Anekant Education Society's
Tuljaram Chaturchand College, Baramati, 413102, Dist-Pune
(Autonomous)

Revised syllabus for
M.Sc. II Analytical Chemistry, Part-II (Semester-III)

Choice Based Credit System Syllabus to be implemented from Academic year 2023-24

PSCHA-231: Analytical Method Development & Validation, Nanotechnology (48 L+ 12 T) (4 Credit)

Learning Objective: At the end of course, students should able to

- ★ Student should know the validation parameters
- ★ Explain validation Parameter, limits of detection, Comparison of two means samples.
- ★ Explain Specific methods and Applications of Dissolution Studies.
- ★ Explain Nanotechnology and their carbon allotropes.
- ★ Explain nanotubes and it's properties, methodologies, Applications.
- ★ Explain biomedical Applications.

Learning outcomes:

- ★ student should be able to apply the knowledge to solve the problems
- ★ Students able to understood method validation parameters.
- ★ Learn about the background on Nanotechnology.
- ★ Apply their learned knowledge to develop Nanomaterial's and study basic tools of nanotechnology

Section I- Analytical Method Development & Validation (24 L+ 06 T) (2 Credit)

1. Assay Validation and Inter Laboratory Transfer

Introduction, fundamental definitions, Essential principles of method transfer, method validation report, the inter-laboratory qualification (ILQ) process, **(4 L, Ref 1 pp. 3 to 14)**

2. Statistical Analysis and analytical Figure of Merit

Introduction, Errors (gross errors, systematic errors, random errors), accuracy, how to reduce systematic errors Validation parameters: Accuracy, precision, mean and standard deviation, calibration, (Linear response functions (linear regression-errors in slope and the intercept, error in the estimate of concentration, standard additions), non-linear response functions and Weighted regression analysis, internal standards), selectivity and specificity (Chromatographic methods),limit of detections (spectrophotometric methods,Chromatographic methods and related techniques, receptor binding assay), limit ofQuantification, sensitivity, ruggedness and robustness, analyte stability in the sample matrix,mean and standard deviation, reliability of results, confidence interval, comparison of results,comparison of two means of two samples **(14 L, Ref-1 pp 15 to 68, Ref- 2 pp 145-197)**

3. Overview of World Wide Regulations **(2 L, Ref-1, pp 75 to 98)**

4. Specific methods and Applications: Dissolution Studies

Introduction, Dissolution test, Apparatus – USP type –I and II, Sampling and analytical instrumentation, Single point test Vs. Dissolution profile, Calibration, regulatory guidelines, analytical validation, linearity, accuracy, precision, specificity.

(4 L, Ref-1, pp 75 to 98 and **169 to 182)**

References:

1. Development and validation of Analytical Methods, Progress Pharmaceutical and Biomedical Analysis, Vol-3, Edited by Chitofer M. Riley and Tomas W. Rosanske (Elvier)
2. Vogel's Textbook of quantitative Chemical Analysis, sixth Ed., Mendham, Denney, Barnes, Thomas, Pub: Pearson Education.
3. Handbook of modern pharmaceutical analysis, edited by SatinderAhuja and Stephen Scypinski, Academic Press, Separation science Series, Vol-3
4. HPLC method Development for pharmaceuticals, Edited by SatinderAhuja and

Henrik Rasmussen, Academic Press, Separation science Series, Vol-8
5. Practical HPLC method Development, Snyder, Kirkiand, Glajch, Wiley India Pvt. Ltd.

Section II- Nanotechnology (24 L+ 06 T) (2 Credit)

1. **Forms of Carbon:** Structure and bonding in Graphite, Diamond like Carbon(DLC) and other allotropes of carbon, carbon nanotubes and structure of C60**.(4L)**

2. **Production of Carbon based nanomaterial:**

Production of carbon nanotubes (Single walled and multi walled), arc discharge method, Laser ablation, Chemical vapour deposition, Pyrolatic technique, purification and separation of carbon nanotubes, diamond synthesis routes, preparation of nanodiamond**.(6L)**

3. **Applications of carbon based nanomaterial:**

Catalysis applications of nanoforms of carbon, supercapacitor, battery applications, water purification, solar cell applications, sensor and FET, Biological applications.**(6L)**

