

SEMESTER II

11.1.4. Course Code: DSC4: ANALYTICAL CHEMISTRY2 (AC2)

Course Title: SEPARATION METHODS-I

Total Credits: 04 (Credits: Theory-02, Practical-02)

(Total Lectures: Theory- 30, Practical-60)

Objectives: To acquire basic knowledge of the analytical chemistry of important techniques that will provide the basis for their industrial production methods. To provide an adequate mastery of analytical methods used for the determination of commercial/domestic raw materials and finished product quality.

Learning Outcomes:

By the end of this course, students will be able to:

- Become familiar with fundamental concepts of partition coefficients and their role in achieving separations across different types of chromatography.
- Develop the core skills to parse existing chromatographic protocols and identify the key factors influencing a chromatography experiment.
- Understand the underlying assumptions of the most common chromatographic separation techniques and approaches to method validation.
- Understand the concept of solubility and their application in separation using distribution law.

Unit 1: Chromatography

Classification of chromatographic methods: Principles of differential migration, description of chromatographic process, distribution coefficients, modes of chromatography. the chromatography (elution time and volume) capacity factor, column efficiency and resolution, sample preparation.

(Lectures: 08)

Unit 2: Techniques of paper chromatography

Experimental modifications, various modes of developments, nature of paper, detections of spots, retardation factors, factors that affect reproducibility of R_f values (due to paper, solvent system, sample, development procedures), selection of solvent, quantitative analysis, applications.

(Lectures:06)

Unit 3: Thin layer chromatography

Stationary phase, adsorbents, liquid phase support, plate preparation, mobile phase, sample application, development, saturation of chamber, detection of spot, R_f values (effect of adsorbent, solvent, solute, development process), quantitative analysis, applications.

(Lectures: 05)

Unit 4: Solvent Extraction

Distribution law, determination of distribution ratio, batch extraction, continuous extraction, discontinuous extraction, counter-current extraction.

(Lectures: 05)

Unit 5: Dialysis and membrane filtration

General laboratory methods, filters-nitrocellulose, fiberglass and polycarbonates.

(Lectures: 06)

PRACTICALS (Credits: 02, Laboratory Periods: 60)

1. Separation and identification of amino acids present in the given mixture by **radial** and **ascending** paper Chromatography (*Perform both*).
2. Separation of ortho-nitrophenol & para-nitrophenol and *o*- and *p*-amino phenol by thin layer chromatography (TLC) and calculation of their R_f values.
3. Separation of constituents of leaf pigments by thin layer chromatography and paper chromatography (*radial & ascending both*).
4. Separation of a mixture of compounds by solvent extraction.
5. Separation of a mixture of naphthalene, benzoic acid and 2-naphthol.
6. Separation of a mixture of 1,4-dimethoxybenzene, 2-chloro benzoic acid and *p*-cresol.
7. Analysis of soil samples (*at least three soil samples to be collected for analysis*) collected from college nursery, sports ground Delhi villages/ Yamuna River bank.
 - (a) Determination of pH of soil samples.
 - (b) Determination of total soluble salts.
 - (c) Determination of carbonate and bicarbonate.
 - (d) Determination of calcium, magnesium and iron.
 - (e) Determination of conductance of the soil samples.
8. Industrial visit to STP plant.

REFERENCES:

- Fifeild, F.W.; Kealey, D. (2000), Principles and Practice of Analytical Chemistry, Wiley.
- Harris, D. C. (2007), Exploring Chemical Analysis, W.H. Freeman and Co.
- Harris, D. C. (2007), Quantitative Chemical Analysis, 6th Edition, Freeman

- 4. Mikes, O. (2000), Laboratory Handbook of Chromatographic methods, D.Van Nostrand Company Inc.

Teaching Learning Process:

- Conventional chalk and board teaching,
- Class interactions and discussions
- Power point presentation on important topics.

Assessment Methods:

- Class Tests at Periodic Intervals.
- Written assignment (s) / Presentation by individual students
- End semester University Theory and Practical Examination

Keywords: Solvent extraction, TLC, Chromatography.