

**SARDAR PATEL UNIVERSITY  
VALLABH VIDYANAGAR**



**SYLLABUS EFFECTIVE FROM: 2018-19  
M.Sc. CHEMISTRY  
SEMESTER-III**

**ANALYTICAL CHEMISTRY**

(Total 650 marks)

Course Code	Course Title	Hours per week	Internal Marks	External Marks	Total Marks
PS03CANC21	Spectroscopy - I	4 hrs	30	70	100
PS03CANC22	Elements of Analytical Chemistry	4 hrs	30	70	100
PS03CANC23	Classical and Thermal of Methods of Analysis	4 hrs	30	70	100
PS03EANC21 and 22	ANY ONE	4 hrs	30	70	100
PS03CANC24	Practicals	8 hrs	30	70	100
PS03CANC25	Project Work	8 hrs	30	70	100
PS03CANC26	Practicals	8 hrs	30	70	100
PS03CANC27	Project Work	8 hrs	30	70	100
PS03CANC28	Comprehensive Viva	1 hrs	-	50	50
<b>Total Marks</b>					<b>650</b>

<b>Paper Code: PS03CANC21</b>	<b>Total Credit: 4</b>
<b>Title of Paper: Spectroscopy-I</b>	

Unit	Description in detail	Weightage (%)
<b>I</b>	<b>Atomic Absorption and Emission Spectroscopy:</b> Absorption of radiation by atoms; equipment: radiation sources (Hollow cathode lamps and electrode less discharge lamps); atomizers (Flame and carbon); wavelength selector and detectors; interferences in atomic absorption spectroscopy; applications and problems: qualitative and quantitative analysis. Introduction to plasma, arc and spark emission spectroscopy; equipment: inductively coupled plasma spectrometer and flame photometer; applications and problems.	<b>25</b>
<b>II</b>	<b>Molecular Luminescence Spectroscopy:</b> Introduction to molecular luminescence (fluorescence, phosphorescence and chemiluminescence); factors affecting on fluorescence and phosphorescence, theory of luminescence; instruments for measuring fluorescence (fluorometer and spectrofluorometer); application and problems.	<b>25</b>

<b>III</b>	<b>Electron Spectroscopy:</b> Introduction to electron spectroscopy (ESCA, Auger and UPS); principle and theory of ESCA; instrumentation; chemical shifts, satellite peaks and spectral splitting; application and problems. Principle and electron transition of Auger electron spectroscopy; equipment; applications and problems.	<b>25</b>
<b>IV</b>	<b>Microscopic Techniques:</b> Introduction to scanning electron microscopy (SEM), Scanning tunneling microscopy (STM) and atomic force microscopy (AFM); basic principles and theory; instrumentation and operating parameters and applications.	<b>25</b>

**Reference books:**

1. Principles of Instrumental Analysis, by Skoog, Holler and Neiman, Sanders College Publishers (USA).
2. Undergraduate Instrumental Analysis, by James W. Robinson, Marcel Dekker, Inc. (Ny.)
3. Introduction to Instrumental Analysis, by Robert D. Braun, Pharms Med Press Hyderabad- India.
4. Instrumental Method of Analysis, Willard, Merritt, Jr., Dean and Settle Jr., CBS Publishers and distributors, New Delhi, India.
5. Microscopic and Spectroscopic Imaging of the Chemical State, Michael D. Morris, Marcel Dekker, Inc. (NY.).
6. Instrumental Methods of Chemical Analysis, 24<sup>th</sup> Edition 2005, by B. K. Sarma, Goel Publishing House, Meerut.
7. Instrumental methods of analysis by B. Sivasanker, Oxford University Press, 2012.
8. Spectroscopy by H. Kaur, 5<sup>th</sup> Edition, Pragati Prakashan, Meerut, 2009.

<b>Paper Code: PS03CANC22</b>	<b>Total Credit: 4</b>
<b>Title of Paper: Elements of Analytical Chemistry</b>	

