WHITE PAPER

Selection Criteria for Analytical Technologies that Meet Polymer Manufacture and Recycling Testing Needs

Polymers used in manufacturing are primarily synthetic organic compounds, such as thermoplastics (e.g., polyethylene, polypropylene, polystyrene, polyvinylchloride, polyester, polyamide, polycarbonate, polyethylene terephthalate), elastomers (e.g., rubber, silicone rubber, cross-linked polyurethanes, neoprene, EVA), and duroplasts (e.g., polyurethane and various resins). Polymers are used in nearly every industry and for purposes as diverse as construction, isolation and insulation materials, medical devices and products, food packaging, consumer goods, and many more.

Every manufacturer that produces or uses polymers needs a comprehensive testing program to ensure the quality of their materials and processes. Some manufacturers prefer to have an in-house analytical workflow, others use contract labs to fill those needs, and many use a combination of the two. Some parts of a company's polymer testing program are driven by the processes they conduct, products they produce, and regulatory and/or end-use requirements for their operations. There are, however, many universal testing needs within the polymer industry.

Some of the drivers are:

- Research and development (R&D) efforts
- Manufacturing quality control (QC)
- End-use specifications
- End-product failure analysis
- Industry and regulatory standards
    - Industry-specific standards such as those for electronics, children's toys, and automotive components
    - Governmental regulating bodies such as those for environmental protection, pharmaceuticals, and food/beverages
    - General industry/manufacturing standards such as ASTM International and ISO
- Consumer and other end-user safety concerns
- Environmental safety concerns

The following sections discuss the testing needs at each step in the polymer production lifecycle, the analytical technologies that will meet those needs, and important considerations to help you select the instrumentation that will provide you with the actionable, reliable data you need. This deep dive is followed by a review of universal considerations for technology evaluation.

Download the companion **Polymer Buyers Guide** to use as a handy reference tool.

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POLYMER PRODUCTION LIFECYCLE

Raw material suppliers provide the chemical feedstocks—e.g., monomers, additives, fillers—that form the basis for polymer production.

Resin producers use combinations of raw materials to manufacture different "master batches" of polymer resins.

Polymer compounders create different plastic formulations by mixing and blending polymer resins, additives, and fillers into process-ready pellets and flakes.

Polymer converters use plastic pellets and flakes to produce an incredibly broad range of products—toys, electronics, automotive, construction, packaging, and many others.

Polymer recyclers reclaim recyclable plastics and return them to compounders and converters for reuse.

Read the companion white paper **Polymer Lifecycle Challenges for the Betterment of Society and the Environment** for a broader discussion of the polymers industry.

Testing Needs and Technology Selection

The primary categories of testing used in polymer production are polymer identification and characterization, additives identification and characterization, physical characterization, residuals analysis, contamination analysis, safety analysis, and evaluation for emissions and leachables.

Polymer Identification

The ability to accurately confirm the identity of a polymer is important at several points in the polymer production lifecycle:

- Polymer converters must confirm the identity of incoming materials before entering them into the final product manufacturing process.
- Polymer recyclers must confirm the identity of polymer recyclates before shipment to customers.
- R&D teams at compounding operations rely on accurate polymer identification for the successful design of new materials and processes, evaluating competitor products, failure analysis, and so forth.

RESEARCH & DEVELOPMENT (R&D) - GENERAL TECH CRITERIA

Considerations for R&D analyses throughout the polymers industry include:

- Performance
- Scalability
- Ability to hyphenate
- Sensitivity and resolution
- Experience of supplier

These things combine to allow greater innovation and uniqueness, which can give you and your company the edge in your field.

Molecular spectroscopy techniques are used to identify polymers. They do so by measuring the absorption/transmission of light and other types of radiation, or the energy emitted from the interactions of excited particles in the material. The absorption or collision energy patterns of the material provide a chemical "fingerprint" that identifies that specific polymer.

Digital polymer libraries have been developed that contain the chemical fingerprints of individual polymers. Such libraries are used to identify known polymers and detect unknown formulations. R&D teams can create their own polymer library or update an existing library as they develop new polymer formulations.

Your laboratory managers and scientists should confirm that the solutions you are considering have:

- The proven ability to generate high-quality data (sensitivity, accuracy, reproducibility
- A comprehensive polymer library with search functions and the ability to combine use with attenuated total reflectance (ATR) should they require it.
- Software that is easy to use and that includes important built-in features, such as cloud connectivity that will be helpful if they need to link up to a master lab.

FT-IR Polymer ID Analyzer - Spectrum Two instrument

MOLECULAR SPECTROSCOPY TECHNOLOGY CONSIDERATIONS

There are a number of important aspects that should be considered when selecting spectroscopy instruments and software. The spectroscopy solution you invest in should be easy to use and move around in a factory environment. It should have the flexibility to grow with your needs, such as being readily upgradeable to include imaging or microscopy hyphenation. Look for the availability of a wide range of plug-and-play accessories for any sample type.

An instrument that is easy to use in the factory environment is an important consideration for converters and recyclers when carrying out tests to confirm polymer identities upon arrival, which they can best do through IR Spectroscopy technology.

Polymer Characterization

Compounders, converters, and recyclers require a full understanding of the chemical makeup and physical characteristics of the polymers they produce or use. For these companies, polymer characterization is an important part of confirming the integrity of input materials and end products, evaluating potential new suppliers, end-to-end production QC, finding the cause of product failures, R&D efforts, and more.

QUALITY CONTROL (QC) - GENERAL TECH CRITERIA

Considerations for production QC analyses throughout the polymers industry include:

- Ease-of-use
- Robustness
- Automation
- Compliance conformity (ASTM, ISO)
- Size and portability
- Common software platform across multiple technologies
- Cloud-based software

The characterization of polymers includes deciphering numerous parameters such as:

- Molecular structure
- Molecular weight distribution
- Thermal characteristics
- Presence of additives and fillers
- Physical morphology
- Composition of mixtures

These diverse properties are evaluated using a number of different technologies including chromatography, spectroscopy, thermal analysis, microscopy, and X-ray diffraction.

Chromatography

Gas chromatography (GC) and liquid chromatography (LC) are routinely used to separate the polymer resins, additives, and fillers in a plastic formulation or mixture. GC is a very popular chromatography technique used to separate volatile compounds or substances that can be vaporized without decomposition. The technique is able to analyze small quantities of material, which means that it is applicable for analysis of residual monomers, initiators, catalysts, some additives and degradation products of polymers.

Very sensitive separation and detection is achieved by coupling GC with Mass Spectrometry for detection, resulting in Gas Chromatography-Mass Spectrometry (GC-MS). Proper sample preparation is necessary before GC/MS. Sensitive and selective techniques are used to separate and extract low-molecular-weight organic compounds from polymers.

LC techniques are very commonly used for molecular characterization of polymers. High Performance Liquid Chromatography (HPLC) is one such technique, used for separation and quantification of small molecules and polymers. HPLC is the method of choice for testing complex mixtures containing non-volatile and semi-volatile organic compounds such as polymer additives, active pharmaceutical ingredients (API), and impurities.

Seize Exclusion (SEC) or Gel Permeation Chromatography (GPC) provides insights into molecular weight distribution.

CHROMATOGRAPHY TECHNOLOGY CONSIDERATIONS

Important aspects to consider when investing in chromatography instruments and software include:

- Are a number of different detectors available?
- Does it have the ability to conduct headspace analysis?
- Is it readily amenable to diverse hyphenations?
- Easy-to-use software

Clarus® SQ 8 GC/MS

Spectroscopy

Spectroscopy is the study of the absorption and emission of light and other radiation by matter, as related to the dependence of these processes on the wavelength of the radiation. Spectroscopy is often used to identify the structure and chemical properties of molecules. Infrared spectroscopy is a classical spectroscopic method based on inducing molecular vibration in matter by infrared light. This yields an astounding amount of chemical and physical information in the form of an infrared spectrum. Common spectroscopic techniques used in polymer characterization include:

- Fourier Transform Infrared (FT-IR) spectroscopy identifies and quantifies polymers, additives, fillers, and their functional groups in pure materials and mixtures.
- Optical emission spectroscopy (OES) identifies and quantifies metals in polymers and polymer mixtures.
- UV-Vis spectroscopy quantifies polymers that absorb UV-VIS-NIR radiation to determine how much light passes through the material.

See the Spectroscopy Technology Considerations feature box.

Thermal Analysis

Thermal analysis techniques are used to characterize the thermal transitions, chemical reactions, and viscoelastic properties of polymers as a function of temperature, heating rate, deformation, and atmosphere. Commonly used thermal techniques used in polymer characterization include:

- Thermogravimetric analysis (TGA) evaluates the thermal stability and composition of a polymer.
- Differential scanning calorimetry (DSC) determines the heat capacity of the sample through the thermal transitions that occur (residual reactivity, evaporation of solvents, melting, crystallization, crystal transitions, and glass transition temperature).
- Dynamic mechanical analysis (DMA) determines the mechanical properties of a polymer as a function of temperature and frequency, simultaneously.
- Thermo-mechanical index analysis (TMA) evaluates a polymer's linear thermal expansion, anisotropy, softening temperatures, glass transitions, and dimensional changes.
- Rheology analysis determine a material's viscosity.

THERMAL TECHNOLOGY CONSIDERATIONS

Considerations when selecting thermal analysis technologies include:

- Does the instrument's heating and cooling rates meet your needs?
- What temperature ranges are available with DSC, TGA, DMA, or TMA?
- Is the performance addressing my needs?

HYPHENATION FOR MORE INFORMATION

Hyphenation refers to the coupling of two or more instruments to increase the power of analysis. Chromatography hyphenated with thermogravimetry and spectroscopy (IR-GC/MS, TG-IR-GC/MS) adds the ability to quantify the separated components. Commonly used applications of this type are gas or liquid chromatography hyphenated with mass spectroscopy (GC/MS, LC/MS).

Hyphenated TG-IR provides powerful insights by coupling a Thermogravimetric Analyzer with your Infrared Spectrometer for evolved gas analysis. Hyphenate further to TG-IR-GC/MS, and you have a formidable combination that provides separation, identification, and trace-level quantification in a single workflow. Using hyphenated workflows such as these provides more information in less time.

Learn more about hyphenated technology at the dedicated landing page.

Microscopy

When analyzing extremely small samples or areas of interest, it's often difficult to get the kind of data that enable better understanding of each sample's material properties. These microscopy methods provide information about material size, shape, structure, surface topography, crystallinity, some types of degradation, and other structural characteristics.

Several microscopy techniques are available such as optical, infrared (IR), and Raman microscopy, to name a few.

Optical (conventional) microscopy is the most commonly used micro analytical technique. It is used in biology, chemistry, material science and many other disciplines across science and technology. It is used in industrial applications for quality, failure and root cause analysis.

Sometimes characteristics are not visible with optical microscopy. In such cases, other microscopy technologies are used to obtain information about a material's physical morphology.

Hyphenating microscopy imaging with FT-IR spectroscopy is a standard technique in many industries. It is used to check the quality of raw materials and products, provide failure analyses such as the presence of impurities or contaminants, and identify unknowns.

Raman microscopy is a technique which allows fast, non-destructive chemical analysis of solids, powders, liquids, and gases. Raman spectroscopy is used in many varied fields, from fundamental research up to applied solutions. It provides detailed information about chemical structure, phase and polymorphy, crystallinity and molecular interactions.

MICROSCOPY TECHNOLOGY CONSIDERATIONS

There are a number of important aspects that should be considered when selecting microscopy instruments and software. The microscopy solution you invest in should be easy to use, robust and reliable. It should be versatile and enable you to run macro and micro measurements on the same platform and should have the flexibility to grow with your needs, such as being readily upgradeable to imaging or hyphenation.

Important aspects to consider:

- Do you need a conventional microscope or are you interested in chemical and physical information as well?
- Scalability and Flexibility
- Sensitivity and resolution
- Can the experiments run in automated mode?
- Is automated data evaluation possible

Spotlight 400 FT-IR Imaging System

XRD TECHNOLOGY CONSIDERATIONS

Two important considerations are space and warranty. XRD instruments come as benchtop and floor units. The benchtop units are compact, while the floor units require a lot of space. R&D teams typically use the robust floor XRD, so keep in mind the end-user needs when selecting XRD technology. The cost of ownership of XRD technology can be significant if adequate warranty coverage is lacking. For example, it can cost $100,000 or more to replace a failed XRD source unit. Make sure the vendor provides you with a full understanding of the scope of the warranty over the expected lifetime of the instrument.

X-ray Diffraction (XRD)

XRD is another useful technology that provides information on a material's crystalline, microcrystalline, or amorphous structure, crystal size and orientation, atomic arrangement, imperfections, and other structural parameters.

Additives Characterization

Additives provide useful characteristics to a polymer, such as stability, flexibility, processing ease, and other important attributes. Polymer materials are routinely analyzed for additives in order to:

- Help R&D teams in their formula development efforts
- Deformulate and compare competitor products
- Confirm additive raw material quality
- Confirm the compounded polymer conforms to design specifications
- Identify the cause of product failure

The technologies used to identify and quantify additives are:

- Spectroscopy for organic and inorganic additives
- XRF for inorganic additives

When characterization of the properties of additives is needed, the technologies used are:

- LC, GC and GC/MS
- Dilute solution viscosity testing
- Rheology testing

Additives characterization is conducted by compounders and convertors to ensure their own final product quality, functionality, and alignment with customer specifications. Compounders use these technologies to conduct QC checks throughout their own production processes. Convertors use these technologies to do mirror tests of polymer formulations upon arrival from the compounder.

Compounders and converters should consider ease-of-use, speed, and automation as well as service and maintenance to get the most from their chosen instruments over time.

At the R&D level, compounders need to evaluate the interactions of different polymer/additive mixtures, or deformulate polymers to identify their makeup and conduct comparative analyses. For this R&D work, the instrumentation you invest in needs to be sensitive and highly accurate, and you should consider their scalability including the ability to hyphenate for increased results insights. Sample preparation method is another important consideration since higher throughput leads to cost savings. The ability to add headspace analysis and use a variety of detectors are also important considerations.

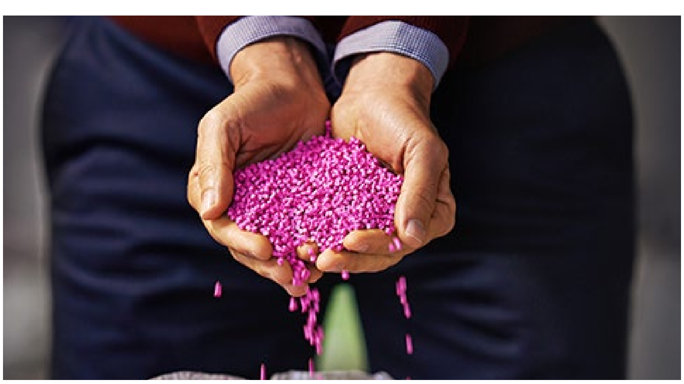
See the **Chromatography Technology Considerations** feature box and the **Thermal Technology Considerations** feature box.

Physical Analysis

Compounders and converters test the polymers they produce and use for physical characteristics. Physical testing includes a number of different thermal and mechanical analyses. Thermal analysis evaluates a polymer's response to various temperature conditions as a function of time and/or frequency. Mechanical analysis evaluates a polymer's response to stressors such as compression, stretching, and others.

These analyses play an important role in R&D design and testing of different polymer formulations and polymer-based products by:

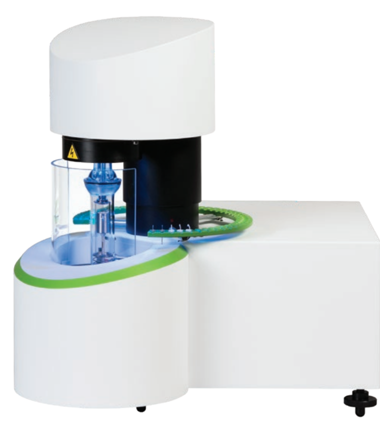
- Evaluating how a material will behave under the intended end-use physical conditions.
- Determining weathering and aging changes in a material's physical characteristics under extreme conditions and over the intended lifetime or the product.
- Evaluating the effects of different processing conditions on a test material.



Compounding R&D teams have the highest demand for thermal and mechanical analyses. They rely upon the physical test data to check polymer batches for strength, flexibility, and biodegradability, to determine usable lifetime, and to evaluate effects on the environment.

Physical testing is also important for product failure analysis, confirming an end-product meets customer specifications, and demonstrating compliance with industry or regulatory standards.Physical testing technologies best suited for thermo-mechanical analyses include:

- Differential scanning calorimetry (DSC) determines the heat capacity of the sample through thermal transitions such as residual reactivity, evaporation of solvents, melting, crystallization, crystal transitions, and glass transition temperature.
- Thermo-gravimetric analysis (TGA) evaluates the material's thermal stability and composition.
- Dynamic mechanical-thermal analysis (DMA/DMTA) determines the material's mechanical properties as a function of temperature and frequency, simultaneously.
- Thermo-mechanical index (TMI) evaluates linear thermal expansion, anisotropy, softening temperatures, glass transitions, and dimensional changes.
- Melt flow index (MFI) determines the ease of flow of plastic materials.
- Rheology measures the melt viscosity, creep, and stress relaxation response of a polymer as a function of time, temperature, and force.



TGA 8000

Other mechanical tests conducted using standard mechanical equipment include:

- Hardness
- Tensile strength
- Yield strength and adhesive strength
- Flexibility
- Compressibility
- Resistance to tearing, shearing, and impacts

Instrument sensitivity is vital for attaining R&D and QC criteria. You should also consider the speed, automation, and product servicing for these technologies.

See the **Thermal Technology Considerations** feature box.

Residuals Determination

Polymers and polymer-containing products sometimes contain residual levels of monomers, oligomers, or solvents. Some monomers and oligomers are known to promote polymer degradation even at residual levels, and residual solvents can lead to downstream polymer failure and/or human health concerns. This makes analysis for residuals an important QC component for resin producers, compounders, and recyclers.

Technologies used to test for the presence of residuals include:

- Spectroscopy hyphenated with microscopy imaging
- GC and GC/MS

R&D teams use these technologies to design process workflows and evaluate the results of mixing different polymers and additives. Residuals testing is also an important part of raw materials testing, processing QC, and final product testing.

See the **Spectroscopy Technology Considerations** feature box and the **Chromatography Technology Considerations** feature box.

Contamination Analysis

The presence of contamination in a polymer can lead to sub-optimal aesthetics or functionality, low product quality, or even product failure. Contaminants of interest in polymer products are typically chemicals, macroparticles, microparticles, and fibers. Contamination analysis can provide the opportunity for remedial action early in the production process. It also provides a final QC check before product shipment.

Testing materials for contamination analysis is important for:

- Raw material suppliers: before shipping products to customers
- Resin producers: before introducing materials into a production process, at checkpoints in the production process, and before shipping a resin master batch to customers

- Compounders: before introducing materials into a production process, at checkpoints in the production process, and before shipping the compounded polymers to customers
- Recyclers: at end of recycling process before shipping recyclates to customers

Technologies used to test for the presence of contaminants include:

- FT-IR spectroscopy hyphenated with microscopy imaging for particulates and fibers
- GC and GC/MS for organic contaminants.
- Inductively coupled plasma (ICP) hyphenated with optical emission spectroscopy (ICP-OES) for trace levels of metals and other elementals.
- ICP hyphenated with mass spectrometry (ICP-MS) for ultra-trace levels of metals and other elementals.
- X-ray fluorescence (XRF) for major and trace levels of elementals and other inorganics.

See the **Spectroscopy Technology Considerations** feature box and the **Chromatography Technology Considerations** feature box and **Microscopy Technology Considerations** feature box.

Safety Analysis

Safety-focused analyses are a crucial part of a company's regulatory compliance program. Polymer convertors must test their final products for the presence of chemicals that are of concern for human and/or environmental health. Some of these chemicals are used in polymer or end-product production, others can unintentionally enter the materials during production.

The primary chemicals of concern for polymer-containing end products and the technologies best suited for their analysis are:

- Toxic metals and other elementals
    - ICP-OES for trace-level analysis
    - ICP-MS for ultra-trace level analysis
- Polychlorinated biphenyls (PCB)
    - GC
    - GC/MS
- Polycyclic aromatic hydrocarbons (PAH)
    - GC
    - GC/MS
- Bisphenol A (BPA)
    - HPLC
- Phthalates
    - GC/MS
    - HPLC
- Perfluorinated and polyfluorinated alkyl substances (PFAS)
    - LC/MS

A crucial consideration when evaluating these technologies is the regulatory criteria that must be met. Make sure the instruments and software you are considering are able to meet or exceed the required quantitation limits.

See the **Spectroscopy Technology Considerations** feature box and the **Chromatography Technology Considerations** feature box.

SAFETY - GENERAL TECH CRITERIA

Considerations for polymer product safety analyses include:

- Sensitivity and resolution
- Ease-of-use
- Robustness
- Automation
- Compliance conformity (ASTM, ISO)
- Size and portability
- Common software platform across multiple technologies

Emissions Testing

Many organic solvents are used in polymer production processes. Volatile and semivolatile organic compounds (VOC and SVOC, respectively) in solvents have the potential to be released from polymers into the surrounding air, presenting a

potential inhalation hazard to humans. Examples of settings in which this can occur are vehicle interiors and hollow areas within children's toys. Convertors must ensure their products do not pose an emissions risk to end users.

