Abstrakt

This thesis deals with optimization of conditions of photochemical generation of volatile species of nickel for atomic absorption spectrometry. The volatile species of nickel were generated in the flow arrangement, when sample was injected to a stream of a reaction medium. Either formic acid was used as the reaction medium or formic acid with the addition of formate anions. Two types of a generator were tested, a generator with a PTFE tube wrapped around a mercury UV lamp and a high efficiency generator with an inside channel. At the same time, two types of a gas-liquid separator were tested. Diffusion flame was used as an atomizer due to its high robustness. Quartz atomizer didn't provide higher sensitivity and measurements had worse repeatability. All the parameters affecting atomization in the diffusion flame were optimized (flow rates and composition of gases, observation height). In the next section, conditions of generation (irradiation time, HCOOH concentration, addition of formate anions) were optimized. Interferences of inorganic acids in photochemical generation were investigated as well. It was found out that the generator with the inside channel provided slightly higher sensitivity and thus generation efficiency than the generator with the PTFE tube wrapped around. Transmission of radiation into sample in the generator with the inside channel was much more efficient, leading to shorter irradiation time and low concentration of HCOOH needed for efficient generation. Generation efficiency, determined by measuring the amount of residual analyte in the waste, limit of detection and quantification and repeatability of measurement were determined for both types of the generator. The lowest limits of detection and quantification were achieved with the high efficiency generator with the inside channel on the instrument ContrAA 300 – 1,5 μg dm^{-3} for HCOOH as the reaction medium and 0,8 μg dm^{-3} for HCOOH with $HCOONH_4$ as the reaction medium. Connection of the generator and a cryogenic trap was tested to further decrease limits of detection. Results were promising, but the method requires thorough optimization.

Key word:

Photochemical generation of volatile species, UV, atomic absorption spectrometry, nickel