



Reliable measurements from sample preparation to data interpretation

**Rheology for Quality Control-
Better rheological results**

Can you rely on your rheological measuring results? This whitepaper provides insight into the importance of standardized measuring procedures (SOPs) and what you should consider when it comes to rheological measurements.

Standard operating procedures (SOP) for routine rheology offer an opportunity to improve efficiency, safety, quality and uniformity through written, step-by-step instructions. An SOP ensures continuous quality in the results, even when untrained operators conduct tests.

This whitepaper describes the development of an effective, complete SOP for rheological measurements in QC. The rheometer, accessory and software are taken into consideration.

- Plates, cones, coaxial cylinder. Which one is most suitable?
- Sample consideration. What do I need to think about for sample history, evaporation, slippage, etc?
- Sample loading, trimming, and gap closing. How can you ensure optimal sample filling?
- User management, measurement and data evaluation. Which capabilities does the software offer to support routine measurements for QC?

This guide provides answers to these and many other questions.

Well prepared - good results

Authors

Cornelia Küchenmeister-Lehrheuer and Klaus Olddörp
Thermo Fisher Scientific, Karlsruhe, Germany

Introduction

In recent years the demands regarding the reliability of rheological test results have grown significantly, like for most analytical methods. Only if correct test results can be produced and easily be reproduced they can be used for the reliable characterization or comparison of substances.

Based on correct test results it is e.g. possible for the QC department to compare different incoming materials or different batches from production no matter whether these results have been produced on different instruments or even on different sites. It is nevertheless essential to use a viscometer or rheometer with a measuring geometry, which gives absolute results, like e.g. coaxial cylinders (CC), parallel plates (PP)- or cone and plate (CP)-geometries. Depending on the sample's nature it can also be necessary to agree upon the test routine and data evaluation method.

Every test result contains a certain error, which is the sum of many effects mainly related to the sample, the instrument and the handling. Using the example of PP- and CP-geometries, this report will describe the preparation of a rheological test aiming to show the possible errors and how to minimize them. It is assumed that the rheometer has been installed correctly and is properly levelled.

Cone or plate? How to choose the right measuring geometry

The classical measuring geometries available for Thermo Scientific™ HAAKE™ rheometers have a notched top (Fig. 1). The motor axis contains a pin, which fits into the notch thus allowing the geometry to be mounted only in always the same position relative to the motor's rotor.



Fig. 1: Classical measuring geometry with a notched cone (left) and a Connect Assist measuring geometry with a mark on its ceramic shaft for defined mounting (right).

The newer Connect Assist measuring geometries have a mark, which can be aligned with a similar mark on the rheometer to achieve the same effect. When a calibration like the MicroStressControl (MSC) [1] is performed to improve the data quality in the low-torque-range (nNm), its results can also be used for later test since the whole setup motor plus measuring geometry is always assembled exactly the same way.

Measuring plates, used as the lower part of PP- or CP-geometries are also designed to be mounted only in one position (Fig. 2) [2].

In a CP-geometry, the shear rate is constant over the whole sample, whereas in a PP-geometry the shear rate decreases from its maximum value at the edge to zero at the centre of the geometry. When non-Newtonian samples are tested with a PP-geometry, viscosity values always contain an intrinsic error because different parts of the sample are exposed to different shear rates. Therefore if possible, a CP-geometry should be used for viscosity tests. Still, due to the bigger flexibility regarding the measuring gap, PP-geometries are the better and sometimes the only choice for many applications. The diameter of the geometry has to be chosen in relation to the sample's viscosity. For water-like samples it is recommended to use cones or plates with the biggest diameter (60 mm). With increasing viscosity smaller diameters have to be used. For e.g. bitumen or hard rubber an 8 mm plate is often the best choice.

For PP- and CP-geometries the correct amount of sample becomes more important for small sample volumes and big edge effects. Therefore to load the correct amount of sample becomes more important for smaller diameters, smaller gaps and higher sample viscosities.

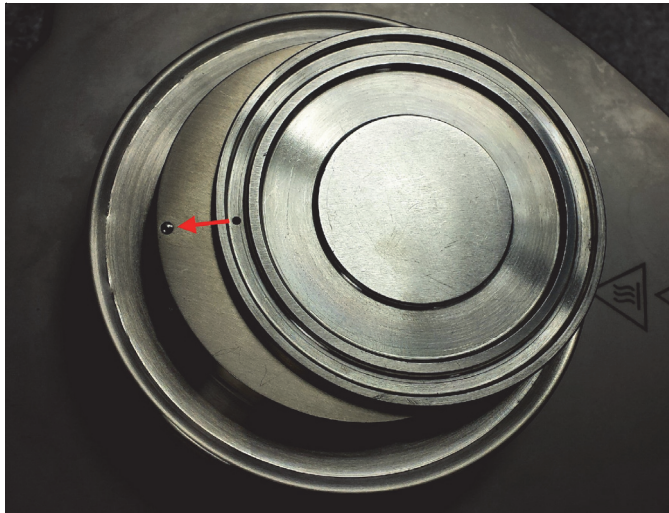


Fig. 2: For our PP- and CP-geometries, measuring plates TMP with the corresponding diameters are available. The notch under the mark on the TMP slides over the pin in the temperature control unit for easy and precise positioning.

Determination of the zero gap - reference for the measuring gap

Whenever a cone, a plate or the lower measuring plate has been mounted, e.g. after it had to be removed from the instrument for cleaning or when a different geometry has been selected, the axial zero point of the geometry has to be determined. In other words, the axial position where the upper part of the measuring geometry touches the lower part is needed as the reference point for the precise setting of the measuring gap. Any error of the zero gap will auto-matically lead to an increased error of the test results due to a wrong gap size during the test.

The zero gap can be determined manually using the monitor mode in the Job Manager of the Thermo Scientific™ HAAKE™ RheoWin™ rheometer software (Fig. 3). When using an instrument with an automatic lift (e.g. a Thermo Scientific™ HAAKE™ MARS™ rheometer) it is

recommended to include the auto-matic zero gap determination into the HAAKE RheoWin Job (Fig. 4). This prevents forgetting this important step and leads to a user-independent precisely determined zero gap.

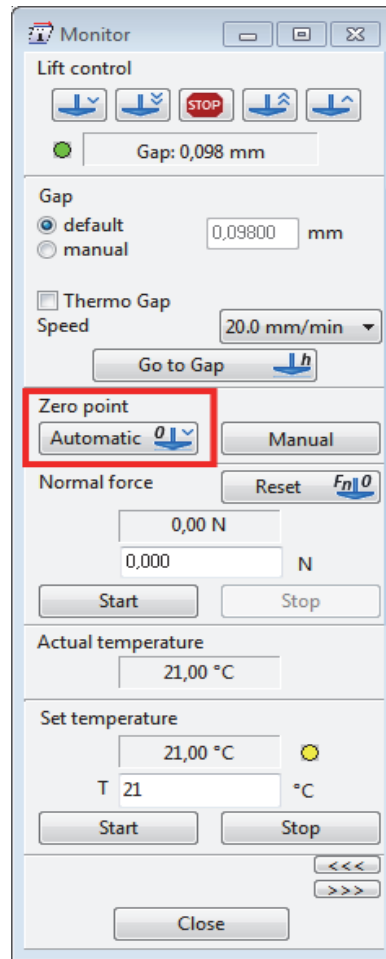


Fig. 3: Automatic determination of the zero gap using the monitor mode.

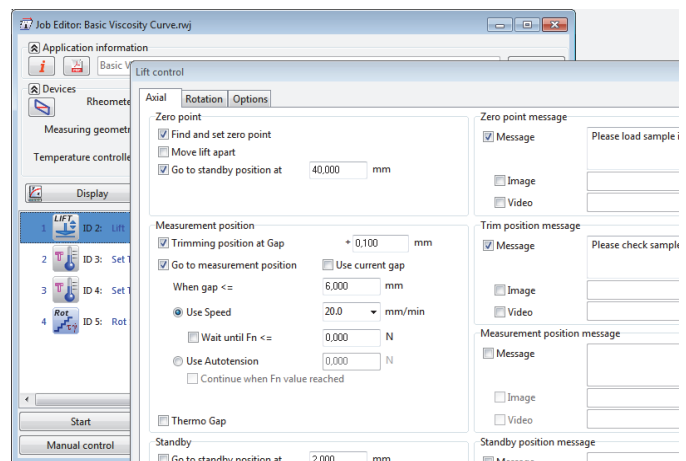


Fig. 4: Automatic determination of the zero gap using the lift control element during a test run. Here a user-defined message has been activated to ask for the sample to be filled into the geometry.

To avoid any error due to thermal expansion or shrinkage of the measuring geometry, the zero gap has to be determined at the temperature, the test is going to be started afterwards. The upper part of the geometry can be put onto the lower measuring plate, which is directly temperature controlled. After the temperature reading remains constant, the upper part of the geometry needs some additional time to adapt to that temperature as well. If

this is done with the upper geometry already mounted, extra care has to be taken that the rheometer's air bearing is not damaged by the expanding geometry for example by setting a small constant normal force. After every part of the measuring geometry has reached the correct temperature, the zero gap can be determined and will be stored as the reference point for the measuring gap.

Sample history

The pre-treatment or history of the sample can play a crucial role for getting correct and reproducible data. The user has to design the test method keeping in mind that the sample needs to be in thermal and mechanical equilibrium before the rheological test starts i.e. by allowing a sufficiently long waiting time between closing the measuring gap and starting the test. During this time the structure of an e.g. thixotropic sample can recover from the partial destruction during the loading and closing procedure.

