ON-LINE-SAMPLE PREPARATION TECHNIQUES (SPE) COUPLED TO HPLC-UV AND MS-DETECTION FOR THE ANALYSIS OF ORGANIC MICROPOLLUTANTS

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Organic micropollutants are a potential threat for water resources all over Europe and require thus continuous monitoring. For this reason, more than 130 micropollutants have been listed in the Directive 76/464/EEC of the European Community which contains mostly organic and organometallic species. Standard methods for the determination of these compounds are gas and liquid chromatography, mostly accompanied by complicated and tedious sample preparation schemes.

For phenols which shall be chosen here as model analytes and are important and practically ubiquitous industrial pollutants, the primary method of analysis is liquid chromatography. Liquid chromatography is preferred in this case to gas chromatography since their reactivity makes them difficult to be quantitatively determined by GC. Furthermore, GC does not lend itself easily to the automation and integration of the entire analytical procedure.

The approach presented here is the *on-line* coupling of solid phase extraction (SPE) with liquid chromatography and parallel UV and mass spectrometric detection (HPLC-UV/MS). Liquid chromatography is capable of separating all of the relevant phenols listed in the US EPA priority list and can be ideally combined with *on-line* sample preparation by solid phase extraction. Yet, this combination requires careful optimisation to achieve maximum sensitivity, both from the sample preconcentration and the separation/detection side, particularly when mass spectrometric detection is used.

The optimisation of the sample enrichment includes the determination of the best suited SPE material and the optimisation of the SPE conditions. The optimisation of the separation and detection should be undertaken jointly: Here, it is often recognised that conditions which are

optimal for the separation of the investigated phenols are not for their detection: Usually good chromatographic separation of the entire range of phenols requires the addition of mobile phase modifiers, e.g. acids or buffers. The addition of mobile phase modifiers however is usually strongly decreasing the MS response for the phenols under study. Therefore, compromise conditions have to be found for achieving good chromatographic separation and sensitivity.

The optimisation of the entire analytical method, its validation and the application of the developed method to waste water samples will be presented. It offers excellent sensitivity for the detection of the range of EPA phenols (in the low ng/L-range in favourable cases), minimises the manual intervention for sample preparation and time required per analysis due to fully automated operation.