<b>Unit</b>	<b>Description in detail</b>	<b>Weightage (%)</b>
<b>I</b>	Introduction to Chemical Instrumental Analysis: Classification of analytical techniques, types of instrumental methods, instruments for analysis, performance characteristics of instruments (Accuracy and precision, Bias, sensitivity, selectivity, Dynamic range), calibration of instrumental methods, sampling and its importance, designing of sampling plan and its implementation.	<b>25 %</b>
<b>II</b>	An Overview Of Electrical Components, Simple DC And AC Circuits, Digital Electronics Computers And Micro Computers: DC and AC current, voltage, resistance and impedance measurements, Definition of basic terms of semiconductors and semi-conducting devices, transducers, transistors, transformers, capacitor, power supplies, regulators and operational amplifiers. Digital electronics-- analog and digital signals, binary numbers and its conversion to decimal numbers, digital circuit components, digital to analog convertor (DAC) and analog to digital convertor (ADC) Computer terminology, components, operational modes of computerized instruments (Inline and On-line), computer software and programming, application of computer in analytical instruments (passive and active applications), computer networking.	<b>25 %</b>
<b>III</b>	Automated Analysis: Introduction, Advantage and disadvantages of automated instruments, Discrete and continuous analyser, unit operation and its automated laboratory analyses, automated laboratory analyzers (elemental analyser, photometric analyser for clinical analysis, centrifugal analyzer), Application of automated systems.	<b>25 %</b>
<b>IV</b>	Data Treatment: Evaluating analytical data-- classification of errors, accuracy and precision, distribution of random errors, normal distribution curve, mean and standard deviation, confidence limits (CL), comparison of results (students-t-test and its application, f-test) paired t-test, Outlier tests, linear regression and correlation coefficient.  Verifying and Validating the Method: Single operator characteristic, blind analysis of standard sample, ruggedness/ robustness and equivalency testing, standards analysis and components of good manufacturing process (GMP), documentation and case study.	<b>25 %</b>

Basic Text & Reference Books:-

- Modern Analytical Chemistry, By David Harvey, Mc Graw-Hill (USA).
- Vogel's Textbook of Quantitative Chemical Analysis, By G. H. Jeffery, J. Bassett, J. Mendham and C. Denney, Longman Singapore Publisher Pte Ltd. (Singapore).
- Principles of Instrumental Analysis, by Skoog, Holler and Neiman, Sanders College Publishers(USA)
- Introduction to Instrumental Analysis, by Robert D. Braun, Pharme Med Press Hyderabad- India.
- Instrumental Methods of Analysis, 7TH Edition Willard, Merit, Dean, Settle, CBS, Publishers & Distribution.
- Analytical Chemistry by Gary D Christian 6th Edition Willey Indorsion
- A Textbook of Analytical Chemistry, Y. Anjaneyulu Pharmmed Publisher 81-88449-19-9

<b>Paper Code: PS03CANC23</b>	<b>Total Credit: 4</b>
<b>Title of Paper: Classical and thermal of methods of analysis</b>	

<b>Unit</b>	<b>Description in detail</b>	<b>Weightage (%)</b>
<b>I</b>	<b>Gravimetric and Thermal Methods of Analysis</b>	
	<b>Gravimetry:</b> Introduction and stoichiometric calculation • Factors governing size and types of gravimetric precipitates • Properties of precipitates and precipitating agent • Requirements of precipitates for Gravimetry • Coagulation, Peptization, Co-precipitation and occlusion • Applications of Gravimetry <b>Thermal Methods:</b> Recapitulation of thermal events and classification of Thermal methods • Factors affecting TA curves and TA analysis results • TGA, DTA, and DSC methods and their applications	<b>25 %</b>
<b>II</b>	<b>Introduction to titrimetric methods of analysis:</b> General principle and classification • Volumetric and gravimetric titrimetric methods and calculation • Requirements of reaction in titrimetry • Endpoint detection • Acid-base, redox, precipitation, and complexation reactions • Dissociation of acids and bases in aqueous and non-aqueous media	<b>25 %</b>
<b>III</b>	<b>Acid-Base and oxidation-reduction titrations</b>	
	Dissociation of acids and bases in water • Theoretical titration curves • Feasibility for titration of mono or dibasic acids with strong alkali solution • Applications <b>Redox titration:</b> Redox system and potentials, Formal potential, Nernst equation • Calculation of redox reaction equilibrium constant • theoretical titration Curves • Feasibility and redox indicators • Applications	<b>25 %</b>
	<b>Precipitation and complexation titrations</b>	
<b>IV</b>	<b>Precipitation titrimetry :</b> Introduction • Factors affecting the titrimetry • theoretical titration curves • Endpoint detection and indications used • Volhard Fajan and Mohr's methods <b>Complexometric titration:</b> Introduction • Ways of detecting end point • Effective stability constant and derivation of titration curves that involved EDTA titrant • Metallochromic indicators • titration error and feasibility • Strategy to titrate mixture of metal ions in solute • Applications.	<b>25 %</b>