In some cases it is impossible to reach a stable equilibrium before starting the rheological test. Common examples are samples undergoing a chemical reaction like e.g. glues or coatings. When dealing with such samples, every step of the sample preparation has to be done following always the same sequence and the same timing to start all tests at the same state of the sample in order to get comparable data. In case of samples with a very long structural recovery like for example some thixotropic coatings, a defined pre-shear in the rheometer helps to start the test at least from the same degree of structural damage thus leading to comparable results. Newtonian fluids do not show any of the effects mentioned above. Here the focus "only" needs to be on correct thermal equilibrium and correct gap filling, which will be looked at in more detail below.

Especially when special accessories are used like for example a sample cover with solvent trap to minimize the evaporation of the solvent in the sample or a measuring geometry with a sand-blasted or serrated surface to avoid slipping, the same set up has to be used to yield comparable results.

The sample volume needed for the correct filling of a measuring geometry can be found in the HAAKE RheoWin software as part of the geometry's properties (Fig. 5) and in the appendix of the rheometer's manual.

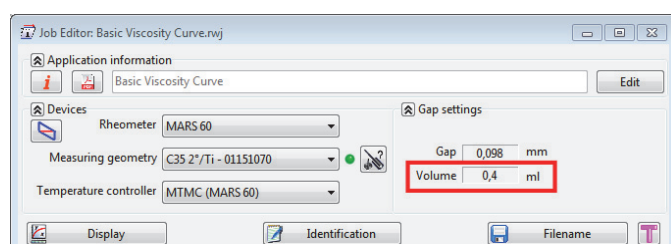


Fig. 5: HAAKE RheoWin software displaying the sample volume for correct gap filling. In this example of a cone C35/2° Ti L, all geometry parameters including the gap and the serial number have been read from the geometry after mounting it into the rheometer.

Choosing a suitable measuring gap

One of the fundamental differences between PP- and CP-geometries is linked to the measuring gap. For every cone only one correct gap exists, equal to truncation of the cone's tip. In case a different gap is needed, a cone with a different cone angle has to be used. For the typical cone angles between 0.5 ° and 4 ° the gap is usually in the range between 25 µm and 140 µm.

In contrast, the measuring gap of a PP-geometry can be varied within a certain range, so the measuring conditions can be adapted to the sample's properties.

The correct measuring gap for any CP- or cylindrical geometry is part of its set of individual parameters, measured and calculated based on its dimensions. After production every part is measured precisely and its diameter, cone angle and truncation are printed into an individual certificate included in the geometry's box. For the classical measuring geometries, these parameters have to be entered manually into HAAKE RheoWin once and from then on are automatically available whenever the geometry is used for a test. With the Connect Assist geometries, all relevant parameters will automatically be transmitted to HAAKE RheoWin when the geometry is mounted into the rheometer.

For both PP- and CP-geometries the rule applies that the measuring gap has to be at least 5 times the diameter of the biggest particle in the sample to be able to measure the sample as whole. In the worst case some bigger particles could pile up under shear and block the gap leading to very noisy data or even damage the surface of the measuring geometry. For example, a suspension with particles up to 100 µm in diameter needs a gap of at least 500 µm. In this case only a PP-geometry can be used because CP-geometries with an angle bigger than 4 ° would not comply with the current standards and do therefore not exist. The factor 5 is just a rule of thumb. Depending on the particle's characteristics, it might become necessary to select an even bigger gap.

When doing tests on foams or emulsions the measuring gap has to be chosen based on the diameter of the biggest bubbles or droplets. Otherwise the sample's properties could already be changed simply because it is squeezed into the measuring gap.

When a sufficiently large measuring gap is used, like e.g. 1 mm with a PP-geometry, any kind of error in parallelism can be neglected due to the tolerance of manufacturing. For very small gaps these small imperfections can lead to an error of the zero gap determination and therefore the measuring gap itself. Either a bigger uncertainty has to be taken into account for test results collected with very small gaps or even greater care has to be invested when producing and adjusting the components for such a test.

Sample loading, sample trimming, closing the gap- the optimum gap filling

Under ideal conditions, the sample fills the measuring gap completely and without any air bubbles. Around the edge of a PP- or CP-geometry the open sample surface should slightly bulge outwards.

Depending on the sample's consistency a suitable tool should be used to fill the sample into the measuring geometry. For low viscous samples a pipette can be used. For samples with a higher viscosity or stronger texture a spatula or spoon is the right tool. Samples with a delicate structure should be sheared as little as possible during the loading procedure to keep damages to the structure as small as possible. In general, the sample should be placed in the centre of the geometry.

The optimum amount of sample can be found amongst the parameters for each measuring geometry listed in HAAKE RheoWin (Fig. 5). It is recommended to slightly overfill the geometry first in order to avoid air remaining in the measuring gap after closing it. Underfilling of the measuring gap has to be avoided under all circumstances!

After closing the measuring geometry the sample has to be trimmed i.e. the excess of sample that was squeezed out of the gap has to be removed with a suitable tool. Since this procedure leads to a straight sample rim, it is recommended first to go to the trimming position above the measuring gap, trim the sample and then close the geometry thus forming a slightly bulged sample rim.

As rule of thumb, a gap 1 – 5% wider than the measuring position is used as trimming position. After loading the sample this position is set either manually or automatically during a running test method in the HAAKE RheoWin software. For sensitive samples the lift speed should be reduced to minimize the damage to the sample's structure during closing the gap.

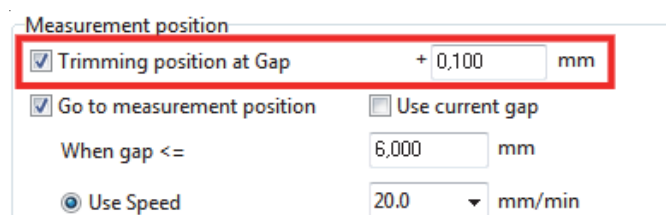


Fig. 6: Parallel plate geometry in measurement position without and with sample cover. The trimming position can be set in HAAKE RheoWin. Recommended are 1 - 5% above the measuring gap chosen for the test (in the example above 100 μm).

When the upper geometry approaches the trimming position using HAAKE RheoWin's lift function, the axis of the rheometer is locked to avoid any damage to the sample's structure by an accidental turning of the measuring geometry. The excess sample can be removed with a lab spatula or a special trimming tool [3]. The lower plate shown in Fig. 7 has been chosen to match the upper geometry's diameter, which makes the necessary trimming procedure much easier.

The tools selected for trimming should be made of a non-absorptive material to avoid any solvent being sucked out of the sample. Any kind of simple paper or wooden tool is therefore excluded. The tool should have a straight edge to form a clean straight sample surface.



Fig. 7: Lower measuring plate TMP with a diameter chosen to match the upper geometry.

For a properly trimmed sample the spatula is moved around the whole sample using the side of the lower plate as a guide (Fig. 8a). Afterwards, the rim is checked visually whether all excess has been removed. If necessary, this step has to be repeated.

Especially when trimming highly viscous samples, it is possible that a bit of the excess sample gets pushed onto the edge of the upper geometry. During the test procedure this material could flow down again, leading to disturbing edge effects thus having a bad influence on the data quality. Therefore it is recommended, especially for highly viscous samples and geometries with small diameters, to strip the edge of the upper geometry from any remaining material (Fig. 8b).

Finally the geometry will move to the measuring position and the sample will get a slightly bulged rim as indicated in Fig. 9.

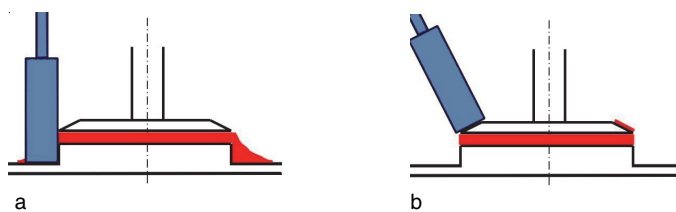


Fig. 8: Removing excess sample with a spatula in trimming position; 8a: Removing excess sample from the edge of the upper geometry.

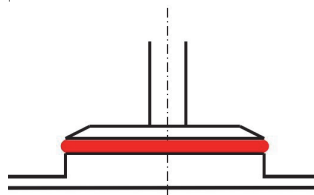


Fig. 9: Correct gap filling after closing the measuring geometry.

A perfect filling is the ideal basis for generating good data with a rheological test. Examples for such a test have been described in detail in [4] and [5] using the examples of testing a calibration oil and a viscoelastic PDMS standard respectively.

Summary

For the determination of reliable rheological data the sample and the rheometer have to be prepared and handled care-fully. Apart from selecting the right measuring geometry and determining the correct zero point, there are some steps not directly linked to the rheometer itself, which are crucial for the data quality. The procedure how to prepare the sample into the rheometer and how carefully the sample is trimmed afterwards are at least equally important. Special care is needed when using small measuring geo-metries, small measuring gaps and high viscosities, since in these cases edge effects have a bigger influence on the data quality. In case of small measuring gaps an individual adjustment of all components involved can improve the data quality. Following the recommendations listed in this report, the accuracy and reproducibility of rheological results can be improved significantly. Especially when rheological results have to be compared with results from other departments or other companies, accurate results are an absolute must.

