

ICP ESCA TGA  
GC MS NMR XRF  
GPC DSC  
FTIR SEM TEM

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- Materials Analysis and Characterization
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## Molecular Identification and Analysis

Technique	Principal Applications	Description	Sample Requirements	Comments, Limitations
<b>Infrared Spectroscopy</b> (Mid-IR, Near-IR, FTIR)	Structure determination, identification, functional group analysis. Qualitative characterization and quantitative analysis.	The absorption of infrared radiation due to vibrational motions in molecules is measured. Characteristic vibrational frequencies in spectra provide distinct identification of molecules.	Solids, liquid, gas. Applicable to most molecules except for homonuclear diatomics (N <sub>2</sub> , etc.).	Transmission, reflectance and micro analysis. Near-IR useful for on-line process monitoring.
<b>Raman Spectroscopy</b>	Structure determination, identification, functional group analysis. Qualitative characterization and quantitative analysis.	The inelastic scattering of UV and visible infrared radiation is measured. Generally used to measure vibrational motions in molecules. Often provides complementary information to IR.	Solids, liquid, gas. Nearly as widely applicable as IR, including homonuclear diatomics.	Not for trace (ppm) analysis in general. Useful for on-line process monitoring.
<b>Ultraviolet and Visible Spectrophotometry</b> (UV/Vis)	Quantitative analysis of trace aromatic components, discoloration problems or detection of UV cure system constituents.	The absorption of ultraviolet or visible light is measured. Molecular absorption in the UV and visible regions of the spectrum is dependent on the electronic structure of the molecule.	Soluble in non-absorbing solvents.	High sensitivity and precision.
<b>Mass Spectrometry</b> (TOF MS, FTMS and 3Q MS coupled with MALDI, EI, CI, ESI, APCI sources)	Structure determination, identification, analysis of trace contaminants; exact mass and empirical formula determination.	Samples are ionized and measured by mass-to-charge ratio.	Solids, liquid, gas soluble in appropriate solvent.	Widely applicable. Can identify materials up to 10 <sup>6</sup> Daltons using a combination of MS techniques.
<b>Nuclear Magnetic Resonance Spectroscopy</b> (Solid and Liquid State NMR)	Structure determination, identification, molecular conformation, dynamic measurements.	Nuclei having a magnetic dipole are observed in a strong magnetic field.	Solids, soluble solids and liquid.	Medium to low sensitivity. Large volume, silicon-free analysis.
<b>X-ray Diffraction</b> (XRD)	Identification of crystalline compounds, crystalline size and percent crystallinity.	The angle and intensity of x-rays diffracted from a material are measured.	Crystalline solids, partially crystalline polymers and crystalline containing liquids.	Generally non-destructive technique.

## Chromatography

<i>Technique</i>	<i>Principal Applications</i>	<i>Description</i>	<i>Sample Requirements</i>	<i>Comments, Limitations</i>
<b>Gas Chromatography (GC)</b>	Analysis of volatile multi-component materials and functionality determination, typically for ppm – percent level quantitation.	Mixtures are separated on an analytical column as components partition between vapor phase and stationary phase.	Molecules with vapor pressure above 1 torr at sample inlet temperature (up to 300° C).	Not applicable to low volatility materials. Sample introduction techniques include headspace, direct injection, thermal desorption, SPME.
<b>Liquid Chromatography (HPLC, SEC/GPC)</b>	Separation, identification and quantitation of individual components of a mixture. Molecular weight (or size) distribution in polymers.	Mixtures are separated by selective retention of components on an analytical column.	Material must be soluble in suitable solvents to enable sample detection.	Available detectors include RI, UV, Viscometry, Light Scattering, FTIR and ELSD; GPC <sup>3</sup> .
<b>Hyphenated Techniques (GC-MS, LC-MS, Headspace GC-MS)</b>	Useful for separation of mixtures and identification of individual components or isomers.	Coupling of chromatography separation techniques with mass spectrometry identification.	Solids, liquid, gas.	Powerful for structural characterization.
<b>Ion Chromatography (IC, CZE)</b>	Cation and anion analysis.	Liquid chromatography separation and quantitation of charged species (inorganic anions and cations, organic acids, etc.).	Ionic materials in aqueous solution or extractable into water.	Useful for trace analysis (ppm – ppb).

