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WHITE PAPER

No. WP02

Better rheological results: Create SOPs that drive sound rheological measurements

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Rheological measurements play an important role in the quality control (QC) of many liquid and semi-solid materials. This includes final product evaluation as well as inspecting the properties of incoming raw materials or intermediates. To be fast and efficient, QC usually demands high sample throughput and a clear pass or fail assessment of the tested sample, regardless of the measurement technique used. To ensure smooth QC operation, the establishment of Standard Operating Procedures (SOPs) can be very helpful.

SOPs are written step-by-step instructions that provide guidance when performing routine operations. They ensure proper sample handling, correct instrument operation (even for untrained personnel) and sound data management and recording. A well-written SOP ensures fast sample assessment for quick product or batch release.

A comprehensive SOP for routine rheological measurements should encompass at least the following elements:

- Sample preparation and sample handling prior to loading samples into the rheometer
- Correct sample loading and sample treatment inside the rheometer prior to measurement
- Measurement procedure and data evaluation
- Report generation and data storage
- Preparation of the rheometer for the next measurement

A modern QC rheometer, together with its control software, provides useful tools and features that facilitate the establishment of a comprehensive SOP and failure-free instrument operation. These features, as well as the required elements listed above, are described in depth in the following paragraphs.

Selecting the right measuring geometry

Selecting the correct measuring geometry plays an important role in rheological measurements and is the first step toward successful measurement. The choices are manifold, and the ideal measuring geometry depends on the measuring range of the rheometer, the sample properties and the measurement parameters. A view of the different types of standard geometries is shown in Figure 1 while Table 1 provides information on what type of geometry can be used for different kinds of materials. More detailed information on how to select the right geometry can be found in our application note, “Well prepared – good results“ [1].

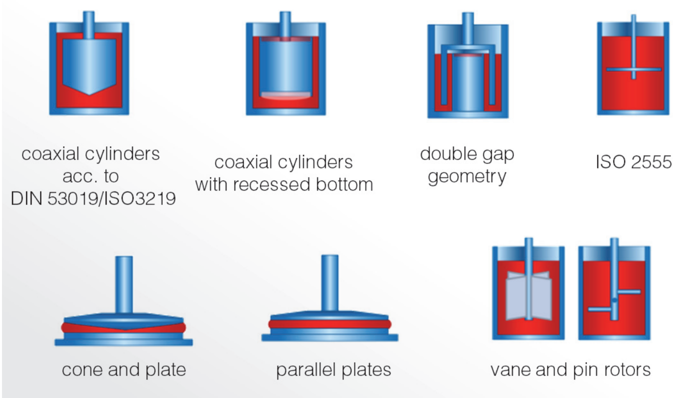

Fig. 1: Overview of standard measuring geometries.

Software tools such as the range calculator in Thermo Scientific™ HAAKE™ RheoWin™ software provide assistance for selecting the best measuring geometry for a specific rheological task (Figure 2). Depending on the viscosity of the tested material, this tool automatically calculates the optimal shear rate and shear stress range for any measuring geometry. If the operational range is too small, a different geometry should be selected.

For reproducible QC measurements, the same type of measuring geometry should always be used for a specific sample test procedure. The rheometer should give a feedback message to the operator if the wrong measuring geometry is attached. Ideally, a measurement procedure cannot be performed unless the correct measuring geometry is attached (Figure 3).

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	low viscous liquids	medium viscous liquids	high viscous liquids	weak semi-solids	gels	larger particles
cone and plate	✓	✓	✓	✓	✗	✗
parallel plates	✓	✓	✓	✓	✓	(✓)
coaxial cylinders	✓	✓	✗	✓	✗	(✓)
vane rotors	✗	✗	✗	✓	✗	✓

Table 1: Overview of measuring ranges for standard geometries.

