

## VOLTAMMETRY

### THE PURPOSE OF THE EXERCISE

Initial introduction to the electrochemical analyzer. Observation of the influence of the carbon electrode surface condition on the voltammetric signal of ascorbic acid. Voltammetric determination of ascorbic acid based on its anodic oxidation.

### APPARATUS AND REAGENTS

PalmSens miniature electrochemical analyzer:

- electrodes: glassy carbon (working electrode), silver chlorine (reference), platinum (counter electrode)
- polarographic vessels, measuring flasks, pipettes, wash bottle, distilled water, ascorbic acid, 0.1 M H<sub>2</sub>SO<sub>4</sub>.

### PROCEDURE

1. Polish the surface of the working electrode with an alumina suspension according to the instructions.
2. Using the dilution method, prepare ascorbic acid solutions in measuring flasks in a concentration range from 1 to 5 mM.
3. Prepare the polarographic cell. Fill it with a 5 mM standard solution
4. Record the differential pulse voltamperogram in the potential range from 0.0 to 1.0 V versus Ag / AgCl.
5. Transfer the electrodes to a second electrochemical cell with 10 mL of basic electrolyte (0.1 M H<sub>2</sub>SO<sub>4</sub>).
6. Electrochemically activate the electrode surface using the cyclic voltammetry technique in the potential range  $0 \rightarrow +2V \rightarrow -0.8 V$ .
7. Record the next baselines for the settings as in p. 4 until stabilization.
8. Re-record the voltamperogram for the 5 mM standard solution.
9. Record voltamperograms for the remaining standard solutions.
10. Record the voltamperogram in the sample solution and after two standard additions.

Processing the results:

1. Determine the peak heights of ascorbic acid oxidation in all samples.
2. Create a calibration curve and determine its equation by linear regression.

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3. Determine the concentration of the analyte in sample by the standard curve method and the standard addition method.

### LITERATURE

1. D. Kealey, P. J. Haines, *Analytical Chemistry*
2. D. Harvey, *Modern Analytical Chemistry*
3. Douglas A. Skoog, Donald M. West, F. James Holler, Stanley R. Crouch, *Fundamentals of Analytical Chemistry*
4. Douglas A. Skoog, F. James Holler, Stanley R. Crouch, *Principles of Instrumental Analysis*
5. B. Sivasankar, *Instrumental Methods of Analysis*