Historical pharmaceutical preparations of caffeine and santonin were analyzed by mass spectrometry, the sample of caffeine was quantified by spectrophotometry, the sample of santonin was quantified by acid-base titration. In the analyzed historical injection solution „Coffeinum natriosalicylicum“ (dated circa 1945) was determined 110 ± 1 % of the declared amount of caffeine and 94.33 ± 0.02 % of the declared amount of sodium salicylate. There were observed neither degradation products of caffeine nor sodium salicylate. The content of santonin in the historical sample (dated prior 1918) was determined by titration to 104 ± 4 %, sample was pure santotnin with no additions. No degradation products of santonin was observed. The analyzed samples were stable for more than seventy, ninety resp., years of storage.