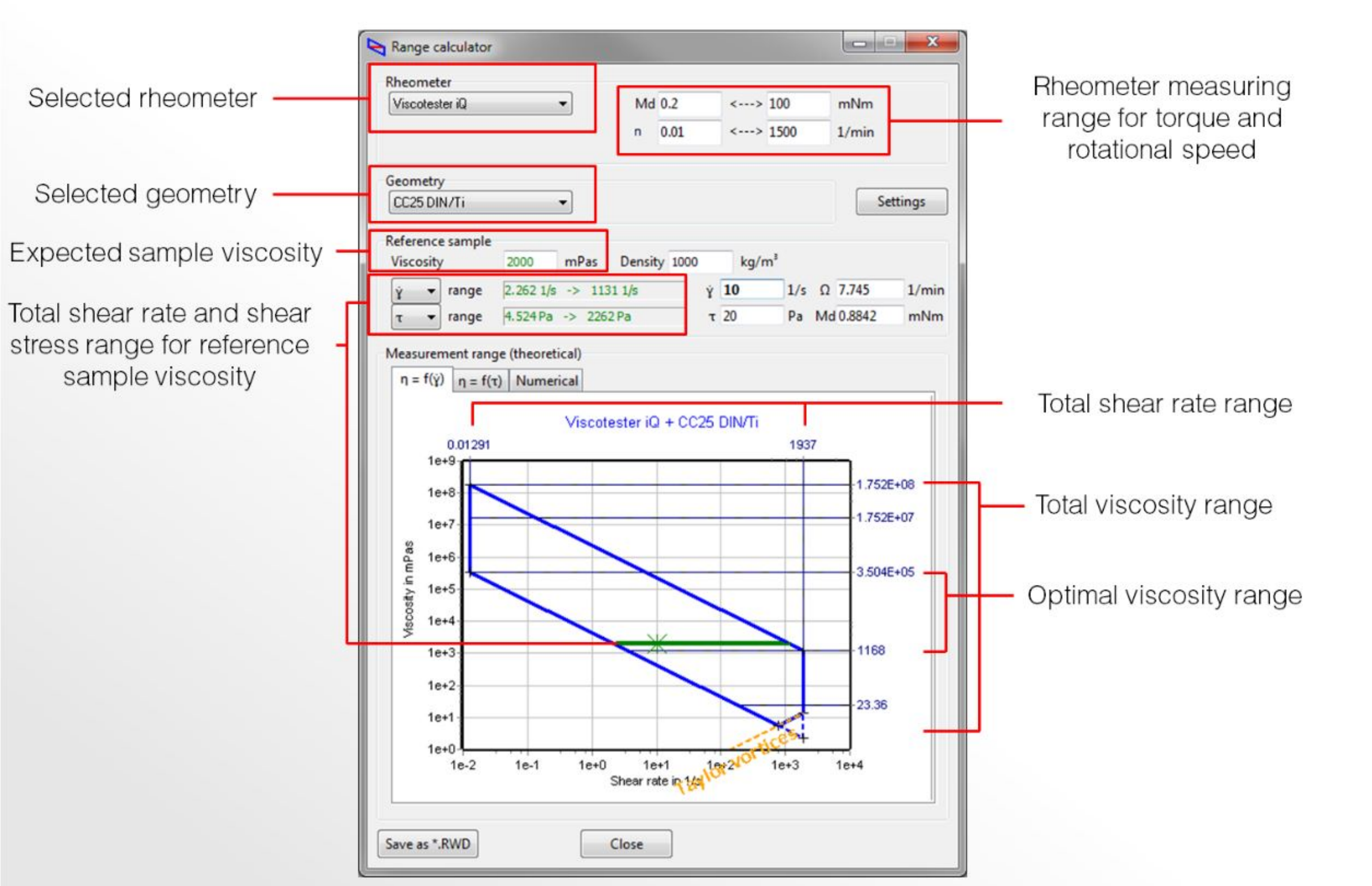

Fig. 2: HAAKE RheoWin software range calculator tool.

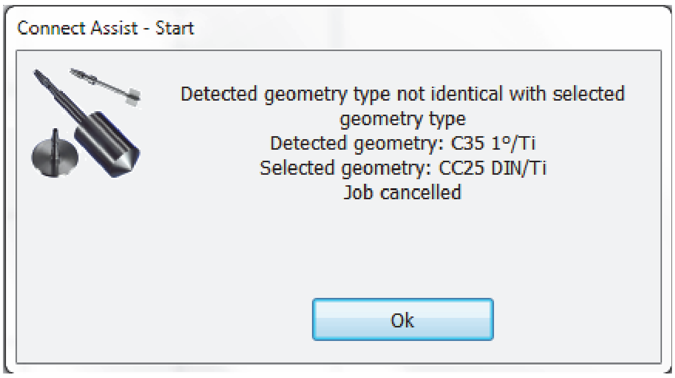

Fig. 3: HAAKE RheoWin software Connect Assist message.

