**COURSE SYLLABUS**

**1. Data about the program**

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| 1.1 Higher education institution | Babeș-Bolyai University |
| 1.2 Faculty | Faculty of Chemistry and Chemical Engineering |
| 1.3 Doctoral school | Chemistry |
| 1.4 Field of study | Chemistry |
| 1.5 Study cycle | Doctorate |
| 1.6 Study program / Qualification | Doctoral training / PhD in Chemistry |

**2. Course data**

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| 2.1 Name of discipline | | | NMR and X-ray diffraction in organometallic chemistry | | | | |
| 2.2 Teacher responsible for lectures | | | Acad. Prof. dr. Cristian SILVESTRU | | | | |
| 2.3 Teacher responsible for seminars | | | Acad. Prof. dr. Cristian SILVESTRU | | | | |
| 2.4 Year of study | I | 2.5 Semester | II | 2.6. Type of evaluation | Ea | 2.7 Course framework | Opb |

a the PhD student can choose to participate in activities without an exam or with an exam. b Op = optional discipline.

**3. Estimated total time of teaching activities** (hours per semester)

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| --- | --- | --- | --- | --- | --- | --- | --- |
| 3.1 Hours per week | 3 | | Out of which: 3.2  Lectures | | 1 | 3.3 Seminars /  Laboratory classes | 2 |
| 3.4 Total hours in the curriculum | 36 | | Out of which: 3.5  Lectures | | 12 | 3.6 Seminars /  Laboratory classes | 24 |
| Allocation of study time: | | | | | | | hrs |
| Study supported by textbooks, other course materials, recommended bibliography and personal  student notes | | | | | | | 120 |
| Additional learning activities in the library, on specialized online platforms and in the field | | | | | | | 120 |
| Preparation of seminars / laboratory classes, topics, papers, portfolios and essays | | | | | | | 44 |
| Tutoring | | | | | | | 30 |
| Examinations | | | | | | | - |
| Other activities: - | | | | | | | - |
| 3.7 Individual study (total hours) | | 314 | |  | | | |
| 3.8 Total hours per semester | | 350 | |
| 3.9 Number of credits | | 14 | |

**4. Preconditions** (where applicable)

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| 4.1 Curriculum | • It's not necessary |
| 4.2 Competences | • It's not necessary |

**5. Conditions** (where applicable)

5.1 Conducting lectures • The courses and seminars take place in conditions of access to internet and

databases

• Students will have access to databases (subsribed by faculty / university)

• Interactive participation will be stimulated

• Students will attend the class with their mobile phones closed

5.2 Conducting seminars / laboratory classes

• Students will attend the class with their mobile phones closed

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**6. Specific competences acquired**

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|  | • Defining and mastering the notions, concepts, theories and basic models used in structural analysis by  Nuclear Magnetic Resonance (NMR), respectively specific Monocrystalline X-ray Diffraction and their  appropriate use in professional communication;  • The use of in-depth knowledge in the field of chemistry to explain and interpret the NMR spectra,  respectively the structural analysis of inorganic (including coordinative) and organometallic compounds;  • Spectroscopic data processing and interpretation;  • Development of the ability to determine the molecular structure in solution or solid state of inorganic,  organic and organometallic compounds using data obtained from NMR spectra;  • Use of solution NMR spectroscopy in the study of dynamic (including fluxional) systems;  • Ability to use structural data of inorganic (including coordinative), organic and organometallic  compounds, obtained by single crystal X-ray diffraction;  • Use of single crystal X-ray diffraction data in identifying aspects in the field of supramolecular chemistry;  • Use of specific software for processing NMR spectra and single crystal X-ray diffraction data;  • Ability to understand and interpret complementary data for the characterization of inorganic (including  coordinative), organic and organometallic compounds, to express and argue the interpretation of data based on the correlation of results and comparison with data from the literature. |
|  | • Execution of the requested tasks according to the specified requirements and within the imposed  deadlines, in compliance with the norms of professional ethics and moral conduct, following a pre-  established work plan;  • Solving the requested tasks in accordance with the general objectives established by integration within a  working group;  • Search of literature data in Romanian and in a language of international circulation, using modern  methods of information and communication (specific software, database search);  • Concern for improving the results of professional activity by getting involved in the activities carried  out;  • Ability to prepare written reports and to present these reports in group seminars. |

