Applied Analytical Chemistry

Module aims

To provide the students with an understanding of the applications of various analytical techniques and their role in modern research. This module will demonstrate the fundamental theoretical principals of selected instrumental analytical techniques (NMR spectroscopy, mass-spectrometry, atomic spectroscopy, separation and hyphenated techniques) in the context of their roles in industrial and academic research, to include chemical and pharmaceutical analysis.

Learning outcomes

Having successfully completed this module students will be able to:
1) To recognise the current trends in the application of the instrumental analytical methods
2) Demonstrate awareness of the theoretical concepts of NMR spectroscopy, Mass-Spectrometry, Chromatography, hyphenated techniques GC/HPLC-MS, and (atomic spectroscopy)-based methods
3) To choose an appropriate technique in order to evaluate the structure, properties and potential applications of materials, or a multi-technique approach to find the solution of a selected experimental problem
4) Improve their confidence in scientific communication and presentation of data to subsequently enhance their employability skills.

Skills

Having successfully completed this module students will acquire the following skills:
1) Students will develop their chemistry-related cognitive ability and skills, relating to intellectual tasks, including problem solving as required by the Chemistry subject benchmark statement
2) Students will improve their confidence in scientific communication and develop presentation skills of analytical data.

Syllabus

Introduction (lectures 1 and 2)

1. Analytical Chemistry: absolute and relative analytical methods, calibration (external calibration, standard additions, internal calibration), characteristics of an analytical procedure (accuracy, precision, specificity and selectivity, linearity range, detection and quantitation limits, robustness).
2. Sample preparation; validation of results

NMR spectroscopy (lectures 3-6)

1. Fundamentals of NMR spectroscopy; sensitivity, resolution, and scope; 1D and 2D NMR methods and techniques. Principal differences between solution and solid-state NMR (magic angle spinning, chemical shift anisotropy, quadrupolar and paramagnetic spins)
2. Applications of NMR spectroscopy
   Applications of NMR spectroscopy in synthetic chemistry, catalysis, and materials study

Mass-spectrometry (lectures 7-9)

1. Fundamentals of mass-spectrometry. Ion sources: Electron Impact Ionization (EI) and Chemical Ionization (CI), Electrospray ionization (ESI), Atmospheric Pressure Chemical Ionization (APCI) and Atmospheric Pressure Photoionization (API). Fast atom bombardment (FAB) and Matrix Assisted Laser Desorption Ionization (MALDI) mass-spectrometry. Mass spectrometry detectors: quadrupole, ion trap, time of flight (TOF).
2. Applications of mass-spectrometry in chemistry and biochemistry

Atomic spectroscopy (lectures 10-11)

1. Fundamentals of atomic spectroscopy; optical emission versus mass-spectrometry detection in ICP spectroscopy; sample preparation and matrixes, standards, speciation analysis, laser ablation.
2. Applications of atomic spectroscopy methods in chemistry and materials studies

Separation science (lectures 12-14)

1. Fundamentals of chromatography

2. Liquid Chromatography: modes of separation, instrumentation used, HPLC vs UHPLC. Method development for LC: optimising separation conditions, selecting an appropriate column and detector for the given analysis. LC detectors: UV (photodiode array versus single wave UV), fluorescence, refractive index, electrochemical methods of detection. Conductivity detectors for Ion Chromatography.
3. Gas Chromatography: modes of separation, instrumentation used. Method development for GC: optimising separation conditions, selecting an appropriate column and detector for the given analysis. GC detectors: flame ionisation detector, thermal conductivity detector.
4. Applications of chromatography in chemical and pharmaceutical analysis

Hyphenated Techniques (lectures 15-16)

1. Introduction to hyphenation (GC/MS, HPLC/MS, MS/MS). GC Interfaces: Electron Impact Ionization (EI) and Chemical Ionization (CI). HPLC Interfaces: Electrospray ionization (ESI), Atmospheric Pressure Chemical Ionization (APCI) and Atmospheric Pressure Photoionization (APPI).
2. Applications of hyphenated techniques in chemical and pharmaceutical analysis.

Recommended texts

E. H. Evans and M. E. Foulkes, Analytical chemistry: a practical approach, Oxford University Press, 2019. ISBN: 9780199651719

R. M. Granger, H. M. Yochum, J. N. Granger, and K. D. Sienerth, Instrumental analysis, Oxford University Press, USA, 2018. ISBN: 9780190865337

Additional texts

D. A. Skoog, D. M. West, F. J. Holler, and S. R. Crouch, Analytical chemistry: an introduction, 7th edition, Fort Worth: Saunders College Publishing, 2000. ISBN: 0030202930

D. A. Skoog, F. J. Holler, and S. R. Crouch, Principles of instrumental analysis, 7th edition, Brooks/Cole, 2017. ISBN: 9781305577213

J. McCullagh and N. Oldham, Mass-spectrometry, Oxford University Press, 2019. ISBN: 9780198789048

J. A. Iggo and K. V. Luzyanin, NMR spectroscopy in inorganic chemistry, 2nd edition, Oxford University Press, 2020. ISBN: 9780198794851

R. Freeman, Magnetic resonance in chemistry and medicine, Oxford University Press, 2003. ISBN 9780199262250

S. M. Nelms (Editor), Inductively coupled plasma mass spectrometry handbook, Oxford: Boca Raton, FL: Blackwell Pub.; CRC Press, 2005. ISBN: 9781405109161 (available as an e-copy online from the library page and DOI: 10.1002/9781444305463, and hard copy from the Brunswick Library Store)

J. F. Rubinson and K. A. Rubinson Contemporary chemical analysis, 2nd edition, Upper Saddle River, NJ: Prentice Hall; Pearson, 2007. ISBN: 9780130920355.

Format of delivery and assessment

Delivery: 16 h lectures, 3x2 h workshops on problem solving, and 53 h of self-directed learning.
Assessment: 1 written examination (80% mark), 3 problem(case)-based learning workshops with summary presentation in small groups (2 x 10% each; one of them is peer-assessed by students using feedback forms; one is assessed by demonstrators; one is not assessed and used for students training in problem(case)-based learning approach/presentation of results).