

SUBJECT GUIDE

Academic year 2017-2018

ANALYTICAL TECHNIQUES

| MODULE   | CONTENT              | YEAR            | TERM   | CREDITS | TYPE      |
|--|----------------------|-----------------|--|---------|-----------|
| Chemistry  | Analytical Chemistry | 1 <sup>st</sup> | 1 <sup>st</sup>  | 6       | Mandatory |
| <b>PROFESSOR(S)</b>  |                      |                 | <b>Postal address, telephone n°, e-mail address</b>  |         |           |
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| <b>DEGREE WITHIN WHICH THE SUBJECT IS TAUGHT</b>   |                      |                 |  |         |           |
| Degree in Science and Technology of Foods  |                      |                 |  |         |           |
| <b>PREREQUISITES and/or RECOMMENDATIONS (if necessary)</b>   |                      |                 |  |         |           |
| Having studied the subjects: Basic Principles of Chemistry, Organic Chemistry, Applied Physics and Physical Chemistry,<br>Have adequate knowledge about: <ul style="list-style-type: none"> <li>• Formulation in Inorganic and Organic</li> <li>• Basic Mathematics</li> </ul>                         |                      |                 |  |         |           |
| <b>BRIEF DESCRIPTION OF CONTENTS</b>   |                      |                 |  |         |           |
| Analytical Methodology<br>Qualitative and Quantitative Chemical Analysis<br>Description of the instrumentation used in food analysis<br>Spectroscopic techniques applied to food analysis<br>Separation techniques applied to food analysis.<br>Electroanalytical techniques applied to food analysis. |                      |                 |  |         |           |
| <b>OBJECTIVES</b>  |                      |                 |  |         |           |
| <ul style="list-style-type: none"> <li>• Introduce students to the study of the methodology of analytical techniques.</li> <li>• Provide students with the criteria for how to choose the appropriate technique of analysis</li> </ul>   |                      |                 |  |         |           |



- Give students the minimum skills necessary for the numerical solution of problems related to Analytical Techniques.
- Train the student for proper handling common Analytical Techniques in chemical analysis laboratories.
- Ensure that the student is able to express themselves before a specialized or unspecialized audience.
- Develop self-learning ability

## DETAILED SUBJECT SYLLABUS

### THEORETICAL PROGRAM:

- **Theme 1. Introduction to Analytical Methodology: The Analytical Process.**

Definition of the analytical problem. Analytical properties. Steps to the analytical process. Difference between process, technique, method and procedure. Background information about the purpose of analysis. Sources of analytical information. Analytical methods: classification. Advantages and disadvantages of analytical methods. Criteria to consider in choosing an analytical method.

- **Theme 2. Collection and sample preparation.**

The sample: Terminology. Sample types, Sampling Plan. Sampling Strategy. Handling and storage of samples: labeling and storage of the sample. Good Sampling Techniques. Sampling plan. Sampling errors. Pretreatment of the sample. Sample preparation for analysis: organic and inorganic constituents. Solution, Solid-Liquid Extraction, Liquid-Liquid Extraction, Solid Phase Extraction.

- **Theme 3. Introduction to analytical measurements. Expression and evaluation of analytical results.**

Analytical property. Analytical property measurement. Interference. Interference control. Relationship between experimental variables. Calculations based on functional relationships. Calibration: (Standard, Standard addition, Internal Standard) quality parameters. precision, accuracy, sensitivity, linear range. Limits of detection and quantification. Truth. Variability of experimental data. Types of experimental errors. Statistics in chemical analysis. Rejection anomalous results. And upper confidence interval of the mean. Comparison of values. Significant figures. Traceability and uncertainty. Validation of an analytical method.

- **Theme 4. Electroanalytical Techniques.**

Introduction. Electroanalytical Instrumentation. Classification of electroanalytical techniques. Techniques not based on electrochemical reactions: Techniques conductivity. Potentiometric techniques. Indicator electrodes. Characteristics of the ion-selective electrodes. Analytical applications. Techniques based on electrochemical reactions: Total electrolysis Techniques. Electrogravimetry. Coulometry. Negligible electrolysis techniques. Introduction to Voltammetric techniques.

- **Theme 5. SEPARATION TECHNIQUES: GAS CHROMATOGRAPHY.**

Introduction. General concepts. Classification of chromatographic techniques. The chromatographic process: Retention, balance and elution. Chromatogram and chromatographic band: chromatographic parameters. Band broadening. The general problem of the elution. Chromatography flat. Gas chromatography. The gas chromatograph. Carrier gas. Systems for sample introduction. Types of columns. Types of detection systems stationary phases. Data processing. Applications of gas chromatography in foods Analysis

- **Theme 6. SEPARATION TECHNIQUES: LIQUID CHROMATOGRAPHY.**

Introduction. Types of chromatography. Strength of the solvent. Elution rates. The liquid chromatograph. Mobile phase Supply Systems high pressure pump. Mobile phase composition, elution gradient formation. Sample injection systems. Chromatographic columns. Detection systems: types and general characteristics. Data processing. Applications of liquid chromatography. Application to the analysis and quality control of foods. Methods of analysis of common components of food.



