

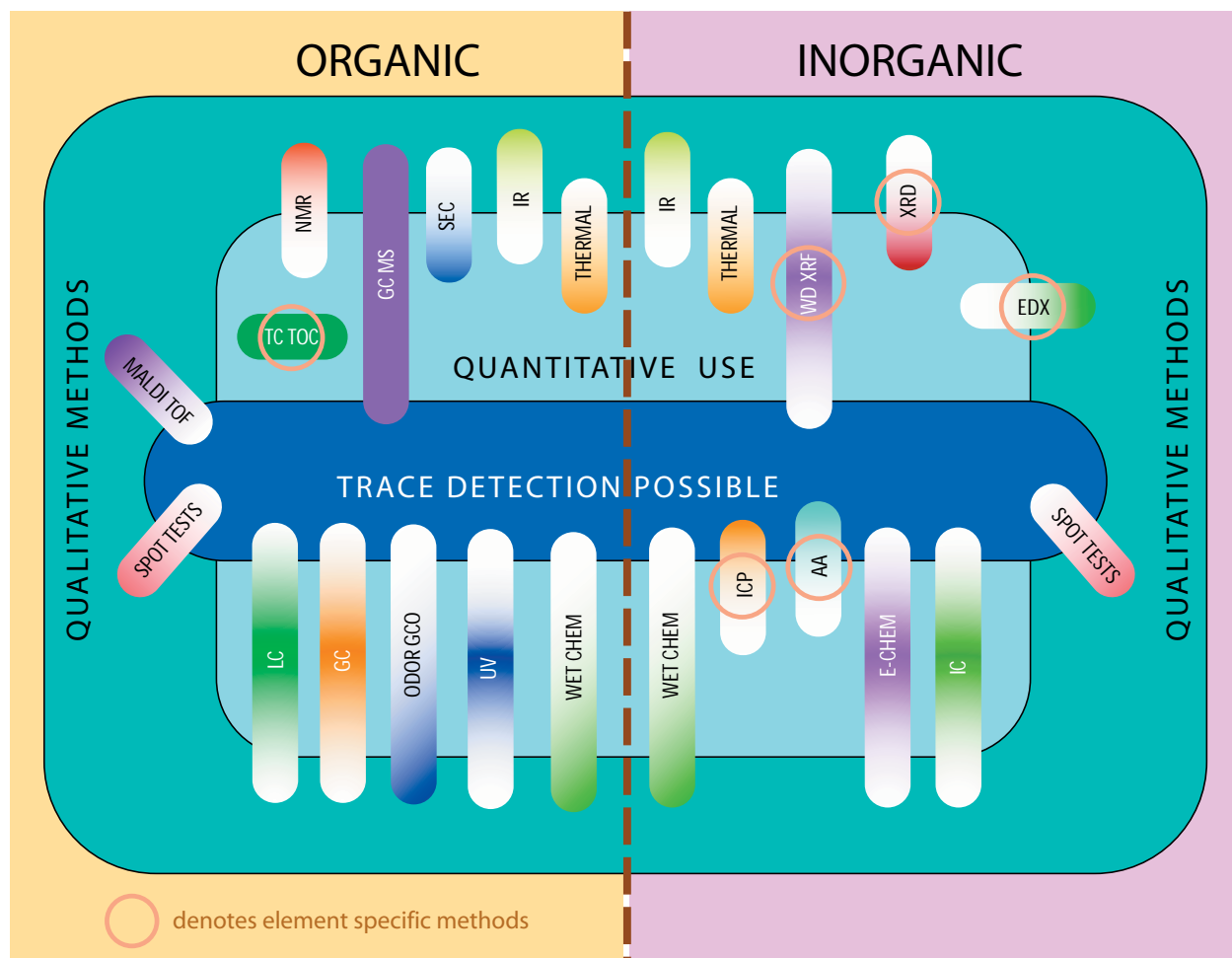
## CHEMICAL METHODS AT A GLANCE

The graphic below depicts many of the methods employed at Aspen Research. They are divided into inorganic and organic methods.

Further, they are depicted to reside in one or more of three categories: qualitative methods,

quantitative methods, and those which have potential for trace detection.

The method abbreviation appears in the area where the method is used most commonly at Aspen Research.



AA	Atomic Absorption	MS	Mass Spectrometry
AFM	Atomic Force Microscopy	NMR	Nuclear Magnetic Resonance Spectrometry
DMA	Dynamic Mechanical Analysis	ODOR GCO	Flavor/Odor/GCO/Sensory Analysis
DSC	Differential Scanning Calorimetry	SEC	Size Exclusion Chromatography
E-CHEM	Electrochemical Analysis	SEM	Scanning Electron Microscopy
EDX	Energy Dispersive X-Ray Analysis	TC TOC	Total Carbon Total Organic Carbon
GC	Gas Chromatography	TGA	Thermo Gravimetric Analysis
GC MS	Gas Chromatography/ Mass Spectrometry	TMA	Thermo Mechanical Analysis
IC	Ion Chromatography	UV	UV-Visible Spectroscopy/Colorimetry
ICP	Inductively Coupled Plasma Spectroscopy	WET CHEM	Chemical Methods, Inorganic
IR	Infrared Spectroscopy	WET CHEM	Chemical Methods, Organic
LC	Liquid Chromatography	WDXRF	X-Ray Fluorescence
MALDI TOF	Matrix Assisted Laser Desorption/Ionization Time of Flight Mass Spectrometry	XRD	X-Ray Diffraction



LC

## Liquid Chromatography (LC) + Liquid Chromatography/ Mass Spectrometry (LC/MS)

### What is measured?

The presence and amount of nonvolatile substances in mixtures based upon comparison to known standards or spectral interpretation.