References

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"Test liquids", Klaus Oldörp
- [5] Thermo Fisher Scientific Application note V264
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APPLICATION NOTE

HAAKE RheoWin software - features for quality control and routine measurements

Author

Fabian Meyer

Thermo Fisher Scientific, Karlsruhe, Germany

Introduction

Rheology is used in more and more industries for product development and quality control. A trend is to develop standard operation procedures like it is common for many other analytical methods. Standardized measuring procedures are necessary to optimize product properties and to determine possible qualitative fluctuations of a product.

By determining the relevant rheological parameters, relationships between structure, process behavior and final product properties can be established. Quality standards can be fulfilled and new products launched on the market faster. This applies to many industrial fields such as plastics and adhesives, paints and coatings, personal care and detergents, foodstuffs or even building materials.

Rheological measurements provide information about storage stability, processability or flow properties of liquid and semi-solid formulations, which are essential for quality control and further product development.

However, due to the increasing complexity of the materials and the high performance requirements, it is not always easy to develop the optimal testing method for a particular application. In this context, a versatile measurement and evaluation software can provide useful support to both, beginners and experts in the field of rheology, in order to establish and execute suitable procedures. The Thermo Scientific™ HAAKE™ RheoWin™ is the instrument control and data evaluation software for all Thermo Scientific™ HAAKE™ rheometers and is used to set up and run measurement procedures with subsequent automatic data evaluation and report generation.

The following article is intended to provide an overview of the possibilities offered by the HAAKE RheoWin software in order to be able to perform a comprehensive and meaningful rheological characterization in quality control.

General structure of the HAAKE RheoWin software

The HAAKE RheoWin software can be changed to 12 different languages with the touch of a button and consists of three different modules:

- The HAAKE RheoWin JobManager for creating and executing measurement and evaluation routines - so-called Jobs. The JobManager also allows for controlling all functionalities of the rheometer individually and outside of complete measurement and evaluation routines.
- The HAAKE RheoWin DataManager for displaying and further evaluating measured data. Different data sets can be overlaid and compared. Graphic and table layout can be formatted and data be transferred into various formats.
- The HAAKE RheoWin UserManager for creating user accounts of different levels and assigning specific and individual user privileges.

In addition, the following optional software modules are available for specific demands and applications:

- A CFR Part 11 tool to meet the requirements of US FDA 21 CFR Part 11
- Additional modules for polymer analysis: Time Temperature Superposition (TTS), generating relaxation spectra and the calculation of molecular weight distribution (MWD)
- Software module for interfacial rheology
- Software module for combination with FT-IR spectrometer

For creating Jobs with the HAAKE RheoWin JobManager, predefined graphical elements are used (Fig. 1). These elements cover all aspects of a complete standard operation procedure, including:

- pre- and post experimental instrument settings
- experimental settings
- data evaluation
- data handling, export
- report creation

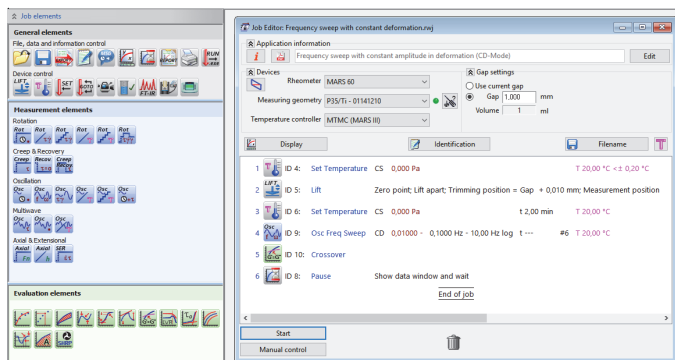


Fig. 1: Graphical elements for creating measurement and evaluation routines in the HAAKE RheoWin JobManager.

All elements can be selected quickly and comfortably via a drag & drop method. All measurements and evaluations can be carried out fully automatically in a single Job sequence. A manual operation mode can be used to perform rheological pre-tests or for gap setting and temperature control outside a measurement and evaluation routine.

The HAAKE RheoWin software allows for simultaneous sample testing and data evaluation or multiple measurements with different rheometers connected to the same PC. Measurement results can be exported and stored in various formats (ASCII, MS-Excel or PDF). The connection to a laboratory information management system (LIMS) is also possible.

Selected measuring and evaluation elements for rheological characterization in quality control and product development

Important product properties for consumers and manufacturers are for instance storage stability, viscosity, yield stress, thixotropy or curing behavior. The determination of these rheological parameters allows for improving product performance as well as for an effective and reproducible evaluation during quality control.

How does a product react under stress or strain? How do different additives, such as fillers or pigments affect the properties of a material? Which thixotropic agent and how much of it has to be added to a product in order to maintain good flow properties or to prevent sagging effects? How should a yield stress be adjusted in order to ensure good shelf life and transportability of a product? How quickly should a micro structure rebuild, after a material was exposed to high shear?

These are just some of the many questions that product manufacturers have to deal with over and over again. The HAAKE RheoWin software provides evaluation routines with the possibility of defining QC criteria for many standard testing methods (Fig.2).

The following section presents selected options offered by the HAAKE RheoWin software to perform effective quality control test routines. The predefined measuring and evaluation elements only have to be adapted to the respective product and the corresponding quality criteria have to be defined accordingly.

Reference curve with deviation tolerance

By using the reference curve element, new measuring results can be compared with previous measurement

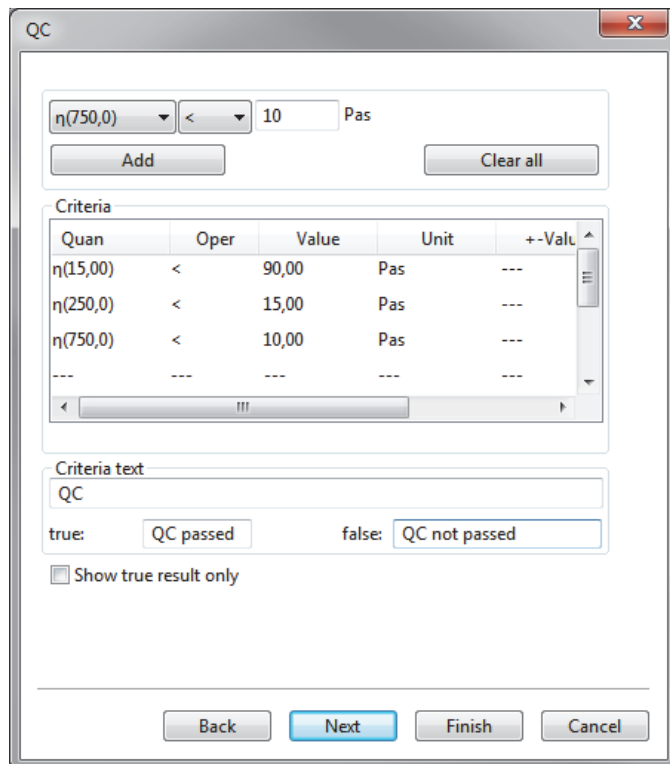


Fig. 2: Definition of Quality Control criteria in a measurement and evaluation routine.

data (Fig.3). A reference curve can be either a fixed data file or selected individually for every measurement by the operator.

The tolerance by which the measured values may deviate from the reference curve can be specified either as percentage or absolute values. Furthermore, the deviation tolerance can be set to either a linear or a logarithmic scale.

Interpolation with deviation tolerance

Interpolation is the calculation of a data point between two measured values. An interpolation can be performed automatically after a measurement (Fig. 4). Various methods are available for this purpose. For the interpolated values, a deviation tolerance can be defined in the HAAKE RheoWin software. Interpolated values need to be inside the deviation tolerance in order for the sample to pass the quality assessment.

Curve fitting

The HAAKE RheoWin software allows for performing curve fitting with various mathematical and rheological models (Fig. 5). Fitted curves can be extrapolated beyond the range of measured data. After a curve fitting was performed all calculated parameters are reported and stored along with the measured data.

Determination of the linear-viscoelastic range

Within the linear-viscoelastic range rheological parameters are independent of the applied stress or strain value. The linear-viscoelastic range of a sample can be determined by performing an oscillation amplitude sweep at constant frequency. The HAAKE RheoWin software allows for the automatic determination of the linear-viscoelastic range from amplitude sweep data (Fig. 6). The end of the linear-viscoelastic range is reached at stress or strain values, where the rheological parameters will start to change from a constant behavior.

Which parameter is used for the evaluation (G' , G'' , \ln^*I , δ or $\tan \delta$) can be selected by the operator. The linear-viscoelastic range can be determined either as a stress or strain value. The HAAKE RheoWin software also allows for the determination of the linear-viscoelastic range according to the DIN 51810-2 standard procedure.

Area under a measuring curve

The area under a measurement curve or a selected curve section can be calculated by the HAAKE RheoWin software (Fig.7). Upper and lower deviation tolerances can be defined as a quality control criterion.