Sample preparation and sample handling prior to loading samples into the rheometer

Before a sample can be tested with a rheometer certain pre-conditioning steps might be necessary to ensure reproducible and comparable results. For inhomogeneous materials that show a tendency toward phase separation, this can include, for instance, pre-measurement stirring or mixing steps. Another possibility is the preheating of the sample in a heating cabinet prior to loading into the rheometer. These steps should be defined in the SOP as precisely as possible to ensure that every sample is prepared for the measurement in exactly the same way.

Correct sample loading and treatment inside the rheometer

For highly thixotropic materials it is also important that all samples are loaded or filled into the measuring geometry in the same way and with the same tools. But correct sample loading also requires that the right amount of material is filled into the measuring geometry. Parallel plate and cone & plate geometries are usually overloaded first and then trimmed to the correct fill amount using a spatula or similar tool after the measurement gap is set.

Fig. 4: Level gauge for coaxial cylinder geometries.

Fig. 5: Fill Assist tool for HAAKE Viscotester iQ rheometer.

For coaxial cylinder geometries, accessories such as level gauges (Figure 4) or tools that measure the filling level by ultrasound (Figure 5) can be used to ensure that the correct sample volume is used. Underfilling as well as excessive overfilling should be avoided in any case. The rheometer software should provide feedback to the operator and guidance for correct sample loading. Figure 6 shows such a message that can be displayed during a rheological test procedure.

Once the sample is loaded into the rheometer geometry and the measuring gap is set correctly, the sample is usually given a defined period of time to release all internal stresses that might have been introduced during sample loading as well as to adjust to the measurement tempe-rature. The duration of these adjustment steps depends on the degree of thixotropy as well as the delta of sample temperature prior to sample loading and the actual test-ing temperature. For highly thixotropic materials the implementation of a pre-shear step followed by a defined resting period can improve the reproducibility of the tests and the comparability of the results.

