



Université  
de Strasbourg

Licence 3

Analytical Chemistry

## Sample preparation

Enseignant : Y. FRANCOIS

## Sample preparation

### Introduction

- Many samples are not injectable directly into the state in the chromatograph
- Analytical science often requires preprocessing of the sample
- Sample preparation can be very complex and represents the bulk of the analyst's work
- It's a real science

## Sample preparation

### Techniques :

- Dissolution
- Distillation
- Extraction
- Adsorption methods
  - ✓ SPE
  - ✓ SPME

## Sample preparation

### Dissolution:

- Solvent choice:
  - ✓ Solubility
  - ✓ Absence of a chemical reaction with the sample
  - ✓ Solvent purity
- Inertia of the solvent
  - ✓ Peroxide formation
- Corrosive properties (chlorinated)
- toxicity

Sample preparationTechniques :

- Dissolution
- Distillation
- Extraction
- Adsorption methods
  - ✓ SPE
  - ✓ SPME

Sample preparationDistillation :

- Simple distillation
- Rotary evaporator
- Fractional distillation

Goal:

- Separate volatile from non-volatile
- Allow to concentrate by eliminating light compounds or removing heavy ones

Sample preparationTechniques :

- Dissolution
- Distillation
- Extraction
- Adsorption methods
  - ✓ SPE
  - ✓ SPME

Préparation de l'échantillonExtraction :

- Conventional solvent extraction
- Extraction by fluid in supercritical phase

Goal:

- Extract the compounds selectively from a complex mixture
- Example : extraction of eugenol contained in clove

Sample preparationTechniques :

- Dissolution
- Distillation
- Extraction
- Adsorption methods
  - ✓ SPE
  - ✓ SPME

Sample preparationAdsorption method:

- Adsorption of liquids: solid phase extraction SPE or SPME

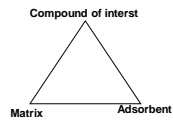
Goal:

- Concentration of minor constituents of a gas matrix
- elective extraction of compounds from a complex mixture
- Direct injection of a compound into a GC

Sample preparationAdsorption of liquids: SPE

- Solid Phase Extraction (SPE): use of cartridges or disks filled with various adsorbents
- Used either to concentrate or to simplify the matrix

The procedure responds to the equilibrium triangle between:  
compound of interest - matrix - adsorbent

Sample preparationSPE : Principle

- Similar to liquid chromatography
- Some molecules are blocked on an adsorbent and are selectively eluted by unhooking them from the support with a specific solvent

### Sample preparation

#### SPE : procedure

First Step: conditioning and solvation

- ✓ Washing with a suitable solvent
- ✓ The cartridge should no longer be dried

Second Step: equilibration

- ✓ Equilibration of the cartridge with the sample solvent

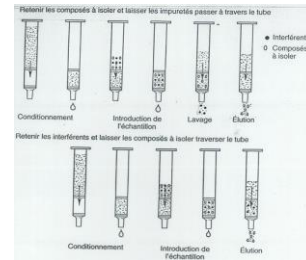
Third Step: deposit of the sample

Fourth Step: elution

- ✓ A solvent of suitable polarity is introduced into the cartridge
- ✓ Allows, depending on the choice of solvent, to elute the products to be isolated and to retain the interferences or vice versa

### Sample preparation

#### SPE : procedure



### Sample preparation

#### SPE : Aims

- Isolation of a compound from a complex mixture
- Sample concentration
- Sample purification
- Solvent change
- The 4 goals at once

### Sample preparation

#### SPE : Advantages

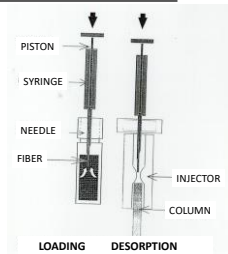
- Fast, selective technique with low solvent consumption
- Extraction of one or more compounds from a complex matrix
- Trace enrichment (preconcentration)
- Desalting solutions
- Online derivatization

Sample preparationSolid Phase Microextraction: SPME

- Use of a special syringe with a hollow needle in which slides a fused silica fiber covered with a thin layer of stationary phase
- Adsorption of a compound of interest on this fiber

**Selective Extraction**Sample preparationSolid Phase Microextraction: SPME

- Fiber can be used either in immersion or in head space
- Fiber then desorbed directly into the GC injector
- Avoids the use of solvents
- Reusable fiber

Sample preparationSolid Phase Microextraction: SPMESample preparationSolid Phase Microextraction: SPMEHead space method

- Volatile constituents analysis of a sample when it is not desirable to introduce the entire sample into the GC