**Basic Text & Reference Books:-**

1. Principles of Instrumental analysis-by D.A. Skoog & F.J. Holler & T.A. NiemenbySaunders College Publishers, 5<sup>th</sup> edition, 1998
2. Analytical Chemistry: Principles-by J.H. Kennedy, Saunders College Publishers, 2<sup>nd</sup> edition, 1990
3. Introduction to Chemical Analysis-by R.D. Braun, Mc-Graw Hill Book Co.2<sup>nd</sup> edition 1995

4. Vogel's Textbook of Quantitative Chemical Analysis-by G.H. Jeffery, J. Mendham, R.C. Denney, 5<sup>th</sup> edition, 1998
5. Analytical Chemistry - by G.D. Christian, Jhon Willey & Sons, 3<sup>rd</sup> edition
6. Quantitative Analysis-by R.A. Day, Prantice hall of India(P)Ltd., New Delhi, 6<sup>th</sup> edition,1993

<b>Paper Code: PS03EANC21</b>	<b>Total Credit: 4</b>
<b>Title of Paper: Separation Methods</b>	

<b>Unit-1</b>	Solvent Extraction: Principles, Mechanism of Extraction, Factors favouring solvent extraction Chromatography: History, Classification, Principles of adsorption and partition chromatography, normal phase and reverse phase chromatography, Column Chromatography, Thin layer Chromatography (TLC), High performance thin layer chromatography (HPTLC), Paper Chromatography applications.	<b>25%</b>
<b>Unit-2</b>	Gas Chromatography: Principle, Instrumentation, Solid/Liquid Stationary phases, Column types, Detectors, Zone-broadening, Van-Deemter equation, factors affecting on Column efficiency, Plate theory, Rate theory and its Applications, derivatization, Temperature programming, Theory of gas chromatography (Basic chromatography equation) and its significant parameters such as distribution coefficient, retention time, relative retention time, retention volume, Kovats retention index. ;Head-space chromatography instrument and applications Problem solving of gas chromatography experiments	<b>25%</b>
<b>Unit-3</b>	High Performance Liquid Chromatography (HPLC): Principle, Instrumentation, Applications. Chiral Chromatography: Stationary phases, mobile phase and Column types, Applications Supercritical Fluid Chromatography and Extraction: Principle, Mechanism, Instrumentation of SFC and SFE, Applications.	<b>25%</b>
<b>Unit-4</b>	Ion Exchange Chromatography: Ion Exchangers principle, Commercial grade Ion-exchangers (cation and anion), key parameter of ion-exchanger such as swelling, selectivity, capacity etc. Applications of IEC Size Exclusion Chromatography (GPC): Principle, Separation mechanism, Methods of Calibration, Applications. Electrophoresis: Principle, Reverse Osmosis, Electro dialysis, Zone Electrophoresis, Curtain Electrophoresis, Capillary Electrophoresis, Applications.	<b>25%</b>

Basic Text & Reference Books:-

- Principles of Instrumental Analysis, 6th Edition 2006, by Douglas A. Skoog, F. James Holler, Timothy A. Nieman.
- Instrumental Methods of Analysis, 6th Edition, by Willard, Merritt, Dean, Settle, CBS Publishers and Distributors.
- Contemporary Chemical Analysis, by J. F. Rubinson and K. A. Rubinson, Prentice-Hall International Inc. 1998.
- Introduction to Instrumental Analysis, by Robert D. Braun, McGraw-Hill Book company, New Delhi.
- Instrumental Methods of Chemical Analysis, 24th Edition 2005, by B. K. Sarma, Goel Publishing House, Meerut.
- Thin Layer Chromatography, A Laboratory Handbook, 1st Edition (2005), by Egon Stahl, Springer (India) Pvt. Ltd. New Delhi
- Chemical Separations: Principles, Techniques and Experiments, ISBN: 978-0-471-35197-9, (1999), by Clifton E. Meloan, Wiley and

<b>Paper Code: PS03EANC22</b>	<b>Total Credit: 4</b>
<b>Title Of Paper: Analytical techniques in Materials characterization</b>	

