Abstract

The general aim of this work was a development of methodology and instrumentation for speciation analysis based on the combination of the selective generation of substituted hydrides with atomic absorption or atomic fluorescence spectrometry detection.

The first topic of this work was the development of methodology and instrumentation for arsenic speciation analysis based on selective generation of substituted arsines with trapping in the cryogenic trap (U-tube packed with chromosorb) with AAS detection (HG-CT-AAS). The conditions of the selective hydride generation approach as well as working procedure of the cryogenic trap were optimized (appropriate approach for hydride generation, set up of heating program of cryogenic trap, new dryer – cartidge with NaOH, elimination of unspecific absorption, decreasing of the detection limits).

The second important part of the work lay in applying of the developed method for arsenic speciation analysis in a homogenized mouse liver tissue. The direct slurry sampling to hydride generator was develop. Moreover the information about oxidation state ($iAs^{III,V}$, $MAs^{III,V}$ a $DMAs^{III,V}$) was obtain. The effect of relevant experimental parameters such as tetrahydroborate concentration, TRIS buffer concentration and time of pre-reduction of the samples by L-cysteine was investigated.

The cryogenic trap made of quartz glass was successfully tested as an alternative to the borosilicate glass U-tube. Narrower peaks with higher resolution were obtained with this kind U-tube. The trapping and volatilization efficiency of arsines in the U-tube packed with chromosorb were 100% determinated by spectrometry and radiometry (with ^{73}As a ^{74}As radioindicators) experiments. All these results showed the possibility of miniaturization of the cryogenic trap. Several materials of the cryogenic trap were tested. An „I“ shaped quartz capillary (0.53 mm i.d.) without filling was found the most suitable reaching 100% trapping and volatilization efficiency.

The final aim of this work was interfacing of the HG-CT system with the atomic fluorescence detector to reach lower detection limits for all arsenic forms. The detection limits 3.2 pg iAs^{III}; 0.8 pg MAs^{V}; 1.1 pg $DMAs^{V}$ were achieved by this approach.