- **Theme 7. - ELECTROPHORESIS.**

Electrokinetic phenomena. Zone electrophoresis. Factors affecting electrophoresis. Types of electrophoresis. Capillary electrophoresis.

- **Theme 8. MASS SPECTROMETRY.**

Mass spectra. Mass Spectrometer Components. Sample Introduction. Ionization Methods: Electron Impact (EI), chemical ionization (CI); FAB -LSIMS, MALDI, Electrospray (ESI) and atmospheric pressure ionization (APCI)). Mass analyzers: magnetic sector, quadrupole, ion trap (ITD) Time of Flight (TOF), Fourier Transform Mass Spectrometry (FTMS). Detectors. Interpretation of mass spectra. Applications of Mass Spectrometry

- **Theme 9. - INTRODUCTION TO SPECTROSCOPY.**

General concepts. Nature and properties of the electromagnetic radiation. Photoelectric effect. Energy levels of molecules. Selection rules. Regions of the electromagnetic spectrum. Radiation absorption: Beer-Lambert Law. Limitations of the Beer. Absorbance range and minimum transmittance basic error. Basic design of an absorption instrument.

- **Theme 10. ELECTRONIC SPECTROSCOPY: UV-VIS.**

Electronic spectra. Selection rules. Main types of electronic transitions in polyatomic molecules. Chromophore and auxochrome groups. Instrumentation. Application to food-dye  $pK_a$  determination.

- **Theme 11. - FLUORESCENCE SPECTROSCOPY.**

Theoretical basis of the fluorescence spectroscopy. Molecular relaxation processes. Radiative and no radiative processes. Quantum yield. Fluorescence lifetimes. Bimolecular deactivation. Factors affecting the fluorescence intensity: Kavanagh Act. Instrumentation: radiation sources, monochromators, detectors. Types of spectra.

- **Theme 12. - ATOMIC SPECTROSCOPY.**

**12.1 Introduction to Atomic Spectroscopy.** Atomic spectra and selection rules. Effect of Temperature in the atomic spectra.

**12.2 Atomic Absorption Spectroscopy.** Radiation Sources. Flame atomization. Electrothermic atomization. Spectrophotometers types. Interferences.

**12.3 Atomic Emission Spectroscopy.** Plasma techniques Excitation Sources: Inductively Coupled. Plasma Direct-Current Plasma. Microwave Plasma. Spark and Arc. Types of Spectrophotometers. Applications. Flame photometry. Determination of trace elements in food.

#### Seminars

- Resolving numeric problems/ Project Exhibition

#### PRACTICAL PROGRAM:

##### Laboratory Practice

- Lab 1: Determination of caffeine in beverages by UV-Vis Spectrometry using the Beer's law.
- Lab 2: Determination of quinine in tonic by Fluorescence Spectroscopy using Kavanagh's law.
- Lab 3: Determination of conductivity, dry residue and fluoride in a water sample.
- Lab 4: Determination of isoflavones in milk by HPLC.

Exhibition / Resolution of Academically Directed Activities



## READINGS

### FUNDAMENTAL BIBLIOGRAPHY

- Principios de Análisis Instrumental. (6<sup>th</sup> Ed.) Skoog-Holler-Nieman, Cengage Learning Editores, 2008.
- Espectroscopia Atómica y Molecular. J. Zúñiga Román. Pearson Educación.
- Fundamentos de Espectroscopia Molecular. C.N. Banwell. Ediciones del Castillo.
- Análisis Instrumental. K.A. Rubinson- J.F. Rubinson. Editorial Prentice Hall.
- Métodos Instrumentales de Análisis. H.H. Willard y col. Grupo Editorial Iberoamérica.
- Análisis Químico, Ramiro Avidad, Ignacio de Orbe. Universidad de Granada 2006
- Análisis Químico Cuantitativo, Daniel C. Harris, 2<sup>a</sup> Edición, Ed. Reverté, 2001.
- Fundamentos de Química Analítica, D. A. Skoog, D. M. West, F. J. Holler y S. R. Crouch, 8<sup>a</sup> Edición, Ed. Thomson, 2005.