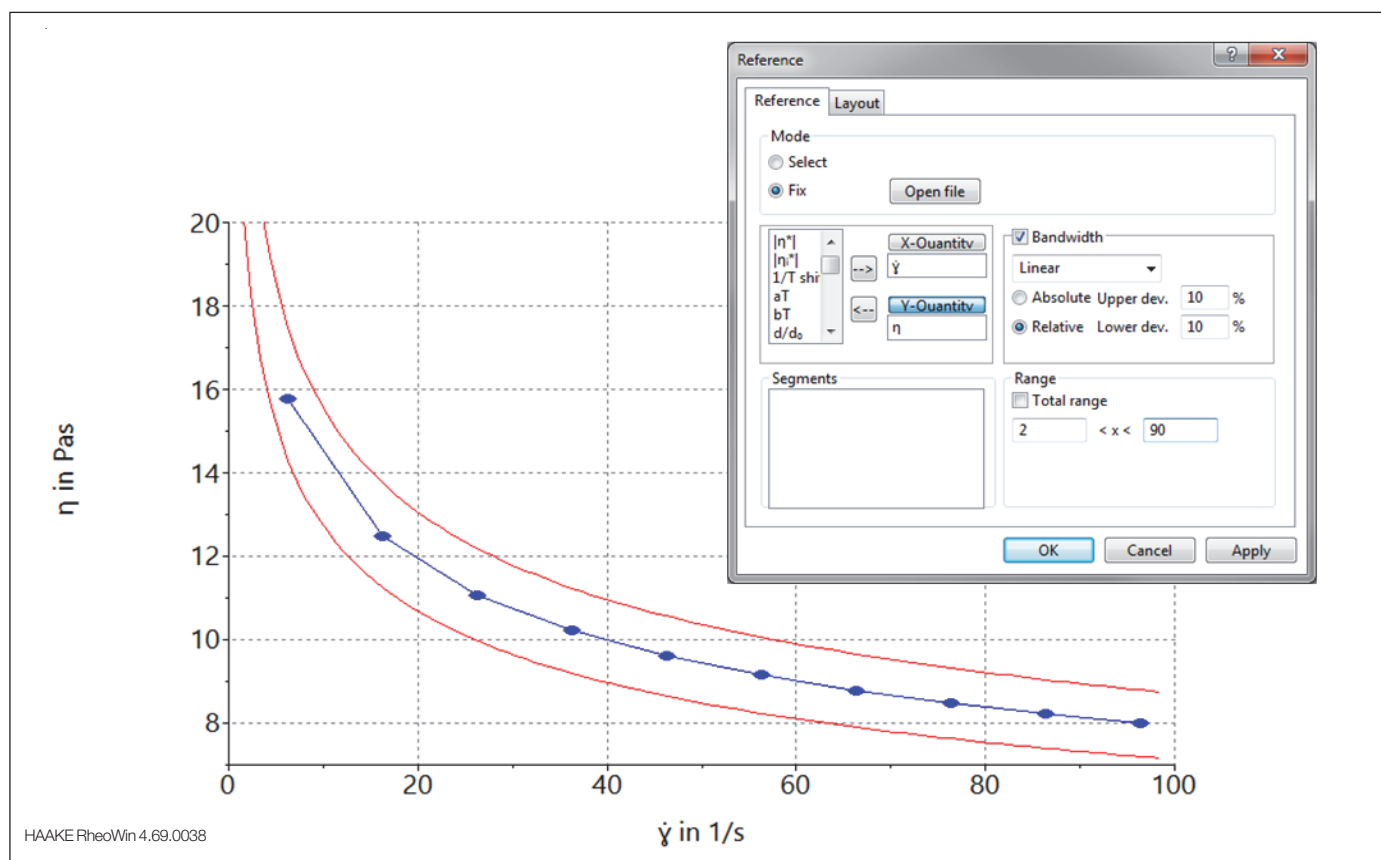


Fig. 3: Reference curve with deviation tolerance.

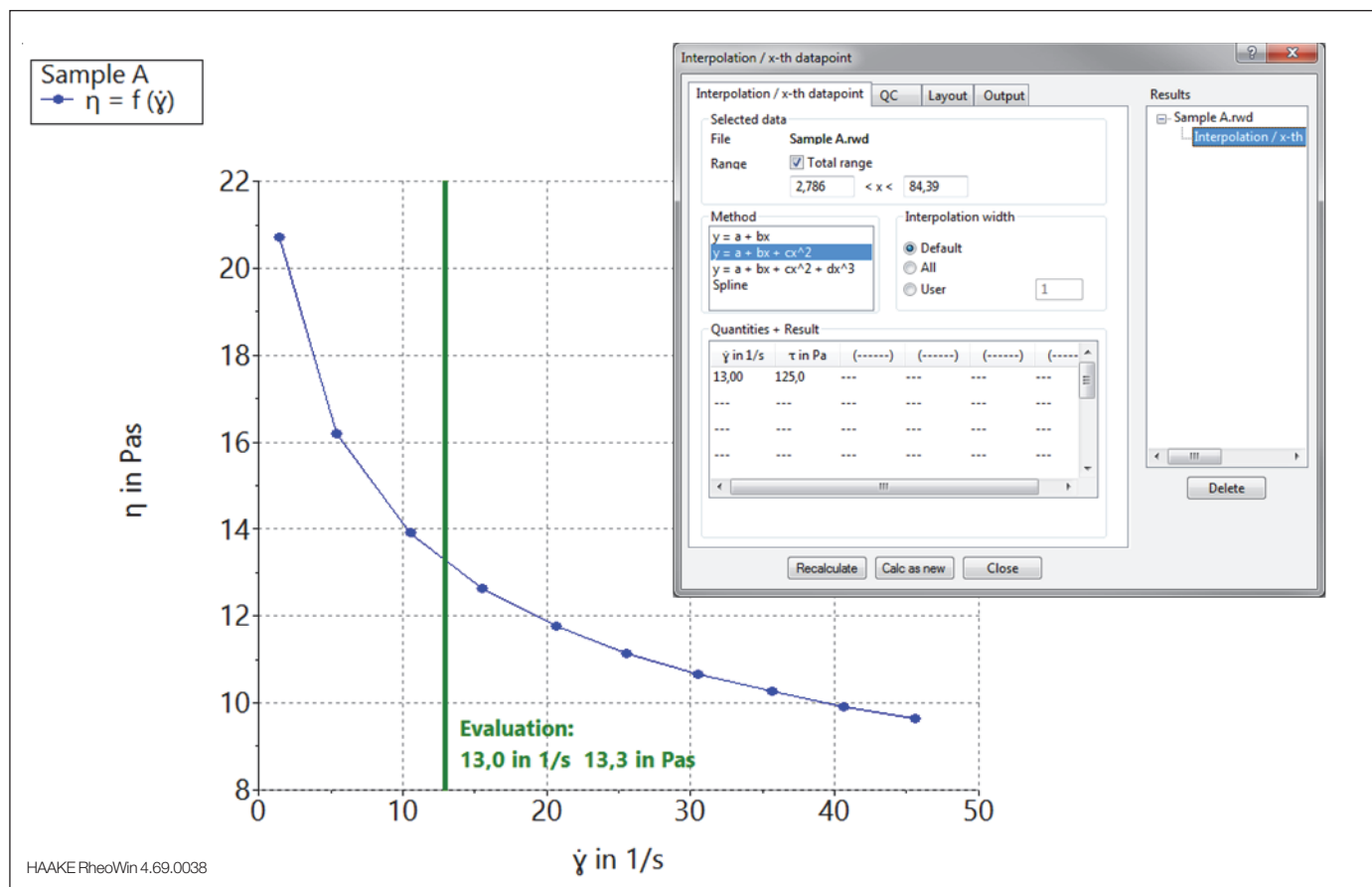


Fig. 4: Interpolation of a viscosity value at a defined shear rate.

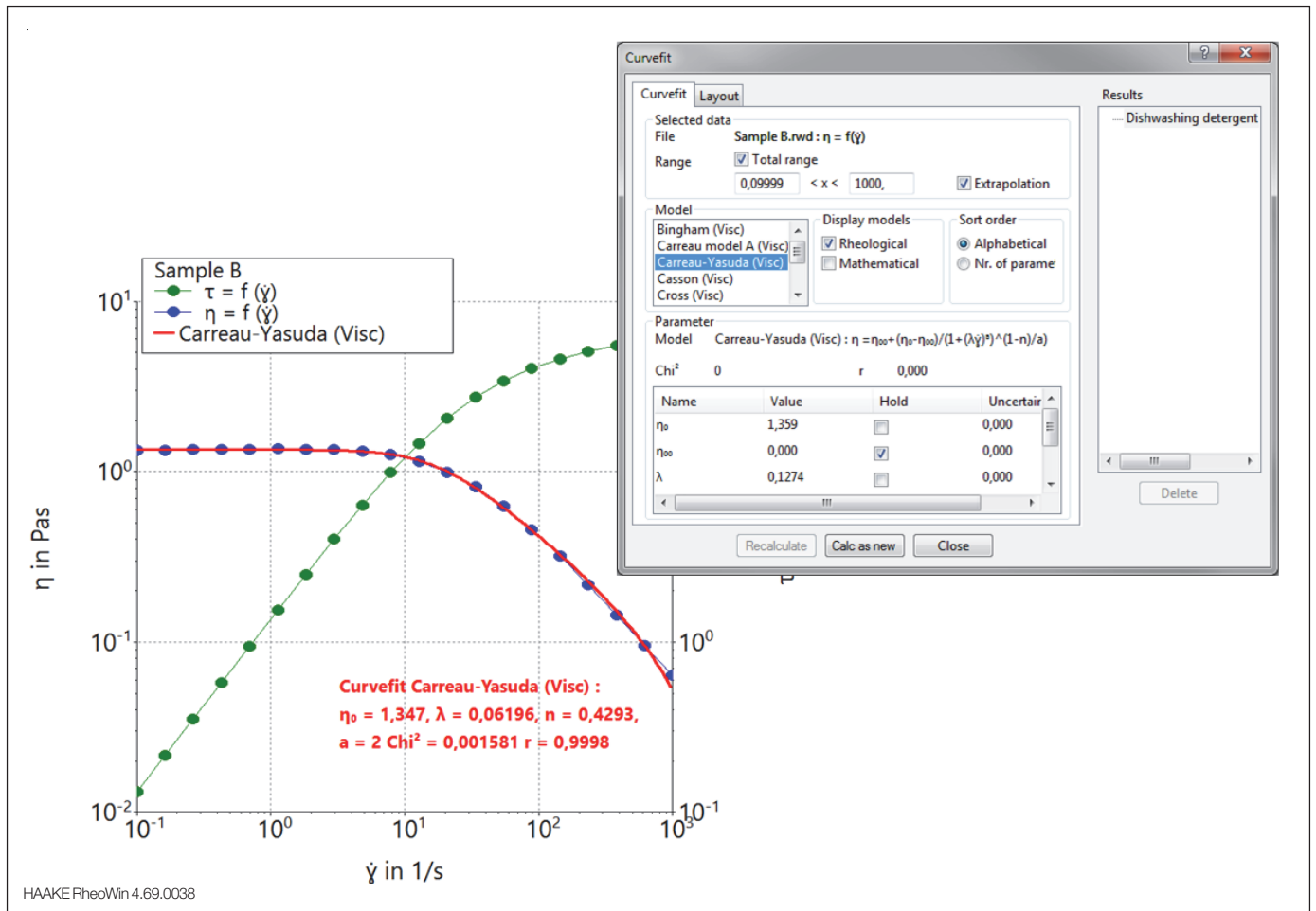


Fig. 5: Curve fitting of viscosity data with the Carreau-Yasuda model.

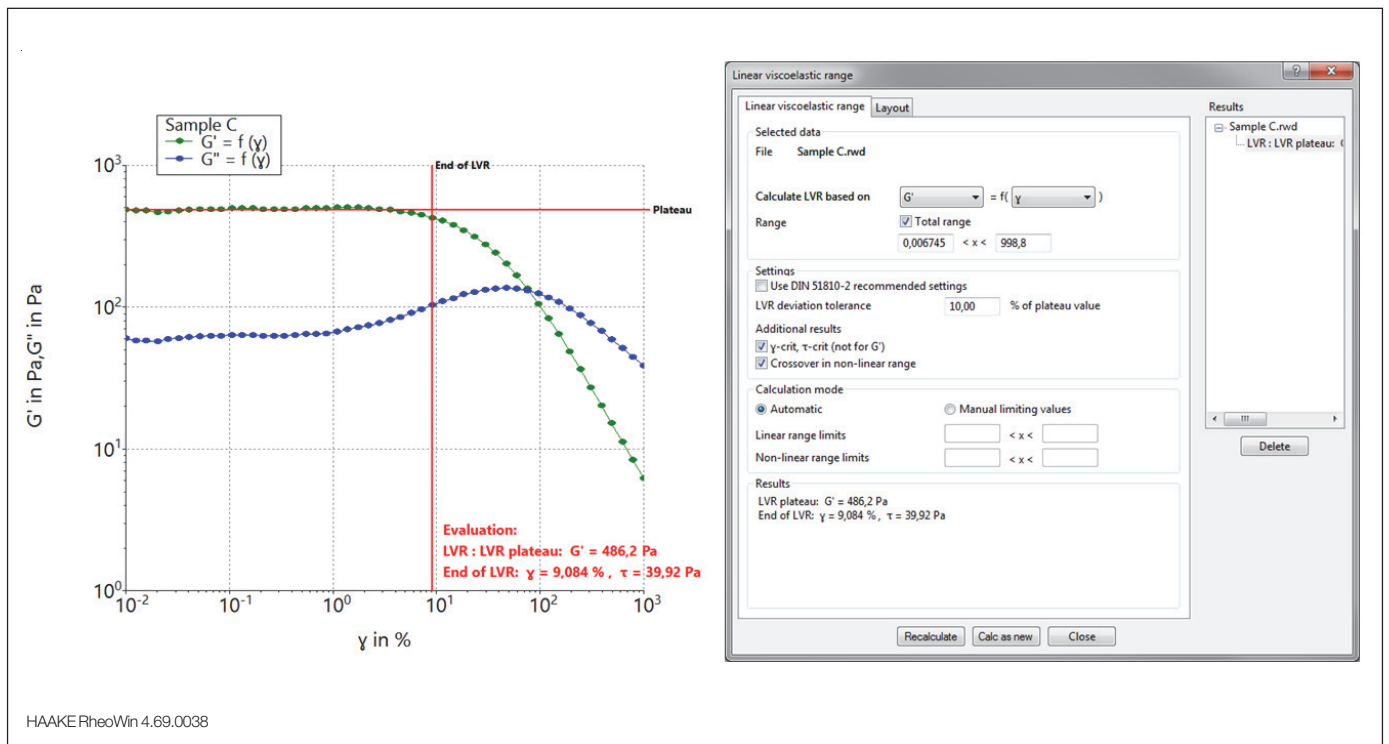


Fig. 6: Automatic determination of the linear-viscoelastic range from an amplitude sweep test.

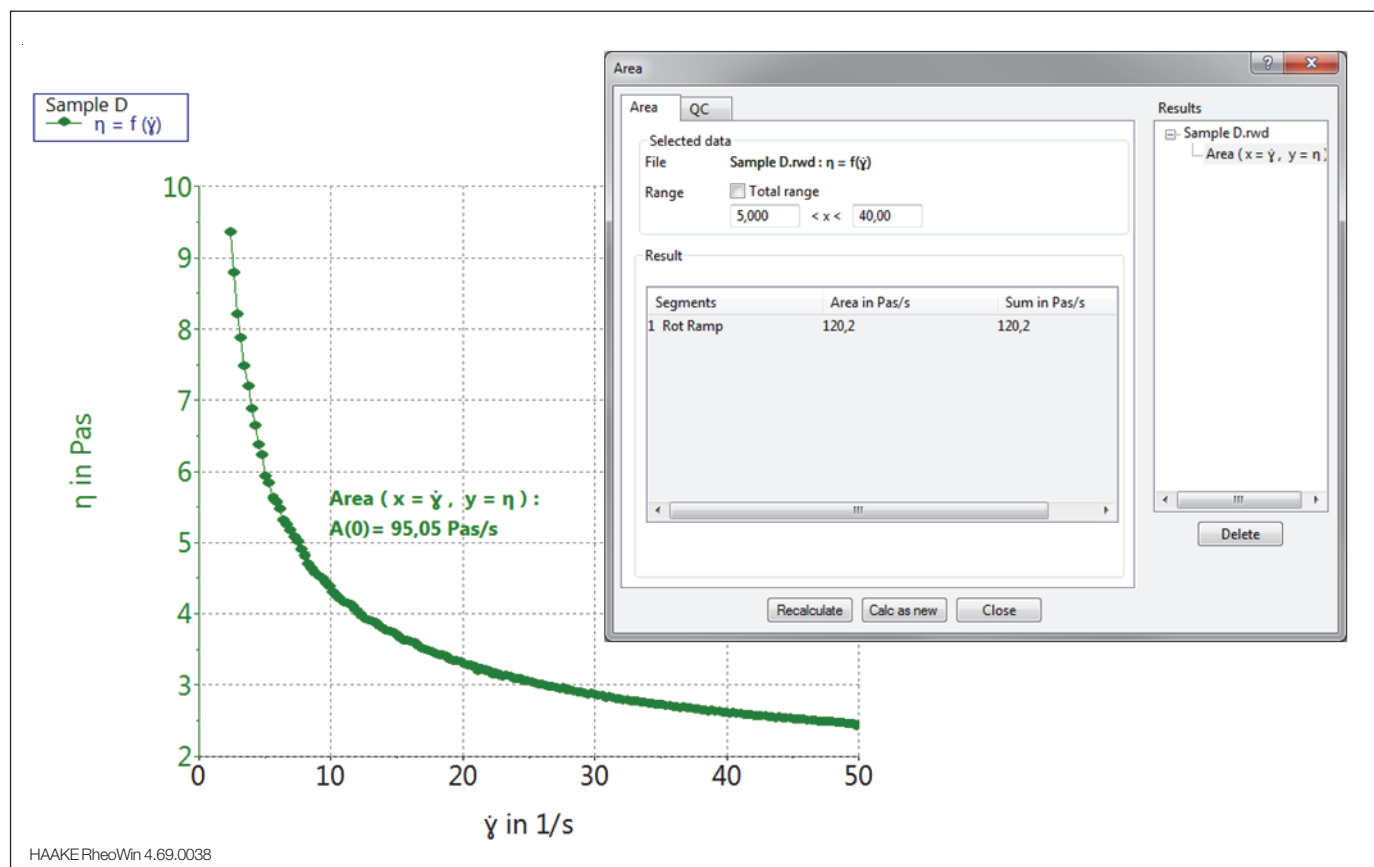


Fig. 7: Calculating the area under a viscosity curve.

Thixotropic loop test

Thixotropy refers to a shear rate and shear time depending decrease in viscosity of structured fluids. In a thixotropic loop test, a sample is exposed to an increasing followed by a decreasing shear rate ramp. The apparent viscosity and the shear stress are recorded as a function of shear rate. The hysteresis area that

forms between the up and the down curve is a measure for the degree of thixotropy of the sample. When performing a thixotropic loop test, the HAAKE RheoWin software can determine the hysteresis area automatically for the viscosity or the shear stress data (Fig.8). Quality control criteria with deviation tolerance can be defined.

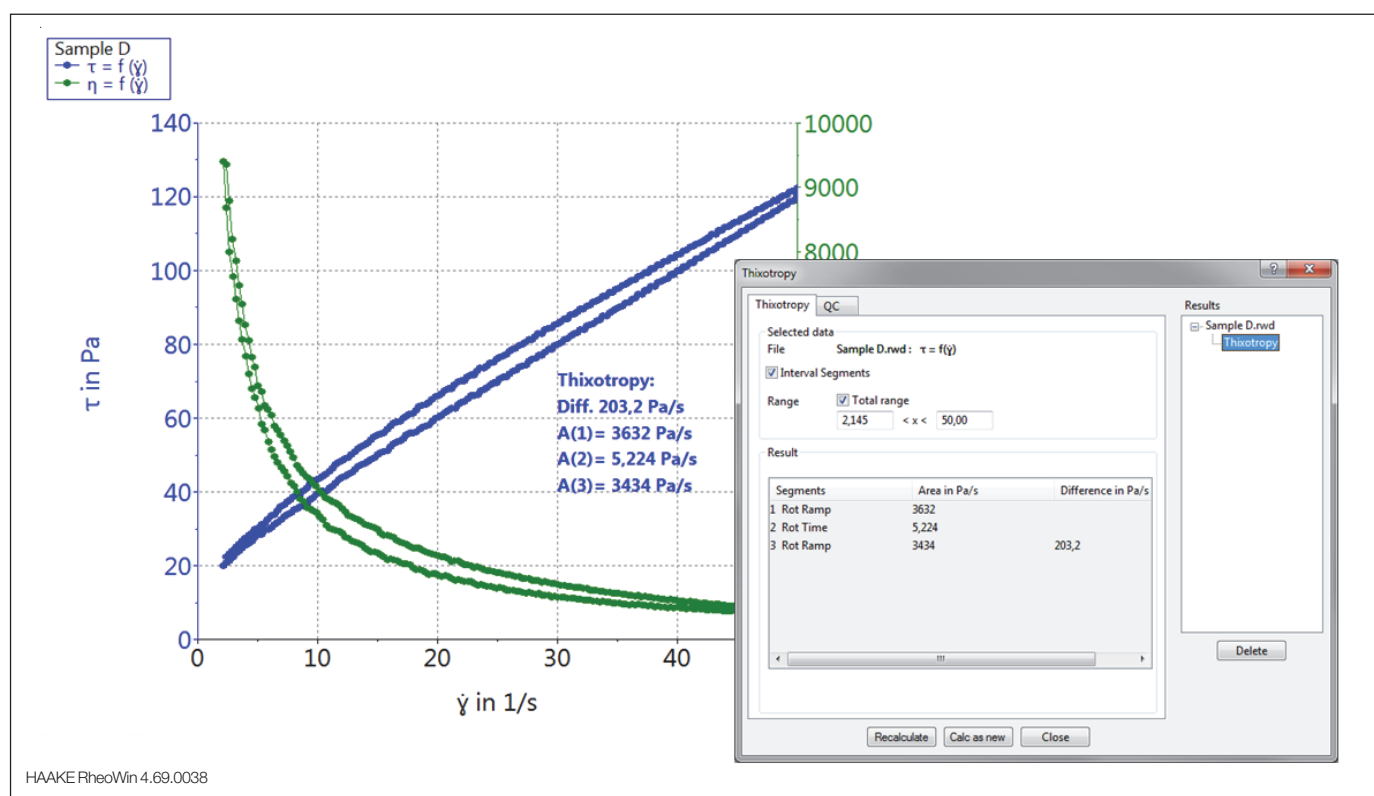


Fig. 8: Determination of the hysteresis area for shear stress data from a thixotropic loop test.

Yield stress

The yield stress of a sample can be determined by performing a shear stress ramp experiment where the deformation is recorded as a function of the increasing stress in a double logarithmic plot. At shear stresses below the yield stress, the deformation will increase linearly (slope of around 1 in a double logarithmic plot) with increasing shear stress. When the shear stress is approaching the yield stress the slope will increase and the sample will start to flow. The yield stress evaluation element in the HAAKE RheoWin software determines the yield stress by means of two tangents that are applied to the measuring curve. The intersection of these tangents

is interpreted as the yield stress of the sample (Fig. 9). QC criteria with a deviation tolerance can be defined.

Curve discussion

Two different elements for curve discussion are available in the HAAKE RheoWin software. With the regular curve discussion element minima, maxima, smallest, highest and mean values of a measurement curve can be determined (Fig. 10). With the advanced curve discussion element absolute and relative slopes, percentage of reference values as well as the intersections of tangents applied to the measured data can be calculated (Fig. 11). For both curve discussion elements quality control criteria can be defined.

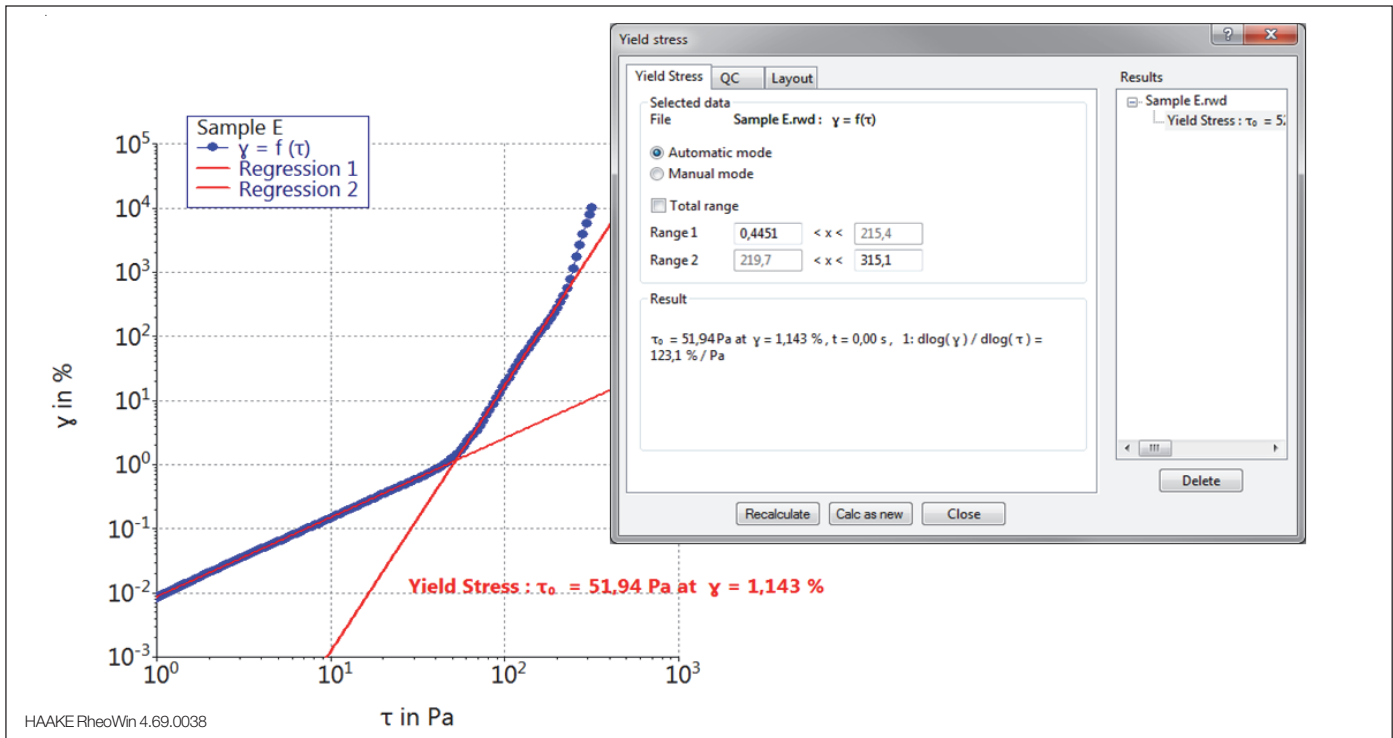


Fig. 9: Automatic determination of yield stress from shear stress ramp data with tangent intersection method.