4. **The basic tools of nanotechnology**

Nanostructure identification by using X-ray diffraction methods (XRD), Scanning Electron Microscope (SEM), Scanning Probe Microscope (SPM), Scanning Transmission Electron Microscopy (STEM), Atomic Force Microscopy (AFM) **(6 L)**

5. Biomedical applications

Introduction, biological science, photodynamic therapy in targeted drugs, biomedical sensorand biosensor, quantum dot technology in cancer treatment, nanoparticles as drug carrier **(2 L)**

References:

1. Introduction to nanotechnology , C.P. Poole, Jr. & F.J. Owens, John Wiley & sons (2009)
2. Nano biotechnology, Subbiah Balagi, MJP publishers, India(2010)
3. The Chemistry of nanomaterials Volume,1 C.N.Rao, A Muller & A.K.Cheetham,
4. Nano, the essentials, T.Pradeep, Tata McGraw Hill new Delhi,2007
5. Nanostructures & nanomaterial's -Synthesis-properties and applications, G. Cao, Imperical college press, London 2004

PSCHA-232: Electrochemical Methods and Food Analysis
(48 L+ 12 T) (4 Credit)

Learning Objective: At the end of course, students should able to

- ★ Define various termis in electrochemistry
- ★ Explain instrumentation in electrochemistry
- ★ Define/understand various terms in food analysis techniques & methods.
- ★ Explain Describe applications of electrochemistry in industry and in analytical laboratory.
- ★ Apply select particular method of analysis for sample to be analysed.
- ★ Explain methods & principles of analysis of food , carbohydrates, proteins and preservatives.

Learning outcomes:

- ★ Students should able to solve numerical problems on electrochemistry
- ★ Interpret polarogram, cyclic voltammogram, pulse polarogram.
- ★ Select appropriate method of food analysis for its quality.
- ★ Solve numerical problems on analysis of food
- ★ Select & describe the parameters required for food quality.

Section I- Electrochemical Methods of Analysis (24 L+ 06 T) (2 Credit)

1 Coulometry

Current voltage relationship during an electrolysis, Operating cell an at fixed applied potential, Electrolysis at constant working electrode potential, Coulometric methods of analysis, Faradays laws of electrolysis, Instrumentations-Constant current and constant voltage instruments, potentiostatic coulometry-Instrumentation and applications, coulometric titrations (Amperostatic coulometry)-Apparatus and applications, advantages and limitations, problems. **(6 L)**

2 Voltammetry and polarographic methods of analysis (14 L)

A) Polarography (linear scan polarography):

Polarographic principles, Instrumentation (different types of microelectrode such as dropping mercury electrode, the static drop mercury electrode, rotating disc and ring disc electrode, cell for polarography, reference and counter electrode and circuit diagram), polarogram and polarographic currents, charging or capacitive current, role of supporting electrolyte, factors affecting on polarographic wave, Ilkovic Equation, advantages and disadvantages of DME, polarographic maxima and maxima suppressors, interference due to dissolved oxygen, Applications (qualitative analysis, quantitative analysis by calibration curve and standard addition methods), specific examples of analysis – analysis of Cu, Cd, Zn, Pb, etc. from tap water and alloys., problems.

B) Hydrodynamic Voltametry and its applications:

Volatametric detectors in chromatography, flow injection analysis, Volatametric oxygen sensors, amperometric titration).

C) Pulse Polarography:

Different types of excitation signals in pulse polarography, Differential pulse polarography, square wave polarography, Stripping method. Voltametry Withultra microelectrode, Applications of these technique Cu and Zn from tap water by differential pulse polarography and by square wave polarography, Vitamin-C by differential pulse polarography, Determination of Pb in tap water by stripping method.

D) Cyclic Voltametry:

Principle of cyclic Voltammetry, cyclic voltamogram of $K_3[Fe(CN)_6]$ and parathion, criteria of reversibility of electrochemical reactions, quasireversible and irreversible processes.