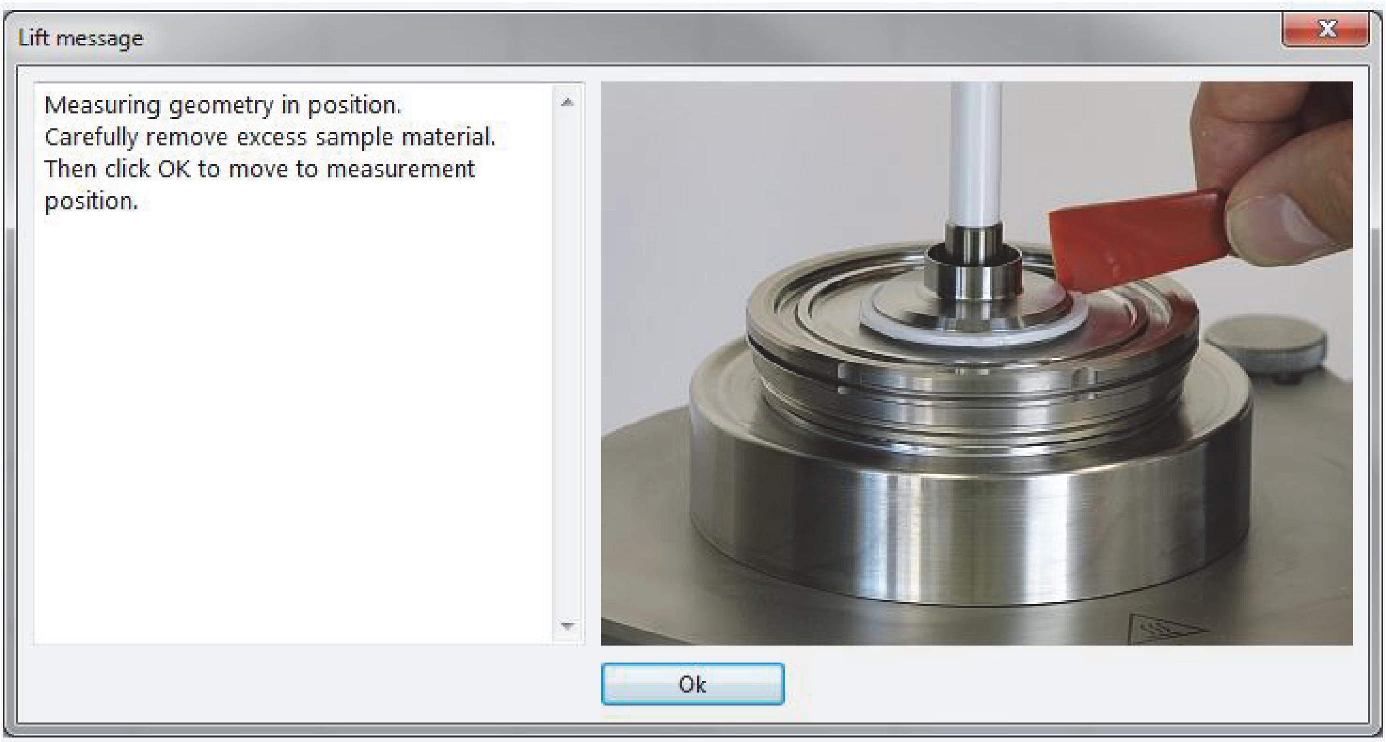

Fig. 6: HAAKE Rheowin software sample trimming message.

Measurement and data evaluation

Once the sample is fully relaxed and in a temperature equilibrium, the actual measurement steps follow. After the last data point has been collected, further data evaluation can be performed to extract QC relevant information from the initial results. This can include simple mathematical operation such as mean value determination or interpolation as well as the application of rheological models. A complete overview of the data evaluation capabilities of HAAKE RheoWin software can be found in our application note, HAAKE RheoWin software – features for quality control and routine measurements [2].

Report generation, data storage, preparation for next measurement

At the end of all data evaluation steps, the rheometer software should provide a clear assessment of the sample, ideally with a "pass or fail" classification as shown in Figure 7. The measurement procedure finishes with the generation of the automated measurement report and/or the data transfer into a laboratory information management system (LIMS) followed by instructions on how to prepare the rheometer for the next measurement. A complete measurement procedure that includes pre-measurement sample treatment, a thixotropic loop measurement procedure, automatic data evaluation and a report generation is shown in Figure 8.

Such a measurement procedure is usually established by the lab manager and executed by various operators.

The user management system in the rheometer software allows for setting up the system with different user levels and different privileges (Figure 9) [3]. This can prevent unwanted manipulation of the measurement procedure thereby ensuring trustworthy QC results.

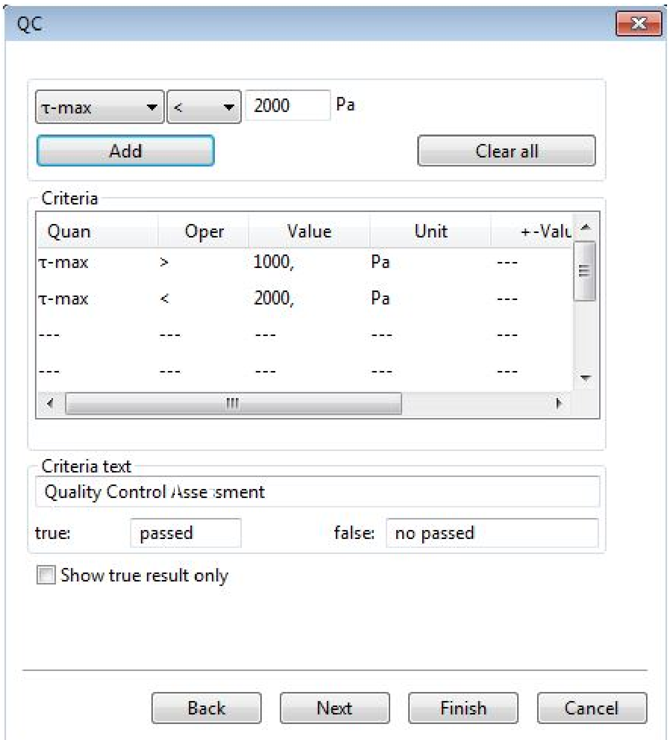

Fig. 7: Definition of QC criteria in HAAKE RheoWin software.