<b>Unit</b>	<b>Description in detail</b>	<b>Weightage (%)</b>
I	<b>Mossbauer Spectroscopy:</b> Principle, Mossbauer nuclides, Spectral parameters required for evaluating Mossbauer spectra, Instrumentation, lamb mossbauer factor, Applications, Structure determination, nature of chemical bond, biological applications.	25%
II	<b>Circular Dichromism (CD) in analysis of materials</b> Introduction, Instrumentation, experimental aspects, CD spectral features and interpretation, relation of CD spectral information to other spectroscopic techniques, Applications	25%
III	<b>Analysis and testing of polymers:</b> <ul style="list-style-type: none"> <li>• Chemical analysis of polymers: X-ray diffraction analysis, thermal analysis, TGA, DTA.</li> <li>• Physical testing of polymers: Mechanical properties, Fatigue testing, impact testing, tear resistance, hardness, abrasion resistance.</li> <li>• Thermal properties: Softening temperature, flammability.</li> <li>• Optical properties: transmittance, color, gloss, haze and transparency.</li> <li>• Electrical properties: dielectric constant and loss factor, resistivity, dielectric strength, electronic properties.</li> <li>• Chemical properties: resistance to solvents, vapor permeability, weathering.</li> </ul>	25%
IV	<b>Analysis of Paints and Pigment</b> <ul style="list-style-type: none"> <li>• Introduction, test on the total coating, water content, separation of pigment binder, thinner of solvent type coating and thinner of latex paints</li> <li>• Identification of the binder, Identification of polymer resins and oils, Identification of plasticizer, Analysis of the vehicle, Identification and Analysis of pigments,</li> <li>• Identification of inorganic pigments, Analysis of white and tinted pigments (outline of general procedure, HCL insoluble, Titanium dioxide, total lead, acid soluble Al and Fe, acid soluble calcium, total zinc, antimony oxide, total sulfate, total carbonate) analysis of colored pigments, Black pigments, other pigments, identification and analysis of thinners.</li> </ul>	25%

**Reference Books:-**

- Instrumental Methods of Chemical Analysis, 24th Edition 2005, by B. K. Sarma, Goel Publishing House, Meerut.
- Spectroscopy by H. Kaur, 5<sup>th</sup> Edition, Pragati Prakashan, Meerut, 2009.
- Analytical Biochemistry, D, J. Homes and H. Peck, Longman (1983)
- Bioanalytical Chemistry, S. R. Mikkelesen and E. Corton, John Wiley and sons 2004
- Analysis of food and beverages, George Charalanbous, Accademic press 1978.
- Encyclopaedia of industrial chemical analysis, snell et al Inter science



- Encyclopedia of Analytical Chemistry: Application, theory and instrumentation by R A Meyeres,, First volume. Pp.344-383 Wily& Sons, UK (2000)
- Textbook of polymer science 3rd edition by F.W.Billmeyer (1994).
- Principles of polymer systems by F. Rodrigue, Tata Mc Graw Hill, New Delhi.
- Principles of polymer systems by P.J.Flory, Cornell University press, New York.
- Polymer chemistry-an introduction Seymour-Carraher-Marcel Dekker. Inc. New York.
- Polymer Science by V.R. Gowarikar, N.B. Vishvanathane, New Age International Ltd. Publisher(1998)
- Polymer Science by Vasant Gowarikar, Wiley Eastern New York (1998).
- Principle of polymer science, Bahadur and sastrri, Narosa publishing house.

<b>Paper Code: PS03CANC24 OR</b>	<b>Total Credit: 4</b>
<b>Title of Paper: Practical</b>	

<b>Unit</b>	<b>Description in detail</b>	<b>Weightage (%) : 100%</b>
<b>Practicals Based On: Classical Methods of Analysis</b>		
1.	Safety and laboratory rules	(S)
2.	Determination of Copper in Brass Alloy.	(S)
3.	To determine % of purity of ferric alum.	(L)
4.	To determine % of protein in a given sample of milk.	(S)
5.	Determination of purity of phthalic anhydride.	(S)
6.	Determination of Total Hardness of water by complexometry using EDTA.	(S)
<b>Practicals Based On: Analysis of Industrial Products</b>		
1.	Calibration of glass wares (Vol. flask, pipette, burette, etc.,).	(S)
2.	Determination of purity of maleic anhydride.	(S)
3.	To determine % of Vitamin C in a given Tablet.	(S)
4.	To determine % of Vitamin C (ascorbic acid) in lemon juice.	(S)
5.	To determine the % of sulfa drugs in the given sample.	(S)
6.	To determine oxidizing power of commercial hydrogen peroxide solution and analysis of commercial hypochlorite.	(S)
<b>Practicals Based On: Instrumental Methods of Analysis</b>		
1.	Calibration of Instruments (pH meter, spectrophotometer, conductometer etc.)	(S)
2.	Determination of milk adulteration by conductivity measurements	(S)
3.	Titrate Cu(II) with EDTA photometrically and Determine the amount of Cu(II) in a given solution	(S)
4.	To determine Co and Ni in a binary mixture by spectrophotometry	(L)
5.	To determine the percentage purity of commercial soda-ash	(L)

