J Chromatogr Sep Tech 2018, Volume: 09
DOI: 10.4172/2157-7064-C2-044

Field-assisted online sample preparation methods for solid sample analysis

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In the past decade, researches in sample preparation techniques have become a significant hot-spot and received much attention. Typically, sample preparation of solid samples is more complicated than the gaseous and liquid samples and more processes in off-line methods will result in lower reproducibility and larger analytical error. The analytes in solid samples must be transferred into a soluble solvent after being extracted from solid matrices and the mass transfer of the interest analytes into a bulk liquid phase, which determines the efficiency of sample preparation, is usually slow. Thus, the most common analytical methods for solid samples are solid-liquid extractions assisted by some field effects and their synergic effects including high pressure, temperature, microwave and ultrasound. A further filtration step was required before the following online or offline chromatographic analysis. Moreover, the solvent conversion problem has to be solved to meet the requirement for the online enrichment procedure. In this work, an online device based on field-assisted extraction, micro-solid phase extraction and high-performance liquid chromatography (FAE-μ-SPE-HPLC, Figure1) was developed. Solid samples were pretreated with ultrasound-microwave synergic effects and the extract was online cleanup with a monolithic column, following analysis with HPLC. The cross actions between ultrasound and microwave along with other parameters were studied systematically. The efficiency of this online device was demonstrated in the determination of polycyclic aromatic hydrocarbons (PAHs) in foods and tetracycline antibiotics (TCAs) in cosmetic samples. The detection limits of nine PAHs including fluorene, phenanthrene, anthracene, fluoranthene, benzo[k]fluoranthene, benz[a]anthracene, benzo[b] fluoranthene, pyrene and benzo[a]pyrene were 0.075-0.30 μg/kg, while that for four TCAs including oxytetracycline, tetracycline, chlortetracycline and doxycycline were 0.02-0.10 μg/g. Six PAHs were found in roast potato and baked fish and the recoveries were ranged in 71.5%-119.7% with the RSDs of 0.2%-10.9% (n=3), respectively. The recoveries for TCAs in cosmetic samples were ranged in 70.2%-128.4% with the RSDs lower than 5.6% (n=3). Compared with offline methods, the present work can not only simplify the experimental process but also increase the sensitivity and analytical speed, showing good potential for analysis of trace analytes in solid and semi-solid samples.

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