Voltammetric methods for the determination of diazepam (DZ) and nordiazepam (NDZ) were developed. Techniques differential pulse voltammetry (DPV) and DC voltammetry were used for determination of DZ and NDZ at meniscus modified silver solid amalgam electrode (m-AgSAE).

The effect of pH[a] on the intensity of signal was observed in the mixture of Britton-Robinson buffer and methanol (9:1), and in the mixture of 0.1 mol.l^{-1} NaOH and methanol (9:1). The stability of the signal during repeated measurements in the mixture of 0.1 mol.l^{-1} NaOH and methanol (9:1), and in the mixture of BR buffer and methanol (9:1) was monitored. Optimal pH[a] 13.2 of medium of 0.1 mol.l^{-1} NaOH and methanol (9:1) was used for determination of DZ with DPV and DCV techniques. Optimal pH[a] 10.1 of medium of BR buffer and methanol (9:1) was used for determination of NDZ with DPV and DCV techniques. Under these conditions linear dependencies calibration were measured.

Concentration range of DZ was measured with DCV in range of $10x10^{-5}$ – $6x10^{-6}$ mol.l^{-1} and with DPV technique in range of $10x10^{-5}$ – $2x10^{-6}$ mol.l^{-1}. Concentration range of NDZ was measured with DCV technique in range of $10x10^{-5}$ – $4x10^{-6}$ mol.l^{-1} and with DPV technique in range of $10x10^{-5}$ – $2x10^{-6}$ mol.l^{-1}. The limit of detection for DZ was calculated $6.6x10^{-6}$ mol.l^{-1} with DCV and $1x10^{-6}$ mol.l^{-1} with DPV. The limit of detection for NDZ was calculated $5.5x10^{-6}$ mol.l^{-1} with DCV and $1.7x10^{-6}$ mol.l^{-1} with DPV.

Developed methods were used to determine the DZ in the drug sample of Diazepam Slovakofarma 2 mg. Drug sample was determined by standard addition method in the mixture of 0.1 mol.l^{-1} NaOH and drinking water (9:1) and in the mixture of 0.1 mol.l^{-1} NaOH and methanol (9:1) with DPV. Concentration of DZ in drinking water was determined at $1.88x10^{-5}$ mol.l^{-1} and in methanol at $1.66x10^{-5}$ mol.l^{-1}.