

SEMESTER VIII

DISCIPLINE SPECIFIC CORE COURSE-20

CREDIT DISTRIBUTION, ELIGIBILITY, AND PRE-REQUISITES OF THE COURSE

Course title & Code	Credits	Credit distribution of the course			Eligibility criteria	Pre-requisite of the course (if any)
		Lecture	Tutorial	Practical/ Practice		
ADVANCED INSTRUMENTAL METHODS II	4	3	0	1	12th Class: Physics, Chemistry, Mathematics	-

Course Objectives:

- To provide fundamental and applied knowledge of thermal and surface analytical techniques.
- To enable students to understand the principles and instrumentation of thermal analysis and nanostructure characterization.
- To interpret data from TGA, DTA, DSC, SEM, and TEM for material identification and behavior analysis.

Learning Outcomes:

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

- Understand the principles and instrumentation of thermal analysis (TGA, DTA, DSC).
- Apply thermal techniques to analyze thermal stability, decomposition, and transitions of materials.
- Understand the working, imaging principles, and resolution differences between SEM and TEM.
- Analyze nanoscale morphology, particle size, and surface features using SEM and TEM data.
- Conduct basic data interpretation and material characterization using experimental tools and simulation software.

THEORY:**UNIT 1: Introduction to Thermal Analysis****(10 Hours)**

Overview of thermal methods: Thermogravimetric Analysis (TGA), Differential Thermal Analysis (DTA), Differential Scanning Calorimetry (DSC), Classification: Dynamic and Isothermal techniques, Comparative overview: TGA, DTA, DSC, TMA (Thermomechanical Analysis), and DMA (Dynamic Mechanical Analysis), Importance of thermal analysis in

materials science, pharmaceuticals, polymers, and food technology, Types of Samples and Sample Handling: Sample forms: powders, solids, films, liquids, Factors affecting thermal behavior: mass, particle size, packing, thermal conductivity, Sample pans: crucible materials (alumina, platinum, aluminum), Calibration and Standardization: Instrument calibration using reference materials (e.g., indium, tin, lead for DSC), Baseline correction, temperature calibration, and sensitivity testing, Importance of reproducibility and blank run procedures, Factors Influencing Thermal Curves: Instrumental parameters: heating rate, gas flow rate, crucible type, Environmental effects: humidity, atmospheric pressure, Interaction with container walls or decomposition gases

UNIT 2: Thermogravimetric Analysis (TGA) (10 Hours)

Thermogravimetric Analysis (TGA) – Basic Concepts, Principle and instrumentation of TGA, Thermogram interpretation: weight loss, multi-step decomposition, Analysis of CaCO_3 decomposition, Thermal stability of polymers and metal complexes, Significance of inert (N_2) and oxidative (O_2 , air) atmospheres, TGA coupled with **mass spectrometry (TGA-MS)** for evolved gas analysis, TGA-FTIR for gas-phase identification of decomposition product.

UNIT 3: DSC and DTA (10 Hours)

Differential Scanning Calorimetry (DSC) – Basic Concepts, Heat flow vs. temperature curves, Principle and comparison of DTA vs. DSC, Endothermic and exothermic transitions, Determination of melting point, glass transition temperature (T_g), crystallinity, Heat capacity and enthalpy measurements, Phase transition of polyethylene, Crystallinity changes in semicrystalline polymers, Enthalpy of fusion for organic solids, Applications in food additives, drug formulation, and polymer quality control. **Differential Thermal Analysis (DTA)** – Basic Concepts, Principle of differential temperature measurement between sample and reference, Typical DTA curves and interpretation: endothermic/exothermic events, Instrument setup and thermocouple configurations, **Classification of Thermal Techniques**, Dynamic vs. Isothermal methods, Static vs. programmed heating, Importance of heating/cooling rates and sample holder selection.

UNIT 4: Introduction to Nanomaterials and Electron Microscopy (15 Hours)

Basics of nanomaterials: size, surface effects, properties, Introduction to imaging: resolution, contrast, magnification, Electron-material interaction and signal generation, **Scanning Electron Microscopy (SEM)**: Working principle and instrumentation of SEM, Secondary electron and backscattered electron imaging, Sample preparation and gold coating, Surface topology, morphology, particle shape analysis, SEM of nanoparticles, corrosion pits, biological materials, **Transmission Electron Microscopy (TEM)**: Principle and instrumentation of TEM, Electron diffraction, contrast mechanisms, High-resolution TEM (HRTEM), Sample thinning techniques, TEM images of metal nanoparticles, nanorods, and carbon nanotubes, Crystallite size and lattice fringes.

PRACTICAL COMPONENTS (30 Hours)

1. Thermogravimetric Analysis (TGA) of hydrated salts or polymers. Record weight loss and analyze decomposition steps.
2. DSC analysis of pure organic compounds and polymers. Measure melting point, glass transition, and enthalpy.

3. DTA of a multicomponent inorganic salt. Identify endothermic and exothermic transitions.
4. Interpretation of recorded TGA/DSC thermograms from literature or instruments.
5. SEM image analysis using provided micrographs. Measure particle size, surface texture, and morphology.
6. TEM micrograph interpretation. Determine lattice fringes, particle shape, and crystallite boundaries.
7. Visit/demo of SEM/TEM facility (if available). Instrument components, vacuum system, sample mounting.
8. Software simulation of electron beam interactions. Use open-source or institutional software for understanding signal generation.

ESSENTIAL/RECOMMENDED TEXTBOOKS

Theory:

1. **Skoog, Holler, and Crouch**, *Principles of Instrumental Analysis* (7th Ed.) – Cengage
2. **Willard, Merritt, Dean, and Settle**, *Instrumental Methods of Analysis* – CBS Publishers
3. **Tiwari, Ashutosh**, *Nanomaterials: A Guide to Fabrication and Applications* – Wiley
4. **Ertl, G., Knözinger, H., & Weitkamp, J.**, *Handbook of Heterogeneous Catalysis (vol. 1: Characterization Techniques)* – Wiley-VCH
5. **Brown, M.E.**, *Introduction to Thermal Analysis: Techniques and Applications* – Springer
6. **N.K. Kaushik and Shulka**, *Thermal Analysis Techniques and Applications*, IK International.

Practicals and Data Interpretation:

1. Practical Manual of Instrumental Techniques for Chemistry Students – University-level lab manuals
2. Lab Guide for Materials Characterization – Any departmental handout or material characterization facility guide
3. SEM/TEM Micrograph Interpretation Manual – Available from JEOL/FEI training resources
4. Online Spectral/Imaging Databases – NIMS, SpringerMaterials, NIST, etc.

Note: Examination scheme and mode shall be as prescribed by the Examination Branch, University of Delhi, from time to time.

DISCIPLINE SPECIFIC ELECTIVE 9

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