P. R. GOVERNMENT COLLEGE (A), KAKINADA <u>B. Sc. (Analytical Chemistry)</u> <u>SEMESTER – III</u>

Paper - III (ANALYTICALCHEMISTRY-3)

SEPARATION METHODS - I

<u>UNIT–I</u>

SOLVENTEXTRACTION:

Introduction, principle, techniques, factors affecting solvent extraction. Different types of Solvent extraction techniques- Batch extraction, continuous extraction and counter current extraction. Application - Determination of Iron (III)

<u>UNIT–II</u>

CHROMATOGRAPHY:

A. Classification of chromatographic methods: Principle of differential migration, description of the chromatographic process, distribution coefficients.
B. Chromatography – theory and practice: Introduction, the chromatograph (elution time and volume), capacity factor, column efficiency and resolution

<u>UNIT-III</u>

A. Techniques of paper chromatography: Experimental modifications, various modes of development, nature of the paper, detection of spots, retardation factors, factors that affect the reproducibility of Rf values (due to paper, solvent system, sample, development procedure), selection of solvent, quantitative analysis. Applications.

B. Thin layer chromatography: stationary phase, adsorbents, liquid phase supports, plate preparation, mobile phase, sample application, development, saturation of chamber, detection of spot, Rf values (effect of adsorbent, solvent, solute, development process). Quantitative analysis: applications

60 hrs. (4h/w)

12hrs

12hrs

12hrs

UNIT-IV

COLUMN CHROMATOGRAPHY.

A. General: columns, matrix materials, stationary phase, column packing, application of sample, column development and sample elution, detectors and fraction collectors, applications.

B. High performance liquid chromatography: Principle, column, matrices and stationary phases, column packing, mobile phase and pumps, application of sample, detectors, applications.

UNIT-V

12hrs

A. Partition chromatography: Principle of liquid – liquid partition chromatography, Normal phase chromatography, Reversed phase liquid chromatography, applications.

B. Adsorption chromatography: Principle, adsorbents, solvents, nature of solute, operating parameters, retention volumes and times, applications.

P. R. GOVERNMENT COLLEGE (A), KAKINADA B. Sc. (Analytical Chemistry) SEMESTER – III

LABORATORYCOURSE –III 30 hrs. (2 h/w)

Max Marks : 50 M

Practical-III: Separation techniques

(At the end of Semester-III)

- 1. Determination of Rf value of amino acids using paper chromatography.
- 2. Separation and identification of monosaccharide present in a given mixture by paper chromatography.
- 3. Determination of Fe (III) by Solvent Extraction
- 4. Separation of organic mixtures through acid/base solvent extraction
- 5. Analysis of soil
 - i. Determination of pH of soil.
 - ii.Determination of total soluble salts.
 - iii.Determination of carbonate and bicarbonate.
 - iv.Determination of calcium, magnesium and iron.
- 6. Determination of adulterant in some common food items:
 - i) Chicory in coffee powder,
 - ii) Foreign resin in asafetida
 - iii) Chilli powder
 - iv) Turmeric powder
 - v) Pulses

Suggested Readings:

- 1. F.W. Fifield and D. Kealy: Analytical Chemistry.
- 2. Daniel C Harris: Exploring chemical analysis.
- 3. Daniel C Harris: Quantitative chemical analysis.
- 4. R.V. Dilts Analytical Chemistry- Methods of Separation.
- 5. O. Mikes, R.A. Chalmers: Laboratory Handbook of Chromatographic Methods.

SCHEME OF VALUATION

Max. Marks: 50

| I. | Procedure to be written in the first 15 min | utes | 10 Marks |
|------|---|--------------|----------|
| II. | Recording of data and reporting the value u | pto 2% error | 20 Marks |
| 111. | Error up to 5% | | 10 Marks |
| | Error greater than 5% | | 5 Marks |
| IV. | Viva – Voice | | 10 Marks |
| V. | Record | | 10 Marks |

P. R. GOVERNMENT COLLEGE, KAKINADA MODEL QUESTION PAPER SEMESTER – III Paper – III (ANALYTICAL CHEMISTRY-3) SEPARATION METHODS-I

Duration: 2hrs. 30Min.

Max. Marks: 60

SECTION – A

Answer any **FOUR** questions. Each question carries **10** marks.

