

## UČNI NAČRT PREDMETA / COURSE SYLLABUS

<b>Predmet:</b>	PRAKTIKUM IZ INSTRUMENTALNIH METOD IN INSTRUMENTALNE ANALIZE
<b>Course Title:</b>	PRACTICAL COURSE IN INSTRUMENTAL METHODS AND INSTRUMENTAL ANALYSIS

Študijski program in stopnja Study Programme and Level	Študijska smer Study Field	Letnik Academic Year	Semester Semester
UŠP Kemija, 1. stopnja	/	3.	5.
USP Chemistry, 1 <sup>st</sup> Cycle	/	3 <sup>rd</sup>	5 <sup>th</sup>

**Vrsta predmeta / Course Type:**

obvezni / Mandatory

**Univerzitetna koda predmeta / University Course Code:**

KE128

Predavanja Lectures	Seminar Seminar	Vaje Tutorial	Klinične vaje Work	Druge oblike študija	Samost. delo Individual Work	ECTS
/	/	75 LV	/	/	75	5

**Nosilec predmeta / Lecturer:**

prof. dr. Irena Kralj Cigić / Dr. Irena Kralj Cigić, Full Professor  
prof. dr. Matija Tomšič / Dr. Matija Tomšič, Full Professor

**Jeziki / Languages:**

**Predavanja / Lectures:** /

**Vaje / Tutorial:** slovenski / Slovenian

**Pogoji za vključitev v delo oz. za opravljanje študijskih obveznosti:**

Študent oz. kandidat mora imeti predmet opredeljen kot študijsko obveznost.

**Prerequisites:**

The course has to be assigned to the student.

**Vsebina:**

Prvi del praktikuma je namenjen spoznavanju merskih metod in principov merjenja osnovnih fizikalno kemijskih količin:

- merjenje tlaka in pretoka;
- merjenje in regulacija temperature (termostat);
- merjenje električnih količin: I, U, R, L, C;
- dinamične karakteristike senzorjev;
- določanje karakteristik elektronskih polprevodniških elementov in elektronskih Podsestavov (dioda, tranzistor, usmernik, stabilizator, operacijski ojačevalnik, integrator, A/D in D/A pretvornik);

**Content (Syllabus outline):**

In the first part of the practicum the student gets acquainted with various experimental methods and measurement principles of basic physic-chemical quantities:

- pressure and flow measurements;
- temperature measurements and temperature control (thermostat);
- measurements of electrical quantities: current, voltage, resistance, inductivity, and capacitance;
- dynamic characteristics of sensors;
- characteristics of electronic semiconductor elements and electronic instrumental

- strmina steklene elektrode, avtomatska regulacija pH;
- določanje karakteristik optičnih elementov;
- preizkus pravilnosti delovanja spektrofotometra.

Drugi del je namenjen uporabi osnovnih instrumentalnih metod v kemijski analizi:

- elektroanalizne metode (amperometrija in voltometrija): separacija in določanje glavnih sestavin, analiza mikroestavin z voltometrijo in stripping voltometrijo);
- spektroskopske metode: karakterizacija materialov z optično emisijsko spektrometrijo, analiza s plamensko atomsko absorpcijsko spektrometrijo in elektrotermično atomsko absorpcijsko spektrometrijo; interference;
- separacijske metode (plinska kromatografija z različno detekcijo – FID, ECD, kombinacija plinska kromatografija-masna spektrometrija);
- radiokemijske metode (beta, gama štetje, števna statistika);
- analiza realnih vzorcev: zajem vzorca, razklopi vzorcev, statistično vzorčevanje in statistično vrednotenje rezultatov.

components (diode, transistor, rectifier, stabilizer, operational amplifier, integrator, A/D and D/A converter);

- the slope of the glass electrode and automatic regulation of pH;

- characteristics of optical elements;

- testing the performance of a spectrophotometer.

In the second part of the practicum the student acquires practical knowledge about instrumental methods in chemical analysis:

- electroanalytical methods (amperometry and voltametry): separation and determination of major constituents, analysis of microcomponents with voltametry and stripping voltammetry.

- spectroscopic methods: characterisation of materials with optical emission spectroscopy, flame atomic absorption spectrometry and graphite furnace atomic absorption spectrometry, interferences.

- separation methods ( gas chromatography with different detectors – FID, ECD, hyphenated techniques – gas chromatography-mass spectroscopy.

- radiochemical methods (beta, gamma counting, counting statistics).

analysis of real samples: sampling, sample digestion, sampling statistics and statistical evaluation of results.

