak Potential

C) Scan Rate

D) Voltage

**Answer: A) Peak Current**

1. **What could be a reason for observing broad peaks in a cyclic voltammogram?**

A) High scan rate

B) Low scan rate

C) High concentration of the analyte

D) Low temperature

**Answer: A) High scan rate**

1. **Which electrode material is commonly used as a working electrode in cyclic voltammetry experiments?**

A) Silver

B) Platinum

C) Glassy Carbon

D) Gold

**Answer: C) Glassy Carbon**

1. **In cyclic voltammetry, if the peak potential shifts to a more positive value with increasing scan rate, what does this indicate?**

A) The reaction is reversible

B) The reaction is irreversible

C) The reaction has a high diffusion coefficient

D) The reaction is at equilibrium

**Answer: B) The reaction is irreversible**

1. **What is the role of a supporting electrolyte in cyclic voltammetry?**

A) To react with the analyte

B) To provide a stable reference potential

C) To maintain ionic conductivity in the solution

D) To increase the pH of the solution

**Answer: C) To maintain ionic conductivity in the solution**