ACCELERATED SOLVENT EXTRACTION (ASE) - A FAST AND AUTOMATED TECHNIQUE WITH LOW SOLVENT CONSUMPTION FOR THE EXTRACTION OF SOLID SAMPLES (T 12)

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Accelerated Solvent Extraction (ASE) is a modern extraction technique that significantly streamlines sample preparation. A common organic solvent as well as water is used as extraction solvent at elevated temperature and pressure to increase extraction speed and efficiency. The entire extraction process is fully automated and performed within 15 minutes with a solvent consumption of 18 mL for a 10 g sample. For many matrices and for a variety of solutes, ASE has proven to be equivalent or superior to sonication, Soxhlet, and reflux extraction techniques while requiring less time, solvent and labor.

First ASE has been applied for the extraction of environmental hazards from solid matrices. Within a very short time ASE was approved by the U.S. EPA for the extraction of BNAs, PAHs, PCBs, pesticides, herbicides, TPH, and dioxins from solid samples in method 3545. Especially for the extraction of dioxins the extraction time with ASE is reduced to 20 minutes in comparison to 18 h using Soxhlet.

In food analysis ASE is used for the extraction of pesticide and mycotoxin residues from fruits and vegetables, the fat determination and extraction of vitamins.

Time consuming and solvent intensive methods for the extraction of additives from polymers as well as for the extraction of marker compounds from herbal supplements can be performed with higher efficiencies using ASE.

For the analysis of chemical weapons the extraction process and sample clean-up including derivatisation can be automated and combined with GC-MS using an online ASE-APEC-GC system.

COMPREHENSIVE 2D CAPILLARY LC AS FRONT END TECHNIQUE FOR MS IN PROTEOMICS (T 13)

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Two-dimensional polyacrylamide gel electrophoresis (2D-PAGE) followed by mass spectrometry (MS) is the most widely used method for protein separation, quantification and identification. However, proteins with extremes in pI and molecular weight, low abundance proteins and membrane associated proteins are rarely seen in 2D-PAGE studies. Therefore, alternative high resolution analytical techniques are needed to analyze complex proteinaceous samples.
One of the promising techniques is the combination of strong cation exchange- (SCX) and reversed phase (RP) chromatography (2D-LC). Proteins are digested in a single step. The resulting peptide mixture is injected onto a SCX column and fractionated with salt steps. The main advantage of the method is the increased peak capacity by which 2D-PAGE can be omitted.
In addition, 2D-LC can be performed fully automated and directly coupled to MS.
In this work we have focused on several variables that affect the overall performance of 2D-LC. These variables include the size of the SCX column, the type of SCX stationary phase, the elution buffer and the arrangement of the SCX and RP column.
All these factors were found to influence the recovery and elution of peptides. Optimized conditions which were used for the identification of proteins in (complex) mixture will be discussed.