3 Amperometry:
Principle, Instrumentation, typical applications, amperometric titrations, chrono-amperometry and chrono-potentiometry. **(4 L)**

Section II- Food analysis (24 L+ 06 T) (2 Credit)

1. Carbohydrates:
Definition, classification, and functions, Analysis of carbohydrates from food sample by different method i) volumetric determination by Fehling's solution, ii) Colorimetric analysis of carbohydrates by Folin Wu method, Nelson Somyogi method, iii) total carbohydrates by Anthrone method, iv) Estimation of starch by anthrone method, v) Determination of amylase, vi) Estimation of pectic substances (gravimetric and colorimetric method), vii) Estimation of crude fibers **(5 L, Ref 9 and 10)**

2. Proteins:
Definitions and functions, Analysis of proteins by Kjedahl's method, analysis of protein by Lowry method, Estimation of amino acids by colorimetric method, Estimation of food grain for methionine content, Protein digestibility in vitro, Protein efficiency and net protein ratio, Determination of net protein utilization, digestibility and biological value, Polyacrylamide gel electrophoresis of proteins **(5 L, Ref-1 and 2)**

3. Analysis of Lipids:
Estimation of oil in oilseeds, Estimation of free fatty acids, Saponification value of oils, iodine value, Determination of acid value of oil, determination of peroxide value of oil, Identification and quantification of fatty acids **(4 L, Ref 1 and 2)**

4. Determination of food preservatives:
Definition, SO_2 legislation and determination by Tanners method, Nitrate and nitrites legislation and determination, boric acid legislation and determination, Benzoic acid legislation and determination, 4-hydroxybenzoate legislation and determination, ascorbic acid legislation and determination. Sweeteners: Saccharine identification and determination, Colours: Identification by general methods, Natural colours.**(6 L, Ref 2)**

5. Milk:
Analysis of milk and milk products: Composition of milk, analysis of milk with respect to pH, acidity, fates, casein content, lactose content, mineral content, adulteration of milk. **(2 L, Ref 1 and 2)**

References:

1) Biochemical Methods, S Sadashivan, A.Manickam; New Age Publication, 3rd Edn
2) Introduction to instrumental analysis, R. D. Broun, Mc Graw Hill (1987)
3) Instrumental methods of chemical analysis, H. willard, L.Merrit, J.A. Dean and F.A. Settle. Sixth edition CBS (1986)
4) Fundamentals of analytical chemistry, D. A. Skoog, D. M. West and H. J. Holler sixth edition (1992)
5) Principles of Instrumental Analysis, Skoog, West, Niemann.
6) Vogel Text Book of quantitative analysis 6th Ed.
7) J. chemical education, 60,302 to 308 (1983)
8) Cyclic Voltammetry and frontiers of electrochemistry, N.Noel and K.I. Vasu IBH,

New Delhi (1990)

PSCHA-233: Pharmaceutical Analysis

(48 L+ 12 T) (4 Credit)

Learning objectives:

The students are expected to learn,

- ★ Define/understand various terms in pharmaceutical raw material and finished product analysis.
- ★ Explain various pharmaceutical dosage forms and types of raw materials used.
- ★ To describe basic principles of methods of pharmaceutical analysis according to IP.
- ★ Explain importance particular test in pharmaceutical raw material and finished product analysis.

Learning Outcomes

- ★ Perform and explain importance of limit tests, identification tests and micobiological limit test of raw materials and finished products.
- ★ Solve numerical problems on analysis pharmaceutical raw material and finished product analysis.
- ★ Interpret IR spectra, HPLC chromatogram. UV-Visible spectra of pharmaceutical materials.
- ★ To perform total analysis of pharmaceutical raw material and finished product analysis according to IP/BP/USP. 9. Standardize analytical instruments according IP /BP/ USP.
- ★ Take a decision on the basis of analytical results regarding quality of raw materials so that material can be accepted for production or rejected.

