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Analytical chemistry and laboratory methods (146024)

Nositelji predmeta

prof. dr. sc. Marko Vinceković, **prof. dr. sc. Marija Romić**

Opis predmeta

The aim of the course "Analytical chemistry and laboratory methods" is to provide students with basic principles of analytical chemistry and physico-chemical processes controlling the fate of pollutants in the environment, and to develop a working knowledge of laboratory methods, chemical analyses of the environmental samples, the supervising or performing laboratory operations, and the interpretation of analytical results. This covers several subject domains: basic principles of analytical methods, analytical procedures to obtain data, methods of chemical separation, measurements', soil chemistry, plant nutrition and environmental pollutants and protection, stressing the importance of quality assurance and quality control. Topics include modern analytical methods used for soil quality testing, soil testing for plant available nutrients, determination of environmental pollutants and the specific methods for the water analysis. Laboratory component will provide hands-on experience of laboratory methods and operation of analytical instrumentation as well as interpretation of the analytical results of testing methods. After finishing the course students should be able to request appropriate chemical analysis, understand procedures and principles of chemical testing methods, perform laboratory operations and report procedures and results in concise written reports. Theoretical lectures are complemented with practical laboratory exercises in small groups working in laboratories of the Faculty, where the students will familiarize with both classical and instrumental methods usually employed in environmental analytical chemistry. Further considerations include the quality of the analytical results and the generation of precise and accurate analytical data.

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ECTS: **6.00**

Engleski jezik: **R1**

E-učenje: **R1**

Sati nastave: 60
Predavanja: 28
Laboratorijske vježbe: 24
Seminar: 8

Izvođač predavanja

- doc. dr. sc. Nenad Jalšenjak
- prof. dr. sc. Marija Romić
- prof. dr. sc. Marko Vinceković

Izvođač vježbi

- doc. dr. sc. Lana Filipović
- prof. dr. sc. Marija Romić
- izv. prof. dr. sc. Monika Zovko

Ocjenjivanje

Dovoljan (2):
Dobar (3):
Vrlo dobar (4):
Izvrstan (5):

Vrsta predmeta

- Graduate studies / Environment, agriculture and resource management (Obvezni predmet, 1. semestar, 1. godina)

Opće kompetencije

Module Analytical chemistry and laboratory methods enables the student with the fundamental knowledge of analytical chemistry and principles of selecting an appropriate analytical method for the determination of certain indicators. Outperforming knowledge of the principles of laboratory methods in the analysis of soil and water, and acquire skills for laboratory work in a group as well as the skills to write reports on chemical analysis and presentation of results.

Oblici nastave

- Lectures
- Laboratory practice/exercises
  conducted in groups (10 students):as part of the laboratory exercise analysis are carried out with different instrumental chemical methods UV/VIS, AAS, SFA, ICP);
- Seminars
  related to stoichiometry, redox processes and how write chemical reactions, evaluation of analytical data, statistics in method validation

Ishodi učenja i način provjere

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Ishod učenja	Način provjere
Development of an appreciation of the underlying chemistry of some important environmental issues;	Mid-term exams, final exams, and tests at the end of course units
Understand the basic methods and relevant parameters in analytical chemistry,	Mid-term exams, final exams, and tests at the end of course units
Be able to apply the methods of the instrumental chemical analysis based on chromatography, spectrometry and spectroscopy	Mid-term exams, final exams, and tests at the end of course units
Understand the role of calibration and quality control in making analytical measurements,	Mid-term exams, final exams, and tests at the end of course units
Be able to prepare reports on the basis of experimental results and draw correct conclusions,	Mid-term exams, final exams, and tests at the end of course units
Demonstrate awareness of the limitations of various methods and select proper technique;	Mid-term exams, final exams, and tests at the end of course units
Apply basic and intermediate statistical concepts that are essential for the interpretation of the data analysis relevant to laboratory experimentation,	Mid-term exams, final exams, and tests at the end of course units
Understand the importance of setting up a quality management system,	Mid-term exams, final exams, and tests at the end of course units
Identify the importance of using standards, reference materials and internal samples for the quality control,	Mid-term exams, final exams, and tests at the end of course units
Be able to define the major components of the quality management system in the analytical laboratory.	Mid-term exams, final exams, and tests at the end of course units