 $4 \ge 10 = 40 M$

1. Question from Unit –I

2. Question from Unit –II

3. Question from Unit –III

4. Question from Unit - IV

5. Question from Unit - V

6. Question from Unit – III

7. Question from Unit – IV

8. Question from Unit - V

SECTION - B

Answer any **four** questions. Each question carries **5** marks.

 $4 \ge 5 = 20M$

9. Question from Unit - I

10. Question from Unit – II

11. Question from Unit – III

12. Question from Unit – IV

13. Question from Unit - V

14. Question from Unit – II

15. Question from Unit – III

16. Question from Unit - III

P. R. GOVERNMENT COLLEGE, KAKINADA SEMESTER – III Paper - III (ANALYTICAL CHEMISTRY - 3) SEPARATION METHODS-I

Duration: 2hrs. 30Min. Max.

Max. Marks: 60

<u>Blue Print:</u>

| 4. | | | | | |
|--------|----------------|--------------------------|--------------------------------|--|--|
| S. No. | Course Content | Essay Questions (10M) | Short Answer Questions (5M) | Total No. Of Questions from each Unit | |
| 1 | Unit -I | 1 | 1 | 2 | |
| 2 | Unit –II | 1 | 2 | 3 | |
| 3 | Unit –III | 2 | 3 | 5 | |
| 4 | Unit –IV | 2 | 1 | 3 | |
| 5 | Unit -V | 2 | 1 | 3 | |
| | TOTAL | 8 | 8 | 16 | |

Note: Questions should be given from Question bank

P. R. GOVERNMENT COLLEGE, KAKINADA SEMESTER – III Paper- III (ANALYTICAL CHEMISTRY-3) SEPARATION METHODS-I

Duration: 2hrs. 30Min.

Max. Marks: 60

QUESTION BANK

ESSAY QUESTIONS – 10 MARKS:

- 1. Explain the principles and applications of Solvent extraction.
- 2. Explain the principle and experimental techniques of solvent extraction
- 3. Explain about different types of Solvent extraction techniques.
- 4. Explain about the Principle and classification of Chromatographic methods
- 5. Explain about the theory and description of Chromatographic process.
- 6. Write about various modes of developments in Paper chromatographic technique.
- 7. Explain about the principle, experimental set up and applications of Paper chromatography.
- 8. Explain about principle, experimental set up and applications of TLC.
- 9. Explain briefly about Plate preparation, Adsorbents and development process in TLC
- 10. Explain about the principle and application of Column chromatography.
- 11. Explain the principle, Column packing and column developments in Column chromatography.
- 12. Explain the principle, Experimental set up and applications of HPLC
- 13. Write about Columns and detectors used in HPLC
- 14. Explain briefly about HPLC Chromatographic technique
- 15. Explain about the Principle, Adsorbents, Solvents used in Adsorption Chromatography.
- 16. Explain about the principle and applications of Adsorption chromatography
- 17. Write about Principle and applications of Liquid –liquid partition chromatography.
- 18. Explain about Normal phase and Reversed phase chromatographic techniques.

QUESTION BANK

SHORT ANSWER QUESTIONS – 05MARKS:

- 1. Explain factors affecting solvent extraction.
- 2. How do you determine Fe(III) by using solvent extraction technique?
- 3. Explain the principle and applications of solvent extraction.
- 4. Explain briefly about efficiency of a chromatographic column.
- 5. What are distribution coefficients? Explain briefly
- 6. Write about the principle of differential migration.
- 7. Explain briefly about Resolution and capacity factor.
- 8. Write about nature of paper, detection of spots in paper chromatography.
- 9. Explain about the Quantitative analysis of Paper chromatography.
- 10. Write about Quantitative analysis of TLC.
- 11. Explain about Sample application and plate preparation in TLC.
- 12. Explain about Stationary phase, Support materials and Liquid phases in TLC.
- 13. Define Rf value and write its significance.
- 14. Write about Columns and Column packing in Column chromatography.
- 15. Explain about Column development and sample elution in Column chromatography.
- 16. Explain about various Detectors used in HPLC.
- 17. Write about Stationary phases and Mobile phases used in HPLC.
- 18. Explain about Retention volumes and Retention times.
- 19. Write the principle and applications of Adsorption chromatography
- 20. What are the differences between NPC and RPC?
- 21. Explain the principle of Partition chromatography.