Section I- Tests, Assay and Roll of FDA (24 L+ 06 T) (2 Credit)

1. A) Apparatus for test and assay, cleaning of glassware
B) Role of FDA in Pharmaceutical Industries:
Definitions of Drug & Cosmetics, Substandard Drugs, Role of FDA, Introduction to New Drug, Development of New Drugs- Selection of Area,, Phase I, Phase II, Phase III Applications to FDA for formulation and marketing of new drug. Stability studies and Shelf life fixation. **(6 L)**

2. Biological Tests & Assay:
Introduction to biological assay, Biological assay of Heparin sodium, Determination of Amylase activity, Determination of Photolytic Activity, Test for Insulin in solution, Biological Assay of Tetanus Antitoxin, Test for Undue Toxicity. **(5 L)**

3. Microbiological Tests and Assays:
Microbiological test for Antibiotics. Standard preparation and units of activity, Test organisms and Inoculums, Cylinder-plate assay receptacles, Turbidimetric assay receptacles, Assay Designs, Cylinder plate or Cup-plate method, Two level fractional assay, Test for Sterility, **(7 L)**

4. Physical Test, Determinations, Limit tests and Sterilization:
A) Disintegration Test for Tablets and Capsules, B) Dissolution Test for Tablets and Capsules, C) moisture / water content by Karl-Fischer titration, limit tests for arsenic, heavy metals, iron, lead, sulphate, chloride, D) Ash, sulphated ash, E) Methods for Sterilization Steam Sterilization, Dry heat sterilization, Sterilization by Filtration, Gas Sterilization, Sterilization by Ionizing radiation, Sterilization by heating with Bactericides, Water for Pharmaceutical use. **(6 L)**

Section II- Analysis and quality control (24 L+ 06 T) (2 Credit)

1. Analysis of vegetable Drugs:

Vegetable drugs: Sampling, foreign organic matter, ash value, acid soluble ash, acid insoluble ash, sulphated ash, Extraction of alkaloids. **(4 L)**

2. Sources of Impurities in Pharmaceutical raw materials & finished products, Shelf life of pharmaceutical product:

Raw materials, Method of manufacture, Atmospheric contaminations, Cross contamination, Microbial contamination, Container contamination, Packaging errors, Chemical instability, Temperature effect and Physical changes, shelf life of pharmaceutical product and determination of shelf life. **(4 L)**

3. Standardization and quality control of different raw materials and dosage form:

Analysis of raw materials with respect to identification, other or related substances, loss on drying, and Assay as per IP, i) adrenaline, ii) Niacin amide iii) Cephalexin, iv) ferrous fumarate, v) isoniazid and vi) paracetamol. Problems based on assay of these materials. Brief introduction to different dosage forms with the IP requirements Analytical methods for the following- Tablets, different types of tablets, uniformity in weight (aspirin) additives used in tablet manufacture, capsules, types of capsules, (Rifampicin) Powders (Sodium benzoate),

Solutions (saline NaCl) Suspensions (barium sulphate –limit test for impurity) Mouthwashes, (Ointments (salicylic acid) and creams Dimethicone by IR) Injections (Mannitol), ophthalmic preparations (sulphacteamine), Aerosols (salbutamol), BProblems based on assay of these materials. **(16 L)**

References:

1) Indian Pharmacopeia, Volume I and II.
2) Practical Pharmaceutical chemistry, A.H.Beckett & J.B.Stenlake, third edition, volume 1.
3) Remington‘s Pharmaceutical sciences.
4) Ansel’s Pharmaceutical Analysis

PSCHA-234: Characterization Techniques
(48 L+ 12 T) (4 Credit)

Learning objectives:

The students are expected to learn,

- ★ Basics of spectroscopic techniques.
- ★ Basic concepts of emmision spectroscopy.
- ★ Terms related to microscopy.
- ★ Principle and applications of various spectroscopy.
- ★ Basic concepts of NMR.
- ★ Applications of NMR, 2-D NMR.

Learning Outcomes

- ★ Student should understand the spectroscopy concepts in detail
- ★ Student should understand Basic concepts of electron spectroscopy.
- ★ Student should know XRD method for chemical analysis.
- ★ Student should understand the difference between chemiluminescence,phosphorescence, fluorescence. .
- ★ Student should solve the numerical based on all the topics included in this course.
- ★ Student should understand the Types of nuclear reactions in detail
- ★ Student should understand Basic concepts of NMR & it's applications in structure elucidation.

Section I- Spectroscopic techniques (24 L+ 06 T) (2 Credit)

1. Electron spectroscopy:

Introduction, principle of electron spectroscopy for chemical analysis (ESCA). Satellite peaks, spectral splitting, chemical shifts in FSCA. Apparatus used for ESCA, X-ray source, samples, Analysers, Detectors, Chemical analysis using ESCA, Applications, Auger electron microscopy and Ultraviolet photoelectron spectroscopy. **(6 L, Ref 1 and 6)**

2. X- ray Methods of Analysis:

Principle, Theory- X-ray spectral lines, X-ray tube, X-ray emission, Absorptive Apparatus: Sources, Collimation, sample handling, wavelength dispersive devices, Energydispersive devices, detectors, readout device, Chemical analysis using X-ray absorption, X-ray Fluorescence- instrumentation and chemical analysis, X-ray Diffraction, Chemical analysis with X-ray diffraction, numerical problems. **(10 L, Ref 1 and 6)**

3. An Introduction to Microscopy (surface characterization techniques):

Limitations of the Human Eye, the X-ray Microscope, the Transmission Electron Microscope, the Scanning Electron Microscope, Scanning Transmission Electron Microscope, Analytical Electron Microscopy, Scanning-Probe Microscopes, the transmission electron microscope **(8 L, Ref 8 and 6)**

Section II- Luminescence and NMR Study (24 L+ 06 T) (2 Credit)

1. Photochemistry:

Introduction, Laws of Photochemistry, interactionof radiation with matter, Theory of Photoluminescence, Jablonski diagram **(2L,Ref 1,9,10)**

2. Chemiluminescence:

Introduction, principle, types. Measurement of chemiluminescence, Instrumentation, quantitative chemiluminescence, Gas phase Chemiluminescence analysis, Chemiluminescent titrations, electro-chemiluminescence. **(6 L, Ref 1)**

3. Fluorescence and phosphorescence:

Introduction, Fluorescence, electron transitions during photoluminescence, factors affecting photoluminescence, luminescent apparatus, optical excitative sources, wavelength selectors, detectors ad readout devices, photo luminescentspectra, photo luminescent analysis, analysis of non-photoluminating compounds, specific examples of analysis using photoluminescence, problems

4. Supramolecular Chemistry: (4L, Ref 1 and 7)

Practical applications, Host-Guest supramolecular photochemistry,Supramolecular chemistry in photosystems:photosynthesis,water oxidation **(4L,Ref 1,9,10)**

5. Nuclear magnetic resonance spectroscopy:

1H-NMR**:** Introduction, theory, Instrumentation, Chemical shifts, spin-spin splitting, protons on heteroatom's, coupling protons with other nuclei, solvents, qualitative and quantitative analysis, problems.

13C NMR**:** Introduction, interpretation 13C NMR spectra, Chemical shifts, Spin coupling, quantitative analysis, problems.

NMR: introduction, 1H - 1H connectivity, 1H - 13C connectivity, 13C - 13C connectivity, Through space 1H - 1H proximity, option and how to use them, problems.

(12 L, Ref 1, 5 and 6)

References:

1) Introduction to instrumental analysis, R.D. Braun, MC. Graw Hill-Interl. edn.
2) Analytical spectroscopy, Kamlesh Bansal- First edition.
3) Instrumental methods of chemical analysis, Willard, Dean and Merittee- Sixth edition.
4) Analytical chemistry principles, John H Kenedey- 2nd edn, Saunders college publ.
5) Spectroscopic identification of organic compounds Silverstrine, Bassler, Morrill, 5th edn. John Wiley and sons.
6) Analytical chemistry, Ed by kellner. Mermet, otto, Valcarcel, Widmer, Second Ed., Wiley- VCH.
7) Vogel's Textbook of quantitative Chemical Analysis,., Mendham, Denney, Barnes, Thomas, Sixth Ed ,Pub: Pearson Education.
8) Electron microscopy in the study of material, P.J Grundy and G.A.Jones, Edward Arnold.

Practical course I
PSCHA-235: Analysis of Materials (4 Credit)

Learning Objective- At the end of course students should able to

★ Define/understand various terms involved practical methods of quantitative analysis.

★ To analyse organic and inorganic materials using appropriate chemical/instrumental methods

★ Explain / describe basic principles of chemical/instrumental methods used for analysis.

★ Able to handle particular instrument according to SOP.

