High-throughput rheometry: Anton Paar's fully automated rheometer product portfolio

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HTR 3000 - Anton Paar's Fully Automated benchtop Rheometer for low viscous samples.

Shortened product development cycles, increased quality control requirements and high sample throughput in synthesis, compounding, and formulation are leading to constantly increasing demand for rheological measurements.

Many of these fully automated benchtop rheometer solutions have been successfully utilised for measurements of e.g. paints and coatings, personal care products, agricultural chemicals, and dairy products.

Fully automated and in operation 24/7, the HTR 3000 is the optimal high-throughput product for cone and bob rheological measurements. The cleaning of the upper measurement geometry ensures continuous measurements of up to 54 samples, ensuring a high walkaway time. The result: no downtime and maximum productivity. Automated measurements reduce the workload of lab staff and allow them to do more important things.

Anton Paar's HTR 3000, the fully automated benchtop rheometer, performs up to 54 continuous rheological measurements without intervention. This high-throughput rheometer product is capable of processing up to 250 samples per day with Anton Paar's MCR 102e or MCR 302e rheometers. It's ideal for measuring all natural flowable samples with a concentric cylinder CC10 – CC27, vane geometries and spindles. Automated measurements guarantee highly accurate, reproducible results while lab employees can focus on more important tasks and the small benchtop size saves you much-needed lab space. And since you're working with one company for your whole measuring system, you can rely on seamless compatibility of our instruments and automated implementation.

The HTR 3000 is a product platform where the standard configuration covers most industry needs. A range of feature upgrades with standardised options allow individual configurations for individual needs. (e.g., automotive, paint, food, and personal care industries). Customised adaptions are available upon request.

Figure 1: High-throughput rheometer for concentric cylinder geometries with 3-axis handling system and the optional bench.

Figure 1: High-throughput rheometer for concentric cylinder geometries with 3-axis handling system and the optional bench. shows a benchtop automation system, which includes a rheometer with a temperature chamber for concentric cylinders, a three-axis handling system with a cup gripper, a cleaning unit for the upper measurement geometry with hot- and/or cold-water brushes and drying with pressured air, and sample racks for up to 54 samples. There's also the option for a code-reader and handheld scanner, a tempered sample rack, a pre-tempering unit, and pH-measuring station with cleaning unit to be added. Instead of water cleaning of the measuring geometry, an advanced cleaning unit is available that uses cleaning detergents.

Measuring bobs with various diameters, spindles or stirrers can be employed. A complete process and data integration into the customer's data network are possible, this ensures full traceability and transparency of each step of operation.

Figure 2: Inside the HTR 3000, storage racks on the left, automated cleaning on the right, MCR 302e in the back.

In a typical workflow for automatic operation, the cup gripper picks a cup from the rack and holds it over a scanner with an integrated code reader. Now, the automated benchtop rheometer has all the sample information ready and can either place the cup into the takeover unit, which finally positions the cup in the rheometer, or place it in a pre-tempering chamber. As soon as the previous measurement is finished and the rheometer is ready for the next sample, the cup is retrieved from the pre-tempering station and inserted in the temperature control unit of the rheometer where it is firmly fixed. As soon as the cup is in position, the measuring head of the rheometer moves down and brings the measuring bob into the measuring position and a predefined rheological measuring profile is started. The test routine can include any measurement the rheometer can perform, such as pre-shear, hold time, rotational measurement, oscillatory measurement, or a complex combination of different measurements. If desired, a different test routine can be assigned to each cup. When the test routine is finished, the lifting motor of the rheometer moves the measuring head with the measuring bob upwards. The cup gripper then removes the old sample and returns it to the rack or to a pH station for further analysis.

The intuitive operating procedure means measurements can be started in just a few steps. Direct transfer of measurement results to the network storage or LIMS saves time and eliminates manual input errors. Errors can also be prevented during cup placement with the option for a unique data matrix code labeled on the bottom of the cups, a handheld scanner, and the integrated code reader cross checking if the sample is in the right spot with the correct template.

After rheological measurements, the HTR 3000 is able to perform a pH measurement with automated pH probe cleaning, and reference measurement performance checks.

A cleaning station is brought in, see *Figure 2*: Inside the HTR 3000, storage racks on the left, automated cleaning on the right, MCR 302e in the back., the bob is rotated and cleaned with brushes, and cold, hot or a combination of cold and hot water in a predefined manner. The cleaning station is decoupled from the rheometer itself, to rule out any influence during the measurement. After cleaning, the bob is dried with compressed air and the cleaning station is removed from the rheometer. For samples that are difficult to clean, a cleaning agent pump can be added to the cleaning unit to ensure effective cleaning of the upper measurement geometry. Now everything is ready for the next measurement, and this entire cycle can be repeated up to 54 times without human intervention. For hard-to-clean samples, the integrated cleaning unit is upgradeable with a cleaning detergent pump, which ensures the upper measurement geometry is cleaned effectively.

