



analytical services
and technology

—
always solving



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We at Ashland are **passionate, tenacious, solvers** who thrive on developing practical, innovative, and elegant solutions to complex problems, always pushing the boundaries of what's possible, and advancing the competitiveness of our customers.

Our people bring exceptional product knowledge, technical support and industry insights to help our customers amplify the **efficacy**, refine the **usability**, add to the **allure**, ensure the **integrity**, and improve the **profitability** of their products and applications.

Our Analytical Services and Technology (AS&T) team is always solving. We provide a full range of services to meet your needs, from routine mechanical and analytical testing to microscopy and material characterization. We serve markets including plastics, composites, adhesives and much more.

spectroscopy and microscopy

The spectroscopy and microscopy group provides quantitative analysis of product formulations, identification of unknown materials, compositional analysis of polymers and resins, and failure analysis of coatings and adhesives.

energy-dispersive X-ray spectroscopy

Energy-dispersive X-ray spectroscopy is capable of surveying small samples or particles for elements from boron through uranium. Line profiles compare element concentration versus depth.

Elemental mapping can be conducted to document the distribution of elements across a sample surface.

applications

- analysis of contaminants or corrosion products in casting defects and composites
- identification of metals used as catalytic, cross-linking or fire-retardant agents in composites and adhesives
- qualitative analysis of powders and unknowns

fourier-transform infrared spectroscopy

Fourier-transform infrared spectroscopy is a commonly used problem-solving tool. The infrared analysis provides general information about a sample's chemical composition. The infrared spectrum can be used to confirm the identity of a material or provide generic information regarding an unknown material's chemical family. A variety of sample handling accessories are available.

applications

- identification of particle contaminants, film inclusions or small samples by infrared microscopy
- identification of polymeric resins and other coatings
- analysis for identification of cured or insoluble materials such as composite materials, sludges, gels, thermoplastic parts and inorganic fillers
- near-surface analysis for the study of adhesion, coating problems and surface contaminants

liquid chromatography / mass spectrometry

Mass spectrometry provides essential chemical information for resolving many complex industrial problems. Various ionization techniques and interface capabilities for HPLC and GPC separations enable effective analyses for a wide range of materials.

applications

- polymer characterization: end-group identification, oligomer distribution and synthetic pathway variations
- identification of surfactants in emulsion adhesives
- identification of various additives, including UV stabilizers, tackifiers and flame retardants in adhesives and composite polymers

nuclear magnetic resonance spectroscopy

Nuclear magnetic resonance (NMR) spectroscopy is an analytical tool that provides detailed information about the molecular structure of a material. NMR is most commonly used to analyze organic materials such as solvents, soluble polymers, surfactants and reaction intermediates. A variety of different NMR experiments help determine chemical information.

applications

- composition information about polymers and resins, including unsaturated polyesters, phenol-formaldehyde resins, acrylic adhesives and other cross-linking polymers
- complete quantitative analyses of product formulations
- purity assays of raw materials and products from experimental reactions
- quantitative assays of individual species in mixtures
- kinetics and reaction completion studies
- identification of unknown organic materials

optical light microscopy

Optical light microscopy is used to examine and document sample appearance and features.

applications

- documentation of particles and fibers
- defect analysis in adhesive coatings and composites
- determination of particle dimensions, particle count, etc., using image analysis software

scanning electron microscopy

Scanning electron microscopy (SEM) offers high magnification and resolution for examination of sample surfaces and cross sections. Imaging modes provide information about sample morphology and texture or information regarding variability in sample composition and density. Variable pressure imaging allows analysis of uncoated or non-conductive samples.

applications

- analysis of variations in surface morphology
- examination of particulates and fibers
- thickness measurements of polymeric coatings and films
- failure analysis of composites and castings
- general high-magnification studies

x-ray diffraction

X-ray diffraction is useful for the identification of crystalline compounds. This nondestructive technique provides semi-quantitative data on mixtures. Computer-based library searching permits positive identification of unknowns. Certain crystalline phases can be quantified.

applications

- analysis of crystalline coatings and films
- identification of fillers, clays and refractory components in coating formulations
- compositional analysis of crystalline solid compounds
- small spot analysis
- high-temperature studies

x-ray fluorescence

Wavelength dispersive X-ray fluorescence can qualitatively and quantitatively determine the presence of elements from carbon through uranium. Both solids and liquids can readily be assayed for composition or contamination. Quantitative analyses can be performed either via a calibration curve or a standardless fundamental parameters technique.

applications

- quantitative analysis of trace metals used as catalysts, cross-linking agents, etc.
- determination of fire-retardant additives
- identification of low-level contaminants
- bulk compositional analysis
- small spot / line scan / mapping capability



materials characterization

The materials characterizations group provides test data to measure the mechanical integrity of molded resin castings and composites as well as offering sophisticated thermal analysis and rheological testing.

