Abstract

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Title of Diploma Thesis: On-line SPE-HPLC method development for determination of zearalenone in beers

High performance liquid chromatography coupled with on-line solid phase extraction (SPE) using the column switching technique for sample preparation was utilized for the development of the method for the determination of mycotoxin zearalenone in beer. Two different SPE sorbents were tested. A volume of 50 µl of beer sample was injected directly into the chromatographic system. After injection, the sample extraction of zearalenone from the matrix was carried out on SPE precolumn Ascentis® Express C18 Guard Cartridge (5 x 4.6 mm, 5 µm), which is based on the reverse phase, or on SPE precolumn (10 x 4.6 mm) filled with Affinimip® SPE Zearalenone sorbent, a specific Moleculary Imprinted Polymer, designed for the selective extraction of zearalenone. 40% methanol – 2% water solution of acetic acid was selected as the washing solution for removing ballast matrix for the C18 extraction column and 10% acetonitrile – 2% water solution of acetic acid for the Affinimip column. These washing solutions were flowed through the extraction column at a flow rate 2 ml/min for 2 minutes. After switching valve, the compounds were further separated on an analytical column Kinetex XB-C18 (150 x 4.6 mm, 5µm). Mobil phase of composition acetonitrile - water (35:65) was passed through the column at 1 ml/min in gradient elution. Wavelengths of fluorimetric detection were set at Ex 270 nm and Em 458 nm. Analysis of one sample including on-line pretreatment was about 13 minutes. Limit of quantification for this method was 5 µg/l. Zearalenone was analyzed in 30 samples of Czech beer. The measured amount of mycotoxin was low, in most cases undetectable and under the maximum allowed level for zearalenone according to EU standards.