GC/MS hyphenated workflows equipped for headspace analysis are most commonly used to identify and quantify emitted VOCs and SVOCs in these and similar settings. Products must be tested under normal use and extreme use conditions to determine the emission rates and concentrations.

A crucial consideration when evaluating these technologies is the regulatory criteria that must be met. Make sure the instruments and software you are considering are able to meet or exceed the quantitation limits.

See the **Chromatography Technology Considerations** feature box.

Electronic data integrity and computer system compliance is a growing focus of regulators in many industries. Ensure the instruments and technology you select will enhance your compliance efforts.

Leachables Testing

Convertors that make polymer-based packaging materials must ensure that no harmful chemicals leach from the materials. This includes any items the convertor adds to the polymer product such as dyes, label adhesives, inks, and so forth. This testing is especially important for packaging intended to hold pharmaceuticals, food, and beverages because leaching of chemicals from such packaging has the potential to impact the safety and efficacy of the pharmaceutical and the safety of the food or beverage.

Packaging materials and add-ons must be tested under normal use and extreme environmental exposure (transport, storage, etc.) conditions. The technologies best suited for these analyses are:

- HPLC and LC/MS for organics
- ICP-OES for trace levels of elementals
- ICP-MS for ultra-trace levels of elementals

A crucial consideration when evaluating these technologies is the regulatory criteria that must be met. Make sure the instruments and software you are considering are able to meet or exceed the quantitation limits.

See the **Chromatography Technology Considerations** feature box.

Other Analyses

In addition to the major categories of analytical technologies already discussed, there are a few other types of analyses that are used in polymer testing when needed.

Wet chemistry, also called applied chemistry, is the preparation of samples for various analyses. Wet chemistry techniques dissolve, filter, extract, and isolate components of a sample for further testing.

Titrations are quantitative chemical analysis techniques for specific analytes. Titration methods and parameters commonly used in polymer analyses include:

- Karl Fischer titration quantifies the water content of solid or liquid samples.
- Potentiometric titration enables quantification of analytes present in dark materials and solvent-based solutions.
- Halide titration quantifies halide ions in solutions (fluoride, chloride, bromide, and iodide).

Titrations such as these are used for quality control of raw materials and production processes, identification of contaminants including moisture, and deformulation studies by R&D teams.

Some polymers and polymer-containing products require specialized testing based on unique end-product use. For example:

- Electrical properties: dissipation, strength, resistivity
- Chemical resistance: determining physical property changes
- Flammability: small-scale flammability, room burn, open calorimeter
- Optical properties: color, yellowness, gloss, haze, refraction, birefringence
- Barrier properties: water vapor, oxygen, other gases

Universal Considerations for Technology Selection

In addition to the analysis-specific considerations already discussed, there are universal considerations that should be kept in mind when evaluating and selecting any analytical instrumentation. Beyond price points and specific test methods, careful attention should be given to hardware, software, and supplier capabilities.

Hardware considerations include user needs and instrument adaptability, such as:

- Range of analytical methods capabilities
- Sensitivity based on customer, industry, and/or regulatory requirements
- Throughput capacity
- Ability to integrate and hyphenate with other technologies
- Ability to upgrade and frequency of upgrades
- Ergonomics

Software considerations continue to expand beyond instrument control and results output. Also determine if the software has:

- Adaptability for multiple project profiles
- Ability to meet regulatory requirements for data integrity, validation, and migration
- Options for cloud-based communications
- Ability to integrate with machine learning and deep learning applications

Supplier considerations go well beyond their hardware and software offerings. Also determine and evaluate their:

- Areas of expertise
- Reputation within the industry
- Innovations track record
- Service and customer support offerings
- Breadth of portfolio
- Global reach

Always keep in mind that the support you receive when evaluating a technology can be an indicator of the level of support you will receive after your purchase.

GENERAL TECHNOLOGY CONSIDERATIONS

- Portfolio of consumables
- Sample preparation
- Finance options
- Variety of training options
- Expert scientific assistance
- Service and maintenance – speed, locality, and turnaround
- Contract options

Conclusion

The polymers industry encompasses the full lifecycle of polymer production and use. From raw materials through intermediate and end products to recycling, each company needs a comprehensive and targeted testing program to ensure the quality of their work and regulatory compliance.

Some polymer industry companies operate their own in-house testing. Others outsource testing to a contract laboratory for all or part of their analytical needs. In both scenarios, it is important that the company or laboratory carefully evaluate their (or their customers') needs and the ability of potential suppliers, instruments, and software to meet those needs.

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