The group performs many ASTM test procedures applied to analyses of plastics, composites, adhesives and coatings. The data are used to qualify materials for customer specifications, to help design materials for optimum end-use performance, or to evaluate the effects of exposure environments on mechanical integrity.

differential scanning calorimetry

Differential scanning calorimetry (DSC) measures the heat flow into and out of a material as a function of time or temperature. This technique can be used to characterize thermal transitions and chemical reactions. Modulated DSC is a single-test technique that allows the separation of reversing phenomena (T_g , melting, etc.) from non-reversing events (volatilization, curing, etc.). Problems or applications that could not be solved by DSC because of interfering events can now be clearly resolved and understood using modulated DSC.

applications

- glass transition temperature (T_g)
- melting points
- crystallization
- heats of fusion and reaction
- specific heat and heat capacity
- rate of cure
- reaction kinetics

dynamic mechanical analysis

Dynamic mechanical analysis (DMA) is a non-destructive mechanical test that measures the stiffness modulus (elastic nature) and energy damping (viscous nature) properties of materials as the materials are deformed under periodic strain or stress.

applications

- modulus
- glass transition temperature (T_g)
- degree of cure

- gelation of prepregs
- damping characteristics
- composite (resin-reinforcement) interactions
- impact resistance correlations
- polymer morphology correlations
- sound absorption correlations

mechanical testing using a video extensometer

The use of a video extensometer avoids materials prematurely failing during mechanical testing due to induced stresses or micro-cracks caused by physical contact with the extensometers used to measure the strain (elongation). The measurement of the ultimate elongation and an accurate modulus for materials which exhibit a very high elongation prior to failure are particularly difficult.

As there is no physical contact between the sample and the extensometer, analyses can be directed to specific regions of the digitized stress-strain curve for detailed and accurate characterization of the material's mechanical performance.

The technique is applicable to measurements in more hostile environments as the absence of physical contact also allows for the unit to be located outside of the sample environment. For example, strain measurements need not be corrected for the effects of temperature. This technique is well-suited for measurements of brittle castings, rubber, high-elongation (tough) polymers and composites.

rheological analysis

Rheological capabilities include both dynamic and constant shear. A wide range of transducers allows the rheological measurements of highly filled viscous pastes and polymer melts, as well as low-viscosity fluids and emulsions. Disposable plates allow the measurement of flow and cure for chemically reacting or cross-linking samples. This information is essential for processing design and end-use property determination.

applications

- viscosity
- rate of cure
- gelation
- yield stress for liquids and pastes

thermogravimetric analysis

Thermogravimetric analysis measures weight changes with temperature and time, and provides information about composition analysis and thermal stability. A complete picture of the amount, rates and composition of volatiles and decomposition products can be ascertained by combining these results with chromatography and/or spectroscopy techniques. Modulated Thermogravimetric analysis and high resolution analysis provide enhanced separation of component thermal decomposition profiles and associated kinetics.

applications

- rates of evaporation
- thermal decomposition
- thermal stability

thermomechanical analysis

Thermomechanical analysis measures the expansion and contraction of materials as a function of temperature. This technique is applicable to coatings, thin films, plastics and composites.

applications

- coefficient of linear thermal expansion
- glass transition temperature and other thermal transitions
- thermal dimensional stability
- residual stress due to processing
- softening and yield temperatures

mechanical testing

This area provides test data to characterize the mechanical integrity of molded resin castings and composites. The group performs many ASTM test procedures. Part of this laboratory is a well equipped sample preparation area, which includes diamond saws, sanders, surface grinders, and special support fixtures to accurately and precisely prepare the specimens for testing. The data are used to qualify materials for customer specifications, to help design materials for optimum end-use performance, or to evaluate the effects of exposure environments on mechanical integrity.

applications

- modulus (stiffness), strength, and elongation of a material
- fatigue information concerning the resilience of a material to cyclical loading is applicable to resin castings, composites, laminates, and sections of molded parts
- creep displacement of a sample as a function of time under a constant load
- lap shear testing to measure the adhesive and/or cohesive strength of a bonded laminate
- automated variable speed instrumental impact testing via High Rate Impact Tester (HRIT) with speeds as high as 20 miles per hour can be achieved; calculated values include the force and energy for crack initiation to the final breaking of the sample.
- heat deflection temperature (HDT) measurements provide distortion temperatures up to 200 °C
- Poisson's Ratio to determine the change in width per unit width relative to the change in length per unit length upon deformation is used for engineering design and characterizing the micromechanics of materials
- flash point testing
- refractive Index



separations and wet chemistry

Specializing in chromatographic techniques for trace analysis and classical wet chemical analysis.