## Physical and Thermal Analysis

<i>Technique</i>	<i>Principal Applications</i>	<i>Description</i>	<i>Sample Requirements</i>	<i>Comments, Limitations</i>
<b>Thermal Analysis (TGA, DSC, DMA, TMA, MDSC)</b>	Oxidative and thermal stability. Physical, chemical, dimensional changes detected.	Changes in weight or heat absorption by transitions are detected and measured.	Solids, liquid.	Temp: 130 - 2670° K Pres: 10 <sup>-5</sup> - 700 KPa
<b>Thermal Degradation Mass Spectrometry (TGA-GC-MS, Pyrolysis GC-MS, HS-GC-MS, TD-GC-MS)</b>	Identification of evolved species from thermal degradation. Useful in understanding cure mechanism at elevated temperatures.	Thermal decomposition followed by mass spectrometry identification.	Solids, liquid.	Mass range < 1000 Daltons; temperature range < 800° C.
<b>Rheology</b>	Viscosity and viscoelastic properties.	Flow, creep and oscillation measurements using either controlled stress or strain.	Liquids to soft solids.	Interpretation of data from complex mixtures is difficult.
<b>Thermodynamic Properties</b>	Engineering calculations, materials characterization.	Density, viscosity, vapor pressure, vapor-liquid equilibrium, etc.; measurement, correlation and estimation.	Liquid.	Measurement range depends on technique. Temp: 100 - 523° K Pres: 0.01 - 3400 KPa Visc: < ~1000 cSt
<b>Flash Point (Pensky-Marten Closed Cup, Setaflash [Small Scale Closed Tester], TAG Open and Closed Cup, Cleveland Open Cup)</b>	Determination of flammability temperature of materials.	Sample is heated in appropriate apparatus based on material viscosity and expected flash point.	Solids and liquid.	Large sample, 100mL minimum.

## Microscopy and Surface Analysis

<i>Technique</i>	<i>Principal Applications</i>	<i>Description</i>	<i>Sample Requirements</i>	<i>Comments, Limitations</i>
<b>Optical Microscopy</b>	Sample appearance including shape, size, texture and uniformity. Contaminant characterization/separation.	Image is enlarged and viewed using reflected or transmitted light. Phase, DIC, polarized light, hot and cold stage capabilities.	Solids, liquid.	Few limitations. Magnification 5X to 1000X. 0.5 micron resolution.
<b>IR Microscopy</b>	Structure determination, identification, functional group analysis on small samples or small features/ phases within a sample.	The absorption of infrared radiation due to vibrational motions in molecules is measured. Characteristic vibrational frequencies in spectra provide distinct identification of molecules.	Solids, liquid. Applicable to most molecules except for homonuclear diatomics (N <sub>2</sub> , etc.).	Sample preparation needs vary. 15 micron spatial resolution.
<b>Raman Microscopy</b>	Structure determination, identification, functional group analysis on small samples or small features/ phases within a sample.	The inelastic scattering of visible or near-IR radiation is measured. Generally used to measure vibrational motions in the molecules.	Solids, liquid. Nearly as widely applicable as IR, including homonuclear diatomics.	Often requires no sample preparation. Tight laser focusing can cause some samples to burn. 6 micron spatial resolution.
<b>Scanning Electron Microscope</b> (Conventional, FE-SEM, LV-SEM)	Micron to sub-micron sample morphology and topography information. Equipped with EDS (elemental analysis) capability.	Secondary and backscattered electrons are used to generate 3D-like, high-resolution images.	Non-volatile solid for conventional SEM and FE-SEM. Solids/semi-solids for LV-SEM; cryo capabilities.	Magnification 50X to 200,000X. 5 nm resolution.
<b>Transmission Electron Microscope</b> (STEM, EDS, electron diffraction and cryo capability)	Provision of fine, micro-structure information including filler/phase dispersion and crystallinity.	High energy electrons are transmitted through a thin sample to obtain high-resolution images.	Most solids and liquids. Cryo microtome and cryo plunge sample preparation.	Extensive sample preparation may be required. ~2 Angstrom resolution.
<b>Atomic Force Microscopy</b> (AFM)	Morphology, surface roughness and topography.  Nano-indentation capabilities.	A fine probe is scanned across a sample surface. Cantilever deflection caused by atomic forces between the surface and probe atoms is used as the imaging mechanism.	Solids no more than 8 inches in diameter and 0.5 inch in height.	Atomic resolution in z direction. 5 nm lateral resolution.
<b>Electron Spectroscopy for Chemical Analysis</b> (ESCA or XPS)	Surface elemental and bonding information including element identification, depth profiling and element mapping.	Sample is irradiated with x-rays and the emitted photoelectrons are measured as a function of binding energy.	Sample must be a vacuum stable, clean solid.	Part per thousand detection limits. 30 micron lateral resolution; 30-50 Angstrom probe depth resolution.
<b>Contact Angle</b>	Can provide wettability information, surface energy and heterogeneity information about surface measured.	The tangent angle of droplets of test liquids (generally water, hexadecane and methylene iodide) are measured against a solid surface.	Clean, solid surface.	Can do dynamic measurements.

