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Utilization of large volume zwitterionic hydrophilic interaction liquid chromatography for the analysis of pharmaceuticals in aqueous environmental samples: benefits and limitations

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Highlights:

- -A sensitive HILIC-ESI-MS/MS method was developed for aqueous environmental samples
- -Freeze-drying was used for the first time as a sample preparation in HILIC
- -Eluent and diluent composition must be strictly controlled to ensure reproducibility
- -Co-elution of chloride or nitrate with the analytes can cause positive matrix effects

Abstract

Benefits and limitations of HILIC were studied for the analysis of extreme polar organic contaminants in aqueous environmental matrices. A sensitive analytical method was developed and validated for the detection of 11 pharmaceuticals, 15 pharmaceutical metabolites and transformation products and the artificial sweetener acesulfame. The analytical method consisted of a simple and non-specific sample preparation based on freeze-drying followed by detection with large injection volume (70 μ L) zwitterionic HILIC-ESI-MS/MS. Robustness studies showed a high sensitivity of the retention times and peak shapes to variations of the acetonitrile/water ratio of both the eluent and the diluent. Thus, a thorough sample and eluent preparation is required to obtain reproducible results. Extreme matrix effects of > 200 % were observed for emtricitabine and acyclovir, which could be traced to the co-elution of nitrate and chloride, respectively. These matrix effects and those of other analytes could be efficiently compensated by using deuterated, ^{13}C and ^{15}N -labeled internal standards. The developed method was able to detect the selected 27 analytes in treated wastewater, surface water and groundwater down to limit of quantification (LOQ) in the lower ng/L range. Appreciable

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