

Bachelor-Abschlussarbeit

Thema: Development of an automated SPE-enrichment for the LC-MS-determination of tetracyclines in water samples

Zusammenfassung

The use of antibiotics is increasing worldwide, especially due to intensive livestock farming, which also increases their entry into the environment. Thereby, it is important to monitor existing concentrations in the environment, to point out possible environmental risks.

The aim of this study was to extend and to optimize an already existing LC-MS/MS method for the analysis of seven antibiotics that are frequently used in veterinary and human medicine: amoxicillin, ciprofloxacin, clotrimazole, chlortetracycline, doxycycline, oxytetracycline and tetracycline.

For this purpose, the solvent composition and solvent gradient on the LC-MS/MS were optimized. The results showed, that the running time of the previously used gradient could be shortened and that Milli-Q water + 0.2 % FA / acetonitrile + 0.2 % FA as eluent composition improved the peak shapes and ionization efficiency for the examined substances.

To optimize the SPE method, the parameters of the previously manually performed SPE method were first transferred to the automated LCTech XANA Freestyle and then adapted to the working range of the instrument. The SPE was performed through Oasis HLB cartridges, including a washing step, the drying of the cartridge and the elution of the analytes with methanol. The extract was then concentrated and adjusted to the LC start gradient with Milli-Q water.

With the optimized parameters, it was tested with matrix-containing water samples from the river Elbe (1 L), whether the addition of EDTA to the samples prior to the SPE-enrichment improves the recovery rates of the analytes. The MQL's for the antibiotics when pre-treated with EDTA was between 2 and 39 ng/L.

It was found, that for ciprofloxacin and clotrimazole the recovery rates using Milli-Q water + 0.2 % FA / acetonitrile + 0.2 % FA as the mobile phase were improved (above 90 %). However, the addition of EDTA during sample preparation deteriorated their recovery rates.

For amoxicillin, no recovery rates could be determined, as it could not be enriched with the optimized SPE-method.

For the tetracyclines, however, the recovery rates were improved by the addition of EDTA, although excessive recovery rates were found.

This is most probably due to signal suppressions during electrospray ionization caused by coelution of matrix constituents and the fact that for most of the analytes no mass-labelled internal standard of the respective compound was available. Therefore, an additional washing step of the SPE cartridge and different compositions for the eluent should be tested, as the use of pure methanol strongly elutes matrix components into the extract, which can increase signal suppressions.

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Datum der Abgabe: 20.08.2018