## Elemental Analysis

<i>Technique</i>	<i>Principal Applications</i>	<i>Description</i>	<i>Sample Requirements</i>	<i>Comments, Limitations</i>
<b>Atomic Absorption Spectroscopy</b> (AAS or FAAS)	Trace (hundreds of ppm to single digit ppm), quantitative elemental analysis for single elements.	Sample is vaporized in flame, forming ground state atoms that absorb light from an element lamp. Amount of absorption is measured.	Virtually any liquid or solid that can be digested to a liquid or dissolved in a plasma sustainable solvent.	May require time-consuming sample preparation (digestion).
<b>Inductively Coupled Plasma-Atomic Emission Spectroscopy</b> (ICP-AES)	Low detection (thousands of ppm down to single ppb), quantitative analysis of approximately 70 elements.	Liquid sample is sprayed into argon plasma, electrons are excited. Photoemission occurs at characteristic wavelengths for elements.	Virtually any liquid or solid that can be digested to a liquid or dissolved in a plasma sustainable solvent.	May require time-consuming sample preparation (digestion). Not suitable for H, He, C, N, O, F, S, Cl, Br, noble gases or actinides.
<b>Inductively Coupled Plasma-Mass Spectrometry</b> (ICP-MS, HR-ICP-MS)	Ultra-trace detection (ppm-ppt), quantitative analysis of most elements.	Liquid sample is ionized in argon plasma; resulting ions are sorted and mass analyzed. HR-ICP-MS housed in class 100 cleanroom.	Virtually any liquid or solid that can be digested to a liquid or dissolved in a plasma sustainable solvent.	May require time-consuming sample preparation (digestion).
<b>Energy Dispersive Spectroscopy</b> (EDS or EDX)	Spatially resolved near-surface (depth of a few microns) elemental analysis for detection of elements B to U. Point, line scan and mapping capabilities.	Sample is irradiated with x-rays generated in an electron microscope. Characteristic x-rays are measured based on the energy of the emitted x-rays.	Solid, non-volatile sample.	Part per thousand detection limits. Semi-quantitative. 250 nm spatial resolution.
<b>Wavelength Dispersive X-ray Fluorescence</b> (WDXRF)	Bulk qualitative and semi-quantitative analysis of elements of atomic no > 5 (B).	Sample irradiated with x-rays from a high-energy source. Characteristic x-rays are measured based on the wavelength of the emitted x-rays.	Solids and liquid.	Sub ppm to percent level detection capabilities (element dependent). Non-destructive technique.



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