

COURSE SYLLABUS

1. Identification

Code and title: QUP 104 – Special Topics in Sample Preparation Methods in Analytical Chemistry
Professors: Tânia Mara Pizzolato, Diogo Pompéu de Moraes and Juliana Severo Fagundes Pereira

Level: Master and Doctorate

Credit hours: 2

Revised: August_2019

2. Summary

Approach to the main methods currently used in sample preparation for analytical measurements of organic compounds and chemical elements.

3. Objective

Provide advanced theoretical training in sample preparation methods, enabling the student to develop specific analytical techniques for the determination of organic compounds and chemical elements.

4. Contents

4.1 Sample preparation methods for organic analysis. Sampling and sample preparation. Supercritical fluid extraction. Solid phase matrix dispersion. Pressurized solvent extraction. Ultrasound extraction. Purge-and-trap. Soxhlet method extraction. Headspace. Derivatization. Liquid-liquid extraction. Solid phase extraction. Evaporation. Centrifugation. Fractionation.

4.2 Sample preparation methods for inorganic analysis. Analytical sequence. Errors in sample preparation. Classification of sample preparation methods. Properties of acids. Combustion methods. Wet decomposition methods. Fusion method. Principle of microwave-assisted decomposition (wet, combustion, use of dilute acids, combination of microwave and ultraviolet radiation) in closed and pressurized systems. Sample preparation for speciation analysis.

5. Assessment

Lectures and seminars. The student, who obtains a final grade of A, B or C, awarded as per the list below, will be considered approved:

A: grade equal to or above 9.0

B: grade equal to or above 7.5 and below 9.0

C: grade equal to or above 5.0 and below 7.5

D: grade below 5

FF: lack of frequency

6. Methodology

Lectures, exercises lists, seminars and examinations.

7. Bibliography

- Peters, D. G.; Hayes, J. M. E Hieftje, G. M. Chemical Separations and Measurements, W.B. Saunders Company, 1974.
- Anderson, R. Sample Pretreatment and Separation (Analytical Chemistry by Open Learning), John Wiley & Sons, 1998.
- R. E. Majors; Trends in Sample Preparation, LC-GC Europe, Fev. 2003, pg 2-8.
- R. E. Majors; New Designs and Formats in Solid-Phase Extraction Sample Preparation, LC-GC Europe, Dec 2001, pg 2-6.
- D. Barceló (Ed), Techniques and Instrumentation in Analytical Chemistry – Volume 21, Sample Handling and Trace Analysis of Pollutants – Techniques, applications and quality assurance, Elsevier, Amsterdam, The Netherlands, First ed., 2000.
- J. Pawliszyn, Solid phase microextraction – Theory and practice., Wiley-VHC, Inc. New York, USA, 1997.
- J. S. Fritz, Analytical Solid-Phase Extraction, John Wiley & Sons, Inc. New York, NY, USA, 1999.
- R. N. Reeve, Environmental Analysis, John Wiley & Sons, Chichester, England, UK, 1994.
- R. P. Schwarzenbach, P.M. Gshwend and D.M. Imboden, Environmental Organic Chemistry, John Wiley & Sons, Inc. New York, NY, USA, 1993.
- Krug, F. J., Rocha, F. R. P., Métodos de Preparo de Amostras Para Análise Elementar, Editora EditSBQ, São Paulo, 2016.
- Flores, E. M. M., Microwave-Assisted Sample Preparation for Trace Element Determination, Elsevier, Oxford, UK, 2014.
- Arruda, M. A. Z., Trends in Sample Preparation, Nova Publisher, New York, 2006.
- Kingston, H. M., Haswell, S. J., Microwave-enhanced Chemistry, American Chemical Society, Washington, 1998.
- Mester, Z., Sturgeon, R., Sample Preparation for Trace Element Analysis, Volume XLI, Comprehensive Analytical Chemistry, Elsevier, Amsterdam, 2003.