#### Temeljna literatura in viri / Readings:

- Skupina avtorjev/Group of authors: Praktikum iz instrumentalnih metod, interno gradivo.
- Drago Kočar, Rubert Susič: Vaje iz instrumentalne analize, interno gradivo.
- D.A. Skoog, F.J. Holler, T.A. Nieman: Principles of Instrumental Analysis 5<sup>th</sup> Ed, Saunders College Publishing

#### Cilji in kompetence:

**Cilji:** Praktikum je namenjen ilustraciji in verifikaciji tematike predstavljene pri predmetih Instrumentalne metode in

#### Objectives and Competences:

The main goal of this practicum is to offer student a suitable illustration and practical verification of the topics presented in the

Instrumentalna analiza. Prvi del je namenjen spoznavanju zakonitosti merjenja osnovnih fizikalnih količin in spoznavanju zgradbe in delovanja najpomembnejših funkcijskih sklopov inštrumentov, ki služijo zajemanju, ojačenju, preoblikovanju, izboljšanju merilnega signala in optimizaciji razmerja signal/šum (S/N).

Drugi del praktikuma je namenjen usposabljanju študentov za delo z zahtevno instrumentacijo v analiznem laboratoriju. Pridobili naj bi zmožnost samostojne izbire in uporabe primerne instrumentalne analizne metode glede na število in vrsto vzorcev, predvideno koncentracijsko območje ter zahtevnost za osebje in instrumentacijo. Navadili naj bi se glavnih vidikov dobre instrumentalne laboratorijske prakse. Študenti si pri predmetu pridobijo naslednje specifične kompetence:

- poznavanje osnovnih sestavnih komponent posameznih instrumentov;
- zmožnost sestavljanja in povezovanja nekaterih aparatov, ki so namenjene profesionalni uporabi v kemijskih laboratorijih;
- pridobivanje praktičnih izkušenj in poglobljanje razumevanja delovanja in karakteristik (tudi omejitev) posameznih aparatov;
- razumevanje zmožljivosti in pravilne rabe instrumentov;
- usposobljenost za samostojno delo z instrumenti ob uporabi navodil proizvajalca;
- zmožnost izbire najprimernejše analizne metode za reševanje specifičnih analiznih problemov;
- zmožnost statistično podprtega načrtovanja in realizacije instrumentalnih analiznih postopkov;
- usposobljenost za statistično analizo podatkov, njihovo pravilno interpretacijo in izdelavo poročil o meritvah in analizi.

course of *Instrumental Methods* and course of *Instrumental Analysis*. The first part of the practicum is devoted to experimental measurements of basic physicochemical quantities and getting acquainted with the composition and principles of important functional components of the instruments for data acquisition, amplification, modulation, filtering and optimisation of signal-to-noise ratio.

The second part (*Instrumental Analysis*) is devoted to qualify student to be able to work with complex instrumentation in analytical laboratory. Students will gain the appropriate knowledge to be able to select the appropriate instrumental method according to the sample size, number of samples, type of analyte and concentration level.

During this practicum the students gain the following competences:

- Knowledge on the basic instrumental components of individual instruments.
- The skills to combine basic chemical instruments into a working instrumental setup.
- Practical skills and enhanced understanding of the basic instrumental functions, characteristics and limitations.
- Understanding the capabilities of the instruments and their proper application.
- Competency to start working with the instruments based on the instructions from the User Manual.
- Competency to select the optimal analytical method for the specific analytical problem.
- Competency to plan the instrumental analysis procedures and the corresponding statistical evaluation.

Statistical evaluation of experimental data, their proper interpretation and preparation of experimental reports.