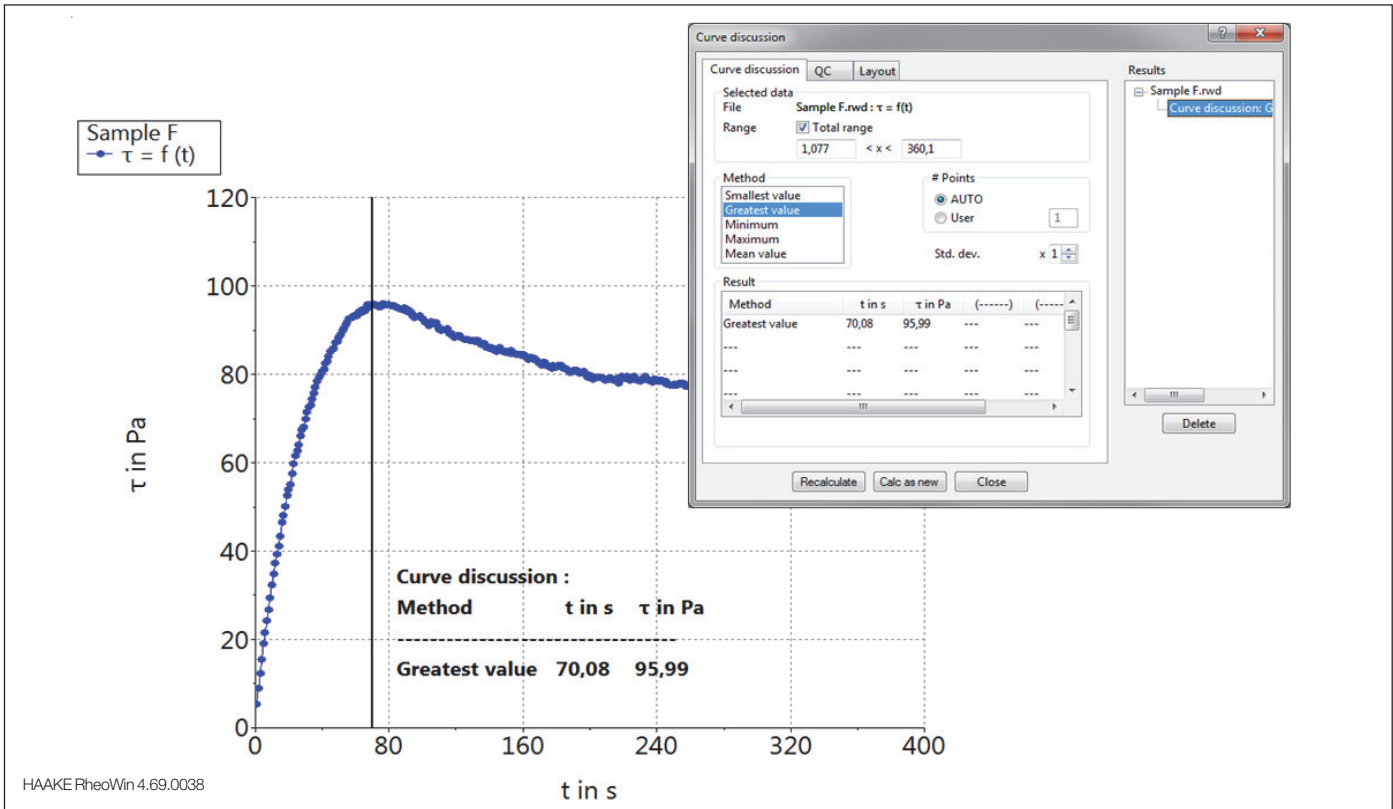


Fig. 10: Determination of the greatest value for the shear stress with the curve discussion evaluation element.

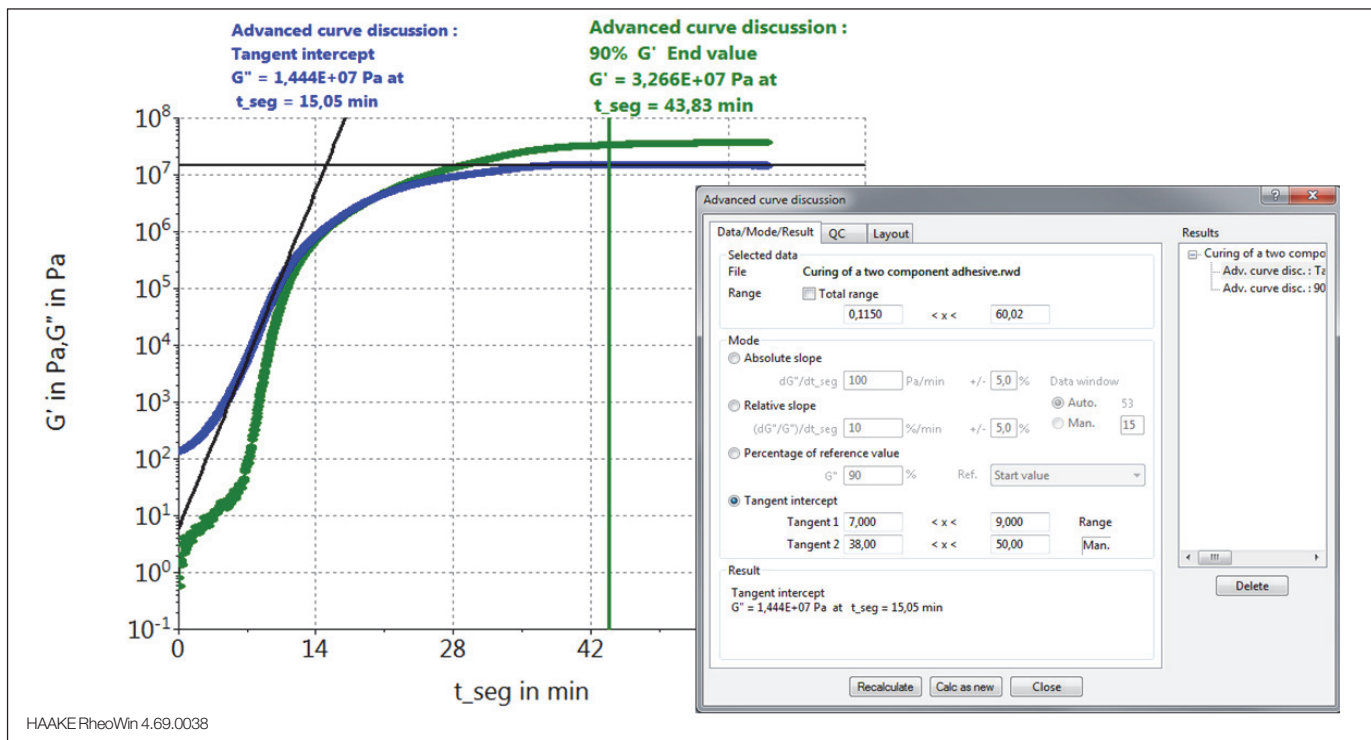


Fig. 11: Advanced curve discussion with G' and G'' data from a curing experiment.

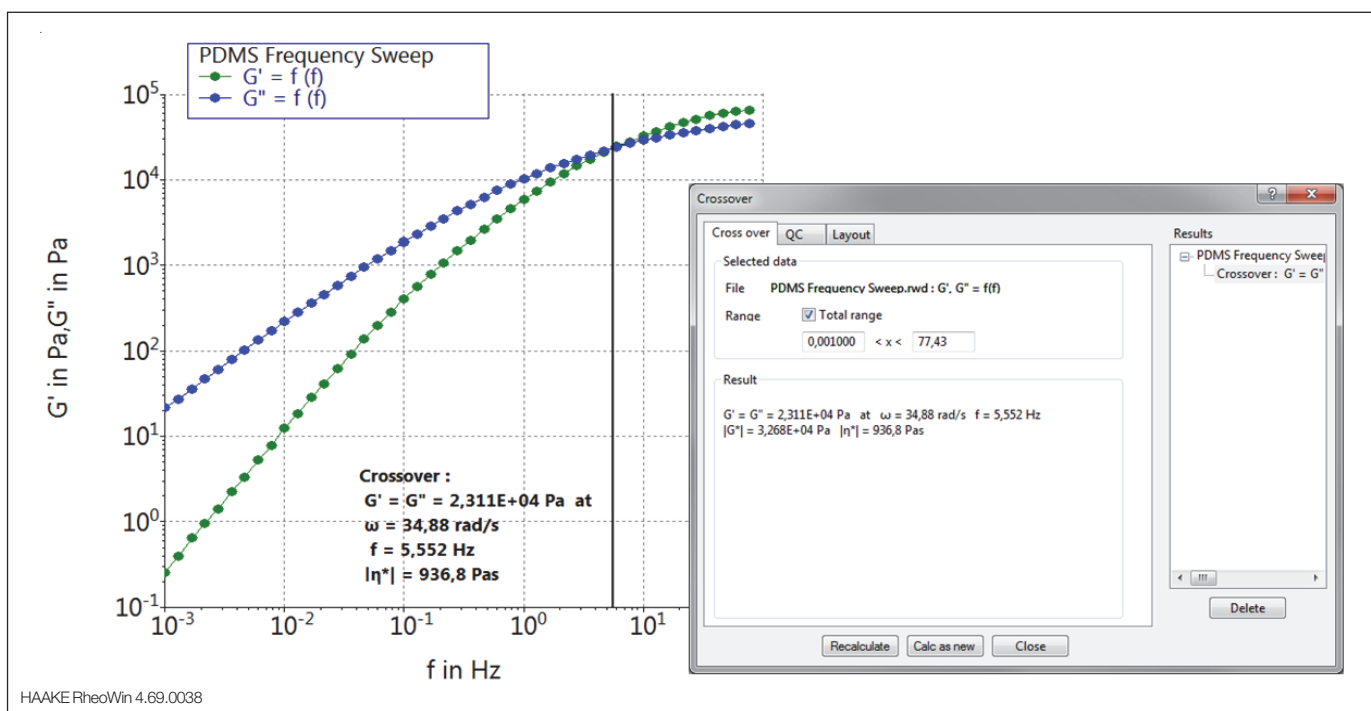


Fig. 12: Determination of the crossover of G' and G'' in a frequency sweep test.

Crossover

The crossover evaluation element determines the intersection point of the storage modulus G' and the loss modulus G'' curve from an oscillatory shear measurement. These include amplitude, frequency, time and temperature sweep experiments (Fig. 12). Quality control criteria with deviation tolerance can be defined.

