Abstract

This dissertation thesis is focused on UV-photochemical generation of volatile species of tellurium, ruthenium, rhenium and iridium coupled to methods of atomic spectrometry. In the first part, attention was paid to the optimization of parameters of UV-photochemical generation of volatile tellurium species and its application for speciation analysis of Te(IV) and Te(VI) in water samples. UV-photochemical generation was carried out in a UV-photoreactor, which consisted of a low-pressure mercury lamp wrapped with a polytetrafluoroethylene reaction coil that served as a reactor. Atomic absorption spectrometry with a continuum source of radiation and high resolution and atomization in a miniature diffusion flame were used to optimize the conditions of generation, which included the composition of the reaction medium, irradiation time and the addition of transition metals as modifiers. In order to achieve a higher sensitivity of determination, the generator was coupled to a triple quadrupole inductively coupled plasma mass spectrometer. Since efficient UV-photochemical generation of volatile species was achieved only from Te(IV), with no response from Te(VI), the feasibility of this technique for simple "non-chromatographic" speciation analysis was tested and a method for determination of Te(IV) and total inorganic Te was developed.

In the second part, the influence of unconventional introduction of a carrier gas to the apparatus for UV-photochemical generation of volatile tellurium species was investigated. Compared to the apparatus from the first part, the carrier gas was introduced upstream the UV-photoreactor, which led to different optimal conditions of generation that are discussed.

The third part is devoted to UV-photochemical generation of volatile iridium species coupled to a inductively coupled plasma mass spectrometer. Unlike the first two parts, UV-photochemical generation was conducted in a special lamp equipped with an internal reaction channel made of quartz glass. An attention was mainly paid to optimization of the concentration of formic acid in the reaction medium without and with added modifiers. Based on the finding that it is possible to efficiently generate volatile Ir species even from a very diluted reaction medium, a more detailed study of UV-photochemical generation of not only Ir, but also Ru and Re was undertaken. In addition to defining the range of low concentrations of formic acid, wherein the most efficient UV-photochemical generation of volatile species from all three elements occurs, the effect of irradiation time, the presence of modifiers and the effect of dissolved gases in the reaction medium (Ar, CO and O₂) were examined. The knowledge gained could help to propose a general mechanism of UV-photochemical generation of volatile species of transition metals.