**Predvideni študijski rezultati:**

**Intended Learning Outcomes:**

<p><u>Znanje in razumevanje</u>          Študent pozna merilne metode, funkcionalno zgradbo in delovanje instrumentov in aparatur in glavne instrumentalne analzne metode. Razume fizikokemijske osnove delovanja in nastavitve analznih instrumentov. Ve, kaj lahko vpliva na analzni postopek, pozna vire motenj in napak. Zna izračunati in preveriti rezultat instrumentalne analize, ga statistično ovrednotiti in napisati ustrezno poročilo.</p>	<p><u>Knowledge and Comprehension</u>          Student is familiar with the measurement techniques, functional composition and operation of the instruments and basic analytical methods. Student understands the physicochemical basics of operation, alignment of the analytical instruments, effects influencing the analytical procedures, and sources of measurement uncertainty and errors. Student is able to calculate and validate the results of instrumental analysis, to treat them statistically, and to prepare the corresponding experimental report.</p>
<p><u>Uporaba</u>          Študent pozna namen in osnove delovanja analznih instrumentov. S pomočjo navodil proizvajalca je sposoben reproducirati izdelano analizo metodo. Zna pripraviti vzorec, potrebne reagente in poskrbi za kalibracijo in vzdrževanje instrumentov. Pridobljeno znanje bo koristno uporabljal pri svojem strokovnem in raziskovalnem delu ali v praksi.</p>	<p><u>Application</u>          Student is familiar with the basic application of analytical instruments and is able to reproduce an individual analytical method on the basis of instructions for users. Student is able to prepare the samples and reagents, to make the necessary instrumental calibrations and alignments and to apply this knowledge in practical research.</p>
<p><u>Refleksija</u>          Študent kritično reflektira in vrednoti vidike dela z analizo instrumentacijo glede števila vzorcev, cene posamezne analize, zahtevnosti glede kemikalij in osebja. Zaveda se pomena instrumentov, njihovega delovanja in iz od tod izhajajočih prednosti in omejitev.</p>	<p><u>Analysis</u>          Student critically reflects and validates the analytical procedures and instrumentation in terms of the necessary number of samples, costs and difficulty of the analysis, special demands for the personnel and handling of the special chemicals. Student is aware of the importance of the precision instrumentation, their limitations and the corresponding advantages or disadvantages in application.</p>
<p><u>Prenosljive spretnosti</u>          Študent se nauči pristopiti k novi instrumentaciji. Razumevanje konkretnih implementacij fizikokemijskih osnov in drugih splošnih principov v realizaciji konkretnega instrumenta mu omogoča realistično oceno zahtevnost načrtovanja novih instrumentalnih rešitev.</p>	<p><u>Skill-transference Ability</u>          Student gains the knowledge on how to approach to the new instrumentation and/or analytical method. Understanding basic physicochemical concepts behind an individual instrument the student is able to realistically assess the possibility to apply such an instrument in new instrumental solutions.</p>

**Metode poučevanja in učenja:**

Praktikum in seminar.

**Learning and Teaching Methods:**

Laboratory practicum and seminar.

Delež (v %) /

**Načini ocenjevanja:**

Weight (in %)

**Assessment:**

Pisni izpit in uspešno opravljen praktični del iz prvega dela praktikuma (FK).	<b>50 %</b>	Written exam and successfully completed practical part in the first part of the practicum (FK).
Pisni izpit in uspešno opravljen praktični del iz drugega dela praktikuma (AK).	<b>50 %</b>	Written exam and successfully completed practical part in the first part of the practicum (AK).

**Reference nosilca / Lecturer's references:**

- Tomšič, M.; Prossnigg, F.; Glatter, O. A thermoreversible double gel: characterization of a methylcellulose and kappa-carrageenan mixed system in water by SAXS, DSC and rheology. *J. Colloid Interf. Sci.* 2008, 322, 41-50.
- Tomšič, M.; Jamnik, A.; Fritz-Popovski, G.; Glatter, O.; Vlček, L. Structural properties of pure simple alcohols from ethanol, propanol, butanol, pentanol, to hexanol: Comparing Monte Carlo simulations with experimental SAXS data. *J. Phys. Chem. B* 2007, 111, 1738-1751.
- Tomšič, M.; Bešter-Rogač, M.; Jamnik, A.; Kunz, W.; Touraud, D.; Bergmann, A.; Glatter, O. Nonionic surfactant Brij 35 in water and in various simple alcohols: Structural investigations by small-angle X-ray scattering and dynamic light scattering. *J. Phys. Chem. B* 2004, 108, 7021-7032.
- MOŽIR, Alenka, GONZALEZ, Lee, KRALJ CIGIČ, Irena, WESS, Tim J., RABIN, Ira, HAHN, Oliver, STRLIČ, Matija. A study of degradation of historic parchment using small-angle x-ray scattering, synchrotron-IR and multivariate data analysis. *Analytical and bioanalytical chemistry*, ISSN 1618-2642, 2012, vol. 402, no. 4, str. 1559-1566, doi: 10.1007/s00216-011-5392-6. [COBISS.SI-ID 35750405]
- KRALJ CIGIČ, Irena, VRŠČAJ VODOŠEK, Tatjana, KOŠMERL, Tatjana, STRLIČ, Matija. Amino acid quantification in the presence of sugars using HPLC and pre-column derivatization with 3-MPA/OPA and FMOC-Cl. *Acta chimica slovenica*, ISSN 1318-0207. [Tiskana izd.], 2008, letn. 55, št. 3, str. 660-664, graf. prikazi. <http://acta.chem-soc.si/55/55-3-660.pdf>. [COBISS.SI-ID 29802245]
- KRALJ CIGIČ, Irena, STRLIČ, Matija, SCHREIBER, André, KOCJANČIČ, Mitja, PIHLAR, Boris. Ochratoxin A in wine : its determination and photostability. *Analytical letters*, ISSN 0003-2719. [Print ed.], 2006, vol. 39, no. 7, str. 1475-1488, Graf. prikazi. [COBISS.SI-ID 27677957]