Polaganje ispita

Elementi praćenja	Maksimalno bodova ili udio u ocjeni	Bodovna skala ocjena	Ocjena	Broj sati izravne nastave	Ukupni broj sati rada prosječnog studenta	ECTS bodovi
Laboratory exercises	10%	0-60 61-70 71-80 81-90 90-100	Nedovoljan (1) Dovoljan (2) Dobar (3) Vrlo dobar (4) Izvrstan (5)	24	0.6	0.6
Seminars	10%	0-60 61-70 71-80 81-90 90-100	Nedovoljan (1) Dovoljan (2) Dobar (3) Vrlo dobar (4) Izvrstan (5)	8	0.6	0.6
Classroom attendance	5 %	0-60 61-70 71-80 81-90 90-100	Nedovoljan (1) Dovoljan (2) Dobar (3) Vrlo dobar (4) Izvrstan (5)		0.3	0.3
I Colloquium	37.5%	0-60 61-70 71-80 81-90 90-100	Nedovoljan (1) Dovoljan (2) Dobar (3) Vrlo dobar (4) Izvrstan (5)	14	2.25	2.25
II Colloquium	37.5%	0-60 61-70 71-80 81-90 90-100	Nedovoljan (1) Dovoljan (2) Dobar (3) Vrlo dobar (4) Izvrstan (5)	14	2.25	2.25
Final (written)	75 %	0-60	Nedovoljan (1)	28	4.5	4.5

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Elementi praćenja	Maksimalno bodova ili udio u ocjeni	Bodovna skala ocjena	Ocjena	Broj sati izravne nastave	Ukupni broj sati rada prosječnog studenta	ECTS bodovi
exam (if colloquiums are not passed)		61-70 71-80 81-90 90-100	Dovoljan (2) Dobar (3) Vrlo dobar (4) Izvrstan (5)			
Total	100 %			60	6	6

Elementi praćenja	Opis	Rok	Nadoknada
Attendance at seminars	At each seminar, students are signed. Periodically control is performed oral roll call.	Before accession on exams.	Students with more than three absences lose the ability to apply for the oral exams. Additional seminars - two pages of text for each lost block lectures. Contact the teacher after announcing record attendance
Activity in class (Seminars and lectures)	Students can participate in discussions at seminars, actively participate in the work of small groups / teams and be active in the exercises in the classroom and laboratory. Special activity in the discussions at the lecture also is scored		
Attendance at laboratory exercises	At each exercise, students are signed.	Before accession on exams.	Students with more than three absences lose the ability to apply for the exams. Additional seminars - two pages of text for each lost block exercises. Contact the teacher after announcing record attendance
Colloquiums OR Written exam	Students answer questions from the teaching materials available in Moodle system	Accession on exams.	Student knowledge and understanding of the subjects covered in lectures, exercises and seminars is examined.

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Tjedni plan nastave

1. Introduction. Chemical equilibrium. L Chemical equilibrium - Classical and advanced methods; analytical concepts and theories: role in science and everyday life; providing qualitative information (what is in a sample) and quantitative information (how much is in a sample) about the matter; importance of environmental issues. Chemical equilibrium in electrolyte solutions, base/acid equilibrium in heterogeneous systems, precipitation/dissolution, complexation, oxidation/reduction.
2. Classical analytical methods. Treatment of analytical data L Classical volumetric, gravimetric and titrimetric analysis. Basic physical units, stoichiometries relations in chemical equations, errors, evaluation of analytical data.
3. Chromatography. Methods and instrumentation in spectrometry and spectroscopy: 1. Instrumentation in optical spectroscopy L Chromatography: basic theory and applications using liquid chromatography and gas chromatography as well as hyphenated techniques such as GC-MS and HPLC-MS. Spectroscopic methods based on ultraviolet and visible radiation; review of characteristics of electromagnetic radiation; absorption of electromagnetic radiation; basic concepts in absorption spectroscopy; Beer's law and its appliance; AAS.
4. Methods and instrumentation in spectrometry and spectroscopy: 2. Interference and atomic emission spectrometry. L Laboratory environment and equipment performance Lab Origin of spectral interferences and their elimination; chemical interferences; flame-AES interference; plasma emission spectrometry - instrumentation and principles. Equipment performance checks; plan what needs to be done for the routine equipment, maintaining; assurance of the measurement traceability to national and international etalons. Laboratory environment and equipment performance: 1. Pipette calibration Lab Pipette calibration procedure; a series of methods for determination of the difference between volumetric measurement value and nominal or selected value of testing pipette; the objective of the procedure is to eliminate errors in measuring process.
5. Laboratory environment and equipment performance: 2. Spectrophotometry - Cuvette calibration Lab Analytical methods and validation Lab The aim of the exercise is to introduce students with the operation of UV/Vis spectrophotometer; reference solution (K2CrO4) with known absorbance (A) values in giving conditions is used. By measuring absorbance (A) of such solution aberration of spectrophotometer can be determined; path length (b) is also tested. Principles of the analytical methods and their confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled.
6. Determination of the cadmium concentration using the standard addition method Lab Soil properties and methods for laboratory analysis L The aim of this exercise is to introduce students with standard addition method which is used for eliminating matrix interference. Matrix interference refers to the components of a sample other than the analyte. Surface chemical properties of soil minerals; Origin of the surface charge; Ion exchange; Mineral surface adsorption sites; Cation exchange capacity (CEC); Analytical methods and principles for cation exchange capacity determination.
7. Soil properties and methods for laboratory analysis: 1. Determination of cation exchange capacity (CEC) and percent base saturation using barium chloride solution Lab In this exercise Cation exchange capacity (CEC) is determined under constant pH conditions of soil sample and low ionic strength of a solution (0,01 mol/l). The exchangeable potassium, calcium, magnesium, aluminium and manganese content is also determined.
8. Soil properties and methods for laboratory analysis: 2. Phosphorus fractionation Lab The chemistry of phosphorus (P) in soils is complex: inorganic phosphorus can react with Ca, Fe and Al to form discrete phosphates, and organic phosphorus can be in different forms as well, with varying resistance to microbial degradation. To investigate the forms of inorganic

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P and transformations of organic P, phosphorus fractionation is applied.
9. Soil properties and methods for laboratory analysis: 3. Ionic composition of the soil solution using segmented flow analysis (SFA) Lab In this exercise chloride (Cl-) ions in water samples are being determined on Skalar San++ Analyzer. The aim of the exercise is to introduce students with method principles, as well as with instrument calibration. An external standard method is being used for an instrument calibration. Calibration straight line is being calculated according to ISO 8466-1.
10. The quality assurance in analytical laboratory L Analytical process; selecting the method; sampling defined; performance criteria for the method used; interferences; calibration and final measurement; the differences between accuracy and precision; reasons for incorrect analytical results, types of errors in experimental dana.
11. Managing quality L Quality control assurance, Quality control management, Quality control, GLP requirements, Proficiency testing schemes; Reference materials.
12. Data treatment: 1. Control Charts Lab Construction and interpretation of control charts for continuous measurement of variable data (total Cr, Zn, Pb i Cu in soil) in the same sample. Application of statistical methods to extract useful information from chemical data, with particular emphasis on the use of control charts.
13. Data treatment: 2. Measurement uncertainity Lab Laboratory session- evaluation of the influence factors which can be applied to the analytical results; estimation of dispersion of the values that could be attributed to the analyte; the process of measurement uncertainty estimation: example determination of pH level in soil;apply statistical procedure dealt with in the quality control through the software application-laboratory session.
14. Statistics used to describe data sets L Handling the data generated by analytical methods; the key statistical parameters used to summerize and describe data sets; correct interpretation of the statistical data.
15. Seminar S Statistics in method validation Written exam WE

Obvezna literatura

1. 1. Evangelou V. P. (1998): Environmental Soil and Water Chemistry: Principles and Applications, Iowa State University (selected chapters).
2. 2. Prichard E., Barwick V. (2008): Quality Assurance in Analytical Chemistry. John Wiley & Sons Inc.
3. 3. Rayment G. E., Lyons D. J. (2011): Soil Chemical Methods - Australasia. CSIRO Publishing, Australia.
4. 4. Skoog D.A. (2011): Principles of Instrumental Analysis, 5th Edition (selected chapters).
5. 5. Silberberg M. S. (2000): Chemistry: The Molecular Nature of Matter and Change, second edition (selected chapters).
6. 6. van Dijk D., Houba V. J. G. (2000): Interlaboratory Analytical Studies and their Evaluation. Wageningen University, The Netherlands.

Sličan predmet na srodnim sveučilištima

- Practical Course in Instrumental Analytical and Physical Chemistry, BOKU
- Instrumental Analytical Chemistry for Master Students, BOKU
- Soil-chemical Analytics (3101-500), University of Hohenheim