### Chemical Analysis

- organic/some inorganic
- substance specific
- qualitative or quantitative
- trace

analytes	sensitivity	sample types/amount
Organic and many Inorganic compounds, components of mixtures	ppb to ppm or above.	soluble materials and liquids  typically gram quantities are sufficient

principle	example applications
<p>Liquid Chromatography separates components of samples by making use of the differential distribution of the various components between a carrier liquid (mobile phase) and the surface or immobile chemical placed upon the surface of a solid column packing (stationary phase).</p> <p>A component, which is preferentially soluble in the liquid mobile phase, will elute from the column quickly whereas an analyte, which interacts more strongly with the stationary phase, will retain for a longer time.</p> <p>Analytes are detected as they elute and their retention time (time in the column) is used as a measure of their identity and the intensity of the detector response can be correlated to the concentration of the analyte in the separated mixture by use of standard materials which also would be analyzed.</p> <p>A mass spectrometer combined with a liquid chromatograph (LC/MS) can provide detailed mass spectral data which can be interpreted to identify unknown compounds or to determine peak purities. LC/MS significantly</p>	<p>expands the effective analytical use of mass spectrometry to a much larger number of organic compounds. LC/MS data may be used to provide information about the molecular weight, structure, identity, and quantity of specific sample components.</p> <p>Detectors include:</p> <ul style="list-style-type: none"> <li>Ultraviolet detector for organics which have UV chromophores in their structure</li> <li>Refractive Index or evaporative light scattering detectors for general detection</li> <li>Electrochemical detector for chemically specific detection of electroactive components.</li> </ul>
	<p>trace quantitation of organic ingredients</p> <p>confirmation of ingredient levels</p> <p>fingerprinting of mixtures (complexity, initial estimates of amounts)</p> <p>isolation of components for other analyses</p> <p>trace monomer analyses</p> <p>additives</p>



LC/MS



**Impact**

## Physical Property Testing of Materials

### What is measured?

Material properties such as material strength, impact and puncture, resistance, flexibility, hardness, moisture transmission, visco-elastic properties, etc. are measured. These tests often are performed by standard protocols.

### Material Evaluation



**Contact Angle**

sample amount	sensitivity	sample types
sufficient to fashion suitable sample for the test to be performed (often described by ASTM or other protocol)	bulk properties are determined	all sorts of materials

principle	example applications
Select physical properties of a material are carefully measured under controlled environmental conditions and related to material performance.	Izod Impact Gardener Impact Contact Angle Moisture Vapor Transmission Microhardness



**Microhardness**



## Scanning Electron Microscopy - Energy Dispersive Spectroscopy (SEM EDS)

### What is measured?

Microscopic features are examined in detail. The presence and relative concentrations of specific atoms (atoms greater than atomic number 5) are determined.

### Material Properties

### Product Inspection

### Chemical Analysis

- qualitative and quantitative

analytes	amounts	sample amount
Elements above atomic number 5 are qualitatively determined. Relative quantitation is possible. Also, high magnification images are obtained.	Depends upon the element. Species in the percent range may be observed	Samples must be physically large enough to place in the instrument. Microscopic features and their contents may be imaged and analyzed.

principle
<p>The scanning electron microscope has variable pressure capabilities that uses a focused stream of electrons to image the sample.</p> <p>The fine beam continuously traverses the region under study to produce contour images by detecting the secondary electrons emanating from the specimen itself.</p> <p>A variable pressure SEM permits the</p>

example applications
<p>Failed component inspection (e.g. electrical contacts for flaws). Flaws can be imaged and classified as corrosion or contamination, etc.</p> <p>EDS (EDX) can help to identify the possible source of a foreign object or the identity of a possible corrodant.</p> <p>Examination of fractures and defects (The extreme depth of field of SEM imaging allows high magnification for the examination of fracture morphology)</p> <p>Chemical analysis of samples too small for other techniques. For example, a tiny chip from a piece of tooling is enough to identify the alloy.</p>



## X-Ray Diffraction (XRD)

### What is measured?

For crystalline materials, the types of crystal structures present and their abundance is assessed. This can serve to identify a substance or to study the nature of crystalline structures in a material.

### Material Properties

#### Chemical Analysis

- substance specific
- qualitative and quantitative

#### amounts

The degree of crystallinity of a sample must be sufficient to produce workable signal. Relative percent of crystalline components may be determined.

#### sample amount

Sub-milligram samples may sometimes be analyzed as may be analyzed as powders. Material samples may be sized to fit the window of the instrument.

#### sample types

Minerals and other crystalline or partially crystalline materials. This includes many synthetic polymers as well as natural polymers such as cellulose

#### principle

Monochromatic x-radiation is directed at the sample. The diffraction of the x-rays at various angles is measured with respect to the incident beam.

The relationship between the wavelength of the x-radiation and the angle(s) of diffraction is used to

determine the distance between each set of atomic planes of the crystal lattice(s) present.

This plane spacing is characteristic of the crystal type(s) in the material. The degree of crystallinity can be measured with appropriate reference materials.

#### example applications

XRD allows you to determine the amount of retained austenite in heat treated steels down to 1% levels

XRD can be used to determine plating (or coating) thickness

Particle size measurements can sometimes be done.

Some chemical analysis are best done by XRD: The percent chiolite ( $\text{Ma}_5\text{Al}_{13}\text{F}_{14}$ ) in cryolite ( $\text{Na}_3\text{AlF}_6$ ) is such an example

Frequently, in the determination of an unknown, chemical analysis can be supplemented by XRD which may allow the analyst to determine the exact compound present, including oxidation state

Distinguishing between different crystal structures in compounds with multiple structures. An example is differentiating the rutile form of  $\text{TiO}_2$  from the anatase crystal form.

# THE ELECTROMAGNETIC SPECTRUM

