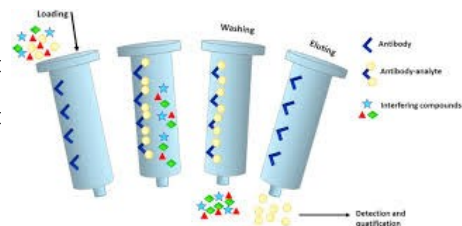


Solid Phase Extraction Quick Reference Guide

Solid-Phase extraction (SPE) is a technique that extracts compounds from a sample matrix based on their physical and chemical properties. The goal of any SPE method should be to efficiently extract compounds of interest removing them from the bulk sample environment. In addition to this the goal should also be to reject unwanted elements of the sample matrix that would otherwise prove problematic in the analytical method in terms of inaccuracies of data or shortening the life time for example of expensive chromatographic columns or potentially excessive instrument downtimes and increased service costs. SPE as a clean-up and concentration technique can be applied to almost any sample type typically these could be urine, blood, water, beverages, soil, and animal tissue. Given the sheer number of chemical compounds required to be extracted and analyzed from a wide range of sample types and also the increase complexity of analytical instrumentation (GC/MS, LC/MS), the greater the need is for amore targeted and efficient clean process.

The SPE Procedure (Non – Polar and Polar Extractions)

- **SPE Pre -Elution**
Removes unwanted compounds that may have been inadvertently absorbed from the environment ahead of the extraction.
- **Conditioning Step**
The solvent penetrates the bond phase silica and opens the C18/C8/C2 chains enabling them to interact with any sample put the through the cartridge at the sample application step.
- **Rinse Step**
Use a solvent usually a buffer that is as close to or as same as the sample type. This creates the best chance for sample analyte(s) of interest to move from the sample matrix when the sample is applied to the SPE sorbent material.
- **Sample Application**
In the case of a C18 based extraction the sample is usually aqueous and looks to drive non-polar compounds from that environment onto the SPE sorbent also known as hydrophobic interaction. This would be classed as a non-polar extraction. Polar extractions would for example exploit the use of bare silica and sample types with their analytes presented to the SPE device in an organic solvent
- **SPE Sample Wash Step**
Rinses from the phase unwanted elements of the sample matrix whilst leaving behind still bound to the phase the analytes/compounds of interest. This step usually requires the most experimentation during method development.
- **SPE Elution Step**
This removes the analytes of interest from the SPE Sorbent phase into a collection vessel as up to this point all other liquids will have been sent to waste.



SPE Hardware

Typically, the SPE sorbent phases are packed into polypropylene or glass syringe barrel type cartridges, 96 well plates or flat disks that are 47 or 90mm in diameter. The type of SPE device used is then usually mounted on a vacuum or positive pressure manifold. A cartridge manifold usually will allow up to 24 samples to be placed in-situ whilst an SPE disk processing station would allow 6 samples to be processed.



Notable Variations

- **Ion exchange SPE**
Exploit retention because of electrostatic interactions between the analyte(s) of interest and charges on the bonded phase silica according to whether they carry a positive or negative charge. For optimal ion exchange to occur the pH of the both the sample and the bonded phase must be charged.
- **Anion exchange**
These sorbents have positive functional groups bonded to the silica and will interact with negatively charged anions. Typically, a strong anion exchanger (SAX) would have a quaternary ammonium ion and is permanently charged in an aqueous solution. Weak anion exchangers (WAX) carry amine groups that are only charged below a pH of 9. Strong anion sorbents are particularly useful as they will irreversibly bind any strongly acidic impurities in the sample that will not then get eluted with target analytes. To recover for example a strong acid an according weak anion exchanger be used. WAX or SAX involves an elution solvent that neutralise the sorbent target analyte(s) or both by breaking the electrostatic interactions that exist and releases the target analyte(s).
- **Cation exchange**
These phases carry a negative charge typically in the case of a strong cation exchanger (SCX) sulphonic acid that is permanently negatively charged in an aqueous solution or weak cation exchanger (WCX) i.e. carboxylic acid. WCX is usually charged above a pH 5. Strongly basic impurities will be irreversibly bound to the sorbent and will not be eluted. To recover a strong bases always use a weak cation exchanger. For both SCX and WCX sorbents to elute compounds of interest an elution solvent should be used that neutralise the charge on the sorbent, analyte, or both washed with a solvent that neutralizes ionic interaction between the analyte and the stationary phase.

Conclusion

If these guidelines are adhered to you will avoid undesirable outcomes and have a fully utilized robust method providing accurate and reliable results fit for purpose.

ChromSolutions Ltd

What we offer at ChromSolutions is our wealth of experience in analytical instrument sales and support (over 110 years distributed through the members of our company). We can help you from defining your requirements to the implementation of a robust SPE method fit for purpose.

For more information on solid phase microextraction please contact us:

