

**SEMESTER -3 (Analytical Chemistry -Theory)**  
**CHE(A) 501**  
**Industrial Analytical Chemistry**

**UNIT-1**

**Automation in Measurements**

Principles of automation, automatic and automated devices, Process control: off-line, at-line and on-line analysis. Continuous and discrete analyzers, feed back mechanism. Flow injection analysis, principles, dispersion coefficient, factors affecting peak height-sample volume, channel length, flow rate and channel geometry. Applications of FIA, stopped flow measurements and gradient FIA.

**UNIT-2**

**Food Analysis**

Introduction to food analysis, regulations and international standards related to food analysis, nutritional labeling, sample and sample preparation. Compositional analysis of foods for moisture, proteins, fat, fiber, ash, vitamins and minerals. Adulteration of fats and oils; milk and milk products.

**UNIT-3**

**Pharmaceutical Analysis**

Instrumental and titrimetric assays for anti-diabetic, anti-cancer, anti-tuberculosis, anti-malarial, anti-hypertensive and anti-HIV drugs based on USP/BP/IP. Heavy metal ion analysis in pharmaceuticals. Importance of UV-Visible spectrophotometry, IR spectroscopy and HPLC with UV, fluorescence and photodiode array detection in pharmaceutical industry.

**UNIT-4**

**Analysis of pesticides, soaps and detergents, fertilizers**

Classification of pesticides. Analysis of different pesticides by classical and instrumental methods. Classification of soaps and detergents with suitable examples. Characterization of soaps and detergents. Types of fertilizers and analysis of different elements like, nitrogen, phosphates, calcium, sodium, potassium and ammonia.

**Reference Books**

1. "Analytical Chemistry" by Gary D. Christian, 6<sup>th</sup> Edition, John Wiley and Sons Inc. New Jersey.
2. "Principles of Instrumental Analysis" by Douglas A. Skoog, 3<sup>rd</sup> Edition, Holt-Saunders International Edition.
3. Flow injection analysis of pharmaceuticals: automation in the laboratory by Jose Martinez Calatayud, Taylor and Francis, 1996.
4. "Food Analysis" by S. Suzanne Nielsen, 3<sup>rd</sup> edition, Springer 2003.
5. "Food Analysis Laboratory Manual" by S. Suzanne Nielsen, 3<sup>rd</sup> edition, Springer 2003.
6. Quantitative Analysis of Drugs in Pharmaceutical Formulation, 3<sup>rd</sup> edition, P.D. Sethi, CBS Publishers, 2008.
7. "Handbook of Modern Pharmaceutical Analysis" by Satinder Ahuja and Stephen Scypinski, Volume 3, Academic Press, 2001.
8. Standard Method of Chemical Analysis by F.J. Welcher, sixth edition, volume 1,2 & 3, Part two, Van Nostrand Reinhold Company.

**SEMESTER -3 (Analytical Chemistry -Theory)**  
**CHE(A) 502**

**Qualitative Optical Spectroscopic Methods**

**Unit – I Infrared Spectroscopy**

- Introduction
- IR Frequency Range and Spectrum Presentation
- Theory of Infrared Absorption
- Dispersive Spectrometers
- Fourier Transform Spectrometers
- Hyphenated Methods Involving Infrared
- Analytical Information : Qualitative and Quantitative
- Applications

**Unit – 2 Raman Spectroscopy**

- Introduction
- Dispersive Spectrophotometers
- Fourier Transform Spectrometers
- Normal Raman
- Resonance Raman
- FT – Raman
- Surface – enhanced Raman Spectroscopy (SERS)
- Raman Microprobe
- Remote Raman Analysis
- Raman Depolarization Ratios
- Analytical Information
- Applications

**Unit – 3 Nuclear Magnetic Resonance Spectroscopy**

- Introduction
- Physical and Chemical Principles
- Instrumentation
- Analytical Information : Qualitative and Quantitative
- Applications

**Unit – 4 X – Ray Diffraction**

- Introduction
- Single – Crystal Diffraction
- Powder Diffraction
- Analytical Information : Qualitative and Quantitative
- Applications

## References

1. “*Handbook of Instrumental Techniques for Analytical Chemistry*”, **Frank Settle**, published by Prentice Hall PTR, New Jersey, 1997.
2. “*Applied Infrared Spectroscopy*”, **Smith A L**, published by Wiley, New York, 1979.
3. “*Instrumental Methods of Analysis*”, **Willard H H**, 7<sup>th</sup> edition, Belmont, CA : Wadsworth, 1987.
4. “*Raman Spectroscopy*”, **Long D A**, McGraw – Hill, New York, 1977.
5. ‘*Laboratory Raman Spectroscopy*’, **Strommen D P, Nakamoto N**, Wiley, New York, 1984.
6. “*Spectrometric Identification of Organic Compounds*”, **Silverstein R M, Bassler G C, Morrill T C**, 5<sup>th</sup> edition, Wiley, New York, 1991.
7. “*Introduction to NMR Spectroscopy*”, **Abraham R J, Fisher J, Loftus P**, Wiley, New York, 1988.
8. “*Elements of X – Ray Crystallography*”, **Azaroff L V**, McGraw – Hill, New York, 1968.
9. “*X – Ray Structure Determination : A Practical Guide*”, **Stout G H, Jensen L H**, 2<sup>nd</sup> edition, Wiley, New York, 1989.

**SEMESTER -3 (Analytical Chemistry -Theory)**  
**CHE(A)503**

**Electroanalytical Techniques**

**UNIT-1      Electroanalytical Measurements**

Voltage.  
Impedance.  
The electric double layer.  
Electrocapillarity.  
Current.  
Diffusion transport.

**UNIT-2      Voltammetry**

Differential pulse polarography.  
Square wave polarography.  
A.C. polarography.  
Stripping analysis.  
Cycling voltametry.  
Amperometric titration.

**UNIT-3      Electrodeposition and Coulometry**

Electrolysis.  
Current-Voltage relation.  
Electrogravimetric analysis at constant current, constant potential and at controlled potential.  
Coulometric analysis.

**UNIT-4      Electrochemical and Bio-sensors**

Potentiometric sensors.  
Potentiometric biosensors.  
Amperometric sensors.  
Conductometric sensors.  
Applications of Field-Effect Transistors sensors.

**REFERENCES**

- 1) Peter T. Kissinger, William R. Heineman, "*Laboratory Techniques in Electroanalytical Chemistry*", Marcel Dekker Inc., New York.
- 2) Basil H. Vassos, Galen W. Ewing, "*Electroanalytical Chemistry*", John Wiley & Sons, New York.
- 3) Allen J. Bard, Larry R. Faulkner, "*Electrochemical Methods – Fundamentals and Applications*", John Wiley & Sons, New York.
- 4) Daniel C. Harris, "*Quantitative Chemical Analysis*", W.H. Freeman and Company, New York.
- 5) I.M. Kolthoff, and P.J. Elving, "*Treatise on Analytical Chemistry*", Wiley-Interscience, New York.
- 6) Brian R. Eggins, "*Chemical Sensors and Biosensors*", John Wiley & Sons, New York.

# **SEMESTER -3 (Analytical Chemistry -Theory)**

## **CHE(A)504**

### **Modern Separation Techniques**

#### **UNIT-1**

##### **Liquid Chromatography**

Principle, theory, instrumentation and applications of high-performance liquid chromatography. LC-MS, preparative chromatography, chiral chromatography, ion-chromatography, ion-pair chromatography, size-exclusion/gel permeation chromatography and affinity chromatography.

#### **UNIT-2**

##### **Gas Chromatography**

GC principle, theory, columns, detector types and applications in pharmaceutical analysis. Head space gas chromatography; Pyrolysis gas chromatography; GC-MS.

#### **UNIT-3**

##### **Electrophoresis**

Principles of electrophoresis, theory and applications of polyacrylamide gel electrophoresis, capillary zone electrophoresis, micellar electrokinetic electrophoresis, capillary electrochromatography and capillary gel electrophoresis. Isoelectric focusing.

#### **UNIT-4**

##### **Specialized chromatographic techniques**

Principle, separation process on special columns, instrumentation and applications of counter current chromatography and ice chromatography. Superheated water chromatography- A green approach for the future. Flash chromatography.

##### **Reference Books**

- 1 "Quantitative Chemical Analysis" by Daniel C. Harris, 5<sup>th</sup> Edition, W.H. Freeman and Company, New York.
- 2 "Analytical Chemistry" by Gary D. Christian, 6<sup>th</sup> Edition, John Wiley and Sons Inc. New Jersey.
- 3 Chiral Separation Techniques: A Practical Approach, 2<sup>nd</sup> edition, edited by Ganapathy Subramanian, Wiley-VCH, 2001.
- 4 Chiral Separations by Chromatography by Satinder Ahuja, American Chemical Society, 2000.
- 5 Chiral Chromatography by Thomas E. Beesley, T.E. Beesley, R.P.W. Scott, John Wiley and Sons, 1999.
- 6 A Practical handbook of preparative HPLC by Donald Wellings, Elsevier, 2006.
- 7 Ion-pair chromatography: Theory and Biological and Pharmaceutical Applications (Chromatographic Science), Milton Hearn (editor), Marcel and Dekker Inc. (1985).
- 8 Advances in Electrophoresis (Volume 2) by Andreas Chrembach, Michael J. Dunn, Bertold J. Radola, Wiley-VCH, 1989.
- 9 High Performance Capillary Electrophoresis: An Introduction by David N. Heiger. Hewlett Packard GmbH, 1992.
- 10 High-speed counter current chromatography by Yoichiro Ito and Walter D. Conway, John Wiley and Sons, 1995.
- 11 Practical Aspects of Gas Chromatography/Mass Spectroscopy by Gordon M. Message, John Wiley & Sons, 1984.

- 12 Modern Practice of Gas Chromatography by Robert L. Grob and Eugene F. Barry, 3<sup>rd</sup> edition, Wiley-Interscience, 1995.
- 13 Basic Gas Chromatography by Harold M. McNair, James M. Miller, John Wiley and Sons, 2008.
- 14 Analytical gas Chromatography by Walter Jennings, Eric Mittlefehldt and Philip Stremple, second edition, Elsevier Science, 1997.
- 15 Modern HPLC for practicing scientists by Michael W. Dong, Wiley Interscience, 2006.

**SEMESTER -3 (Analytical Chemistry -Practicals)**  
**CHE(A)505 & 506 PR**

**Industrial Analytical Chemistry-Practicals**  
**Semester III**  
**Practical (Core subject)**

1. pK<sub>a</sub> determination of drugs by spectrophotometry
2. Characterisation of drugs substance by IR.
3. Flame Photometry and Atomic Absorption Spectrophotometry.
4. Simultaneous determination of metal ions by spectrophotometry.
5. Solvent extraction of transition metal ions
6. Ion-exchange separation of cations and anions
7. Fluorimetric determination of vitamins and drugs
8. Method validation for linearity, accuracy and precision.
9. Characterization of drug substances by IR Spectroscopy.
10. High performance liquid chromatography.
11. Gas chromatography.

## **SEMESTER -4 (Analytical Chemistry -Theory)**

### **CHE(A) 507 Selected topics in Analytical Chemistry**

#### **UNIT-1**

##### **Clinical Chemistry**

Composition of blood, collection and preservation of samples, common determinations- serum electrolytes, blood glucose, blood urea nitrogen, uric acid, albumins and globulins, acid and alkaline phosphatases, barbiturates. Principles of immunoassays, radioimmunoassay, fluorescence immunoassay, enzyme immunoassay.

#### **UNIT-2**

##### **Analytical Aspects of Drug Discovery**

Discovery of new chemical entity, Identity and purity assessment, bioavailability/dissolution requirement, high-throughput screening, degradation and impurity analysis of drug substances, residual solvent and its classification, stability studies, pre-formulation studies, method development and validation.

#### **UNIT-3**

##### **Clinical and Regulatory Aspects of Drug Discovery**

Clinical trials - Phase I, II, III and IV. Quality control and quality assurance. Regulatory considerations, regulatory compliance, International Conference on Harmonization (ICH) classification and FDA guidelines, global CMC NDA.

#### **UNIT-4**

##### **Bioanalysis**

Components of bioanalytical methodology: extraction from biological matrices, chromatography and detection systems. Bioanalytical method validation parameters: sensitivity, selectivity, accuracy and precision, linearity (calibration curves), recovery, matrix effect and stability. Bioavailability and bioequivalence study, incurred sample reanalysis test for subject samples. USFDA guidelines for bioanalytical method validation and the acceptance criteria.