classical wet chemistry

AS&T provides analytical testing using classical wet chemistry techniques that conform to procedures of the ASTM and the Society of the Plastics Industry. These procedures are applied to analyses of coating resins, plastics, adhesives and phenolics. They include Karl Fischer water determination, pH measurements and auto titration capabilities.

specific techniques

- Karl Fischer water analysis; both volumetric and coulometric with oven capability
- automated titrimetric analyses to colorimetric and potentiometric endpoints
- measurement of volatile organic compounds (VOCs)

gas chromatography

Gas chromatography (GC) is a universal separation technique applicable to complex mixtures. The technique uses differences in volatility and analyte-column interaction to achieve the desired separation.

GC is commonly used to analyze mixtures for identification and quantification. Various ancillary GC techniques, such as headspace/GC-MS, and other multidisciplinary techniques, are available to solve client problems. GC techniques are cost effective and encompass a wide range of analytical problems. Assay and trace-level analyses are achieved with minimal sample preparation. A wide variety of special detectors is available for quantification and specific identification of various classes of compounds.

applications

- quantification by direct injection or headspace/gas chromatography to determine composition of solvent blends and additives in manufactured products
- identification and quantification of residual monomers and solvents in product formulations, coated films and solid materials
- trace-level analysis of analytes for product contamination, environmental studies and waste analysis



gel permeation chromatography

Gel permeation chromatography (GPC), also known as size exclusion chromatography, is an analytical technique that determines the molecular weight distribution (MWD) of a polymer by virtue of its hydrodynamic volume. Molecular weight averages, such as M_n , M_w and polydispersivity, are also characterized by GPC. The size and shape of polymeric molecules determine physical and processing properties and are critical to producing a product with the desired performance characteristics. This technique applies to any solvent soluble polymeric material.

applications

- characterize MWDs of polymers (polyesters, polyurethanes, polyols, etc.)
- determine polymeric properties of incoming materials for processing suitability
- monitor effect of processing conditions on MWDs
- troubleshoot product failure
- collect polymer fractions for subsequent analysis
- absolute molecular weight

high-performance liquid chromatography

The strength of high-performance liquid chromatography (HPLC) lies in its versatility, sensitivity and selectivity. This chromatographic technique uses different mobile phases and columns to achieve the necessary separation of polar, thermally labile, non-volatile and other analytes that cannot be analyzed by gas chromatography. HPLC is non-destructive and analytes can be isolated for further spectroscopic analysis. Very low detection levels are achievable and multiple detectors can be used in series to monitor specific analytes. A wide variety of detectors are used including electrochemical, variable wavelength UV/visible, diode array and mass spectrometers. These are used to measure electrochemically active compounds, high molecular-weight analytes and non-UV absorbing materials.

applications

- quantification and identification of inhibitors and other electrochemically active components in products
- trace analysis of polymeric extracts using advanced LC-MS technology
- characterization of isocyanates, sulfonic acids, biocides and peroxides through the application of derivatization techniques, ion pairing, normal and reversed-phase HPLC

- isolation of specific analytes for further spectroscopic analysis and use of preparative liquid chromatography columns permitting simultaneous analysis and trapping

inductively coupled plasma-optical emission spectroscopy

Inductively coupled plasma-optical emission spectroscopy (ICP) is used for high-sensitivity elemental analysis. The spectrometer has the capability to measure from lithium through the periodic table (excluding the halogens) at mid-part-per-billion (ppb) levels. Semi-quantitative scans can provide elemental composition information at several hundred ppb levels for most elements. Samples can be analyzed in aqueous as well as organic matrices.

applications

- quantitative analysis of trace metals used as catalysts, cross-linking agents, etc.
- identification of low-level elemental contaminants

ion chromatography

Ion chromatography is a separation technique used to examine aqueous samples for anions, cations and trace levels of organic acids. Ion chromatography is similar to high-performance liquid chromatography in that it requires a liquid mobile phase and a column with a stationary phase. Ions that have a higher affinity for the stationary phase elute more slowly than ions that have a lower affinity for the stationary phase.

applications

- identification and quantification of ionic contaminants, such as formate, nitrate, chloride or hydrogen cyanide
- trace-level analysis of organic acids in aqueous process streams
- analysis for hydrogen chloride, sulfuric acid or sulfur dioxide and other organic and mineral acids in air
- polyester resins analysis for halogens or phosphorus components via conversion to halides and phosphate, respectively
- process waste analysis for mono- and di-valent cations such as ammonium, sodium, potassium, calcium and magnesium

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