GUJARAT TECHNOLOGICAL UNIVERSITY
PHARM.D
3rd Year

Subject Name: PHARMACEUTICAL ANALYSIS
Subject Code: 838802

Scope: This subject will delve upon concepts of quality assurance, various analytical methods used for pharmaceutical formulations.

Objectives:

a. To study quality assurance concepts, validation methods, ICH guidelines, GLP
b. To study various chromatographic techniques.
c. To study electrometric methods and spectroscopy methods.

Theory (3 Hours/ Week, Total: 90 Hours)

Teaching Scheme				Evaluation Scheme				Total Marks
Theory	Tutorial	Practical	Total	Theory		Practical		
				External	Internal	External	Internal	
3	1	3	7	70	30	70	30	200

Sr.	Topic	Hr	% Weigh tage
1.	**Quality Assurance:** a. Introduction, sources of quality variation, control of quality variation. b. Concept of statistical quality control. c. Validation methods- quality of equipment, validation of equipment and validation of analytical instruments and calibration. d. GLP, ISO 9000. e. Total quality management, quality review and documentation. f. ICH- international conference for harmonization-guidelines. g. Regulatory control.	**10**	**11.1%**
2.	**Chromatography:** Introduction, history, classification, separation techniques, choice of methods. The following techniques be discussed with relevant examples of pharmaceutical products involving principles and techniques of separation of drugs from excipients. a. **Column Chromatography**: Adsorption column chromatography, Operational technique, frontal analysis and elution analysis. Factors affecting column efficiency, applications and partition chromatography. b. **TLC**: Introduction, principle, techniques, Rf value and applications. c. **PC:** Introduction, principle, types of paper chromatography, preparation techniques, development techniques, applications. d. **Ion-exchange chromatography**: Introduction, principles, types of ion exchange synthetic resins, physical properties, factors affecting ion exchange, methodology and applications. e. **HPLC**: Introduction, theory, instrumentation, and applications. f. **HPTLC**: Introduction, theory, instrumentation, and applications. g. **Gas Chromatography**: Introduction, theory, instrumentation-carrier gases, types of columns, stationary phases in GLC & GSC. Detectors-Flame ionization detectors,	**30**	**33.3%**

	electron capture detector, thermal conductivity detector. Typical gas chromatogram, derivatisation techniques, programmed temperature gas chromatography, applications. h. **Electrophoresis**: Principles of separation, equipment for paper and gel electrophoresis, and application. i. **Gel filtration** and **affinity chromatography**: Introduction, technique, applications.		
3.	**Electrometric Methods:** Theoretical aspects, instrumentation, interpretation of data/spectra and analytical applications be discussed on the following topics. a. **Potentiometry**: Electrical potential, electrochemical cell, reference electrodes, indicator electrodes, measurement of potential and pH, construction and working of electrodes, Potentiometric titrations, methods of detecting end point, Karl Fischer titration. b. **Conductometry**: Introduction, conductivity cell, conductometric titrations and applications. c. **Polarography**: Instrumentation, DME, residual current, diffusion current and limiting current, polarographic wave, Ilkovic's equation, Effect of oxygen on polarographic wave, Polarographic maxima and suppressors and applications. d. **Amperometric Titrations:** Introduction, types of electrodes used, reference and indicator electrode, instrumentation, titration procedure, advantages and disadvantages of Amperometry over potentiometry. Pharma applications	**15**	**16.7%**
4.	**Spectroscopy:** Theoretical aspects, instrumentation, elements of interpretation of data/spectra and application of analytical techniques be discussed on: a. **Absorption Spectroscopy: -** Theory of electronic, atomic and molecular spectra. Fundamental laws of photometry, Beer-Lambert's Law, application and its deviation, limitation of Beer law, application of the law to single and multiple component analysis, measurement of equilibrium constant and rate constant by spectroscopy. Spectra of isolated chromophores, auxochromes, batho-chromic shift, hypsochromic shift, hyperchromic and hypochromic effect, effect of solvent on absorption spectra, molecular structure and infrared spectra. **Instrumentation** – Photometer, U.V.-Visible spectrophotometer – sources of U.V.-Visible radiations, collimating systems, monochromators, samples cells and following detectors-Photocell, Barrier layer cell, Phototube, Diode array, applications of U.V.-Visible spectroscopy in pharmacy and spectrophotometric titrations. - **Infrared Spectroscopy**: Vibrational transitions, frequency – structure correlations, Infrared absorption bands, Instrumentation–IR spectro-meter – sources of IR, Collimating systems, monochromators, sample cells, sample handling in IR spectroscopy and detectors–Thermocouple, Golay Cells, Thermistor, Bolometer, Pyroelectric detector, Applications of IR in pharmacy. - **Fluorimetric Analysis:** Theory, luminescence, factors affecting fluorescence, quenching. Instrumentation, Applications, fluorescent indicators, study of pharmaceutically important compounds estimated by fluorimetry b. **Flame Photometry:** Theory, nebulisation, flame and flame temperature, interferences, flame spectrometric techniques and instrumentation and pharmaceutical applications. c. **Atomic Absorption Spectrometry:** Introduction, Theory, types of electrodes, instrumentation and applications. d. **Atomic Emission Spectroscopy**: Spectroscopic sources, atomic emission spectrometers, photographic and photoelectric detection.	**35**	**38.9%**

