In living organisms, reactive nitrogen species such as peroxynitrite can nitrate specific amino acids, whether or protein bound, and 3-nitrotyrosine (3-NT) is believed to be one marker of this radicalic reaction [1]. Different methods such as immune techniques, gas and liquid chromatographic methods with mass spectrometry, tandem mass spectrometry, electrochemical and UV detectors have been used to determine 3-NT[2-4]. Immune techniques and chromatographic methods are both time consuming and expensive. These drawbacks lead researchers to investigate new sensitive monitoring methods for 3-NT. To the best of our knowledge voltammetric determination of 3-NT has not been studied. Here in this work we aimed to develop a voltammetric assay for 3-NT in biological fluids. Using Square wave voltammetry (SWV), various supporting electrolytes with different pH values including 0.1 M HCl, 0.1 M NaOH, 0.1 and 1.0 M KCl, 0.5M acetate (pH 3-5), 0.5 M borate (pH 8-10), 0.05 M Britton-Robinson (pH 2-12), 0.5 M phosphate (pH 2-12) were examined. Phosphate buffer (0.5 M pH 9) was selected as supporting electrolyte considering the height of the peak current and the peak shape. Other experimental conditions were optimized with preliminary experiments and chemometric design. SWV conditions were selected as 0.01 V, 0.1 V, 140 Hz for potential step, amplitude and frequency respectively. A well defined cathodic peak was observed at -0.476 V versus Ag/AgCl/ 3 M KCl for 3-NT and a linear calibration curve was obtained in 2.02x10^-8 - 1.05x10^-6 M concentration range. As a result of cyclic voltammetric(CV) studies the reduction of 3-NT was found to be adsorption controlled. Therefore we used adsorptive stripping square wave voltammetric method (AdSWV). Different electrochemical behaviour of 3-NT was studied using cyclic voltammetry. Obtained results showed that observed current by reduction of 3-NT is absorption controlled. Therefore we tried adsorptive stripping square wave method. Different deposition times and deposition potentials were evaluated and 20 s time was chosen for deposition time and -0.15 V versus Ag/AgCl/ 3M KCl potential for deposition potential. In these conditions a calibration curve was plotted in 1.61x10^-10 – 1.00x10^-8 M concentration range. AdSWV method seems to be promising for determination of 3-NT in biological fluids.