Name of the Programme: M.Sc. Part-II (Analytical Chemistry)

Course Code: CHA-624 **Title of the course:** Calibration and Validation in Analytical Chemistry

Number of Credits: 4

Effective from AY: 2023-24

Prerequisites for the course:	Students should have studied M.Sc. Part-I.	
Course Objectives:	1. To understand the terminologies used in measurement science 2. To classify the nature of errors involved in measurements 3. To study the concept of calibration and matrix effect in Analysis 4.To comprehend the role method validation and development in Analytical laboratories of pharmaceutical, clinical, environmental and forensic studies. 5. To gain the knowledge on application of statistical tools in Analysis	
Content	**1. Introduction** a. The vocabulary of analytical chemistry: Analysis, determination and measurement; techniques, methods, procedures, and protocols b. Classifying analytical techniques: Qualitative, quantitative and structural determination, separation and hyphenated techniques, basic principle of analysis and limitations c. Selecting an analytical method: Identification of analytical problem, understanding the selection criteria viz. accuracy, precision, sensitivity, selectivity, robustness, ruggedness, scale of operation, analysis time, availability of equipment, and cost; developing analytical procedure d. Errors in analytical measurements: Classification, methods of minimization of errors, significance of gaussian curve, probability distribution of errors.	No of hours 10
	2. Calibration and Statistical treatment of data a. Calibration in analytical chemistry: Significance and need for calibration, compensating for interferences (method blank), chemical standard, reference material, calibration of glassware and its tolerance limit (standard deviation) b. Matrix effect: Effect of matrix on signal measurement, importance of correlation coefficient, concept of curve fitting, linear regression of good data, linearity and sensitivity of instrumental measurement c. Calibration methods: External standard, standard additions and Internal standard method, case scenario to understand	22

	the suitability of each method for a given analysis. d. Statistical evaluation of analytical results: Confidence limits and interval, testing for significance, detection of bias and presence of outliers, control charts e. Calibration of important analytical instruments: UV-visible spectrophotometer, FTIR spectrophotometer, conductivity meter, GC, HPLC.	
	3. Validation a. Quality in Analytical Laboratories: Good laboratory practices, quality control, quality assurance, accreditation system. b. Validation and qualification: Overview of installation, operation, and performance qualification (IQ, OQ, PQ) of analytical equipment. c. Method validation in pharmaceutical industry: Regulatory requirements for analytical method validation International conference on harmonization (ICH) guideline Q2R1, method validation parameters and timeframe as per ICH guidelines, linearity and range criteria and their role in instrumental method validation, detailed discussion on accuracy and precision role in the method validation, Role of quantification limit and specificity -Limit of Detection (LOD) and Limit of Quantification (LOQ) for a given method.	18
	4. Case study of method development and modifications a. Environment sample monitoring: Estimation of nitrite, lead in wastewater, Measurement of calcium by flame emission spectroscopy b. Food and medicine: Generic drugs, health supplements, nutritional labels and daily nutritional requirement c. Clinical studies: Determination of glucose in human blood and urine, preservation of biological fluid for analysis of different analytes. b. Forensic analysis: Determination of blood alcohol content, Analysis of narcotic drugs, adulterations.	10
Pedagogy:	Mainly lectures and tutorials, Seminars / assignments / presentations / self-study or a combination of some of these can also be used. ICT mode should be preferred. Sessions shall be interactive in nature to enable peer group learning.	

References/ Readings	1. M. E. Swartz, I. S. Krull, Analytical method development & validation, CRC Press book, 1997. 2. G. H. Jeffery, J. Bassett, J. Mendham, R C. Denney, Vogel's Text Book of Quantitative Chemical Analysis, 5th Ed. Wiley, 1989. 3. A. H. Wachter, R. A. Nash, Pharmaceutical Process Validation, Marcel Dekker Inc, 2003. 4. L. Huber, Validation and Qualification in Analytical Laboratories, Informa Healthcare USA Inc; 2007. 5. M. Valcarcel, Principles of analytical chemistry: A text book, Springer Publications, 2000. 6. D. Harvey, Modern Analytical Chemistry, MC Graw Hill, 2000. 7. D. A. Skoog, D. M. West, F. J. Holler, Fundamentals of Analytical Chemistry, 9th Ed. Sounders College publishing, 2014. 8. B. W. Wenclawiak, M. Koch, E. Hadjicostas, Quality Assurance in Analytical Chemistry, Springer, 2004. 9. G. D. Christian, Analytical Chemistry, 6th Ed.; Wiley, 2004. 10. J. H. Kennedy, Analytical Chemistry: Principles, 2nd Ed.; Saunders College Publishing, 1990. 11. B. Magnusson, U. Ornemark, The Fitness for Purpose of Analytical Methods – A Laboratory Guide to Method Validation and Related Topics, 2nd Ed; Eurachem, 2014 12. Willard, Instrumental Methods of Analysis, 7th Ed., CBS Publishers, 1986
Course Outcomes:	1. Students will be able to differentiate between technique, method, protocol and procedure. 2. Students should be able to identify and correct any measurement errors. 3. Students will be able to analyse the reliability of results for a chosen method of analysis 4. Student will be able to evaluate the suitability of method for intended purpose 5. Student will learn to draw conclusions based on statical method.