Polarographic Analysis for Cadmium

I. Introduction

The concentration of cadmium ion in an unknown solution will be determined by measuring the polarographic diffusion current of this solution and relating the magnitude of this current to the diffusion current of a solution containing a known amount of cadmium ion.

II. Equipment

A. PAR Model 263A Potentiostat with Model 270 computer interface
B. Model 303A dropping mercury electrode assembly with Model 507 interface
C. 7.000-mL glass transfer pipet (obtain from instructor)
D. 200-µL adjustable autopipet and disposable tips (obtain from instructor)

III. Reagents (Cadmium standard and unknown solutions are obtained from the instructor.)

A. Potassium chloride
B. Cadmium sulfate, standard solution, 1.001×10^{-2} M
C. Unknown cadmium solution
D. Nitrogen gas

IV. Procedure

A. Weigh out the necessary mass of KCl to prepare 50 mL of a 0.20 M solution and transfer to a 50-mL volumetric flask. Add deionized water and dilute to the mark.

B. Carefully pipet 7.000 mL of the 0.20 M KCl solution into a clean, dry polarography cell.

C. The instructor will demonstrate the software used to control the potentiostat. Select the setup conditions under filename “422Cdsc.set”. Run a sampled current scan from 0.00 V to -1.20 V at a scan rate of 2 mV/sec. This is a check on the presence of O_2 and the residual current level. Save the data using a unique filename.

D. Repeat the sampled current scan as in step (C) except <u>slowly</u> bubble N_2 through this solution for at least 4 minutes to remove most of the dissolved oxygen before starting the scan. Save the data. Overlay this polarogram with that obtained in step (C) and print out the overlay. Note the elimination of (decrease in) the O_2 polarographic waves.

E. Carefully add exactly 200 µL of 1.000×10^{-2} M $CdSO_4$ solution to the cell. Rinse the autopipet tip with some of the solution in the cell.

F. Repeat step (C) with the standard solution except bubble N_2 through the solution for 4

minutes before starting the scan. Print out the sampled current polarogram for the standard.

G. Switch to the square wave polarography mode by selecting the setup conditions under filename "422Cdsw.set". Scan the solution containing the standard at least twice to obtain a reproducible value for the height of the peak. Use a 10-second purge time with each scan. Save the data. It is only necessary to scan from an initial potential just before the peak (-0.4V) to a potential slightly after the peak (-0.8V). The peak height is proportional to the concentration of Cd^{2+}.

H. Add 4 additional samples of the standard $CdSO_4$ solution to the cell, deaerate with N_2 for 1 minute and repeat as in step (G) for **each** addition. Overlay representative polarograms for each addition of standard and print out the overlay.

I. Prepare a clean cell with 7.000 mL of KCl solution as in step (B) and add 200 µL of an unknown Cd^{2+} solution. Deaerate with N_2 for 1 min. and repeat steps (G) and (H) above.

V. Treatment of Data

A. Use the software to carefully determine the height of each square wave polarogram peak in all samples and average those for replicate scans. For the cadmium standards, prepare a plot of peak height versus the total Cd^{2+} concentration in the cell (remember to allow for cell volume changes upon addition of each aliquot) and fit with a first-order least squares line. Normally the 0,0 point is included with the 5 data points for the standards to construct this calibration curve.

B. From the equation for the calibration plot and the observed peak heights of the solutions containing the unknown, calculate the concentration of cadmium in the cell for each unknown aliquot. Then determine the cadmium concentration in the original unknown solution. Report the mean $[Cd^{2+}]$ in your unknown and the RMD of the result.

VI. Questions

1. What would be the effect on your square wave voltammetry results if you did not purge the solution with N_2 after each new addition of standard or unknown solution? Explain.

2. What would be the effect on the height of each square wave polarogram peak if you used a larger drop size? Explain.

Lab Cautions for Polarographic Analysis for Cadmium

1. Mercury spills are a major concern in this experiment. ALL WORK INVOLVING MERCURY SHOULD BE DONE OVER A TRAY TO CONTAIN ANY SPILLS.

2. When you are finished using the dropping mercury electrode, thoroughly rinse the cell and electrodes, fill the cell with dilute KCl solution and replace the cell.

3. Before you leave, turn off the power to the Model 303A dropping electrode, the Model 263A potentiostat and the Model 270 computer interface.

4. The last person in the lab using the nitrogen gas for purging should close the valve to the tank before leaving.