The option of a third sample rack increases the standard storage capacity from 36 to 54 samples, offering increased walkaway time and higher throughput. An automated Peltier unit pre-tempers the sample before it's placed into the rheometer, shortening the tempering time in the rheometer and, again, increasing throughput.

A tempered storage rack for 36 cups, shown *Figure 3*, keeps samples at a temperature range between +4°C and +45°C, so items like dairy products don't go bad during storage.

Figure 3: Tempered rack in the HTR 3000.

High-throughput rheometer for cone-plate and parallel-plate geometries

For paste-like samples or polymer melts or in cases where only small sample volumes are available, cone-and-plate or parallel-plate geometries are a better choice compared to concentric cylinders or vanes. Anton Paar offers suitable configurations for the adhesive, polymer, personal care, paint and food industry. In addition, with the fully automated benchtop rheometer, the samples must be prepared and placed in the measuring cups before automation and afterwards the option to clean the upper and lower measuring geometry exists.

Since a robotic arm offers greater motion flexibility compared to a three-axis motion system, a six-axis robotic arm with gripper is used in a floor-based high-throughput solution for fully automated rheological measurements. This allows the implementation of automation solutions for cone-and-plate and parallel-plate solutions, and offers the possibility of additional automatic sample preparation and loading, see the sample pipetting in *Figure 5*. Some of the steps in *Figure 5* show implementation for liquid-like samples with cone-and-plate geometries.

Figure 4: The HTR 7000: Fully Automated Rheometer for CC, CP or PP measurements with the automatic trimming tool, a robotic arm, and storage racks for sample discs and sets of measurement geometries, option for sample preparation and fully cleaning the upper and lower measurement geometries.

Figure 5: Attaching the cone, dispensing the sample, and loading in the cleaning unit for a high-throughput rheometer with a six-axis robotic arm and a rheometer equipped with a Peltier temperature device and cone-plate geometries.

The sample vials are placed on a rack by the operator and a vial is gripped by the robotic arm for a measurement, and the code on the vial is read by a reader and placed in a mixing station for automatic homogenisation. During sample mixing, a clean measuring cone is mounted in the coupling of the rheometer and a clean plate is fixed to the bottom in a Peltier temperature device. The Peltier hood is then closed, the corresponding rheometer routine determines the zero-gap position, and then the Peltier hood and the rheometer head with the upper measuring geometry are moved into the loading position. After mixing the sample, the vial is placed in a decapper, the cap is removed and the required amount of sample is drawn off in a disposable pipette or syringe. The sample is then dispensed precisely onto the bottom plate in the exact volume required for the respective measurement geometry. The measuring cone is subsequently brought into the measuring position and the automated sample trimming tool performs the trimming process in order to achieve the highest-possible reproducibility of the measurement results. The Peltier hood is then closed and the rheological measurement begins after temperature equilibrium has been reached. During the measurement, an automated pH measurement can also be performed on the same sample vial. As soon as the rheological measurement is completed, the Peltier hood and the measuring head are moved back to the loading position and the measuring cone and the base plate are placed in a cleaning station tailored to the respective sample type and are cleaned according to a predefined routine and dried afterwards. To reduce rheometer downtime, two sets of measurement geometries can be used, so that while one set is being cleaned the other can be used for the measurement.

High-throughput rheometer for polymer melts

While for liquid-like materials the specimens can be dispensed onto the bottom plate and the measurement geometries can be cleaned relatively easily after the measurements, for materials with high viscosity, paste-like samples, or polymer melts, which are typically solid at room temperature, different strategies for perfect loading of the specimen in the rheometer and providing clean geometries are required. *Figure 4* shows a high-throughput system that includes a sample storage rack and a rack for storing measuring geometries.

As clean geometries are required for each new measurement and for the implementation of polymer melt samples, 30 pairs of parallel-plate measurement geometries, consisting of a bottom plate and an upper measurement geometry, are stored in a rotary vertical storage rack. After loading the geometries into the rheometer, the temperature control unit is closed and after temperature equilibrium at the desired temperature is reached, the zero-gap position is established via the corresponding rheometer routine. The prepared solid-like polymer discs, which are stored in a sample layer rack, are gripped with a vacuum gripper and positioned in the centre of the bottom plate. Now the sample is heated up to the measurement temperature and trimmed, and then the rheological measurement starts according to the predefined order. After the measurement is completed, the measurement geometries are transferred back to the storage rack.