Learning Outcomes

★ Student should understand the principles & applications of instruments in detail
★ students should able to analyse different minerals from soil, water and fertilizer sample

★ Students should able to Apply/select particular method/instrumental parameters for analysis of given sample.

★ Student should able To perform 1) selective analysis of particular component from sample. ii) Analysis at trace level from sample.

★ To conclude the results able to take the decision regarding quality of sample.

1	Analysis of ilmenite ore
2	Analysis of Dolomite ore for Ca, Mg and silicate material
3	Analysis of Bronze with respect to copper and tin
4	Analysis of nichrome alloy with respect to nickel and chromium
5	Determination of nitrogen , phosphorous and potassium from fertilizer
6	Determination of iron detergent sample
7	Determination of organic carbon from soil sample
8	Determination of COD from waste water sample
9	Determination of magnesium from talcum powder
10	Determination of calcium from plaster of paris
11	Determination of total cation concentration in waste water sample by cation exchange resin
12	To determine phosphoric acid in cold drink by molybdenum blue method
13	Analysis of Zn- chrome pigment for zinc and chromiu

14 Quantitative analysis using cyclic voltammetry of anyone – Vit.C / nitrobenzene/ any other substance for which your department has develope cyclic Quetta method

(Note: Minimum 12 experiments should be completed in this course.)
Report on industrial visit or study tour.

References:

1. Lab manual: selected experiments of Pharmaceutical analysis, Anees A Siddiqui.
2. Experiments in chemistry, D.V.Jahagirdar.
3. Pharmacopeia of India
4. Vogel's textbook of quantitative chemical analysis, sixth Ed.
5. Environmental chemistry by A.K.De.
6. Biochemical methods, Sadashivam and Manickem, Narosa publication
7. Quantitative inorganic analysis: Elementary Instrul. Analysis A. Vogel, 3rd ed. ELBS

Practical course II
PSCHA-236: Instrumental Analysis (4 Credit)

Learning objectives:

The students are expected to learn,

- ★ Basic principles of instruments.
- ★ Handling of different instruments.
- ★ Inculcate scientific knowledge.
- ★ Applications of instruments in sample analysis.
- ★ Strengthen basic concepts.
- ★ Develop critical thinking.

Learning Outcomes

- ★ Student should understand the principles & applications of instruments in detail
- ★ Student should understand handling of UV Visible spectrophotometer.
- ★ Student should know handling of Photoflurometer, potentiostat, nephelometer, pH meter, potentiometer, conductometer, polarimetry, etc.
- ★ Student should be able to find out minerals by using Flame photometer from soil, water.
- ★ Student should know principle, applications of HPLC & Gas chromatography.

1. To determine amount of each p-nitrophenol from the given sample by spectrophotometric titration.
2. Determination of strength of phosphoric acid by potentiometric titration using standard solution of sodium hydroxide.
3. Determination of Na and K from water sample by flame photometry binary method/ internal standard method.
4. Determination of boric acid by conductomtery.
5. Determination of relative strength of acetic acid, chloro acetic acid and trichloro acetic acid through measuring their Ka value by conductivity measurement method.
6. Determination of amount each copper and bismuth or copper and iron (III) from given mixture by spectrophotometric titration using standard EDTA solution.
7. P^H metric titration of anthranilic acid and glycine with NaOH.
8. Analysis of alcohol from wine by GC.
9. Analysis of paracetamol/caffeine/metformin hydrochloride by HPLC
10. Determination of sulphate and chloride and turbidimteric method
11. Determination of Ranitidine content in tablet.
12. Cyclic voltammetric study of Fe(II)/Fe(III) system basic principles and calculation of basic parameters from CV

(Note: Minimum 16 experiments should be completed in this course)

References:

1. Lab manual: selected experiments of Pharmaceutical analysis, Anees A Siddiqui.
2. Experiments in chemistry, D.V.Jahagirdar.
3. Pharmacopeia of India
4. Vogel's textbook of quantitative chemical analysis, sixth Ed.
5. Environmental chemistry by A.K.De.
6. Biochemical methods, Sadashivam and Manickem, Narosa publication
7. Senior practical physical chemistry. B.D. Khosla and V.S. Garge (R.Chand and Co).
8. Analytical chemistry by Gary Christian, 6^{th} edition, 2008