**7. Course objectives** (based on the acquired competencies grid)

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| --- | --- |
| 7.1 The general objective of the course | • Acquisition of knowledge regarding the structural characterization of inorganic  (including coordinative), organic and organometallic compounds |
| 7.2 Specific objectives | • Acquisition of theoretical knowledge on NMR spectroscopy and single crystal X-  ray diffraction for the investigation and identification of molecular structure in  solution and in solid state;  • Use of NMR techniques for structural characterization in solution of organic,  organometallic compounds and coordinating compounds, including the study of  dynamic processes in solution;  • Use of single crystal X-ray diffraction, including in investigating aspects of  supramolecular chemistry. |

**8. Content**

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| 8.1 Lectures | Teaching methods | Comments |
| 8.1.1. Nuclear Magnetic Resonance - the principle of the method; active  NMR isotopes, internal and external standards, spectral parameters | Lecture; explication;  conversation | 1 hour |
| 8.1.2. Multinuclear NMR spectroscopy – 1H, 13C | Lecture; explication;  conversation | 1 hour |
| 8.1.3. Multinuclear NMR spectroscopy – – 19F, 31P and other active NMR  isotopes of non-metals | Lecture; explication;  conversation | 1 hour |
| 8.1.4. Multinuclear NMR spectroscopy – active NMR isotopes of some metals; spin-spin coupling with nuclei with an abundance of 100% or less than <100% - satellites | Lecture; explication; conversation | 1 hour |
| 8.1.5. 2D NMR spectroscopy | Lecture; explication;  conversation | 1 hour |

