HAAKE PolyLab – The Versatile, Application-Oriented Torque Rheometer

Polymers in Practical Application

An ever-increasing complexity of polymers and polymer blends is leading to a growing number of specific demands with regards to measurement technology.

In addition, measuring devices need to meet different sets of requirements for each department of an organization.

Product Development ...

... focuses on research of various additives and formulations, i.e. special emphasis is given to small-scale twin screw extruders (16 mm).

Process Engineering ...

... has the task of mastering new applications. This department therefore needs to carry out tests under field conditions in order to gain a better understanding of process characteristics and process relationships.

Quality Control ...

requires regular testing throughout all processing stages. For this reason, most Quality Control applications include HAAKE Postex units.

Complex needs

Torque rheometers cater for all of these requirements in real-world laboratory testing. Torque rheometers need to meet exact specifications with regard to accuracy, handling and reproducibility of test results, as well as usability and process orientation. At the same time, today’s markets call for application-oriented and thus extremely versatile system solutions.

HAAKE PolyLab systems are designed with the demands for sophisticated products in mind. The HAAKE Rheocord family of products offers significant enhancements, integrating innovative new functionalities with tried and tested know-how.

Amalgamation of experience and innovation

The HAAKE PolyLab System consists of a HAAKE Rheocord torque rheometer including all drive and control functions for various measuring sensors.

A broad variety of measuring sensors, such as mixer torque rheometers and measuring extruders with application-oriented control mechanisms, enable the acquisition of various types of measurement data. Data thus collected is transferred to the HAAKE Rheocord torque rheometer for analysis. This way, even large amounts of information can be securely processed, enabling trouble-free and quick implementation of new applications.

HAAKE PolyLab Systems are extremely flexible and measuring sets can be tailored to the specific requirements of a given application.
Born from long experience

Thermo Electron Corporation was among the pioneers in the field of rheological measuring engineering. Expertise gained over many years, in combination with the wide-ranging knowledge and feedback from our customer base, has lead to the development of HAAKE PolyLab into a system that caters even to the most highly specialized needs.

Thermo offers to be your competent partner for rheology, quality assurance and production, providing an extensive range of products and services that will assist you in remaining competitive in your particular market:

- development of efficient concepts in cooperation with our specialists
- support during installation and commissioning
- consulting services
- software updates
- global service network

Safety and convenience

The HAAKE PolyLab system is based on the Plug&Play concept.

Mobile mixer torque rheometer, extruder and compounding units are designed for easy and safe handling in the laboratory. Once a measuring sensor has been docked on to the base unit, the control module automatically detects its configuration, and all unit-specific limits and characteristic values are set accordingly. The measuring ranges of sensors are automatically adopted for measuring and calibration purposes as are the measuring set visualization and GUI settings.

Manual pre-testing and process optimization can be remotely controlled for more flexibility, giving operators the opportunity to control the process from where they have the best vantage point.
HAAKE Rheocord – The Base Unit

HAAKE Rheocord - one base unit for all applications

The HAAKE Rheocord base unit contains all elements of a torque rheometer that either drive the measuring sensors or detect the resulting torque. Built-in interfaces enable communication with:
- a control and analysis module
- a remote control unit
- measuring sensors
- external devices
- a local area network

Due the flexibility of the measuring bus technology, a single unit satisfies all needs – both of current and future applications.

The Technology

Measuring sensors are driven by a state-of-the-art power unit, enabling operation at the optimum bias point of the drive unit at all times. This leads to load characteristics that can compensate even peak demands under extreme operating conditions, e.g. in high torque/high speed conditions.

Torque measurement is carried out via a measuring cell with integrated amplifier that has been custom-designed for the HAAKE Rheocord, thus providing the highest level of measuring accuracy and tolerance to interference for your application. The measuring accuracy of the unit is sufficient for all applications.

Additionally, a number of easy-to-install, interchangeable, coded torque sensors provide extra high resolution for application threshold ranges, should this be required. A real-time operating system with the look and feel of a standard Microsoft Windows GUI provides functions for drive unit control, measuring data bus control and data transfer.

Remote control

In addition to the field bus and PC interfaces, the standard version also features three RS-232 and two analog I/O ports for external devices or network communications.
HAKE PolyLab System - Comprehensive System Safety

The notion of state-of-the-art “system safety” comprises protective measures for operators, hardware and data. This concept has been consistently implemented into all HAAKE Rheocord control modules and is ensured by the following means:

- full enclosure of all rotating parts
- a safety cut-out engages if rotating parts are exposed
- internal motor temperature monitoring with advance warning and auto-stop function
- torque control for drive unit and measuring sensors
- real-time operating system ensures data integrity
- real-time fault handling, i.e.:
  - system ensures immediate response and generates alarms
  - alarm messages are displayed instantly in the foreground – even during measuring cycles.
- permanently available online data buffer for measuring data.
- user-defined start conditions that guarantee:
  - system safety, e.g. ensure adherence to temperature start conditions
  - high degree of reproducibility of results
  - automatic testing cycles
- CONTROL terminal control for peripheral devices:
  - easy-to-use
  - short preparation times
- No predetermined mechanical breaking points (shear pins) have been incorporated into the design in order to ensure proper operation of the system for application threshold ranges.

Convenience and ease of use...

... begin with instrument layout and end with device cleaning.

The HAAKE Rheocord offers many control functions that make laboratory work more efficient. Once a measuring sensor is docked, the control module automatically recognizes and dynamically indicates its hardware configuration on screen. A user-friendly menu structure guides operators through the configuration process for each measuring cycle. A self-optimizing temperature control feature ensures that process conditions are achieved more quickly. The optimum operating point for subsequent switching to programmed operation can be set “on the spot” using a remote control.

Predefined start conditions ensure that no limit values are exceeded. Once these conditions are fulfilled, the measuring cycle is started automatically. After completion of the measuring program, the unit automatically switches into a user-defined status. When the automatic storage procedure has been completed, cleaning can commence.

Increased work efficiency through parallel analysis or preparation of test series

You can evaluate and document measuring data for previous test cycles even while the unit is operating. Alternatively, you might prepare a second measuring sensor for a follow-up measuring cycle.

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### Technical data

<table>
<thead>
<tr>
<th>Parameter</th>
<th>RC 300p</th>
<th>RC 400p</th>
<th>RC 500p</th>
</tr>
</thead>
<tbody>
<tr>
<td>Motor power</td>
<td>4.0 kW</td>
<td>9.2 kW</td>
<td>16.0 kW</td>
</tr>
<tr>
<td>Torque</td>
<td>300 Nm</td>
<td>400 Nm</td>
<td>400 Nm</td>
</tr>
<tr>
<td>Max. speed</td>
<td>250 rpm</td>
<td>300 rpm</td>
<td>550 rpm</td>
</tr>
</tbody>
</table>

Torque load cells: interchangeable

Remote control: standard

Temperatures integrated into sensors

Ports:
- 2 x analog I/O
- 3 x RS232

Mains supply:
- 3 x 400/230V 50/60 Hz, N, PE, 32 A

Dimensions (W/H/D): 42/125/95 cm

Weight: 240 kg net

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### Safety Specifications

According to the EU laboratory instrument directive 98/37 EU

Safety warnings and cut-offs for:
- Motor
- Torque
- Control temperature
- Melt temperature
- Melt pressure
- Speed
HAAKE Rheomix/HAAKE Rheomex – The Application Specialists

Thermo Haake offers a comprehensive program of measuring extruders and mixer torque rheometers which can be attached to the Rheocord base unit.

The actual measuring sensor to be used depends on the task at hand. Typical applications in the polymer, food products, pharmaceutical and ceramics industry include:

- testing of processing behavior
- evaluation of processibility
- formulation research
- compounding
- small-batch production of profiles and films for additional tests
- rheological testing

All measuring sensors are equipped with essential measuring devices and automatic control technology for their designated application! Another advantage of the system is an automated data management that use a fast field bus for communications between the HAAKE Rheocord base unit and its measuring sensors. All measuring sensors are coded, thus ensuring trouble-free interaction and communications between all system components. The measuring sensors will notify the base unit – and thus the user – automatically of its presence and current configuration, i.e. number and location of the temperature control zones and measuring ports, unit-specific limit values and possible sensor options.

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All pressure transducers connected to the system are also coded. A new concept simplifies handling in the laboratory and ensures safe operation:

- mobile measuring sensors avoid the necessity of handling heavy and hot components
- integrated heating and cooling circuits reduce the number of plug-in connections and thus the chance of a mix-up
- control functions for multiple peripheral devices, such as feeders and post-extrusion units, lead to improved reproducibility and documentation of results.

HAAKE Rheomix - mobile measuring sensors

Mixer torque rheometers are ideal for testing processes such as mixing, compounding or plastifying of polymers, chemicals, ceramics or pigments in a production-oriented environment. HAAKE PolyLab systems with HAAKE Rheomix units can generate so-called mixer rheograms, which are standard tools for quality control. Production-related information can be derived from these files including:

- interrelation between dynamic viscosity and shear load
- melt and degradation behavior in the extruder
- the influence of additives

Mixers for various standard applications are available for testing of thermoplastics, thermosetting plastics and elastomers. An electric temperature control facilitates testing of high-temperature plastics up to 500°C. Additional features include:

- interchangeable rotors
- three-section mixer chamber
- forced-air cooling

The large volume chamber version of the mixer torque rheometer is suited for the production of small batches for additional testing. Thermo mixer torque rheometers are available with special testing equipment for products with non-standard requirements:

- liquid temperature control
- anti-wear surface treatment against abrasion or chemically aggressive materials
- conical mixer chamber for material / thermoset curing
- pneumatic feeder units
- electric conductivity sensor
- gas volume for quantification of propellants during the plastification phase

Technical Specifications

<table>
<thead>
<tr>
<th>Model</th>
<th>Description</th>
<th>Temperature Control</th>
<th>Volume, ccm</th>
</tr>
</thead>
<tbody>
<tr>
<td>R600</td>
<td>Electric temperature control up to 450°C for: thermoplastics, elastomers, thermosetting plastics</td>
<td>70 - 100 ccm</td>
<td></td>
</tr>
<tr>
<td>R610</td>
<td>Liquid temperature control up to 150°C for: food products, ceramics</td>
<td>70 - 100 ccm</td>
<td></td>
</tr>
<tr>
<td>R3000</td>
<td>Like R600 except for: Volumes: 300 - 540 ccm</td>
<td></td>
<td></td>
</tr>
<tr>
<td>R3010</td>
<td>Like R610 except for: Volumes: 300 - 540 ccm</td>
<td></td>
<td></td>
</tr>
<tr>
<td>R540</td>
<td>Electrical temperature control up to 450°C for: thermosetting plastics</td>
<td>70 ccm</td>
<td></td>
</tr>
</tbody>
</table>
HAAKE Rheomex – Specialized Measuring Extruders

Measuring extruders of the HAAKE Rheomix family enable researchers to analyze rheological parameters and process characteristics as well as the production of samples for additional test or small batch production. For this purpose, extruders can be equipped with transducers that measure melt temperature, pressure and process parameters along the extruder barrel as well as upstream measuring and profile dies.

All HAAKE post-extrusion systems ensure a defined conveying of the extruded material beyond the die. Both single screw extruders and twin screw extruders are available.

Typical applications include:
- extrusion of PVC compounds for various profiles or using PVC dies designed by means of FEM simulation
- sheet blowing, e.g. for LDPE or HDPE films
- compounding and production of master batches
- metered feeding of additives and evaporation of volatile constituents
- reactive extrusion

Single screw extruders

Many different screw geometries are available for custom configuration of the extruder to the processing application at hand, e.g.:
- standard screw screws with a 1:1, 2:1, 3:1, 4:1, or 5:1 compression ratio
- venting and mixing screws
- wear-reduced screws

Single and twin screw extruders

Various feeder devices (volumetric and gravimetric) ensure a trouble-free handling of pourable, powdery, sticky and paste-like materials.

Single screw extruder

<table>
<thead>
<tr>
<th>Model</th>
<th>Application</th>
</tr>
</thead>
<tbody>
<tr>
<td>R102p</td>
<td>for thermosetting plastics</td>
</tr>
<tr>
<td></td>
<td>D = 19.05 mm, L/D = 10</td>
</tr>
<tr>
<td></td>
<td>for curing materials</td>
</tr>
<tr>
<td>R104p</td>
<td>for rubber and elastomers</td>
</tr>
<tr>
<td></td>
<td>D = 19.05 mm, L/D = 10</td>
</tr>
<tr>
<td></td>
<td>with roll-feeder for elastomer rolling sheets</td>
</tr>
<tr>
<td>R202p</td>
<td>for thermoplastics and ceramics-compounds</td>
</tr>
<tr>
<td></td>
<td>D = 19.05 mm, L/D = 20</td>
</tr>
<tr>
<td>R203p</td>
<td>with roll-feeder for elastomer rolling sheets</td>
</tr>
<tr>
<td></td>
<td>D = 19.05 mm, L/D = 20</td>
</tr>
<tr>
<td>R252p</td>
<td>for thermoplastics</td>
</tr>
<tr>
<td></td>
<td>D = 19.05 mm, L/D = 25</td>
</tr>
<tr>
<td></td>
<td>Evaporation: optional</td>
</tr>
<tr>
<td>R332p</td>
<td>for thermoplastics</td>
</tr>
<tr>
<td></td>
<td>D = 19.05 mm, L/D = 33</td>
</tr>
</tbody>
</table>

Conical / counterrotating twin screw extruder

<table>
<thead>
<tr>
<th>Model</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>CTW100p</td>
<td>for conical twin screw, PVC dry blends, thermoplastics</td>
</tr>
<tr>
<td></td>
<td>wood compounds</td>
</tr>
<tr>
<td></td>
<td>D = 31.8/20 mm, L = 300 mm</td>
</tr>
<tr>
<td></td>
<td>for cont. compounding</td>
</tr>
</tbody>
</table>

