ABSTRACT

Charles University in Prague Faculty of Pharmacy in Hradec Králové Department of Analytical chemistry

Candidate: Bc. Eva Krchňáková

Supervisor: PharmDr. Petr Chocholouš, Ph.D.

Title of diploma thesis: Solid phase extraction and its miniaturization by method Lab-On-Valve.

This thesis deals with the ability to automate solid phase extraction (SPE), in connection with the bead injection (BI). The aim was to test the replacement of microparticles in the bead injection with sorbent from SPE cartridges, determinate the behavior of sorbent in this system and then determine whether it is possible to miniaturize all the steps of solid phase extraction that are necessary to implement the method.

In the work was done manual solid phase extraction, where were tested seven sorbents: Lichrolut Merck RP-18, DSC-18 DSC-18Lt, DSC-8, PH-DSC, DSC-CN and Oasis HLB. The experiment resulted in the best possible conditions for carrying out solid phase extraction as follows: activation of the sorbent and elution of analyte 100% acetonitrile, solvation of the sorbent, the dissolution of vitamin D and ballast from the sorbent washed with dilute acetic acid of pH 3. Oasis HLB from Waters Company is the most suitable sorbent for the system, which is water-wettable and should not be dispersed by the system, which was confirmed by the use of sorbent. Other sorbents were inappropriate for the system because sorbents were not water-wettable. Sorbents were dispersed system, got into the pump and then pump was clogged. It was reason for stopped the work and other testing of this sorbents was not possible.

Further development of the method consisted in transfer the conditions from manual solid phase extraction to the Lab-On-Valve system, in the program were compliance all phases of the general procedure for solid phase extraction. In the program were tested various dosing volumes, flow speed and repeatability of column packing. Optimized method was tested by analysis of blood plasma spiked with vitamin D. After (analysis) extraction of plasma in bead injection was performed sequential injection analysis, recovery of the method was 41.07 %. In the optimal conditions, was result the possibility of extraction of low concentrations of vitamin D in blood plasma. The disadvantage of the method was low repeatability analysis.

The method tried to automate and accelerate the solid phase extraction in combination with the bead injection was experimental. Under defined conditions and in the current implementation of the method is ineffective. For increase efficiency of this analysis is need further method development.