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# A guide to the right rheological device for quality control

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The requirements for quality control in manufacturing grow constantly. As novel materials and formulations change and turnaround times for batches accelerate, the quality control (QC) process must continue to optimize to ensure that products are produced to the highest quality standards. The demands on production require the QC expert needs to perform more measurements in a given production cycle, and sometimes with less personnel than before. Rheological properties play an essential role in both product development and quality control. These properties are used to adjust or predict storage stability, processability or application behaviour. Rheological properties, for example, control haptic or sensory properties such as spreadability, mouthfeel and swallowing behaviour.

Whilst viscometers are used to measure the viscosity and flow behaviour of a sample in rotation, rheometers can be used for further measurements in oscillation. Rheological measuring instruments are capable of managing increasing requirements. In addition to handling a wider measuring range or by providing new measuring possibilities, simplified operation by the user and error prevention procedures enable optimized instrument use for better QC measurements. This Buying Guide covers entry-level rheometers and rotational viscometers designed for QA procedures and reviews criteria that may make the selection of the next rheological instrument ideal for your QC situation.

## How viscosity is determined, in absolute or relative numbers

Viscosity cannot be measured directly but must be calculated based on measured quantities. In a rotational viscometer, for example, the resistance of the sample against the motion of a measuring geometry rotating at a defined speed is detected and quantified.

We make the distinction between absolute and relative measurement results. If a cone-plate or coaxial cylinder measuring geometry is used, the raw data can be converted into absolute viscosity values. These results are independent of the measuring system used and can be compared with other absolute viscosity values. In case of relative measuring geometries such as spindles or vane rotors, the flow profile is unknown, which means that the rheological parameters cannot be calculated. The results obtained in this way can only be compared with each other if the same measuring instruments, the same measuring geometries and identical measuring procedures were used. This can make the comparison of viscosity values considerably more difficult, for example between different manufacturing locations within a company or between suppliers and processors. Relative measuring geometries usually represent a compromise and are often the only way to perform reproducible measurements on complicated samples, such as those that tend to separate, e.g., sediment or contain very large particles.

Viscosity is not a constant. It depends on various parameters such as temperature and pressure. In addition, in case of non-Newtonian flow behaviour, viscosity may depend on the stress (shear rate, shear stress) and time. Flow behavior that is independent of these parameters is called Newtonian. To differentiate between Newtonian and non-Newtonian flow behaviour, several viscosity values must be measured at different shear rates a single-point measurement is not sufficient.

## Your requirement profile for the new measuring device

In purchasing a new measuring device, you need to evaluate a number of important aspects, including daily use and the ability to maintain measurement data over several years. For example: Which samples should be measured, and which product properties should be investigated? Who will operate this instrument? Which standards or measurement regulations (DIN, ISO, in-house) must be taken into account? What investment costs, maintenance and follow-up costs are associated with the purchase of the instrument? In the following, the possible applications of various rotational viscometers and entry-level rheometers are presented. The transition between the two instrument classes is smooth. With a viscometer, rotational measurements can be carried out, whereas with a rheometer it is also possible to measure in oscillation and to determine viscoelastic properties.

## Simplicity hides in the details

The simplest way of determining the viscosity of a material is by a single-point measurement. The viscometer can be operated at the push of a button and a relative value for the viscosity is determined at a constant rotational speed. Different rotors are available depending on the viscosity range. The lower the viscosity of the sample to be measured, the larger the rotor that is used. In order to avoid possible errors due to the selection of an unsuitable rotor, the user can be offered assistance, such as the indication of the measuring range of the respective rotor on the display of the instrument and a note as soon as the permissible measuring range is exceeded and the use of another rotor is recommended. A colour coding of the rotors simplifies their identification. A viscometer can be used as a hand-held device “in the field” or with a stand as a bench top device in the laboratory. A comparison of the current measured value with a reference value on the viscometer’s display enables the immediate evaluation of a measurement result as “in spec” or “out of spec”. Viscometers such as the Thermo Scientific™ HAAKE™ Viscotester™ 3 are ideally suited wherever measurements at ambient temperature have to be carried out quickly.


Figure 1: HAAKE Viscotester 3 Viscometer as hand-held device (left), with laboratory stand (middle) and detailed view of the display with measured value and comparison with reference value (right)

## Standard-compliant relative measurements

In order to determine the flow behaviour, in other words, the dependence of viscosity on shear stress, a rotational viscometer with variable rotational speed is necessary. In the case of viscometers that comply with ISO 2555, for example, the adjustable speeds, the shape of the measuring spindles and the measuring cup with a volume of 600 ml are standardized to ensure the comparability of measurement results. The application range of these models can be extended by accessories, such as temperature-controlled adapters for small sample volumes or low viscosities. The various instruments in this class differ, for example, in the possibility of software connection, in the measuring range – whether for low, medium or higher viscosities – and the rotational speed range. If measurements according to this standard have to be carried out additionally, but not exclusively, the use of these measuring geometries with a rheometer can be useful. The ISO spindle can be inserted into an air-bearing rheometer by means of an adapter, while a holder that can be adapted to the shape of the container fixes the 600 ml measuring cup. This opens up the possibility of carrying out rheological measurements in addition to the standard-compliant viscosity measurements.

