

Analytical Chemistry
Subject Code: 02CY0505
Subject Name: Separation Techniques
M.Sc. Sem - III
Objectives:

- To understand the scope and principle of Analytical Chemistry and various chromatographic methods including LC, GC, TLC, PC and Hyphenated techniques.

Credits Earned: 6 Credits
Course Outcomes: After completion of this course, student will be able

- To develop an understanding of the range and theories of instrumental methods available in analytical chemistry.
- To develop knowledge pertaining to the appropriate selection of instruments for the successful analysis of complex mixtures.
- To develop an understanding of the role of the chemist in measurement and problem solving in chemical analysis.
- To provide practical experience in selected instrumental methods of analysis.
- To extend skills in procedures and instrumental methods applied in analytical tasks.
- To expand skills in the scientific method of planning, developing, conducting, reviewing and reporting experiments.

Pre-requisite of course: NA.
Teaching and Examination Scheme

| Teaching Scheme (Hours) | | | Credits | Theory Marks | | | Tutorial/ Practical Marks | | Total Marks |
|-------------------------|----------|-----------|---------|--------------|-------------|-------------------|---------------------------|----------------|-------------|
| Theory | Tutorial | Practical | | ESE (E) | Mid Sem (M) | CSE/ Internal (I) | Viva (V) | Term work (TW) | |
| 4 | 0 | 3 | 6 | 50 | 30 | 20 | 25 | 25 | 150 |

Contents:

| Unit | Topics | Contact Hours |
|------|---|---------------|
| 1. | Liquid Chromatography Introduction, History, Classification, Principle & basic theory of chromatography, Column adsorption chromatography, Partition chromatography, Band broadening & column efficiency, Factors affecting, Plate theory & Rate theory of chromatography, Types of Liquid chromatography, Theory, principle and Instrumentation of HPLC, Types of column, Column efficiency, Pumps, Various types of detector, Injection system, Isocratic and gradient elution, Normal phase and Reverse phase liquid chromatography, Development of HPLC and UPLC method, Choice of stationary and mobile phase, Difference between HPLC and UPLC, Applications. | 15 |
| 2 | Gas Chromatography Introduction, Types of Gas chromatography, Theory principle and Instrumentation of gas chromatography, Carrier gas, Injection port, Types columns, Solid inert support, Stationary phase, Mobile phase, Role of Detectors, Thermal conductivity detector, Flame Ionization detector, Flame photometric detector, Development of GC method, Column silanization, Factor affecting to separation, Temperature programming, Application. | 15 |
| 3 | Thin Layer Chromatography Introduction, Theory, Principle and Instrumentation of TLC, Method for the preparation of thin layers on plates, Application of sample on the chromoplates, Choice of adsorbent, Choice of mobile phase, Detecting reagent, Developing chamber, Developing and detection, Ascending, Descending and two dimensional TLC development, Impurity profiling with the help of TLC, Applications. | 15 |
| 4 | Paper Chromatography and Hyphenated techniques Paper Chromatography - Introduction, Types of PC, Theory, principle and technique of PC, Types of paper, Modification of the paper, Choice of solvents, R_f value measurement, Sample application, Precautions in PC, Quantitative estimation by PC, Applications. Hyphenated techniques - GC-MS, HP-TLC, LC-MS etc. Principle & Applications. | 15 |
| | Total Hours | 60 |

References:

1. Introduction to instrumental analysis –R.D.Broun, McGraw Hill (1987)
2. Instrumental methods of chemical analysis – H. Willard, L.Meritt, J.A. Dean and F.A. Settle. Sixth edition CBS (1986)
3. Thermal analysis –W.W. Wendlandt, John Wiley, (1986)
4. Fundamentals of analytical chemistry –D.A.Skoog, D.M. West and H.J. Holler sixth edition (1992)
5. Cyclic Voltammetry and frontiers of electrochemistry –N.Noel and K.I. Vasu IBH, New delhi (1990)
6. Nuclear Analytical Chemistry – J. Tolgyessy and S. Verga vol. 2, university Park press, (1972)
7. Radiochemistry and Nuclear methods – W.D. Ehmann and D.E. Vance, John Wiley and Sons.

Analytical Chemistry

8. Instrumental methods of chemical analysis, In; Introduction to Analytical chemistry: Sharma BK. Goel Publishing House Meerut, 23thedition; 2004

Suggested Theory distribution:

The suggested theory distribution as per Bloom's taxonomy is as per follows. This distribution serves as guidelines for teachers and students to achieve effective teaching-learning process

| Distribution of Theory for course delivery and evaluation | | | | | |
|---|------------|-------|---------|----------|--------|
| Remember | Understand | Apply | Analyze | Evaluate | Create |
| 30% | 25% | 15% | 15% | 10% | 5% |

Suggested List of Experiments:

- 1) Determine quantitatively the amino acids (Cysteine, Theonine, Valine) present in the given mixture by the technique of Ascending Paper Chromatography.
- 2) Determine quantitatively the amino acids (Cysteine, Glycine, Valine) present in the given mixture by the technique of Descending Paper Chromatography.
- 3) To determine the R_f value of given amino acids (Cysteine, Glycine, Valine) by Ascending Paper Chromatography.
- 4) To determine the R_f value of given amino acids (Cysteine, Theonine, Valine) by Descending Paper Chromatography.
- 5) To determine the R_f value of given amino acids (Alanine, Glycine, Valine) by Circular Paper Chromatography.
- 6) To determine the R_f value of given amino acids (Cysteine, Theonine, Valine) by Circular Paper Chromatography.
- 7) To determine the R_f value of given inorganic cations (Co^{+2} , Cu^{+2} and Ni^{+2}) by Descending Paper Phromatography.
- 8) To determine the R_f value of given inorganic cations (Co^{+2} , Mn^{+2} and Zn^{+2}) by Aescending Paper Chromatography.
- 9) Determine the R_f values of the inorganic cations (Co^{+2} , Cu^{+2} and Ni^{+2}) present in the given mixture by the technique of Circular Paper Chromatography.
- 10) Separation of amino acids by Thin Layer Chromatography.
- 11) Separation of dyestuffs by Thin Layer Chromatography.
- 12) Separation of KMnO_4 and $\text{K}_2\text{Cr}_2\text{O}_7$ by column chromatography.

Instructional Method:

- a. The course delivery method will depend upon the requirement of content and need of students. The teacher in addition to conventional teaching method by black board, may also use any of tools such as demonstration, role play, Quiz, brainstorming, MOOCs etc.
- b. The internal evaluation will be done on the basis of continuous evaluation of students in the laboratory and class-room.

Analytical Chemistry

- c. Practical examination will be conducted at the end of semester for evaluation of performance of students in laboratory.
- d. Students will use supplementary resources such as online videos, NPTEL videos, e-courses, Virtual Laboratory.
- e. Use of hazardous/toxic chemicals should be avoided as far as possible in laboratory.
- f. All students in the laboratory must wear safety goggles and lab coats during lab session.

Supplementary Resources:

1. <http://www.nptel.ac.in/courses/104103069/#>
2. <http://ocw.mit.edu/courses/chemistry/>
3. <http://vlab.amrita.edu/index.php?sub=2>
4. http://www.vlab.co.in/ba_labs_all.php?id=9