

CHE-HE-5026: ANALYTICAL METHODS IN CHEMISTRY

(Credits: Theory-04, Lab-02)

Theory: 60 Lectures

Course Objective: This is an elective course designed to complement the needs of students who wish to learn more about the qualitative/quantitative characterization and separation techniques. The content of this course aims to cover some of the widely used instrumental techniques for characterization of samples. Experiments included aim at giving students hands on experience using different instrumental techniques and chemical analysis.

Learning outcome: On successful completion students will have theoretical understanding about choice of various analytical techniques used for qualitative and quantitative characterization of samples. At the same time through the experiments students will gain hands on experience of the discussed techniques. This will enable students to take judicious decisions while analyzing different samples.

Qualitative and quantitative aspects of analysis:

Sampling, evaluation of analytical data, errors, accuracy and precision, methods of their expression, normal law of distribution if indeterminate errors, statistical test of data; F, Q and t test, rejection of data, and confidence intervals.

(5 Lectures)

Optical methods of analysis:

Origin of spectra, interaction of radiation with matter, fundamental laws of spectroscopy and selection rules, validity of Beer-Lambert's law.

UV-Visible Spectrometry: Basic principles of instrumentation (choice of source, monochromator and detector) for single and double beam instrument;

Basic principles of quantitative analysis: estimation of metal ions from aqueous solution, geometrical isomers, keto-enol tautomers. Determination of metal complex composition using Job's method of continuous variation and mole ratio method.

Infrared Spectroscopy: Basic principles of instrumentation (choice of source, monochromator & detector) for continuous wave and Fourier transform spectrometers; sampling techniques.

Structure elucidation through interpretation of data. Effect and importance of isotope substitution.

Flame Atomic Absorption and Emission Spectrometry: Basic principles of instrumentation (choice of source, monochromator, and detector, choice of flame and Burner designs. Techniques of atomization and sample introduction. Method of background correction, sources of chemical interferences and their method of removal. Techniques for the quantitative estimation of trace level of metal ions from water samples.

(25 Lectures)

Thermal methods of analysis:

Theory of thermogravimetry (TG), basic principle of instrumentation.
Techniques for quantitative estimation of Ca and Mg from their mixture.

(5 Lectures)

Electroanalytical methods:

Classification of electroanalytical methods, basic principle of pH metric, potentiometric and conductometric titrations. Techniques used for the determination of equivalence points. Techniques used for the determination of pK_a values.

(10 Lectures)

Separation techniques:

Solvent extraction: Classification, principle and efficiency of the technique.

Mechanism of extraction: extraction by solvation and chelation.

Technique of extraction: batch, continuous and counter current extractions.

Qualitative and quantitative aspects of solvent extraction: extraction of metal ions from aqueous solution, extraction of organic species from the aqueous and nonaqueous media.

Chromatography: Classification, principle and efficiency of the technique.

Mechanism of separation: adsorption, partition & ion exchange.

Development of chromatograms: frontal, elution and displacement methods.

Qualitative and quantitative aspects of chromatographic methods of analysis: IC, GLC, GPC, TLC and HPLC.

Stereoisomeric separation and analysis: Measurement of optical rotation, calculation of Enantiomeric excess (ee)/ diastereomeric excess (de) ratios and determination of enantiomeric composition using NMR, Chiral solvents and chiral shift reagents. Chiral chromatographic techniques using chiral columns (GC and HPLC).

Role of computers in instrumental methods of analysis.

(15 Lectures)

Recommended Books:

1. Mendham, J. et al.: Vogel's Text Book of Quantitative Chemical Analysis ; 6th Ed. Pearson Education, 2009.
2. Willard, Hobert H. et al.: Instrumental Methods of Analysis, 7th Ed. CBS Publishers & Distributors, 2004.
3. Christian, Gary D: Analytical Chemistry, 6th Ed. Wiley India (P) Ltd., 2004.
4. Harris, Daniel C: Exploring Chemical Analysis, 4th Ed. W. H. Freeman, 2008.

5. Khopkar, S.M.: Basic Concepts of Analytical Chemistry, 3rd Ed. New Age, International Publisher, 2009.
 6. Skoog, D.A. Holler F.J. and Nieman, T.A. Principles of Instrumental Analysis, 6th Ed. Thomson Asia Pvt. Ltd. Singapore.
 7. Mikes, O. and Chalmes, R.A. Laboratory Hand Book of Chromatographic & Allied Methods, Elles Harwood Ltd. London. 1979
 8. Ditts, R.V. *Analytical Chemistry: Methods of separation*. Van Nostrand, New York, 1974.
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LAB

60 Lectures

1. Separation Techniques

I. Chromatography:

(a) Separation of mixtures

(i) Paper chromatographic separation of Fe^{3+} , Al^{3+} , and Cr^{3+} .

(ii) Separation and identification of the monosaccharides present in the given mixture (glucose & fructose) by paper chromatography. Reporting the R_f values.

(b) Separate a mixture of Sudan yellow and Sudan Red by TLC technique and identify them on the basis of their R_f values.

(c) Chromatographic separation of the active ingredients of plants, flowers and juices by TLC

II. Solvent Extractions:

(i) To separate a mixture of Ni^{2+} & Fe^{2+} by complexation with DMG and extracting the Ni^{2+} - DMG complex in chloroform, and determine its concentration by spectrophotometry.

(ii) Solvent extraction of zirconium with amberliti LA-1, separation from a mixture of irons and gallium.

3. Determine the pH of the given aerated drinks fruit juices, shampoos and soaps.

4. Determination of Na, Ca, Li in cola drinks and fruit juices using flame photometric techniques.

5. Analysis of soil:

(i) Determination of pH of soil.

(ii) Total soluble salt

(iii) Estimation of calcium, magnesium, phosphate, nitrate

6. Ion exchange:

(i) Determination of exchange capacity of cation exchange resins and anion exchange resins.

- (ii) Separation of metal ions from their binary mixture.
- (iii) Separation of amino acids from organic acids by ion exchange chromatography.

7. Spectrophotometry

- (i) Determination of pKa values of indicator using spectrophotometry.
- (ii) Structural characterization of compounds by infrared spectroscopy.
- (iii) Determination of dissolved oxygen in water.
- (iv) Determination of chemical oxygen demand (COD).
- (v) Determination of Biological oxygen demand (BOD).
- (vi) Determine the composition of the Ferric-salicylate/ ferric-thiocyanate complex by Job's method.

Recommended Books:

1. Vogel, Arthur I: A Test book of Quantitative Inorganic Analysis (Rev. by G.H. Jeffery and others) 5th Ed. The English Language Book Society of Longman .
 2. Willard, Hobert H. et al.: Instrumental Methods of Analysis, 7th Ed. Wardsworth Publishing Company, Belmont, California, USA, 1988.
 3. Christian, Gary D; Analytical Chemistry, 6th Ed. John Wiley & Sons, New York, 2004.
 4. Harris, Daniel C: Exploring Chemical Analysis, Ed. New York, W.H. Freeman, 2001.
 5. Khopkar, S.M. Basic Concepts of Analytical Chemistry. New Age, International Publisher, 2009.
 6. Skoog, D.A. Holler F.J. and Nieman, T.A. Principles of Instrumental Analysis, Thomson Asia Pvt. Ltd. Singapore.
 7. Mikes, O. & Chalmes, R.A. Laboratory Hand Book of Chromatographic & Allied Methods, Elles Harwood Ltd. London.
 9. Ditts, R.V. *Analytical Chemistry: Methods of separation*. Van Nostrand, New York, 1974.
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