##### **Reference Books**

- 1 "Quantitative Chemical Analysis" by Daniel C. Harris, 5<sup>th</sup> Edition, W.H. Freeman and Company, New York.
- 2 "Analytical Chemistry" by Gary D. Christian, 6<sup>th</sup> Edition, John Wiley and Sons Inc. New Jersey.
- 3 "Principles of Instrumental Analysis" by Douglas A. Skoog, 3<sup>rd</sup> Edition, Holt-Saunders International Editions.
- 4 "Handbook of Modern Pharmaceutical Analysis" by Satinder Ahuja and Stephen Scypinski, Volume 3, Academic Press, 2001.
- 5 "Handbook of Modern Pharmaceutical Analysis" (Drug and the Pharmaceutical Sciences) by Lena Ohannesian and Anthony Streeter, Marcel Dekker Inc., New York, 2001.
- 6 Quantitative Analysis of Drugs in Pharmaceutical Formulation, 3<sup>rd</sup> edition, P.D. Sethi, CBS Publishers, 2008.
- 7 Bioanalytical Chemistry by S. Mikkelsen and E. Corton, John Wiley and Sons, 2004.
- 8 Clinical Chemistry: Principles, Procedures, Correlations, 4<sup>th</sup> edition by Michael L. Bishop, Janet L. Duben-Engelkrik, Edward P. Fody, Lippincott Williams and Wilkins, 2000.

**SEMESTER -4 (Analytical Chemistry -Theory)**  
**CHE(A) 508**  
**Quantitative Optical Spectroscopic Methods**

**Unit – I Atomic Absorption Spectrometry**

- Introduction
- Basic Principles
- Instrumentation
- Interferences
- Techniques for Quantification of Elements
- Recent Developments
- Applications

**Unit – 2 Inductively Coupled Plasma Atomic Emission Spectroscopy**

- Introduction
- Physical and Chemical Principles
- Spectrometers
- Detection, Calculation, and Output
- Analytical Information : Qualitative and Quantitative
- Applications

**Unit – 3 Atomic Fluorescence Spectroscopy**

- Introduction
- Physical and Chemical Principles
- Instrumentation
- Accuracy
- Precision
- Detection Limits
- Analytical Information : Quantitative
- Applications

**Unit – 4 X – Ray Fluorescence Spectrometry**

- Introduction
- Instrumentation
- Analytical Information : Qualitative and Quantitative
- Applications

**References**

1. “*Handbook of Instrumental Techniques for Analytical Chemistry*”, **Frank Settle**, published by Prentice Hall PTR, New Jersey, 1997.
2. “*Spectrochemical Analysis by Atomic Absorption and Emission*”, **Lajunen L H J**, Cambridge, UK : The Royal Society of Chemistry, 1992.
3. “*Advances in Atomic Spectroscopy*”, **Sneddon J**, CT : JAI Press, Greenwich, 1992.



4. “*CRC Handbook of Inductively Coupled Plasma Atomic Emission Spectrometry*”, **Varma A**, FL : CRC Press, Boca Raton, 1991.
5. “*Multielement Detection Systems for Spectrochemical Analysis*”, **Busch K W, Busch M A**, Wiley, New York, 1990.
6. “*Principles and Practice of X – Ray Spectrometric Analysis*”, 2<sup>nd</sup> edition, **Bertin, Eugene**, Plenum Press, New York, 1975.
7. “*An Introduction to X –Ray Spectrometry*”, **Jenkins, Ron**, Heyden & Sons, London, 1974.
8. “*Principles of Quantitative X – Ray Fluorescence*”, **Tertian R, Claisse F**, Heyden, London, 1982.

## **SEMESTER -4 (Analytical Chemistry -Theory)**

### **CHE(A) 509**

### **Advanced Analytical Instrumentation**

#### **UNIT-1**

##### **UHPLC and SFC**

Principle, theory, instrumentation and applications of ultra high-performance liquid chromatography (UHPLC) and super critical fluid chromatography (SFC). Comparison with HPLC.

#### **UNIT-2**

##### **Advanced mass spectrometry**

Ion sources- matrix assisted laser desorption ionization (MALDI), thermospray, electrospray, atmospheric pressure chemical ionization (APCI), atmospheric pressure photo ionization (APPI) and atmospheric pressure secondary ion ionization (APSI); Mass analyzers- quadrupole, ion-trap, time of flight (TOF), ion-cyclotron resonance and Fourier transform mass spectrometry. Tandem mass spectrometry. Applications in the analysis of biomolecules.

#### **UNIT-3**

##### **LC-NMR and LC-MS**

Principle, theory, instrumentation and applications of liquid chromatography-nuclear magnetic resonance and liquid chromatography-mass spectrometry. Application of LC-NMR-MS.

#### **UNIT-4**

##### **ICP-MS**

Principle, theory, instrumentation and applications of inductively coupled plasma-mass spectrometry.

