UNIVERSITÀ
DEGLI STUDI DI MILANO-BICOCCA

COURSE SYLLABUS

Laboratory of Experimental Chemistry

2122-3-E2701Q064

Aims

The aim of the course is the introduction to the most important spectroscopic techniques and commonly-used methodologies for the analysis of different classes of materials.

Knowledge and understanding
At the end of the course the student knows the main analytical techniques, such as solution and solid state nuclear magnetic spectroscopy, infrared and UV-vis spectroscopies, calorimetric and thermogravimetic methods, and X-ray diffraction from single crystal and polycrystalline powders.

Applying knowledge and understanding

At the end of At the end of the course the student is able to analyze and explain the experimental data obtained during laboratory experiments:

1) solution and solid state NMR spectra, 2) IR and UV-vis spectra, 3) calorimetric analysis, 4) diffraction patterns of polycrystalline powders.

Making judgments

At the end of the course the student is able to identify the most suitable chemical characterization methods for the description of materials.

Communication skills
To be able to present the topics of the course in a suitable language and describe the procedure and the results of the experiments carried out in the laboratory in a clear and concise scientific report.

Learning skills
To be able to apply the acquired knowledge to contexts different from those presented during the course, and to understand the topics covered in the scientific literature concerning the analytical techniques described during the course.

Contents

The course consists of theoretical lessons and laboratory experiences. The course encompasses general principles of the main analytical techniques, especially Nuclear Magnetic Resonance spectroscopy in solution and in the solid state, Infrared spectroscopy, calorimetric methods, and single and powder X-ray diffraction techniques.

Moreover the corse includes collection and analysis of:

1) exemplifications of the Nuclear Magnetic Resonance spectroscopy in solution and in the solid state,

2) exemplifications of Infrared and UV-vis Spectroscopies,

3) calorimetric and thermogravimetric traces,

4) powder X-ray diffraction patterns

and quantitative analysis of the experimental data.

Detailed program

The course includes a general overview of the most appropriate methodologies for the analysis of the different classes of materials and hints on the methods of collecting experimental data, followed by the development of laboratory experiences through the use of some analytical and structural recognition techniques. The laboratory activity will be preceded by a series of lessons to recall the general principles on which each technique is based, the description of instrumental methodologies, the collection and interpretation of specific data, and the performance of qualitative and quantitative analyzes. Students will develop the processing capacity also through the use of appropriate software. Some categories of materials will be considered and the most appropriate techniques for quantitative characterization and analysis will be explored.

In particular, the following exercises are planned:

- X-ray diffraction on polycrystalline systems. Collection and interpretation of the diffractograms (for example quartz), identification of the unit cell, and refinement of the lattice parameters with the least squares method. Quantification of the crystalline phases in multi-component systems or in systems containing polymorphs.

- NMR in solution. Preparation of the sample, collection of the spectra, transformation of the signal from the time

domain to the frequency domain and interpretation of the spectra with particular regard to the 1H nucleus. During the laboratory experience students will learn the methodology to acquire the experiments that allow to identify the molecular structure.

- Solid state NMR. Preparation of the samples and collection of the carbon-13 and silicon-29 spectra under magic angle spinning, cross polarization method and high power decoupling. Interpretation of the multiplicity of signals and symmetry. The signals acquired on these nuclei will allow to identify the organic and inorganic microphases and their evolution in a chosen reactive system.

- Infrared and UV-vis spectroscopies. Preparation of the samples for the study of organic materials and recognition of the main functional groups. The same samples of which the crystalline structure has been previously determined will be used.

- Thermogravimetric analysis combined with mass spectrometry. Release and identification of volatile species adsorbed on materials, the study of reactive processes and recognition of the emitted species.

Prerequisites

The students should have gained the knowledge of basic principles of chemistry and physics.

Teaching form

Oral lessons and practice exercises/laboratory experiences.

In the COVID-19 emergency period, the lessons will be delivered in a mixed-mode: partly in presence, lessons recorded in live streaming, and deferred. Other methods may be proposed in accordance with the University ordinances.

Textbook and teaching resource

1) Chimica Analitica Strumentale K. A. Rubinson, J. F. Rubinson Zanichelli

2) Understanding NMR spectroscopy, Understanding NMR spectroscopy, J. Keeler, Wiley 2010

3) NMR of Polymers, F. A. Bovey and P. Mirau, Academic Press.

4) Lecture Notes by the Professor.

The lecture notes will allow students to follow and deepen the topics covered during the lessons.

Semester

3rd year, 1st semester

Assessment method

The oral exam will cover the following topics:

1) the theoretical aspects of the most important spectroscopic and diffractometric analysis techniques

2) the interpretation of the spectra collected during the exercises and those of some model molecules.

The written report of laboratory experiences will be analyzed and discussed.

In the Covid-19 emergency period, the exams will be carried out using the WebEx platform and on the e-learning page of the course there will be a public link for access to the examination of possible virtual spectators.

Office hours

By appointment.

In the COVID-19 emergency period, the students are invited to send an email to the teacher (angiolina.comotti@unimib.it). They will be contacted for a videocall.