COMPARATIVE STUDY OF MODERN EXTRACTION TECHNIQUES FOR THE DETERMINATION OF ENVIRONMENTAL SAMPLES (M 14)

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The aim of any extraction method in analytical chemistry is, to effectively separate the analytes from the matrix. The whole step should be fast and quantitative with minimal solvent and time required. The classical Soxhlet extraction usually requires large volumes (up to 200 mL) of solvent to be refluxed through the solid samples for several hours. Therefore, in the last decades, alternatives for this extraction method have been presented and investigated such as ultrasonic extraction (UE), supercritical fluid extraction (SFE), accelerated solvent extraction (ASE), microwave-assisted extraction (MAE) and fluidized-bed extraction (FBE). The actual choice for analytical application is frequently the initial capital cost, operating costs, simplicity of operation, amount of organic solvent required and sample throughput.
Since sample preparation is a critical step in the analytical cycle, special care has to be taken for an accurate choice and optimization of extraction techniques and clean-up procedures. Therefore, MAE and FBE were investigated for their influential extraction parameters and these parameters were optimized for the extraction of organochlorine biocides [1], polychlorinated biphenyls [2] and polycyclic aromatic hydrocarbons [3] from environmental matrices like soil, sediment and sewage sludge. The extraction yields were compared with those obtained by Soxhlet extraction performed following DIN-methods. Finally, the optimized modern methods were validated by systematic experiments with certified reference materials.

[1] Martens D, Gfrerer M, Wenzl T, Zhang A, Gawlik BM, Schramm K-W, Lankmayr E, Kettup A. Anal Bioanal Chem 372 (2002) 562-568
[2] Gfrerer M, Stadlober M, Gawlik BM, Wenzl T, Lankmayr E. Chromatographia 53 7/8 (2001) 442-446
[3] Gfrerer M, Serschen M, Wenzl T, Gawlik BM, Lankmayr E. Chromatographia 55 7/8 (2002) 467-473

MICROWAVE ACCELERATED EXTRACTION OF FATS WITHOUT CO-SOLVENTS (M 15)

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Microwave Accelerated Extraction has proven to shorten the time required extracting analytes from solid matrices. By using extraction solvents above their normal atmospheric boiling points, Microwave Accelerated Extraction drastically reduces the volume of solvent used from 150 – 500mL to 20 –50mL per sample. This paper demonstrates the application of Microwave Accelerated Extraction, in coordination with a novel sample stirring mechanism and the newly improve GreenChem *Plus* Glass Vessel Polymer samples. The newly improved glass vessels now incorporate temperature and pressure monitoring during the extraction.

This presentation will review the methods optimization process when using a closed vessel microwave accelerated extraction technique for the extraction of fat from food matrices using only non-polar solvents. Derivatization reactions will also be examined. It will focus on extraction temperature and time. Recovery data from "real world" and quality control samples will be presented comparing the microwave technique to the conventional Soxhlet technique.

EVALUATION OF SOLID PHASE EXTRACTION PROTOCOLS FOR ISOLATION OF ANALGESIC COMPOUNDS FROM BIOLOGICAL FLUIDS PRIOR TO HPLC DETERMINATION (M 16)

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A comparative study of various sorbents, reversed phase silica based C_{18}, C_8, and copolymeric hydrophilic-lipophilic balanced, from different manufacturers and various eluting solvents (methanol, acetonitrile and isopropanol) was conducted for optimization of isolating the constituents of multi-component analgesic mixtures by means of Solid Phase Extraction (SPE). Optimized SPE protocols were subsequently applied to human serum and urine samples. Traditional minicolumns and disc formats of C_{18} sorbent were studied as well. The effect of sorbent bed conditioning was also investigated when using novel copolymeric sorbent materials such as OASIS and NEXUS as both claim to function under no conditioned sorbent bed as being water wettable.
An analgesic mixture containing: paracetamol, caffeine and codeine was selected as a model for this survey, since they very often co-exist in pharmaceutical formulations. Analytes were monitored at 240 nm, after isocratic elution from a C_8 Inertsil analytical column. The mobile phase was a mixture of methanol and ammonium acetate (0.05 M) at a volume ratio of 40:60. Statistical evaluation revealed satisfactory accuracy, repeatability and intermediate precision. Pharmaceutical formulation analysis yielded high recoveries ranging from 95.4 to 107.5 %. Various recovery rates were obtained when the different protocols were applied. Reversed phase C_{18} sorbent yielded a 80-90% recovery, while copolymeric sorbents reached the 100 % of analyte concentration in optimizing extraction conditions concerning the activation step and the eluting solvent.