



## Review article

## Solvent microextraction: A review of recent efforts at automation

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Automation

## ABSTRACT

Innovations and trends in sample pretreatment techniques are currently focused on the development of methods that satisfy the principles of Green Analytical Chemistry. The increase in the number of publications devoted to solvent microextraction (SME) techniques testifies to the growing interest of analytical chemists in this field. In addition to miniaturization, we can see a greater than ever effort being made towards the automation of SME procedures.

Rather than providing an exhaustive overview of the current state of this trend, we instead offer readers a look at what we consider to be some of the more interesting recently published solutions.

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## 1. Introduction

In the mid-1990s, several remarkable works were published [1–5] in which the potential of the microdrop in analytical chemistry was discussed. These and several other articles [6–16] published during the decade that followed initiated the subsequent extensive development

of solvent microextraction/liquid-phase microextraction techniques (SME/LPME). We can distinguish several methods that are included in the SME group, for example, single-drop microextraction (SDME), dispersive liquid–liquid microextraction (DLLME) and hollow fiber liquid-phase microextraction (HF-LPME). These methods differ in design, but they all have one common feature: namely, they use only microvolumes of organic solvent and thus comply with the requirements of green analytical chemistry [17–19]. Apart from miniaturization, one interesting and challenging task for analytical chemists is automation, the direct coupling of the sample preparation step with the detection system. Automated systems offer a number of advantages, such as minimizing the errors associated with manual handling, reducing sample and reagent consumption and improving sensitivity and precision, among others. Readers can easily find numerous articles focused on the evolution, advantages, applications and recent advances in SME techniques [20–31]; therefore, no detailed discussion of developments

*Abbreviations:* DLLME, dispersive liquid–liquid microextraction; DLLSME, dynamic liquid–liquid–solid microextraction; ETAAS, electrothermal atomic absorption spectrometry; FAAS, flame atomic absorption spectrometer; FIA, flow injection analysis; HF, hollow fiber; HPLC, high-performance liquid chromatography; IL, ionic liquid; LAV, lab-at-valve; LLLME, liquid–liquid–liquid microextraction; LPME, liquid-phase microextraction; MIP, molecularly imprinted polymer; SI, sequential injection; SDME, single-drop microextraction; SME, solvent microextraction; SPME, solid-phase microextraction; USAEME, ultrasound-assisted emulsification microextraction.

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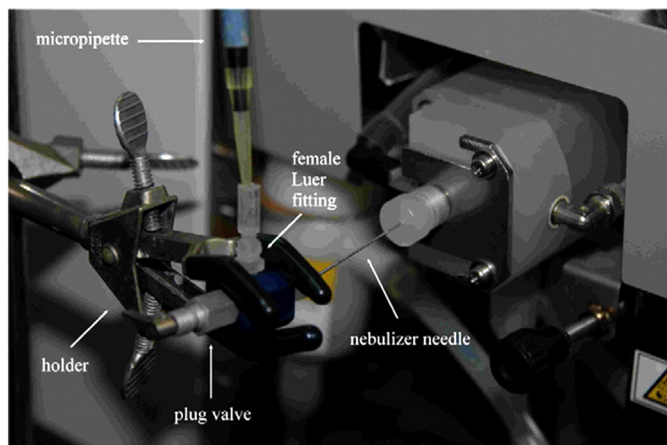


Fig. 1. The microsample introduction system in FAAS [32]. Reprinted from [32], with permission from Elsevier.

in SME will be included herein. This review focuses rather on the capabilities of the automation of liquid-phase microextraction techniques, and the more recent advances are illustrated with selected examples.

## 2. Automation of DLLME

Conventional, manually performed DLLME consists of the following steps: (i) sampling, that is the adding of an aliquot of the sample solution into a screw-cap glass test tube with a conical bottom; (ii) adding of all the required reagents (buffer, salt, ligand, etc.); (iii) rapid injection of a mixture of dispersive and extraction solvents (thus forming the dispersion, the so-called cloudy state); (iv) separation of the phases, usually through centrifugation; (v) withdrawal of the sedimented phase enriched by target analytes; (vi) if necessary, pretreatment of the collected organic phase so that it is compatible with the detection technique used (often this step consists of dilution of the sedimented phase or evaporation of the extract and subsequent reconstitution of analytes in a solvent compatible with the analytical instrument); and (vii) transferring of the extraction phase to the analytical instruments used for performing detection. Several of these steps are fairly simple and can be accomplished using a flow injection manifold. The greatest difficulties are likely to arise (i) in the automation of phase separation, which is typically done off-line and is the most time-consuming process, and (ii) in the transfer of the collected organic phase into the measuring