## Modular devices for maximum flexibility

The investment in a modular rheometer may seem unattractive at first glance due to its higher purchase price. However, this apparent disadvantage turns into a major advantage when the tasks to be performed by the instrument change at a later time. Instead of purchasing another instrument with different features, only the additionally required modules such as measuring geometries, temperature control units or software modules have to be added to the already existing rheometer. Conversely, it is also possible to purchase the desired rheometer configuration planned over a certain period of time. Initially, the user can start with a basic rheometer configuration and further modules can be added later on according to requirements or available budget.

With a modular instrument concept, all application-relevant components, such as the temperature control unit or the measuring geometry, are exchangeable and a wide range of accessories is available. The selection of a suitable temperature control unit is based on the requirements for temperature range, dynamics or temperature stability. Peltier temperature control is very often chosen because this type of temperature control combines performance and comfortable handling, especially if an air-cooled version is sufficient. For measurements well below 0 °C, a liquid-cooled Peltier temperature control with a suitable cooling thermostat is used.

When selecting the measuring geometries, various aspects need to be considered: how much sample is available for a measurement? At which shear rates should the measurement be performed? Does the sample contain particles? Is the sample prone to drying out or sedimentation? Is the cleaning of the measuring geometry easy or time-consuming? Several variants of the different measuring geometries are available: cones, plates, coaxial cylinders, double-gap cylinders, vane, spiral and pin rotors; made of various materials such as steel, titanium, chemically extremely resistant Hastelloy® or disposable aluminium versions; with different surfaces such as smooth, sandblasted or serrated to prevent wall slip. New measuring geometries are constantly being added due to new applications and problems. Rheology application experts from the instrument vendor [or: Thermo Fisher Scientific] can provide support in selecting measuring geometries based on experience and knowledge of new applications and samples.

In quality control, oscillation measurements determine visco-elastic properties of a product. Changing dynamics in manufacturing and measurement technology requires users who need to become familiar with and master various devices and their operation within a very short time. This means that measuring instruments and their software must be intuitive: compact, flexible and extremely easy to use rheometer models, such as the Thermo Scientific™ HAAKE™ Viscotester™ iQ Rheometer series, available with ball- or air-bearings and a uniquely simple manual lift concept. Numerous user assist functions, such as quick coupling or the automatic recognition of the rotor and the temperature control unit, facilitate error-free installation and quick configuration changes. The rheometers can be operated either stand-alone with measuring and evaluation routines stored in the device or via software on a connected PC. Both rheometer types can measure in rotation and in oscillation. The ball-bearing version operates in a limited frequency range for oscillation measurements but is sufficient for many applications and the demands on the lab infrastructure are lower, as no compressed air supply is required.

**Figure 2: HAAKE Viscotester iQ Rheometer in different configurations**

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## SOPs for user-independent measurement results of highest quality

The essential interface between user and measuring instrument is the software. This is where measuring methods are created, measurements are started, and results are evaluated. With the various measuring possibilities available with a rheometer, the software supports a wide range of options. In environments with several everyday users and materials, it is important that the software adapt to individual uses and tasks. The Thermo Scientific™ HAAKE™ RheoWin™ Software offers a simple graphical user interface for the beginner. Behind this interface, advanced users can find a wide range of options to adapt a measuring method to their individual requirements and fully utilize the rheometer's capabilities.


**Figure 3: Execution of a standardized measuring and evaluation routine directly at the rheometer**

Measurement methods may include instructions in the form of text, images and videos to assist the user in performing measurements and data evaluation based on standardized measurement routines (SOPs) or established industry standards. In addition, the software can be switched to one of 15 languages during operation with just one click and can thus be adapted to changing users. A wide range of evaluation routines including automated evaluation criteria is available for data evaluation, for user-independent and reliable measurement results,[1] as required by QA. Ideally, the standardized methods can be performed directly on the rheometer, such as using the touch screen of the Thermo Scientific™ HAAKE™ MARS™ iQ Rheometer (Figure 3). Finally, requirements for database connections such as LIMS should also be considered when selecting a suitable measurement software.

## Secure purchase through right of return

Device descriptions are often very extensive and detailed. Nevertheless, they do not replace the experience gained from handling the device. For this purpose, on-site tests and loans are an easy way to get to know devices and software. In the case of the HAAKE Viscotester iQ Rheometer, a complete measuring setup is available for beginners in rheology. This consists of the rheometer, a laptop with installed software and measuring and evaluation routines matched to three samples, which are also included in the delivery. The intuitive rheometer can be easily installed and used without any previous rheological knowledge by means of a video tutorial. This allows an easy introduction to rheology.

## Conclusion

The requirements for rheological measurements in quality control are very diverse. In order to select a suitable measuring instrument, various aspects should be considered. For simple and fast measurements at ambient conditions a viscometer for single-point measurements is sufficient. If requirements change, the investment in a higher quality modular rheometer can be the more economical alternative, as the measuring device and thus the measuring possibilities can be extended. Irrespective of the instrument class, intuitively operated instruments and their software allow for quick familiarization. Standardized measuring and evaluation routines for rheological characterization ensure user-independent results of the highest quality. An instrument decision has long-term effects. Use the opportunity to test the instrument of your choice in your own company and under everyday conditions.


**References**

1. Fabian Meyer, Cornelia Küchenmeister-Lehrheuer, Thermo Fisher Scientific application report V223 "HAAKE RheoWin Software – features for quality control and routine measurements"


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