

Voltammetric determination of iodide in pharmaceutical samples using multi-walled based screen printed electrodes (SPE)

1. Aim of the work:

The purpose of the exercise is the electrochemical determination of iodide content in medicinal preparations using an advanced screen-printed electrode system.

2. Preparation of the exercise:

1. Prepare a series of potassium iodide solutions with the following concentrations: 0.02 mM, 0.04 mM, 0.06 mM, 0.08 mM, 0.1 mM using a standard solution with a concentration of 1 mM KI in phosphate buffer (0.1 M PBS) with pH = 7.4.
2. Connect the electrodes. Start the measuring system (follow the instructor's instructions).
3. To stabilize the working electrode, record cyclic voltammograms in the blank electrolyte (0.1 M PBS). For this purpose, 60 μl of the buffer should be dropped on the surface of the SPE. **Note:** All 3 electrodes should be in contact with the electrolyte. Perform 10 scans from 0.2 V to 1.0 V at a potential shift speed of $v = 20 \text{ mV} / \text{s}$. Voltamperograms should show a capacitive current with no visible redox peaks.
4. For each test solution containing KI, perform 3 voltammetric scans ranging from 0.2 V to 1.0 V at a potential shift rate $v = 20 \text{ mV s}^{-1}$. Measure the height of the KI oxidation peak for the third voltammetric scan.
5. Grind a tablet of Jodid 100 in a mortar. Quantitatively transfer the resulting powder into a 10 ml vial and dissolve in 0.1 M PBS. Place the suspension in the ultrasound for 10 minutes.
6. Filter the resulting suspension on a filter paper or a syringe and use a clear solution for further tests.
7. Record the voltammograms for the real sample tested under test conditions identical to those in 4. Measure the height of the KI oxidation peak.

Analysis of the results:

1. On the basis of the measured current peaks, plot a calibration curve $I = f(c)$
2. From the calibration curve, calculate the KI content of the slide.

Issues for learning:

Reduction/oxidation reactions, reversibility of electrode reactions, electromotive force, half-cell potentials, electroanalytical methods (amperometry, voltammetry), electrochemical properties of microelectrodes.