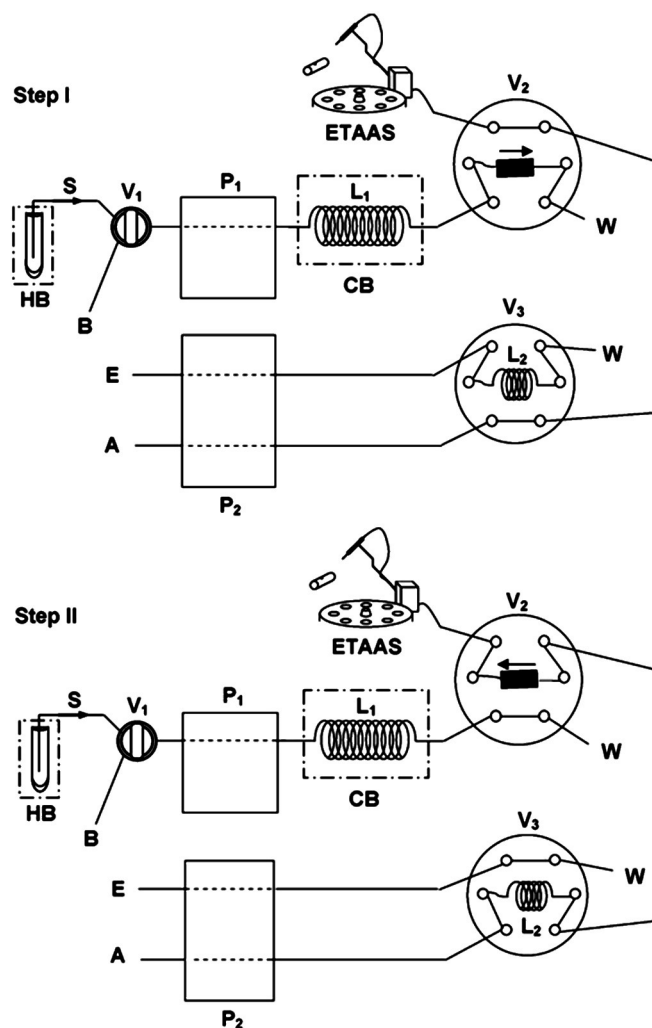


Fig. 3. A flow injection device and its operation sequence for on-line microextraction and determination of vanadium. P1 and P2, peristaltic pumps; V1, V2, and V3, injection valves; HB, hot bath; CB, cold bath; L1, reaction loop; L2, solvent loop; S, sample; B, buffer; E, eluent; A, air; W, waste [34]. Reprinted from [34], with permission from Elsevier.

equipment, because the volume of sedimented phase may be too small for some detection techniques.

To overcome the difficulties that arise in the connection of DLLME and FAAS as a result of the insufficient volume of sedimented phase,

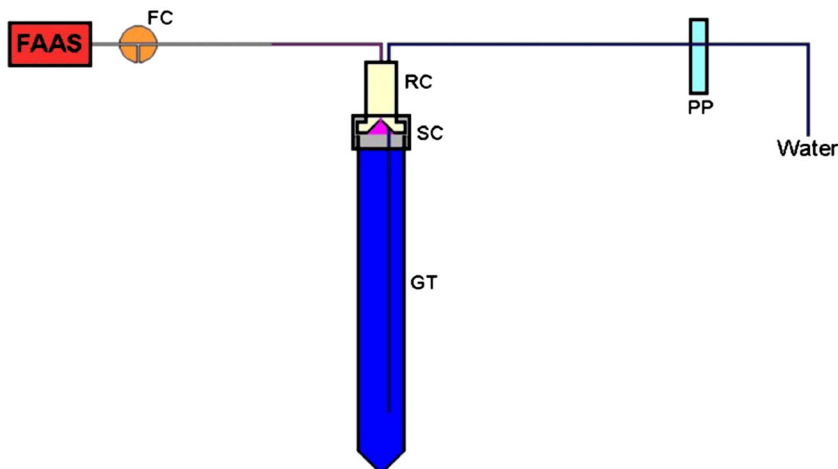


Fig. 2. Schematic of the on-line microvolume introduction system for the delivery of an extraction solvent into the FAAS. PP, peristaltic pump; GT, glass tube; SC, screw cap; RC, restriction cap; FC, flow compensation [33]. Reprinted from [33], with permission from Elsevier.

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