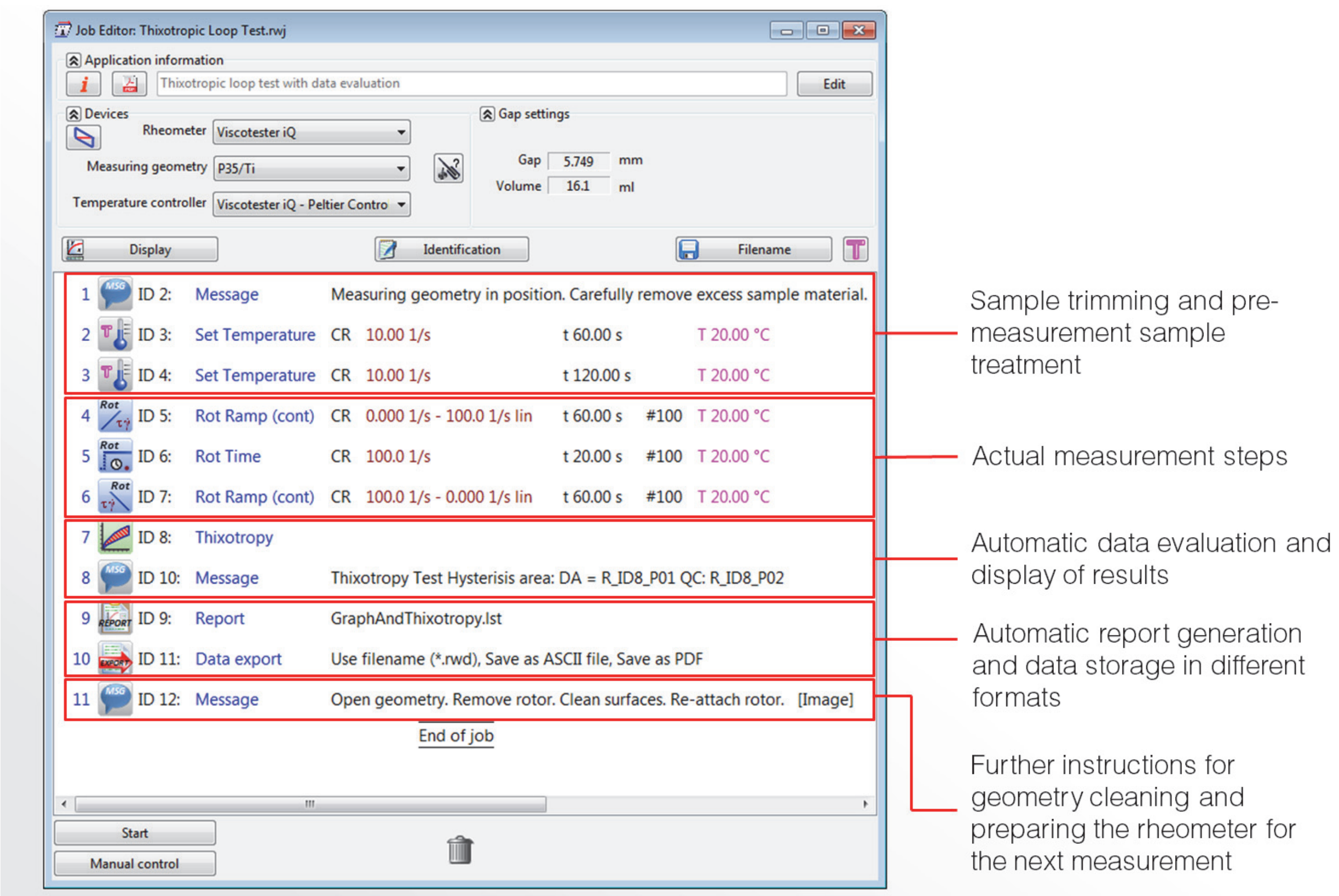

Fig. 8: Complete HAAKE RheoWin software test procedure.

