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Review

# Solid-phase microextraction: a promising technique for sample preparation in environmental analysis

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## Abstract

Solid-phase microextraction (SPME) is a simple and effective adsorption and desorption technique, which eliminates the need for solvents or complicated apparatus, for concentrating volatile or nonvolatile compounds in liquid samples or headspace. SPME is compatible with analyte separation and detection by gas chromatography and high-performance liquid chromatography, and provides linear results for wide concentrations of analytes. By controlling the polarity and thickness of the coating on the fibre, maintaining consistent sampling time, and adjusting other extraction parameters, an analyst can ensure highly consistent, quantifiable results for low concentration analytes. To date, about 400 articles on SPME have been published in different fields, including environment (water, soil, air), food, natural products, pharmaceuticals, biology, toxicology, forensics and theory. As the scope of SPME grew, new improvements were made with the appearance of new coatings that allowed an increase in the specificity of this extraction technique. The key part of the SPME fibre is of course the fibre coating. At the moment, 27 variations of fibre coating and size are available. Among the newest are a fibre assembly with a dual coating of divinylbenzene and Carboxen suspended in poly(dimethylsiloxane), and a series of 23 gauge fibres intended for specific septumless injection system. The growth of SPME is also reflected in the expanding number of the accessories that make the technology even easier to use. Also available is a portable field sampler which is a self-contained unit that stores the SPME fibre after sampling and during the shipment to the laboratory. Several scientific publications show the results obtained in inter-laboratory validation studies in which SPME was applied to determine the presence of different organic compounds at ppt levels, which demonstrates the reliability of this extraction technique for quantitative analysis. © 2000 Elsevier Science B.V. All rights reserved.

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## 1. Introduction

The analytical procedure has several steps: field sampling, field sample handling, laboratory sample preparation, separation and quantitation, statistical evaluation, decision, and finally, action. Each one of these steps is important for obtaining correct results. Also, it is important to keep in mind that the analytical steps follow one after another, and the next one cannot begin until the preceding one has been completed. If one of these steps is not properly done, the overall performance of the procedure will be poor, errors will be introduced, and consequently variability in the results can be expected. On the other hand, the slowest step determines the overall speed of the analytical process, and if it is important to improve the throughput of the analysis, all steps need to be considered. If an instrument could perform all the analytical steps in the field, without human intervention, then no problems of human error would arise; but, in fact, the reality is quite different.

At the moment several sophisticated instruments are available to separate and to quantify very complex mixtures, such as gas chromatography–mass spectrometry (GC–MS) and liquid chromatography (LC)–MS. The automation and the applicability of chemometric methods to this instrumentation may be considered as very useful. In fact, traditional sample preparation methods are time and labour intensive, have multi-step procedures which lead to loss of compounds, and require the use of toxic solvents. These characteristics make such methods very difficult to combine with hyphenated and automated techniques. The result is that over 75% of analysis time is spent on sampling and preparation steps. Anything we can do to make improvements in this area will translate into advances in time saving and convenience. The phasing out of solvents constitutes

a challenge to the analytical chemist in particular, and to the scientific community in general. Consequently, a great change in analytical methodology will be necessary. There is a great need for change in the current sample preparation methodology, and solvent-free alternatives are needed. These needs have driven the development of a solvent-free preparation technique: solid-phase microextraction (SP< E).

## 2. Sample preparation techniques

Despite the advances in separation and quantitation techniques, many sample preparation practices are based on traditional technologies such as Soxhlet extraction [1] and liquid–liquid extraction (LLE) [2] which are time consuming, labour intensive, and also require the use of toxic solvents [3]. The operating principle of any sample preparation method is to allow analytes to partition between the sample matrix and an extracting phase [4]. Sample preparation techniques which use a small quantity or no organic solvent have been available for some time. They can be classified according to the extracting phase used: gas, membrane, or solvent [5]. Table 1 shows the main steps followed in different sample preparation techniques. As we can see, LLE is a multi-step procedure that often result in loss of analytes during the process, frequently making sample preparation the major source of errors in the analysis, and making it impeditive for integration with the rest of the analytical process. Solid-phase extraction (SPE) was developed in the 1980s, and has emerged as a powerful tool for chemical isolation and purification. From trace levels to industrial scale, SPE plays an important role in a broad range of applications. SPE generically uses an adsorbent material to extract trace organic compounds from aqueous samples. It is