Abstract

This diploma thesis is focused on the construction of the electrochemical cells for the lead determination using electrochemical generation of volatile compounds connected with the atomic absorption spectrometry as a detection technique. Three different electrochemical cells were constructed: membrane electrochemical cell (MEC), non-membrane electrochemical cell 1 (BEC1) and non-membrane electrochemical cell 2 (BEC2). Experimental conditions were optimized in continuous flow mode with cells BEC1 and BEC2. The optimization include flow rate of carrier gas, composition and concentration of the electrolytic solution, atomization temperature, generation current and voltage applied to the electrode material. A 1.0 mm diameter cadmium wire was used as cathode material and the anode material was composed of platinum wire of 0.5 mm in diameter. Under the optimal experimental conditions, the calibration was constructed and basic characteristics of proposed method were determined for the BEC1 and BEC2 cells: limit of detection, limit of quantification, repeatability, sensitivity, linear dynamic range and correlation factor of linear dynamic range. The cell MEC was found to be unsuitable for the lead determination by the electrochemical generation of volatile compounds. Using the BEC1 cell, the experimental system was simplified compared to the MEC cell. The cell BEC1 has integrated gas/liquid separator. A mixture of hydrochloric acid (0.25 $mol \cdot l^{-1}$) and potassium chloride (0.1 $mol \cdot l^{-1}$) was used as a optimal electrolytic solution. The detection limit of the method with the BEC1 cell was 1.08 $mg \cdot l^{-1}$, sensitivity $7.4 \cdot 10^{-3}$ $l \cdot mg^{-1}$ and repeatability 14.3 %. The BEC2 cell was designed with minor modifications compared to BEC1 cell. The volume of the electrode chamber has been reduced and the design of integrated phase separator has been modified to facilitate the transport of the generated gaseous phase into the detection part. The limit of detection of the method using BEC2 cell was 0.38 $mg \cdot l^{-1}$, sensitivity $11.2 \cdot 10^{-3}$ $l \cdot mg^{-1}$ and repeatability 7.9 %. The BEC1 and BEC2 cell comparisons shows that better parameters were achieved for the BEC2 cell.