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| 8.1.6. NMR spectroscopy - correlations between coordination geometry, number of signals and chemical displacement in the spectra of inorganic, coordination and organometallic compounds | Lecture; explication; conversation; discussion; exercises | 1 hour |
| 8.1.7. Fluxional molecules | Lecture; explication;  conversation | 1 hour |
| 8.1.8. NMR spectroscopy – dynamic NMR, kinetic and thermodynamic parameters | Lecture; explication. conversation; discussion; exercises | 1 hour |
| 8.1.9. Single crystal X-ray diffraction (principle of the method and  instrumentation) | Lecture; explication.  conversation; discussion | 1 hour |
| 8.1.10. Interpretation of data obtained by single crystal X-ray diffraction. Description and interpretation of cif files, crystallographic tables, atomic parameters. Covalent atomic rays, Van der Waals rays | Lecture; explication. conversation; discussion | 1 hour |
| 8.1.11. Intra- and intermolecular interactions. Non-covalent interactions - hydrogen bonds, hydrogen-pi bonds, metal-pi, pi-pi stacking, metallophilic interactions, etc. | Lecture; explication. conversation; discussion; exercises | 1 hour |
| 8.1.12. Supramolecular architectures obtained by self-assembly | Lecture; explication.  conversation; discussion | 1 hour |
| **Bibliography**  1. D. W. Rankin, N. W. Mitzel, C. A. Morrison, *Structural Methods in Molecular Inorganic Chemistry*, John Wiley &  Sons, Chichester, 2013.  2. R. V. Parish, *NMR, NQR, EPR and Moessbauer Spectroscopy in Inorganic Chemistry*, Ellis Horwood, New York,  1990.  3. H. Friebolin, *Basic One- and Two-Dimensional NMR Spectroscopy*, Wiley-VCH, Weinheim, 1998.  4. W. Massa, *Crystal Structure Determination*, Springer, Berlin, 2004.  **Optional bibliography**  1. I. Haiduc, J. J. Zuckerman, *Basic Organometallic Chemistry*, Walter de Gruyter, Berlin, 1985.  2. Ch. Elschenbroich, A. Salzer, *Organometallics - A Concise Introduction Chemistry*, VCH Verlag, Weinheim, 1992.  3. R. H. Crabtree, *The Organometallic Chemistry of The Transition Metals*, 3rd Ed., John Willey & Sons, New York,  2001.  4. Kin-ya Akiba (Ed.), *Chemistry of Hypervalent Compounds*, Wiley-VCH, New York, 1999.  5. E. A. V. Ebsworth, D. W. H. Rankin, S. Cradock, *Structural Methods in Inorganic Chemistry*, Blackwell, Oxford, 1987.  6. A. F. Wells, *Structural Inorganic Chemistry, 4th Ed.*, Oxford Univ. Press, London, 1975. | | |
| 8.2 Seminars / laboratory classes | Teaching methods | Comments |
| 8.2.1-2. Applications of NMR spectroscopy in the structural analysis of organometallic and coordinative compounds. Use of specific software (MestRENova). Interpretation of 1H and 13C spectra - structural considerations based on 1H and 13C chemical shifts, value of integrals of 1H resonance signals, spin-spin couplings from 1H NMR spectra of organometallic compounds | Explication; Conversation; Description; Problems | 4 hours |
| 8.2.3-4. Applications of NMR spectroscopy in the structural analysis of organometallic and coordination compounds. Interpretation of two- dimensional spectra. Assignment of 1H and 13C resonance signals. | Explication; Conversation; Description; Problems | 4 hours |
| 8.2.5-6. Interpretation of NMR spectra (19F, 31P, etc.) of some inorganic and organometallic compounds. Assignment of the structure in solution based on multinuclear NMR spectra. Simulation of NMR spectra. | Explication; Conversation; Description; Problems | 4 hours |
| 8.2.7-8. NMR applications in the study of dynamic processes. Variable  temperature spectra. Kinetic and thermodynamic parameters. | Explication; Conversation;  Description; Problems | 4 hours |
| 8.2.9-10. Analysis and interpretation of single crystal X-ray diffraction data. Use of specific software (Diamond, Mercury, Platon, etc.) and specific databases (CSD, ICSD, etc.). | Explication; Conversation; Description; Problems | 4 hours |
| 8.2.11-12. Use of specific programs to prepare data for publication. | Explication; Conversation;  Description; Problems | 4 hours |
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**Bibliography**:

1. Problems provided by the teacher responsible for lectures and seminars.

2. The collections from the last 5 years of journals **Angew. Chem. Int. Ed.; Chem. Eur. J.; Eur. J. Inorg. Chem; Chem. Commun.; Chem. Sci.; Inorg. Chem.; Dalton Trans. And Organometallics**

**9. Aligning the contents of the discipline with the expectations of the epistemic community**

**representatives, professional associations and standard employers operating in the program field**

• The content of this discipline is very useful in evaluating / ensuring the quality of synthetic chemical

products and materials, meeting the needs of employers located both in the field of production and in the

sale of synthetic chemical products.

• Knowledge and use of these modern and complex methods for characterizing inorganic, organic,

organometallic compounds or complexes is consistent with the partial competencies required for possible occupations provided in Grid 1 - RNCIS.

**10. Examination**

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| Activity type | 10.1 Evaluation criteria | 10.2 Evaluation methods | 10.3 Weight in the  final grade |
| 10.4 Lectures | Correctness of answers - proper acquisition and understanding of the issues addressed | Oral examination | 60% |
| The way of thinking, the correctness, and the argumentation of the proposed solutions |
| 10.5 Seminars / laboratory classes | Activity during seminars | Presentation of the analysis of spectra and structures | 40% |
| 10.6 Minimum performance standard | | | |
| • exam grade 5 (five) | | | |

Date of issue

Signature of the teacher

responsible for lectures

Signature of the teacher

responsible for seminars

October 1st, 2021

Date of approval by the doctoral school council

Signature of the doctoral school director

Prof. Dr. Ion Grosu

Corresponding Member of Romanian Academy

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