Why Automated Solid-Phase Extraction Is Superior to Traditional Extraction

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Executive Summary

In modern analytical chemistry laboratories, automated solid-phase extraction (SPE) instruments have been widely used in determining organic pollutants in large-volume water samples. In comparison to traditional methods such as liquid-liquid extraction (LLE) or manual SPE using vacuum a manifold, the automated SPE techniques save time, solvent and labor and ensures high productivity for laboratories.

Keywords

Solid-phase extraction, automated SPE, U.S. EPA water analysis methods, sample preparation

Solid-phase extraction (SPE) is a separation process by which the compounds of interest in a liquid mixture are immobilized on a bed and then eluted off. The beds are made of a polymeric or silica-based stationary phase in a cartridge or disk format. SPE is one of the simplest, most cost-effective and versatile methods of sample preparation. The traditional SPE techniques involve a four-step process: conditioning the stationary phase, loading samples onto the SPE cartridge, rinsing unwanted materials, and eluting the compounds of interest into collection vials.







SPE has widely been used in many environmental laboratories to pre-concentrate and cleanup samples. It is primarily performed with inexpensive vacuum pumps, manifolds, and a variety of low-cost, prepackaged, disposable cartridges or disks. Cartridges are the most popular format having a broader selection of solid phases than discs and plates. Discs are slightly more expensive than cartridges, but offer the ability to accommodate higher flow rates, which is advantageous in many environmental applications.

In modern environmental laboratories, chromatography has become faster and more sensitive. This requires sample preparation techniques to increase productivity and quality to keep pace with advancements in chromatography. The automated SPE technique was developed to address these challenges.

Analytical Precision and Accuracy

Vacuum design introduces variation in loading and elution flow rates. In a vacuum manifold, flow rates will change as the amount of solvent in SPE cartridges changes, and the flow rate can vary if one cell goes dry, which causes deterioration of reproducibility. Automated SPE also eliminates human error. With manual SPE, precision and accuracy can vary from operator to operator, batch to batch, and sample to sample. Automated SPE eliminates this variation. Modern instrument designs, especially positive pressure systems, consistently outperform manual vacuum manifold in terms of reproducibility and recovery rates.

	Dionex AutoTrace 280 SPE		Vacuum Manifold SPE	
Compound	Recovery %	%RSD	Recovery %	%RSD
Atrazine	88	1.8	54	12.2
Propazine	91	1.5	80	7.3
Alachlor	99	3.4	96	4.1
Metachlor	99	4.3	96	2.9

Table 1. Pesticide recovery study¹: Thermo Scientific[™] Dionex[™] AutoTrace[™] 280 Solid-Phase Extraction Instrument workstation vs. vacuum manifold SPE.

 $^{1}N = 6$



Figure 2. Dionex AutoTrace 280 SPE instrument.

Productivity

Environmental laboratories often use SPE to extract a small amount of organic pollutants from large volumes of drinking water or waste water samples. These processes can take up to 2–3 hours to complete. If the chemist or lab technician uses manual SPE, they have to spend hours on operating the manifold, turning the vacuum on and off, adding solvents, disposing waste, and then waiting. Fortunately, automated SPE instruments automate all four steps of SPE (cartridge conditioning, sample loading, and elution) for large-volume aqueous solution extractions. Automated SPE requires only 15–30 minutes of an operators' time, followed by 2–3 hours of unattended operation. This timesaving allows chemists to redirect their time to other tasks while the SPE is performed. More than half of the sample preparation cost for a typical vacuum manifold extraction is labor. Automated SPE provides unattended operation, thereby reducing the cost of analysis, while increasing productivity significantly.

Table 2. Total cost of liquid sample extraction.

Liquid-Liquid Technique	Extraction Cost per Sample (USD)	
Dionex AutoTrace 280 SPE Instrument	\$3.67	
Vacuum Manifold Cartridge SPE	\$12.50	
Vacuum Manifold Disk SPE	\$18.00	
Separation Funnel	\$24.75	

Vacuum manifolds have been widely used with the commercial available SPE cartridges for the separations. Dionex AutoTrace 280 SPE instrument is a high-throughput workstation dedicated specifically for automating SPE.

The Dionex AutoTrace 280 SPE instrument provides customers with the following advantages::

- Lab safety: Manual SPE requires a fume hood to minimize operators' exposure to harmful reagents including organic solvents and hazardous samples. For example, in the experiments of extracting semivolatile organic compounds from drinking water (U.S. EPA Method 5250), the operators are at risk of potentially being exposed to mutagenic or carcinogenic polycyclic aromatic hydrocarbons (PAHs). Some closed systems don't even require a fume hood to operate. Automated SPE allows operators' safer handling of these hazardous materials, therefore minimizing potential health risks.
- Method development: Method developments using manual SPE are time consuming and tedious. Due to human factors and poor reproducibility, they are often unreliable. Even when using the same manifold and SPE cartridges, a method developed in one lab may not be reproduced in another. Automated SPE makes fast method development possible. Automation from start to finish removes operator intervention and enables different laboratories to achieve reproducible analytical results.

Manual SPE will remain popular because it's easy-to-use, inexpensive, and flexible. However, the demand for high performing automated SPE instruments has increased in the last few decades due to the advantages summarized in this whitepaper. The Dionex AutoTrace 280 automated SPE instrument is superior to conventional SPE methods for large volume aqueous samples. It saves time, labor and solvent with the benefits of unattended automation, ensures analytical precision from constant flow, positive pressure operation, and improves productivity to meet the demands of modern chromatography technology.

Please visit the www.thermoscientific.com/autotrace for more information on the Dionex Auto-Trace 280 SPE instrument, and contact your local Thermo Fisher Scientific sales representative for an instrument demonstration or information on current sales promotions.

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