Abstract

This thesis is focused on increasing the senstitivity of gold determination by electrochemical volatile species generation using two different types of electrolytic cells in continuous flow setting. Externally heated quartz tube atomizer was used as means of atomization and detection of gold with atomic absorption spectrometer. Generation parameters were optimized for electrolytic cell with an ion exchange (nafion) membrane. After selection of new cathode material (Cu) the carrier gas (Ar) flow rate was optimized, where an additional inlet of carrier gas was found to have possitive effect on increasing the sensitivity of determination of gold and efficiency of volatile specie transport to the atomizer. Experiments with Antifoam B showed possitive effect on generation, thus calibration was carried out for optimized experimetal conditions reaching detection limit of 0,53 $mg.dm^{-3}$. The efficiency of volatile specie transfer from liquid to gaseous phase was determined between 60 – 65 % by measuring the residual gold content in liquid waste by F-AAS method. Subsequently the effieciency of electrolytic generation of volatile gold specie with radioactive tracer isotopes and autoradigraphy was determined to 0,6 %. These methods confirmed adsorption of generated species on apparatus surface. Using ICP-MS as a detection technique, limit of detection 0,12 $mg.dm^{-3}$ was determined. As an alternative a new non-membrane electrolytic cell with Pt electrodes was constructed for which the generation parameters were optimized. Limit of detection of 0,49 $mg.dm^{-3}$ was determined for optimized conditions. Using the same F-AAS method, liquid waste was measured for residual gold volatile specie content and using an improvised filter, efficiency of 1,3 % was determined.