##### **Reference Books**

1. “Quantitative Chemical Analysis” by Daniel C. Harris, 7<sup>th</sup> Edition, W.H. Freeman and Company, New York.
2. “Analytical Chemistry” by Gary D. Christian, 6<sup>th</sup> Edition, John Wiley and Sons Inc. New Jersey.
3. On-line LC-NMR and related techniques, Klaus Albert (editor), John Wiley and Sons, 2002.
4. Practical Guide to ICP-MS by Robert Thomas, Marcel Dekker Inc., 2004.
5. Packed columns SFC by T.A. Berger, RSC Chromatography Monographs, RSC, 1995.
6. Introduction to Mass Spectrometry: Instrumentation, Applications, and Strategies for Data Interpretation by J. Throck Watson, O. David Sparkman, Wiley, 2007.
7. Interpretation of Mass Spectra by Fred W. McLafferty, Turecek University Science Books, 1993.
8. Mass spectrometry-Principles and Applications by Edmond de Hoffmann and Vincent Stroobant, John Wiley and Sons, 2007.

# SEMESTER -4 (Analytical Chemistry -Theory)

## CHE(A)510

### Environmental Chemistry

#### **UNIT-1**      **Performance and Structure of Analytical Methods**

Choosing method, A statistical view of analytical procedures, Criteria for selecting a method, Sources of error in trace analysis, Sampling, Dissolution and decomposition of samples, Separation, Determination, The complete analytical procedure.

#### **UNIT-2**      **THE ATMOSPHERE**

Composition of the atmosphere, Common air pollutants and their sources, Gases, vapours and particles, Air pollution and health, Sampling of air borne solids, Examination of airborne solids, Direct instrumental methods for gaseous pollutants, Sampling of gases and the atmosphere, Gas chromatography, Some chemical methods for determining trace gases, Some case studies of air pollution.

#### **UNIT-3**      **THE HYDROSPHERE**

The hydrological cycle and pollution, The oxygen balance in natural waters, Observations on sampling, Storage of samples and prevention of contamination, The analysis of water, Selected analytical methods for water quality control, pH measurement – the glass electrode, Conductivity, Dissolved oxygen (DO), Biochemical oxygen demand (BOD), Chemical oxygen demand (COD), Methods for the determination of inorganic nitrogen, Determination of phosphate, Automation of colorimetric procedures, The determination of chloride by titrimetry (visual), Ion-selective electrodes, Ion chromatography, The determination of heavy metals, The importance of chemical species – speciation, Trace organics in water – total organic carbon (TOC), Determination of some individual compounds or groups of compounds in polluted water, Gas chromatography / mass spectrometry (GC / MS), The EPA survey procedure : priority pollutants.

#### **UNIT-4**      **THE LITHOSPHERE AND THE BIOSPHERE**

Introduction, The need for chemical analysis of solids and rocks, Available elements, Particles size distribution in solids, Soil analysis versus visual symptoms shown by plants, Sampling problems with rocks and soils, Subsampling, Dissolution for total element analysis, Some selected chemical methods in soil analysis, Flame atomic absorption spectroscopy, Flame emission spectroscopy, Other emission techniques, Identification of minerals, The nature of the biosphere, The need for analysis of zoological specimens, The merits of treating the biosphere as a whole, Sampling problems, Sample preparation problems, Sample dissolution, Analysis of plant tissues for N, P, K, Ca and Mg, Boron in plant tissue, Cobalt in plant tissue, Sulphur in plant tissue, Simultaneous multi-element analysis, The role of trace element in living systems, Trace element determinations on very small samples, Cold vapour and hydride generation systems in AAS.

#### **REFERENCES**

- 1) Lain L. Marr, Malcolm S. Cresser, *“Environmental Chemical Analysis”*, Published by International Textbook Company, New York.
- 2) Laitinen, H. A. and Harris, W. E., *“Chemical Analysis”*, 2dn Edition, McGraw-Hill, New York.
- 3) Katz, M., *“Methods of Air Sampling and Analysis”*, 2ed Edition, American Public Health Association, Washington, DC.
- 4) Israel, H. and Israel, G.W., *“Trace Elements in the Atmosphere”*. Ann Arbor, Michigan.
- 5) Z. Marczenko, *“Spectrophotometric Determination of the Elements”*, Ellis Horwood, Chichester.

- 6) Wilson, A.L., "*The Chemical Analysis of Water : General Principles and Techniques*", The Society for Analytical Chemistry, London.
- 7) Black, C.A., "*Methods of Soil Analysis*", American Society of Agronomy, Madison, Wisconsin.
- 8) Brooks, R.R., "*Geobotany and Biogeochemistry in Mineral Exploration*", Harper and Row, New York.

**SEMESTER -4 (Analytical Chemistry –dissertation/  
industrial Training  
CHE(A)511 & 512 PR**