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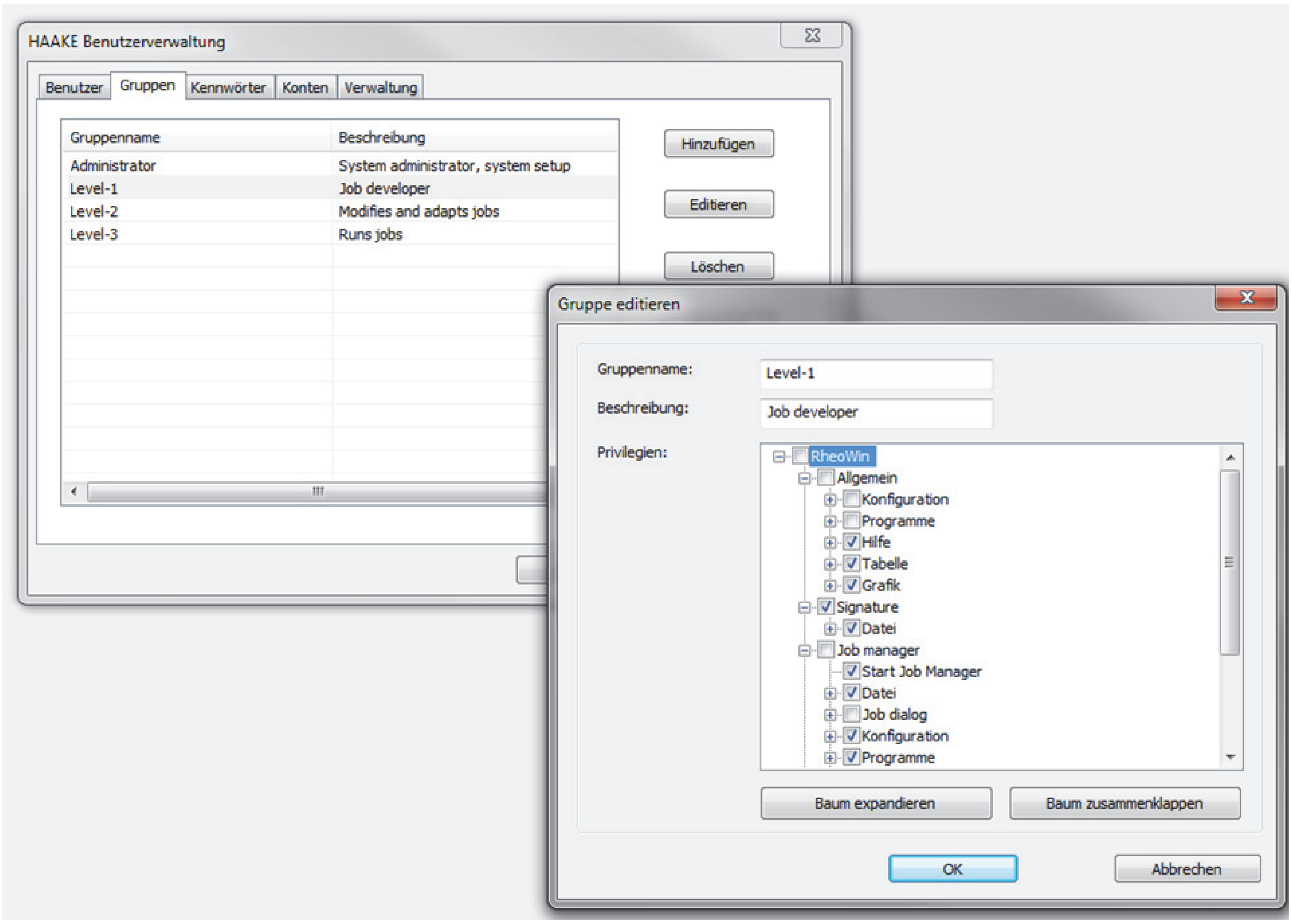

Fig. 9: HAAKE RheoWin software User Manager tool.

Conclusion

Standard Operation Procedures guarantee a smooth and identical QC operation with reliable data and a clear “pass or fail“ assessment of the sample even for untrained personnel.
Fast, efficient QC requires high sample throughput and a “pass or fail“ assessment that’s clear for all users. SOPs help ensure smooth, consistent QC operation when written comprehensively to include key elements: sample preparation and handling, sample loading, measurement and evaluation, reports and data storage, and preparation for future measurements. From automatically recognized geometries to feedback functionalities of the control software, modern rheometers have many tools to support the development of a comprehensive, effective SOP.

References

[1] Thermo Fisher Scientific Application note V248 “Well prepared-good results,“ Cornelia Küchenmeister-Lehrheuer and Klaus Oldörp

[2] Thermo Fisher Scientific Application note V223 “HAAKE RheoWin software - features for quality control and routine measurements,“ Fabian Meyer

[3] Thermo Fisher Scientific Product specifications “HAAKE RheoWin software 21 CFR part 11 tools“

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