

## Abstract

This diploma thesis examines the optimization of the determination of nitrophenylhydrazines using high performance liquid chromatography with electrochemical detection on dual thin-layer electrode. The spectrophotometric detection was used for the comparison. The developed method was also tested for the determination of hydrazone produced by the reaction of 2,4-dinitrophenylhydrazine (DNPH) with benzaldehyde as a member of substances containing carbonyl group.

The conditions for the determination of nitrophenylhydrazines were optimized; based on the hydrodynamic voltammograms, pH 3 of aqueous part of the mobile phase and potentials of +1,0 V and -1,0 V were chosen. Under these conditions, the repeatability was tested.

The concentration dependences were also measured and detection limits were obtained. The detection limits were obtained from the peak heights both in anodic and cathodic potential range. For 2-nitrophenylhydrazine these limits were  $9,5 \cdot 10^{-8}$  mol/L for oxidation and  $6,2 \cdot 10^{-7}$  mol/L for reduction. For 2,4-dinitrophenylhydrazine the limits were  $2,1 \cdot 10^{-8}$  mol/L and  $1,8 \cdot 10^{-7}$  mol/L for oxidation and for reduction, respectively.

For the preparation of hydrazone, DNPH was chosen due to its better cathodic response and it was mixed with benzaldehyde. The electrochemical behaviour of benzaldehyde-2,4-dinitrophenylhydrazone was examined in mobile phase with pH 3 of aqueous part. From the hydrodynamic voltammogram, working potential of -1,0 V was selected. The yield of the reaction was examined and it was found that at least double concentration of DNPH than benzaldehyde is needed.