Comprehensive Material Characterization for Polymers

Moving science forward
In the **R&D Laboratory** a new polymer is created by synthesizing small amounts of polymers or by blending existing raw materials to get the best performance for the final product. A wide range of test methods and tools is required to classify a new polymer and to allow accurate predictions of its future behavior during compounding and production. Precise tests done in the laboratory reduce costs at later stages.

The **Lab Scale Processing** is the second stage in the scale-up process. Kilograms vs. grams of the new precious polymer are produced. Material tests can be performed now to give detailed information on the processability and material strength of the new polymer.

During **Pilot Plant** and **Small Scale Production**, the developed and tested plastic material is transformed into the end product. To meet high-end-user requirements, a thorough and precise quality control process is mandatory.

With all your responsibilities, it’s something you don’t have time to think about on a daily basis: worrying about equipment performance and servicing. We created customer-centered service modules to cover every type of service challenge – from installation, inspection, validation, repairs, maintenance, and training.

No matter what stage of the polymer’s life cycle, eliminating waste and keeping production levels at peak capacity are crucial to ensuring profitability in today’s highly competitive environment. Our goal is to provide objective measurement and high quality testing to improve and speed the development and production of polymers.
To ensure your success, it is not enough for us to just sell an instrument. By establishing cooperative relationships with our customers, we ensure that our instruments and services are focused on your needs.

Our flexible, high value instruments enable measurements and small scale production for a variety of specific requirements – giving you high quality results in a very short time.

Not only do we know the capabilities and limitations of our instruments, but we also understand polymer-related processing parameters. Our product specialists have the years of experience necessary to interpret your test results and to help you with your applications.

Thermo Scientific material characterization solutions offer comprehensive, worldwide support and innovative instruments that span the complete life cycle of a polymer: from the development of a polymer in the laboratory to the pilot plant where small batches of test materials are compounded, to online quality control in production, to processing small quantities of special polymers or composites. In addition, we offer products and services to address the latest trends in plastics recycling.
Today the challenge is to develop inexpensive base materials with the properties of expensively engineered plastic. This is accomplished by blending inexpensive mass plastic material with other components and additives. The most difficult task is mixing and extruding several polymers that are immiscible by nature. Special additives have to be tested and evaluated to reduce the interfacial barriers between the different blends. The key to our customers’ success is using our instruments in the development phase to subsequently produce high-tech polymer products at a low cost.
The concept is reflected by the name: Modular Advanced Rheometer System. The HAAKE MARS rheometer platform features a modular design to enable it to be adapted quickly and flexibly to the requirements of various different applications or tests. All rheological measurements can be performed in CR (Controlled Rate) mode, CS (Controlled Stress) mode and CD (Controlled Deformation) mode, in rotation and in oscillation. A new normal force sensor also allows measurements of negative normal forces. Another innovation is the temperature control unit covering a range of -150 to 600°C.

The emphasis is placed on modularity and user-friendliness. This rheometer is easy to handle, while easy access for sample preparation and optional adjustments to the base stand allow individual additional modules to be connected. All the relevant components, including the measuring head and electronics systems, can be interchanged, even at a customer’s own location.

With a rheometer, the mechanical properties such as the viscosity and the dynamic moduli of polymer solutions, polymer melts as well as semi-solids can be measured as a function of stress, strain, time, frequency, temperature etc., not in only in shear but also in extensional flow.

Thermo Scientific rheometers can help to more efficiently characterize a polymer.

Thermo Scientific HAAKE MARS

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Thermo Scientific thermogravimetric analyzers (TGA)

Our Thermogravimetric analyzers measure the change in weight of a material as it is heated, cooled or kept at constant temperature as a function of time and temperature. These highly sensitive balances continuously measure the weight of the sample. The results can be related to thermal or oxidative stability, material composition, moisture content, reaction rate, oxidation, thermal decomposition and activation energy.

The TGAs accommodate the highest sample mass with loads up to 100 grams. When specially coupled with evolved gas analysis systems like FT-IR, MS and GC/MS, TGAs provide detailed information about the composition, additives used, and characteristics of all types of polymers.
Thermo Scientific HAAKE MiniLab
The HAAKE MiniLab micro-compounder combines compounding and viscosity tests for small volume samples (5 g or 7 cm³). It is based on proven, conical twin-screw technology with co- and counter-rotating screws. The pneumatic force feeder guarantees easy sample loading. An integrated bypass valve allows the sample to be recirculated in a slit capillary backflow channel or extruded for further tests. With fast cooling and a hinged barrel, the user can freeze the compound for further examination. The HAAKE MiniLab works as a stand-alone unit with data export or as a fully computer-controlled system.

Micro-Compounder compounding the smallest quantities for R&D

Thermo Scientific HAAKE MiniJet II
The HAAKE MiniJet II system allows you to optimize your development process and realize cost reduction opportunities:
- The production of tests specimens from as little as 5 g of material
- Test specimens can be produced from various material forms (powders, pellets, or melts)
- Specimen geometries offered from standard to unique, customized forms
- A control and design concept that provides simplistic handling with consistent, reproducible results

The Thermo Scientific HAAKE MiniJet system offers a complementary solution to product development investigations when coupled with the HAAKE MiniLab micro-compounder, Thermo Scientific HAAKE MARS, or HAAKE RheoStress 6000 rheometers.

R&D of New Polymers
Thermo Scientific
HAAKE RheoStress 6000

The HAAKE RheoStress 6000 rheometer is a highly sophisticated, research-grade instrument. Due to its wide measurement range and its modularity, this rheometer is equally well-suited for measuring highly viscous polymer melts and low viscous polymer solutions.

Rotational and oscillatory measurements can be run in CS, CR and CD mode. The HAAKE RheoStress 6000 is capable of performing all standard rheological tests including normal force measurements.

Lab Scale Processing

Once polymer testing is completed in the R&D laboratory, materials with promising properties are manufactured in small scale at the pilot plant. Newly developed polymers must pass the first test: Can the polymer be produced with reproducible quality at moderate cost? What problems can be expected when tons of raw materials are fed into an extruder instead of grams or kilograms?

The pilot plant delivers initial quantities of a new polymer to ensure the desired properties in respect to mechanical stability, strength and appearance. Grams or kilograms of a new polymer are essential to conduct standard tests that compare the product with existing materials. Creating product prototypes with the newly developed material further justifies future investments.
**Thermo Scientific HAAKE PolyLab OS System**

The HAAKE Rheodrive torque rheometer is the heart of the HAAKE PolyLab OS system. The rheometer includes all functionality to drive and control “intelligent” measuring sensors, i.e. mixers, extruders and compounders. The Windows®-based Thermo Scientific HAAKE PolySoft monitor software is easy to use and guarantees high test repeatability. Thermo Scientific HAAKE Rheomix OS mixers are used during batch mixing to examine process-relevant data such as melt behavior, influence of additives, heat and shear stability under shear load. Single and modular twin-screw extruders are used for process simulation such as compounding and extrusion of strands, profiles or films combined with rheological measurements.

**The HAAKE PolyLab OS is key for polymer testing**

**POSTExtrusion equipment for the Thermo Scientific HAAKE PolyLab OS System**

Specific downstream equipment and dies complete the HAAKE PolyLab OS system. A variety of POSTExtrusion equipment is available for a wide range of applications. Blown-film and chill roll take-off systems for films and sheets, including camera systems, are offered for online quality testing.

Water baths and pelletizers are available for strand extrusion lines. Calibration dies and conveyor belts allow pipe extrusion. Wire coating dies and take-off systems are available as well as a melt pump with catheter die for high precision products like medical tubes and catheters.

**Postextrusion devices maintain the shape of extruded material**

**Dies**

A variety of dies is available and can be adapted to all HAAKE PolyLab extruders. The dies range from simple strand dies to round and slit capillary dies that convert extruders into capillary rheometers. Special rheology dies, e.g. extensional dies, are designed to test the extensional flow behavior of polymer melts. Slit and blown film dies can be used in combination with camera systems for online testing to monitor polymer processability and quality. Special slit dies are also available to extrude sheets of PVC for further surface inspection.

**The most important part of extrusion is at the end of the process – the die**
Filter Test Software
According to the standard EN-13900-5, the Thermo Scientific HAAKE Filter Pressure Value test consists of the HAAKE PolyLab QC extruder, the melt pump and the filter die in which standard die filter packages can be inserted. A pressure control loop integrated within the extruder guarantees constant feeding conditions for an attached melt pump with filter die. This powerful and cost-effective filter test setup is a unique way to analyze the performance of polymer master batch mixtures. The formula generator allows the user to customize the evaluation mathematics of the filter test to individual requirements. Those settings are stored so that quality control (QC) test reports can be printed on demand. Routine QC data also can be evaluated and superimposed according to individual needs.

Customized mixer test data evaluation at the push of a button

Production is the environment where human beings are only needed in case of problems. Production runs 24 hours a day and is measured by high output and low waste. Testing machines are required to check the output of the production machines in order to get a constant quality. If something runs out of specifications due to a machine failure, the tests should quickly indicate a change in quality and ideally suggest a countermeasure to eliminate the problem. The MVR (Melt Volume Rate) is an accepted value to guarantee the quality of the polymer production. Production can also be understood as the manufacture of small quantities of special polymer compositions which are expensive, difficult to handle or needed in kilos only. This is required for pharmaceutical applications, for polymers with special material properties and for special applications. Thermo Scientific products enclose testing instruments and preproduction machines for all stages in the life of a polymer.
**Thermo Scientific HAAKE PolyLab QC Extruder System**

The most common methods in processing polymers are extrusion and injection molding. For testing quality and processability of those materials, the single-screw laboratory extruder – the Thermo Scientific HAAKE Rheomex 19/25 QC is a proven tool. With over 35 years of expertise, we’ve developed a wide variety of different screw designs that are guaranteed to provide the best performance. Special solutions such as chemical or wear resistance are also available to extrude harsh materials. To shape a polymer for further processes and tests, different dies can be adapted to the extruder such as rod dies and water bath for strand extrusion, sheet and ribbon dies in various dimensions to produce bands. Cast films and blown film dies and take offs complete the testing equipment. Besides the standardized filter test (EN 13900-5), comprehensive rheological studies can be conducted.

For continuous compounding and plasticising, the Thermo Scientific HAAKE CTW 100 QC is the ideal extruder in the HAAKE PolyLab QC system. This is a counter-rotating conical twin-screw compounder, with intermeshing screws, that will give well-defined residence times for faultless production of process-critical polymers.

**Thermo Scientific Compounders**

Thermo Scientific twin-screw compounders offer flexible compounding configurations for small batches for either pilot scale production or low volume manufacturing. The co-rotating twin screw compounders feature segmented screws, a modular, barrel construction with side feeding, liquid injection and venting ports that can be tailored to individual requirements.

The full-length opening barrel allows easy access to the screws for easy reconfiguration and cleaning. A touch-screen operator interface with data logging options and recipe storage guarantees repeatable results. Feeding and HAAKE POSTEXtrusion systems are controlled via the extruder.

**Thermo Scientific compounders are the most flexible, small scale, twin-screw compounding systems on the market**
Quality Control

Thermo Scientific HAAKE MeltFlow
Thermo Scientific HAAKE MeltFlow indexers comply with the ISO 1133, ASTM D 1238, ASTM D 3364, JIS K 7210 and referring standards. Based on a compact design, the HAAKE MeltFlow is the ideal table-top instrument for small labs and for frequent use in a quality control environment. Temperature stability and temperature uniformity (over the barrel length) are superior to these standards!

MVR and MFR measurements
Equipped with a digital displacement sensor the HAAKE MeltFlow measures the melt volume rate (MVR) automatically. The HAAKE Meltflow can also automatically measure the melt flow rate (MFR) with software-controlled balance.

Rheological measurements
The Thermo Scientific HAAKE MeltFlow software allows users to automatically measure apparent viscosity data (shear rate, viscosity, shear stress).

Recycling
Recycling is only newsworthy when raw materials are expensive or when politics draw attention to this field. Consumer awareness of the necessity for recycling by sorting plastics tubes, bottles and packaging materials – such as PE, PET, PS, etc. – is a challenge that requires proper product coding, time and education. The basic concept of recycling is to feed used plastic into the supply chain for manufacturing new materials with less costs and without compromises in material specifications. These expectations have to be supported by objective measurements and proven in small-scale recycling and re-production trials.

We can help you adapt existing machines to meet new recycling requirements in respect to stability, sturdiness and sensitivity. With our instruments, you can detect even small degradations caused by reworking polymers. Molecular weight and molecular weight distribution, together with the traditional MVR value, are the grades of quality during R&D.
Thermo Scientific HAAKE PCR

The HAAKE PCR-620 is a twin-pump bypass rheometer that provides on-line rheological measurements of melt index and viscosity in polymer processing. With more than 200 successful installations of the HAAKE PCR-620 and its predecessors around the world, we are a leader in advanced process control technology for the polymer processing industry.

The HAAKE PCR-630 patented 3-pump design is especially suited for fractional and low melt index polymers, as well as applications sensitive to residence time.

The HAAKE PCR-630 is the fastest bypass rheometer design available and provides near real-time data with its slit die design and three independently driven and controlled melt pumps.

Thermo Scientific HAAKE PolyLab QC Mixer

A typical mixer test is run at a defined rotor speed. The material’s response to the shear is recorded as torque and displayed versus time.

As a material’s properties are very sensitive to temperature, the mixer chamber is separated into different sections. These are individually temperature-controlled by the Thermo Scientific HAAKE PolyLab QC.
When you look at service options, you often find manufacturers that enforce inflexible options in their support plans and demand you pay for services you don’t use.

Imagine the peace of mind of getting cost-effective installation, servicing, and training handled by the trusted experts in material characterization.

We created customer-centered service modules to cover every type of service challenge – from installation, inspection, validation, repairs, maintenance, and training. We also provide you with enormous flexibility.
Select only the service modules you want. Add modules at the time of equipment purchase, during warranty, or after. Combine modules. Or create a customized service plan that matches your unique requirements. We can offer comprehensive consultation to help choose the right options.

And if you require services not described here, please call us. We’ll develop customized solutions for you.

Visit www.thermo.com/mc_service for more detailed information. And then phone your Thermo Fisher Scientific sales representative to arrange for your free service assessment.