

Abstract

This thesis deals with the study of UV-photochemical generation of volatile compounds (UV-PVG) in connection with atomic absorption spectrometry (AAS). Selenium (Se(IV)) was selected as a model analyte and many experiments were carried out to expand the current sum of knowledge of the topic.

The study was commenced by assembling the UV-PVG apparatus in the continuous flow mode. The detection method used was AAS with externally heated quartz furnace atomizer. The focus of the first step of the study was on the construction of the volatile compounds generator (UV-photoreactor) with emphasis on the material used; tubes made of teflon or quartz of different diameters were tested. The construction of the apparatus was followed by optimization of the reaction conditions (the type and concentration of the photochemical agent and other agents, which increase the analytical signal; the carrier gas and the auxiliary hydrogen gas flow rate; the sample flow rate). Eventually, the analytical figures of merit of the selenium determination using the proposed method were found. The results showed that teflon reaction tubes are a good competitor to those made of quartz. The accuracy of the method has been successfully verified by analysis of certified reference material and its applicability has been further tested on analysis of samples in various matrices; selenium was determined in samples of drinking water and selected dietary supplements. Series of experiments using radiotracer ^{75}Se have been carried out to estimate the overall efficiency of the conversion of the analyte to the volatile compound. Moreover, it was possible to observe the distribution of the analyte in the apparatus. Further improvement of sensitivity and the limits of detection was achieved by combining the UV-PVG with *in situ* collection of the generated volatile compound in a graphite furnace prior to its electrothermal atomization. Another set of experiments carried out was an interference study addressing the effects of selected inorganic acids and salts, transition metals and other hydride forming elements on the determination of selenium.

For comparison, many experiments were performed using traditional chemical generation with sodium borohydride as a reducing agent. The figures of merit and the efficiency achieved with the optimized conditions clearly show that UV-PVG is fully comparable alternative to the conventional approach to the generation of volatile compounds. The main disadvantage at the current state of its research lies in the lower tolerance of UV-PVG to interference effects.