

# Solvent Selection

- Influences the retention of the compound on the sorbent and its subsequent elution.
- The solvent polarity determines the solvent strength.
- For a normal phase solvent, both solvent polarity and solvent strength are coincident whereas this is not the case for a reversed phase sorbent.
- In practice, the solvents normally used for reversed phase sorbents are restricted to water, methanol, isopropyl alcohol and acetonitrile.
- For ion exchange sorbents, solvent strength is not the main effect.



# Factors Affecting SPE

- The choice of SPE sorbent is highly dependent upon the compound of interest.
- The number of active sites available on the sorbent cannot be exceeded by the number of molecules of compound.
- It is important to assess the capacity of the SPE cartridge or disc for its intended application.
- The flow rate of the sample through the sorbent is important.
- The choice of solvent is critical.



# Applications of Normal Phase SPE

- Normal phase (NP) SPE refers to the sorption of the functional groups of the compound (solute) from a non-polar solvent to the polar surface of the stationary phase.
- The stationary phase such as silica gel, Florisil ( $\text{MgSiO}_3$ ) and alumina ( $\text{Al}_2\text{O}_3$ )
- The mechanism of sorption involves polar interactions such as hydrogen bonding, dipole–dipole interactions,  $\pi$ – $\pi$  interactions and induced dipole–dipole interactions.



# Applications of Reversed Phase SPE

- Reversed phase (RP) SPE refers to the sorption of organic solutes from a polar mobile phase, such as water or aqueous solvent, into a non-polar stationary phase, such as a C8 or C18 sorbent.
- The sorption mechanism involves the interaction of the solute within the chains of the stationary phase, i.e. van der Waals or dispersion forces.



# Applications of Ion Exchange SPE

- Ion exchange SPE has been used in the separation of ionic compounds from either a polar or non-polar solvent to the oppositely charged ion exchange sorbent, such as benzenesulfonic acid, propanesulfonic acid and quaternary amines.
- The separation mechanism involves ionic interaction; hence, a polar compound may be effectively separated from polar solvents, including water, as well as less polar organic solvents.



# Solid Phase Microextraction

- Solid phase microextraction (SPME) is the process whereby an organic compound is adsorbed onto the surface of a coated-silica fibre as a method of pre-concentration.
- This is followed by desorption of the organic compounds into a suitable instrument for separation and quantitation.
- The most important stage of this two-stage process is the adsorption of a compound onto a suitably coated-silica fibre or stationary phase.
- The choice of sorbent is essential.



- The most reported stationary phase for SPME is polydimethylsiloxane (PDMS). This non-polar phase has been utilized for the extraction of a range of non-polar compounds.
- The fused-silica polydimethylsiloxane-coated fibre is stable at high temperatures. This stability and its small physical diameter and cylindrical geometry allow the fibre to be incorporated into a syringe-like holder.
- SPME has been exploited most effectively when coupled to gas chromatography (GC).