	e. **NMR & ESR (introduction only):** Introduction, theoretical aspects and applications. f. **Mass Spectroscopy**: (**Introduction only**) – Fragmentation, types of ions produced mass spectrum and applications. g. **Polarimetry: (Introduction only**) – Introduction to optical rotatory dispersion, circular dichroism, polarimeter. h. **X-RAY Diffraction: (Introduction only**) – Theory, reciprocal lattice concept, diffraction patterns and applications. i. **Thermal Analysis**: Introduction, instrumentation, applications, and DSC and DTA.		

Prcaticals:

List of Experiments:

1. Separation and identification of Amino Acids by Paper Chromatography.
2. Separation and identification of Sulpha drugs by TLC technique.
3. Effect of pH and solvent on the UV spectrum of given compound.
4. Comparison of the UV spectrum of a compound with that of its derivatives.
5. Determination of dissociation constant of indicators using UV-Visible spectroscopy.
6. Conductometric titration of mixture of acids with a strong base.
7. Potentiometric titration of a acid with a strong base.
8. Estimation of drugs by Fluorimetric technique.
9. Study of quenching effect in fluorimetry.
10. Colourimetric estimation of Supha drugs using BMR reagent.
11. Simultaneous estimation of two drugs present in given formulation.
12. Assay of Salicylic Acid by colourimetry.
13. Determination of Chlorides and Sulphates in Calcium gluconate by Nepheloturbidimetric Method.
14. Determination of Na/K by Flame Photometry.
15. Determination of pKa using pH meter.
16. Determination of specific rotation.
17. Comparison of the IR spectrum of a compound with that of its derivatives.
18. Demonstration of HPLC.
19. Demonstration of HPTLC.
20. Demonstration of GC-MS.
21. Demonstration of DSC.
22. Interpretation of NMR spectra of any one compound.

Reference Books:

1. Text Book of Pharm. Analysis by Higuchi. T and Hasen. E. B., New York Inter Science Publishers.
2. Quantitative Pharma. Analysis by Jenkins, The Blakiston division, New York. Latest edition
3. Quantitative Drug Analysis, by Garrot. D, Chapman & Hall Ltd., London. Latest edition
4. Undergraduate Instrumental Analysis by James. E., CBS Publishers. Latest edition
5. Instrumental Analysis by Willard and Merritt, EWP, East West Press Ltd., Delhi/Madras. Latest edition
6. Pharm Analysis by Skoog and West, Sounders Manipal College Publishing. Latest edition
7. Text Book of Chemical Analysis, by A.I.Vogel, ELBS with Macmillan press, Hampshire. Latest edition
8. Textbook of Pharm. Analysis by K.A.Connors, John Wiley & Sons, New York, Brisbane, Singapore. Latest edition

9. Textbook of Pharm. Analysis (Practical) by Beckett & Stenlake, CBS Publishers, Delhi. Latest edition
10. Textbook of Drug Analysis by P.D. Sethi., CBS Publishers, Delhi. Latest edition
11. Spectroscopy by Silverstein, John & Wiley & Sons. Inc., Canada & Singapore. Latest edition
12. How to practise GMP-A Plan for total quality control by P.P. Sharma, Vandana Publications, Agra. Latest edition
13. The Science & Practice of Pharmacy by Remington Vol-I & II, Mack Publishing Co. Pennsylvania. Latest edition
14. TLC by Stahl, Spring Verlay. Latest edition
15. Text Book of Pharm. Chemistry by Chatten, CBS Publications. Latest edition
16. Spectroscopy by William Kemp, ELBS with Macmillan Press, Hampshire. Latest edition
17. I.P.-1996, The Controller of Publications, New Delhi. Latest edition
18. BPC- Dept. of Health, U.K. for HMSO. Latest edition
19. USP - Mack Publishing Co., Easton, PA. 20. The Extra Pharmacopoeia – The Pharm. Press, London Latest edition

Scheme of Practical Examination:

	Sessional	Annuals
Synopsis	05	10
Major Experiment	08	30
Minor Experiment	05	15
Viva	02	15
Max Marks	**20**	**70**
Duration	**3hrs**	**4hrs**

Note : Total sessional marks is 30 (20 for practical sessional plus 10 marks for regularity, promptness, viva-voce and record maintenance).