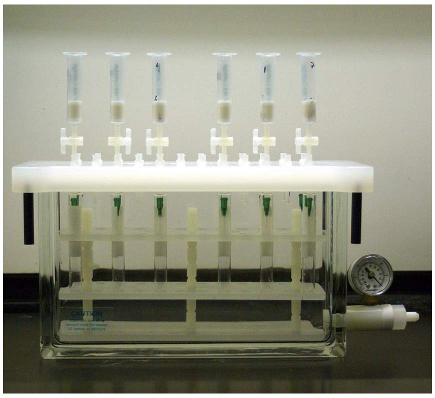
Solid phase extraction

Solid-phase extraction (**SPE**) is a separation process by which compounds that are dissolved or suspended in a liquid mixture are separated from other compounds in the mixture according to their physical and chemical properties. Analytical laboratories use solid phase extraction to concentrate and purify samples for analysis. Solid phase extraction can be used to isolate analytes of interest from a wide variety of matrices, including urine, blood, water, beverages, soil, and animal tissue.[1]



A typical solid phase extraction manifold. The cartridges drip into the chamber below, where tubes collect the effluent. A vacuum port with gauge is used to control the vacuum applied to the chamber.

SPE uses the affinity of solutes dissolved or suspended in a liquid (known as the mobile phase) for a solid through which the sample is passed (known as the stationary phase) to separate a mixture into desired and undesired components. The result is that either the desired analytes of interest or undesired impurities in the sample are retained on the stationary phase. The portion that passes through the stationary phase is collected or discarded, depending on whether it contains the desired analytes or undesired impurities. If the portion retained on the stationary phase includes the desired analytes, they can then be removed from the stationary phase for collection in an additional step, in which the stationary phase is rinsed with an appropriate eluent.

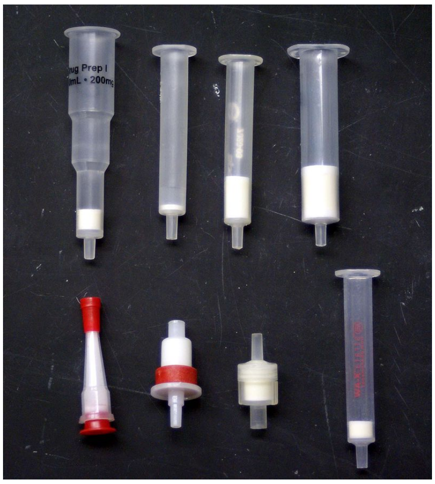
The stationary phase comes in the form of a packed syringe-shaped cartridge, a 96 well plate or a 47- or 90-mm flat disk, each of which can be mounted on its specific type of extraction manifold. The manifold allows multiple samples to be processed by holding several SPE media in place and allowing for an equal number of samples to pass through them simultaneously. A typical cartridge SPE manifold can accommodate up to 24 cartridges, while a typical disk SPE manifold can accommodate 6 disks. Most SPE manifolds are equipped with a vacuum port. Application of vacuum speeds up the extraction process by pulling the liquid sample through the stationary phase. The analytes are collected in sample tubes inside or below the manifold after they pass through the stationary phase.

Solid phase extraction cartridges and disks are available with a variety of stationary phases, each of which can separate analytes according to different chemical properties. Most stationary phases are based on silica that has been bonded to a specific functional group. Some of these functional groups include hydrocarbon chains of variable length (for reversed phase SPE), quaternary ammonium or amino groups (for anion exchange), and sulfonic acid or carboxyl groups (for cation exchange).[2]

Normal Phase SPE procedure

A typical solid phase extraction involves four basic steps. First, the cartridge is equilibrated with a non-polar solvent or slightly polar, which wets the surface and penetrates the bonded phase. Then water, or buffer of the same composition as the sample, is typically washed through the column to wet the silica surface. The sample is then added to the cartridge. As the sample passes through the stationary phase, the analytes in the sample will interact and retain on the sorbent while the solvent, salts, and other impurities pass through the cartridge. After the sample is loaded, the cartridge is washed with buffer or solvent to remove further impurities. Then, the analyte is eluted with a non-polar solvent or a buffer of the appropriate pH.

A stationary phase of polar functionally bonded silicas with short carbons chains frequently makes up the solid phase. This stationary phase will adsorb polar molecules which can be collected with a more polar solvent.[3]



A selection of solid phase extraction cartridges, available in many sizes, shapes, and types of stationary phase.

