Abstract

This master's thesis is focused on development of a new analytical method based on photochemical generation of volatile species (PCVG) of rhodium as an efficient sample introduction technique for inductively coupled plasma mass spectrometry. At first, the conditions affecting the PCVG efficiency were optimized, namely carrier gas flow rate, irradiation time, composition of the reaction medium as well as the impact of transition metal ions as potential sensitizers was examined. The combination of Cu^{2+} and Co^{2+} was found optimal, enhancing the PCVG efficiency more than 60-fold. Additionally, a serious effect of the residence time of volatile species in the reaction medium, after exiting the irradiated part of the photoreactor, was found. The PCVG efficiency was doubled by modifying the output part of the photoreactor and another two-fold increase in the PCVG efficiency was achieved using the addition of nitrate anions to the reaction medium. Under chosen optimal conditions, the limit of detection and quantification were 13 pg dm^{-3} (3σ, n = 11) and 42 pg dm^{-3} (10σ, n = 11), respectively, and the PCVG efficiency was determined to be $13.5 \pm 0.1\%$. The repeatability (n = 10) at 20 ng dm^{-3} was 2.9%. These experiments were followed by a study of interferences commonly occurring in the PCVG methods (chloride and sulfuric anions, hydrogen peroxide and selected transition metal ions). Finally, the method was validated by analyses of real water samples and solid certified reference materials (OREAS 684 and SRM 2556), which were prepared by a fusion with sodium peroxide and subsequent dissolution in nitric acid.

Key words

photochemical generation of volatile species, inductively coupled plasma mass spectrometry, rhodium