### COMPLEMENTARY BIBLIOGRAPHY

- Estadística y Quimiometría para Química Analítica, Miller y Miller. Ed. Prentice Hall. 2002.
- Toma y tratamiento de muestra, C. Cámara, P. Fernández, A. Martín-Esteban, C. Pérez-Conde y M. Vidal, Ed. Síntesis, Madrid 2004.
- Garantía de calidad en los laboratorios analíticos, R. Compañó y A. Ríos. Editorial Síntesis, 2002.
- Espectroscopia molecular. V. Luaña. Servicio de publicaciones de la Universidad de Oviedo.
- Fluorescent Spectroscopy. A.J. Pesce. Marcel Dekker. New Cork.
- Técnicas Instrumentales Físicoquímicas. S. Senent. Publicaciones UNED
- Química Física (Vol. 1). M. Díaz Peña, A. Roig Muntaner. Editorial Alhambra. Química Física. P. Atkins. (8<sup>a</sup> Ed). Editorial Médica Panamericana.
- Físicoquímica. (Vol. 2). Ira N. Levine. 5<sup>a</sup> Ed. Editorial Mc. Graw Hill.
- Química Física. J. Morcillo Rubio. 2<sup>a</sup> Ed. Publicaciones UNED.
- Aplicación de las Nuevas Tecnologías a la Enseñanza Práctica de Técnicas Instrumentales. Proyecto de Innovación Docente 07-02-08 subvencionado por la UGR Ref. MVGPI2007
- Aplicación de las Nuevas Tecnologías a la Enseñanza Práctica de Técnicas Instrumentales (2<sup>a</sup> parte). Proyecto de Innovación Docente con Código N° 08-22 subvencionado por la UGR Ref. MVG/PI2008
- Nutrición y Alimentación humana. José Mataix Verdu Ed. ERGON (2<sup>a</sup> Edición) 2009 Madrid.
- Aplicación de las Nuevas Tecnologías a la Enseñanza Práctica de Técnicas Instrumentales. Proyecto de Innovación Docente 07-02-08 subvencionado por la UGR Ref. MVGPI2007
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- Analisis Instrumental, Isabel Sierra Alonso, Netbiblo, 2010
- Técnicas analíticas de contaminantes químicos: Aplicaciones toxicológicas, medioambientales y alimentarias Miguel Ángel Sogorb Sánchez, Eugenio Vilanova Gisbert, Ediciones Díaz de Santos S.A., 2004
- Instrumental Methods in Food Analysis, J.R.J. Paré, J.M.R. Bélanger, Elsevier, 1997
- Food Analysis: Theory and Practice, Yeshajahu Pomeranz, Clifton E. Meloan, An Aspen Publishers, 1994, 2000

### BIBLIOGRAPHY FOR PRACTICES:

- Laboratorio de análisis instrumental Adela Mauri Aucejo, María José Llobat Estellés, Rosa Herráez



- Hernández, Universidad de Valencia, 2010
- Química de Alimentos: Manual de laboratorio. Nuria Bolaños V., Giselle Lutz C., Carlos H. Herrera R. Universidad de Costa Rica "Rodrigo Facio". 2003

### RECOMMENDED INTERNET LINKS

General Bibliography:

<http://www.ugr.es/~biblio/>

Chromatography :

<http://www.sci.sdsu.edu/TFrey/Bio750/Chromatography.html>

<http://ull.chemistry.uakron.edu/chemsep/>

<http://www.files.chem.vt.edu/chem-ed/sep/gc/gc.html>

<http://teaching.shu.ac.uk/hwb/chemistry/tutorials/chrom/gaschr.htm>

<http://www.forumsci.co.il/HPLC/topics.html>

<http://caspar.bgsu.edu/~courses/HPLC/HPLCManual.html>

<http://www.instrumentalchemistry.com/index.htm>

<http://www.science.oas.org/RLQ/tutoriales/cromatografia/croma.htm>.

General Spectroscopy:

<http--fisica.usach.cl--jammann-LabOpticaGuias-G3-PrismaAWEb.pdf>

[http://condor.cida.ve/~briceno/cursos/astrof\\_observ/clase3/](http://condor.cida.ve/~briceno/cursos/astrof_observ/clase3/)

[http://jchemed.chem.wisc.edu/JCESoft/Issues/Series\\_B/9B1/prog3-9B1.html](http://jchemed.chem.wisc.edu/JCESoft/Issues/Series_B/9B1/prog3-9B1.html)

[http://nautilus.fis.uc.pt/wwwfi/hipertextos/espectro/hiper\\_espectro.html](http://nautilus.fis.uc.pt/wwwfi/hipertextos/espectro/hiper_espectro.html)

<http://www.chem.vt.edu/chem-ed/spec/beerslaw.html>

<http://www.chm.davidson.edu/ChemistryApplets/spectrophotometry/BeersLaw.html>

<http://www.inform.umd.edu/EdRes/Topic/Chemistry/ChemConference/Software/Spreadsheets/WWW/BeersLaw.html>

<http://www.sc.ehu.es/sbweb/fisica/cuantica/fotoelectrico/fotoelectrico.htm>

<http://www.shu.ac.uk/schools/sci/chem/tutorials/molspec/beers1.htm>

[Prism Applet- Refraction and Dispersion](#)

[WebSpectra - Problems in NMR and IR Spectroscopy](#)

Flourescence Spectroscopy:

[http://www.infochembio.ethz.ch/links/en/spectrosc\\_luminescence.html](http://www.infochembio.ethz.ch/links/en/spectrosc_luminescence.html)

<http://www.inform.umd.edu/EdRes/Topic/Chemistry/ChemConference/Software/Spreadsheets/WWW/Flourescence.html>

<http://teaching.shu.ac.uk/hwb/chemistry/tutorials/molspec/lumin3.htm>

