StressDetect - A method and process development to improve the limits of detection of pharmaceuticals in water - SPME coupled GC-MS

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Water is the most important resource in the world. Over 70 percent of our planet is covered with water, the share of fresh water of the entire water deposit however only amounts to 3.5 percent. Of these, less than 0.007 percent is available for direct human usage, even though water is indispensable for us humans – everyone drinks about 75,000 liters in the course of their life. Therefore, the pollution of rivers, lakes, seas and groundwater is a major threat to mankind. Currently thousands of substances are known to contaminate the waters and pharmaceuticals play in this group an important role. Their active ingredients and degradation products are excreted and come with the sewage into the water cycle. Therefore it is necessary to find an effective method by which it is possible to detect the drugs in the aquatic system. Currently the typical limit of detection for drugs in water oscillates in the range of µg/L or ng/L and our goal is to improve these detection limits.

Due to the low concentration of drug residues in water, an enrichment technique before the analysis is needed. The SPME (*Solid Phase Microextraction*) technique seems to be a perfect choice. This is a simple and innovative technology wherein the analytes adsorb onto the solid phase fibers and can thus be easily extracted/separated from the environmental samples. This method can be used for many classes of compounds and matrices, as extraction and desorption of the compounds are carried out quickly and no solvents are needed [1].

The fiber, the most important part of the SPME system, consists of a coated quartz glass fiber having a diameter of about 0.3mm and a length of 15mm. Typical SPME phases (for coating) are polydimethylsiloxane, polydimethylsiloxane/divinylbenzene, carboxene and polyacrylates. Our goal is to produce our own hybrid material, which consists of an organic and inorganic unit. The organic unit has the selective functionalisation, which favors the necessary sorption properties of the material. The fiber should posses a certain film thickness (ca. 60µm) and stability over a wide pH range. In addition, the fiber should have a high temperature resistance and thermal shock resistance. This is necessary because of the high temperature of the GC-Injector (280ºC). The other important features of the fiber are high material purity and stability in the presence of the derivatizing agent. The derivatization reaction of the pharmaceuticals must be carried out before the GC analysis to change non-volatile into volatile analytes. Some derivatizing agents can be employed to confer improved detectability to sample compounds. For example, EDC and MS-NCI detectors hve very good detectability for analytes with the halogen and nitro substituents [2] [3]. We intend to use the GC with MS-NCI detector, which can be much more sensitive in comparison to typical MS-EI detector.

References:

[1] Pawliszyn J., *Theory of Solid-Phase Microextraction.* Journal of Chromatographic Science, Vol. 38 (2000).
[2] Knapp D. R., *Handbook of Analytical Derivatization Reactions*. John Wiley & Sons (1979).
[3] Moldoveanu S., David V., *Modern Sample Preparation for Chromatography*. Elsevier (2014).