

## 60623 - Sample preparation for analysis

### Información del Plan Docente

Academic Year	2016/17
Academic center	100 - Facultad de Ciencias
Degree	542 - Master's in Chemical Research
ECTS	3.0
Course	1
Period	First semester
Subject Type	Optional
Module	---

### 1. Basic info

#### 1.1. Recommendations to take this course

#### 1.2. Activities and key dates for the course

### 2. Initiation

#### 2.1. Learning outcomes that define the subject

#### 2.2. Introduction

### 3. Context and competences

#### 3.1. Goals

#### 3.2. Context and meaning of the subject in the degree

#### 3.3. Competences

#### 3.4. Importance of learning outcomes

### 4. Evaluation

### 5. Activities and resources

#### 5.1. General methodological presentation

The learning process that is designed for this subject is based on the following:

\* 12.5 hours of participatory lectures. The contents of each issue are discussed, alternating theory with examples, issues and problems. The dynamics of these sessions will be fundamentally exhibition, open to discussion and questions about the material available (books, articles, material prepared by the teacher). The keynote sessions should serve to establish the theoretical bases of the different techniques are developed.

\* 5 hours of problems and cases. Each teaching unit will have questionnaires for independent student work, which also will assess your progress.

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\* 2.5 hours of educational work. It is the study of real cases. Students compile reports and subsequently make an oral presentation.

\* 10 hours of laboratory practices. The laboratory sessions will be made individually or in small groups supervised by the teacher.

### 5.2.Learning activities

### 5.3.Program

#### BLOCK I. Introduction

Unit 1. Global approach to sample preparation in the analytical process. Change of state of aggregation. Separation. Concentration. Interference resolution. Derivatization

Unit 2. Treatments before sample preparation. Storage and preservation of samples. Ground and dried solid sample. Filtering liquid and gaseous samples.

#### BLOCK II. Preparation of samples for inorganic analysis

Unit 3. Dissolution / leaching. Inorganic matrices: dissolution without chemical change, acid dissolution, disintegration. Organic and biological matrices: calcination, acid digestion. Microwave assisted dissolution. Preparation of suspensions or slurries.

Unit 4. Separation / preconcentration. Liquid-liquid extraction. Solid phase extraction.

Unit 5. Derivatization. Metal complex formation. Formation of volatile species.

Unit 6. Preparation of samples for speciation analysis

#### BLOCK III. Preparation of samples for organic analysis

Unit 7. Liquid matrices. Liquid-liquid extraction. Liquid extraction (drop) -gas (static headspace). Solid-liquid extraction (SPE) directly or headspace (static and dynamic -purga and trap-). Solid-liquid microextraction (SPME) or direct headspace. Extraction Stir Bar

Unit 8. Gas matrices. Sampling site: balls, sorbents (traps, SPME) and membranes. Direct analysis. Thermal desorption (thermal, traps, SPME). Desorption solvent.

Unit 9. Solid matrices. Solid-liquid extraction: Soxhlet and variants (Soxtec), accelerated solvent extraction (ASE PLE), microwave assisted extraction (MAE), ultrasonic assisted extraction (bath and probe). Supercritical fluid extraction (SFE). QuEChERS

Unit 10. Special techniques for volatile analytes. Headspace (HS, Purge and trap). SPME

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Unit 11. Derivatization. Formation of volatile compounds for GC analysis (acylation, alkylation, silylation). Formation of fluorescent compounds for HPLC analysis

### 5.4.Planning and scheduling

### 5.5.Bibliography and recommended resources

#### Bibliography

1. Chemical Separations. Principles, techniques and experiments. Clifton E. Meloan. Willey-Interscience. 1999
2. Sample Preparation Techniques in Analytical Chemistry, Edited by Somenath Mitra, 2003 John Wiley & Sons, Inc.
3. Handbook of Sample Preparation. Janusz Pawliszyn (Editor), Heather L. Lord (Editor), 2010, Wiley-Blackwell.
4. Microwave-Enhanced Chemistry. Fundamentals, Sample Preparation and applications. Edited by H.M. Kingston, S.J. Haswell, 1997, American Chemical Society.