The aim of the thesis was to analyze the historical pharmaceutical preparations, including the determination of the active substance and identify their possible degradation products. A historical pharmaceutical preparation of naloxone was analyzed by mass spectrometry. Historical pharmaceutical preparations of adrenaline and ephedrine were analyzed by UHPLC-MS and were quantified using a calibration curve. In the historical injection solution of naloxone, “NARCAN”, dated around 1980, there were no significant degradation products and the measured mass and UV spectrum was consistent with the spectrum of naloxone. The analyzed sample of naloxone was stable even after 35 years of storage. In the analyzed historical injection solution of adrenaline, “Adrenalin Hydrochlor., Dr. Heisler“ (dated between 1917 and 1938) was determined 5.26 ± 0.11 % of the declared amount of adrenaline. In the measured spectra were noticeable degradation products, which have not been described in the literature yet and their identification was beyond the scope of this paper. The analyzed sample of adrenaline was almost completely degraded during about ninety years. The stability test carried out with four standard solutions of adrenaline proved influence of oxygen, light, temperature and time on the degradation of adrenaline. In the historic injectable form of ephedrine, „Ephedrin“ (dated between 1945 and 1948) was determined 107.58 ± 0.39 % of the declared amount of ephedrine and in the measured spectra were no apparent degradation products. The analyzed sample of ephedrine was stable for nearly seventy years of storage.