Reversed phase SPE

Reversed phase SPE separates analytes based on their polarity. The stationary phase of a reversed phase SPE cartridge is derivatized with hydrocarbon chains, which retain compounds of mid to low polarity due to the hydrophobic effect. The analyte can be eluted by washing the cartridge with a non-polar solvent, which disrupts the interaction of the analyte and the stationary phase.[4]

A stationary phase of silicon with carbon chains is commonly used. Relying on mainly non-polar, hydrophobic interactions, only non-polar or very weakly polar compounds will adsorb to the surface.[5]

Ion exchange SPE

Ion exchange sorbents separate analytes based on electrostatic interactions between the analyte of interest and the positively charged groups on the stationary phase. For ion exchange to occur, both the stationary phase and sample must be at a pH where both are charged.

Anion exchange

Anion exchange sorbents are derivatized with positively charged functional groups that interact and retain negatively charged anions, such as acids. Strong anion exchange sorbents contain quaternary ammonium groups that have a permanent positive charge in aqueous solutions, and weak anion exchange sorbents use amine groups which are charged when the pH is below about 9. Strong anion exchange sorbents are useful because any strongly acidic impurities in the sample will bind to the sorbent and usually will not be eluted with the analyte of interest; to recover a strong acid a weak anion exchange cartridge should be used. To elute the analyte from either the strong or weak sorbent, the stationary phase is washed with a solvent that neutralizes the charge of either the analyte, the stationary phase, or both. Once the charge is neutralized, the electrostatic interaction between the analyte and the stationary phase no longer exists and the analyte will elute from the cartridge.[6]

Cation Exchange

Cation exchange sorbents are derivatized with functional groups that interact and retain positively charged cations, such as bases. Strong cation exchange sorbents contain aliphatic sulfonic acid groups that are always negatively charged in aqueous solution, and weak cation exchange sorbents contain aliphatic carboxylic acids, which are charged when the pH is above about 5. Strong cation exchange sorbents are useful because any strongly basic impurities in the sample will bind to the sorbent and usually will not be eluted with the analyte of interest; to recover a strong base a weak cation exchange cartridge should be used. To elute the analyte from either the strong or weak sorbent, the stationary phase is washed with a solvent that neutralizes ionic interaction between the analyte and the stationary phase.[7]

References

[1] Supelco (1998), *Guide to Solid Phase Extraction* (http://www.sigmaaldrich.com/Graphics/Supelco/objects/4600/4538.pdf), pp. 9,
[2] Supelco (1998), *Guide to Solid Phase Extraction* (http://www.sigmaaldrich.com/Graphics/Supelco/objects/4600/4538.pdf), pp. 2–12,
[3] Supelco (1998), *Guide to Solid Phase Extraction* (http://www.sigmaaldrich.com/Graphics/Supelco/objects/4600/4538.pdf), pp. 3–4,
[4] Supelco (1998), *Guide to Solid Phase Extraction* (http://www.sigmaaldrich.com/Graphics/Supelco/objects/4600/4538.pdf), pp. 3,
[5] Supelco (1998), *Guide to Solid Phase Extraction* (http://www.sigmaaldrich.com/Graphics/Supelco/objects/4600/4538.pdf), pp. 3,
[6] Supelco (1998), *Guide to Solid Phase Extraction* (http://www.sigmaaldrich.com/Graphics/Supelco/objects/4600/4538.pdf), pp. 3,
[7] Supelco (1998), *Guide to Solid Phase Extraction* (http://www.sigmaaldrich.com/Graphics/Supelco/objects/4600/4538.pdf), pp. 4,

Further reading

E. M. Thurman, M. S. Mills, Solid-Phase Extraction: Principles and Practice, Wiley-Interscience, 1998, ISBN 978-0471614227

Nigel J.K. Simpson, Solid-Phase Extraction: Principles, Techniques, and Applications, CRC, 2000, ISBN 978-0824700218

James S. Fritz, Analytical Solid-Phase Extraction, Wiley-VCH, 1999, ISBN 978-0471246671

Watch a slide show and learn Why Prepare a Sample (http://www.appliedseparations.com/wiki/SPE01/player.html).
Watch a slide show and learn about SPE Functional Groups (http://www.appliedseparations.com/wiki/SPE02/player.html).
Watch a slide show and learn Three Simple Steps to SPE (http://www.appliedseparations.com/wiki/SPE03/player.html).
Watch a slide show and video: Making an SPE Sample (http://www.appliedseparations.com/wiki/SPE04/player.html).
Watch a slide show and learn about SPE Sorbent and Cartridge Selection (http://www.appliedseparations.com/wiki/SPE05/player.html).

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