Abstract

This master's thesis deals with the optimization of conditions of chemical hydride generation (HG) of bismuth, its atomization and detection by atomic absorption spectrometry (AAS) and atomic fluorescence spectrometry (AFS). Two types of atomizers were used for atomization of volatile species, a miniature diffusion flame for AAS as well as for AFS and a flame-in-gas-shield atomizer for AFS. At first, the parameters of HG in a flow injection mode were optimized – the concentration of hydrochloric acid, the concentration of sodium borohydride and the volume of the reaction coil. Subsequently, the atomization conditions were optimized using both atomizers. The parameters optimized were hydrogen fraction, total gas flow rate and observation height. Due to the more complicated construction of the FIGS atomizer, more parameters were studied, such as the oxygen flow rate through the capillary and the flow rate of shielding argon required for shielding the free atoms. A special part of the thesis dealt with the optimization of the optical path of the atomic fluorescence spectrometer, the selection of an interference filter and the optimization of a power supply of an electrodeless discharge lamp. It was found that under optimum conditions of generation, atomization and detection excellent detection limits (2,3 pg cm^{-3} for MDF and 1,6 pg cm^{-3} for FIGS atomizer) and excellent repeatability (below 1%) were obtained for both atomizers. The accuracy of HG-AFS method with both MDF and FIGS atomizers was verified by determination of bismuth in the certified reference material of water NIST 1643f with a relatively good agreement of the results.

Keywords: bismuth, chemical hydride generation, miniature diffusion flame, flame-in-gas-shield atomizer, atomic absorption spectrometry, atomic fluorescence spectrometry.