An essential step in preparing a sample in a cone-and-plate or parallel-plate geometry is removing the excess material by trimming the sample after lowering the geometry. Only trimming ensures the correct loading conditions with an ideal sample shape at the edge. To enable trimming in an automated situation, a special device is used where a blade is brought into contact with the edge of the geometry at an angle and rotated around the entire diameter of the sample by a drive motor and gear wheels [1]. This automated trimming tool as shown in *Figure 6* can be incorporated in the electrically heated Peltier base plates, which in combination with the corresponding tempering control hoods ensures good temperature control. To achieve the best trimming results, the blade is mounted within the tempered sample environment and is therefore at the same temperature as the sample before trimming. For trimming, the hood is opened and trimming is started. After trimming, the blade is separated from the geometry, a photo is taken by a camera to document the trimming result, and the hood is then closed again.

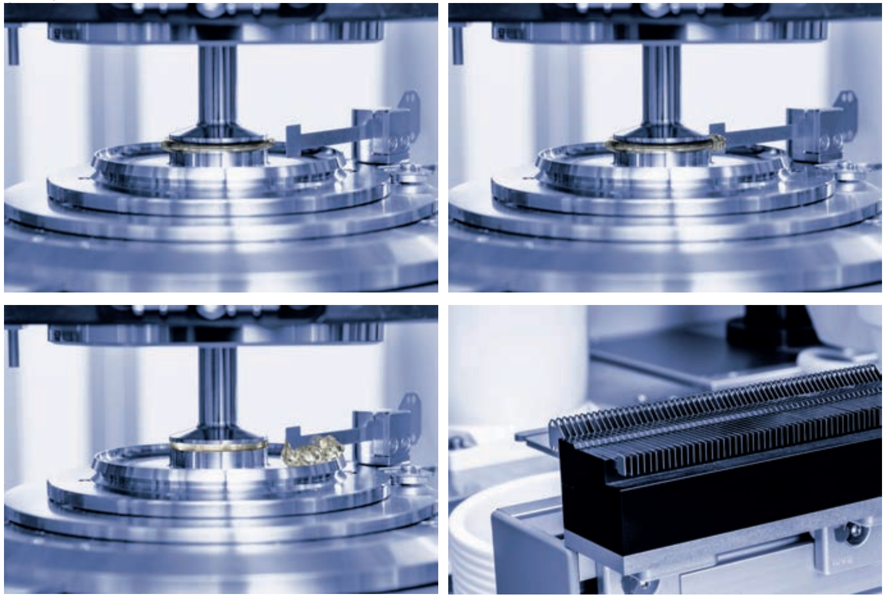

Figure 6: Blade of the trimming tool before, during and after the trimming process; and blade rack with disposable blades.

The whole process is fairly quick, keeping the sample close to the set temperature and reducing the time to reach temperature equilibrium after trimming. After the measurement, the used blade is transferred to a waste box where all disposable blades are collected after use. A new blade is picked up from a trimming blade rack that stores the disposable trim blades.

Results of the trimming process lead to very good reproducibility of the rheological measurements. In an extensive internal study on various polymer materials, it was found that automatic trimming using the trimming tool increased measurement accuracy significantly compared to manual trimming by the same experienced operator. Since different operators may trim slightly differently with manual trimming, the actual increase in measurement quality is even greater. In addition, the camera snapshots after trimming allow documentation of the trimming results and ensure complete traceability of the trimming process.

Benefits of automation

In summary, with the rheology automation products, the HTR 3000 and the HTR 7000, 24/7 availability of automated Anton Paar precision measurement technology is ensured, as is 24/7 availability of automated sample handling and sample preparation, and 24/7 availability of automated data analysis resulting in increased productivity.

The effect on quality is powerful. Automation leads to the highest-possible reproducibility for sample preparation, sample handling, and measurement data analysis and data controlling.

There are major safety benefits too: reduction of human-to-hazard contact points, reduction of safety-impacting human error, and alignment with the CE / UL Safety Concept.

And finally, on the cost front, the advantages are notable as well: HTR 3000 and HTR 7000 enhance resource planning security, reduce the requirements for cost-intensive laboratory resources and the time-to-market for formulations, decrease process times, and allow staff to focus on more important tasks.

References

1. H. Hohensinner and M. Krenn, US patent, US8061240B2, Nov. 22, 2011.