(S) = Short exercise ; (L) = Long Exercise

#### References:

1. J. G. Dick, Analytical Chemistry, p.640, International student Edn., Mc Grow Hill, Kogaksusha Ltd., 1973.
2. Analytical chemistry by S. Shapiro Ya., Gurvich Eng. Transition, Mir Publisher, Moscow P. 279, 1975.
3. A Textbook on Experiments Calculation in Chemical Engineering By S. S. Dara, S. Chand & Company Ltd., New Delhi, 1997,
4. Quantitative Analytical Chemistry, P. 596, 15 Edition by James S. Fritz, George II. Schenk.
5. Experimental Physical Chemistry by R. C. Das and B. Behera. P. 27.
6. Quantitative Organic Analysis, Part-3 First Edition By A. I. Vogel (1958) p. 724 , p. 797..
7. Encyclopedia of Industrial methods of analysis, Vol. 8, p. 166.
8. Analytical Chemistry by G. D. Christian , 3<sup>rd</sup> Edition, p. 278, p. 411.

<b>Paper Code: PS03CANC26 OR</b>	<b>Total Credit: 4</b>
<b>Title of Paper: Practical</b>	

<b>Unit</b>	<b>Description in detail</b>	<b>Weightage (%): 100%</b>
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<b>Practicals Based On: Classical Methods of Analysis</b>		
1.	To determine Cl <sup>-</sup> ion concentration in a given sample of water.	(S)
2.	To study the influence of ionic strength on solubility of CaSO <sub>4</sub> and determine its thermodynamic solubility product and mean ionic activity using Debye-Huckel equation.	(L)
3.	Determination of crystallizing point of given naphthaline ball sample.	(S)
4.	To determine total iodide content (in ppm) in a given iodised edible salt.	(S)
<b>Practicals Based On: Analysis of Industrial Products</b>		
1.	To determine the percentage purity of glucose by iodimetry.	(S)
2.	Determination of acid value of polyester resin.	(S)
3.	To determine the epoxy equivalent weight of given epoxy resin.	(S)
4.	To determine chemical oxygen demand (COD) in a given water sample.	(S)
5.	Extraction of caffeine from dry tea leaves and its quantitative determination.	(L)
<b>Practicals Based On: Instrumental Methods of Analysis</b>		
1.	To determine the pKa Value of an indicator by spectrophotometric method.	(L)
2.	To determine nicotine in a given tobacco sample by potentiometric and titrimetric method.	(L)
3.	To determine R <sub>f</sub> value and/OR of amino acids in mixture by using Ascending and Circular paper chromatography	(S)
4.	To determine R <sub>f</sub> value of pigments of ink using paper/thin layer chromatography.	(S)

(S) = Short exercise ; (L) = Long Exercise

#### References:

1. Encyclopedia of Industrial methods of analysis, Vol. 19, p. 365.
2. Text book of Quantitative Chemical Analysis by A. I . Vogel.
3. Chemical experiments for Instrumental methods by Sawyer, Heineman and Beebe Ed., 1984.
4. A Textbook on Experiments Calculation in Chemical Engineering By S. S. Dara, S. Chand & Company Ltd., New Delhi, 1997.
5. Introduction to the chemical analysis of plastics, by A. Krause & A. Lange (Modified) p. 121.
6. Advanced Practical Physical Chemistry by J. B. Yadav. P. 99.
7. Vogel's "Textbook of Quantitative chemical analysis" by G. H. Jeffery, J. Basserr Edition. 1989.
8. Analytical Chemistry by G. D. Christian , 2<sup>rd</sup> Edition.
9. Encyclopedia of Industrial chemical analysis, Vol. 14, p. 601.
10. Analytical Chemistry by G. D. Christian , 3<sup>rd</sup> Edition, p. 506.

**OR**

**PS03CANC25 and PS03CANC27:**

\* **Project work** (as optional) in place of practicals; to be offered to some of the students, based on their merit, interest and placement with the teachers (Marks : 200). The project shall have to be carried out under the allotted teacher(s) and a dissertation shall be submitted and will be assessed for internal (60 marks) and external (140 marks), in the usual manner.

<b>Paper Code:</b> PS03CANC28	<b>Total Credit: 1</b>
<b>Title of Paper:</b> Comprehensive Viva	

<b>Description in detail</b>	<b>Weightage (%)</b>
Viva Voce From the Subjects Studied in Semester - III	100%