Structure recovery

The structure recovery element provides information on how quickly and how well the structure of a sample recovers after it was exposed to a high shear rate. A structure recovery test consists of three steps. In an initial step the viscosity or complex viscosity of a material with an intact structure is measured as a reference. The second

step is a high shear rate period to break down the microstructure of the sample. In the third step the applied stress or strain signal is reduced to the initial value again and the recovery of the sample after a high shear impact is monitored. The structure recovery evaluation element of the HAAKE RheoWin software compares the data from the first and the third element to assess the recovery (Fig. 13). The evaluation options include the absolute change from the first to the end of the third step, relative recovery after a defined period of time and a relative recovery back to a defined percentage. Additionally, the time until the crossover of G' and G'' occurs (sample becomes predominantly elastic again) in the recovery step can be detected automatically.

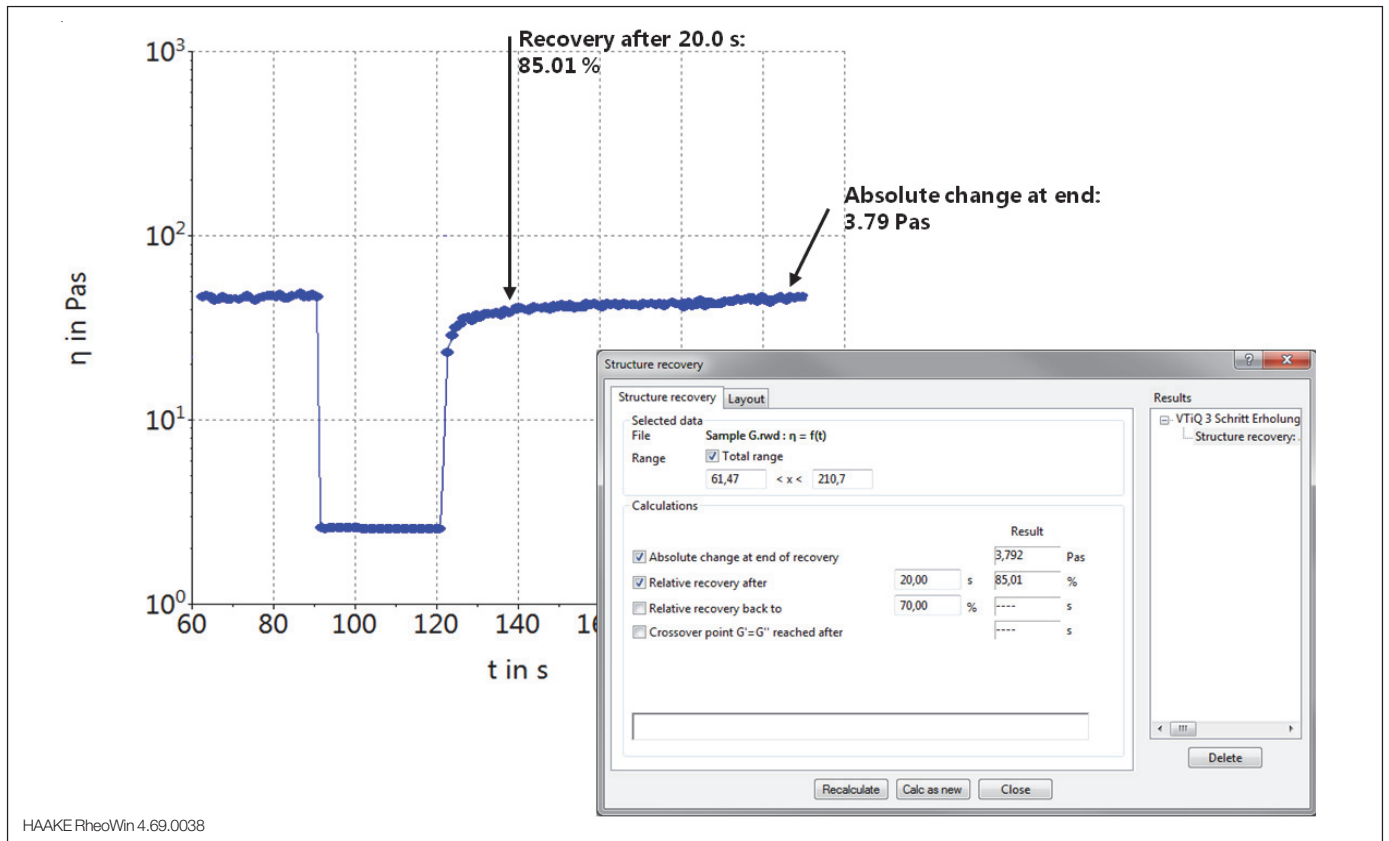


Fig. 13: Evaluation of the structure recovery after a sample was exposed to a high shear rate.

Creep recovery

Creep and recovery tests are the most direct way in rheology to qualify and quantify the elasticity of a material. The experiment is divided into two segments. During the first part, the creep, an instantaneous stress signal is applied to the sample for a defined period of time. In the second part, the stress is removed again and the recovery of the sample is monitored. The response of the sample is a deformation curve with a shape depending

on both, the amount of stress applied by the rheometer and the microstructure of the sample. The creep and recovery evaluation element allows for an automatic and comprehensive creep analysis (see Fig. 14). Parameters like the zero shear viscosity, recoverable deformation or equilibrium compliance can be extracted from the creep and recovery curves. Quality control criteria with deviation tolerance can be defined for the different evaluation parameters.

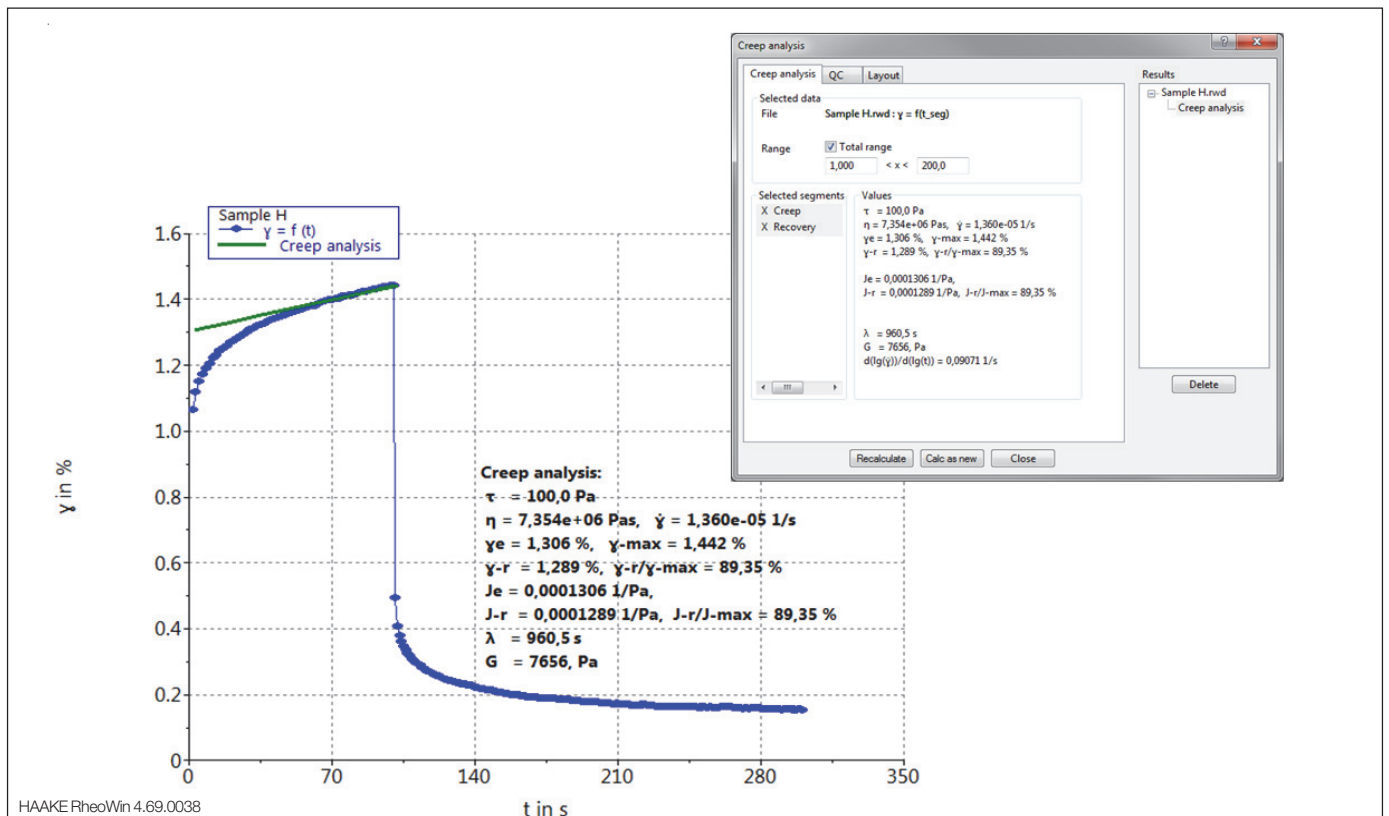


Fig. 14: Analysis of a creep and recovery test.

Summary

The HAAKE RheoWin software enables a comprehensive rheological characterization of liquid, semi-solid and solid materials. Users can quickly and easily create suitable measurement procedures for various products and applications. A broad range of data evaluation routines allow for an automatic data analysis and the integrated QC criteria for a convenient operation in Quality Control.

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The logo for ThermoFisher Scientific, with "ThermoFisher" in red and "SCIENTIFIC" in grey.

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