

ADVANCED INSTRUMENTAL ANALYSIS (MPA 201T)

Scope

This subject deals with various hyphenated analytical instrumental techniques for identification, characterization and quantification of drugs. Instruments dealt are LC-MS, GC-MS, and hyphenated techniques.

Objectives

After completion of course student is able to know,

- interpretation of the NMR, Mass and IR spectra of various organic compounds
- theoretical and practical skills of the hyphenated instruments
- identification of organic compounds

THEORY

60 Hrs

1. HPLC: Principle, instrumentation, pharmaceutical applications, peak shapes, capacity factor, selectivity, plate number, plate height, resolution, band broadening, pumps, injector, detectors, columns, column problems, gradient HPLC, HPLC solvents, trouble shooting, sample preparation, method development, New developments in HPLC-role and principles of ultra, nano liquid chromatography in pharmaceutical analysis. Immobilized polysaccharide CSP's: Advancement in enantiomeric separations, revised phase Chiral method development and HILIC approaches. HPLC in Chiral analysis of pharmaceuticals. Preparative HPLC, practical aspects of preparative HPLC. 12 Hrs
- 2 Biochromatography: Size exclusion chromatography, ion exchange chromatography, ion pair chromatography, affinity chromatography general principles, stationary phases and mobile phases. 12 Hrs
Gas chromatography: Principles, instrumentation, derivatization, head space sampling, columns for GC, detectors, quantification.
High performance Thin Layer chromatography: Principles, instrumentation, pharmaceutical applications.
- 3 Super critical fluid chromatography: Principles, instrumentation, pharmaceutical applications. 12 Hrs
Capillary electrophoresis: Overview of CE in pharmaceutical analysis, basic configuration, CE characteristics, principles of CE, methods and modes of CE. General considerations and method

- development in CE, Crown ethers as buffer additives in capillary electrophoresis. CE-MS hyphenation.
- 4 Mass spectrometry: Principle, theory, instrumentation of mass spectrometry, different types of ionization like electron impact, chemical, field, FAB and MALD, APCI, ESI, APPI mass fragmentation and its rules, meta stable ions, isotopic peaks and applications of mass spectrometry. LC-MS hyphenation and DART MS analysis. Mass analysers (Quadrupole, Time of flight, FT-ICR, ion trap and Orbitrap) instruments. MS/MS systems (Tandem: QqQ, TOF-TOF; Q-IT, Q-TOF, LTQ-FT, LTQ-Orbitrap). 12 Hrs
- 5 NMR spectroscopy: Quantum numbers and their role in NMR, Principle, Instrumentation, Solvent requirement in NMR, Relaxation process, NMR signals in various compounds, Chemical shift, Factors influencing chemical shift, Spin-Spin coupling, Coupling constant, Nuclear magnetic double resonance, Brief outline of principles of FT-NMR with reference to ¹³CNMR: Spin spin and spin lattice relaxation phenomenon. ¹³CNMR, 1-D and 2-D NMR, NOESY and COSY techniques, Interpretation and Applications of NMR spectroscopy. LC-NMR hyphenations. 12 Hrs

REFERENCES

1. Spectrometric Identification of Organic compounds – Robert M Silverstein, Sixth edition, John Wiley & Sons, 2004.
2. Principles of Instrumental Analysis – Douglas A Skoog, F. James Holler, Timothy A. Nieman, 5th edition, Eastern press, Bangalore, 1998.
3. Instrumental methods of analysis – Willards, 7th edition, CBS publishers.
4. Organic Spectroscopy – William Kemp, 3rd edition, ELBS, 1991.
5. Quantitative analysis of Pharmaceutical formulations by HPTLC – P D Sethi, CBS Publishers, New Delhi.
6. Quantitative Analysis of Drugs in Pharmaceutical formulation – P D Sethi, 3rd Edition, CBS Publishers, New Delhi, 1997.
7. Pharmaceutical Analysis– Modern methods – Part B – J W Munson, Volume 11, Marcel Dekker Series.
8. Organic Spectroscopy by Donald L. Paviya, 5th Edition.