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| Exp. No. 6 | Experiment/Subject CHEM 438 (ECD) | Date 10/18/2024 |
| Name Abdul Fayed | Lab Partner | Locker/Desk No. Course & Section No. 021L |

Prelab Questions

1. Square-wave stripping voltammetry is more sensitive than regular square-wave experiment because it employs the preconcentration step which accumulates the desired analyte on the surface of the electrode through electrodeposition. Stirring increases the sensitivity as it allows the desired analyte in the solution to constantly move and eventually reach the surface of the anode. However, too much stirring will also cause the lack of deposition of analyte on the surface as it is constantly moving around instead being reduced onto the surface. Thus, the stirring has to be controlled carefully.
2. Citrate buffer is used to control the pH of the solution @ pH = 3. pH plays an important role when working with electrochemical system as it determines the range of appropriate voltage to be applied on the system to achieve the intended goal of the experiment (electrodeposition of Cu^{2+} on the surface before anodic stripping). It is possible that the citrate buffer has a small trace of Cu. The presence of Cu in buffer will interfere ^{with} the original sample (tap water) and gives out ~~false~~ wrong concentration of Cu in the ~~tap~~ water. To correct this standard addition method can be used to determine the concentration as it takes into account the "matrix" effect of the system.

3. Sigma aldrich:


citric acid: $[\text{Pb}] = 2 \text{ ppm}$

ammonium citrate: $[\text{Pb}] = 5 \text{ ppm}$

$$0.0465 \text{ L} \times \frac{0.1 \text{ mol}}{\text{L citric acid}} \times \frac{2 \text{ mol Pb}}{10^6 \text{ mol of solution}} = 9.3 \times 10^{-9} \text{ mol Pb}$$

$$0.0035 \text{ L} \times \frac{0.1 \text{ mol}}{\text{L ammonium}} \times \frac{5 \text{ mol Pb}}{10^6 \text{ mol soln}} = 1.75 \times 10^{-9} \text{ mol Pb}$$

$$\frac{(9.3 \times 10^{-9} + 1.75 \times 10^{-9}) \text{ mol}}{0.05 \text{ L}} \times \frac{207.2 \text{ g}}{\text{mol}} \left(\frac{1 \mu\text{g}}{10^{-6} \text{ g}} \right) \left(\frac{1 \text{ ppm}}{1000 \mu\text{g/L}} \right)$$

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THE HAYDEN-McNEIL STUDENT LAB NOTEBOOK

Note: Insert Divider Under Copy Sheet Before Writing

$$[\text{Pb}]_{\text{mix}} = 0.046 \text{ ppm}$$

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Objective

Purpose of experiment is to determine the copper concentration in drinking water by anodic stripping voltammetry. ~~Plot~~

Introduction

There is a trace of Cu in drinking water that can be lethal to human health if not controlled properly. Anodic stripping voltammetry is one of the technique to determine the concentration of Cu in drinking water. IAF or X-ray fluorescence have the better detection limit but they involve complex instrumentation. The idea of this experiment is to deposit the Cu in the drinking water onto an anode which will then be oxidized and the amount of Cu that leaves the surface reflects the concentration of Cu in the solution, which will be detected by the amount of current produced.

Anodic stripping voltammetry procedure.


→ Procedure (Brief general overview only).

1. Collect the water sample from the tap.
2. Determine the concentration of Cu by standard addition method ($n=3$).
3. Use ammonium citrate buffer ($\text{pH} \approx 8$) in the sample.

Data for voltage vs. current are provided in TXT file (in this lab, all data should be provided → why lab).

Standard Addition Method

| Sample | Trial | Added vol (mL) | Voltage \downarrow V | Current \downarrow A |
|--------|-------|----------------|------------------------|------------------------|
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4. Assemble the electrochemical apparatus
5. Set up the experiment in the software. (deposition, equilibration, stripping).

Calibration of micropipette

1. Weigh bottle and cap.
2. Micropipette 20 μ L water into the bottle.
3. Cap the bottle then reweigh.
4. Repeat 10 times to obtain the volume average volume and standard deviation.
5. Dispose of all chemicals appropriately and clean the glassware.

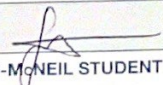
Conclusions

Anodic stripping voltammetry employs the setup of electrochemical cells, where it can ~~test~~ be used to determine the concentration of desired analyte in a system.

Micropipette calibration

~~# of calibra~~

| Number | Mass (g) | Mass of H ₂ O (g) |
|--------|----------|------------------------------|
| 0 | | |
| 1 | | |
| 2 | | |
| 3 | | |
| 4 | | |
| 5 | | |
| 6 | | |
| 7 | | |
| 8 | | |
| 9 | | |
| 10 | | |

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