Parallel / corotating twin screw compounders

<table>
<thead>
<tr>
<th>Model</th>
<th>Diameter</th>
<th>Length Ratio</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>PTW16p</td>
<td>D = 16 mm</td>
<td>L/D = 15...40</td>
<td>for cont. compounding</td>
</tr>
<tr>
<td>PTW24p</td>
<td>D = 24 mm</td>
<td>L/D = 15...40</td>
<td>for cont. compounding</td>
</tr>
<tr>
<td>PTW25p</td>
<td>D = 25 mm</td>
<td>L/D = 16...52</td>
<td>for cont. compounding</td>
</tr>
</tbody>
</table>
Dies & HAAKE Post-extrusion Units for Extrusion Processing

**Production-oriented testing**

Additional equipment used for the testing of rheological characteristics and the processibility of polymers as well as for the lab-based production of test samples includes require dies and post-extrusion units.

Dies define the geometry of profiles, sheets/films and rheological measurements.

HAAKE post-extrusion units – or HAAKE Postex systems for short – enable a defined downstream transport of the extruded material and preparation for additional analytical testing. The design characteristics of dies and HAAKE Postex units differ only slightly from their production-scale counterparts. This guarantees that all tests are performed under conditions similar to those in the actual production environment. Integrated measuring technology extends the number of application possibilities in the laboratory.

Various dies are available for extruders of the HAAKE Rheomex family of products including:
- blown film
- flat sheets
- pellets
- pipes
- fibers
- coated wires
- screen life tests

Blown Film Die And Cooling Ring

Catheter Die
Combined with capillary dies, the HAAKE PolyLab system can be transformed into an efficient extrusion capillary rheometer. Rheological parameters such as shear stress and viscosity functions can be determined this way. This type of data is of great importance for the design of injection molds.

HAAKE X-dies is used to measure of the extension rate / strain viscosity. Rod and slit capillary dies provide coverage for the entire range of shear rates of the various processes used in the polymer industry.
The Rheometry Application Examples

Thermo can supply a range of measuring systems for the rheological testing of your polymers:

- a HAAKE PolyLab system in combination with a HAAKE Rheomex measuring extruder and capillary dies as an extrusion capillary rheometer
- HAAKE PolyLab system in combination with a HAAKE Rheomix mixer torque rheometer for relative measurements of rheological properties
- a HAAKE RheoStress unit equipped with a HAAKE RotoVisco rotation rheometer for plate-plate or plate-cone measuring geometries

**Example 1: Determination of a PE reference curve**

A reference curve illustrates the viscosity behavior of a polymer over several decades of shear rate. The diagram in this example shows the comparability of two entirely different measuring methods - capillary measurement with an extruder and oscillation measurement in a controlled stress rheometer - for a LLD-PE melt.

The diagram in Fig. 1 shows the viscosity of the capillary measurement and the complex viscosity of the oscillation measurement against the shear rate. Capillary and oscillation measurement curves clearly overlap in the transitional area of structural viscosity. This indicates a high correlation of individual measuring segments and the COX-MERZ rule can thus be confirmed.

**Viscosity curve of PE-LLD at 220 °C measuring temperature**

![Viscosity curve graph](image)

Fig. 1
Example 2:
A differentiation of compounds

Mixer torque rheometers are often used for the differentiation of compounds. For instance, new additives may affect the viscosity of your compounds and thus the power consumption of your production plant and lead to processing and manufacturing problems. Fig. 2 illustrates the torque curve and the energy input for two similar compounds with different pigments. The differences in the diagram are clearly visible. Measurements of this type are often used to quickly solve problems such as selecting the best supplier and as such can help to avoid costly production testing.

Example 3:
Viscosity/Elasticity

The viscosity behavior of the melt greatly influences the processing characteristics of a material. For instance, the viscosity determines whether a mold is completely filled up during the injection molding process while elasticity influences the geometry of your finished product. Two different elastomers with similar flow characteristics during processing (i.e. similar viscosity) can result in completely different product cross sections. An analysis of these materials may show that the viscosity of both elastomers decreases to virtually the same level with increasing load while the impact of elasticity clearly increases.

Examples from our technical reports:

- Examination of the Plastifying and Degradation Behavior in PVC
- Testing the Flow Characteristics of Glass Fiber Reinforced TPU
- Characterization of Master Batches Using Screen Life Testing
- Flow and Cross-Linking Behavior of Cross-Linking Polyethylenes (XLPE)
- Lab scale extruder for pharmaceutical development (LR 47)
- Differentiation of PVC Dry Blend Batches Using an Extruder Sensor
- Correlation of Measuring Test Results for Production Problems in Elastomer Processing
- Pigment Differentiation in Master Batches