Solid Phase Extraction

- Popular sample preparation method used for isolation, enrichment and/or clean-up of components of interest from aqueous samples.
- SPE normally involves bringing an aqueous sample into contact with a solid phase or sorbent whereby the compound is selectively adsorbed onto the surface of the solid phase prior to elution.
- The solid phase sorbent is usually packed into small tubes or cartridges.

Types of SPE Media (Sorbent)

- SPE sorbents can be divided into three classes, i.e. normal phase, reversed phase and ion exchange.
- The most common sorbents are based on silica particles to which functional groups are bonded to surface silanol groups to alter their retentive properties.
- It is the nature of the functional groups that determines the classification of the sorbent.

- **Normal phase sorbents** have polar functional groups.
- **Reversed phase sorbents** have non-polar functional groups.
- **Ion exchange sorbents** have either cationic or anionic functional groups and when in the ionized form attract compounds of the opposite charge.

Multimodal Extractions

Multimodal SPE can be done in one of two ways: either by connecting two alternate phase SPE cartridges in series or by having two different functional group sorbents present within one cartridge.

SPE Formats and Apparatus

- The most common arrangement is the syringe barrel or cartridge.
- The cartridge itself is usually made of polypropylene (although glass and polytetrafluorethylene, PTFE, are also available) with a wide entrance, through which the sample is introduced, and a narrow exit.
- The appropriate sorbent material, ranging in mass from 50 mg to 10 g, is positioned between two frits, at the base (exit) of the cartridge, which act to both retain the sorbent material and to filter out particulate matter.
- Typically the frit is made from polyethylene with a 20 μm pore size.

- Solvent flow through a single cartridge is typically done using a side-arm flask apparatus.
- Multiple cartridges can be simultaneously processed (from 8 to 30 cartridges) using a commercially available vacuum manifold.

In both cases a vacuum pump is required to affect the movement of solvent/sample through the sorbent.

Method of SPE Operation

What are the five stages of SPE operation?

- wetting the sorbent;
- conditioning of the sorbent;
- loading of the aqueous sample;
- rinsing or washing the sorbent
- elution of the compound of interest.

- **Wetting the sorbent** allows the bonded alkyl chains to be solvated so that they 'spread open' to form a 'bristle'.
- **Conditioning of the sorbent** in which solvent or buffer, similar in composition to the aqueous sample that is to be extracted, is pulled through the sorbent.
- **Sample loading** where the sample is forced through the sorbent material by suction, a vacuum manifold or a plunger.
- **Washing with a suitable solvent** that allows unwanted extraneous material to be removed without influencing the elution of the compound of interest.
- **The compound of interest is eluted** from the sorbent using the minimum amount of solvent to affect quantitative release.